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PLANT
PRODUCTION
AND PROTECTION
PAPER

222

Pesticide residues in food 2014

**Joint FAO/WHO Meeting
on Pesticide Residues**

**EVALUATIONS
2014**

PART I - RESIDUES

Pesticide residues in food 2014

Evaluations Part I - Residues

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Sponsored jointly by FAO and WHO

Joint meeting of the
FAO Panel of Experts on Pesticide Residues
in food and the Environment
and the
WHO Core Assessment Group
Rome, Italy 16-25 September 2014

WORLD HEALTH ORGANIZATION
FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS
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* New compound

** Evaluated within the periodic review programme of the Codex Committee on Pesticide Residues

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ABBREVIATIONS

ADI	acceptable daily intake
ae	acid equivalent
ai	active ingredient
AR	applied radioactivity
ARfD	acute reference dose
asp gr fn	aspirated grain fraction
AU	Australia
BAM	2,6-dichlorobenzamide
BBCH	B iologischen Bundesanstalt, B undessortenamt und C hemische Industrie
bw	body weight
CAC	Codex Alimentarius Commission
CAS	Chemical Abstracts Service
CCN	Codex classification number (for compounds or commodities)
CCPR	Codex Committee on Pesticide Residues
CEC	Cation exchange capacity
cGAP	Critical GAP
CPCA	cyclopropane carboxylic acid
CXL	Codex MRL
DAFT	Days after first treatment
DAP	days after planting
DAT	days after treatment
DC	Dispersible concentrate
DM	dry matter
DT ₅₀	time required for 50% dissipation of the initial concentration
dw	dry weight
2D-TLC	Two dimensional thin layer chromatography
ECD	electron capture detector
ETU	ethylene thiourea
EPO	early post-emergence
eq.	equivalent
EU	European Union
FAO	Food and Agriculture Organization of the United Nations
fw	fresh weight
GAP	good agricultural practice
GC	gas chromatography

GC-ECD	gas chromatography with electron capture detection
GC/MS	gas chromatography/mass spectrometry
GC/MSD	gas chromatography/mass selective detector
GC-NPD	gas chromatography coupled with nitrogen-phosphorus detector
GEMS/Food	Global Environment Monitoring System – Food Contamination Monitoring and Assessment Programme
GI	gastrointestinal
GLC	gas liquid chromatography
GLP	good laboratory practice
GPC	gel permeation chromatography
HPLC	high performance liquid chromatography
HR	highest residue in the edible portion of a commodity found in trials used to estimate a maximum residue level in the commodity
HR-P	highest residue in a processed commodity calculated by multiplying the HR of the raw commodity by the corresponding processing factor
IEDI	international estimated daily intake
IESTI	international estimate of short-term dietary intake
IPCS	International Programme on Chemical Safety
ISO	International Organization for Standardization
IUPAC	International Union of Pure and Applied Chemistry
JECFA	Joint FAO/WHO Expert Committee on Food Additives
JMPR	Joint FAO/WHO Meeting on Pesticide Residues
JP	Japan
LC	liquid chromatography
LC ₅₀	median lethal concentration
LD ₅₀	median lethal dose
LOAEL	lowest-observed-adverse-effect level
LOD	limit of detection
log P _{ow}	octanol-water partition coefficient
LOQ	limit of quantification
MTE	Mancozeb toxicity equivalents
MOA	mode of action
MRL	maximum residue limit
MS	mass spectrometry
MS/MS	tandem mass spectrometry
m/z	mass to charge ratio
ND	non-detect - below limit of detection
NMR	Nuclear magnetic resonance

OECD	Organisation for Economic Co-operation and Development
om	Organic matter in soil
OP	organophosphorus compound
PAI	Pure active ingredient
PAM	Pesticide Analytical Manual
PBI	plant back interval
Pf	processing factor
PH	pre-harvest
PHI	pre-harvest interval
ppm	parts per million
PRE	pre-emergence
r	correlation coefficient
r ²	coefficient of determination
RAC	raw agricultural commodity
RSD	relative standard deviation
RTI	re-treatment interval
SC	suspension concentrate
S _f	Scaling factor
SL	soluble liquid
SPE	solid phase extraction
STMR	supervised trials median residue
STMR-P	supervised trials median residue in a processed commodity calculated by multiplying the STMR of the raw commodity by the corresponding processing factor
SUA	Supply Utilisation Account
TAR	total administered radioactivity
TF	transfer factor
TGAI	Technical grade active ingredient
TLC	thin-layer chromatography
TRR	total radioactive residues
U	uniformly (labelled)
UK	United Kingdom
USA	United States of America
US/CAN	United States and Canada
USEPA	United States Environmental Protection Agency
UV-VIS	Absorption spectrometry in ultraviolet and visible part of the spectrum
%v/v	Percentage volume:volume
WG	wettable granule

WHO	World Health Organization
WP	wettable powder
w/w	Percentage weight:weight

USE OF JMPR REPORTS AND EVALUATIONS BY REGISTRATION AUTHORITIES

Most of the summaries and evaluations contained in this report are based on unpublished proprietary data submitted for use by JMPR in making its assessments. A registration authority should not grant a registration on the basis of an evaluation unless it has first received authorization for such use from the owner of the data submitted for the JMPR review or has received the data on which the summaries are based, either from the owner of the data or from a second party that has obtained permission from the owner of the data for this purpose.

INTRODUCTION

A Joint Meeting of the Food and Agriculture Organization of the United Nations (FAO) Panel of Experts on Pesticide Residues in Food and the Environment and the World Health Organization (WHO) Core Assessment Group on Pesticide Residues (JMPR) was held at FAO Headquarters, Rome (Italy), from 16 to 25 September 2014. The FAO Panel Members met in preparatory sessions on 11–15 September.

The Meeting was opened by Dr Clayton Campanhola, Director, Plant Production and Protection Division, FAO. On behalf of FAO and WHO, Dr Campanhola welcomed and thanked the participants for providing their expertise and for the significant time and effort devoted to such an important activity. He also expressed his sincere gratitude to the respective national authorities, institutes and organizations that have allowed their experts to contribute to the important work of the JMPR.

Dr Campanhola said that as the scientific advisory body for the Codex Committee on Pesticide Residues, the JMPR plays a central role in the establishment of global pesticide residue standards. For more than 50 years, the JMPR has been operating with two main objectives: firstly, risk assessment of pesticide residues and secondly scientific advice on the acceptable levels of pesticide residues in food and feed. Dr Campanhola further acknowledged that the deliberations of the JMPR were recognized as being authoritative, technically sound and provided an invaluable contribution to the collective efforts to provide safe food for consumers and to facilitate international trade. The growing demand for safe food and that agricultural production be sustainable underline now more than ever the continued relevance of the objectives underlying the work of the JMPR.

Given the FAO's goal of eradicating hunger Dr Campanhola observed that this could not be achieved without having safe food in the hands of consumers. And that it was essential that fair practices existed in food trade allowing all producers equitable access to markets. Consequently, the work of JMPR makes an important contribution to the global fight against hunger.

Dr Campanhola also acknowledged that the demand for JMPR evaluations, in support of Codex Maximum Residue Limits, reflected the importance accorded by member countries to the scientific assessments undertaken by the JMPR, and the growth in the international trade in agricultural products. Finally, Dr Campanhola addressed his best wishes to the Meeting on behalf of the two parent organizations.

During the meeting, the FAO Panel of Experts was responsible for reviewing residue and analytical aspects of the pesticides under consideration, including data on their metabolism, fate in the environment and use patterns, and for estimating the maximum levels of residues that might occur as a result of use of the pesticides according to good agricultural practice (GAP). Maximum residue levels and supervised trials median residue (STMR) values were estimated for commodities of animal origin. The WHO Core Assessment Group was responsible for reviewing toxicological and related data in order to establish acceptable daily intakes (ADIs) and acute reference doses (ARfDs), where necessary.

The Meeting evaluated 33 pesticides, including eight new compounds and three compounds that were re-evaluated within the periodic review programme of the Codex Committee on Pesticide Residues (CCPR), for toxicity or residues, or both.

The Meeting allocated ADIs and ARfDs, estimated maximum residue levels and recommended them for use by CCPR, and estimated STMR and highest residue (HR) levels as a basis for estimating dietary intake.

The Meeting also estimated the dietary intakes (both short-term and long-term) of the pesticides reviewed and, on this basis, performed dietary risk assessments in relation to their ADIs or ARfDs. Cases in which ADIs or ARfDs may be exceeded were clearly indicated in order to facilitate the decision-making process of CCPR. The rationale for methodologies for long- and short-term dietary risk assessment are described in detail in the FAO manual on the submission and evaluation of pesticide residue data for the estimation of maximum residue levels in food and feed (2009).

The Meeting considered a number of current issues related to the risk assessment of chemicals, the evaluation of pesticide residues and the procedures used to recommend maximum residue levels.

AMINOCYCLOPYRACHLOR (272)

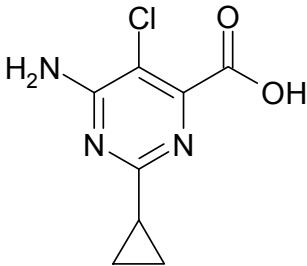
The first draft was prepared by Dr Dugald MacLachlan Australian Government Department of Agriculture, Fisheries and Forestry, Australia

EXPLANATION

Aminocyclopyrachlor is a new pyrimidine carboxylic acid herbicide used for the control of broadleaf weeds and woody vegetation. Aminocyclopyrachlor mimics the naturally occurring phytohormone indole acetic acid (auxin) disrupting plant growth. At the 45th Session of the CCPR (2013), it was scheduled for the evaluation as a new compound by 2014 JMPR.

The Meeting received information on the metabolism of aminocyclopyrachlor and also its methyl ester (aminocyclopyrachlor-methyl DPX-KJM44) in lactating goats and grass, methods of residue analysis, freezer storage stability, GAP information, supervised residue trials, and a cattle feeding study.

IDENTITY

Common name	Aminocyclopyrachlor
Chemical name	
IUPAC:	6-amino-5-chloro-2-cyclopropylpyrimidine-4-carboxylic acid
CAS:	6-Amino-5-chloro-2-cyclopropyl-4-pyrimidinecarboxylic acid
Manufacturer's code numbers:	DPX-MAT28
CAS number:	858956-08-8
CIPAC Code:	
Molecular formula:	C ₈ H ₈ ClN ₃ O ₂
Molecular mass:	213.62 g/mole
Structural formula:	

Formulations	Active ingredient content
SG	50% aminocyclopyrachlor
WG	39.5% aminocyclopyrachlor, 12.6% metsulfuron methyl
WG	35.3% aminocyclopyrachlor, 17.6% metsulfuron methyl

PHYSICAL AND CHEMICAL PROPERTIES*Specifications*

Specifications for aminocyclopyrachlor have not been developed by FAO.

Physical and chemical properties (pure aminocyclopyrachlor 99.7%)

Property	Results (method)	Reference
Appearance	Pure aminocyclopyrachlor is a white solid with a mild fruity odour	Shalini 2007 23196
Melting point	140.5 ± 0.1 °C	Shalini 2007 23196

Property	Results (method)	Reference
Relative density	1.4732 ± 0.0777	Shalini 2007 23196
pH	3.34 ± 0.01 for a 1% aqueous suspension	Moorthy 2011 22543 rev 1
Vapour pressure	6.9215 × 10 ⁻³ kPa (5.192 × 10 ⁻⁸ mm Hg) at 20 °C	Moorthy 2007a 22537
Henry's Law constant (Pa/m ³ /mol at 20 °C)	5.24 × 10 ⁻⁷ (Milli-RO-water) 4.71 × 10 ⁻⁷ at pH 4 3.51 × 10 ⁻⁷ at pH 7 3.82 × 10 ⁻⁷ at pH 9	Hirata 2008 22545
Solubility in water at 20 °C	2.81 ± 0.1 g/L Milli-Q® water 3.13 ± 0.26 g/L pH 4 4.20 ± 0.14 g/L pH 7 3.87 ± 0.17 g/L pH 9	Moorthy 2007b 22541
Solubility in organic solvents (at 20 °C) (g/L)	Methanol 36.747 ± 0.281 Ethyl acetate 2.008 ± 0.002 n-octanol 1.945 ± 0.027 acetone 0.960 ± 0.007 acetonitrile 0.651 ± 0.003 o-xylene 0.005 ± 0.000 n-hexane (9.7 ± 2.1) × 10 ⁻⁶ dichloromethane 0.235 ± 0.003	Anand 2007 22542
Partition coefficient n-octanol/water (20 ± 1 °C)	log K _{ow} -1.12 ± 0.04 Milli-Q® water log K _{ow} -1.01 ± 0.01 pH 4 log K _{ow} -2.48 ± 0.02 pH 7	Manjunatha 2007 22544
Hydrolysis	Stable at pH 4, 7 and 9 and at 25 °C	Manjunatha 2008 22116
Photolysis	Half-life: 7.3 days at pH 4, 25 °C and 1.3 days in sterile natural spring water	Lowrie 2008 22117 rev 1
Quantum yield	Φ = 2.5521 × 10 ⁻⁴ molecules degraded/photon	Lowrie 2008 22117 rev. 1
Dissociation constant	pK _a 4.65 ± 0.04 at 20 ± 1 °C	Anand 2011a 22555 rev 1
Stability	Aminocyclopyrachlor was tested for stability at normal (ambient) and elevated (54 ± 2 °C) temperatures and in the presence of metal and metal ions for a period of 14 days. The results of the test show that the aminocyclopyrachlor is stable to normal and elevated temperatures and metal and metal ions	Yogeesha 2011 22539 rev 1

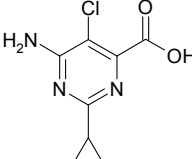
Technical grade material (aminocyclopyrachlor 92.2%)

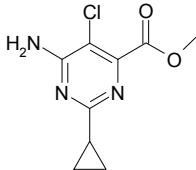
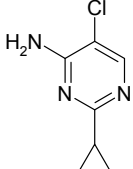
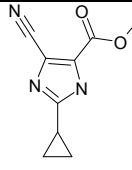
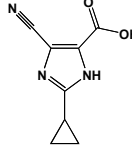
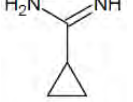
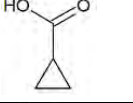
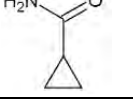
Property	Results (method)	Reference
Melting point	138.9 ± 0.1 °C	Anand 2011b 22551 rev 1
Density at 20 °C	1.3476 ± 0.0813 g/mL	Anand 2011b 22551 rev 1

METABOLISM AND ENVIRONMENTAL FATE

Metabolites are given various abbreviations and code numbers in the studies. Structures and abbreviations and codes are shown below.

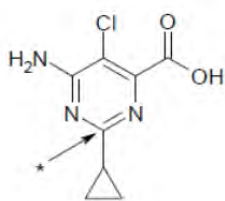
Degradation compounds from metabolism of aminocyclopyrachlor in plants, animals, soil, or water

Compound Name	Structure	Found in:
Aminocyclopyrachlor 6-Amino-5-chloro-2-cyclopropyl-4-pyrimidinecarboxylic acid CAS Number: 858956-08-8 DPX-MAT28 MW 213.62		grass, goat, rotational crops, soil

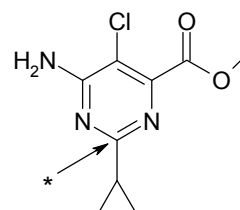
Compound Name	Structure	Found in:
Aminocyclopyrachlor-methyl Methyl 6-amino-5-chloro-2-cyclopropyl-4-pyrimidinecarboxylate CAS Number: 858954-83-3 DPX-KJM44, DPX-MAT28 methyl ester MW 227.65		
5-Chloro-2-cyclopropyl-pyrimidin-4-ylamine IN-LXT69 MW 169.61		Grass, goat, photolysis, rotational crops, soil
5-Cyano-2-cyclopropyl-3H-imidazole-4-carboxylic acid methyl ester IN-QGC48 MW 191.19		grass
5-Cyano-2-cyclopropyl-3H-imidazole-4-carboxylic acid IN-QFH57 MW 177.16		Photolysis, grass Structural formulae changed
Cyclopropane carbamidine IN-YY905		Photolysis
Cyclopropane carboxylic acid CAS NO.: 1759-53-1 IN-V0977		Photolysis, grass
Cyclopropane carboxamide CAS No.: 6228-73-5 IN-Q3007		Photolysis, grass

Animal metabolism

The Meeting received studies on the metabolism of DPX-KJM44 (aminocyclopyrachlor-methyl) in rats and lactating goats. The metabolism of aminocyclopyrachlor in animals was investigated using [^{14}C]-DPX-KJM44 while in plants studies were available for metabolism of [^{14}C]aminocyclopyrachlor and [^{14}C]-DPX-KJM44. The structural formula and the positions of the ^{14}C label are shown below. The studies on rats were evaluated by the WHO Core Assessment Group.



[pyrimidine-2- ^{14}C]aminocyclopyrachlor



[pyrimidine-2- ^{14}C]DPX-KJM44

Figure 1 Label positions of aminocyclopyrachlor and DPX-KJM44: marked as *

The identification of residue components in the animal and plant metabolism studies was achieved using authentic standards of the compounds involved.

Laboratory animal (rat) studies

Metabolism of aminocyclopyrachlor-methyl (DPX-KJM44) in rats was evaluated by the WHO Core Assessment Group of the 2014 JMPR. The metabolic conversion of DPX-KJM44 to aminocyclopyrachlor by plasma esterases is rapid (*in vitro* plasma $t_{1/2}$ approximately 5 minutes). As a result, administration of DPX-KJM44 to animal models will result in systemic exposures to primarily aminocyclopyrachlor. Both aminocyclopyrachlor and DPX-KJM44 have comparable pharmacokinetic profiles. Each is rapidly absorbed, and rapidly and essentially completely eliminated. For both substances the terminal compound in laboratory animals is aminocyclopyrachlor.

Lactating goat

Melville and Vance (2009 22837) studied the metabolism of DPX-KMJ44 in a lactating goat (British Saanen $\times$, 51.3 kg bw; 2.0 kg milk/d) that was dosed orally via gelatin capsule twice daily with ring labelled [^{14}C] DPX-KMJ44 for 5 consecutive days at the equivalent of 97 ppm in the feed (feed consumption 1.45–1.73 kg DM/d). Doses were administered following milking (ca. 8:00 and 20:00 hours). Milk was collected twice daily and urine and faeces were collected daily. The goat was sacrificed approximately 6 h after the administration of the final dose and tissues taken post-mortem for quantification and analysis of radioactivity.

Composite liver, kidney, muscle, and fat samples were ground to a powder in dry ice and initially extracted with buffered acetonitrile:water (9:1, v/v). The extracted liver and kidney samples were digested with pepsin and protease to liberate additional ^{14}C -residues. Milk samples were initially partitioned with hexane and subsequently extracted with acetone. Aliquots of the extracts were analysed using liquid scintillation counting (LSC) while the post-extraction solids were dried under nitrogen, and triplicate portions were analysed using combustion and LSC.

Radioactive residues in the various extracts from each matrix were analysed by HPLC, using a C_{18} column with a mobile phase gradient of 10 mM ammonium formate and methanol. Radioactive residues were quantified by LSC analysis of the collected HPLC fractions. Radiolabelled components identified by HPLC and/or TLC co-chromatography with authentic unlabelled reference standards were later confirmed in selected samples using mass spectrometry.

All samples were extracted and analysed within 8 months of frozen storage. Reanalysis of liver and kidney extracts, stored for 11 months, showed no significant changes in the metabolite profile indicating metabolites in the extracts were stable to frozen storage.

The total recovery of dosed radioactivity was 85% with the majority of the radioactivity excreted in urine (54%) and faeces (20%). Approximately 8% remained in the contents of the gastrointestinal tract and a further 3.1% was found in the cage wash.

TRR in milk samples increased during the dosing period and reached a maximum of 0.031 mg equiv/kg in the milk collected at Day 5 of the study. Acetone extractions recovered ca. 80.5% of the total milk [^{14}C] residues. Aminocyclopyrachlor accounted for 15.9% TRR (0.004 mg equiv/kg). Other milk components accounting for 0.002 to 0.007 mg equiv/kg were not identified. Composite sample of milk from days 1 to 5 was separated into skim milk and cream with the majority of ^{14}C located in the cream fraction (0.016 mg equiv/kg skim milk, 0.12 mg equiv/kg cream). Residues in skim milk and cream were too low for identification.

Radioactive residues in tissues ranged from 0.01 mg equiv/kg in the omental fat to 1.67 mg equiv/kg in the kidneys. Good extractability was achieved for the milk and tissue samples with greater than 80% TRR recovered in solvent extracts (except omental fat = 47.4%). Residues remaining after solvent extraction of liver and kidney were subject to sequential treatment with pepsin and protease enzymes liberating an additional 1 to 11.6% of the TRR.

The identified metabolites in the tissue and milk samples are summarised in Table 1.

Table 1 Identification of radioactivity in tissue and milk of goats dosed orally with DPX-KJM44

Matrix	Milk ^a	Liver	Kidney	Muscle	Renal Fat	Omental fat	SC fat
TRR (mg equiv/kg)	0.023	0.30	1.67	0.042	0.016	0.010	0.026
			%TRR				
Solvent extracted	80.5	86.7	98.3	87.5	83.8	47.4	80.7
Aminocyclopyrachlor	15.9	55.6 ^b	55.3 ^c	43.3	83.8	47.4	80.7
unknowns	64.6 ^d	ND	ND	32.7 ^e	ND	–	–
Pepsin/protease digest	NC	11.6	1.0	NC	NC	NC	NC
Aminocyclopyrachlor		10.1	NA				
Unextracted	19.5	1.7	0.7	12.5	16.2	52.6	19.3
Losses during processing	–	29.3 ^f	43.0 ^f	11.5	–	–	–

^a composite day 1–5 d sample

^b Total characterised as aminocyclopyrachlor by TLC (concentrated extract prior to SPE clean-up) was 87.5% TRR (0.26 mg equiv/kg); confirming aminocyclopyrachlor as the only component present

^c Total characterised as aminocyclopyrachlor by TLC (concentrated extract prior to SPE clean-up) was 93.2% TRR (1.56 mg equiv/kg); confirming aminocyclopyrachlor as the only component present

^d Three components ranging from 0.002–0.007 mg equiv/kg

^e Two components ranging from 0.006–0.008 mg equiv/kg

^f Concentrated muscle and liver solvent extracts required SPE clean-up prior to HPLC analysis; efforts to improve low recoveries following SPE clean-up were unsuccessful.

ND = Not detected

NC = Not conducted

NA = Not analysed

Solvent extraction of liver recovered 86.7% TRR (0.259 mg equiv/kg). The concentrated solvent extract was subjected to SPE clean-up which resulted in losses such that the concentrated cleaned extract accounted for 57.4% TRR. Pepsin and protease digestion of the liver PES liberated a further 11.6% TRR (0.034 mg equiv/kg). Unextracted liver residues were 1.7%TRR (0.006 mg equiv/kg).

Aminocyclopyrachlor was the major extractable component in liver accounting for 55.6% TRR (0.167 mg equiv/kg) in the SPE cleaned solvent extract by HPLC. Aminocyclopyrachlor was the only component observed by TLC analysis of the concentrated extract prior to SPE clean-up accounting for 87.5% TRR (0.262 mg equiv/kg). A single radiolabelled component was detected in the pepsin digest which co-chromatographed with aminocyclopyrachlor and accounted for 10.1% TRR (0.030 mg equiv/kg).

Total liver residues characterised as aminocyclopyrachlor by a combination of TLC (concentrated extract prior to SPE clean-up) and HPLC of the pepsin digest was 97.6% TRR (0.292 mg equiv/kg).

Solvent extraction of kidney recovered 98.3% TRR (1.64 mg equiv/kg). The concentrated solvent extract was subjected to SPE clean-up which resulted in losses such that the concentrated cleaned extract accounted for 55.3% TRR. Pepsin and protease digestion of the kidney PES only liberated an additional 1.0% TRR (0.017 mg equiv/kg) and were not analysed further. Unextracted kidney residues were 0.7% TRR (0.011 mg equiv/kg). Aminocyclopyrachlor was the major extractable component in kidney accounting for 55.3% TRR (0.925 mg equiv/kg) in the SPE cleaned solvent extract by HPLC. Aminocyclopyrachlor was the only component observed by TLC analysis of the concentrated extract prior to SPE clean-up accounting for 93.2% TRR (1.560 mg equiv/kg). Total kidney residues characterised as aminocyclopyrachlor by TLC (concentrated extract prior to SPE clean-up) was 93.2% TRR (1.56 mg equiv/kg).

Solvent extraction of muscle recovered 87.5% TRR (0.037 mg equiv/kg). The HPLC profile of the concentrated muscle extract showed that aminocyclopyrachlor was the major component accounting for 43.3% TRR (0.018 mg equiv/kg). Two minor components accounted for 14.4% TRR (0.006 mg equiv/kg) and 18.3% TRR (0.008 mg equiv/kg) were not identified. Unextracted muscle residues were 12.5% TRR (0.005 mg equiv/kg).

Solvent extraction of the three fat samples ranged from 47.4–83.8% TRR with lower extractability in omental fat. The HPLC profile of the renal, omental, and subcutaneous fat extracts showed that aminocyclopyrachlor was the major extractable component accounting for 83.8% TRR (0.013 mg equiv/kg), 47.4% TRR (0.005 mg equiv/kg), and 80.7% TRR (0.021 mg equiv/kg), respectively. Unextracted fat residues were ≤ 0.005 mg equiv/kg.

In summary, DPX-KJM44 was rapidly metabolised and eliminated as aminocyclopyrachlor, primarily in the excreta (74% of the administered total dose). Identified residues in tissues were exclusively aminocyclopyrachlor. A proposed metabolic pathway for DPX-KJM44 in the lactating goat is presented in Figure 2.

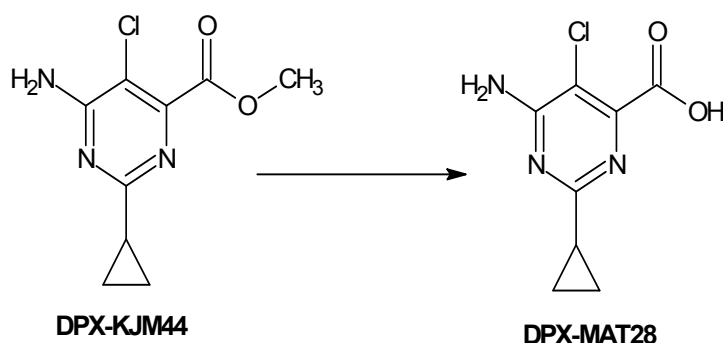


Figure 2 Metabolism of DPX-KJM44 and aminocyclopyrachlor (DPX-MAT28) in lactating goats

Plant metabolism

The Meeting received plant metabolism studies with DPX-KJM44 on grass, aminocyclopyrachlor on grass and DPX-KJM44 and aminocyclopyrachlor on a variety of weeds. When applied to plants, the methyl esters of aminocyclopyrachlor (DPX-KJM44) is quickly broken down to release free acid which is readily translocated throughout the plants.

Grass

Lowrie (2009, 22393) studied the metabolism of [^{14}C]-DPX-KJM44 in grass (Amenity mix: 30% Disco perennial ryegrass, 30% Franklin strong creeping red fescue, and 40% Vienna perennial ryegrass) grown under field conditions. A single application at a nominal rate of 373 g DPX-KJM44/ha (spray volume 1000 L/ha) of a WP formulation was made to a 1 m² plot of grass growing in a sandy loam soil (67.6% sand, 15.1% silt, 17.4% clay; pH 6.5; 5.8% organic matter; 17.5 cmol/kg CEC). At the time of application the grass was approximately 30 cm in height. The grass plot was enclosed during the experiment. Grass samples were collected at 0, 3, 7, 14, 30 and 60 days after application and were separately homogenised and radioactivity determined by combustion/LSC. The identity of DPX-KJM44, aminocyclopyrachlor, and the various grass metabolites were investigated by chromatographic (HPLC) comparison of the extracted residues with authentic reference standards of DPX-KJM44, aminocyclopyrachlor, IN YY905, IN V0977, IN Q3007, IN LXT69, IN QFH57, and IN QGC48. All samples were stored frozen (ca. -20°C). Analyses of ^{14}C residues were completed within 4 months.

The grass foliage collected at each sampling point was gently washed with acetonitrile to remove the surface residues. The rinsed tissues were homogenised and portions of plant tissue were extracted twice with acetonitrile:0.2% formic acid in water (80:20, v/v; ca 150 mL) then twice with acetonitrile:0.2% formic acid in water (1:1, v/v; ca 150 mL). Levels of radioactivity in the wash and extract samples were determined by liquid scintillation counting (LSC). The unextracted radioactivity in the post extraction solids (PES) was determined by combustion analysis followed by LSC. Total

radioactive residues in each sample were determined by summing radioactivity in the surface wash, extracts and post extraction solids.

Unextracted ^{14}C residues in PES of tissues harvested 0, 7, and 60 DAT were subject to enzymatic and chemical hydrolysis. Post-extraction solids were incubated twice with a 2% solution of α -amylase in 0.2 M sodium phosphate buffer (pH 7 at 50 °C for 72 hours). After the second incubation with α -amylase, the remaining pellet was incubated with a mixture of 0.5% amyloglucosidase and 2% cellulase in 0.2 M sodium acetate buffer (pH 5 at 50 °C for 48 hours). Following each incubation the solution was separated from the pellet by centrifugation followed by decantation. The pellet remaining following enzyme hydrolysis was sequentially incubated with sodium hydroxide (0.1 N, 60 °C, 6 hours) and hydrochloric acid (1 M, 60 °C, 6 hours). The hydrolysates were separated from the remaining solids on each occasion by centrifugation and decanting.

Surface wash, solvent extracts, enzyme digests and hydrolysates were analysed using reversed phase HPLC. Radioactivity in collected fractions was quantified by LSC. LC-MS was conducted to confirm the presence of DPX-KMJ44, aminocyclopyrachlor and related metabolites.

Table 2 Characterisation of ^{14}C residues in grass following application of DPX-KJM44

				%TRR		
Days after application	TRR (mg equiv DPX-KJM44/kg)	Surface wash and extracted				Unextracted
		Surface wash	Extract 1	Extract 2	Total	
0	15.6	13.0	76.6	8.6	98.2	1.8
3	15.4	10.3	76.2	8.3	94.8	5.2
7	12.0	5.8	75.8	10.4	92.0	7.9
14	5.9	3.1	70.3	12.0	85.4	14.6
30	4.1	4.5	57.2	11.3	73.0	27.0
60	2.4	1.2	35.4	11.6	48.2	51.7

Table 3 Identification of ^{14}C residues in grass following application of DPX-KJM44

Days after application	0	3	7	14	30	60
TRR (mg equiv DPX-KJM44/kg)	15.6	15.4	12.0	5.9	4.1	2.4
%TRR						
Extracted	99.4 ^a	94.8	95.3 ^a	85.4	73.0	76.7 ^a
IN-Q3007	< 0.1	< 0.1	0.1	0.2	0.2	1.3
IN-V0977	ND	ND	ND	ND	ND	0.5
Aminocyclopyrachlor	64.2	67.7	64.4	50.5	53.9	32.9
IN-QFH57	< 0.1	0.5	0.6	1.0	2.0	1.9
IN-LXT69	4.9	4.2	4.8	6.1	4.7	5.6
IN-QGC48	< 0.1	1.1	0.5	ND	4.1	0.6
DPX-KJM44	24.7	13.7	5.9	8.6	6.5	8.6
Unextracted	0.6	5.2	4.7	14.6	27.0	23.4
Total identified/characterised	93.8	87.5	77.1	67.4	72.7	56.2

^a Figures differ from Table 2 as it also includes material released from enzyme digestion and acid/base hydrolysis of post-extraction solids

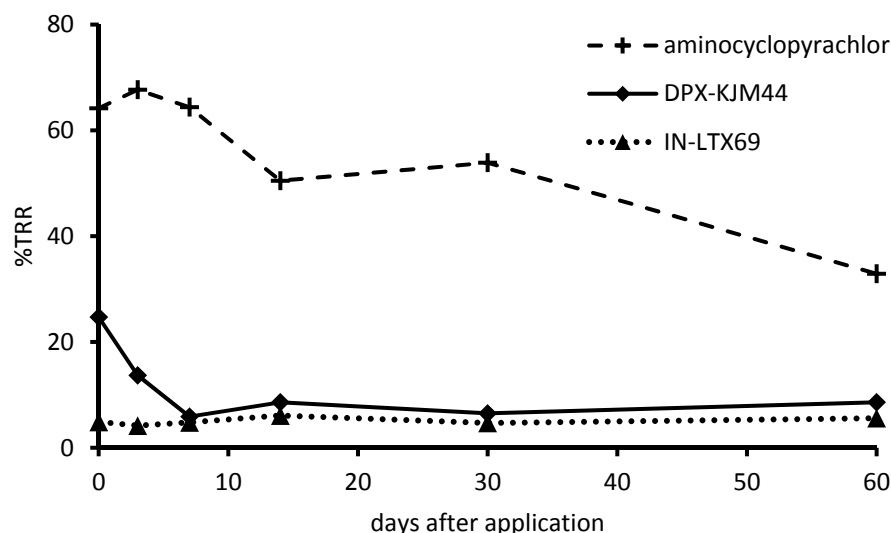


Figure 3 Plot of DPX-KJM44, aminocyclopyrachlor and IN-LTX69 residues in grass following foliar application of DPX-KJM44

Total surface wash and tissue extracted residues in grass ranged from 15.3 to 1.18 mg equiv/kg (98.2 to 48.3% TRR) at 0 DAT to 60 DAT, respectively. The unextracted residue accounted for 0.28 and 1.27 mg equiv/kg (1.8 and 51.7% TRR, respectively) at 0 DAT and 60 DAT, respectively. Only a very small amount of the TRR was released from PES by enzyme and chemical hydrolysis (1.2%) at 0 days after application; 28.5% of the TRR was released from PES by enzyme and chemical hydrolysis at 60 days after application.

TRR in grass harvested immediately following application was 15.6 mg equiv/kg. Acetonitrile surface washes removed 13.0% TRR (2.0 mg equiv/kg). Solvent extracted residues accounted for 85.2% TRR (13.3 mg equiv/kg). Unextracted residues were further investigated by enzyme 1 (amylase), enzyme 2 (a mixture of amyloglucosidase and cellulose), base and acid hydrolysis, resulting in the liberation of 0.7% TRR (0.11 mg equiv/kg), 0.3% TRR (0.041 mg equiv/kg), 0.1% TRR (0.016 mg equiv/kg), and 0.1% TRR (0.013 mg equiv/kg), respectively. Unextracted residues remaining following enzyme/chemical hydrolysis of PES accounted for 0.6% TRR (0.102 mg equiv/kg).

The identification of extracted residues was confirmed by LC-MS including several minor components including IN Q3007, IN V0977, IN QFH57, IN LXT69, and IN QGC48. Aminocyclopyrachlor (32.9 to 64.2%TRR) was the principal extracted residue in the grass samples with DPX-KJM44 (5.9 to 24.7%TRR) present at lower levels. IN-LXT69, IN V0977, IN Q3007, IN QFH57, and IN QGC48 were identified as minor grass components that individually accounted for no greater than 6.1% TRR (0.14 to 0.77 mg equiv/kg; Table 3).

Lewis *et al.* (2013) studied the absorption and translocation of ^{14}C -aminocyclopyrachlor in tall fescue (*Lolium arundinaceum* (Schreb.) SJ Darbyshire, cv. Confederate) following application to a tiller leaf. Plants were grown to the 5 to 7 leaf stage (single tiller) and the third leaf from the crown was covered with aluminium foil and the plants sprayed with commercially formulated aminocyclopyrachlor at 79 g ai/ha with the addition of 0.25% non-ionic surfactant (NIS). The foil was removed and five 1 μL droplets of ^{14}C -aminocyclopyrachlor and 0.25% NIS were applied to the previously covered leaf. Plants were harvested 3, 12, 24, 48, 96 and 192 hours after application and separated into ^{14}C -treated leaf, remaining above ground foliage, crown and roots. The treated leaf was washed with 50% v/v methanol:water to remove surface residues. Similarly, roots were washed with methanol:water to remove root exudates.

Absorption of ^{14}C - aminocyclopyrachlor was high accounting for 63% (100%-leaf wash %) of the applied ^{14}C by 24 hours after application (Table 4). Translocation of absorbed ^{14}C was observed as evidenced by ^{14}C accumulation in the untreated foliage and in the roots. Accumulation of ^{14}C -radioactivity in other foliage ranged from 25 to 41% of the absorbed ^{14}C and for roots from 22 to 27% at 24 to 72 hours after treatment. Metabolism was not observed with 100% of ^{14}C in solvent extracts present as aminocyclopyrachlor at 192 hours after application.

Table 4 Distribution of ^{14}C following foliar application of ^{14}C -aminocyclopyrachlor to tall fescue

Hours after application	Leaf wash	^{14}C -treated leaf	Other foliage	%applied ^{14}C Crown	Roots	Root exudates	Total recovery
3	49	25	5	4	0	3	86
12	38	35	11	4	0	1	89
24	37	41	14	3	0	0	95
48	32	35	21	2	1	8	99
96	29	30	34	2	1	1	97
192	26	26	25	1	2	3	83

Following application to grass, DPX-KMJ44 was rapidly metabolised (demethylated) to form aminocyclopyrachlor. Aminocyclopyrachlor was the principal radiolabelled component found in grass samples collected immediately after treatment and up to 60 days after treatment. Aminocyclopyrachlor was decarboxylated to a lesser extent to IN-LXT69. Other minor routes of metabolism included photo-induced simultaneous elimination of hydrogen chloride and pyrimidine ring contraction yielding the imidazole-nitriles, IN-QGC48 and IN-QFH57 (from DPX-KJM44 and aminocyclopyrachlor, respectively), and pyrimidine ring opening with subsequent oxidation providing the amide IN-Q3007. Formation of the carboxylate, IN V0977 could occur through photolysis and is a minor pathway or it could be an artefact of the extraction process as hydrolysis experiments on IN-Q3007 under alkaline and acidic conditions showed that IN V0977 is produced under conditions comparable to exhaustive extraction procedures employed in the grass metabolism study (Brown 2011 31316).

The proposed metabolic pathway for DPX-KMJ44 and aminocyclopyrachlor in grass is provided in Figure 4.

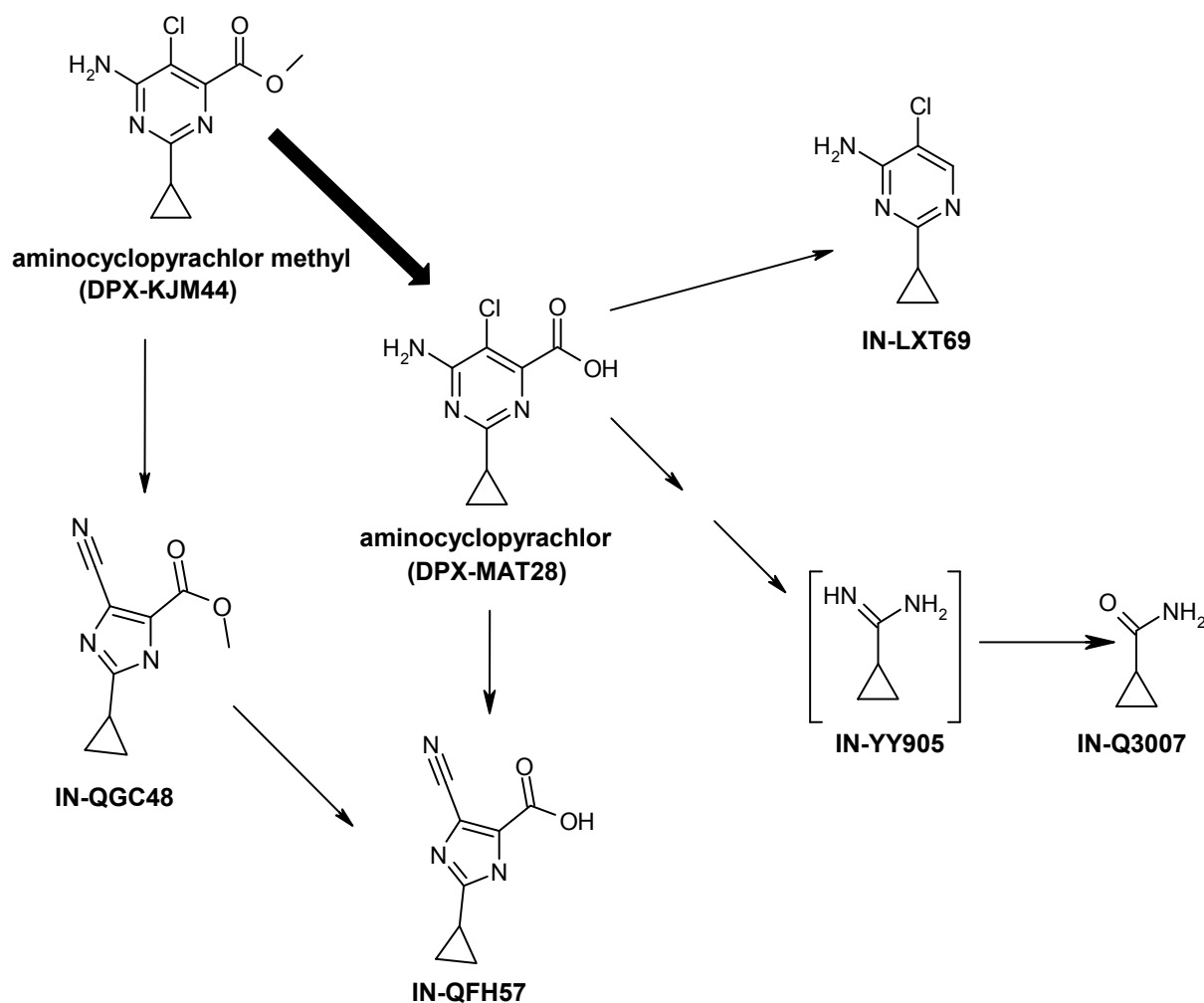


Figure 4 Metabolic pathway of DPX-KJM44 and aminocyclopyrachlor following foliar application to grass

Studies on weeds

A number of literature studies have investigated the absorption, translocation and metabolism of aminocyclopyrachlor and DPX-KJM44 in various weed species.

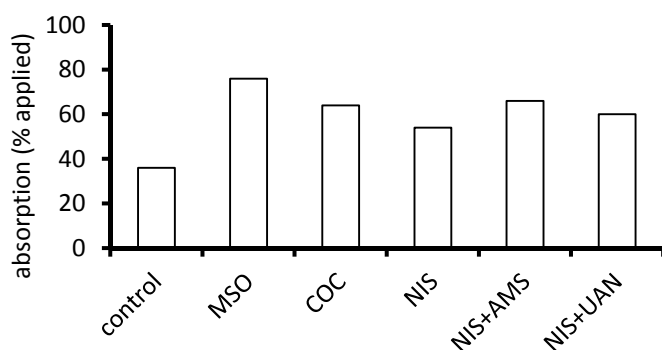
Bukun *et al.* (2010) studied absorption and translocation of both aminocyclopyrachlor and DPX-KJM44 in Canada thistle (*Cirsium arvense*). Plants at the five- to six-rossette stage were selected and the youngest fully expanded leaf on each plant was covered with aluminium foil. The plants were sprayed with formulations containing aminocyclopyrachlor or DPX-KJM44 at the equivalent of 0.14 kg ai/ha together with 1% v/v methylated seed oil (MSO). Following spray application the protected leaves were exposed and treated with formulations containing ^{14}C -herbicide. Plants were harvested at 24, 48, 96 and 192 hours after application and separated into ^{14}C -treated leaf, and above and below ground tissue. In studies on metabolism, plants were harvested at 0, 2, 6 and 24 hours after application with residues analysed by HPLC.

Metabolism of ^{14}C - DPX-KJM44 was exclusively to the free acid (aminocyclopyrachlor). By 2 hours after application, 65% of the ^{14}C in plant extracts was aminocyclopyrachlor and by 6 hours after treatment 82% of the absorbed DPX-KJM44 had been metabolised to aminocyclopyrachlor.

The effect of adjuvants on absorption was studied in a separate experiment where [^{14}C]aminocyclopyrachlor or [^{14}C]DPX-KJM44 were formulated in treatment solutions containing methylated seed oil (MSO), crop oil concentrate (COC), non-ionic surfactant (NIS), NIS plus ammonium sulphate (AMS) or NIS plus urea ammonium nitrate (UAN). Solutions were 1% MSO (v/v), 1% COC (v/v), 0.25% NIS (v/v), 0.25% NIS (v/v) plus 0.2% AMS (w/v) and 0.25% NIS (v/v) plus 1% UAN (v/v) combined with formulated product to provide a concentration when applied to leaves that was equivalent to application at 0.14 kg ai/ha. Leaves were harvested 96 hours after application and surface residues measured by washing the leaves with 90% water, 10% methanol and 0.25% NIS. Quantitation of radioactivity was by liquid scintillation spectroscopy. The fraction absorbed was determined as the difference between applied radioactivity and radioactivity in the surface wash divided by applied radioactivity.

Absorption of both DPX-KJM44 and aminocyclopyrachlor was maximised in formulations containing methylated seed oil (MSO). While absorption of DPX-KJM44 by leaves was greater than for aminocyclopyrachlor, once absorbed DPX-KJM44 is rapidly metabolised to aminocyclopyrachlor and translocation from the application site to other parts of the plant was almost identical for the two formulations.

A aminocyclopyrachlor



B DPX-KJM44

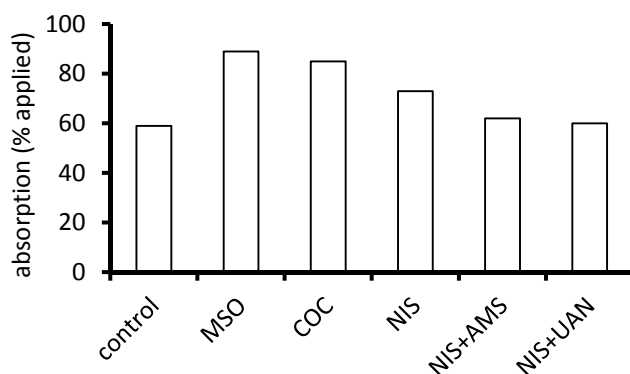


Figure 5 Effect of adjuvants on absorption of A aminocyclopyrachlor and B DPX-KJM44. MSO = methylated seed oil, COC = crop oil concentrate, NIS = non-ionic surfactant, NIS+AMS = NIS + ammonium sulphate, NIS+UAN = NIS + urea ammonium nitrate

Lindenmayer (2012) studied the absorption, translocation and metabolism of aminocyclopyrachlor in field bindweed (*Convolvulus arvensis*). Field bindweed plants were grown from seed, pruned to maintain a single shoot and prevent flowering. When plants reached 12–15 leaf

stage, and vined to a height of 30 cm on a wooden stake, a single leaf midway up the stem was covered by aluminium foil and the plant sprayed with aminocyclopyrachlor (with 1% v/v MSO) at the equivalent of 0.14 kg ai/ha and a spray volume equivalent to 187 L/ha. Following the spray application, the aluminium foil was removed and the protected leaves were treated with a solution of [^{14}C]aminocyclopyrachlor plus 1% MSO v/v. Plants were harvested at 0, 6, 12, 24, 48, 96 and 192 hours after application and separated into ^{14}C -treated leaves, above-ground tissues, below ground tissue.

To determine absorption, treated leaves were washed with 90% water, 10% methanol and 0.25% NIS and the wash solution analysed by liquid scintillation spectroscopy. Radioactivity in distant plant parts was extracted by homogenising with 90% methanol, centrifuging and decanting the extract for LSC. Samples of the extracts were also subject to HPLC to determine metabolites.

Absorption of ^{14}C was rapid, reaching a maximum of 48% of the applied radioactivity by 48 hours after application. Translocation to above and below ground plant parts increased with time after application and reached 14% of the applied radioactivity by 192 hours after application. No soluble metabolites of aminocyclopyrachlor were detected in the plant extracts suggesting limited or no metabolism of aminocyclopyrachlor within 192 hours of application.

The results for Canada thistle and field bindweed described above are similar to those reported by Bell *et al.* (2011) who studied the absorption, translocation and metabolism of DPX-KJM44 and aminocyclopyrachlor on yellow star thistle (*Centaurea solstitialis* L.), rush skeleton weed (*Chondrilla juncea* L.) and prickly lettuce (*Lactuca serriola* L.). Following application of DPX-KJM44 there was rapid formation of the free acid (aminocyclopyrachlor) with a half-life of 3.5 hours and no subsequent metabolism in yellow starthistle, rush skeleton weed and prickly lettuce. Proportional herbicide movement between species was similar, with the majority translocating to developing shoots. However, in rush skeleton weed, early translocation was directed to root tissue. Highly sensitive species such as prickly lettuce absorb and translocate less material than relatively less sensitive species such as rush skeleton weed. De-esterification of DPX-KJM44 appeared to delay translocation of the resulting aminocyclopyrachlor in yellow star thistle and rush skeleton weed.

Vargas *et al.* (2014) studied the absorption, translocation, and metabolism of ^{14}C -DPX-KJM44 in large crabgrass (*Digitaria sanguinalis* (L.) Scop) and black nightshade (*Solanum nigrum* L) applied with and without diflufenzopyr (35 g ai/ha). Diflufenzopyr had minimal effects on translocation of radioactivity in either species. Accumulation of radioactivity in above ground parts of black nightshade was greater than, or equal to, that in large crabgrass by 72 h after treatment. In both species, metabolism of ^{14}C -DPX-KJM44 was rapid such that 60 to 78% of the extracted radioactivity was present as aminocyclopyrachlor at 8 hours after application.

Lym (2014) studied the absorption and translocation of aminocyclopyrachlor in leafy spurge (*Euphorbia esula*) and yellow toadflax (*Linaria vulgaris*). Application of [^{14}C]aminocyclopyrachlor was made approximately 8 weeks after planting and when plants were 10–12 cm tall. The application was to a single leaf midway on the stem of each plant. Samples were collected at 24, 48, 96 and 192 hours after application with plants separated into treated leaf, leaves above and leaves below the treated leaf as well as roots. In both weed species absorption was rapid averaging 63 and 71% of the applied ^{14}C in yellow toadflax and leafy spurge respectively at 24 hours after application. By 96 hours after application, 28 and 16.5% of the applied ^{14}C for yellow toadflax and leafy spurge respectively had translocated to other above ground plant parts. Relatively low levels of ^{14}C translocated to the roots of both weeds (< 12% applied ^{14}C).

In summary, DPX-KJM44 and aminocyclopyrachlor are rapidly absorbed and translocated by plants. Absorption is improved by the addition of spray adjuvants such as MSO or NIS. Following administration of DPX-KJM44 to plants, DPX-KJM44 is rapidly metabolised to aminocyclopyrachlor which is the major component of the residue. Following administration of aminocyclopyrachlor to plants, aminocyclopyrachlor is the major component of the residue.

ENVIRONMENTAL FATE

The FAO Manual on the Submission and Evaluation of Pesticide Residues Data for the Estimation of Maximum Residue Levels in Food and Feed (2009) explains the data requirements for studies of environmental fate. The focus should be on those aspects that are most relevant to MRL setting. For aminocyclopyrachlor, supervised residue trials data are available for grass. Aerobic degradation in soil is relevant, as well as the normal requirements for hydrolysis, photolysis and rotational crop studies.

The Meeting received information on soil aerobic metabolism and soil photolysis properties of aminocyclopyrachlor and DPX-KJM44. Studies were also received on the behaviour of [^{14}C]-DPX-KJM44 in a confined rotational crop situation.

Aminocyclopyrachlor residues are persistent in soils. Aminocyclopyrachlor residues in soils resulting from recommended uses could contribute to the residues in succeeding crops.

Aerobic degradation in soil

The rate and route of degradation of [^{14}C]-DPX-KJM44 was investigated in a sandy loam soil (Sassafras, Maryland USA, pH 5.4, 2.1% OM) under aerobic conditions by Hirata *et al.* (2011, 22435). [^{14}C]-DPX-KJM44 was applied at a nominal rate equivalent to a single application of 0.336 kg ai/ha. The soil samples were incubated under aerobic conditions in the laboratory and maintained under moist, dark conditions at $20 \pm 2^\circ\text{C}$ for up to 360 days. Samples were extracted twice with 0.2% formic acid in water:acetonitrile (20:80 v:v). For samples from day 40 onwards, an additional extraction was conducted using 0.15M ammonium acetate:acetonitrile (30:70 v:v). This extraction step was repeated to demonstrate exhaustive extraction.

The mean total recoveries of radioactivity for the 0 to 360 DAT soil samples were between 91.8 and 93.7% of applied radioactivity (AR). By 360 days, 20.4 to 25.8% of the AR was mineralised to $^{14}\text{CO}_2$ with 46.4–46.6% AR extracted with the solvent systems used and 21.4 to 25.0% remaining unextracted.

DPX-KJM44 was rapidly degraded, accounting for less than 1% AR by Day 3 as it was converted to aminocyclopyrachlor which accounted for 91.0–91.4% AR. By day 360, aminocyclopyrachlor accounted for 42.8–43.1% AR. The only other product observed was IN-LXT69 which reached a maximum between days seven and 122 accounting for 2.8–3.1% AR and declining to 2.2–2.4% AR by day 360.

The rate of degradation was estimated using single first-order (SFO) kinetics. The DT_{50} and DT_{90} values obtained are presented in Table 5.

Table 5 Summary of DT_{50} for DPX-KJM44 and aminocyclopyrachlor in Sassafras sandy loam (20°C in the dark)

Soil: Sassafras sandy loam	DT_{50} (days)	DT_{90} (days)	r^2
DPX-KJM44	0.1	0.2	0.9999
aminocyclopyrachlor	275	912	0.9492

The proposed degradation pathway of DPX-KJM44 in soil is presented in Figure 6 and involves the rapid de-methylation of DPX-KJM44 to form aminocyclopyrachlor which then undergoes de-carboxylation to form IN-LXT69. Both $^{14}\text{CO}_2$ and bound residue are significant degradation products of DPX-KJM44 in soil.

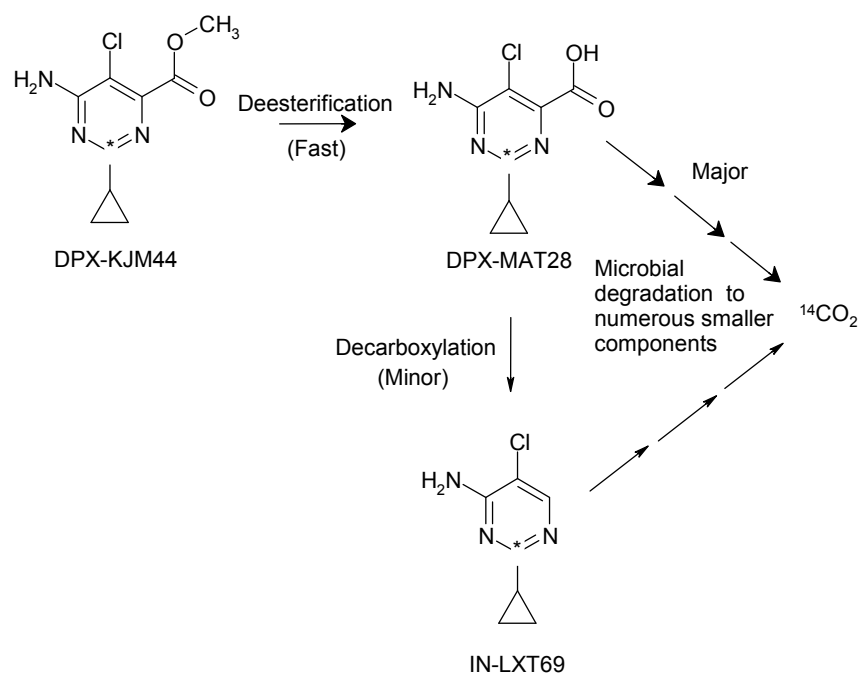


Figure 6 Proposed degradation pathway for DPX-KJM44 in soil under aerobic conditions

Manjunatha (2010, 22119) studied the degradation of aminocyclopyrachlor in three soils maintained under aerobic conditions in the dark at a nominal temperature of $20 \pm 2^\circ\text{C}$ for 120 days. The moisture content of the soils was adjusted to between 40 and 60% maximum water holding capacity and as close as possible to 75% water holding capacity at 1.033 MPa. Relevant properties of the soils used are presented in Table 6.

Table 6 Properties of soils used to study aerobic degradation of aminocyclopyrachlor

Soil Name	Texture	Origin	pH Water	0.01 M CaCl_2	% Organic Matter Walkley-Black	Soil initial	biomass $\mu\text{g/g}$ final
Nambsheim	Sandy Loam	Europe	8.09	7.48	2.2	1394	4796
Tama	Silty Clay	USA	6.45	6.13	4.6	650	4234
Drummer	Clay Loam	USA	6.40	5.79	6.5	284	4181

[Pyrimidine-2- ^{14}C] aminocyclopyrachlor was applied at a nominal rate of 0.448 mg/kg dry soil. Samples were analysed after 0, 7, 15, 30, 60, 90 and 120 days incubation. Residues in samples, other than day 0, were sequentially extracted using: (1) phosphate buffer (pH 7.4); (2) and (3) acetonitrile: pH 7.4 phosphate buffer 70:30 v/v; 4 acetonitrile: The extraction for the day 0 Drummer soil sample omitted the acetonitrile:ammonium acetate extraction. The extraction for the day 0 Nambsheim and Tama samples utilised: 1 $2\times$ acetonitrile: 0.2% (v/v) formic acid 80:20 v/v; 2 acetonitrile: 0.15 M ammonium acetate 70:30 v/v; 3 acetonitrile: pH 7.4 phosphate buffer 70:30 v/v followed by: 4 acetonitrile. Analysis of residues was by LSC and LC-MS/MS.

Extracted radioactivity recovered from samples ranged from *ca* 98 to 110% AR at day 0 decreasing to *ca* 49–81% AR after 120 days. Unextracted residues accounted for *ca* 0.6–6.9% AR at Day 0, increasing to a maximums *ca* 13–43% AR after 120 days while radioactivity associated with volatile traps reached 0.1–0.6% AR at Day 120. Following extraction, the organic matter from the Tama and Drummer soils of day 120 were fractionated into humin, fulvic acid, and humic acid. The mean radioactivity in the unextracted residue associated with humin, fulvic acid and humic acid were 13–20%, 11% and 0.3% AR, respectively.

The overall material balance ranged between *ca* 90 and 111% AR.

The amount of aminocyclopyrachlor in the solvent extracts declined from 105, 93 and 94% AR at zero time to 76, 42 and 45% AR after 120 days in the Nambenheim, Tama and Drummer soils, respectively. IN-LXT69 accounted for 4.0–6.4% AR at Day 0 declining thereafter to 0.2–0.4% AR at Day 120.

The dissipation half-lives were calculated using nonlinear first-order regression and are summarised in Table 7.

Table 7 DT₅₀ and DT₉₀ times for aminocyclopyrachlor in three soils (SFO kinetics)

Soil	<i>k</i> (day ⁻¹)	DT ₅₀ (days)	DT ₉₀ (days)	r ²
Nambenheim sandy loam	0.0016	433	1439	0.448
Tama silty clay	0.0058	120	397	0.964
Drummer clay loam	0.0055	126	419	0.938

Conklin and Lym (2013) studied the effect of temperature and moisture on the soil half-life of aminocyclopyrachlor on four soils from the Northern Great Plains. Relevant properties of the soils used are presented in the table below.

Table 8 Properties of Northern Great Plains soils used to study the degradation of aminocyclopyrachlor

Location	Soil	Sand	Silt	% by weight Clay	Organic Matter	FCWC ^a	pH
Fargo	Fargo	5	45	50	7.0	55	7.2
Jamestown	Svea-Barnes	37	42	21	6.4	51	5.7
Medora	Glendive-Havre	5	35	60	1.2	38	8.1
Walcott	Lamoure	86	9	5	2.6	49	7.8

^a Field capacity gravimetric water content

Ten mL of a 36 µg/kg aminocyclopyrachlor solution was mixed thoroughly with 500 g air-dry soil. The treated soil was maintained in the dark at different soil moisture contents and temperatures for 8 weeks. At the end of this period, the soil was frozen to reduce or eliminate microbial activity until soybean bioassay for aminocyclopyrachlor residues. DT₅₀ values for the decline of aminocyclopyrachlor were calculated based on the soybean bioassay results (Table 9).

The rate of aminocyclopyrachlor dissipation generally increased as soil moisture content or temperature increased however, there were also effects that may be due to organic matter and clay content. The DT₅₀ times for aminocyclopyrachlor are not easily predicted by a single soil characteristic.

Table 9 Effect of moisture content and temperature on DT₅₀ values (days) for four soils after treatment with aminocyclopyrachlor at 39 µg/kg.

Moisture content (FCWC ^a)	Temperature (°C)	Fargo	Glendive-Havre	Lamoure	Svea-Barnes
22.5%	16	> 50	20	44	7
45%	16	17	54	30	3
90%	16	5	19	21	11
45%	8	37	> 112	> 80	> 88
45%	16	18	54	52	22
45%	24	11	51	13	13

^a Field capacity gravimetric water content

The soil dissipation of aminocyclopyrachlor occurs at a slow rate. Aminocyclopyrachlor is persistent in the environment.

Field dissipation

In a series of studies, the degradation of DPX-KJM44 was studied following a single application of a wettable powder formulation to bare soil (Shepard 2009 22528, 2010a 22527) or to turf grass (Shepard 2012 22529, 2010b 22526). Treated plot areas ranged from 279 to 372 m². In each experiment, five replicate soil core samples at each sampling time were collected from each of three treated plots. Soil samples were collected at various intervals after depending on the site and up to 540 days after treatment. Samples were collected from depths of 0–5, 5–15, 15–30, 30–50, 50–70, and 70–90 cm. Where available, grass samples were taken from a 30 × 30 cm area by cutting the grass as close as possible to the soil surface. Soil and grass samples were extracted and analysed for residues of DPX-KJM44 and its degradation products aminocyclopyrachlor and IN-LXT69 by LC-MS/MS. The LOQ and LOD for DPX-KJM44 aminocyclopyrachlor and IN-LXT69 were 0.001 mg/kg and 0.0003 mg/kg respectively. Table 10 contains a summary of the experimental conditions.

Table 10 Experimental conditions for soil field dissipation studies on DPX-KJM44

	Location	Application rate g as/ha (g ai/ha)	Spray volume (L/ha)	Soil type	Soil pH ^a	Soil OC% ^b
Bare soil	Ontario	336 (315)	980	Brant silt loam	6.0–8.4	0.23–1.0
	California	336 (315)	890	Tagus silt loam	8.4–8.7	0.1–0.4
Turf	Georgia	168 (158)	944	Tifton sandy loam	6.3–6.4	0.7–0.9
	Ontario	336 (316)	980	Brant silt-loam	6.4–7.1	1.1–2.6

^a Range of soil pH measured in water for soil fraction collected from 0–90 cm depth

^b Range of soil organic carbon measured in soil fraction collected from 0–90 cm depth

Analysis of the total residue (DPX-KJM44 + aminocyclopyrachlor + IN-LXT69) in soil indicated that DPX-KJM44 was rapidly hydrolysed to aminocyclopyrachlor and quickly declined to soil concentrations that were below the limit of detection. Within 14 days after application, aminocyclopyrachlor was the major residue present. The degradation product IN-LXT69 generally showed concentrations < LOD throughout the study. Figure 7 summarises visually the degradation of DPX-KJM44 and aminocyclopyrachlor. Total residues, calculated as the sum of DPX-KJM44, aminocyclopyrachlor and IN-LXT69, are almost entirely due to aminocyclopyrachlor.

Air sampling did not recover significant quantities of DPX-KJM44 or aminocyclopyrachlor in volatile traps suggesting loss through volatilisation is not a significant dissipation mechanism.

The DPX-KJM44 concentrations in soil in units of % mass of applied parent equivalents were used to calculate the DT₅₀ and DT₉₀ values tabulated below. DPX-KJM44 is rapidly degraded to aminocyclopyrachlor with DT₅₀ values of 0.7 to 1.6 days for the four sites studied. In contrast the DT₅₀ values for aminocyclopyrachlor are much longer ranging from 55 to 163.6 days. Meaningful kinetic information could not be generated for IN-LXT69.

The dissipation of DPX-KJM44 in established grass foliage was investigated at two sites with DT₅₀ values for DPX-KJM44 of 0.4 days while the DT₅₀ values for aminocyclopyrachlor were 4.8 to 8.9 days (Figure 8, Table 11).

The modelling results are summarized in Table 11.

Table 11 DT₅₀ and DT₉₀ values for DPX KJM44 and aminocyclopyrachlor in four soils

Site	Matrix	Compound	Model	DT ₅₀ (days)	DT ₉₀ (days)	r ²
Ontario	Soil	DPX-KJM44	SFO	1.6	5.2	0.9408
		aminocyclopyrachlor	SFO	79.9	265	0.7721
California	Soil	DPX-KJM44	SFO	0.7	2.4	0.9707
		aminocyclopyrachlor	SFO	163.6	543.5	0.8048
Sycamore, Georgia	Foliage	DPX KJM44	SFO	0.4	1.4	0.8537

Site	Matrix	Compound	Model	DT ₅₀ (days)	DT ₉₀ (days)	r ²
		aminocyclopyrachlor	SFO	4.8	16.1	
	Soil	DPX-KJM44	SFO	0.8	2.7	
		aminocyclopyrachlor	SFO	55	183	
Ontario	Foliage	DPX KJM44	SFO	0.4	1.4	0.983
		aminocyclopyrachlor	DFOP	8.9	30.2	0.972
	Soil	DPX-KJM44	DFOP	0.8	3.4	0.951
		aminocyclopyrachlor	DFOP	141.4	644	0.754

SFO = Simple first-order model

DFOP = Double first-order parallel model

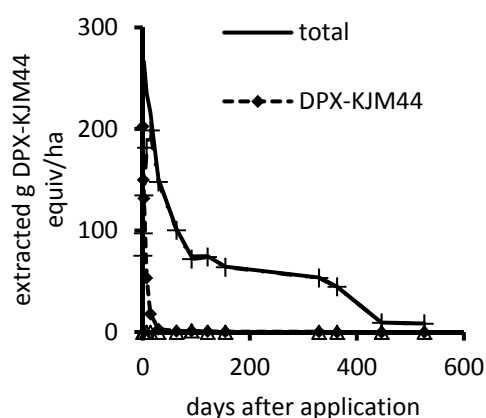
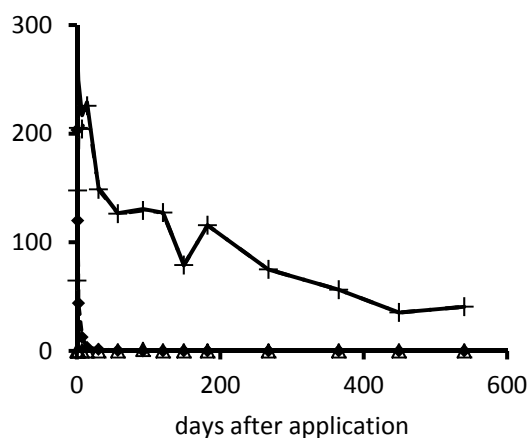
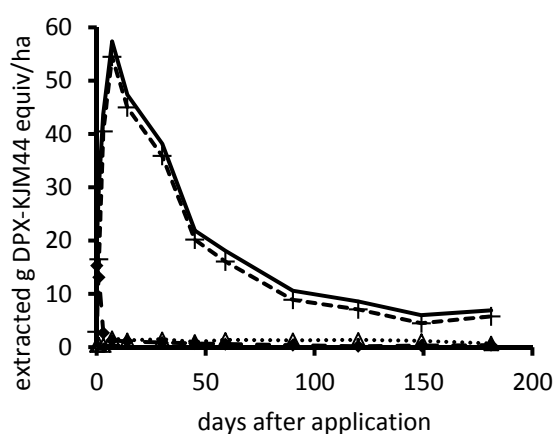
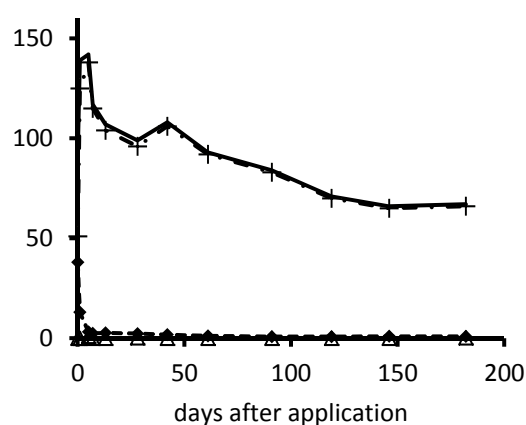
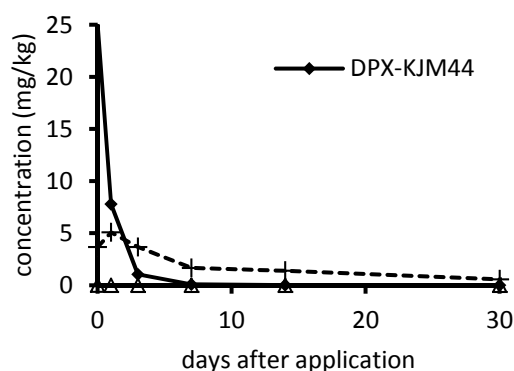
A Ontario Canada (bare soil)**B** California (bare soil)**C** Sycamore Georgia (grass)**D** Ontario Canada (grass)

Figure 7 Evolution of residues of DPX-KJM44, aminocyclopyrachlor and IN-LXT69 in soil following application of DPX-KJM44 to bare soil (A, B) and to plots of grass (C, D)

A Sycamore Georgia



B Ontario

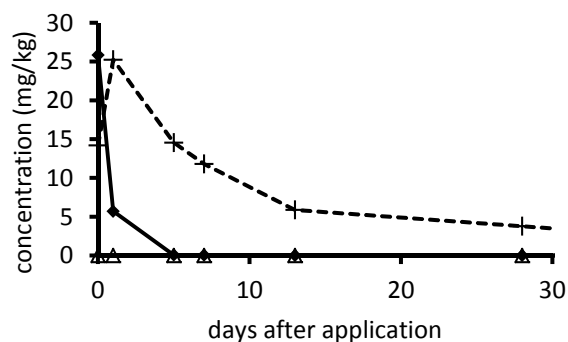


Figure 8 Evolution of residues of DPX-KJM44, aminocyclopyrachlor and IN-LXT69 following application of DPX-KJM44 to plots of grass, residues in grass

Soil photolysis

Wardrope (2007 22118) studied the soil photolysis of aminocyclopyrachlor. [Pyrimidine- ^{14}C]aminocyclopyrachlor was applied by pipette to thinly-layered soil (*ca* 2 mm thick) to achieve a nominal concentration of 448 $\mu\text{g ae/kg}$ soil (448 ppm), equivalent to a field use rate of 336 g ai/ha soil, and irradiated under artificial irradiation (Xenon light) equipped with filters to eliminate wavelengths of < 290 nm. $^{14}\text{CO}_2$ and ^{14}C non-specific organic volatiles were collected. Samples were taken for analysis at 0, 1, 3, 5, 7, 11, and 15 days of irradiation. The soil samples were extracted twice with acetonitrile: 0.2% formic acid (8:2 v/v) followed by an extraction with acetonitrile: 0.15 M ammonium acetate (7:3 v/v). Identification of the transformation products was performed by HPLC and LC-MS analysis using authentic reference standards.

The material balance ranged from 96 to 102% and 95 to 102% of the applied radioactivity (AR) in the irradiated and dark (non-irradiated) samples, respectively. The total extracted radioactivity for irradiated samples decreased during the irradiation period, from a mean of 97% AR at Day 0 to a mean of 83% AR after 15 days. Unextracted ^{14}C -residues increased from $< 4\%$ to a maximum mean value of 17% AR after 15 days. Throughout the experimental phase, evolved $^{14}\text{CO}_2$ and ^{14}C -organic volatile radioactivity were below the limit of quantification. In the non-irradiated (dark control) samples the total extracted radioactivity remained relatively constant at all time points, with means of 97% AR at Day 1 and 92% AR at Day 15. Unextracted ^{14}C -residues increased from *ca* 2 to 5.1% AR after 15 days. Throughout the experimental phase, evolved $^{14}\text{CO}_2$ and ^{14}C -organic volatile radioactivity were below the limit of quantification.

In the irradiated samples, aminocyclopyrachlor decreased from a mean value of 94% AR at day 0, to a mean of 74% AR after 15 days. The only significant transformation product was identified as IN-LXT69, with a maximum mean concentration of 4.9% AR observed at Day 7. Multiple (a total of fourteen but up to six for any one sample) minor transformation products were detected, accounting for a combined mean maximum value of 8.7% AR at Day 11. No individual unidentified component accounted for greater than 5% AR at any time point. In the non-irradiated samples, the parent compound decreased from a mean of 94% AR at day 1 to 87% AR after 15 days. One major transformation product was identified as IN-LXT69, with a mean maximum value of 5.2% AR observed at Day 11. Eight minor transformation products were detected, none of which accounted for greater than 5% AR with a majority less than 1% AR at any time point.

To calculate the rate of degradation of aminocyclopyrachlor due to photolysis only, the first-order rate of degradation constant in the non-irradiated samples was subtracted from the first-order rate of degradation constant in the irradiated samples. The resulting corrected DT_{50} and DT_{90} values

were 61 and 204 days, respectively. These estimated DT_{50} and DT_{90} values are extrapolated beyond the limits of the observed data, 15 days.

Soil photolysis is expected to be a significant route of aminocyclopyrachlor degradation when compared to aerobic soil degradation.

Hydrolysis

Manjunatha (2008, 22116) studied the hydrolytic stability of aminocyclopyrachlor at 50 ± 0.5 °C for 5 days in dark, sterile, aqueous buffered solutions at pH 4, pH 7 and pH 9. Aqueous solutions of aminocyclopyrachlor showed less than 10% degradation at the end of the 5 day study period. Aminocyclopyrachlor is considered stable to hydrolysis at pH 4, 7 and 9 ($t_{1/2}$ at 25 °C > 1 year).

Hydrolysis of aminocyclopyrachlor is not expected to be a significant process under environmental conditions.

Aqueous Photolysis

Lowrie (2008, 22117) studied the photolysis of aminocyclopyrachlor in sterile natural water. Solutions containing 2 µg [^{14}C]aminocyclopyrachlor/mL were continuously irradiated using light from a xenon arc lamp filtered to give a spectral distribution close to that of natural sunlight. The samples were maintained at 20 °C and were irradiated for periods up to 360 h.

The mass balance (mean across all samples) was 99% of the AR. In the pH 4 buffer system, aminocyclopyrachlor was relatively rapidly degraded accounting for 28.1% AR after 360 hours of continuous irradiation. A number of photo-degradates were formed; IN-QFH57 (13.8% AR) and IN-LXT69 (16.1% AR), IN-YY905 (8.0% AR), IN-Q3007 (6.8% AR) and IN-V0977 (12.4% AR) together with several minor components that individually accounted for < 10% AR. No significant degradation was apparent in the dark controls indicating that the degradation in irradiated samples was the result of photodegradation only.

Degradation was also rapid in the irradiated natural water test system. Aminocyclopyrachlor accounted for 5.6% AR after 144 hours of continuous irradiation and was not detected at subsequent sampling times. IN-QFH57 was the major degradation product accounting for 29.7% AR at 360 hours with other significant degradates IN-YY905, IN-Q3007 and IN-V0977 accounting for 11.7, 24.4 and 14.6% AR at 360 hours. Two other components that each accounted for more than 10% AR could not be identified though from their properties are thought to be small aliphatic molecules. Other components individually accounted for less than 10% AR.

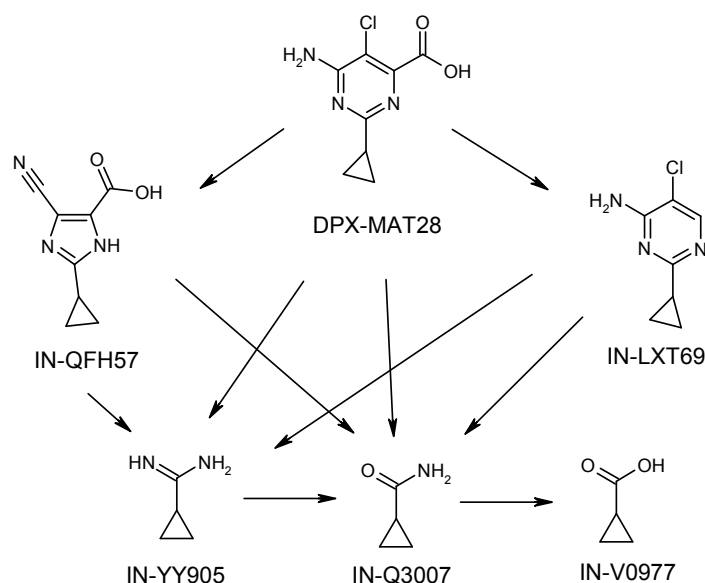


Figure 9 Proposed photodegradation pathway for aminocyclopyrachlor in pH 4 buffer and natural water.

Calculated degradation rate constants (half-lives) for aminocyclopyrachlor under constant irradiation are 7.3 days and 24.2 days for the DT_{50} and DT_{90} values in pH 4 buffer and 1.3 and 4.5 days for the DT_{50} and DT_{90} values in natural water.

Aminocyclopyrachlor is rapidly degraded by photolysis in both pH 4 sterile buffer and natural water solutions. Aminocyclopyrachlor is photodegraded through at least three separate pathways: decarboxylation to form IN-LXT69, photo-induced simultaneous elimination of hydrogen chloride and pyrimidine ring contraction to form the imidazole-nitrile IN-QFH57 and pyrimidine ring destruction to form the cyclopropyl amidine, IN-YY905 with subsequent hydrolysis to IN-Q3007 and IN-V0977. Unidentified products are likely to be other low molecular weight compounds.

Photolysis of aminocyclopyrachlor could potentially be a significant route of degradation under environmental conditions.

Confined rotational crop studies

A confined rotational crop study was conducted on a sandy loam soil (65.9% sand, 18.7% silt, 15.4% clay; pH 6.3; 3.2% organic carbon; 15.4 meq/100 g CEC) treated with [^{14}C]-DPX-KJM44 at either 75 or 369 g ai/ha (MacDonald and Hall 2009 22559). The lower application rate was used for rotations of 15, 30, 60, 120, and 300 days while the high application rate was used with a 300 day rotation only. Seeds of cabbage and turnip were sown into the soil at 30, 60, 120 and 300 (both treatment rates) days after application (DAA). Seeds of maize (field corn) were sown into the soil at 15, 120 and 300 (both treatment rates) DAA. The trials were conducted under greenhouse conditions. The growth stages for harvest of immature crop fractions were maize forage (whole aerial portion, collected at late dough/early dent, BBCH 85), ca 50% expected mature head size (BBCH 45) for cabbage and ca 50% expected tuber diameter (BBCH 45) for turnip. Tissues were homogenised and, if necessary, extracted sequentially with acetonitrile:water (9:1, v/v) and acetonitrile:water (7:3 v/v, twice). For select samples, the post extraction solids (PES) were further extracted sequentially with enzymes (α -amylase, amyloglucosidase/cellulase combination), alkali (0.1 N NaOH at 60 °C) and acid (1 N HCl at 60 °C). The total radioactive residues (TRR) were expressed as mg equiv DPX-KJM44/kg.

Radioactive residues detected in immature and mature cabbage ranged from 0.005 to 0.027 mg equiv/kg (at both treatment rates). The majority of the ^{14}C was recovered in solvent extracts with unextracted residues amounting to no more than 0.005 mg equiv/kg. Aminocyclopyrachlor accounted for 60–61%TRR (0.008–0.014 mg equiv/kg) in mature cabbage from plots treated 30 and

60 days before sowing (low application rate) and for 83% TRR (0.010 mg equiv/kg) from plots treated 300 days before sowing (high application rate). DPX-KJM44 was only observed at trace levels (0.001 mg/kg) in mature cabbage. Unidentified residues accounted for ≤ 0.004 mg equiv/kg.

TRR in immature turnip tops at the 75 g ai/ha soil treatment rate were ≤ 0.029 mg equiv/kg while TRRs in immature turnip tubers were ≤ 0.010 mg equiv/kg. The TRR in immature turnip tops at the 369 g ai/ha soil treatment rate were 0.085 mg equiv/kg with no residues detected in the immature tubers. TRR in turnips grown from seeds sown 30, 60, 120 and 300 DAA at the lower treatment rate were ≤ 0.011 mg equiv/kg in mature turnip tops and ≤ 0.003 mg equiv/kg in the tubers. TRR in mature turnip tops and tubers from the high rate were each 0.004 mg equiv/kg.

Aminocyclopyrachlor accounted for 40 to 59% TRR (0.004–0.007 mg equiv/kg) of the ^{14}C present in mature turnip tops from crops sown 60 and 120 DAA. DPX-KJM44 (17% TRR, 0.002 mg equiv/kg) was only detected in the mature tops from the 60 DAA planting interval. Unidentified components were individually present at < 0.01 mg equiv/kg.

TRR in immature maize (forage) from seeds sown at 15, 120, 300 (75 g ai/ha) and 300 (369 g ai/ha) DAA were 0.091, 0.011, 0.028 and 0.246 mg equiv/kg, respectively; the corresponding values for stover were 0.149, 0.023, 0.058 and 0.262 mg equiv/kg, for cobs were 0.024, 0.003, ND and 0.012 mg equiv/kg, and for grain were 0.067, 0.012, 0.018 and 0.085 mg equiv/kg. The majority of the radioactivity from all maize tissues was able to be extracted with the solvent systems used (61–99% TRR). TRR in post-extraction solids did not exceed 0.009 mg equiv/kg.

Aminocyclopyrachlor was the principal component (63–70% TRR) in forage from the 15 and 120 DAA plots. DPX-KJM44 was only observed in the 15 DAA forage (10% TRR; 0.009 mg/kg) and not in forage of crops sown at later intervals after soil application. Unidentified components were also present accounting for ≤ 0.01 mg equiv/kg in total.

Aminocyclopyrachlor was also the major ^{14}C component in forage from the 300 DAA plots (low and high treatments groups) comprising 68–71% TRR. A trace amount of DPX-KJM44 (0.7% TRR; 0.002 mg/kg) was detected in forage from the high soil treatment rate. A component with similar chromatographic properties to IN LXT69 was detected at ≤ 0.01 mg equiv/kg at both soil treatment rates. Multiple (5–26) unidentified and apolar components totalling 5–21% TRR (≤ 0.05 mg equiv/kg) with individual components ≤ 0.01 mg equiv/kg were also observed in the various forage samples at both soil treatment rates. Chromatographic analysis of enzyme digests of the post extraction solids from the high soil treatment rate demonstrated the presence of aminocyclopyrachlor (11% TRR, 0.026 mg equiv/kg).

Aminocyclopyrachlor was the major component detected in all stover samples ranging from 0.014 to 0.151 mg equiv/kg. DPX-KJM44 accounted for 0.013 mg/kg in the stover sample from the 15 DAA plot, and ≤ 0.002 mg/kg in all other stover samples. A late eluting apolar unknown accounted for 2–7% TRR (≤ 0.018 mg equiv/kg) whereas a component with similar chromatographic properties to IN-LXT69 was detected at ≤ 0.001 mg equiv/kg. Multiple (12 to 27) unidentified components totalled 6–25% TRR (0.001–0.064 mg equiv/kg), with individual components ≤ 0.012 mg equiv/kg, were also observed in the various stover samples.

In grain, aminocyclopyrachlor accounted for 71–72% TRR (0.01–0.05 mg equiv/kg) in samples from the 15 and 120 DAA plots. DPX KJM44 was observed at a trace level (< 0.01 mg/kg) in the 15 DAA plot grain. Aminocyclopyrachlor accounted for 72–76% TRR in the grain from maize sown at 300 DAA at the low and high treatment rates. DPX-KJM44 (2.5% TRR) was detected in the low treatment rate sample only. Unidentified components totalled 2.2–4.2% TRR with no single component comprising greater than 0.001 mg equiv/kg were also detected.

Table 12 Identification of ^{14}C residues in rotational crops following application of ^{14}C -DPX-KJM44 to soil

	Application	Sowing	TRR			Extracted	(%TRR)			PES
RAC	rate g ai/ha	interval (DAA)	mg equiv/kg	Total	KJM44	ACP	LXT69	Apolar unknown	Un-identified	(%TRR)
Cabbage	75	30	0.023	82.4	3.3	60.0			19.1 ^a	17.6

	Application	Sowing	TRR			Extracted	(%TRR)			PES
RAC	rate g ai/ha	interval (DAA)	mg equiv/kg	Total	KJM44	ACP	LXT69	Apolar unknown	Un-identified	(%TRR)
		60	0.013	72.4	4.6	60.6			7.2 ^b	27.6
		120	ND							
		300	ND							
	369	300	0.012	83.0	ND	83.0			ND	17.0
Turnip tops	75	30	0.006							
		60	0.010	100	17.1	40.5		ND	42.4 ^c	ND
		120	0.011	87.7	ND	59.3		23.3	5.1 ^d	12.3
		300	0.003							
	369	300	0.004							
Turnip roots	75	30	ND							
		60	ND							
		120	ND							
		300	0.003							
	369	300	0.004							
Maize forage	75	15	0.091	95.3	10.0	62.7	ND	4.0	1.9 ^e	4.6
		120	0.011	74.9	ND	69.7	ND	5.2	ND	25.1
		300	0.028	82.9	ND	68.2	1.5	4.9	8.3 ^f	17.1
	369	300	0.246	98.7	0.7	71.4	1.9	5.0	16.3 ^g	1.4
Maize stover	75	15	0.149	96.7	9.1	71.9	0.2	5.1	5.6 ^h	3.3
		120	0.023	60.9	ND	58.8	ND	2.1	ND	39.1
		300	0.058	95.2	1.2	45.9	ND	6.5	19.6 ⁱ	4.7
	369	300	0.262	96.8	0.9	58.0	ND	6.9	25.3 ^j	3.3
Maize grain	75	15	0.067	98.1	4.5	72.3		2.6	ND	1.9
		120	0.012	70.9	ND	70.9		ND	ND	29.1
		300	0.018	78.2	2.5	71.5		ND	4.2 ^k	21.8
	369	300	0.085	98.9	ND	76.0		1.0	1.2 ^l	1.1

^a Two components, no single component greater than 17.5% TRR, 0.004 mg equiv/kg

^b Three components, no single component greater than 3.4% TRR, 0.001 mg equiv/kg

^c Three components, no single component greater than 16.4%TRR, 0.002 mg equiv/kg

^d One single component

^e Five components, no single component greater than 0.5%TRR, < 0.001 mg equiv/kg

^f Eight components, no single component greater than 2.0%TRR, 0.001 mg equiv/kg

^g 25 components, no single component greater than 1.4%TRR, 0.003 mg equiv/kg

^h 12 components, no single component greater than 1.5% TRR, 0.002 mg equiv/kg

ⁱ 25 components, no single component greater than 3.0% TRR, 0.002 mg equiv/kg

^j 27 components, no single component greater than 4.7% TRR, 0.012 mg equiv/kg

^k Three components, no single component greater than 1.7%TRR, < 0.001 mg equiv/kg

^l Three components, no single component greater than 0.5%TRR, < 0.001 mg equiv/kg

ACP = aminocyclopyrachlor

The proposed metabolic pathway for DPX-KJM44 in confined rotational crops is provided in Figure 10. The pathway involves de-esterification of DPX-KJM44 to form aminocyclopyrachlor. Aminocyclopyrachlor undergoes decarboxylation (to a much lesser extent) to form IN LXT69.

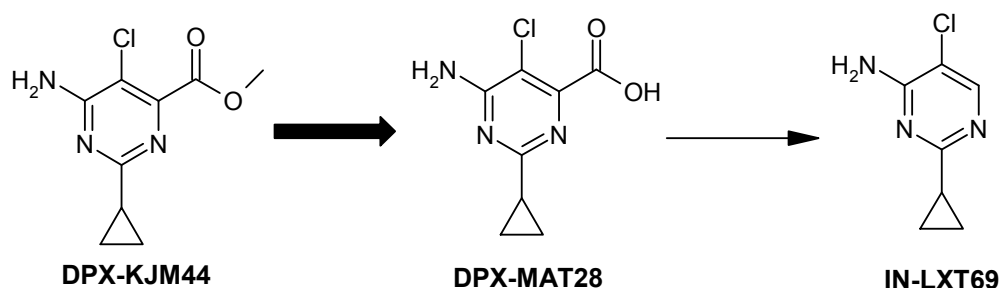


Figure 10 Proposed metabolic pathway for [¹⁴C]DPX-KJM44 in confined rotational crops

Field crop rotational studies

No field crop rotational studies were made available to the meeting. From the confined rotational crop study, low levels of residues are expected in rotational crops.

METHODS OF RESIDUE ANALYSIS*Analytical methods*

The Meeting received descriptions and validation data for analytical methods for residues of aminocyclopyrachlor in animal and plant matrices. The methods are suitable for analysis of DPX-KJM44, aminocyclopyrachlor and metabolites in plant matrices as well as for DPX-KJM44, aminocyclopyrachlor and IN-LXT69 in animal commodities.

A brief description of the methods used in the residue trials is given in Table 13.

Table 13 Summary of major analytical methods used for the determination of aminocyclopyrachlor and metabolites in various matrices

Method/ reference	Matrix	Extraction	Clean-up	Detection, LOQ
22582	Plant commodities	0.15 M ammonium acetate/acetonitrile. Combined extracts are diluted with 0.1 N HCl and water	aminocyclopyrachlor , DPX-KJM44, IN-LXT69, IN-QGC48: Extract aliquots concentrated under a stream of N ₂ at 40 °C, diluted with 0.5% formic acid, passed through SAX SPE columns and loaded onto an Oasis® MCX SPE cartridge, eluted with 75 mM ammonium hydroxide in methanol into tubes containing 0.2% formic acid. Eluates are concentrated under stream of N ₂ at 40 °C, diluted with 0.01% formic acid and filtered through 0.45 µm PTFE filter ready for analysis IN-QFH57: Extract aliquots concentrated under a stream of N ₂ at 40 °C, diluted with 0.01% formic acid, loaded onto an SAX SPE cartridge, eluted with 1% formic acid in methanol into tubes containing 0.2% formic acid. Eluates are concentrated under stream of N ₂ at 40 °C, diluted with 0.01% formic acid and filtered through 0.2 µm PTFE filter ready for analysis	LC-MS/MS LOQ 0.01 mg/kg
25836	Animal commodities	Milk: acetonitrile/0.1% formic acid (aq) (90:10, v/v). An aliquot is diluted with 0.01% formic acid (aq) for analysis. Tissues: acetonitrile/0.1% formic acid (aq) 90:10, 70:30 then 50:50 v/v. Extraction is on ice. Kidney, liver, fat:	Muscle: An aliquot of extract is diluted with 0.01% formic acid (aq) and passed through a MCX SPE cartridge.	LC-MS/MS LOQ 0.01 mg/kg

*Plant materials**Residue Analytical Method 22582 (Penz and Nanita 2008 22582)*

After grinding the samples in dry ice to a powder, the samples should be returned to the freezer until ready for analysis. The analytes were extracted sequentially from a 10 g sample by (1) soaking in 35 mL of 0.15 M ammonium acetate (aq), addition of 80 mL of acetonitrile and homogenization; and

(2) homogenization in 100-120 mL of acetonitrile/0.15 M ammonium acetate (aq) 70/30. Extracts were combined and diluted with 0.1 N HCl and water.

For analysis of DPX-KJM44, aminocyclopyrachlor, IN-LXT69, and IN-QGC48, 1–2 mL of extract aliquots were concentrated to 0.5 mL (stream of N₂, 40 °C) and diluted with 0.5% formic acid (aq). The extracts were filtered using SAX SPE cartridges and loaded onto Oasis® MCX SPE cartridges. The analytes were eluted with 15–20 mL of 75 mM ammonium hydroxide in methanol into tubes containing 1 mL of 0.2% formic acid (aq). The eluates were concentrated to a volume of 1–2 mL (stream of N₂, 40 °C), diluted with 0.01% formic acid (aq), filtered through a 0.45 µm PTFE filter and analysed by LC-MS/MS.

For analysis of IN-QFH57, 1 mL of extract aliquots were concentrated to a volume of 0.5 mL (stream of N₂, 40 °C), diluted with 0.01% formic acid (aq) and loaded onto SAX SPE cartridges. IN-QFH57 was eluted with 1% formic acid in methanol into tubes containing 0.5 mL of 0.2% formic acid (aq). The eluates were concentrated to a volume of 0.5 mL (stream of N₂, 40 °C), diluted with 0.01% formic acid (aq), filtered through a 0.2 µm PTFE filter and analysed by LC-MS/MS.

Transitions monitored for quantitation were m/z 214 → 68 for aminocyclopyrachlor (confirmation m/z 214 → 101), 228 → 68 for DPX-KJM44 (confirmation m/z 228 → 101), 170 → 76 for IN-LXT69 (confirmation m/z 170 → 103), 192 → 178 for IN-QGC48 (confirmation 192 → 132) and 176 → 132 for IN-QFH57 (confirmation 176 → 105).

Good linearity was observed for the calibration curves of DPX-KJM44, aminocyclopyrachlor, IN-LXT69, IN-QGC8 and IN-QFH57 solvent standards at 0.02–2.0 ng/mL, i.e. each had correlation coefficient, r , ≥ 0.99 . Inferences were not observed for DPX-KJM44, aminocyclopyrachlor, IN-LXT69, IN-QFH57, and IN-QGC48 at their respective chromatographic retention times in the samples tested.

The LOQ determined in this method for all analytes was 0.01 mg/kg for all matrices tested. In addition, at this fortification level, the analyte peak consistently represents a signal-to-noise ratio of approximately 5–20 to 1 for the least responsive analyte.

Table 14 Aminocyclopyrachlor and metabolite recovery data obtained during validation of 22582 (recovery $\pm$ RSD)

Matrix	Level (mg/kg)	N	DPX-KJM44	Aminocyclopyrachlor	IN-QFH57	IN-QGC48	IN-LXT69	Reference
grass	0.01	5	82 $\pm$ 3	80 $\pm$ 8	91 $\pm$ 7	79 $\pm$ 14	83 $\pm$ 10	22582
forage	0.1	5	83 $\pm$ 1	79 $\pm$ 2	88 $\pm$ 5	82 $\pm$ 1	89 $\pm$ 4	Supplement No. 1,
grass	0.01	5	85 $\pm$ 6	77 $\pm$ 7	80 $\pm$ 13	84 $\pm$ 17	89 $\pm$ 4	Revision No. 2
hay	0.1	5	84 $\pm$ 3	71 $\pm$ 4	93 $\pm$ 4	87 $\pm$ 2	88 $\pm$ 2	
lettuce	0.01	5	90 $\pm$ 5	87 $\pm$ 6	75 $\pm$ 7	86 $\pm$ 10	88 $\pm$ 3	22582
	0.1	5	86 $\pm$ 3	86 $\pm$ 2	80 $\pm$ 6	93 $\pm$ 4	89 $\pm$ 4	Supplement No. 2
oranges	0.01	5	94 $\pm$ 3	94 $\pm$ 5	77 $\pm$ 6	86 $\pm$ 9	92 $\pm$ 5	
	0.1	5	93 $\pm$ 2	89 $\pm$ 3	85 $\pm$ 4	98 $\pm$ 3	93 $\pm$ 1	
sweet	0.01	5	90 $\pm$ 3	88 $\pm$ 7	87 $\pm$ 9	80 $\pm$ 11	91 $\pm$ 6	
corn	0.1	5	87 $\pm$ 8	89 $\pm$ 3	88 $\pm$ 11	90 $\pm$ 9	92 $\pm$ 7	

Rodgers (2010 30574) conducted an independent laboratory validation of the method. Two slightly different versions were studied: one, following purification, based on strong anion exchange (SAX) filtration and the other using SAX and mixed cation exchange (MCX) solid phase extraction (SPE). Additionally, the effect of replacing the 0.5% formic acid flushing solvent for the SAX cartridge with 0.1 N HCl was investigated. The simple SAX filtration method (no MCX step) combined with 0.1 N HCl flushing solvent gave satisfactory performance (Table 15).

Boughton (2011 31151) conducted an independent laboratory validation of method 22582 in grass and representative oily (corn), watery (lettuce) and acidic (oranges) crops. Acceptable recoveries were obtained for all analytes in all matrices except for IN-QFH57 in oranges.

Table 15 Aminocyclopyrachlor and metabolite recovery data obtained during independent laboratory validations of 22582

Matrix	Level (mg/kg)	N	DPX-KJM44	Aminocyclopyrachlor	IN-QGC48	IN-LXT69	IN-QFH57
Grass hay	0.01	5	69 ± 4	77 ± 5	71 ± 7	74 ± 5	–
(SAX + 0.5% formic acid)	0.1	5	75 ± 8	74 ± 8 ^a	78 ± 9	80 ± 7	–
Grass hay	0.01	5	83 ± 4	105 ± 6	87 ± 2	101 ± 17	–
(SAX + 0.1N HCl)	0.1	5	85 ± 4	108 ± 4	92 ± 3	77 ± 5	–
Lettuce	0.01	5	79 ± 4	94 ± 10	86 ± 8	85 ± 5	95 ± 5
	0.1	5	78 ± 3	84 ± 3	89 ± 2	82 ± 5	103 ± 2
Orange	0.01	5	88 ± 2	94 ± 6	95 ± 3	91 ± 5	171 ± 4
	0.1	5	88 ± 3	90 ± 3	85 ± 2	90 ± 4	171 ± 3
Corn	0.01	5	75 ± 2	77 ± 6	76 ± 10	70 ± 2	81 ± 2.5
	0.1	5	75 ± 2	76 ± 2	80 ± 1	74 ± 3	89 ± 1

^a n=4

Nanita *et al.* (2013) reported the results of an inter-laboratory study comprising five participating laboratories in the USA that was used to evaluate the method reproducibility and ruggedness. Method validation sets for the matrix white pine were analysed at the five laboratories. One laboratory also analysed grass forage and hay samples. The results are shown in Table 16.

Table 16 Summary of aminocyclopyrachlor recoveries during method validation and concurrent fortifications for routine sample analysis at five laboratories in the USA

Laboratory	Matrix	N	%Recovery (mean ± SD) ^a
A	White pine	6	111 ± 10
B	Conifers (Norway spruce 16, white pine 7, white spruce 4)	27	100 ± 15
C	White pine	6	101 ± 7
D	White pine	10	88 ± 14
E	White pine	9	102 ± 13
A	Grass forage	10	103 ± 13
A	Grass hay	10	107 ± 5

^a Fortification levels for aminocyclopyrachlor included 0.01, 0.1 and 1 mg/kg.

It was reported that acceptable analyte calibration curves were routinely obtained at each laboratory ($r > 0.99$, RSD response factors $< 20\%$) and when combined with the satisfactory mean recoveries and standard deviations it is concluded the method is suitably validated for use as a single analyte method in regulatory laboratories.

RESIDUES IN FOOD OF ANIMAL ORIGIN

Residue Analytical Method 25836

The extraction procedure for milk involves extraction twice using acetonitrile/0.1% aqueous formic acid (90:10, v/v). The extracts are combined and an aliquot diluted with 0.01% aqueous formic acid prior to analysis. For tissue samples, after grinding the samples in dry ice to a powder, the samples should be returned to the freezer until ready for analysis. Muscle, liver, kidney and fat are extracted sequentially with acetonitrile/0.1% aqueous formic acid 90:10, 70:30 then 50:50 v/v (liver, kidney and muscle are extracted on ice). An aliquot of the extract of fat, liver or kidney is analysed following a dilution step with 0.01% aqueous formic acid. An aliquot from the extract of muscle is analysed following a clean-up step using mixed mode cation exchange solid phase extraction.

Aminocyclopyrachlor, DPX-KJM44, and IN-LXT69 are quantified by liquid chromatography with tandem mass spectrometry employing turbo ion spray ionisation in positive mode. Transitions monitored for quantitation were m/z 214 → 68 for aminocyclopyrachlor (confirmation m/z 214 →

101), 228 → 68 for DPX-KJM44 (confirmation m/z 228 → 101) and 170 → 76 for IN-LXT69 (confirmation m/z 170 → 103). The method LOQs were 0.01 mg/kg for each analyte.

Good linearity was observed for the calibration curves of DPX-KJM44, aminocyclopyrachlor and IN-LXT69 solvent standards at 0.02–2.0 ng/mL, i.e. each had correlation coefficient, r , ≥ 0.99 . Inferences were not observed for DPX-KJM44, aminocyclopyrachlor and IN-LXT69 at their respective chromatographic retention times in the samples tested.

As the solvent extraction procedure is close to that used in the metabolism study for which greater than 80% of the TRR were recovered from tissues and milk, the extraction procedure is considered suitable.

Recovery data reported by Pentz and Nanita (2009 25836) and also by Ward and Spence (2010 27162 rev 1) are shown in Table 17.

Table 17 DPX-KJM44, aminocyclopyrachlor and metabolite recovery data obtained during validation of the method 25836 (recovery $\pm$ RSD)

Matrix	Level (mg/kg)	N	DPX-KJM44	Aminocyclopyrachlor	IN-LXT69	Reference
muscle	0.01	5	94 $\pm$ 1	93 $\pm$ 7	74 $\pm$ 4	25836
	0.1	5	93 $\pm$ 4	97 $\pm$ 5	78 $\pm$ 3	
liver	0.01	5	81 $\pm$ 9	91 $\pm$ 4	83 $\pm$ 5	
	0.1	5	79 $\pm$ 14	89 $\pm$ 9	84 $\pm$ 13	
kidney	0.01	5	89 $\pm$ 3	99 $\pm$ 9	87 $\pm$ 14	
	0.1	5	83 $\pm$ 14	102 $\pm$ 8	91 $\pm$ 10	
fat	0.01	5	94 $\pm$ 5	87 $\pm$ 4	79 $\pm$ 3	
	0.1	5	96 $\pm$ 6	103 $\pm$ 5	87 $\pm$ 4	
milk	0.01	5	90 $\pm$ 12	95 $\pm$ 5	90 $\pm$ 10	
	0.1	5	94 $\pm$ 16	101 $\pm$ 5	97 $\pm$ 14	
Skim milk	0.01	5	106 $\pm$ 2	96 $\pm$ 3	100 $\pm$ 4	
	0.1	5	103 $\pm$ 1	101 $\pm$ 1	101 $\pm$ 2	
Heavy cream	0.01	5	97 $\pm$ 2	91 $\pm$ 2	95 $\pm$ 5	
	0.1	5	93 $\pm$ 3	100 $\pm$ 5	102 $\pm$ 1	
eggs	0.01	5	102 $\pm$ 6	94 $\pm$ 9	95 $\pm$ 5	
	0.1	5	101 $\pm$ 6	93 $\pm$ 8	99 $\pm$ 7	
fish	0.01	5	83 $\pm$ 5	84 $\pm$ 8	74 $\pm$ 6	
	0.1	5	83 $\pm$ 8	81 $\pm$ 12	78 $\pm$ 6	
muscle	0.01	3	77 $\pm$ 6	76 $\pm$ 4	73 $\pm$ 5	27162
	0.1	3	80 $\pm$ 5	75 $\pm$ 3	92 $\pm$ 10	revision no. 1
liver	0.01	3	91 $\pm$ 1	95 $\pm$ 6	91 $\pm$ 5	
	0.1	3	84 $\pm$ 4	97 $\pm$ 4	94 $\pm$ 2	
kidney	0.01	3	95 $\pm$ 7	111 $\pm$ 8	89 $\pm$ 8	
	0.1	3	100 $\pm$ 2	98 $\pm$ 5	103 $\pm$ 4	
fat	0.01	3	100 $\pm$ 4	107 $\pm$ 17	101 $\pm$ 4	
	0.1	3	100 $\pm$ 2	95 $\pm$ 4	97 $\pm$ 2	
milk	0.01	3	104 $\pm$ 1	89 $\pm$ 5	103 $\pm$ 2	
	0.1	3	94 $\pm$ 3	78 $\pm$ 1	93 $\pm$ 3	

In a study describing the independent laboratory validation of the method (Sheehan 2010 28151), acceptable recoveries were obtained with fortified samples though it is noted that analysis of DPX-KJM44 requires special care to ensure the compound does not hydrolyse during the analytical procedure. Initially DPX-KJM44 was hydrolysed during analysis of liver samples fortified with a mixture of DPX-KJM44, aminocyclopyrachlor and IN-LXT69 leading to low recoveries for DPX-KJM44 and high recoveries for aminocyclopyrachlor. Subsequent minor modifications such as smaller sample sizes lead to satisfactory recoveries being obtained (liver 2). Special care should be taken to keep liver and kidney samples chilled on ice where practical during the extraction procedure.

Table 18 DPX-KJM44, aminocyclopyrachlor and metabolite recovery data obtained during validation of the method and independent laboratory validation of 25836 (recovery $\pm$ RSD)

Matrix	Level (mg/kg)	N	DPX-KJM44	Aminocyclopyrachlor	IN-LXT69	Reference
liver 1	0.01	5	31 $\pm$ 25 ^a	168 $\pm$ 20	106 $\pm$ 5	28151
	0.1	5	39 $\pm$ 31 ^a	148 $\pm$ 7	102 $\pm$ 2.8	independent
	0.01	5	33 $\pm$ 8 ^b			laboratory
	0.1	5	43 $\pm$ 13 ^b			validation
liver 2	0.01	5	83 $\pm$ 5 ^a	93 $\pm$ 19	–	
	0.1	5	80 $\pm$ 6 ^a	86 $\pm$ 9	–	
	0.01	5	97 $\pm$ 6 ^b	–	–	
	0.1	5	92 $\pm$ 5 ^b	–	–	
milk	0.01	5	107 $\pm$ 2 ^a	97 $\pm$ 11	107 $\pm$ 7	
	0.1	5	108 $\pm$ 1 ^a	101 $\pm$ 3	107 $\pm$ 2	
	0.01	5	107 $\pm$ 4 ^b			
	0.1	5	110 $\pm$ 1 ^b			
egg	0.01	5	99 $\pm$ 2 ^a	98 $\pm$ 8	101 $\pm$ 5	
	0.1	5	100 $\pm$ 1 ^a	92 $\pm$ 3	96 $\pm$ 2	
	0.01	5	99 $\pm$ 4 ^b			
	0.1	5	101 $\pm$ 3 ^b			

^a Gradient elution^b Isocratic elution*Applicability of multiresidue methods*

Aminocyclopyrachlor is not suitable for inclusion in QuEChERS and US FDA pesticide multi-residue methods (Nanita *et al.* 2013).

STABILITY OF RESIDUES IN STORED ANALYTICAL SAMPLES

The freezer storage stability of aminocyclopyrachlor in homogenised plant, animal tissues, milk and egg samples fortified with DPX KJM44, aminocyclopyrachlor or metabolites was studied. Except for liver and kidney, residues were stable for the duration of the storage studies.

Stability of residues in plant products

Vogl (2009b 24605) studied the freezer storage stability of DPX-KJM44, aminocyclopyrachlor and IN-LXT69 in samples of grass forage and hay spiked separately with the compounds at a nominal rate of 0.5 mg/kg. Samples were analysed using procedures based on method 22582. The data indicate residues of DPX-KJM44, aminocyclopyrachlor and IN-LXT69 are stable at approximately -20 °C for at least 500 days in grass forage and grass hay.

Table 19 Storage stability results for samples spiked with aminocyclopyrachlor, DPX-KJM44 and IN-LXT69

Commodity	Storage	aminocyclopyrachlor		DPX KJM44	IN-LXT69	
	Period (days)	(mg/kg)	Procedural Recovery (%)	(mg/kg)	Procedural Recovery (%)	(mg/kg)
Grass forage	0	0.46/ 0.47	–	0.45/ 0.46	–	0.44/ 0.46
	7	0.46/ 0.45	93/ 92	0.44/ 0.42	96/ 93	0.49/ 0.46
	14	0.40/ 0.41	86/ 91	0.40/ 0.41	85/ 89	0.43/ 0.39
	30	0.41/ 0.43	87/ 83	0.41/ 0.40	84/ 82	0.38/ 0.37
	60	0.37/ 0.40	75/ 76	0.36/ 0.36	75/ 80	0.40/ 0.40
	90	0.45/ 0.46	86/ 79	0.45/ 0.45	87/ 81	0.49/ 0.45
	150	0.41/ 0.41	79/ 89	0.41/ 0.37	82/ 90	0.41/ 0.39
	210	0.38/ 0.39	76/ 77	0.39/ 0.38	81/ 80	0.40/ 0.41
	300	0.39/ 0.42	79/ 79	0.41/ 0.38	82/ 80	0.40/ 0.45
	360	0.44/ 0.44	89/ 91	0.45/ 0.42	94/ 94	0.41/ 0.46
	500	0.41/ 0.43	88/ 87	0.44/ 0.42	90/ 92	0.44/ 0.45
						93/ 93

	Storage	aminocyclopyrachlor		DPX KJM44		IN-LXT69	
Commodity	Period (days)	(mg/kg)	Procedural Recovery (%)	(mg/kg)	Procedural Recovery (%)	(mg/kg)	Procedural Recovery (%)
Grass hay	0	0.85/ 0.83	85/ 83	0.87/ 0.84	87/ 84	0.88/ 0.81	88/ 81
	7	0.78/ 0.79	81/ 81	0.89/ 0.85	85/ 82	0.79/ 0.82	85/ 85
	14	0.80/ 0.76	82/ 75	0.84/ 0.86	84/ 80	0.86/ 0.90	85/ 81
	30	0.82/ 0.84	83/ 80	0.90/ 0.93	88/ 85	0.78/ 0.82	80/ 75
	60	0.84/ 0.76	75/ 77	0.85/ 0.81	81/ 81	0.91/ 0.82	85/ 85
	90	0.90/ 0.93	92/ 89	0.95/ 0.96	99/ 98	0.93/ 0.950	96/ 92
	150	0.73/ 0.77	74/ 77	0.87/ 0.82	80/ 85	0.84/ 0.86	67/ 74
	210	0.84/ 0.81	79/ 80	0.85/ 0.86	86/ 85	0.90/ 0.91	85/ 88
	300	0.74/ 0.77	79/ 77	0.81/ 0.87	83/ 80	0.80/ 0.80	80/ 73
	360	0.88/ 0.95	95/ 97	0.89/ 0.92	97/ 95	0.91/ 0.86	89/ 90
	500	0.83/ 0.87	98/ 87	0.91/ 0.93	99/ 92	0.89/ 0.89	97/ 91

In another study, separate grass forage and grass hay control sample replicates were individually fortified with IN-QFH57 or IN-QGC48 (Vogl 2010 27890). Grass samples were fortified with the respective analyte at 0.30 mg/kg for forage and hay, and stored at approximately -20 °C.

The data indicate residues of IN-QFH57 and IN-QGC48 are stable at approximately -20 °C for at least 400 days in grass forage and grass hay.

Table 20 Storage stability results for samples spiked with aminocyclopyrachlor at 0.2–0.3 mg/kg

	Storage	IN-QFH57		IN-QGC48	
Commodity	Period (days)	(mg/kg)	Procedural Recovery (%)	(mg/kg)	Procedural Recovery (%)
Grass forage	0	0.28/ 0.27	–	0.33/ 0.33	–
	7	0.20/ 0.19	86/ 65	0.33/ 0.32	105/ 97
	14	0.23/ 0.23	91/ 90	0.31/ 0.36	119/ 118
	30	0.22/ 0.22	88/ 86	0.30/ 0.30	102/ 109
	60	0.21/ 0.21	89/ 87	0.27/ 0.27	101/ 100
	90	0.23/ 0.23	91/ 87	0.27/ 0.27	106/ 97
	150	0.23/ 0.22	86/ 77	0.28/ 0.28	117/ 109
	210	0.24/ 0.23	87/ 79	0.29/ 0.31	107/ 109
	300	0.21/ 0.20	73/ 75	0.28/ 0.28	99/ 102
	360	0.23/ 0.22	80/ 79	0.27/ 0.28	98/ 99
	400	0.25/ 0.26	101/ 94	0.29/ 0.27	105/ 91
Grass hay	0	0.35/ 0.35	–	0.31/ 0.32	–
	7	0.28/ 0.28	99/ 97	0.27/ 0.28	96/ 99
	14	0.26/ 0.26	82/ 86	0.30/ 0.30	100/ 100
	30	0.33/ 0.33	107/ 109	0.26/ 0.25	83/ 80
	60	0.25/ 0.25	94/ 81	0.23/ 0.24	76/ 79
	90	0.22/ 0.21	71/ 73	0.28/ 0.31	96/ 98
	150	0.25/ 0.28	93/ 85	0.28/ 0.29	85/ 81
	210	0.26/ 0.25	86/ 85	0.24/ 0.24	79/ 79
	300	0.30/ 0.29	114/ 113	0.27/ 0.26	92/ 98
	360	0.28/ 0.28	103/ 104	0.33/ 0.33	105/ 109
	400	0.26/ 0.26	93/ 95	0.29/ 0.31	106/ 107

Animal matrices

Aliquots of homogenized bovine muscle, liver, kidney, fat, milk and hens eggs were fortified with DPX-KJM44, aminocyclopyrachlor or IN-LXT69 at 0.2 mg/kg (Roberts and Ward 2010 26273). All samples were stored at approximately -20 °C prior to analysis after approximate intervals of 1 week, 2 weeks, 1, 2, 3 and 5 months, with the exception of kidney which was not analysed at 5 months. In addition, extracts of liver and kidney were stored at approximately -20 °C for ca. 7 and 14 days.

The freezer stability of DPX-KJM44, aminocyclopyrachlor and IN-LXT69 was demonstrated in milk, muscle, fat and hens eggs for at least 133 days. Aminocyclopyrachlor and IN-LXT69 were

stable in liver and kidney for at least 147 and 88 days, respectively. DPX-KJM44 was not stable in liver and kidney, being converted to aminocyclopyrachlor either during storage or subsequent analysis. However, DPX-KJM44 was stable in liver and kidney extracts stored at *ca.* -20 °C for at least 14 days.

Table 21 Storage stability results for egg, milk and tissue samples spiked with DPX-KJM44, aminocyclopyrachlor or IN-LXT69

Matrix	Storage Interval (days)	DPX-KJM44 (mg/kg)	Procedural Recovery (%)	aminocyclopyrachlor (mg/kg)	Procedural Recovery (%)	IN-LXT69 (mg/kg)	Procedural Recovery (%)
Milk	0	0.22/ 0.22	98/ 101	0.18/ 0.18	87/ 94	0.22/ 0.21	99/ 106
	10	0.22/ 0.22	106/ 112	0.19/ 0.18	91/ 98	0.21/ 0.22	102/ 111
	19	0.21/ 0.21	105/ 106	0.18/ 0.18	98/ 95	0.20/ 0.21	108/ 103
	31	0.20/ 0.19	101	0.18/ 0.16	96	0.24/ 0.20	101
	59	0.19/ 0.19	96/ 95	0.19/ 0.19	97/ 106	0.21/ 0.21	106/ 107
	94	0.20/ 0.21	103/ 104	0.19/ 0.19	91/ 91	0.19/ 0.19	96/ 99
	146	0.19/ 0.20	111/ 108	0.20/ 0.18	100/ 101	0.20/ 0.20	101/ 98
	138 ^a	0.23/ 0.24	111/ 125	0.16/ 0.15	71/ 82	0.17/ 0.16	84/ 86
muscle	0	0.23/ 0.24	111/ 125	0.16/ 0.15	71/ 82	0.17/ 0.16	84/ 86
	7	0.18/ 0.17	89/ 91	0.15/ 0.16	82/ 84	0.14/ 0.16	78/ 73
	13	0.17/ 0.18	97/ 94	0.17/ 0.19	89/ 92	0.17/ 0.17	83/ 90
	39	0.18/ 0.18	97/ 102	0.20/ 0.26	104/ 105	0.10/ 0.18	87/ 99
	55	0.15/ 0.16	86/ 90	0.12/ 0.17	103/ 100	0.16/ 0.14	88/ 91
	83	0.14/ 0.14	80/ 76	0.15/ 0.16	80/ 80	0.15/ 0.15	77/ 68
	138 ^a	0.02/ 0.02	9/ 9	0.18/ 0.18	100/ 98	0.04/ 0.04	19/ 20
	138 ^a	0.02/ 0.02	9/ 9	0.18/ 0.18	100/ 98	0.04/ 0.04	19/ 20
liver	0	0.17/ 0.18	93/ 91	0.18/ 0.18	97/ 99	0.19/ 0.19	93/ 93
	9	0.09/ 0.09	90/ 91	0.17/ 0.16	88/ 90	0.17/ 0.17	91/ 89
	16	0.17/ 0.15	92/ 83	0.17/ 0.14	103/ 107	0.19/ 0.18	98/ 96
	28	0.15/ 0.18	95/ 92	0.18/ 0.20	119/ 113	0.19/ 0.19	99/ 101
	57	0.18/ 0.19	97/ 96	0.20/ 0.20	133/ 130	0.16/ 0.17	106/ 104
	91	0.18/ 0.17	92/ 92	0.19/ 0.19	101/ 95	0.13/ 0.16	77/ 82
	147	0.18/ 0.17	93/ 90	0.19/ 0.21	95/ 107	0.21/ 0.21	101/ 105
	147	0.18/ 0.17	93/ 90	0.19/ 0.21	95/ 107	0.21/ 0.21	101/ 105
kidney	0	0.15/ 0.15	90/ 84	0.18/ 0.17	92/ 92	0.17/ 0.17	92/ 89
	8 ^b	0.11/ 0.11	96/ 94	0.18/ 0.18	92/ 90	0.18/ 0.17	85/ 85
	14 ^c	0.11/ 0.13	99/ 97	0.20/ 0.19	96/ 100	0.22/ 0.21	97/ 96
	26 ^d	0.11/ 0.13	102/ 99	0.21/ 0.22	108/ 108	0.18/ 0.17	102/ 93
	60	0.18/ 0.18	102/ 103	0.20/ 0.21	115/ 114	0.16/ 0.16	87/ 92
	88	0.16/ 0.17	92/ 93	0.20/ 0.22	93/ 94	0.15/ 0.15	75/ 76
	147	0.16/ 0.17	92/ 93	0.20/ 0.22	93/ 94	0.15/ 0.15	75/ 76
	147	0.16/ 0.17	92/ 93	0.20/ 0.22	93/ 94	0.15/ 0.15	75/ 76
Fat ^f	0	0.25/ 0.19	92/ 91	0.18/ 0.17	81/ 83	0.22/ 0.22	95/ 104
	12	0.20/ 0.20	104/ 97	0.18/ 0.18	96/ 91	0.19/ 0.20	97/ 92
	19	0.18/ 0.18	92/ 86	0.18/ 0.19	102/ 90	0.19/ 0.18	100/ 92
	46	0.20/ 0.21	102/ 104	0.20/ 0.21	113/ 116	0.22/ 0.22	111/ 118
	60	0.21/ 0.20	108/ 105	0.17/ 0.18	101/ 102	0.19/ 0.19	104/ 104
	95	0.20/ 0.20	97/ 97	0.18/ 0.15	98/ 97	0.18/ 0.17	97/ 93
	147	0.21/ 0.21	102/ 96	0.18/ 0.19	103/ 97	0.17/ 0.18	94/ 88
	147	0.21/ 0.21	102/ 96	0.18/ 0.19	103/ 97	0.17/ 0.18	94/ 88
egg	0	0.20/ 0.19	100/ 96	0.18/ 0.17	89/ 94	0.19/ 0.19	97/ 100
	6	0.21/ 0.28	104/ 105	0.18/ 0.18	95/ 95	0.20/ 0.18	96/ 96
	18	0.19/ 0.20	101/ 103	0.18/ 0.16	100/ 101	0.17/ 0.20	102/ 107
	33	0.19/ 0.20	108/ 108	0.20/ 0.18	107/ 106	0.21/ 0.21	112/ 119
	46	0.19/ 0.19	104/ 103	0.19/ 0.18	100/ 100	0.20/ 0.20	108/ 110
	81	0.19/ 0.18	99/ 99	0.17/ 0.18	88/ 89	0.18/ 0.18	94/ 91
	133 ^e	0.16/ 0.13	89/ 87	0.19/ 0.18	101/ 99	0.21/ 0.19	100/ 99
	133 ^e	0.16/ 0.13	89/ 87	0.19/ 0.18	101/ 99	0.21/ 0.19	100/ 99

^a DPX-KJM44 muscle 138 d also contained 0.012 and 0.01 mg/kg aminocyclopyrachlor. No evidence of significant hydrolysis. Possible incorrect fortification. In addition, the procedural recoveries for DPX-KJM44 and IN-LXT69 were very low indicating an issue with the fortification of the muscle samples. Data for muscle day 138 should be discarded.

^b DPX-KJM44 kidney 8 d, also contained 0.068 and 0.074 mg/kg aminocyclopyrachlor indicating significant hydrolysis of DPX-KJM44 either during storage or during sample analysis

^c DPX-KJM44 kidney 14 d, also contained 0.070 and 0.060 mg/kg aminocyclopyrachlor indicating significant hydrolysis of DPX-KJM44 either during storage or during sample analysis

^d DPX-KJM44 kidney 26 d, also contained 0.088 and 0.086 mg/kg aminocyclopyrachlor indicating significant hydrolysis of DPX-KJM44 either during storage or during sample analysis

^e DPX-KJM44 egg 133 d, also contained 0.026 and 0.02 mg/kg aminocyclopyrachlor indicating significant hydrolysis of DPX-KJM44 either during storage or during sample analysis

^f storage intervals are for aminocyclopyrachlor and IN-LXT69. Storage intervals for DPX-KJM44 were 0, 6, 13, 40, 54, 89 and 141 days.

USE PATTERNS

Aminocyclopyrachlor is a broad-spectrum herbicide that is rapidly absorbed by leaves and roots and translocated to the meristematic regions of the plant, where it is thought to act as an auxin mimic. Aminocyclopyrachlor is active on most broadleaf weeds and brush species with selectivity to most grasses and is used for the control of a range of broadleaf weeds in pasture, rangeland and industrial non-crop situations.

Table 22 Selected registered uses of aminocyclopyrachlor

Crop	Country	Formulation a		Application				PHI (days)
		g ai/L or g ai/kg	type	Method	Rate g ai/ha	Water L/ha	No	
Pasture, rangeland, industrial non-crop, industrial grassed	Canada	50%	WG	Broadcast	70 (max 70/season)	200 grd 30–50 air	1	0 b, c
Rangeland, non-crop	Canada	39.5% + 12.6% metsulfuron-methyl	WG	Broadcast	264 (max 264/season)	≤ 2000 grd 30–50 air	1	0 b, c
Pasture, rangeland, industrial non-crop	Canada	35.3% + 17.6% metsulfuron-methyl	WG	Broadcast	60	200 grd 30–50 air	1	0 b, c

^a Product labels require the use of adjuvants: non-ionic surfactant (NIS) at 0.25% v/v or Merge Adjuvant at 1% v/v or crop oil concentrate at 1% v/v.

^b There are no grazing or haying restrictions for non-lactating or lactating animals (including cattle, horses, sheep and goats). Grazing animals do not have to be moved off pasture or rangeland before, during or after applying aminocyclopyrachlor.

^c If range or pasture or non-crop sites treated with aminocyclopyrachlor are to be converted to a food, feed, or fibre agricultural crop, or to a horticultural crop, a field bioassay should be completed before planting the desired crop.

grd = For application using ground based equipment

air = For application using aircraft

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Aminocyclopyrachlor is a non-selective herbicide that is highly active against growing weeds. The Meeting received information on supervised field trials for aminocyclopyrachlor on the following crops or crop groups:

Commodity	Table No.
Grass forage	Tables 24, 25
Grass hay	Tables 26, 27

Trials were generally well documented with laboratory and field reports. Laboratory reports included method validation with procedural recoveries from spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Control samples are indicated in the summary tables with a "c". Unless stated otherwise, residue data are recorded unadjusted for recovery.

The application rates for DPX-KJM44 and aminocyclopyrachlor are expressed in terms of acid equivalents (i.e., aminocyclopyrachlor).

Residues and application rates have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Residue values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. Those results included in the evaluation are underlined.

Conditions of the supervised residue trials were generally well reported in detailed field reports. Trial designs used non-replicated plots. Field reports provided data on the sprayers used, plot size, field sample size and sampling date.

Table 23 Summary of sprayers, plot sizes and field sample sizes in the supervised trials (Shepard 2010c 24323)

Location	Year	Sprayer	Plot size	Sample size	Sample to analysis interval (days)
New Glasgow, PEI Canada	2008	CO ₂ hand sprayer	20×4 m	Grass 1.1–1.2 kg hay 0.62–0.75 kg	336–376
Paris, ON, Canada	2008	CO ₂ backpack sprayer	20×3 m	Grass 1.3–1.4 kg hay 0.48–0.60 kg	358–400
Elora, ON, Canada	2008	Tractor mounted boom	30×8 m 30×5 m	Grass 1.0–1.2 kg Hay 2.1–2.4 kg	142–249
Tipton, MO, USA	2008	Tractor mounted boom	9.1×15.2 m	Grass 1.5–1.9 kg Hay 0.7–0.8 kg	286–371
Springfield, NE, USA	2008	CO ₂ backpack sprayer	6.1×15.2 m	Grass 1 kg Hay 0.5 kg	290–372
East Bernard, TX, USA	2008	CO ₂ backpack sprayer	11.0×45.7 m	Grass 1.0–1.2 kg Hay 0.5–0.8 kg	273–401
Hinton, OK, USA	2008	CO ₂ backpack sprayer	7.3×12.2 m	Grass 2.2 kg Hay 1.1 kg	247–331
Frederick, SD, USA	2008	CO ₂ backpack sprayer	6.1×18.3 m	Grass 1.0–1.2 kg Hay 0.5–0.7 kg	274–365
Jamestown, ND, USA	2008	CO ₂ backpack sprayer	6.1×12.2 m	Grass 1.0 kg Hay 0.5 kg	287–379
Uvalde, TX, USA	2008	CO ₂ backpack sprayer	6.1×21.3 m	Grass 1.5–3.2 kg Hay 1–2 kg	295–380
Lamed, KS, USA	2008	CO ₂ backpack sprayer	6.1×15.2 m 5.5×15.2 m	Grass 1.0–1.4 kg Hay 0.5–0.6 kg	282–344
Saskatoon, SK, Canada	2008	Side mounted tractor boom	4×25 m	Grass 1.2–1.6 kg Hay 1.1–1.3 kg	297–360
Hepburn, SK, Canada	2008	Tractor mounted boom	8×50 m	Grass 1.1–2.1 kg Hay 0.8–1.4 kg	257–397
Innisfail, AB, Canada	2008	Side mounted tractor boom	4×25 m	Grass 0.7–0.8 kg Hay 0.5–0.7 kg	278
Innisfail, AB, Canada	2008	Side mounted tractor boom	4×25 m	Grass 1.3–1.8 kg Hay 0.6 kg	273–377
Minto, MB, Canada	2008	Side mounted tractor boom	8×29 m	Grass 1.1 kg Hay 0.7–0.8 kg	183–300
St Andrew, MB, Canada	2008	Tractor mounted boom	3×20 m	Grass 1.0 kg Hay 0.3–0.5 kg	272–365
Arborg, MB, Canada	2008	ATV offset mounted boom	3×20 m	Grass 1.1–1.2 kg Hay 0.12–0.54 kg	268–360
Germansville, PA, USA	2008	CO ₂ backpack sprayer	6.1×9.1 m	Grass 1.0–1.5 kg Hay 0.6–0.8 kg	38–97
Germansville, PA, USA	2008	CO ₂ backpack sprayer	6.1×9.1 m	Grass 1.0–1.5 kg Hay 0.5–0.9 kg	38–55
Frenchtown, NJ, USA	2008	CO ₂ backpack sprayer	6.1×7.6 m	Grass 1.3 kg Hay 0.5	37–65
Richland, IA, USA	2008	CO ₂ backpack sprayer	15.2×12.2 m	Grass 1.0–1.2 kg Hay 0.7–0.9 kg	40–44

Where duplicate field samples from an un-replicated plot were taken at each sampling time and were analysed separately, the mean of the two analytical results was taken as the best estimate of the residues in the plot and only the means are recorded in the tables. Similarly where samples were collected from replicate plots the mean result is reported (see general consideration JMPR 2010).

Animal feeds**Grass**

Twenty-two supervised residue trials with aminocyclopyrachlor or DPX-KJM44 on grass pastures were conducted in Canada and the USA in 2008 and 2009 (Shepard 2010c 24323). In these trials aminocyclopyrachlor (SL formulation) or DPX-KJM44 (WG formulations) were applied once at rates of 303–335 g ai/ha to pasture. All applications included an adjuvant at the recommended rate. Two replicate samples were collected from each treated plot. Samples of grass and hay were stored for a maximum of 401 days. Residues of aminocyclopyrachlor, DPX-KJM44, IN-LXT69, IN-QFH57 and IN-QGC48 samples were determined using method 22528. Mean concurrent recoveries for the analytes in samples fortified at 0.01 to 120 mg/kg ranged from $83 \pm 11\%$ to $95 \pm 9\%$ per analyte/matrix combination ($n = 32$ to 39).

Table 24 Application of DPX-KJM44 to grass: Residues of DPX-KJM44, aminocyclopyrachlor and IN-LXT69 (mg/kg, mean of two replicate samples) in grass forage (rates expressed in terms of aminocyclopyrachlor equivalents)

Location, year, variety	Form	g ai/ha ^e	l/ha	GS	DAT (days) ^a	DPX-KJM44	Aminocyclopyrachlor	IN-LXT69	Moisture (%)
New Glasgow, PEI, Canada, 2008, Timothy	WG	335	209	BBCH 41	0	14 ^b	1.1 ^b	0.007 ^b	75.4
Paris, ON, Canada, 2008, Bromegrass	WG	315	200	boot	0	10	0.77	0.004	77.6
Elora, ON, Canada, 2008, Bromegrass breeder-seed EXP	WG	316	200	BBCH 39–41	0	34	1.5	0.018	60.4
	WG	308	9	BBCH 39–41	0	9.9	0.75	0.007	
Tipton, MO, USA, 2008, fescue	WG	317	258	Stem elongation	0	8.8	1.0	0.005	70.6
Springfield, NE, USA, 2008, fescue K-31	WG	311	130	23 cm	0	29	5.6	0.017	77.1
East Bernard, TX, USA, 2008, coastal Bermuda grass	WG	308	136	boot	–0	< 0.01	0.01	< 0.01	
					0	24	4.3	0.013	69.9
					3	4.6	8.9	0.004	
					7	1.8	7.7	< 0.01	
					14	0.099	5.6	0.004	58.0
					21	0.05	3.6	0.004	
Hinton, OK, USA, 2008, Bermuda grass	WG	310	131	boot	0	16	1.9	0.01	69.3
Frederick, SD, USA, 2008, bluegrass/smooth bromegrass	WG	317	94	85–90% pre-boot	0	29	4.0	0.018	58.0
Jamestown, ND, USA, 2008, bromegrass	WG	309	140	BBCH 37	0	14	3.1	0.01	70.5
Uvalde, TX, USA, 2008, Bermuda grass	WG	317	188	BBCH 45	0	29	3.2	0.017	53.1
Larned, KS, USA, 2008, Bermuda grass	WG	317	187	BBCH 45	0	26	1.9	0.02	65.3
2008, Bermuda grass	WG	303	9	BBCH 45	0	11	0.53	0.006	

Location, year, variety	Form	g ai/ha ^c	l/ha	GS	DAT (days) ^a	DPX-KJM44	Aminocyclopyrachlor	IN-LXT69	Moisture (%)
grass									
Saskatoon, SK, Canada, 2008, brome grass	WG	314	200	BBCH 32-37	0	27	2.3	0.012	69.6
Hepburn, SK, Canada, 2008, meadow brome grass	WG	307	195	BBCH 32-37	0	< 0.01	< 0.01	< 0.01	
					0	13	2.9	0.007	77.2
					3	3.9	2.8	< 0.01	
					7	0.4	2.7	< 0.01	
					14	0.15	1.6	< 0.01	73
					21	0.027	0.92	< 0.01	
Innisfail, AB, Canada, 2008, Timothy climax	WG	319	200	BBCH 33-34	0 ^c	18	0.94	0.009	74.2
Innisfail, AB, Canada, 2008, Timothy drummond	WG	326	205	BBCH 32-34	0 ^c	18	1.8	0.01	74.8
Minto, MB, Canada, 2008, meadow brome grass 52%, fleet and crested wheat grass 48% Fairway	WG	310	158	6 leaves, 20 tillers	20	26	2.2	0.011	78.0
St. Andrew, MB, Canada, 2008, meadow fescue	WG	318	280	BBCH 39-40	0	13	2.0	0.008	76.6
Arborg, MB, Canada, 2008, Barrelex tall fescue	WG	317	281	Early boot	0	14	1.1	0.007	80.9
	WG	314	9	Early boot	0	13	0.71	0.006	
Germansville, PA, USA, 2008, fescue triplet mix bravo, Biltmore, Laramie	WG	309	234	Pre-boot	0 ^d	38	5.6	0.02	75.6
Germansville, PA, USA, 2008, bluegrass-unique midnight Blacksburg	WG	321	234	Pre-boot	0 ^d	47	6.6	0.028	72.2
Frenchtown, NJ, USA, 2008, tall fescue Rebel	WG	325	257	Pre-head 6-8 in	0	37	13	0.02	77.8
Richland, IA, USA, 2008, Kentucky blue grass	WG	315	181	Pre-boot	0	20	2.3	0.011	78.3

^a DAT = days after last application (days between last application and sampling)

^b Comparison of residues support the conclusion that control and replicate A samples were inadvertently switched. The values reported have been corrected to account for this.

^c plot locations are the same with spray applications within one week for the two plots

^d plot locations are the same with spray applications within one week for the two plots

^e Adjuvants added to spray solutions are listed in order of trials in the table: 1. Agral 90 0.05% and soluble urea 2.5%; 2. Agral 90 0.05% and 28-0-0 1.0%; 3. Agral 90 0.2%, UAN 2.5%; 4. 80-20 0.25% and 28% N 1 qt/acre; 5. MSO 10% and UAN 2.5%; 6. Induce 0.25%, 32-0-0 0.25%; 7. Baron 0.9%, AMS 2.0%; 8. Preference 0.25%, N-Pa-K28 3.7%; 9. Activator 90 0.25%, 28-0-0 0.25%; 10. Dyne-Amic 0.23%, 32% liquid nitrogen 2.2%; 11. Spreader 90 0.25%, 32%UAN 0.125%; 12 Ag-Surf 0.2%, 28-0-0 0.25%; 13 Ag-Surf 0.2%, 28-0-0 0.25%; 14 Ag-Surf 0.2%, 28-0-0 0.25%; 15 Ag-Surf 0.2%, 28-0-0 0.25%; 16 Ag-Surf 0.2%, 28-0-0 0.25%; 17. Merge 0.5%, 28-0-0 0.25%; 18. Merge 0.5%, 28-0-0 0.25%; 19. Induce 0.25%, UAN 2.5%; 20. Induce 0.25%, UAN 2.5%; 21. Induce 0.25%, urea 2.5%; 22 Preference 0.25%, UAN 1 qt/ac.

Table 25 Application of aminocyclopyrachlor to grass: Residues of DPX-KJM44, aminocyclopyrachlor and IN-LXT69 (mg/kg, mean of two replicate samples) in grass forage

Location, year, variety	Form	g ai/ha	L/ha	GS	DAT (days) ^a	Aminocyclopyrachlor	IN-LXT69	DPX-KJM44	Moisture (%)
Paris, ON, Canada 2008, Bromegrass	SL	308	200	Boot	0	13	< 0.01	0.027	77.6
Hinton, OK, USA, 2008, Bermuda grass	SL	311	131	Boot	0	22	0.019	0.028	69.3
Frederick, SD, USA, 2008, bluegrass/ smooth bromegrass	SL	317	94	85–90% pre-boot	0	25	0.02	0.026	58.0
Uvalde, TX, USA, 2008, Bermuda grass	SL	319	190	BBCH 45	0	39	0.035	0.14	53.1
Hepburn, SK, Canada, 2008, meadow bromegrass	SL	310	197	BBCH 32–37	-0	< 0.01	< 0.01	< 0.01	
					0	18	0.004	0.016	77.2
					3	9.0	0.008	0.016	
					7	4.7	0.004	0.034	
					14	2.6	< 0.01	0.026	73
					21	1.2	< 0.01	0.024	
St. Andrew, MB, Canada, 2008, meadow fescue	SL	317	280	BBCH 39–40	0	18	0.006	0.062	76.6

^a DAT = days after last application (days between last application and sampling)

^b Trials are conducted in association with trials using DPX-KJM44. Residues of DPX-KJM44 are thought to arise from movement of DPX-KJM44 vapour from DPX-KJM44 treated plots to aminocyclopyrachlor treated plots.

^c Adjuvants added to spray solutions are listed in order of trials in the table: 1. Agral 90 0.05% and 28-0-0 1.0%; 2. Baron 0.9%, AMS 2.0%; 3. Preference 0.25%, N-Pa-K28 3.7%; 4. Dyne-Amic 0.23%, 32% liquid nitrogen 2.2%; 5 Ag-Surf 0.2%, 28-0-0 0.25%; 6. Merge 0.5%, 28-0-0 0.25%;.

No residues of IN-QGC48 were detected while only occasional trace (< LOQ) residues of IN-QFH57 were detected.

Table 26 Application of DPX-KJM44 to grass: Residues of DPX-KJM44, aminocyclopyrachlor and IN-LXT69 (mg/kg, mean of two replicate samples) in grass hay (rates expressed in terms of aminocyclopyrachlor equivalents)

Location, year, variety	Form	g ai/ha	L/ha	GS	DALA (days) ^a	DPX-KJM44	Aminocyclopyrachlor	IN-LXT-69	Moisture (%)
New Glasgow, PEI, Canada, 2008, Timothy	WG	335	209	BBCH 41	0 + 8	29	15	0.018	37.9
Paris, ON, Canada, 2008, Bromegrass	WG	315	200	boot	0 + 10	30	3.9	0.012	34.6
Elora, ON, Canada, 2008, Bromegrass breeder-seed EXP	WG	316	200	BBCH 39–41	0 + 18	34	4.5	0.018	19.2
	WG	308	9	BBCH 39–41	0 + 18	23	2.4	0.014	
Tipton, MO, USA, 2008, fescue	WG	317	258	Stem elongation	0 + 2	25	2.6	0.014	25.2
Springfield, NE, USA, 2008, fescue K-31	WG	311	130	23 cm	0 + 4	41	35	0.037	20.2

Location, year, variety	Form	g ai/ha	L/ha	GS	DALA (days) ^a	DPX-KJM44	Aminocyclopyrachlor	IN-LXT-69	Moisture (%)
East Bernard, TX, USA, 2008, coastal Bermuda grass	WG	308	136	boot	0 + 4	0.042	0.013	< 0.01	
					0 + 4	46	16	0.029	19.0
					3 + 5	9.2	20	0.01	
					7 + 4	2.8	13	0.006	
					14 + 4	0.21	9.1	0.01	15.2
					21 + 3	0.13	6.4	0.006	
Hinton, OK, USA, 2008, Bermuda grass	WG	310	131	Boot	0 + 3	31	7.1	0.022	23.4
Frederick, SD, USA, 2008, bluegrass/ smooth brome grass	WG	317	94	85–90% pre-boot	0 + 3	38 ^b	12 ^B	0.03 ^B	12.8
Jamestown, ND, USA, 2008, brome grass	WG	309	140	BBCH 37	0 + 2	21	9.2	0.015	28.3
Uvalde, TX, USA, 2008, Bermuda grass	WG	317	188	BBCH 45	0 + 2	45	6.2	0.028	6.6
Larned, KS, USA, 2008, Bermuda grass	WG	317	187	BBCH 45	0 + 1	46	4.1	0.028	15.0
Saskatoon, SK, Canada, 2008, brome grass	WG	314	200	BBCH 32–37	0 + 0	33	17	0.025	13.6
Hepburn, SK, Canada, 2008, meadow brome grass	WG	307	195	BBCH 32–37	0 + 11	0.005	0.007	< 0.01	
					0 + 25	14	22	0.021	11.8
					3 + 22	2.5	15	0.008	
					7 + 18	0.57	9.7	0.005	
					14 + 28	0.031	4.9	< 0.01	10.4
					21 + 21	0.038	3.1	< 0.01	
Innisfail, AB, Canada, 2008, Timothy climax	WG	319	200	BBCH 33–34	0 + 7 ^c	41	16	0.03	25.3
Innisfail, AB, Canada, 2008, Timothy drummond	WG	326	205	BBCH 32–34	0 + 9 ^c	34	24	0.024	21.7
Minto, MB, Canada, 2008, meadow brome grass 52%, fleet and crested wheatgrass 48% Fairway	WG	310	158	6 leaves, 2 tillers	0 + 7	68	11	0.028	23.0
St. Andrew, MB, Canada, 2008, meadow fescue	WG	318	280	BBCH 39–40	0 + 20	14	22	0.016	18.1
Arborg, MB, Canada, 2008, Barrelex tall fescue	WG	317	281	Early boot	0 + 14	15	27	0.014	26.7
	WG	314	9	Early boot	0 + 14	27	12	0.016	
Germansville, PA, USA, 2009, fescue triplet mix bravo, Biltmore, Laramie	WG	309	234	Pre-boot	0 + 2 ^d	102	26	0.075	17.9
Germansville, PA, USA, 2009,	WG	321	234	Pre-boot	0 + 2 ^d	71	24	0.053	10.4

Location, year, variety	Form	g ai/ha ^c	L/ha	GS	DAL (days) ^a	DPX-KJM44	Aminocyclopyrachlor	IN-LXT-69	Moisture (%)
bluegrass-unique midnight Blacksburg									
Frenchtown, NJ, USA, 2008, tall fescue Rebel	WG	325	257	Pre-head 6–8 in	0 + 3	36	79	0.031	28.0
Richland, IA, USA, 2008, Kentucky blue grass	WG	315	181	Pre-boot	0 + 8	28	17	0.02	27.0

^a DAT = days after last application (days between last application and sampling) + interval between cutting and sampling (windrowing time)

^b Comparison of residues support the conclusion that control and replicate A samples were inadvertently switched. The values reported have been corrected to account for this.

^c plot locations are the same with spray applications within one week for the two plots

^d plot locations are the same with spray applications within one week for the two plots

^e Adjuvants added to spray solutions are listed in order of trials in the table: 1. Agral 90 0.05% and soluble urea 2.5%; 2. Agral 90 0.05% and 28-0-0 1.0%; 3. Agral 90 0.2%, UAN 2.5%; 4. 80-20 0.25% and 28% N 1 qt/acre; 5. MSO 10% and UAN 2.5%; 6. Induce 0.25%, 32-0-0 0.25%; 7. Baron 0.9%, AMS 2.0%; 8. Preference 0.25%, N-Pa-K28 3.7%; 9. Activator 90 0.25%, 28-0-0 0.25%; 10. Dyne-Amic 0.23%, 32% liquid nitrogen 2.2%; 11. Spreader 90 0.25%, 32%UAN 0.125%; 12 Ag-Surf 0.2%, 28-0-0 0.25%; 13 Ag-Surf 0.2%, 28-0-0 0.25%; 14 Ag-Surf 0.2%, 28-0-0 0.25%; 15 Ag-Surf 0.2%, 28-0-0 0.25%; 16 Ag-Surf 0.2%, 28-0-0 0.25%; 17. Merge 0.5%, 28-0-0 0.25%; 18. Merge 0.5%, 28-0-0 0.25%; 19. Induce 0.25%, UAN 2.5%; 20. Induce 0.25%, UAN 2.5%; 21. Induce 0.25%, urea 2.5%; Preference 0.25%, UAN 1 qt/ac.

Table 27 Application of aminocyclopyrachlor to grass: Residues of DPX-KJM44, aminocyclopyrachlor and IN-LXT69 (mg/kg, mean of two replicate samples) in grass hay (rates expressed in terms of aminocyclopyrachlor equivalents)

Location, year, variety	Form	g ai/ha ^c	L/ha	GS	DAT (days) ^a	Aminocyclopyrachlor	IN-LXT-69	DPX-KJM44 ^b	Moisture (%)
Paris, ON, Canada, 2008, Bromegrass	SL	308	200	Boot	0 + 10	<u>30</u>	0.011	0.2	34.6
Hinton, OK, USA, 2008, Bermuda grass	SL	311	131	Boot	0 + 3	<u>46</u>	0.08	0.11	23.4
Frederick, SD, USA, 2008, bluegrass/ smooth bromegrass	SL	317	94	85–90% pre-boot	0 + 3	<u>35</u>	0.05	0.084	12.8
Uvalde, TX, USA, 2008, Bermuda grass	SL	319	190	BBCH 45	0 + 2	<u>46</u>	0.11	0.22	6.6
Hepburn, SK, Canada, 2008, meadow bromegrass	SL	310	197	BBCH 32–37	–0 + 11	< 0.01	< 0.01	< 0.01	
					0 + 25	<u>40</u>	0.03	0.08	11.8
					3 + 22	25	0.021	0.081	
					7 + 18	12	0.012	0.082	
					14 + 28	5.9	0.005	0.035	10.4
					21 + 21	3.7	< 0.01	0.045	
St. Andrew, MB, Canada, 2008, meadow fescue	SL	317	280	BBCH 39–40	0 + 20	<u>48</u>	0.015	0.72	18.1

^a DAT = days after last application (days between last application and sampling) + interval between cutting and sampling (windrowing time)

^b Trials are conducted in association with trials using DPX-KJM44. Residues of DPX-KJM44 are thought to arise from movement of DPX-KJM44 vapour from DPX-KJM44 treated plots to aminocyclopyrachlor treated plots.

^c Adjuvants added to spray solutions are listed in order of trials in the table: 1. Agral 90 0.05% and 28-0-0 1.0%; 2. Baron 0.9%, AMS 2.0%; 3. Preference 0.25%, N-Pa-K28 3.7%; 4. Dyne-Amic 0.23%, 32% liquid nitrogen 2.2%; 5 Ag-Surf 0.2%, 28-0-0 0.25%; 6. Merge 0.5%, 28-0-0 0.25%.

No residues of IN-QGC48 were detected while only occasional trace (< LOQ) residues of IN-QFH57 were detected.

PRIMARY FEED COMMODITIES OF PLANT ORIGIN

Livestock feeding studies

Dairy cow feeding study

The transfer of aminocyclopyrachlor-methyl (DPX-KJM44) residues from feed to tissues and milk of dairy cows was studied by Roberts and Ward (2010, 26273).

DPX KJM44 was administered orally to four groups of three Holstein/Friesian cattle (4–9 yrs old; 504–704 kg bw) by gelatine capsule (with one control cow fed capsules only) for 28 days. Mean daily feed consumption during the exposure period was 14.3–16.4 kg DM (hay, *ad libitum* and 8 kg/day protein concentrate). Mean daily milk yield during the exposure period was 8.1 to 29 kg/cow/day. Based on mean daily feed consumption, the exposure was equivalent to 73, 160, 454 and 1594 ppm in the feed (1.9, 3.8, 11.4, and 37.9 mg/kg bw/day). Milk was collected twice daily (pm sampling pooled with am sampling the next day) at 11 intervals through the 28 days of dosing. Selected samples were analysed for residues of DPX-KJM44 and aminocyclopyrachlor and a metabolite, IN-LXT69. Cream and skim milk were produced from selected milk samples taken at two intervals after milk residue plateau and these samples were analysed for DPX-KJM44, aminocyclopyrachlor and IN-LXT69. Muscle, liver, kidney and fat samples were collected at sacrifice 22–24 hours after the last dose; 14–16 days in the case of the depuration animals. Due to potential instability of DPX-KJM44 in frozen storage, liver and kidney samples were extracted shortly after sacrifice. Milk, skim milk and cream samples were all analysed within approximately 2 months of collection. The maximum frozen storage intervals were 65 days for milk, 30 days for skim milk and 59 days for cream. The maximum storage intervals for tissues were 44 and 36 days for muscle and fat, respectively. Liver and kidney samples were extracted on the day of collection with the exception of selected repeat samples.

Table 28 Concurrent recovery results for DPX-KJM44, aminocyclopyrachlor and IN-LXT69 for the dairy cow feeding study

Matrix	Fort level (mg/kg)	N	DPX-KJM44		aminocyclopyrachlor		IN-LXT69	
			Recoveries (%)		Recoveries (%)		Recoveries (%)	
			Range	Mean $\pm$ SD	Range	Mean $\pm$ SD	Range	Mean $\pm$ SD
Milk	0.01	31–36	70–116	92 $\pm$ 11	59–115	91 $\pm$ 15	63–126	100 $\pm$ 12
	0.10	31–37	86–124	108 $\pm$ 9	65–118	94 $\pm$ 10	62–113	102 $\pm$ 12
Muscle	0.01	3–4	74–82	77 $\pm$ 4	75–85	80 $\pm$ 5	64–82	75 $\pm$ 8
	0.10	3–4	80–92	85 $\pm$ 5	74–90	83 $\pm$ 9	75–82	79 $\pm$ 3
Liver	0.01	8–11	61–96	77 $\pm$ 11	88–126	107 $\pm$ 12	94–116	107 $\pm$ 8
	0.10	8–11	82–111	90 $\pm$ 8	65–121	102 $\pm$ 18	92–120	103 $\pm$ 9
Kidney ^b	0.01	8–10	85–124	93 $\pm$ 7 ^a	102–242	109 $\pm$ 7 ^a	90–110	101 $\pm$ 8
	0.10	8–10	79–113	93 $\pm$ 13	87–107	99 $\pm$ 6	92–110	100 $\pm$ 7
Fat ^b	0.01	3–4	84–93	87 $\pm$ 4	68–89	77 $\pm$ 11	91–102	95 $\pm$ 5
	0.10	4	97–102	99 $\pm$ 2	86–100	93 $\pm$ 6	81–105	95 $\pm$ 10

^a Outliers (recovery values > 160%) omitted from calculations

^b Note: There were 2 additional aminocyclopyrachlor kidney and fat fortifications, each, at 2 mg/kg (96 and 99% and 97 and 104% recoveries for kidney and fat, respectively).

Average aminocyclopyrachlor milk residues at each sampling interval are presented below for the two dose rates that routinely had milk residues above the LOQ.

Table 29 Daily average residues of aminocyclopyrachlor in milk of cows dosed with DPX-KJM44.

DPX-KJM44 dose	Average residue (mg/kg) in milk at various sampling intervals (days)									
	1	3	5	7	10	14	17	21	24	28
454 ppm	0.014	0.017	0.021	0.024	0.019	0.018	0.020	0.021	0.022	0.022
1594 ppm	0.038	0.050	0.055	0.053	0.050	0.053	0.061	0.059	0.071	0.077

These data suggest that residues of aminocyclopyrachlor detected in whole milk reached a plateau by 5 to 7 days of dosing.

The maximum individual milk residue for all groups at each dose level over the 28 days of dosing is summarized below (Table 30).

Table 30 Maximum residues of aminocyclopyrachlor in milk of individual cows dosed with DPX-KJM44

Analyte	Maximum individual cow daily residue in milk in mg/kg			
	73 ppm	160 ppm	454 ppm	1594 ppm
DPX-KJM44	< 0.01	< 0.01	< 0.01	< 0.01
aminocyclopyrachlor	< 0.01	0.012	0.026	0.098
IN-LXT69	< 0.01	< 0.01	< 0.01	< 0.01

The average cream and skim milk residues are presented below.

Table 31 Partitioning of residues of DPX-KJM44, aminocyclopyrachlor and IN-LXT69 in skim milk and cream

Analyte	Average residue in cream and skim milk (mg/kg)				
	Matrix	73 ppm	160 ppm	454 ppm	1594 ppm
Milk day 14					
DPX-KJM44	Cream	ND	ND	ND	ND
	Skim milk	ND	ND	ND	ND
aminocyclopyrachlor	Cream	< 0.01	< 0.01	0.011	0.033
	Skim milk	< 0.01	< 0.01	0.018	0.065
IN-LXT69	Cream	ND	< 0.01	< 0.01	< 0.01
	Skim milk	ND	ND	ND	< 0.01
Milk day 21					
DPX-KJM44	Cream	ND	ND	< 0.01	< 0.01
	Skim milk	ND	ND	ND	ND
aminocyclopyrachlor	Cream	ND	< 0.01	0.012	0.033
	Skim milk	< 0.01	< 0.01	0.019	0.063
IN-LXT69	Cream	ND	< 0.01	< 0.01	< 0.01
	Skim milk	ND	ND	ND	< 0.01

ND = Not detected

Table 32 Residues of DPX-KJM44, aminocyclopyrachlor and IN-LXT69 in tissues of cows dosed with DPX-KJM44

Dose (ppm)	Analyte	residue in tissues in mg/kg (average)			
		Muscle	Fat	Liver	Kidney
73	DPX-KJM44	ND ND ND (ND)	ND < 0.01 ND (< 0.01)	0.014 < 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 < 0.01 (< 0.01)
	aminocyclopyrachlor	< 0.01 ND ND (< 0.01)	0.015 ND < 0.01 (< 0.01)	0.028 0.082 < 0.01 (0.039)	0.092 0.11 0.17 (0.12)
	IN-LXT69	ND ND ND (ND)	ND ND ND (ND)	< 0.01 < 0.01 ND (< 0.01)	ND ND ND (ND)
160	DPX-KJM44	ND < 0.01 ND (< 0.01)	ND ND < 0.01 (< 0.01)	< 0.01 ND < 0.01 (< 0.01)	< 0.01 < 0.01 < 0.01 (< 0.01)
	aminocyclopyrachlor	< 0.01 < 0.01 0.012 (< 0.01)	< 0.01 < 0.01 0.040 (0.015)	0.064 0.02 0.042 (0.042)	0.40 0.29 0.23 (0.31)
	IN-LXT69	ND ND ND (ND)	ND ND ND (ND)	ND < 0.01 ND (< 0.01)	ND ND ND (ND)

454	DPX-KJM44	ND < 0.01 < 0.01 (< 0.01)	ND ND ND (ND)	ND ND ND (ND)	< 0.01 < 0.01 < 0.01 (< 0.01)
	aminocyclopyrachlor	< 0.01 < 0.01 < 0.01 (< 0.01)	0.040 0.029 0.12 (0.062)	0.075 0.025 0.046 (0.049)	0.54 0.20 0.28 (0.34)
	IN-LXT69	< 0.01 < 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 < 0.01 (< 0.01)
1594	DPX-KJM44	ND ND ND (ND)	ND ND ND (ND)	ND ND ND (ND)	< 0.01 < 0.01 < 0.01 (< 0.01)
	aminocyclopyrachlor	0.021 0.10 0.030 (0.051)	0.025 0.61 0.74 (0.46)	0.091 0.088 0.11 (0.096)	0.68 0.85 1.4 (0.98)
	IN-LXT69	< 0.01 < 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 < 0.01 (< 0.01)
1594	DPX-KJM44	ND	ND	ND	< 0.01
(-14 days)	aminocyclopyrachlor	0.019	0.030	< 0.01	< 0.01
	IN-LXT69	ND	ND	ND	ND
1594	DPX-KJM44	ND	ND	ND	ND
(-16 days)	aminocyclopyrachlor	ND	0.056	ND	< 0.01
	IN-LXT69	ND	ND	ND	ND

ND = Not detected.

Aminocyclopyrachlor was the only analyte with residues greater than the LOQ (0.01 mg/kg). In tissue samples obtained within approximately 24 hrs of dose completion, quantifiable aminocyclopyrachlor residues levels were highest in kidney followed by fat, liver and muscle. Where quantifiable, residue levels in fat, liver and kidney were dose dependent.

Following cessation of dosing and where present, residues in milk and tissues rapidly declined. The average residues of DPX-KJM44, aminocyclopyrachlor and IN-LXT69 in milk obtained 1 day post last dose were < 0.01, 0.013 mg/kg and not detected, respectively. These residues declined to non-detectable in milk obtained 7 days post last dose.

Three days after withdrawal of dosing, residues of DPX-KJM44, aminocyclopyrachlor and IN-LXT69 declined to not detected, < 0.01 mg/kg and not detected in cream and not detected, < 0.01 mg/kg and not detected in skim milk, respectively. All residues were not detected 10 days post last dose.

Following a 15 day withdrawal period, all residues of DPX-KJM44 in tissues were not detected with the exception of kidney (< 0.01 mg/kg). Residues of aminocyclopyrachlor were < 0.01, 0.056, < 0.01 and 0.019 mg/kg in kidney, fat, liver and muscle, respectively. No residues of IN-LXT69 were detected.

Following a 17 day withdrawal period, all residues were not detected with the exception of aminocyclopyrachlor in kidney (< 0.01 mg/kg) and fat (0.03 mg/kg)

NATIONAL RESIDUE DEFINITIONS

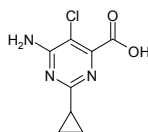
Canada: aminocyclopyrachlor

APPRAISAL

Aminocyclopyrachlor is a new pyrimidine carboxylic acid herbicide used for the control of broadleaf weeds and woody vegetation. Aminocyclopyrachlor mimics the naturally occurring phytohormone indole acetic acid (auxin) disrupting plant growth. At the Forty-fifth Session of the CCPR (2013), it was scheduled for evaluation as a new compound by the 2014 JMPR.

The Meeting received information on the metabolism of aminocyclopyrachlor and also its methyl ester (aminocyclopyrachlor-methyl, DPX-KJM44) in lactating goats and grass, methods of residue analysis, freezer storage stability, GAP information, supervised residue trials, and a cattle feeding study.

Aminocyclopyrachlor is 6-amino-5-chloro-2-cyclopropyl-4-pyrimidinecarboxylic acid (IUPAC).



Metabolites referred to in the appraisal were addressed by their codes:

DPX-KJM44	(methyl 6-amino-5-chloro-2-cyclopropyl-4-pyrimidinecarboxylic acid)	
IN-LXT69	(5-chloro-2-cyclopropyl-pyrimidin-4-ylamine)	
IN-QGC48	(5-cyano-2-cyclopropyl-3H-imidazole-4-carboxylic acid methyl ester)	
IN-QFH57	(5-cyano-2-cyclopropyl-3H-imidazole-4-carboxylic acid)	
IN-Q3007	(cyclopropane carboxamide)	

The methyl ester of aminocyclopyrachlor (DPX-KJM44) was initially explored as a potential herbicide before finally settling on aminocyclopyrachlor (free acid) as the compound to be commercialised. As DPX-KJM44 is rapidly converted to aminocyclopyrachlor in both animals and plants, studies on DPX-KJM44 were submitted to the Meeting to support the evaluation of aminocyclopyrachlor.

Animal metabolism

The current meeting evaluated laboratory animal (rat) metabolism studies of orally administered aminocyclopyrachlor-methyl (DPX-KJM44) and reported that the ester is rapidly converted to the free acid (aminocyclopyrachlor). Therefore, studies with DPX-KJM44 also serve to demonstrate the metabolism of aminocyclopyrachlor.

A lactating goat was orally dosed twice daily for five consecutive days with [pyrimidine-2-¹⁴C]DPX-KJM44 at a dose equivalent to 97 ppm in the feed. Approximately 85% of the administered dose was recovered in the excreta (20% faeces, 54% urine) or gastrointestinal tract (8%). The radioactivity in the tissues ranged from 0.01 mg DPX-KJM44 equivalents/kg in fat to 1.67 mg DPX-KJM44 equivalents/kg in kidney. TRR values in milk reached 0.031 mg DPX-KJM44 equivalents/kg after five days of dosing.

With the exception of one fat sample, greater than 80% of the TRR was extracted with the solvent systems used (acetone for milk and acetonitrile/water for tissues).

No intact DPX-KJM44 was detected in tissues or milk. The major component of the ^{14}C residues were aminocyclopyrachlor (kidney 55% TRR, liver 66% TRR, muscle 43% TRR, fat 47–84% TRR, milk 16% TRR). A number of minor metabolites were unable to be identified but were generally only present at low levels (< 0.01 mg DPX-KJM44 equivalents/kg).

In summary, the metabolism of DPX-KJM44 and aminocyclopyrachlor in goats is similar to metabolism in laboratory animals in the respect that the ester is de-esterified with little further breakdown of the acid (aminocyclopyrachlor).

Plant metabolism

A study on the metabolic fate of DPX-KJM44 in grass was made available to the meeting and a number of studies were located in the literature where aminocyclopyrachlor or DPX-KJM44 were sprayed onto grass and also a variety of weed species. While the literature studies were not conducted according to the protocols developed for submission of data to regulatory authorities, they were used to contribute to the weight of evidence regarding metabolism of aminocyclopyrachlor in plants. As with animal systems, following treatment with DPX-KJM44, rapid degradation occurs with the formation of aminocyclopyrachlor, thus studies with DPX-KJM44 also provide evidence on the metabolism of aminocyclopyrachlor in plants.

Grass

The metabolic fate of [pyrimidine-2- ^{14}C]DPX-KJM44 in mixed grass (30 cm high) was examined following a single foliar application at 373 g ai/ha. Absorption following spraying was rapid with only 13% of the TRR in leaves recovered in surface washes from samples collected on the day of application. DPX-KJM44 was rapidly degraded, representing 25% TRR on the day of application, 14% TRR after three days and less than 9% TRR thereafter. Aminocyclopyrachlor was the major component of the ^{14}C residue comprising 64% TRR at day 0, declining to 33% TRR at 60 days after application. Minor metabolites were IN-LXT69 (4–6% TRR), IN-QGC48 (0–4% TRR), IN-QFH57 (0–2% TRR) and IN-Q3007 (0–1% TRR). Combined, the minor metabolites accounted for no more than 6.1% TRR in individual grass samples.

In a study of [^{14}C]-aminocyclopyrachlor metabolism in tall fescue grass sprayed once at 79 g ai/ha with aminocyclopyrachlor with the addition of 0.25% non-ionic surfactant, absorption from a single leaf treated with [^{14}C]-aminocyclopyrachlor was rapid with only 37% TRR recovered in surface washes 1 day after application. At eight days after application, aminocyclopyrachlor was the only compound detected in solvent extracts of plant material.

The metabolism of DPX-KJM44 and aminocyclopyrachlor was also studied in a range of weeds (black nightshade, Canada thistle, field bindweed, large crabgrass, prickly lettuce, rush skeleton weed, yellow star thistle). In those cases where DPX-KJM44 was applied, there was rapid hydrolysis to form aminocyclopyrachlor which was translocated in the plant. There was no further transformation of aminocyclopyrachlor within the three to eight day duration of the studies.

The metabolism of aminocyclopyrachlor by plants is well understood. Following application to grass (and weeds) the major residue component consists of parent aminocyclopyrachlor.

Environmental fate

The Meeting received information on the soil aerobic metabolism, soil photolysis, aqueous hydrolysis and aqueous photolysis properties of [^{14}C]-aminocyclopyrachlor and [^{14}C]-DPX-KJM44. Studies were also received on the behaviour of [^{14}C]-DPX-KJM44 in a rotational crop situation.

In soil incubation studies under aerobic conditions in the dark at 20 °C, ^{14}C -DPX-KJM44 degraded to form aminocyclopyrachlor with a DT_{50} of 0.1 days. Subsequently aminocyclopyrachlor degraded with a DT_{50} of 275 days. In studies following the aerobic degradation of ^{14}C -aminocyclopyrachlor applied to soil, the DT_{50} for degradation ranged from 120–433 days the sandy loam, clay loam and silty clay soils studied. IN-LXT69 accounted for 4.0–6.4% of the applied radioactivity (AR) at day 0 declining to 0.2–0.4% AR by day 120 of the study. Further analysis of the

unextracted portion of ^{14}C demonstrated incorporation into humin (13–20% AR), fulvic acid (11% AR) and humic acid (0.3% AR) fractions present in the soil.

In four field dissipation studies where DPX-KJM44 was applied to bare soil at two sites and to grass plots at two sites, the soil DT_{50} values were 0.4–1.6 days for DPX-KJM44 and 55 to 163 days for aminocyclopyrachlor. The DT_{50} values for grass foliage were 0.4 days for DPX-KJM44 and 4.8–8.9 days for aminocyclopyrachlor.

In a soil photolysis study with application of ^{14}C -aminocyclopyrachlor on the surface of a silt loam soil the estimated DT_{50} was 61 days suggesting photolysis will contribute to soil degradation.

Aminocyclopyrachlor was stable to hydrolysis in aqueous solutions at pH 4, 7 and 9 suggesting hydrolysis plays a negligible role in its degradation. A study on the aqueous photolysis of aminocyclopyrachlor showed it is degraded on irradiation. Aminocyclopyrachlor accounted for 28% AR after 360 hours continuous irradiation. Photodegradates formed at levels above 5% AR were IN-QFH57 (14% AR), IN-LXT69 (16% AR), IN-YY905 (8% AR), IN-Q3007 (7% AR) and IN-V0977 (12% AR).

In a confined rotational crop study with cabbage, turnip and maize, a plot of sandy loam soil was treated with [^{14}C]-DPX-KJM44 at the equivalent of 75 g ai/ha with some plots treated at 369 g ai/ha and crops sown 30, 60, 120 and 300 days after soil application for cabbage and turnip and 15, 120 and 300 days after application for maize. TRR in cabbage ranged from < LOD to 0.023 mg eq./kg with 60 to 83% of the ^{14}C accounted for by aminocyclopyrachlor. For turnips, negligible ^{14}C residues were detected in roots (maximum 0.004 mg eq./kg) while ^{14}C residues in tops ranged from 0.003 to 0.011 mg eq./kg. In the two samples with sufficient residues for identification, the major components of the ^{14}C residue in tops were aminocyclopyrachlor (41–59% TRR) and DPX-KJM44 (0–17% TRR). Radioactive residues in maize ranged from 0.011 to 0.246 mg equiv/kg for forage, 0.023–0.262 mg eq./kg for stover and 0.012–0.085 mg eq./kg for maize grain. In all cases aminocyclopyrachlor was the major component of the ^{14}C residue (46–71% TRR) with DPX-KJM44 present at $\leq 10\%$ TRR together with small amounts of IN-LXT69 (< 2% TRR). Residues of aminocyclopyrachlor present in soil are able to be taken up the rotational crops.

In summary, aminocyclopyrachlor residues in soil may contribute to residues observed in rotational crops. A field crop rotation study is desirable.

Methods of Analysis

The Meeting received description and validation data for analytical methods suitable for residue analysis of DPX-KJM4, aminocyclopyrachlor and related metabolites IN-LXT69, IN-QFH57 and IN-QGC48 in grass and DPX-KJM4, aminocyclopyrachlor and IN-LXT69 in animal commodities.

Grass samples are homogenised with 0.15 M ammonium acetate (aq) and acetonitrile, extracted with acetonitrile/0.15 M ammonium acetate (aq) 70/30 and the extracts acidified with dilute HCL. Extracts are cleaned up using SPE cartridges before analysis by LC-MS/MS. The LOQs for all analytes are 0.01 mg/kg.

Animal commodities are analysed using a different procedure. Milk samples are extracted using acetonitrile/0.1% aqueous formic acid (90:10, v/v) with analysis by LC-MS/MS. Tissue samples are homogenised and extracted with acetonitrile/0.1% aqueous formic acid with analysis by LC-MS/MS. In the case of muscle the samples are analysed following a clean-up step using solid phase extraction. The LOQs for all analytes are 0.01 mg/kg.

QuEChERS and the US FDA pesticide multiresidue methods are not suitable for analysis of aminocyclopyrachlor.

In conclusion, suitably validated methods are available for the analysis of aminocyclopyrachlor and selected metabolites in animal and plant matrices although currently there are no multi-residue methods available for aminocyclopyrachlor.

Stability of pesticide residues in stored analytical samples

The Meeting received information on the stability of DPX-KJM-44, aminocyclopyrachlor, IN-LXT69, IN-QFH57 and IN-QGC48 in grass and hay stored frozen. The compounds were all stable in grass and hay for the duration of the stability studies; 500 days for DPX-KJM-44, aminocyclopyrachlor, IN-LXT69 and 400 days for IN-QFH57 and IN-QGC48.

In animal matrices fortified separately with DPX-KJM44, aminocyclopyrachlor and IN-LXT69, residues were stable in milk, muscle, fat and hens eggs for at least 133 days. Aminocyclopyrachlor and IN LXT69 were stable in liver and kidney for at least 147 and 88 days, respectively. DPX-KJM44 was not stable in liver and kidney, being converted to aminocyclopyrachlor either during storage or subsequent analysis.

The periods of demonstrated stability cover the frozen storage intervals used in the residue studies.

Definition of the residue

Livestock may be exposed to residues present in feeds. In a lactating goat metabolism study with DPX-KJM44, the ester was rapidly converted to aminocyclopyrachlor which was the major component of the residue in all tissues and milk (kidney 55% TRR, liver 66% TRR, muscle 43% TRR, fat 47–84% TRR, milk 16% TRR) with no individual metabolite of aminocyclopyrachlor was identified as present at levels above 0.01 mg/kg.

Residues of aminocyclopyrachlor were higher in muscle than fat in the metabolism study while in the livestock feeding study they were much higher in fat compared to muscle (2.4 to 9.0×). Levels of aminocyclopyrachlor were higher in skim milk compared to cream. The log K_{ow} for aminocyclopyrachlor is -2.48 (pH 7) suggesting the compound is not fat soluble. Taken as a whole, the Meeting considered that residues of aminocyclopyrachlor are not fat soluble.

Following foliar application of aminocyclopyrachlor (and also DPX-KJM44) to grass, the major component of the residue is aminocyclopyrachlor (33–68% TRR). All components formed from aminocyclopyrachlor were minor (<6.1% TRR).

Based on the above the Meeting decided the residue definition for compliance with MRLs and estimation of dietary intake should be as follows:

Definition of the residue for compliance with MRL and estimation of dietary intake (for animal and plant commodities): *aminocyclopyrachlor*.

The residue is not fat soluble.

Results of supervised residue trials on crops***Grass***

The Meeting received supervised residue trial data for aminocyclopyrachlor on grass (including hay). GAP in the Canada is one application at up to 264 g ai/ha with a PHI of 0 days. In trials approximating critical GAP in Canada residues in grass forage were (n=6): 13, 18, 18, 22, 25, 39 mg/kg (on an as received basis) and 58, 60, 72, 77, 79 and 81 mg/kg when corrected for reported moisture contents.

The Meeting estimated a highest and median residue of 81 and 74.5 mg/kg for grass (dry weight basis).

Residues in grass hay from field trials performed in Canada and the USA approximating GAP in Canada were (n=6): 30, 35, 40, 46, 46, 48 mg/kg (on an as received basis) and 40, 45, 46, 49, 60 and 60 mg/kg when corrected for reported moisture contents.

The Meeting estimated a maximum residue limit and median and high residues of 150, 47.5 and 60 mg/kg for grass hay.

Rotational crop residues

Soil residues of aminocyclopyrachlor are moderately persistent. The use-pattern (Canadian GAP) does not specify plant-back intervals for follow-crops. In the confined rotational crop study, where DPX-KJM44 was applied to soil at the equivalent of 70 to 346 g aminocyclopyrachlor/ha, residues of aminocyclopyrachlor in cabbage and turnip from crops planted 300 days after application to soil were below practical LOQs of 0.01 mg/kg. Residues of aminocyclopyrachlor were above 0.01 mg/kg in maize commodities (forage, stover, grain). The Meeting considered the available information on residues in rotational crops to be inadequate for the purposes of estimating maximum residue levels and STMR values to cover potential residues in such crops. The Meeting also noted that the livestock dietary burden is dominated by residues in grass and hay (100% of the diet for Australia) and that any contribution from potential residues in feeds in follow crops is minor. To enable completion of the risk assessment, the Meeting noted that residues in follow crops are unlikely to average more than 0.01 mg/kg and agreed that a value of 0.01 mg/kg could be used in estimates of consumer exposure for commodities from non-permanent crops.

Residues in animal commodities

Farm animal feeding studies

The Meeting received information on the residue levels in tissues and milk of dairy cows dosed with DPX-KJM44 at the equivalent of 73, 160, 454 and 1594 ppm in the feed for 28 consecutive days.

Residues of DPX-KJM44 and IN-LXT69 in tissues and milk and aminocyclopyrachlor in muscle were < 0.01 mg/kg at all sampling intervals and doses.

Aminocyclopyrachlor residues in milk were < 0.01 mg/kg for the 73 ppm dose group and most of the samples for the 160 ppm dose group. The maximum daily mean residues were 0.024 mg/kg for the 454 ppm dose group and 0.077 mg/kg for the 1594 ppm dose group.

In kidney mean aminocyclopyrachlor residues were 0.12, 0.31, 0.34 and 0.98 mg/kg for the 73, 160, 454 and 1594 ppm dose groups respectively. Mean residues liver residues were 0.039, 0.042, 0.049 and 0.096 mg/kg and mean fat residues were < 0.01, 0.015, 0.062 and 0.46 mg/kg for the 73, 160, 454 and 1594 ppm dose groups respectively.

Animal commodity maximum residue levels

Dietary burden calculations for beef cattle and dairy cattle and poultry are provided below. The dietary burdens were estimated using the OECD diets listed in Appendix IX of the 2009 edition of the FAO Manual.

Potential cattle feed items include: grass and grass hay.

Summary of livestock dietary burden (ppm of dry matter diet)

	US-Canada		EU		Australia		Japan	
	max	mean	Max	mean	Max	Mean	Max	Mean
Beef cattle	9	7.2	41.5	37.2	81	74.5	21.2	20.4
Dairy cattle	36.4	33.5	48.6	44.7	81 ^a	74.5 ^b	44.3	35.6
Broilers	0	0	0	0	0	0	0	0
Layers	0	0	8.1 ^c	7.4 ^d	0	0	0	0

^a Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian meat and milk

^b Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian meat and milk

^c Highest maximum poultry dietary burden suitable for MRL estimates for poultry meat and eggs

^d Highest mean poultry dietary burden suitable for STMR estimates for poultry meat and eggs

Animal commodity maximum residue levels

A lactating dairy cow feeding study was made available to the Meeting. A review of the laboratory animal and lactating goat metabolism studies showed that DPX-KJM44 is rapidly converted to

aminocyclopyrachlor and significant differences are not expected in residues arising from dosing with DPX-KJM44 or aminocyclopyrachlor. The meeting decided the DPX-KJM44 feeding study could be used to estimate aminocyclopyrachlor residues in meat, edible offal and milk and agreed that in estimating residues levels, the feed levels should be expressed in terms of aminocyclopyrachlor acid equivalents.

The calculations used to estimate highest total residues for use in estimating maximum residue levels, STMR and HR values are shown below.

	Feed level (ppm) for milk residues	Residues (mg/kg) in milk	Feed level (ppm) for tissue residues	Residues (mg/kg) in			
				Muscle	Liver	Kidney	Fat
MRL beef or dairy cattle							
Feeding study ^a	68.5 (73) ^c	< 0.01	68.5 (73) ^c	< 0.01	0.064	0.17	0.015
	150 (160) ^c	0.012	150 (160) ^c	< 0.01	0.082	0.4	0.04
Dietary burden and high residue	81	0.01	81	< 0.01	0.067	0.21	0.019
STMR beef or dairy cattle							
Feeding study ^b	68.5 (73) ^c	< 0.01	68.5 (73) ^c	< 0.01	0.039	0.12	0.01
	150 (160) ^c	< 0.01	150 (160) ^c	< 0.01	0.042	0.31	0.015
Dietary burden and residue estimate	74.5	< 0.01	74.5	< 0.01	0.039	0.13	0.01

^a highest residues for tissues and mean residues for milk

^b mean residues for tissues and mean residues for milk

^c feeding level expressed as acid equivalents = aminocyclopyrachlor, figure in brackets is feed level expressed in terms of DPX-KJM44

The Meeting estimated the following STMR values: milk 0.01 mg/kg; muscle 0.01 mg/kg; 0.039 mg/kg for liver and 0.13 mg/kg for kidney and fat 0.01 mg/kg.

The Meeting estimated the following maximum residue levels: milk 0.02 mg/kg; meat (mammalian except marine mammals) 0.01 mg/kg, fat 0.03 mg/kg and edible offal 0.3 mg/kg.

No information on residues of aminocyclopyrachlor in poultry were available to the meeting, therefore no maximum residue levels can be estimated for poultry commodities. However, Europe was the only region for which the poultry dietary burden was greater than zero. As aminocyclopyrachlor is not approved for use in Europe, the meeting considered there is no likelihood of residues in poultry commodities.

RECOMMENDATIONS

On the basis of the data obtained from supervised residue trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue (for compliance with MRL and estimation of dietary intake) for animal and plant commodities: *Aminocyclopyrachlor*

The residue is not fat soluble.

Commodity		Recommended MRL (mg/kg)		STMR or STMR-P (mg/kg)	HR, HR-P, highest residue (mg/kg)
CCN	Name	New	Previous		
MO 0105	Edible offal (mammalian)	0.3		0.039Liver 0.13Kidney	
MF 0100	Mammalian fats	0.03		0.01	
	Grass forage			74.5 dw	81 dw
AS 0162	Hay and fodder (dry) of grass	150			
MM 0095	Meat (from mammals other than marine mammals)	0.01		0.01	
ML 0106	Milks	0.02		0.01	

dw = dry weight basis

Table of recommendations animal feeds.

Commodity		Recommended MRL (mg/kg)		STMR or STMR-P (mg/kg)	HR, HR-P, highest residue (mg/kg)
CCN	Name	New	Previous		
	Grass forage			74.5 dw	81 dw

dw = dry weight basis

DIETARY RISK ASSESSMENT

Long-term intake

The WHO Panel of the 2014 JMPR established an Acceptable Daily Intake (ADI) of 0-3 mg/kg bw for aminocyclopyrachlor.

The evaluation of aminocyclopyrachlor resulted in recommendations for MRLs and STMR values for raw and processed commodities. Where data on consumption were available for the listed food commodities, dietary intakes were calculated for the 17 GEMS/Food Consumption Cluster Diets.

The IEDIs in the seventeen Cluster Diets, based on the estimated STMRs were 0% of the maximum ADI (3 mg/kg bw). The Meeting concluded that the long-term intake of residues of aminocyclopyrachlor from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The Meeting decided that an ARfD is unnecessary. The Meeting therefore concluded that the short-term intake of residues of aminocyclopyrachlor is unlikely to present a public health concern.

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BENZOVINDIFLUPYR (261)

First draft prepared by Ms T. van der Velde-Koerts, Centre for Nutrition, Prevention and Health Services (VPZ), National Institute for Public Health and the Environment (RIVM), The Netherlands

EXPLANATION

Benzovindiflupyr was scheduled for residue evaluation as a new compound by the 2014 JMPR at the Forty-fifth Session of the CCPR (2013). The toxicological review was conducted in 2013, which established an ADI of 0–0.05 mg/kg bw and an ARfD of 0–0.1 mg/kg bw. Additional toxicological data were provided for the metabolites SYN546039 and SYN545720. Benzovindiflupyr was defined by the WHO panel as the only toxicologically significant compound in animals, plants and the environment.

Benzovindiflupyr is a broad-spectrum fungicide belonging to the chemical class of pyrazole carboxamides. Benzovindiflupyr acts as an inhibitor of the fungal complex II respiratory chain, where it inhibits the succinate dehydrogenase enzyme (succinate dehydrogenase inhibitor, SDHI) by blocking the ubiquinone-binding sites in the mitochondrial complex.

The Meeting received information from the manufacturer on identity, metabolism, storage stability, residue analysis, use pattern, residues resulting from supervised trials on soya beans, fate of residue during processing, and livestock feeding studies.

IDENTITY

ISO common name:	benzovindiflupyr
Chemical name	
IUPAC:	N-[(1RS,4SR)-9-(dichloromethylene)-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1-methylpyrazole-4-carboxamide. In the IUPAC convention the symbol 1RS,4SR represents a racemic mixture of 1R,4S and 1S,4R [Syngenta, 2014b],
CAS:	1H-pyrazole-4-carboxamide, N-[9-(dichloromethylene)-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1-methyl-
CAS Registry No:	1072957-71-1
CIPAC No:	-
Synonyms and trade names:	SYN545192; CSCD064398; N-[(1RS,4SR)-9-(dichloromethylidene)-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxamide (name used in the WHO monograph) N-[(1RS,4SR)-9-(dichloromethylene)-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxamide; N-[9-(dichloromethylene)-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxamide; N-[11-(dichloromethylene)tricyclo[6.2.1.0 ^{2,7}]undeca-2,4,6-trien-3-yl]-3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxamide 3-difluoromethyl-1-methyl-1H-pyrazole-4-carboxylic acid ((1RS,4SR)-9-dichloromethylene-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl)-amide SMILES: <chem>Cn1cc(c(n1)C(F)F)C(=O)Nc2cccc3c2C4CCC3C4=C(Cl)Cl</chem>
Structural formula:	Structure for PAI (99.4% purity) confirmed by UV-VIS, IR, ¹ H-NMR and MS [Oggenfuss, 2011, SYN545192_10087]

		or
		or
Molecular formula:	C ₁₈ H ₁₅ Cl ₂ F ₂ N ₃ O	
Molecular weight:	398.2 g/mol	

Benzovindiflupyr contains chiral centres at both the bridgehead carbon atoms potentially resulting in four stereoisomeric forms. However, the bicyclic ring is a rigid structure, and therefore only two stereoisomers exist (an enantiomeric pair). Technical benzovindiflupyr consists of a racemic mixture of two enantiomers SYN546526 and SYN546527, at a ratio of 50:50. SYN546526 represents N-[(**1R,4S**)-9-(dichloromethylene)-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1-methylpyrazole-4-carboxamide. SYN546527 represents N-[(**1S,4R**)-9-(dichloromethylene)-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1-methylpyrazole-4-carboxamide [Syngenta, 2014b]. The enantiomers were unresolved using the chromatographic solvent systems in the reports. Both enantiomers were measured within the same single chromatographic peak and they were collectively reported as one concentration [Syngenta 2014b]. Both enantiomers are fungicidally active. No toxicological studies were performed on the individual enantiomers.

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient (PAI)

Parameter	Result	References	Guidelines/method
Purity	99.4% (PAI)		
Appearance:	odourless white powder at 25 °C purity 99.4% (PAI)	[Das, 2010a, SYN545192_10057]	organoleptic and visual assessment
Vapour pressure:	measured 2.0×10^{-5} Pa at 70 °C 7.6×10^{-5} Pa at 80 °C 3.9×10^{-4} Pa at 90 °C extrapolated 3.2×10^{-9} Pa at 25 °C	[Weissenfeld, 2010, SYN545192_10067]	OECD 104 gas saturation method

Parameter	Result	References	Guidelines/method
	purity 99.4% (PAI) The vapour pressure at 25 °C was extrapolated from high temperature measurements to allow the completion of the various experiments within a suitable period of time [Syngenta, 2014a, SYN545192_10468].		
Melting point:	148.4 °C ± 0.5 °C purity 99.4% (PAI)	[Kühne, 2010b, SYN545102_10060]	OECD 102 thermal analysis (differential scanning calorimetry)
Octanol/water partition coefficient:	$\log K_{ow} = 4.3 \pm 0.3$ at 25 °C ± 1 °C in pure water (pH 6.59) purity 99.4% (PAI) The $\log K_{ow}$ value calculated from the ratio of solubilities (19 g/L: 0.98 mg/L) is 4.3 which agrees well with the value determined using the OECD 107 [Syngenta, 2014a, SYN545192_10468].	[Vijayakumar, 2010b, SYN545102_10070]	OECD 107 shake flask method
Solubility:	0.98 mg/L RSD 8.9% in pure water at pH 6.7–7.5, at 25 °C purity 99.4% (PAI)	[Vijayakumar, 2011, SYN545102_10072]	OECD 105 column elution method
	solubility at 25 °C ± 0.5 °C purity 97.7% (TGAI) Acetone 350 g/L Dichloromethane 450 g/L Ethyl acetate 190 g/L Hexane 270 g/L Methanol 76 g/L Octanol 19 g/L Toluene 48 g/L No data are available for PAI [Syngenta, 2014a, SYN545192_10468].	[Vijayakumar, 2010a, SYN545102_10066]	CIPAC MT 157.3 flask method
Specific gravity:	1.466 g/cm ³ at 20.4 °C $D_{4}^{20} = 1.466$ (rel. density) purity 99.4% (PAI)	[Kühne, 2010d, SYN545192_10068]	OECD 109 gas comparison pycnometer
Hydrolysis in water:	0.35 mg/L [pyrazole-¹⁴C]-SYN545192 (radiochemical purity 98.7%) Hydrolytically stable in sterile buffer at pH 4, 5, 7 and 9 for up to 5 days at 50 °C in the dark. Stability was confirmed at all pH values for up to 30 days at 25 °C. No degradation products were detected by HPLC, TLC or HPLC-MS.	[Lowrie, 2009, SYN545192_10010]	OECD 111
Photolysis in water:	0.3 mg/L [phenyl- ¹⁴ C]-SYN54519 (purity 98.5%) or 0.3 mg/L [pyrazole- ¹⁴ C]-SYN545192 (purity 98.6%)	[Wardrope, 2011, SYN545192_10132]	OECD 316 EPA 161-2 OPPTS 835.224

Parameter	Result	References	Guidelines/method
	Moderate decline of parent to 74.8% TAR or 74.4% TAR for phenyl or pyrazole labels, respectively, after 15 days in sterile buffer at pH 7 at 25 °C ± 1 °C using a xenon arc lamp at 53.1 W/m² continuously DT₅₀ = 44.2 days (average of both labels). Photodegradation of the phenyl labelled compound resulted in the production of multiple polar components (11.5% TAR) and diffuse radioactivity (6.3% TAR). No individual compound or region accounted for > 5% TAR. For the pyrazole label, the most significant degradates were NOA449410 at 8.5% TAR and SYN508272 at 2.6% TAR, both at 15 DAT. No other single degradate was observed at > 5% TAR. Photodegradation resulted in the production of ¹⁴CO₂, which reached a maximum mean value of 6.3% TAR in the phenyl samples and 1.1% TAR in the pyrazole samples at 15 DAT.		
Dissociation constant:	no dissociation at pH 2.0–12.0 purity 99.4% (PAI)	[Kühne, 2010e, SYN545192_10069]	OECD 112 titration method

Technical material (TGAI)

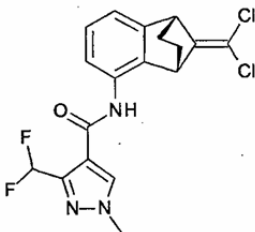
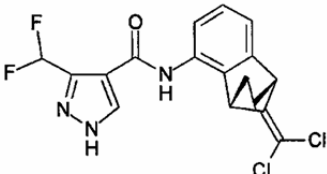
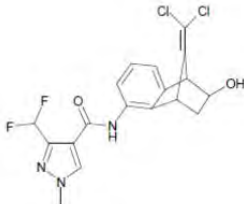
Parameter	Result	References	Guidelines
Purity	97.7% (TGAI)		
Appearance:	odourless off-white powder at 25 °C purity 97.7% (TGAI)	[Das, 2010b, SYN545192_10058]	organoleptic and visual assessment
Density:	1.42 g/cm ³ at 22.0 ± 1.0 °C purity 97.7% (TGAI)	[O'Connor, 2013a, SYN545192_10444]	OECD 109 gas comparison pycnometer
Melting range:	145.7 °C (418.9 K), with RSD 0.07% purity 97.7% (TGAI)	[O'Connor, 2013b, SYN545192_10445]	OECD 102 differential scanning calorimetry
Thermal stability	PAI (purity 99.4%) decomposition started at about 285 °C before boiling occurred. No data are submitted for the TGAI.	[Kühne, 2010a, [SYN545192_10059]]	OECD 103, thermal analysis (differential scanning calorimeter)
Stability:	TGAI (purity 97.7%) is stable in nitrogen or air at room temperature.	[Williams, 2011, SYN545192_10088]	OECD 113 differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA)

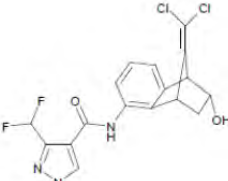
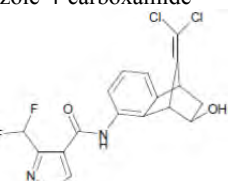
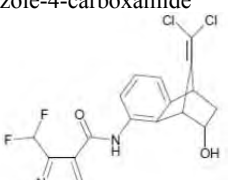
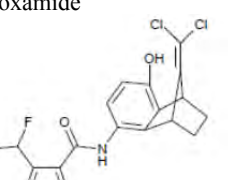
Formulations

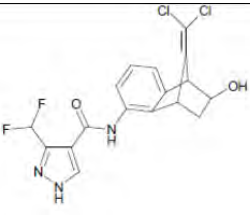
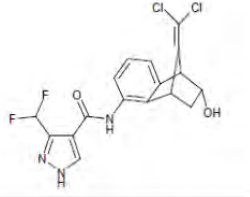
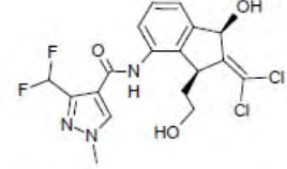
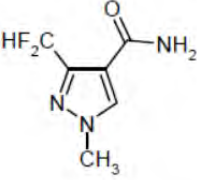
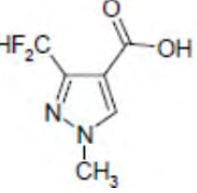
Formulation authorised for use: WG (150 g/kg benzovindiflupyr + 300 g/kg azoxystrobin).

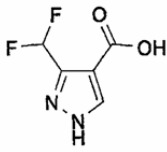
Benzovindiflupyr has not been evaluated by JMPS and therefore no FAO specifications for technical and formulated benzovindiflupyr have been published.

Table 1 List of reference compounds used in various study reports [Syngenta, 2014c]

Abbreviation	Trivial and systematic chemical names Other abbreviations used in study reports	Found as or in
SYN545192 BVFP (parent)	benzovindiflupyr CSCD064398 IUPAC: N-[(1RS,4SR)-9-(dichloromethylene)-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxamide 	rat goat (milk, liver, kidney, muscle, fat) hen (eggs, liver, muscle, skin with fat) tomato fruit wheat forage/hay/straw wheat grains soya bean forage/hay soya bean seeds rotational crops: lettuce leaves, turnip roots/leaves, wheat forage/hay/straw photolysis in water hydrolysis in water
SYN546206 (N-demethyl-BVFP)	CSCD711742; N-demethyl-benzovindiflupyr IUPAC: N-[(1RS,4SR)-9-(dichloromethylene)-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1H-pyrazole-4-carboxamide  MW 384.22	rat goat (liver, kidney, muscle, fat) hen (eggs, liver, muscle, skin with fat) tomato fruit wheat forage/hay/straw wheat grains soya bean forage/hay soya bean seeds rotational crops: lettuce leaves, turnip roots/leaves, wheat forage/hay/straw
SYN546039 (BVFP-OH)	CSCD695908 hydroxy-benzovindiflupyr isomer of SYN546040 IUPAC: N-[(1RS,2RS,4SR)-9-(dichloromethylene)-2-hydroxy-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxamide  MW 414.20	rat goat (milk, liver, kidney, muscle, fat) hen (eggs, liver, muscle, skin with fat) tomato fruit wheat forage/hay/straw wheat grains soya bean forage/hay soya bean seeds rotational crops: lettuce leaves, turnip roots/leaves, wheat forage/hay/straw
SYN546040 (BVFP-OH)	CSCD696468 hydroxy-benzovindiflupyr isomer of SYN546039	rat goat (milk, liver, kidney, muscle, fat)

Abbreviation	Trivial and systematic chemical names Other abbreviations used in study reports	Found as or in
	IUPAC: N-[(1RS,2SR,4SR)-9-(dichloromethylene)-2-hydroxy-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1-methyl-1<i>H</i>-pyrazole-4-carboxamide  MW 414.20	hen (eggs, liver, muscle, skin with fat) tomato fruit wheat grains soya bean forage/hay soya bean seeds rotational crops: lettuce leaves, turnip roots/leaves, wheat forage/hay/straw
SYN546322	CSCD733585 hydroxy-benzovindiflupyr isomer of SYN546323 IUPAC: N-[(1RS,3SR,4SR)-9-(dichloromethylene)-3-hydroxy-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1-methyl-1<i>H</i>-pyrazole-4-carboxamide  MW 414.20	not found in wheat
SYN546323	CSCD733586 hydroxy-benzovindiflupyr isomer of SYN546322 IUPAC: N-[(1RS,3RS,4SR)-9-(dichloromethylene)-3-hydroxy-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1-methyl-1<i>H</i>-pyrazole-4-carboxamide  MW 414.20	not found in wheat
SYN546360	CSCD737493 hydroxy-benzovindiflupyr IUPAC: N-[(1RS,4SR)-9-(dichloromethylene)-8-hydroxy-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1-methyl-1<i>H</i>-pyrazole-4-carboxamide  MW 414.20	rat goat (bile) hen (eggs, liver, muscle)
SYN546041 (N-demethyl-BVFP-OH)	CSCD695909 N-demethyl-hydroxy-benzovindiflupyr isomer of SYN546042 IUPAC: N-[(1RS,2RS,4SR)-9-(dichloromethylene)-2-hydroxy-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1<i>H</i>-pyrazole-4-carboxamide	rat goat (milk, liver, kidney, muscle, fat) hen (eggs, liver, muscle, skin with fat) wheat straw wheat grain

Abbreviation	Trivial and systematic chemical names Other abbreviations used in study reports	Found as or in
		soya bean forage/hay soya bean seeds rotational crops: turnip roots/leaves, wheat forage/hay/straw
SYN546042 (N-demethyl-BVFP-OH)	CSCD695910 N-demethyl-hydroxy-benzovindiflupyr isomer of SYN546041 IUPAC: N-[(1RS,2SR,4SR)-9-(dichloromethylene)-2-hydroxy-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1H-pyrazole-4-carboxamide 	rat goat (milk, liver, kidney, muscle, fat) hen (eggs, liver, muscle, skin with fat) soya bean hay soya bean seeds rotational crops: turnip roots/leaves, wheat forage/hay/straw
SYN546422	CSCD743668; IUPAC: N-[(1SR,3RS)-2-(dichloromethylene)-1-hydroxy-3-(2-hydroxy-ethyl)-indan-4-yl]-3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxamide  MW 432.3	goat (milk, liver, kidney, muscle, fat) not found in hen postulated in rat
SYN508272	CSCC210616 pyrazole amide IUPAC: 3-difluoromethyl-1-methyl-1H-pyrazole-4-carboxylic acid amide  MW 175.1	goat (milk, liver, kidney, muscle, fat) hen (eggs, liver, muscle, skin with fat) wheat hay/straw wheat grains soya bean forage soya bean hay rotational crops: turnip leaves, wheat forage/hay/straw photolysis in water common metabolite with other pyrazole fungicides like bixafen, fluxapyroxad, isopyrazam and sedaxane
NOA449410	pyrazole acid CSAA798670 IUPAC: 3-difluoromethyl-1-methyl-1H-pyrazole-4-carboxylic acid 	not detected in goat and hen commodities tomato fruit wheat hay/straw wheat grains soya bean forage/hay soya bean seeds rotational crops: lettuce leaves, turnip roots/leaves, wheat

Abbreviation	Trivial and systematic chemical names Other abbreviations used in study reports	Found as or in
		forage/hay/straw photolysis in water common metabolite with other pyrazole fungicides like bixafen, fluxapyroxad, isopyrazam and sedaxane
SYN545720	N-desmethylpyrazole-acid CSCD465008, R958945, IUPAC: 3-difluoromethyl-1 <i>H</i> -pyrazole-4-carboxylic acid CASnr 151734-02-0  MW 162.1	not detected in rat, goat and hen commodities tomato fruit soya bean forage/hay soya bean seeds rotational crops: lettuce leaves, turnip roots/leaves, wheat forage/hay/straw (not found in photolysis in water) common metabolite with other pyrazole fungicides like bixafen, fluxapyroxad, isopyrazam and sedaxane

METABOLISM AND ENVIRONMENTAL FATE

The Meeting received information on the fate of benzovindiflupyr in livestock, plant commodities, soil and rotational crops. The test items used in the studies were benzovindiflupyr ^{14}C uniformly labelled in the phenyl ring or at the 5-position of the pyrazole ring as shown in Figure 1.

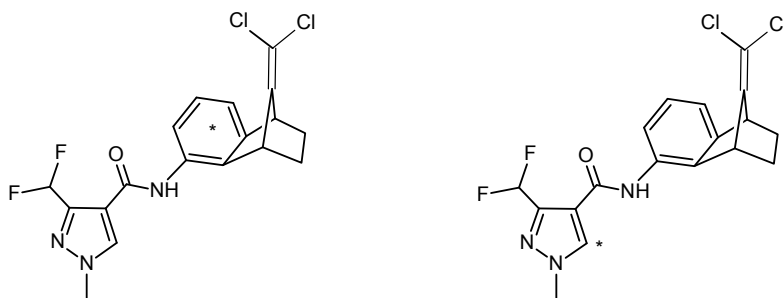


Figure 1 Benzovindiflupyr ^{14}C labelled in the phenyl ring or the pyrazole ring

Animal metabolism

The Meeting received information on the metabolic fate of benzovindiflupyr in ruminants (lactating goats) and poultry (laying hens). The metabolism in laboratory animals was summarized and evaluated by the WHO panel of the JMPR in 2013.

Lactating goats

One lactating goat (Saanen × Toggenburg) per radiolabel was dosed orally once daily for 7 consecutive days with a gelatin capsule containing [phenyl-¹⁴C]-benzovindiflupyr or [pyrazole-¹⁴C]-benzovindiflupyr [Green, 2012, SYN545192_10184 and additional info in Syngenta, 2014c]. The actual mean daily dose administered was 41 (range 34–50) and 32 (range 30–34) ppm in the dry feed for the phenyl and pyrazole label, respectively. Average feed consumption was 1.5 kg/animal (phenyl label) and 1.9 kg/animal (pyrazole label experiment) as dry weight feed. Average dose rates were 0.89 and 0.98 mg/kg bw/day for the phenyl and pyrazole label, respectively. The age of the goats was 3.0 and 4.5 years for the phenyl and pyrazole label, respectively. Body weights were 70 and 63 kg at the start of the study and 66 and 59 kg at the end of the study for the phenyl and pyrazole label, respectively. Milk was collected twice daily; urine and faeces were collected once daily. Average milk production during application was 2.5 and 2.1 kg/day for the phenyl and pyrazole label experiments, respectively. Aliquots of the PM and AM milk were mixed and this composite 24 h sample was used to generate cream and skimmed milk for that time period following centrifugation. The goats were sacrificed approximately 12 hours after the administration of the final dose and liver, kidney, muscle (hindquarter, forequarter and tenderloin), fat (subcutaneous, omental and perirenal), blood, GI tract and contents, bile were taken post mortem. Samples were stored frozen at –20 °C for 82–139 days until analysis.

Homogenised samples were analysed by combustion LSC. The mean total recovery of the dosed radioactivity was 90% and 86% for the goats dosed with phenyl and pyrazole radiolabelled forms, respectively. The majority of the radioactivity was recovered in faeces (79%/73% TAR, phenyl/pyrazole). The remainder of the dose was recovered in urine (4.5%/5.2% TAR phenyl/pyrazole), GI tract contents (6.8%/7.2% TAR), cage wash (< 0.28% TAR), milk (< 0.16% TAR) and liver (0.33%/0.22% TAR), while only very low levels were found in kidney, muscle, blood or fat (≤ 0.01% TAR each).

Total radioactive residues are summarised in Table 2. The highest radioactivity concentrations in edible tissues were found in the liver (1.3/0.70 mg/kg eq), kidney (0.28/0.18 mg/kg eq), followed by fat (0.098/0.070 mg/kg eq) and muscle (0.070/0.032 mg/kg eq). Total radioactive residues in milk from the phenyl label dosed goat reached a plateau concentration of approximately 0.046 mg/kg eq following 96 hours dosing. Total radioactive residues in milk from the pyrazole label dosed goat reached a plateau concentration of approximately 0.035 mg/kg eq following 72 hours dosing. Residue levels in cream and skimmed milk derived from combined PM and AM milk samples (24 hour samples) from within the plateau milk residue period are summarised in Table 3. The ratio of cream total radioactive residue to skimmed milk total radioactive residue ranged from 3.7:1 to 4.8:1 in all analysed samples of both radiolabelled experiments except one sample (phenyl: 144 hour sampling) where the ratio was larger (12.2:1). No explanation for the increase in ratio in this single sample was evident. No constituent ¹⁴C-residue analysis of the separated cream and aqueous (whey) fractions was undertaken [Syngenta 2014b].

Homogenised samples of liver, kidney and composite muscle were extracted sequentially with acetonitrile (1×), acetonitrile/water 4:1 v/v (1–4×), acetonitrile/water 3:7 v/v (1–2×), water (1×) and acetonitrile (1×). Composite fat was extracted first with dichloromethane (1×) and subsequently with the other solvents. Whole milk (144 hrs) was extracted with dichloromethane (3×), ethyl acetate (2×) and acetone (1×). Extracts and remaining solids were analysed by (combustion) LSC. Extracted residues were, where appropriate, combined and fractionated by liquid/liquid partition. Liver and kidney extracts were subject to β-glucuronidase (pH 5, 37 °C, 18 hrs) to cleave glucuronide and sulphate conjugates and subsequently partitioned with ethyl acetate. Fractions containing significant radioactive residues were subject to TLC with bio-imaging analysis to enable quantification and identification of residues. Residues were identified by co-chromatography with authentic reference standards for parent, SYN546206, SYN546039, SYN546040, SYN546041, SYN546042, SYN546360, SYN546422, SYN508272, NOA449410 and SYN545720.

Extractability of the radioactive residues was ≥ 94% TRR except that of liver (78–89% TRR). Identified radio-components present in extracted residues are summarised in Table 4.

Comparison of 2D-TLC chromatograms of pre- and post-hydrolysis fractions in liver and kidney shows that the released ^{14}C -exocons after hydrolysis originate predominantly from radio-components present on the origin (baseline) of the chromatogram. Unassigned radio-components that chromatograph away from the origin are unlikely to be conjugated metabolites for two reasons:

i) The solvent systems employed in the normal phase 2D-TLC analyses are quite non-polar in nature and the sulphate and glucuronide conjugated metabolites (or other logical conjugates) are intrinsically quite polar in nature and it would therefore be expected that those conjugates would be located on or very close to the origin of these chromatograms,

ii) Significant proportions of the “unassigned” chromatographed radio-components chromatograph well away from the origin of the 2D-chromatograms and in the region of identified “non-conjugated” and non-polar metabolites. The unassigned radio-components are therefore too non-polar to be characteristic of the conjugates characterised in i) above. [Syngenta, 2014c]

Solids remaining after extraction from liver (phenyl PES: 11% TRR, pyrazole PES: 22% TRR,) were further characterised in three parallel experiments. Incubation under mild acid hydrolysis conditions (0.1 M HCl, 37 °C, 4 hrs) rendered only very minor proportions ($\geq 0.9\%$ TRR) of the PES soluble. Following protein solubilisation using sodium dodecyl sulphate (SDS) and then a protein precipitation step, 6.8% TRR (phenyl) and 15% TRR (pyrazole) was shown to be associated with protein. Protease digestion (pH 7, 37 °C, 24 hrs + 48 hrs) solubilised almost all the PES. TLC analysis of the protease hydrolysates showed them to comprise a very complex mixture of highly polar metabolites of which no single one accounted for $> 2.0\%$ TRR (phenyl) or $> 4.4\%$ TRR (pyrazole).

Initial radio-component profiles of fractions of liver and whole milk samples were produced within 82–131 days of sacrifice. While there is no information on the first 82 days of storage, re-analysis of the milk fractions after completion of the analyses (730 days of total storage) demonstrated the radio-component profiles not to have changed significantly during the interim period of frozen storage. Re-analysis of the liver fractions after 694 days of total storage showed a different profile, indicating some change in composition during storage.

Note: The identified metabolite levels in muscle, fat and milk relate only to the free form of the metabolites. Since the unassigned fractions of these matrices were not subjected to glucuronidase treatment, it is not clear how much conjugated metabolites need to be added to these figures. It will however i) not be more than the sum of the “unassigned and baseline” figures and ii) in reality be closer to the polar “baseline” only figure due to the predominantly non-polar character of the “unassigned” residues precluding them from being conjugates.

Table 2 Total Radioactive Residues and extractability in samples from goats treated with [^{14}C]benzovindiflupyr

Radiolabel	Sample	Extracted Radioactivity		Post-extracted solids		TRR ^a
		%TRR	mg/kg eq	%TRR	mg/kg	
Phenyl	Milk (144 h)	98	0.040	2.0	0.001	0.041
	Liver	89	1.1	11	0.14	1.3
	Kidney	96	0.27	4.0	0.011	0.28
	Composite muscle ^b	98	0.068	2.3	0.002	0.070
	Composite fat ^c	100	0.098	0.3	< 0.001	0.098
Pyrazole	Milk (144 h)	100	0.034	NA	NA	0.034
	Liver	78	0.54	22	0.16	0.70
	Kidney	94	0.18	5.9	0.011	0.18
	Composite muscle ^b	96	0.031	3.7	0.001	0.032
	Composite fat ^c	99	0.069	1.0	0.001	0.070

NA Not Applicable

^a TRR in mg/kg eq calculated from radioactivity extracted and radioactivity in the PES.

^b Forequarter, hindquarter and tenderloin muscle combined in the ratio 4:4:1 by weight to produce a single composite sample.

^c Subcutaneous, omental and perirenal fat combined in the ratio 2:1:1 by weight to produce a single composite sample.

Table 3 Total Radioactive Residues in Cream and Skimmed Milk

Milk Sample Time Point	TRR am samples (mg/kg eq)		Ratio (C/S)	TRR am samples (mg/kg eq)		Ratio (C/S)
	Cream	Skimmed		Cream	Skimmed	
96 hours	0.20	0.041	4.8	0.12	0.029	4.2
120 hours	0.21	0.043	4.8	0.11	0.029	3.9
144 hours	0.31	0.025	12	0.13	0.034	3.7
156 hours	0.19	0.042	4.6	0.11	0.031	3.7

C/S = ratio cream / skimmed

Table 4 Characterisation and Identification of Components in samples from Goats Treated with [¹⁴C]BVFP

	phenyl- ¹⁴ C-BVFP					pyrazole- ¹⁴ C-BVFP				
	Milk %TRR	Liver %TRR	Kidney %TRR	Muscle %TRR	Fat %TRR	Milk %TRR	Liver %TRR	Kidney %TRR	Muscle %TRR	Fat %TRR
TRR mg/kg eq ^a	0.041	1.3	0.28	0.070	0.098	0.034	0.70	0.18	0.032	0.070
parent	5.5	11	13	24	41	7.3	10	7.5	25	44
SYN546206 (N-demethyl-BVFP)	ND	0.4	0.9	1.3	0.8	ND	0.5	0.3	0.7	0.3
		(ND)	(ND)				(0.2)	(0.1)		
SYN546039 (BVFP-OH)	24	50	23	39	36	22	38	22	39	25
		(45)	(ND)				(35)	(7.8)		
SYN546040 (BVFP-OH)	2.3	5.4	3.5	8.1	3.5	0.4	1.3	0.9	1.8	0.9
		(4.3)	(0.1)				(1.0)	(0.3)		
SYN546360 ^f	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
SYN546041 (N-demethyl-BVFP-OH)	7.5	6.2	7.6	2.8	0.7	8.2	6.9	10.5	4.4	1.1
		(5.7)	(1.1)				(6.3)	(5.0)		
SYN546042 (N-demethyl-BVFP-OH)	6.6	3.0	7.0	4.4	0.8	7.1	0.9	1.8	1.0	0.3
		(1.9)	(ND)				(0.6)	(1.1)		
SYN546422	25	4.4	19	8.2	1.6	20	6.0	16	8.9	1.5
		(3.5)	(1.6)				(5.6)	(4.5)		
SYN508272	NA	NA	NA	NA	NA	2.5	0.2	0.7	3.3	0.3
NOA449410	NA	NA	NA	NA	NA	ND	ND	ND	ND	ND
SYN545720	NA	NA	NA	NA	NA	ND	ND	ND	ND	ND
Unassigned ^b	13	2.2	3.9	0.7	1.0	16	2.3	11	6.5	10
Baseline ^c	2.3	2.7	5.9	2.0	0.6	3.5	3.6	7.1	5.3	1.4
Other fractions ^d	8.7	3.3	2.3	2.5	2.5	7.0	4.5	2.6	2.5	4.9
PES ^e	2.0	11	4.0	2.3	0.3	NA	22	5.9	3.7	1.0
Total	97	100	89	95	88	95	97	86	102	91

ND Not detected (< 0.001 mg/kg eq)

NA Not applicable for the label indicated

() = The values within parentheses indicate the proportion of the TRR that is in the conjugated forms. Values without parentheses are the sum of both the free and conjugated forms.

^a TRR determined by summation of radioactivity present in the extracts and solids following solvent extraction

^b Unassigned radio components which chromatographed away from the origin in normal phase 2D-TLC.

Milk comprising at least 4–8 discrete components, no single one ≥ 6.5% TRR (≥ 0.003 mg/kg eq)

Liver post-enzyme hydrolysis, comprising at least eight discrete components, no single one ≥ 0.7% TRR (≥ 0.006 mg/kg eq).

Kidney post-enzyme hydrolysis comprising at least 10–19 discrete components, including a poorly resolved region of 14 components which accounted for a total of 8.5% TRR (0.016 mg/kg). All other unidentified components were < 1.3% TRR (< 0.004 mg/kg eq).

Muscle, comprising at least 2–3 discrete components, no single one ≥ 5.2% TRR (≥ 0.002 mg/kg eq).

Fat, comprising at least 2–4 discrete components, no single one ≥ 6.2% TRR (≥ 0.004 mg/kg eq).

^c Polar material on the origin of the radio-chromatogram using normal phase 2D-TLC

^d Extracted residues in fractions that were not analysed because levels of radioactivity were too low for analysis

Milk (2 fractions), No single fraction comprised $\geq 6.9\%$ TRR (≥ 0.002 mg/kg eq)

Liver (3–4 fractions), No single fraction comprised $\geq 2.4\%$ TRR (≥ 0.030 mg/kg eq)

Kidney (3 fractions), No single fraction comprised $\geq 1.7\%$ TRR (≥ 0.004 mg/kg eq)

Muscle (5 fractions), No single fraction comprised $\geq 2.4\%$ TRR (≥ 0.002 mg/kg eq).

Fat (4 fractions), No single fraction comprised $\geq 1.6\%$ TRR (≥ 0.001 mg/kg eq).

^e Radioactivity remaining in the solids after extraction

^f The presence of SYN546360 in goat bile was shown by HPLC-MS. Its location in 2D-TLC chromatograms (the primary technique used for quantitative analysis of Milk and Tissue fractions) was not established in the goat metabolism study. However comparison of goat 2D-TLC chromatograms for milk and goat tissue fractions with those of the rat biotransformation study (where a metabolite standard of SYN546360 was shown to chromatograph between SYN546039 and SYN546206) demonstrated that either no residues or if present only a very minor residue (Goat liver: 0.4% TRR; 0.005 mg/kg eq) could be attributed to this metabolite. Its presence in milk and goat tissues can therefore be assumed to be negligible [Syngenta, 2014b].

Laying hens

Five laying hens (ISA Warren) per radiolabel were dosed orally once daily for 14 consecutive days via gelatin capsules containing [phenyl- ^{14}C]-benzovindiflupyr or [pyrazole-5- ^{14}C]-benzovindiflupyr [Lowrie and Kadow, 2012, SYN545192_10170 and additional information in Syngenta, 2014c]. The actual mean daily dose administered based on measured feed consumption of individual birds was 16–20 and 17–20 ppm in the dry feed, for the phenyl and pyrazole experiments, respectively. Average feed consumption ranged from 0.13–0.15 kg/animal (phenyl treatment) and 0.12–0.14 kg/animal (pyrazole experiment) on dry weight basis. Body weights ranged from 1.6–1.9 kg at the start and 1.7–2.1 kg at the end of the dosing period. Average dose rates were 1.4 mg/kg bw/day for the phenyl and pyrazole labels, each. Hens were pullets (young hens < 1 year old and greater than 23 weeks) and demonstrated good egg laying performance. Excreta were collected daily; eggs were collected twice daily. Eggs were separated into yolk and white. The hens were sacrificed approximately 12 hours after the administration of the final dose and liver, kidney, leg and thigh muscle, breast muscle, peritoneal fat, subcutaneous fat with skin attached, blood, bile, partially formed eggs, carcass and GI tract and contents were taken post mortem. Samples were stored frozen at -20°C for 314–426 days until analysis.

Homogenised samples were analysed by combustion LSC. The kidneys, carcass and bile samples were not analysed. The average recovery of radioactivity was 88% and 93% TAR from birds of the phenyl and pyrazole label experiment, respectively. Of this on average 88% and 92% TAR was accountable in the hen excreta plus cage wash, while 0.28%/1.1% TAR was found in the gastrointestinal tract contents, 0.10%/0.13% TAR in egg yolks + whites, < 0.04% in liver, < 0.02% in muscle samples, < 0.01% in fat samples.

Total radioactive residues are summarised in Table 5. The highest radioactivity concentrations in edible tissues were found in the liver (0.19/0.25 mg/kg eq), followed by fat (0.033/0.045 mg/kg eq) and muscle (0.025/0.036 mg/kg eq). Mean total radioactive residues in egg yolks achieved a plateau concentration of 0.17 mg/kg eq (phenyl label) after 240 hrs dosing and 0.18 mg/kg eq (pyrazole label) after 168 hours dosing. Mean total radioactive residues in egg whites achieved a plateau concentration of 0.04 mg/kg eq (phenyl label) after 168 hrs dosing and 0.03 mg/kg eq (pyrazole label) after 120 hours dosing.

Homogenised composite samples from the five birds per radiolabel were prepared for liver, fat, muscle, egg yolk and egg white. Sub-samples of liver, composite muscle, and egg white were extracted sequentially with acetonitrile (1 $\times$), acetonitrile/water 4:1 v/v (2–3 $\times$), acetonitrile/water 3:7 v/v (1–4 $\times$), water (1 $\times$) and acetonitrile (1 $\times$). Egg yolk and composite fat with skin were extracted first with dichloromethane (1 $\times$) and subsequently with the other solvents. Extracts and remaining solids were analysed by (combustion) LSC. Extracted residues were, where appropriate, combined and fractionated by liquid/liquid partition. Aqueous fractions from liver extracts were subject to β -glucuronidase (pH 5, 37°C , 18 hrs) to cleave glucuronide and sulphate conjugates and subsequently partitioned with diethyl ether. Fractions containing significant radioactive residues were subject to TLC with bio-image analysis to enable quantification and identification of residues. Residues were

identified by chromatographic comparison with authentic synthetic reference standards of parent, SYN546206, SYN546039, SYN546040, SYN546041, SYN546042, SYN546360, SYN508272, NOA449410, SYN545720.

Following solvent extraction, residue extractabilities were $\geq 83\%$ TRR for egg yolk and egg white and 68–73% TRR for skin with subcutaneous fat. Residue extractability was lowest in liver (48–49% TRR) and composite muscle (24–37% TRR). Identified radio-components present in extracted residues are summarised in Table 6.

Comparison of 2D-TLC chromatograms of pre- and post-hydrolysis fractions in liver shows that the released [^{14}C]exocons after hydrolysis originate predominantly from radio-components present on the origin (baseline) of the chromatogram. Unassigned radio-components that chromatograph away from the origin are unlikely to be conjugated metabolites for two reasons:

i) The solvent systems employed in the normal phase 2D-TLC analyses are quite non-polar in nature and the sulphate and glucuronide conjugated metabolites (or other logical conjugates) are intrinsically quite polar in nature and it would therefore be expected that those conjugates would be located on or very close to the origin of these chromatograms,

ii) Significant proportions of the “unassigned” chromatographed radio-components chromatograph well away from the origin of the 2D-chromatograms and in the region of identified “non-conjugated” and non-polar metabolites. The unassigned radio-components are therefore too non-polar to be characteristic of the conjugates characterised in i) above. [Syngenta, 2014c]

Solids remaining after extraction from liver (PES 51–52% TRR) and muscle (PES 63–76%TRR) in both radiolabelled experiments were further characterised in three parallel experiments. Liver and muscle PES were characterised to be similar in nature. Incubation under mild acid hydrolysis conditions (0.1 M HCl, 37 °C, 4 hrs) rendered only very minor proportions of the PES soluble. Following protein solubilisation using sodium dodecyl sulphate solution (SDS) and then a protein precipitation step, almost all of PES were shown to be associated with protein. Protease digestion (pH 7, 37 °C, 24 hrs + 48 hrs) solubilised almost all the PES. Analysis of protease hydrolysates showed them to comprise almost entirely of radio-components that were highly polar in nature.

All tissues, egg yolk and egg white samples were initially extracted and a 2D-TLC radio-component profile obtained from the principal residue-containing fractions within 104–107 days of sacrifice. The same fractions were again analysed using the same 2D-TLC conditions at the end of the analytical phase of the study (after 413–559 days of total storage). While there is no information on the first 104 days of storage, the results demonstrated that there had been no significant changes in the respective radio-component profiles during the interim storage period.

Note: The identified metabolite levels in muscle, skin/fat, egg yolk and egg white relate only to the free form of the metabolites. Since the unassigned fractions of these matrices were not subjected to glucuronidase treatment, it is not clear how much conjugated metabolites need to be added to these figures. It will however i) not be more than the sum of the “unassigned and baseline” figures and ii) in reality be closer to the polar “baseline” only figure due to the predominantly non-polar character of the “unassigned” residues precluding them from being conjugates.

Table 5 Total Radioactive residues and extractability in samples from laying hens treated with [^{14}C]benzovindiflupyr

Radiolabel	Sample	Extracted Radioactivity		Post-extracted solids		TRR ^a
		%TRR	mg/kg eq	%TRR	mg/kg eq	
[Phenyl- ^{14}C]-BVFP	Liver	48	0.090	52	0.098	0.19
	Egg Yolk	85	0.14	15	0.024	0.16
	Egg White	98	0.033	2.3	0.001	0.034
	Muscle	24	0.006	76	0.019	0.025
	Skin and Fat	68	0.022	32	0.011	0.033
[Pyrazole- ^{14}C]-BVFP	Liver	49	0.12	51	0.13	0.25
	Egg Yolk	83	0.15	17	0.030	0.18

Radiolabel	Sample	Extracted Radioactivity		Post-extracted solids		TRR ^a
		%TRR	mg/kg eq	%TRR	mg/kg eq	
	Egg White	98	0.031	2.3	0.001	0.032
	Muscle	37	0.013	63	0.023	0.036
	Skin and Fat	73	0.033	27	0.012	0.045

^a TRR calculated by summation of extracted radioactivity and post extracted solids (PES)

Table 6 Characterisation and Identification of components in samples from laying hens treated with [¹⁴C]-BVFP

	phenyl-[¹⁴ C]-BVFP					[pyrazole ¹⁴ C]-BVFP				
	Liver %TRR	Egg yolk %TRR	Egg White %TRR	Muscle %TRR	Skin and fat %TRR	Liver %TRR	Egg yolk %TRR	Egg White %TRR	Muscle %TRR	Skin and fat %TRR
TRR mg/kg eq ^a	0.19	0.16	0.034	0.025	0.033	0.25	0.18	0.032	0.036	0.045
parent	0.2	14	11	3.3	38	0.2	14	12	3.3	42
SYN546206 (N-demethyl- BVFP)	0.2 (< 0.1)	3.8	2.8	0.4	0.7	0.9 (0.5)	3.4	2.7	0.6	1.4
SYN546039 (BVFP-OH)	1.3 (0.6)	14	22	2.2	3.1	2.3 (0.5)	12	21	2.4	5.2
SYN546040 (BVFP-OH)	0.2	5.8	11	0.4	0.4	0.6	3.9	8.1	0.4	1.0
SYN546360	0.4 (0.2)	2.9	1.2	0.4	ND	0.5 (0.3)	2.5	1.7	0.4	ND
SYN546041 (N-demethyl- BVFP-OH)	1.6 (0.2)	12	11	3.5	1.8	2.3 (0.2)	11	11	2.7	2.2
SYN546042 (N-demethyl- BVFP-OH)	1.7	12	9.0	3.0	1.0	2.6	9.3	6.6	2.3	1.2
SYN546422 ^j	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
SYN508272	NA	NA	NA	NA	NA	0.6 (0.2)	0.9	4.0	4.4	1.1
NOA449410	NA	NA	NA	NA	NA	ND	ND	ND	ND	ND
SYN545720	NA	NA	NA	NA	NA	ND	ND	ND	ND	ND
Unassigned ^b		7.7	15.2	6.0	11.4		8.0	17.5	9.3	7.4
Organo soluble ^c	1.6					2.8				
Organo soluble ^d	1.5					2.5				
Water soluble ^e	23					18				
Baseline ^f	2.6	4.2	5.1	1.3	1.6	1.6	6.2	2.5	4.0	1.2
Remainder ^g	< 0.1	2.1	2.8	0.5	1.8	1.1	1.3	2.5	0.3	1.5
Other fractions ^h	2.1	9.9	2.3	1.5	1.6	1.7	11	< 0.1	2.2	1.6
PES ⁱ	52	15	2.3	76	32	51	17	2.3	63	27
Total	88	104	95	98	93	88	100	91	96	92

ND = Not detected (< 0.001 mg/kg eq)

NA = Not applicable for the label indicated

() = The values within parentheses indicate the proportion of the TRR that is in the conjugated forms. Values without parentheses are the sum of both the free and conjugated forms.

^a TRR calculated by summation of extracted radioactivity and post extracted solids (PES)

^b Unassigned radio-components which chromatographed away from the origin in normal phase 2D-TLC

Egg yolk, comprising at least 11–12 discrete components, no single one $\geq 1.3\%$ TRR (≥ 0.002 mg/kg eq)

Egg white, comprising at least 6–10 discrete components, no single one $\geq 4.9\%$ TRR (≥ 0.002 mg/kg eq)

Muscle, comprising at least 5–6 discrete components, no single one $\geq 3.7\%$ TRR (≥ 0.001 mg/kg eq)

Fat/skin, comprising at least 4–6 discrete components, no single one $\geq 6.0\%$ TRR (≥ 0.002 mg/kg eq)

^c Liver unassigned radio-components in pre-enzyme hydrolysis organo soluble fraction, which chromatographed away from the origin in normal phase 2D-TLC, comprising at least 8–11 discrete components, no single one $\geq 0.6\%$ TRR (≥ 0.001 mg/kg eq)

^d Liver unassigned radio-components in post enzyme hydrolysis organo soluble fraction which chromatographed away from the origin in normal phase 2D-TLC, comprising at least 10–16 discrete components, no single one $\geq 0.4\%$ TRR (≥ 0.001 mg/kg eq)

^e Liver unassigned highly polar radio-components in post enzyme hydrolysis water soluble partition phase, which

remained at the origin in normal phase 1D-TLC, with a small proportion moving slightly in a more polar solvent system normal phase 1D-TLC.

^f Polar material on origin of the radio-chromatogram using normal phase 2D-TLC

^g Diffuse areas of radioactivity within the chromatogram which cannot be assigned to discrete radioactive components

^h Extracted residues in fractions that were not analysed because levels of radioactivity were too low for analysis.

Liver (2 fractions), no single fraction comprised $\geq 1.4\%$ TRR (≥ 0.003 mg/kg eq)

Egg yolk (4 fractions), no single fraction comprised $\geq 6.1\%$ TRR (≥ 0.011 mg/kg eq)

Egg white (4 fractions), no single fraction comprised $\geq 1.2\%$ TRR (≤ 0.001 mg/kg eq)

Muscle (4 fractions), no single fraction comprised $\geq 1.5\%$ TRR (≤ 0.001 mg/kg eq)

Fat/skin (4 fractions), no single fraction comprised $\geq 1.6\%$ TRR (≤ 0.001 mg/kg eq)

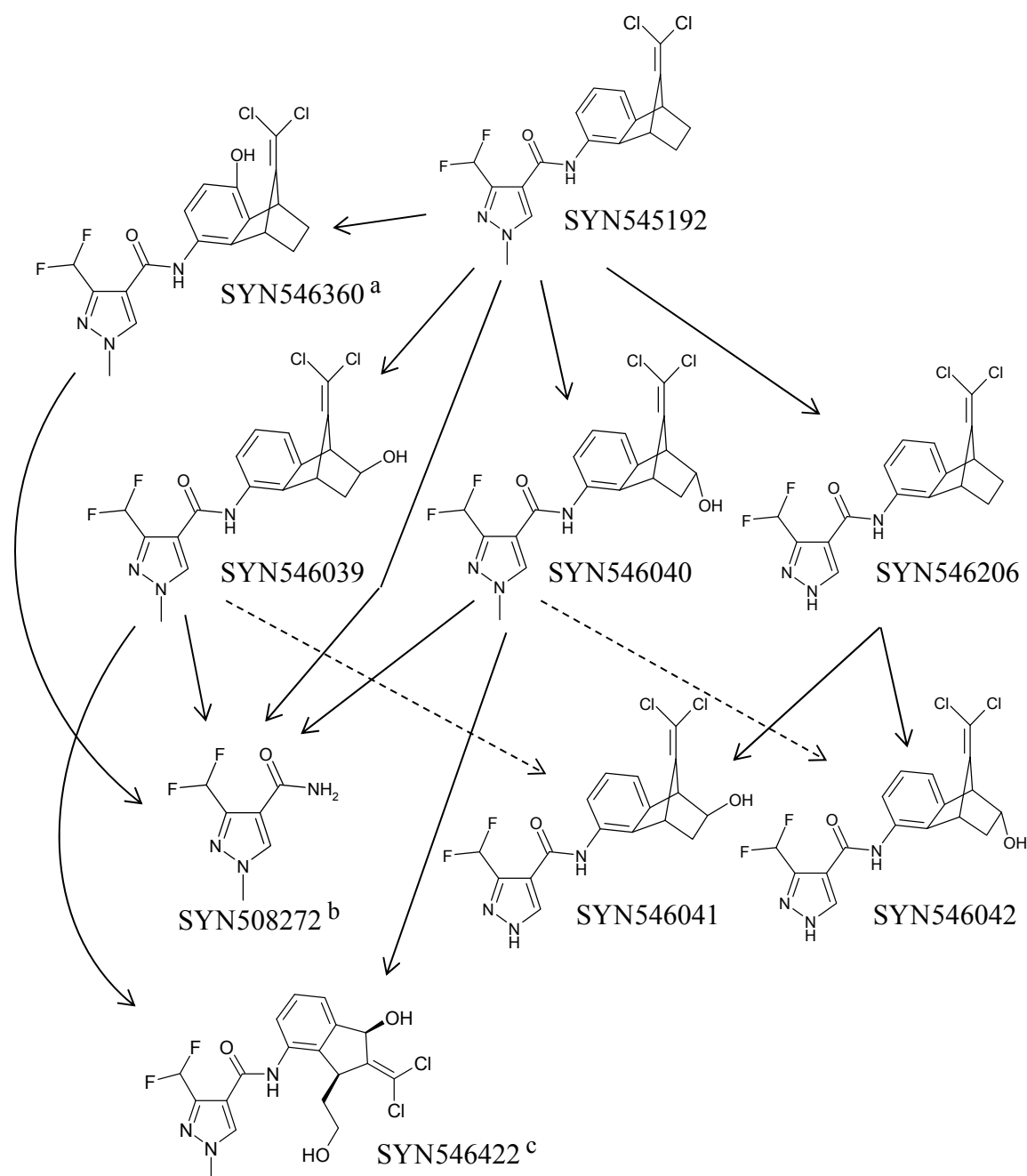
ⁱ Post extraction solids

^j A reference standard of SYN546422 was not chromatographed in the hen study fractions. However, the location of this metabolite in 2D-TLC is very characteristic as it consistently chromatographs between SYN546040 and SYN546042 (just below SYN546040) in the goat metabolism study. In the hen metabolism study no radio-components were observed in this region of the corresponding 2D-TLC analyses of egg and tissue fractions. It was therefore concluded that SYN546422 was not present in the hen egg and tissues [Syngenta, 2014b].

Overview of the metabolic pathway in livestock

Metabolism studies conducted with ruminants (lactating goats) and poultry (laying hens) and based on oral dosing, show that the metabolic pathways in ruminants and poultry are similar. Metabolism observed arose via:

- N-demethylation of the pyrazole ring to form SYN546206
- mono hydroxylation on the alicyclic ring to form the SYN546039 and SYN546040 isomers
- mono hydroxylation on the phenyl ring to form SYN546360
- N-demethylation of the pyrazole ring and subsequent mono hydroxylation of the alicyclic ring or mono hydroxylation of the alicyclic ring and subsequent N-demethylation of the pyrazole ring to form SYN546041 and SYN546042 isomers
- oxidative opening of the alicyclic ring to form SYN546422 (ruminants only)
- cleavage between the pyrazole and phenyl rings of parent or mono hydroxylated metabolites SYN546039/SYN546040 to form SYN508272
- conjugation of N-demethylated metabolite SYN546206, predominantly as its glucuronide conjugate
- conjugation of dihydroxylated metabolite SYN546422, predominantly as its glucuronide and/or sulphate conjugate (ruminants only)
- conjugation of mono hydroxylated metabolites SYN546039/SYN546040 and SYN546360, predominantly as their glucuronide and/or sulphate conjugates
- conjugation of mono hydroxylated N-demethylated metabolites SYN546041/SYN546042, predominantly as their glucuronide and/or sulphate conjugates
- conjugation of cleavage metabolite SYN508272 predominantly as its glucuronide conjugate.



a = Identified in hen tissues and eggs and goat bile as a very minor metabolite.

b = Identified in hen and goat commodities as a very minor metabolite

c = Identified in goat commodities only

Dotted arrows indicate an alternative pathway to continuous arrows.

All metabolites were present in their free (non-conjugated) forms in milk, egg, muscle and fat containing tissues; possible conjugates have not been released in the metabolism study. In liver and kidney all metabolites were present in their free and conjugated forms. The conjugates were characterised as glucuronides and/or sulphate esters.

Figure 2 Metabolic pathway of benzovindiflupyr in livestock

Plant metabolism

The Meeting received information on photolysis on leaves and the metabolic fate of benzovindiflupyr after foliar application on fruits (tomatoes), cereals (wheat) and pulses & oilseeds (soya beans).

Photolysis on leaves

To determine whether leaf photolysis is an appreciable route of degradation, the leaves of six green bean plants were treated topically with an unspecified formulation containing ^{12}C and ^{14}C -pyrazole-labelled-benzovindiflupyr [Syngenta, 2014d]. Four suitable healthy leaves of each plant were marked with a 5 cm diameter circle. The leaves on three plants were sprayed with the formulated material without adjuvant. The leaves on three other plants were sprayed with the formulated materials with adjuvant. Three different treatments were performed:

- The treated leaves of two of the plants (one with and one without adjuvant) were placed underneath artificial sunlight derived from a xenon lamp irradiation source. The treated leaves were placed as flat as possible underneath a quartz sheet and illuminated with a 14 hr light and 10 hr dark cycle.
- Two of the plants (one with and one without adjuvant) were placed in the greenhouse with a 14 hr light and 10 hr dark cycle.
- Two of the plants (one with and one without adjuvant) were placed outdoors between 3–10 July 2008, with the treated leaves facing south where possible.

Treated leaves from each plant were sampled at 2 hrs after treatment and at DAT=1, 4 and 7. The leaves underneath the Suntest were scorched brown and dried in some cases, especially at the 7 DAT sampling. Whole leaves were extracted twice with methanol/water (70:30, v/v) and radioactivity in the extracts and remaining solids were quantified by (combustion) LSC.

Extract recoveries were 70–94% TAR, while remaining solids represented 0.1–4.4% TAR. The missing radioactivity was assumed to have been translocated into the areas of the plant and/or lost by evaporation. At DAT =7 total recovery was > 98% TAR.

HPLC chromatograms of the extracts demonstrated very minor degradation in all three situations at DAT=7. The minor degradates formed gave qualitatively similar chromatographic pattern. The highest level of degradation was found on the leaves grown in a greenhouse, either with or without adjuvant.

Metabolism in tomatoes

The metabolic fate of benzovindiflupyr was studied in indoor-grown tomatoes (variety F1 Orange Pixie, a dwarf variety) after four foliar spray applications to the plants [Oddy, 2011, SYN545192_10117]. Two radio-labelled forms were used in the study, [phenyl- ^{14}C]-BVFP and [pyrazole- ^{14}C]-BVFP (see Figure 1). Plants were grown in containers in sandy loam soil. The phenyl and pyrazole test items were applied at an actual rate of $0.138 + 0.132 + 0.129 + 0.128$ kg ai/ha and $0.138 + 0.131 + 0.129 + 0.127$ kg ai/ha, respectively, at weekly intervals. The spray solution was applied as an EC formulation to 6 week old plants (BBCH 71–72) in a spray volume of 600 L/ha using a lance and trigger sprayer with CO_2 propellant to give as even a cover as possible. Mature tomato fruits (176–663 g) were harvested 1 and 14 days after the final application. At each harvest interval fruits were surface washed immediately following sampling by immersing the fruit for 30 sec in acetonitrile (2 $\times$). Samples washes and samples were stored frozen at -18°C for 17–23 days until extraction and 21–22 months until last analysis.

The washed frozen fruit was homogenised and sequentially extracted with acetonitrile (1 $\times$), acetonitrile/water 4/1 v/v (3 $\times$), acetonitrile/water 3/7 v/v (1 $\times$), water (1 $\times$), and acetonitrile (1 $\times$). Radioactivity in wash, extracts and post-extraction solids (PES) was determined by LSC and combustion/LSC. The total radioactive residues (TRR) in tomato fruits taken at 1 and 14 days after the final application are shown in Table 7. The TRRs in the samples derived from the pyrazole experiment were higher than those derived from the phenyl experiment. The majority of the radioactivity (65–79% TRR) could be washed off by acetonitrile. The total extractability of residues into wash and solvents for all samples was $\geq 99\%$ TRR.

Proportionate aliquots of the acetonitrile wash and extracts 1–4 were combined; extracts 5–7 were not analysed further. The combined extracts were concentrated by evaporation and residues were

partitioned into diethyl ether/hexane (3:1, v/v). The resulting organic phase was characterised using 2D-TLC with phosphor image analysis. The remaining aqueous phase was not analysed further. Radiolabelled components were co-chromatographed with authentic reference standards of parent, SYN546206, SYN546039, SYN546040, SYN546041, SYN546042, NOA449410, SYN545720, SYN508272 using two dissimilar solvent systems. Results are shown in Table 8. In total a high level of identification was achieved in all tomato fruits (> 91.8% TRR).

The surface wash was analysed by 2D-TLC at the start and end of the analytical phase of the study to demonstrate storage stability. The original samples were re-analysed at the end of the analytical phase of the study, following 21–22 months of storage. Comparison of the initial and final radiolabelled component profiles obtained showed that no significant change in the profiles had occurred during the interim period of storage. Storage stability data do not exist for the 1 DALT 1 and 14 DALT extracts (confirmed, Syngenta, 2014b). However, since the majority of the radioactive residue (65–79% TRR) is derived from the surface wash, and 90.8–95.1% TRR is identified as parent, this is considered to have no impact on the results of this study.

Table 7 Total Radioactive Residues and extractability in tomatoes treated with [^{14}C]benzovindiflupyr

Radiolabel	Mature fruit Commodity conditions	Initial Fruit Wash		Washed Fruit				TRR ^a
				Extracted Radioactivity		Post-extracted solids		
		%TRR	mg/kg eq	%TRR	mg/kg eq	%TRR	mg/kg eq	mg/kg eq
[Phenyl- ¹⁴ C]- BVFP	4× (0.128– 0.138) kg ai/ha, DALT = 1	79	0.037	20	0.0094	0.8	0.0004	0.047
	4× (0.127– 0.138) kg ai/ha, DALT = 14	77	0.071	22	0.020	0.5	0.0005	0.092
[Pyrazole- ¹⁴ C]- BVFP	4× (0.128– 0.138) kg ai/ha, DALT = 1	73	0.13	26	0.048	0.3	0.0005	0.18
	4× (0.127– 0.138) kg ai/ha, DALT = 14	65	0.095	34	0.050	0.8	0.0012	0.15

^a TRR determined by summation of radioactivity present in the surface wash, solvent extracts and debris

Table 8 Identification and characterisation of residues in tomatoes treated with [^{14}C]benzovindiflupyr

	Phenyl- ^{14}C		Pyrazole- ^{14}C	
	DALT = 1	DALT = 14	DALT = 1	DALT = 14
TRR, mg/kg eq	0.047	0.092	0.18	0.15
Component	% TRR	%TRR	% TRR	%TRR
parent	95	93	91	92
SYN546206 (N-demethyl-BVFP)	ND	0.4	0.4	0.5
SYN546039 (BVFP-OH)	ND	0.2	0.3	0.4
SYN546040 (BVFP-OH)	ND	ND	ND	0.1
SYN546041 (N-demethyl-BVFP-OH)	ND	ND	ND	ND
SYN546042 (N-demethyl-BVFP-OH)	ND	ND	ND	ND
SYN508272	NA	NA	ND	ND
NOA449410	NA	NA	0.2	0.2
SYN545720	NA	NA	0.1	0.1
Unassigned ^a	ND	0.7	0.7	0.7
Baseline ^b	ND	0.5	0.3	0.5

	Phenyl- ¹⁴ C		Pyrazole- ¹⁴ C	
	DALT = 1	DALT = 14	DALT = 1	DALT = 14
Other fractions ^c	3.6	2.0	4.7	5.4
PES ^d	0.8	0.5	0.3	0.8
Total	100	97	98	101

ND = Not detected, i.e. < 1.5 times the background radioactivity in samples with no radioactivity (< 0.0001 mg/kg eq)

NA = Not applicable to this radiolabel

^a Unassigned radio-components which chromatographed away from the origin in 2D-TLC (normal phase) and comprised at least 3 compounds

^b Polar material on origin of the radiochromatogram using 2D-TLC (normal phase).

^c Extracted residues in fractions that were not analysed because levels of radioactivity were too low for analysis. These comprised solvent extracts 5–7 and the aqueous partition of extracts 1–4.

^d Post-extraction solids; radioactivity remaining in the debris after extraction with solvent.

Metabolism in wheat

The metabolic fate of benzovindiflupyr was studied in indoor-grown spring wheat (variety Tybalt) after two foliar spray application to the plants [Simmonds and Mackenzie, 2011, SYN545192_10136]. Two radiolabelled forms were used in the study, [phenyl-¹⁴C]-BVFP and [pyrazole-¹⁴C]-BVFP (see Figure 1). Plants were grown in containers in sandy loam soil. The phenyl and pyrazole test items were applied at an actual rate of 2 times 0.14 kg ai/ha, at a 35 day interval. The first application was conducted at BBCH 31 and the second at BBCH 69. The spray solution was applied as an EC formulation in a spray volume of 300 L/ha using a lance and trigger sprayer with CO₂ propellant to spray from above. Samples were collected at BBCH 39 (forage, 9 days after the first application), BBCH 77 (hay, 10 days after the second application) and BBCH 89 (mature grain and straw, 40 and 41 days after the second application). The hay samples were laid out in the greenhouse to dry for 4 days. Sample sizes were 0.71–0.91 kg forage, 0.61–0.70 kg hay, 1.2–2.0 kg straw and 2.1 kg grain. Samples were stored frozen at –18 °C for 18–60 days until extraction and 19–23 months until analysis.

Wheat forage, hay and straw were homogenised and sequentially extracted with acetonitrile (1×), acetonitrile/water 4/1 v/v (2×–4×), acetonitrile/water 3/7 v/v (1×), water (1×) and acetonitrile (1×). Wheat grain was homogenised and sequentially extracted with acetonitrile (1×), acetonitrile/water 4/1 v/v (3×) and acetonitrile/water 1/1 v/v (2×). Radioactivity in extracts and post-extraction solids (PES) was determined by LSC and combustion/LSC. The total radioactive residues (TRR) in forage, hay, straw and grain are shown in Table 9. Residue levels derived from the two radiolabelled experiments were generally similar on a commodity basis. The total level of extractability was ≥ 97%TRR for all commodities.

Proportionate aliquots of the acetonitrile and acetonitrile/water (4/1) extracts were combined; the other fractions were not analysed further. The combined extracts were concentrated by evaporation and if necessary partitioned between water and diethyl ether/hexane (3:1, v/v).

All fractions were analysed by 2D-TLC with phosphor image analysis. Identification was conducted by co-chromatography with authentic reference standards of parent, SYN546206, SYN546039, SYN546040, SYN546322, SYN546323, SYN546041, SYN546042, NOA449410, SYN545720, SYN508272 using two dissimilar solvent systems. Results are shown in Table 10.

Selected straw and grain fractions were subjected to hydrolysis procedures involving the use of bovine rumen fluid, pectinase enzyme or mild base followed by pectinase enzyme to release the metabolites and to characterise the nature of the conjugates. Cleavage following pectinase hydrolysis (pH 5, 18 hrs at 30 °C) alone characterised the residue as a glycoside conjugate. Cleavage following mild base (0.05 M NaOH, 18 hrs, room temperature) and pectinase hydrolysis (pH 5, 18 hrs at 30 °C) characterised the residue as malonyl glycoside conjugate. Cleavage following bovine rumen fluid (pH 7, 24 hrs at 39 °C), effected greater hydrolytic conversion compared to pectinase and base pectinase. Hydrolysates were adjusted to pH 2–3 and partitioned into diethyl ether. The remaining aqueous phase was not analysed further. The resulting organic phase was characterised using 2D-TLC. Results

are shown in Table 10. Several of metabolites were present in the free and conjugated form. Significant proportions of the SYN546039 conjugates in straw and grain were characterised as glycoside and malonyl glycoside conjugates. In total > 86% TRR was identified for each commodity.

Combined extracts were analysed by TLC within 1 to 5 months of sample harvest and were analysed again following 19 to 23 months frozen storage to demonstrate storage stability. Comparison of the initial and final radio-component profiles obtained showed no significant change in the profiles had occurred during the interim period of storage.

Table 9 Total Radioactive Residues and Extractability in Wheat Treated with [^{14}C]benzovindiflupyr

	Wheat Commodity	Conditions	Extracted Radioactivity		Post-extracted solids		TRR ^a
			%TRR	mg/kg eq	%TRR	mg/kg eq	mg/kg eq
[Phenyl- ^{14}C]-BVFP	Forage	1 × 0.137 kg ai/ha; DAFT = 9	100	3.0	0.3	0.009	3.0
	Hay	2 × (0.135–0.137) kg ai/ha; DALT = 11	98	4.8	1.5	0.074	4.9
	Straw	2 × (0.135–0.137) kg ai/ha; DALT = 40	97	7.9	2.7	0.22	8.1
	Grain	2 × (0.135–0.137) kg ai/ha; DALT = 40	97	0.12	2.6	0.003	0.12
[Pyrazole- ^{14}C]-BVFP	Forage	1 × 0.142 kg ai/ha; DAFT = 9	100	2.1	0.2	0.004	2.1
	Hay	2 × (0.135–0.142) kg ai/ha; DALT = 11	99	6.3	1.1	0.070	6.4
	Straw	2 × (0.135–0.142) kg ai/ha; DALT = 41	98	8.9	2.2	0.20	9.0
	Grain	2 × (0.135–0.142) kg ai/ha; DALT = 41	97	0.090	2.7	0.002	0.092

^a TRR derived from the summation of the radioactivity present in the solvent extracts and debris

DALT = Days after last treatment

DAFT = Days after first treatment

Table 10 Identification and characterisation of residues in wheat commodities treated with [^{14}C]benzovindiflupyr

	Forage	Forage	Hay	Hay	Straw	Straw	Grain	Grain
	Phenyl- ^{14}C	Pyrazole- ^{14}C	Phenyl- ^{14}C	Pyrazole- ^{14}C	Phenyl- ^{14}C	Pyrazole- ^{14}C	Phenyl- ^{14}C	Pyrazole- ^{14}C
TRR mg/kg eq	3.0	2.1	4.9	6.4	8.1	9.0	0.12	0.092
Component	% TRR	% TRR	% TRR	% TRR	% TRR	% TRR	% TRR	% TRR
parent	100	103	89	93	81	84	87	84
SYN546206 (N-demethyl-BVFP)	0.3	0.3	0.9	0.8	1.4 (0.1)	1.1 (ND)	2.3	2.0
SYN546039 (BVFP-OH)	0.1	0.1	0.7	0.5	2.8 (1.8)	2.2 (1.3)	3.6 (1.7)	2.0 (0.7)
SYN546040 (BVFP-OH)	ND	ND	ND	ND	< 0.1	ND	< 0.1	0.1
SYN546322	ND	ND	ND	ND	ND	ND	ND	ND
SYN546323	ND	ND	ND	ND	ND	ND	ND	ND
SYN546041 (N-demethyl-BVFP-OH)	ND	ND	ND	ND	0.3 (0.2)	0.3 (0.3)	0.8 (0.5)	0.5 (0.3)
SYN546042	ND	ND	ND	ND	ND	ND	ND	ND

	Forage	Forage	Hay	Hay	Straw	Straw	Grain	Grain
	Phenyl- ¹⁴ C	Pyrazole- ¹⁴ C	Phenyl- ¹⁴ C	Pyrazole- ¹⁴ C	Phenyl- ¹⁴ C	Pyrazole- ¹⁴ C	Phenyl- ¹⁴ C	Pyrazole- ¹⁴ C
(N-demethyl-BVFP-OH)								
SYN508272	NA	ND	NA	0.2	NA	0.4 (0.1)	NA	0.3 (0.1)
NOA449410	NA	ND	NA	0.2	NA	0.4 (0.1)	NA	0.6
SYN545720	NA	ND	NA	ND	NA	ND	NA	ND
Unassigned ^a	1.0	0.8	2.2	1.8	2.1	2.0	3.7	3.3
Baseline ^b	1.2	1.0	1.3	1.5	5.3	6.1	2.1	3.8
Remainder ^c	0.0	0.2	0.8	0.6	0.3	1.3	0.1	0.4
Other fractions ^d	1.2	1.2	1.1	1.0	2.8	2.7	2.2	3.2
Unextracted ^e	0.3	0.2	1.5	1.1	2.7	2.2	2.6	2.7
Total	104	106	98	101	99	103	104	103

ND = Not detected, i.e. < 1.5 times the background radioactivity in samples with no radioactivity (< 0.00015 mg/kg eq)

NA = Not applicable to this radiolabel

() = The main figures are the sum of both the free and conjugated forms, the figures in parentheses indicate the proportion of the residue that was found in the conjugated form

^a Unassigned radio-components which chromatographed away from the origin in 2D-TLC (normal phase)

Forage, phenyl label: comprising at least 4 discrete components, no single one > 0.6% TRR (> 0.018 mg/kg eq)

Forage, pyrazole label: comprising at least 4 discrete components, no single one > 0.5% TRR (> 0.011 mg/kg eq)

Hay, phenyl label: comprising at least 4 discrete components, no single one > 1.5% TRR (> 0.074 mg/kg eq)

Hay, pyrazole label: comprising at least 4 discrete components, no single one > 1.3% TRR (> 0.083 mg/kg eq)

Straw, phenyl label: comprising at least seven discrete components, no single one ≥ 0.4% TRR (≥ 0.032 mg/kg eq)

Straw, pyrazole label: comprising at least six discrete components, no single one ≥ 0.5% TRR (≥ 0.045 mg/kg eq)

Grain, phenyl label: comprising at least eight discrete components, no single one ≥ 0.8% TRR (≥ 0.001 mg/kg eq)

Grain, pyrazole label: comprising at least seven discrete components, no single one ≥ 0.9% TRR (≥ 0.001 mg/kg eq)

^b Polar material on origin of the radiochromatogram using 2D-TLC (normal phase)

Straw, pyrazole/phenyl label from fraction F4 (≤ 0.8% TRR, ≤ 0.065 mg/kg eq) and F5 (≤ 5.4% TRR, ≤ 0.489 mg/kg eq).

Polar material from phenyl label F5, likely to comprise more than one radio-component. Polar material from pyrazole label F5: Further 2D TLC using a more polar solvent system (2D-TLC SSC) showed this to comprise at least three discrete components no single one of which ≥ 0.9% TRR (≥ 0.081 mg/kg).

^c The remainder comprises diffuse areas of radioactivity within the chromatogram which cannot be assigned to discrete radioactive components.

^d Extracted residues in fractions that were not analysed, because these fractions contained very low levels of radioactivity.

No single fraction comprised > 0.9% TRR (> 0.027 mg/kg eq) in forage, > 0.7% TRR (> 0.044 mg/kg eq) in hay, ≥ 1.7% TRR (≥ 0.154 mg/kg eq) in straw, ≥ 2.5% TRR (≥ 0.002 mg/kg eq) in grains in either radiolabelled experiment.

^e Radioactivity remaining in the debris after extraction with solvent.

Metabolism in soya beans

The metabolic fate of benzovindiflupyr was studied in indoor-grown soya beans (variety S12-C2) after two foliar spray application to the plants [Oddy, 2012, SYN545192_10102]. Two radiolabelled forms were used in the study, [phenyl-¹⁴C]-BVFP and [pyrazole-¹⁴C]-BVFP, subsequently referred to as phenyl and pyrazole test item (see Figure 1). Plants were grown in containers in sandy loam soil. The phenyl and pyrazole test items were applied at an actual rate of two times 0.12–0.13 kg ai/ha, at a 22 day interval. The first application was conducted at BBCH 55–60 and the second at BBCH 75. The spray solution was applied as an EC formulation in a spray volume of 300 L/ha using an automated track sprayer. Commodities were taken at BBCH 70 (forage, 11 days after first application), BBCH 85 (hay, 13 days after second application), and BBCH 89 (mature bean, 30 days after second application). Forage hay samples were left in the greenhouse to air dry for 6 days. Mature soya bean pods were dried for 2 days, after which the seeds were taken from the pods. Samples (0.11 kg fresh forage, 0.10 kg dry hay, 0.25 kg dry seeds) were stored frozen at –15 °C for 20–37 days until extraction and 29–31 months until analysis.

Soya bean forage was homogenised and sequentially extracted with acetonitrile (1×), acetonitrile/water 4:1, v/v (2×), acetonitrile/water 3:7, v/v (1×), water (1×) and acetonitrile (1×). Soya bean hay and beans were homogenised and sequentially extracted with hexane (3×), diethyl ether

(2×), acetonitrile (2×), acetonitrile/water 4:1 v/v (2×–7×), acetonitrile/water 3:7 v/v (1×–2×), water (1×), and acetonitrile (1×). Radioactivity in extracts and post-extraction solids (PES) was determined by LSC and combustion/LSC. Total radioactive residues (TRR) present in the soya bean commodities are shown in Table 11. Residue levels in the soya bean seeds derived from the pyrazole experiment were approximately three times higher than those in the phenyl experiment. The total level of extractability was $\geq 99\%$ TRR (forage), $\geq 98\%$ TRR (hay), $\geq 89\%$ TRR (beans, phenyl experiment) and $\geq 98\%$ TRR (beans, pyrazole experiment).

For soya bean forage proportionate aliquots of the acetonitrile and acetonitrile/water 4:1, v/v, extracts were combined and the resulting fraction ($\geq 98\%$ TRR) was analysed by 2D-TLC. An aliquot of the combined extracts was subjected to hydrolysis procedures involving the use of bovine rumen fluid (pH 7, 24 hrs, 39 °C) to release the metabolites. The hydrolysate was analysed by 2D-TLC.

For soya bean hay proportionate aliquots of hexane, diethyl ether and acetonitrile extracts were combined and resulting fractions containing 76% TRR (phenyl experiment) and 67% TRR (pyrazole experiment) were analysed by 2D TLC. Similarly, proportionate aliquots of the acetonitrile/water extracts were combined and the resulting fractions containing 18% TRR (phenyl experiment) and 26% TRR (pyrazole experiment) were analysed by 2D TLC and 1D-TLC. An aliquot of the combined acetonitrile/water extracts was subjected to hydrolysis procedures involving the use of bovine rumen fluid, pectinase enzyme (pyrazole experiment only), and mild base followed by pectinase enzyme (pyrazole experiment only) to release the metabolites and to characterise the nature of the conjugates. The hydrolysates were analysed by 2D-TLC.

Cleavage following bovine rumen fluid (pH 7, 24 hrs at 39 °C) was the most effective at hydrolysing baseline conjugate material and effected greater hydrolytic conversion compared to pectinase and base pectinase. Cleavage following pectinase hydrolysis (pH 5, 18 hrs at 30 °C) alone is the least effective hydrolysis method and characterised the residue as a glycoside conjugate. Cleavage following mild base (0.05 M NaOH, 18 hrs, room temperature) and pectinase hydrolysis (pH 5, 18 hrs at 30 °C) characterised the residue as malonyl glycoside conjugate.

For soya bean seeds, proportionate aliquots of hexane, diethyl ether and acetonitrile were combined. The resulting fraction (phenyl: 35% TRR; pyrazole: 10% TRR) was concentrated by evaporation to an oil, dissolved in hexane and partitioned into acetonitrile/water (9:1, v/v) to remove endogenous oil. The hexane phase (phenyl: 7.2% TRR; pyrazole: 1.2% TRR) was not further analysed. The aqueous acetonitrile fraction (phenyl: 24% TRR; pyrazole: 8.7% TRR) was analysed by 2D-TLC. Similarly, proportionate aliquots of the primary acetonitrile/water extracts were combined and the resulting fractions (phenyl: 48% TRR; pyrazole: 85% TRR) were analysed by 2D-TLC. An aliquot of the combined primary acetonitrile/water extracts was subjected to a protein precipitation clean-up step: addition of acetone and cooling for 18 hrs at 4 °C. The resulting supernatant (phenyl: 46% TRR; pyrazole: 82% TRR) was analysed by 2D-TLC and 1D-TLC. A separate aliquot of the combined primary acetonitrile/water extracts was concentrated and subjected to hydrolysis procedures involving the use of bovine rumen fluid (pH 7, 24 hrs at 39 °C). The resulting hydrolysate was partitioned between water and diethyl ether/hexane (9:1 v/v). The organic phase (phenyl: 41% TRR; pyrazole: 55% TRR) was analysed by 2D-TLC and 1D-TLC. The aqueous phase derived from the phenyl experiment (8.1% TRR) was not analysed further. The aqueous phase derived from the pyrazole experiment (30% TRR) was subjected to acid hydrolysis (0.5 M HCl, 6 hrs, 100 °C) and the resulting hydrolysate was partitioned into diethyl ether/hexane 4:1 v/v. The organo soluble fraction (22% TRR) was analysed by 2D-TLC. The aqueous phase (0.0% TRR) was not analysed further.

All fractions were analysed by 2D-TLC and/or 1D-TLC with phosphor image analysis. Identification was conducted by co-chromatography with authentic reference standards of parent, SYN546206, SYN546039, SYN546040, SYN546041, SYN546042, NOA449410, SYN545720, SYN508272, SYN545720-aspartic acid conjugate, SYN545720-monosaccharide conjugate using two dissimilar solvent systems. Results are shown in Table 12.

In soya bean forage and hay, high levels of identification of the extracted residues were achieved ($\geq 83\%$ TRR). A significant proportion of the conjugates of SYN546039 in hay was characterised as glycoside (minor proportion) and malonyl glycoside (major proportion) conjugates.

In soya bean seeds, high levels of identification of the extracted residues were achieved for the pyrazole experiment (67% TRR) but lower levels of identification were found for the phenyl experiment (41% TRR) where absolute residue levels were lowest. Significant proportions of the conjugated residue of SYN545720 in seeds were identified as the aspartic acid conjugate (12% TRR) and monosaccharide conjugate (11% TRR).

Combined extracts were analysed by TLC within 3 months of sample harvest and were analysed again following 29-31 months frozen storage to demonstrate storage stability. Comparison of the initial and final radio-component profiles obtained showed no significant change in the profiles during the interim period of storage.

Table 11 Total Radioactive Residues and extractability in soya bean commodities treated with [^{14}C]benzovindiflupyr

Radiolabel	Soya bean commodity	Conditions	Extracted Radioactivity		Post-extracted solids		TRR ^a
			%TRR	mg/kg eq	%TRR	mg/kg eq	mg/kg eq
[Phenyl- ^{14}C]-BVFP	forage	1 × 0.121 kg ai/ha; DAFT = 11	99	3.3	1.0	0.033	3.4
	hay	2 × (0.121–0.123) kg ai/ha; DALT = 13	98	14	2.3	0.32	14
	seeds	2 × (0.121–0.123) kg ai/ha; DALT = 30	89	0.026	11	0.003	0.029
[Pyrazole- ^{14}C]-BVFP	forage	1 × 0.126 kg ai/ha; DAFT 11	99	4.0	0.9	0.037	4.1
	hay	2 × (0.120–0.126) kg ai/ha; DALT 13	98	12	2.4	0.31	13
	seeds	2 × (0.120–0.126) kg ai/ha; DALT = 30	98	0.099	2.1	0.002	0.10

^a TRR derived from the summation of the radioactivity present in the solvent extracts and debris

DALT = Days after last treatment

DAFT = Days after first treatment

Table 12 Identification and characterisation of residues in soya bean commodities treated with [^{14}C]benzovindiflupyr

	Forage	Forage	Hay	Hay	Seeds	Seeds
	Phenyl- ^{14}C	Pyrazole- ^{14}C	Phenyl- ^{14}C	Pyrazole- ^{14}C	Phenyl- ^{14}C	Pyrazole- ^{14}C
TRR, mg/kg eq	3.4	4.1	14	13	0.029	0.10
Component	% TRR	% TRR	% TRR	% TRR	% TRR	% TRR
parent	83	85	72	67	31	15
SYN546206 (N-demethyl-BVFP)	1.4	1.6	1.5	1.6	4.6	2.0
			(0.1)	(0.2)	(ND)	(2.0)
SYN546039 (BVFP-OH)	9.9	9.2	11	12	1.2	0.4
	(9.0)	(8.5)	(10)	(12)	(1.0)	(0.4)
SYN546040 (BVFP-OH)	0.1	0.2	0.2	0.3	0.8	0.1
	(< 0.1)	(0.1)	(0.2)	(0.3)	(0.4)	(0.1)
SYN546041 (N-demethyl-BVFP-OH)	0.4	0.5	0.6	0.7	3.2	1.1
	(0.3)	(0.4)	(0.5)	(0.6)	(3.2)	(1.1)
SYN546042 (N-demethyl-BVFP-OH)	ND	ND	0.1	0.2	0.2	ND
			(0.1)	(0.2)	(0.2)	
SYN508272	NA	0.1	NA	0.1	NA	ND
		(< 0.1)		(0.1)		
NOA449410	NA	0.3	NA	0.3	NA	1.2
		(0.1)		(0.1)		(1.2)
SYN545720	NA	0.3	NA	0.3	NA	47

	Forage	Forage	Hay	Hay	Seeds	Seeds
	Phenyl- ¹⁴ C	Pyrazole- ¹⁴ C	Phenyl- ¹⁴ C	Pyrazole- ¹⁴ C	Phenyl- ¹⁴ C	Pyrazole- ¹⁴ C
		(< 0.1)		(ND)		(30)
Unassigned ^a	ND	1.4	1.2	1.5	23	11
Baseline ^b	1.8	1.1	2.7	2.0	5.0	6.6
Remainder ^c	4.3	1.2	4.3	6.7	5.6	2.6
Other fractions ^d	1.6	1.8	1.0	1.7	8.9	4.3
PES ^e	1.0	0.9	2.3	2.5	11.1	2.1
Total	104	103	97	97	95	93

Not detected, i.e. < 1.5 times the background radioactivity in samples with no radioactivity (< 0.00013 mg/kg eq)

NA = Not applicable to this radiolabel

() = The main figures are the sum of both the free and conjugated forms, the figures in parentheses indicate the proportion of the residue that was found in the conjugated form

^a Unassigned radio-components which chromatographed away from the origin in 2D-TLC (normal phase)

Forage phenyl label: no discrete components;

Forage pyrazole label: comprising at least 5 discrete components, no single one of which > 0.8% TRR (> 0.03 mg/kg eq);

Hay phenyl label: comprising at least 4 discrete components, no single one > 0.6% TRR (> 0.084 mg/kg eq);

Hay pyrazole label: comprising at least 6 discrete components, no single one > 0.6% TRR (> 0.075 mg/kg eq);

Seeds phenyl label: comprising at least 7 discrete components, no single one > 14.1% TRR (> 0.0041 mg/kg eq);

Seeds pyrazole label: comprising at least 5 discrete components, no single one > 2.5% TRR (> 0.0025 mg/kg eq)

^b Polar material on origin of the radiochromatogram using 2D-TLC (normal phase)

^c Diffuse areas of radioactivity within the chromatogram which cannot be assigned to discrete radioactive components.

^d Extracted residues, produced during processing that were too low for analysis. No single fraction comprised > 1.4% TRR (> 0.06 mg/kg eq) in forage or > 1.2% TRR (> 0.151 mg/kg eq) in hay or > 3.5% TRR (> 0.0004 mg/kg eq) in seeds in either radiolabelled experiment.

^e Post extraction solids; radioactivity remaining in the debris after extraction.

Overview of the metabolic pathway in plants

Metabolism studies conducted with crops representative of cereal/grass (spring wheat), fruits (tomato) and pulses/oilseeds (soya bean) and based on foliar treatment show that the metabolic pathways in the three crop groups are similar. The nature of the terminal residue depends on the relative importance of the various routes of metabolism and the nature and extent of the conjugation reactions. The level of metabolism of benzovindiflupyr was most extensive in pulses/oilseeds (soya bean). Metabolism observed arose via:

- N-demethylation of the pyrazole ring to form SYN546206
- mono hydroxylation on the alicyclic ring to form the SYN546039 and SYN546040 isomers
- subsequent N-demethylation of the pyrazole ring and mono hydroxylation of the alicyclic ring or subsequent mono hydroxylation of the alicyclic ring and N-demethylation of the pyrazole ring to form SYN546041 and SYN546042 isomers
- cleavage between the pyrazole and phenyl rings of parent or mono hydroxylated metabolites SYN546039/SYN546040 to form SYN508272 and subsequent hydrolysis of the amide functionality of SYN508272 to form NOA 449410 and subsequent demethylation to form SYN545720
- cleavage between the pyrazole and phenyl rings of the N-demethylated metabolites SYN546206 or SYN546041/SYN546042 to form SYN545720
- conjugation of N-demethylated metabolite SYN546206
- conjugation of mono hydroxylated metabolites SYN546039/SYN546040, predominantly as their glycoside and malonyl glycoside conjugates
- conjugation of mono hydroxylated and N-demethylated metabolites SYN546041/SYN546042
- conjugation of hydroxylated cleavage metabolites NOA449410 and SYN545720, the latter metabolite found predominantly as its aspartic acid and sugar conjugates.

In tomato fruits, wheat grains, wheat forage/hay/straw, soya bean forage/hay the major compound in the residue is parent. In soya bean seeds the major compound is the cleavage compound SYN545720 with significant amounts of parent present. The major pathway in soya bean seeds is cleavage between the pyrazole and phenyl rings combined with N-demethylation of the pyrazole moiety (either before or after cleavage) to form SYN545720 followed by conjugation of SYN545720 to aspartic acid and sugar. The overall metabolic pathways in rotated crops are similar to those observed in primary crops.

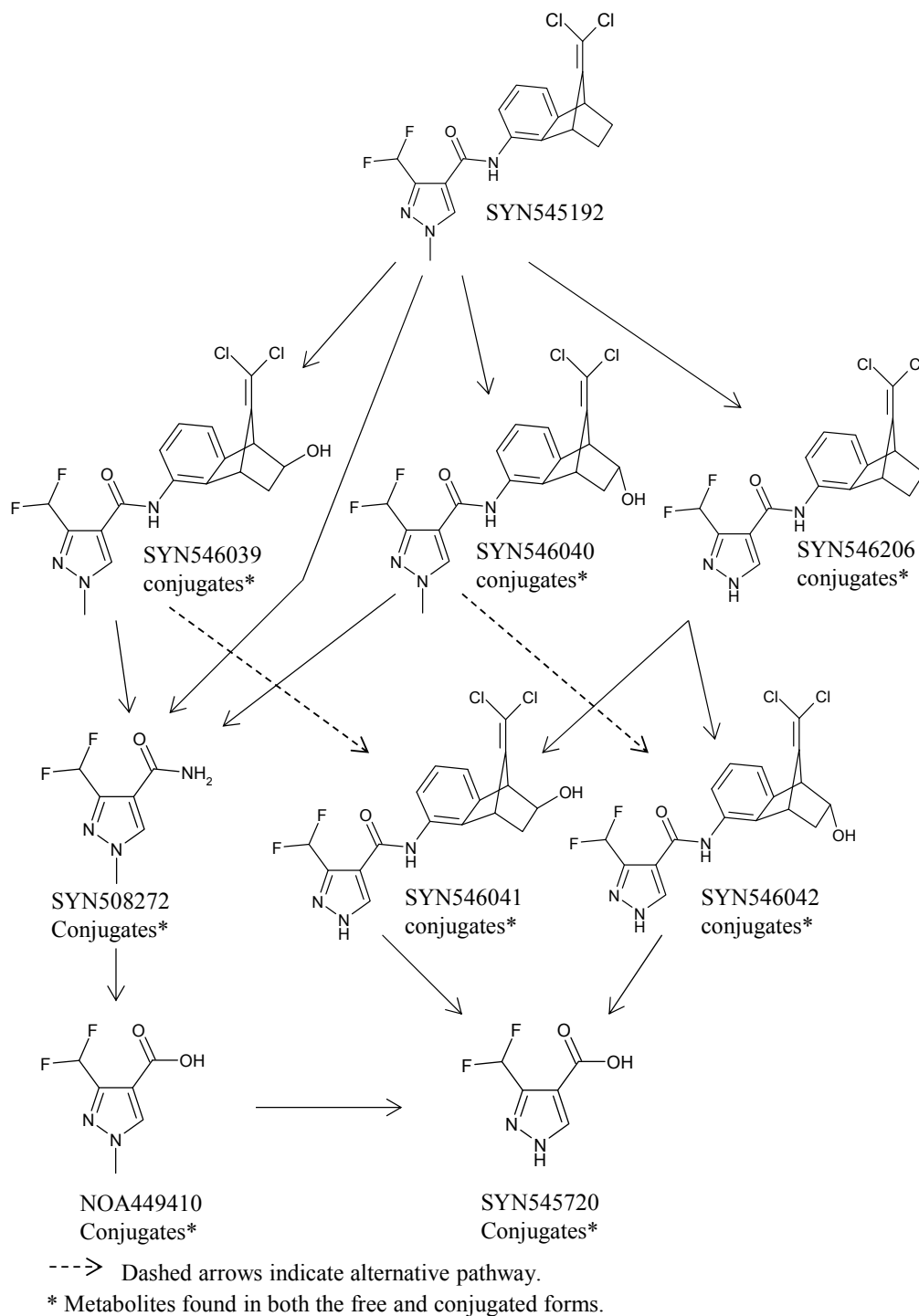


Figure 3 Metabolic pathway of benzovindiflupyr in plant commodities

Environmental fate in soil

The Meeting received information on aerobic/anaerobic degradation in soil, field dissipation studies, photolysis in soil and confined/field rotational crop studies. In conformance with the JMPR manual 2009 for foliar treatments, only confined and field rotational crops studies were considered for the current evaluation. In addition, The Meeting considered photolysis studies in/on soil relevant for the present evaluation. The fate and behaviour of benzovindiflupyr in the environment was investigated using the [^{14}C]pyrazole and [^{14}C]phenyl labelled compound (see Figure 1).

Photolysis studies in/on soil

The photolysis of [^{14}C]phenyl labelled and [^{14}C]pyrazole labelled benzovindiflupyr was investigated on both dry and moist soil surfaces [McCorquodale and White, 2012, SYN545192_10186]. Soil characteristics are indicated in Table 13. At day 0 moist soil was adjusted to pF2 moisture tension. Dry soil tests used air dried soil. In each case, benzovindiflupyr was applied, at rates equivalent to 0.25 kg ai/ha, to thin layers (ca 2 mm) of loamy soil in individual photolysis vessels. The treated soils were irradiated using light from a xenon arc lamp. The emitted light was filtered to give a spectral distribution close to that of natural sunlight at a mean light intensity of 48–58 W/m². The samples were maintained at 20 ± 2 °C and were continuously irradiated for 30 days, the equivalent of 56–69 days summer sunlight at latitudes 30–50 °N. CO₂ and volatile organics were trapped with 2 mol (M) NaOH. In each test, duplicate irradiated samples were taken for analysis at 0, 5, 10, 15, 20, 25 and 30 days after treatment (DAT) and traps for volatiles were replaced with fresh solution. Dry soils received no moisture maintenance. Moist soils were checked daily and moisture adjusted by addition of water if required to maintain soil close to the target weight at pF2 moisture tension. Dark control samples were also prepared and maintained at 20 °C and analysed at 0 and 30 DAT.

Extraction procedures were started on the day of sampling. Each soil sample was extracted with 0.01 M CaCl₂ and twice with acetone. Extracts, traps and remaining post-extracted soil residue were analysed by (combustion) LSC. Extracts were analysed by HPLC. Selected extracts were analysed by 1D-TLC to confirm both the co-chromatography with reference standards and the quantification of the main components. HPLC-MS-MS was also used to provide qualitative confirmation of the identification of parent, SYN546039, SYN508272 and NOA449410.

A mass balance for each sample was determined by summation of the radioactivity recovered in the soil extracts, the total ¹⁴CO₂ evolved and the post-extracted soil residues. The mean mass balance from the irradiated soils was 99% and from the dark controls was 99%. Benzovindiflupyr degraded slowly under irradiated conditions with no significant degradation in the dark controls.

Under dry, irradiated conditions, the amount of parent declined over the irradiation period to levels of 88.4% and 74.4% TAR by 30 DAT in soil treated with the [^{14}C]phenyl and [^{14}C]pyrazole labelled parent compound, respectively. In moist, irradiated soil, the amount of parent declined over the course of the irradiation period to levels of 89% and 88% TAR by 30 DAT in the phenyl and pyrazole labelled experiments, respectively. The DT₅₀ values were calculated using non-linear regression and single first-order kinetics (SFO) and are 144 days in dry soil and 244 days in moist soil. Corrected for latitude, the DT₅₀ is 298 and 501 for dry and moist soil, respectively, at 30–50 °N (OECD).

In general, a similar degradation pattern was observed in both dry and moist irradiated soil. Photodegradation of benzovindiflupyr resulted in the cleavage of the carboxamide link to produce the pyrazole amide, SYN508272, and the pyrazole acid, NOA449410. Levels of SYN508272 and NOA449410 were higher in dry soil, with amounts reaching maximum mean values of 6.5% and 2.8% TAR, respectively. In moist soil, the maximum mean levels of SYN508272 and NOA449410 were 1.1% and 2.3% TAR, respectively. The demethylated pyrazole acid, SYN545720, was identified in soil extracts from dry soil only, and reached a maximum level of 0.6% TAR. The hydroxylated degradate, SYN546039, was detected at a mean maximum level of 3.7% TAR, but only in the moist irradiated soil treated with the phenyl label. Minor, unidentified components accounted for a combined mean maximum value of 3.5% TAR across all the samples. Carbon dioxide was a minor

($\leq 2.5\%$ TAR) product of photolysis. Unextracted residues increased during the irradiation but did not exceed $> 9.0\%$ TAR in any of the samples.

Table 13 Physico-chemical characteristics of the test soil

Soil origin	Gartenacker Switzerland
Texture (USDA) ^a	loam
Particle size distribution (%)	
—Sand (2000–50 μm)	45
—Silt (50–2 μm)	43
—Clay ($< 2 \mu\text{m}$)	12
Organic carbon (%)	2.0
Organic matter ^b (%)	3.5
CEC (meq/100 g)	10.8
pH (in Water)	7.3
pH (in 0.01 M CaCl_2)	7.0
Moisture Holding Capacity at 0.33 bar ^c (% w/w)	28.8
Moisture Holding Capacity at pF2 (% w/w); study conditions	39.0
Maximum Water Holding Capacity (% w/w)	ns
Microbial biomass (mg carbon/100 g dry soil)	ns

^a Classification according to United States Department of Agriculture

^b Calculated from organic carbon content $\times 1.724$

^c Equivalent to pF = 2.5

Confined rotational crop studies

A confined rotational crop study was designed to provide information on the uptake and metabolism of benzovindiflupyr in rotational crops [Roohi and Adams, 2012, SYN545192_10246]. Two radiolabelled forms were used in the study: [phenyl- ^{14}C]-benzovindiflupyr and [pyrazole- ^{14}C]-benzovindiflupyr. For each radiolabelled form, a single spray application onto the surface of bare soil was performed employing an EC formulation (A170560) at a spray volume of 300 L/ha. The soil characteristics were: USDA sandy loam, pH (H_2O) 5.8, 3.2% om, CEC = 9.4 meq/100 g. The actual application rates achieved were 0.54 kg ai/ha and 0.53 kg ai/ha for the pyrazole and phenyl experiments respectively. After application, the soil was kept in containers under greenhouse conditions and at intervals of 30, 90 and 300 days after application three representative rotational crops were sown into the treated soil. The crops selected were lettuce (variety Tonova), spring wheat (variety Tybalt) and turnip (variety Tokyo Cross), respectively. For each plant back interval and for each radiolabelled experiment the following crop samples were taken: immature lettuce (BBCH 41–43), mature lettuce (BBCH 49), mature turnip roots and leaves (BBCH 49), wheat forage (BBCH 39), wheat hay (BBCH 39–69, sampled 59 days after planting); wheat straw and grain (BBCH 89). Soil samples (0–20 cm) were taken at the time of each sowing (i.e. DAT = 30, 90, 300). Lettuce samples (0.07–1.1 kg) were harvested by cutting just above the soil surface using a knife and any dead leaves and loose soil were removed. Turnips were harvested by hand and any loose soil was removed. The leaves (1.6–2.9 kg) and roots (0.55–1.1 kg) were separated using a knife. If necessary, large roots were cut into smaller pieces. Wheat forage (0.30–1.3 kg) was harvested by cutting the plants with scissors 1 cm above the soil surface. The forage was cut into pieces of 5 cm length. Wheat hay (0.13–0.73 kg) was harvested as wheat forage and then left to dry for 6–10 days in a glasshouse. Mature wheat was harvested as wheat forage and intact ears of grain were removed from the straw using scissors. Grain (0.34–0.70 kg) and chaff were separated and the chaff was added to the straw samples (0.34–0.55 kg). The samples were stored at -18°C for 6–45 months.

Soil samples were not analysed (confirmed, Syngenta, 2014b). Plant commodities were homogenised and the total radioactive residue (TRR) determined by combustion LSC. In the pyrazole experiment, with the exception of the 30 day mature lettuce and the 90 and 300 day grain, all samples contained radioactive residues $> 0.01 \text{ mg/kg eq}$ (see Tables 14–21). In the phenyl experiment, with the exception of the lettuce and grain, all samples contained radioactive residues $> 0.01 \text{ mg/kg eq}$ (see

Tables 14–21). Maximum levels were 0.030 mg/kg eq in lettuce leaves, 0.031 mg/kg eq in turnip roots, 0.026 mg/kg eq in turnip leaves, 0.11 mg/kg eq in wheat forage, 0.34 mg/kg eq in wheat hay, 0.77 mg/kg eq in wheat straw and 0.014 mg/kg eq in wheat grain.

The samples with radioactive residues > 0.01 mg/kg eq were sequentially extracted with solvents of increasing polarity: acetonitrile (1×), acetonitrile/water (4:1, v/v) (2–3×), acetonitrile/water (3:7, v/v) (1×), water (1×), acetonitrile (1×). For grain this sequence was extended with an additional sequence of acetonitrile/water (3:7, v/v) (1×), water (1×), acetonitrile (1×). Radioactivity in extracts and solids was determined by (combustion) LSC. The overall levels of extractability were high with > 80% TRR extracted in all commodities.

For each commodity, extracts containing significant levels of radioactivity were combined, concentrated and re-suspended in water (pH 2). This aqueous concentrate was then partitioned with hexane/diethyl ether (1:3 v/v) in the ratio 3:1 v/v with the aqueous fraction. The resulting organic phase was analysed by 1D- or 2D-TLC for quantification and identification of the components. Identified components in these organic partition fractions were thereby shown to be present in the free form (unconjugated). When the residue in the aqueous fraction was considered significant, a sub-sample was subjected to hydrolysis by rumen technology (bovine rumen fluid, pH 7, 24 hrs at 39 °C, under nitrogen). The resulting hydrolysates were analysed by 2D-TLC and 1D-TLC methods. Identified components were thereby shown to have originally been present in the conjugated form in the initial sample extracts. Where significant radioactivity remained in the post-extraction solids, selected samples were subjected to additional extraction procedures employing 0.1 M HCl (at 37 °C for 6 hrs) or 0.5 M HCl (at 90 °C for 6 hrs) or sodium dodecyl sulphate (SDS), (at 90 °C for 2 hours) in parallel experiments. Identification was by comparison with authentic reference standards of parent, SYN546206, SYN546039, SYN546040, SYN546041, SYN546042, NOA449410, SYN545720 and SYN508722 using two dissimilar solvent systems. Analytical standards for parent, SYN546039, SYN546042, NOA449410, SYN545720 and SYN508722 were confirmed to be stable during hydrolysis by rumen technology.

Results are shown in Tables 14–21. Overall, residue identification accountability was > 80%. In samples where the TRR was low (< 0.03 mg/kg eq) the level of identification was lower: averaging 70% for lettuce, 45% for turnip leaves. In wheat commodities accountability was lower: identification averaged 66% in wheat forage, 61% in wheat hay and 50% in wheat straw. Wheat grain had very low residues (< 0.01–0.014 mg/kg eq) and most extracts were not amenable to chromatographic examination.

The pattern of metabolites found in the samples from the pyrazole experiment was different from that found in the samples from the phenyl experiment because of the presence of metabolites that retained only the pyrazole ring (i.e. NOA449410, SYN545720 and SYN508722).

Storage stability

Chromatographic profiles of all initial crop extracts were obtained within 190 days of harvest. The original extracts were stored at ≤ −18 °C and were then re-analysed at 155 weeks after harvest (i.e. at the end of the analytical phase of the study). Selected samples were immature lettuce (analysed 94 and 1079 days after harvest), turnip leaves (analysed 42 and 1079 days after harvest), turnip roots (analysed 90 and 1082 days after harvest), turnip leaves (analysed 90 and 1082 days after harvest), turnip roots (analysed 90 and 1082 days after harvest) and wheat straw (analysed 48 and 1016 days after harvest). Comparison of the initial and final radio-component profiles obtained from the selected samples showed that no significant change in the profiles had occurred during the interim period of storage.

Table 14 Residues in immature lettuce grown in soil Treated with [¹⁴C]benzovindiflupyr

immature lettuce	30 day PBI		90 day PBI		300 day PBI	
harvest, DAT	DAT 74	DAT 75	DAT 148	DAT 149	DAT 350	DAT 351
harvest, DAS	DAS 44	DAS 45	DAS 58	DAS 59	DAS 50	DAS 51
harvest, BBCH	41–43	41–43	41–43	41–43	41–43	41–43
	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C
TRR (sum) mg/kg eq ^a	N/A	0.012	N/A	0.023	N/A	0.019

immature lettuce	30 day PBI		90 day PBI		300 day PBI	
harvest, DAT	DAT 74	DAT 75	DAT 148	DAT 149	DAT 350	DAT 351
harvest, DAS	DAS 44	DAS 45	DAS 58	DAS 59	DAS 50	DAS 51
harvest, BBCH	41–43	41–43	41–43	41–43	41–43	41–43
	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C
TRR (direct) mg/kg eq ^b	0.005	0.013	0.009	0.023	0.003	0.018
Component	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR
Parent	N/A	38 (N/A)	N/A	13 (N/D)	N/A	6.5 (N/D)
SYN546206 (N-demethyl-BVFP)	N/A	4.9 (N/A)	N/A	0.5 (N/D)	N/A	N/D (N/D)
SYN546039 (BVFP-OH)	N/A	4.3 (N/A)	N/A	2.6 (2.1)	N/A	3.6 (N/D)
SYN546040 (BVFP-OH)	N/A	N/D (N/A)	N/A	5.9 (N/D)	N/A	N/D (N/D)
SYN546041 (N-demethyl- BVFP-OH)	N/A	N/D (N/A)	N/A	N/D (N/D)	N/A	N/D (N/D)
SYN546042 (N-demethyl- BVFP-OH)	N/A	N/D (N/A)	N/A	N/D (N/D)	N/A	N/D (N/D)
SYN508272	N/A	N/D (N/A)	N/A	N/D (N/D)	N/A	N/D (N/D)
NOA449410	N/A	11 (N/A)	N/A	15 (10)	N/A	39 (32)
SYN545720	N/A	N/D (N/A)	N/A	38 (16)	N/A	34 (N/D)
Unassigned ^c	N/A	0.7	N/A	4.2	N/A	1.7
Baseline ^d	N/A	0.9	N/A	14	N/A	13
Remainder ^e	N/A	1.7	N/A	0.2	N/A	3.5
Other Fractions ^f	N/A	23	N/A	N/A	N/A	2.8
PES ^g	N/A	5.0	N/A	9.8	N/A	5.7
Total	N/A	89	N/A	103	N/A	109

The figures in Tables 14–21 are the total radioactive residue for each metabolite reported expressed as mg/kg parent equivalents. The main figures are the sum of both the free and conjugated forms. The figures in parentheses indicate the portion of the radioactive residue in the conjugated form(s).

N/A = Not applicable (Sample not analysed or metabolite not possible in phenyl label).

N/D = Not detected

DAT = Days after soil treatment

DAS = Days after sowing of the rotational crops

^a TRR determined by summation of radioactivity present in the extracts and solids following solvent extraction and set as 100% TRR.

^b TRR determined by direct quantification employing combustion/LSC.

^c Unassigned radio-components which chromatographed away from the origin using 2D-TLC SSD, comprising 1–41 discrete components (depending on matrix), none greater than 5.0% TRR (each < 0.01 mg/kg eq)

^d Polar material remaining on the origin on the TLC plate using 2D-TLC SSD

^e The remainder consists of areas of the chromatogram which cannot be assigned to discrete radioactive components.

^f Extracts or aqueous partition fractions that were considered too low for analysis: < 5% TRR or < 0.01 mg/kg eq

^g Radioactivity remaining in the solids after the initial solvent extractions.

Table 15 Residues in mature lettuce grown in soil treated with [¹⁴C]benzovindiflupyr

Mature lettuce	30 day PBI		90 day PBI		300 day PBI	
Harvest, DAT	DAT 88	DAT 89	DAT 158	DAT 159	DAT 362	DAT 363
Harvest, DAS	DAS 58	DAS 59	DAS 68	DAS 69	DAS 62	DAS 63
Harvest, BBCH	49	49	49	49	49	49
	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C
TRR (sum) mg/kg ^a	N/A	N/A	N/A	0.030	N/A	0.020
TRR (direct) mg/kg ^b	0.004	0.008	0.006	0.027	0.005	0.018
Component	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR
Parent	N/A	N/A	N/A	24 (N/D)	N/A	13 (N/D)
SYN546206 (N-demethyl-BVFP)	N/A	N/A	N/A	0.2 (N/D)	N/A	0.2 (N/D)

Mature lettuce	30 day PBI		90 day PBI		300 day PBI	
Harvest, DAT	DAT 88	DAT 89	DAT 158	DAT 159	DAT 362	DAT 363
Harvest, DAS	DAS 58	DAS 59	DAS 68	DAS 69	DAS 62	DAS 63
Harvest, BBCH	49	49	49	49	49	49
	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C
SYN546039 (BVFP-OH)	N/A	N/A	N/A	1.9 (1.5)	N/A	0.5 (N/D)
SYN546040 (BVFP-OH)	N/A	N/A	N/A	8.0 (2.9)	N/A	6.8 (N/D)
SYN546041 (N-demethyl-BVFP-OH)	N/A	N/A	N/A	N/D (N/D)	N/A	N/D (N/D)
SYN546042 (N-demethyl-BVFP-OH)	N/A	N/A	N/A	N/D (N/D)	N/A	N/D (N/D)
SYN508272	N/A	N/A	N/A	N/D (N/D)	N/A	N/D (N/D)
NOA449410	N/A	N/A	N/A	9.5 (7.2)	N/A	21 (19)
SYN545720	N/A	N/A	N/A	24 (10.9)	N/A	25 (N/D)
Unassigned ^c	N/A	N/A	N/A	7.4	N/A	10.6
Baseline ^d	N/A	N/A	N/A	9.7	N/A	14.4
Remainder ^e	N/A	N/A	N/A	ND	N/A	ND
Other fractions ^f	N/A	N/A	N/A	N/A	N/A	0.5
PES ^g	N/A	N/A	N/A	12	N/A	9.3
Total	N/A	N/A	N/A	97	N/A	101

^a TRR determined by summation of radioactivity present in the extracts and solids following solvent extraction and set as 100% TRR.

^b TRR determined by direct quantification employing combustion/LSC.

^c Unassigned radio-components which chromatographed away from the origin using 2D-TLC SSD, comprising 1–41 discrete components (depending on matrix), none greater than 5.0% TRR (each < 0.01 mg/kg eq)

^d Polar material remaining on the origin on the TLC plate using 2D-TLC SSD

^e The remainder consists of areas of the chromatogram which cannot be assigned to discrete radioactive components.

^f Extracts or aqueous partition fractions that were considered too low for analysis: < 5% TRR or < 0.01 mg/kg eq

^g Radioactivity remaining in the solids after the initial solvent extractions.

Table 16 Residues in turnip leaves grown in soil treated with [¹⁴C]benzovindiflupyr

Turnip leaves	30 day PBI		90 day PBI		300 day PBI	
harvest, DAT	DAT 74	DAT 75	DAT 140	DAT 141	DAT 351	DAT 352
Harvest, DAS	DAS 44	DAS 45	DAS 50	DAS 51	DAS 51	DAS 52
Harvest, BBCH	49	49	49	49	49	49
	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C
TRR (sum) mg/kg ^a	0.010	0.021	0.014	0.027	0.008	0.022
TRR (direct) mg/kg ^b	0.012	0.020	0.015	0.031	0.012	0.024
Component	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR
Parent	37 (N/A)	26 (N/A)	17 (N/A)	26 (N/A)	22 (N/D)	10 (N/D)
SYN546206 (N-demethyl-BVFP)	2.8 (N/A)	2.4 (N/A)	1.1 (N/A)	2.1 (N/A)	9.2 (6.7)	1.2 (N/D)
SYN546039 (BVFP-OH)	2.0 (N/A)	2.4 (N/A)	1.3 (N/A)	1.5 (N/A)	3.4 (N/D)	1.0 (N/D)
SYN546040 (BVFP-OH)	2.5 (N/A)	3.0 (N/A)	2.5 (N/A)	1.9 (N/A)	2.7 (N/D)	0.9 (N/D)
SYN546041 (N-demethyl-BVFP-OH)	0.7 (N/A)	1.2 (N/A)	0.3 (N/A)	0.6 (N/A)	1.0 (N/D)	0.3 (N/D)

Turnip leaves	30 day PBI		90 day PBI		300 day PBI	
SYN546042 (N-demethyl-BVFP-OH)	1.9 (N/A)	N/D (N/A)	1.2 (N/A)	1.5 (N/A)	1.9 (N/D)	0.7 (N/D)
SYN508272	N/A	1.4 (N/A)	N/A	4.2 (N/A)	N/A	8.5 (7.4)
NOA449410	N/A	7.3 (N/A)	N/A	3.9 (N/A)	N/A	14 (8.1)
SYN545720	N/A	12 (N/A)	N/A	12 (N/A)	N/A	15 (N/D)
Unassigned ^c	2.1	0.5	1.7	4.1	1.6	14
Baseline ^d	1.4	1.2	0.9	1.2	12	11
Remainder ^e	ND	ND	0.9	1.5	19	3.5
Other fractions ^f	30	40	34	28	38	N/A
PES ^g	4.9	2.8	7.9	4.9	5.0	3.0
Total	85	99	67	93	116	82

^a TRR determined by summation of radioactivity present in the extracts and solids following solvent extraction and set as 100% TRR.

^b TRR determined by direct quantification employing combustion/LSC.

^c Unassigned radio-components which chromatographed away from the origin using 2D-TLC SSD, comprising 1–41 discrete components (depending on matrix), none greater than 5.0% TRR (each < 0.01 mg/kg eq)

^d Polar material remaining on the origin on the TLC plate using 2D-TLC SSD

^e The remainder consists of areas of the chromatogram which cannot be assigned to discrete radioactive components.

^f Extracts or aqueous partition fractions that were considered too low for analysis: < 5% TRR or < 0.01 mg/kg eq

^g Radioactivity remaining in the solids after the initial solvent extractions.

Table 17 Residues in turnip roots grown in soil treated with [¹⁴C]benzovindiflupyr

Turnip roots	30 day PBI		90 day PBI		300 day PBI	
harvest, DAT	DAT 74	DAT 75	DAT 140	DAT 141	DAT 351	DAT 352
harvest, DAS	DAS 44	DAS 45	DAS 50	DAS 51	DAS 51	DAS 52
harvest, BBCH	49	49	49	49	49	49
	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C
TRR (sum) mg/kg ^a	0.019	0.028	0.026	0.031	0.011	0.014
TRR (direct) mg/kg ^b	0.020	0.027	0.022	0.028	0.011	0.013
Component	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR
Parent	90 (N/A)	81 (N/A)	69 (N/A)	72 (N/A)	71 (N/A)	64 (N/A)
SYN546206 (N-demethyl-BVFP)	5.0 (N/A)	5.8 N/A	5.1 (N/A)	5.6 (N/A)	5.8 (N/A)	8.0 (N/A)
SYN546039 (BVFP-OH)	(N/D) (N/A)	1.8 (N/A)	1.3 (N/A)	1.6 (N/A)	1.8 (N/A)	1.4 (N/A)
SYN546040 (BVFP-OH)	1.4 (N/A)	1.5 (N/A)	2.1 (N/A)	1.1 (N/A)	1.2 (N/A)	1.0 (N/A)
SYN546041 (N-demethyl-BVFP-OH)	(N/D) (N/A)	0.4 (N/A)	0.8 (N/A)	0.3 (N/A)	0.4 (N/A)	0.4 (N/A)
SYN546042 (N-demethyl-BVFP-OH)	(N/D) (N/A)	0.5 (N/A)	(N/D) (N/A)	0.5 (N/A)	0.2 (N/A)	0.6 (N/A)
SYN508272	N/A	N/D (N/A)	N/A	N/D (N/A)	N/A	0.1 (N/A)
NOA449410	N/A	3.5 (N/A)	N/A	1.9 (N/A)	N/A	6.1 (N/A)
SYN545720	N/A	0.4 (N/A)	N/A	0.4 (N/A)	N/A	0.8 (N/A)
Unassigned ^c	1.2	< 0.1	1.2	< 0.1	< 0.1	0.4
Baseline ^d	2.5	0.9	1.8	1.5	1.4	1.2
Remainder ^e	ND	ND	0.1	ND	ND	ND
Other fractions ^f	1.0	4.7	4.8	5.3	3.5	6.4
PES ^g	2.2	1.5	2.5	3.1	3.8	2.7
Total	103	102	89	93	89	93

^a TRR determined by summation of radioactivity present in the extracts and solids following solvent extraction and set as

100% TRR.

^b TRR determined by direct quantification employing combustion/LSC.

^c Unassigned radio-components which chromatographed away from the origin using 2D-TLC SSD, comprising 1–41 discrete components (depending on matrix), none greater than 5.0% TRR (each < 0.01 mg/kg eq)

^d Polar material remaining on the origin on the TLC plate using 2D-TLC SSD

^e The remainder consists of areas of the chromatogram which cannot be assigned to discrete radioactive components.

^f Extracts or aqueous partition fractions that were considered too low for analysis: < 5% TRR or < 0.01 mg/kg eq

^g Radioactivity remaining in the solids after the initial solvent extractions.

Table 18 Residues in wheat forage grown in soil treated with [¹⁴C]benzovindiflupyr

wheat forage	30 day PBI		90 day PBI		300 day PBI	
harvest, DAT	DAT 74	DAT 75	DAT 138	DAT 139	DAT 348	DAT 349
harvest, DAS	DAS 44	DAS 45	DAS 48	DAS 49	DAS 48	DAS 49
harvest, BBCH	39	39	39	39	39	39
	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C
TRR (sum) mg/kg ^a	0.044	0.099	0.067	0.11	0.064	0.090
TRR (direct) mg/kg ^b	0.040	0.098	0.067	0.097	0.051	0.090
Component	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR
Parent	31 (N/D)	26 (N/D)	35 (N/D)	19 (N/D)	29 (N/D)	17 (N/D)
SYN546206 (N-demethyl-BVFP)	13 (N/D)	10 (N/D)	13 (N/D)	11 (2.5)	15 (N/D)	8.3 (N/D)
SYN546039 (BVFP-OH)	19 (15.8)	7.4 (5.1)	8.0 (4.7)	8.7 (6.9)	20 (16)	10 (8.5)
SYN546040 (BVFP-OH)	0.8 (N/D)	2.1 (1.4)	2.3 (N/D)	1.0 (N/D)	3.1 (N/D)	1.0 (N/D)
SYN546041 (N-demethyl-BVFP-OH)	6.1 (5.7)	1.2 (0.8)	0.6 (N/D)	1.5 (1.5)	2.1 (N/D)	N/D (N/D)
SYN546042 (N-demethyl-BVFP-OH)	N/D (N/D)	0.4 (N/D)	1.0 (N/D)	2.3 (1.8)	1.7 (1.0)	N/D (N/D)
SYN508272	N/A	5.4 (4.6)	N/A	2.4 (2.0)	N/A	5.7 (3.9)
NOA449410	N/A	6.1 (N/D)	N/A	12 (9.9)	N/A	14 (9.5)
SYN545720	N/A	1.3 (N/D)	N/A	12 (10)	N/A	12 (10)
Unassigned ^c	2.3	17	6.2	3.6	9.2	4.8
Baseline ^d	8.7	3.4	25	19	9.0	27
Remainder ^e	7.3	ND	0.7	2.5	0.3	ND
Other fractions ^f	4.4	1.3	N/A	4.2	N/A	N/A
PES ^g	4.4	5.9	6.8	5.3	5.5	5.5
Total	96	88	99	104	95	106

^a TRR determined by summation of radioactivity present in the extracts and solids following solvent extraction and set as 100% TRR.

^b TRR determined by direct quantification employing combustion/LSC.

^c Unassigned radio-components which chromatographed away from the origin using 2D-TLC SSD, comprising 1–41 discrete components (depending on matrix), none greater than 5.0% TRR (each < 0.01 mg/kg eq)

^d Polar material remaining on the origin on the TLC plate using 2D-TLC SSD

^e The remainder consists of areas of the chromatogram which cannot be assigned to discrete radioactive components.

^f Extracts or aqueous partition fractions that were considered too low for analysis: < 5% TRR or < 0.01 mg/kg eq

^g Radioactivity remaining in the solids after the initial solvent extractions.

Table 19 Residues in wheat hay grown in soil treated with [¹⁴C]benzovindiflupyr

wheat hay	30 day PBI		90 day PBI		300 day PBI	
Harvest	DAT 89	DAT 89	DAT 149	DAT 149	DAT 359	DAT 359
Harvest, DAS	DAS 59	DAS 59	DAS 59	DAS 59	DAS 59	DAS 59
Harvest, BBCH	41-69	39-65	41-69	39-65	41-69	39-65
	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C
TRR (sum) mg/kg ^a	0.21	0.34	0.26	0.34	0.20	0.29

wheat hay	30 day PBI		90 day PBI		300 day PBI	
Harvest	DAT 89	DAT 89	DAT 149	DAT 149	DAT 359	DAT 359
Harvest, DAS	DAS 59	DAS 59	DAS 59	DAS 59	DAS 59	DAS 59
Harvest, BBCH	41-69	39-65	41-69	39-65	41-69	39-65
	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C
TRR (direct) mg/kg ^b	0.18	0.35	0.22	0.33	0.20	0.29
Component	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR
Parent	33 (N/D)	24 (N/D)	32 (N/D)	25 (N/D)	23 (N/D)	15 (N/D)
SYN546206 (N-demethyl-BVFP)	18 (N/D)	9.3 (N/D)	15 (N/D)	14 (2.6)	12 (N/D)	7.6 (N/D)
SYN546039 (BVFP-OH)	14 (11)	7.0 (5.1)	9.8 (7.3)	1.9 (N/D)	16 (12)	9.4 (7.0)
SYN546040 (BVFP-OH)	0.8 (N/D)	1.2 (0.7)	1.2 (N/D)	0.9 (N/D)	3.8 (1.8)	1.0 (N/D)
SYN546041 (N-demethyl- BVFP-OH)	4.6 (3.7)	1.6 (1.3)	0.8 (N/D)	0.5 (N/D)	2.2 (1.4)	0.6 (N/D)
SYN546042 (N-demethyl- BVFP-OH)	0.5 (N/D)	0.8 (0.5)	0.8 (N/D)	0.4 (N/D)	2.1 (1.3)	0.6 (N/D)
SYN508272	N/A	3.9 (3.3)	N/A	2.8 (2.3)	N/A	3.4 (2.9)
NOA449410	N/A	4.3 (3.1)	N/A	5.6 (4.9)	N/A	8.8 (6.7)
SYN545720	N/A	2.0 (1.6)	N/A	5.8 (5.4)	N/A	11 (9.8)
Unassigned ^c	4.7	14	3.8	12	7.7	14
Baseline ^d	8.6	13	18	30	7.1	14
Remainder ^e	ND	ND	ND	ND	1.5	1.2
Other fractions ^f	1.2	4.2	4.4	1.1	3.2	0.7
PES ^g	6.9	0.2	9.0	10	11	13
Total	92	86	95	111	90	100

^a TRR determined by summation of radioactivity present in the extracts and solids following solvent extraction and set as 100% TRR.

^b TRR determined by direct quantification employing combustion/LSC.

^c Unassigned radio-components which chromatographed away from the origin using 2D-TLC SSD, comprising 1–41 discrete components (depending on matrix), none greater than 5.0% TRR (each < 0.01 mg/kg eq)

^d Polar material remaining on the origin on the TLC plate using 2D-TLC SSD

^e The remainder consists of areas of the chromatogram which cannot be assigned to discrete radioactive components.

^f Extracts or aqueous partition fractions that were considered too low for analysis: < 5% TRR or < 0.01 mg/kg eq

^g Radioactivity remaining in the solids after the initial solvent extractions.

Table 20 Residues in wheat straw grown in soil treated with [¹⁴C]benzovindiflupyr

wheat straw	30 day PBI		90 day PBI		300 day PBI	
Harvest	DAT 140	DAT 141	DAT 200	DAT 201	DAT 420	DAT 421
Harvest, DAS	DAS 110	DAS 111	DAS 110	DAS 111	DAS 120	DAS 121
Harvest, BBCH	89	89	89	89	89	89
	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C
TRR (sum) mg/kg ^a	0.71	0.77	0.34	0.33	0.20	0.23
TRR (direct) mg/kg ^b	0.71	0.77	0.26	0.31	0.23	0.22
Component	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR
Parent	15 (N/D)	14 (N/D)	16 (N/D)	18 (N/D)	14 (N/D)	12 (N/D)
SYN546206 (N-demethyl-BVFP)	18 (N/D)	13 (1.4)	14 (N/D)	15.1 (1.3)	14 (1.1)	13 (N/D)
SYN546039 (BVFP-OH)	7.1 (4.2)	9.0 (6.2)	5.2 (2.2)	5.8 (3.9)	9.6 (7.4)	9.6 (8.0)
SYN546040 (BVFP-OH)	2.0 (1.2)	2.7 (1.8)	1.5 (N/D)	1.4 (0.8)	3.7 (1.7)	2.5 (1.6)
SYN546041 (N-demethyl- BVFP-OH)	3.5 (2.5)	3.9 (3.1)	1.0 (N/D)	2.1 (1.5)	2.2 (1.4)	1.9 (1.2)

wheat straw	30 day PBI		90 day PBI		300 day PBI	
Harvest	DAT 140	DAT 141	DAT 200	DAT 201	DAT 420	DAT 421
Harvest, DAS	DAS 110	DAS 111	DAS 110	DAS 111	DAS 120	DAS 121
Harvest, BBCH	89	89	89	89	89	89
	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C
BVFP-OH)						
SYN546042 (N-demethyl- BVFP-OH)	1.7 (1.00)	2.0 (1.5)	0.9 (N/D)	2.6 (2.0)	2.1 (1.4)	1.3 (0.8)
SYN508272	N/A	1.1 (0.8)	N/A	2.1 (1.9)	N/A	1.3 (1.0)
NOA449410	N/A	4.5 (3.6)	N/A	2.5 (2.2)	N/A	1.7 (1.0)
SYN545720	N/A	2.3 (2.0)	N/A	0.3 (N/D)	N/A	1.4 (1.1)
Unassigned ^c	12	20	15	9.3	17	14
Baseline ^d	15	17	16	12	12	13
Remainder ^e	5.5	1.9	2.3	0.9	ND	0.2
Other fractions ^f	1.5	1.3	3.0	1.6	4.1	5.3
PES ^g	9.5	9.5	12	12	13	19
Total	90	102	87	85	92	96

^a TRR determined by summation of radioactivity present in the extracts and solids following solvent extraction and set as 100% TRR.

^b TRR determined by direct quantification employing combustion/LSC.

^c Unassigned radio-components which chromatographed away from the origin using 2D-TLC SSD, comprising 1–41 discrete components (depending on matrix), none greater than 5.0% TRR (each < 0.01 mg/kg eq)

^d Polar material remaining on the origin on the TLC plate using 2D-TLC SSD

^e The remainder consists of areas of the chromatogram which cannot be assigned to discrete radioactive components.

^f Extracts or aqueous partition fractions that were considered too low for analysis: < 5% TRR or < 0.01 mg/kg eq

^g Radioactivity remaining in the solids after the initial solvent extractions.

Table 21 Residues in wheat grain grown in soil treated with [¹⁴C]benzovindiflupyr

	30 day PBI		90 day PBI		300 day PBI	
Harvest	DAT 140	DAT 141	DAT 200	DAT 201	DAT 420	DAT 421
Harvest, DAS	DAS 110	DAS 111	DAS 110	DAS 111	DAS 120	DAS 121
Harvest, BBCH	89	89	89	89	89	89
	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C	phenyl- ¹⁴ C	pyrazole- ¹⁴ C
TRR (sum) mg/kg ^a	N/A	0.014	N/A	N/A	N/A	N/A
TRR (direct) mg/kg ^b	0.003	0.013	0.002	0.004	0.007	0.007
Component	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR
Extracts ^c	N/A	80	N/A	N/A	N/A	N/A
PES ^d	N/A	20	N/A	N/A	N/A	N/A
Total	N/A	100	N/A	N/A	N/A	N/A

^a TRR determined by summation of radioactivity present in the extracts and solids following solvent extraction and set as 100% TRR.

^b TRR determined by direct quantification employing combustion/LSC.

^c Extracts or aqueous partition fractions that were considered too low for analysis: < 5% TRR or < 0.01 mg/kg eq

^d Radioactivity remaining in the solids after the initial solvent extractions.

Field rotational crop studies

Four rotational crop field trials have been conducted in the EU to investigate the magnitude of residues in rotational crops following a single application to bare soil at 0.200 kg ai/ha. Additionally, four rotational crop field trials have been conducted in the USA to investigate the magnitude of residues in rotational crops following three foliar applications at 14 day intervals and at a rate equivalent to 0.100 kg ai/ha per application to target crops of peanuts and soya beans.

Study 1

Two field crop rotation trials were carried out in Northern Europe during 2010 and 2011 [Ellis, 2012b, A17056B_10004]. Plots (72–75 m² each) were treated with a single application to bare soil at a nominal rate of 0.20 kg ai/ha using an EC formulation (A17056B). Plots received minimal cultivation with a harrow or rototill to 5 cm depth 8–13 days prior to drilling of the rotational crops. Rotational crops were planted into the treated soil at 28–30, 60–61 or 355–365 days after application. The 355–365 day rotation crops were planted on the same plot as the 28–30 day plots, after the first rotation crops were harvested and removed. Experimental conditions for trial S10-00261-01 and S10-00261-02 are indicated in Table 22. Crops sampled at immature stages were spinach (leaves) at BBCH 42–43 (DAT 81/71, 91/99, 420/420) and spring wheat (whole plant) at BBCH 39 (DAT 88/90, 98/107, 429/436) for trial 01/02. Mature samples were taken for spinach at BBCH 49 (DAT 91/86, 100/111, 434/432), for spring wheat (grain and straw) at BBCH 89 (DAT 144/155, 161/170, 505/525) and for carrot (root and tops with leaves) at BBCH 49 (DAT 149/131, 156/170, 491/467) for trial 01/02. Samples were taken randomly across each plot from at least 12 points. Spinach leaves were cut above ground using scissors, a knife or a sickle. Wheat forage whole plants were taken by hand or with scissors/secateurs or a sickle. Wheat grain and straw samples were produced by threshing the whole plant samples using a plot harvester. Carrot plants were taken by hand and the soil washed off. Sample sizes were in accordance with appendix V of the FAO manual: 1.0–2.0 kg spinach leaves from at least 12 plants, 1.0–1.4 kg wheat forage from at least 12 plants, 1.0–1.4 kg wheat grain and 0.50–0.73 kg wheat straw from at least 50 plants, 2.0–2.3 kg carrot roots and 1.0–1.2 kg carrot tops from at least 12 plants. Samples were stored frozen within 1–6 hrs after harvest at –18 °C for a maximum period of 10 months until analysis. The storage period of 10 months is covered by the storage stability studies on parent, SYN546039 and SYN546206 in commodities with high water content, high starch content and wheat straw. During shipment, temperatures were out of range for short periods of less than 12 hrs. Since the samples remained frozen, this is considered to have no impact on the study.

Soil samples have not been analysed for parent or its soil metabolites (confirmed, Syngenta, 2014b). Wheat forage, grain and straw from the 30 and 60 day plant back intervals were analysed for parent and SYN546039 using HPLC-MS-MS method GRM042.03A with matrix matched standards for grain and straw and minor modifications for parent. Parent compound was analysed directly in the initial acetonitrile/water extract. This modification (A) was shown to have no impact on the validity of the analysis. All other crop samples were analysed for parent, SYN546039 and SYN546206 using HPLC-MS-MS method GRM042.08A with matrix matched standards. Methods GRM042.03A and GRM042.08A are considered valid for the purpose of this study (commodity type and concentration level of the analytes). Average concurrent recoveries were within 70–120% for each analyte and matrix and control samples had residues < 0.01 mg/kg, showing adequate performance at the time of analysis of the samples.

No residues of parent, SYN546039 or SYN546206 were found at or above the LOQ of 0.01 mg/kg in any of the treated samples.

Study 2

Two field crop rotation trials were carried out in Southern Europe during 2010 and 2011 [Ellis, 2012c, A17056B_10005]. Plots (24–90 m²) were treated with a single application to bare soil at a nominal rate of 0.20 kg ai/ha using an EC formulation (A17056B). Plots were lightly cultivated (plot 01) or cultivated with a motor hoe to a depth of 20 cm (plot 02) 1–8 days prior to drilling rotational crops. Rotational crops were planted into the treated soil at 28–32, 60–69 or 357–366 days after application. The 357–366 day rotation crops were planted on the same plot as the 28–32 day plots, after the soil was cultivated to a depth of 20 cm, including the remains of the first rotation crops. Experimental conditions for trials S10-00262-01 and S10-00262-02 are indicated in Table 22. Crops sampled at immature stages were spinach (leaves) at BBCH 41–43 (DAT 79/66, 108/97, 416/416) and spring wheat (whole plant) at BBCH 39 (DAT 69/75, 111/97, 416/423) for trial 01/02. Mature samples were taken for spinach at BBCH 49 (DAT 86/75, 118/104, 429/423), for spring wheat (grain and straw) at BBCH 89 (DAT 118/125, 192/152, 471/480) and for carrot (root and tops with leaves) at BBCH 49

(DAT 154/152, 169/159, 483/483) for trial 01/02. Spinach leaves were cut above ground using a knife. Wheat forage whole plants were cut above ground using a knife, scissors or secateurs. Wheat grain and straw samples were threshed by using a plot or portable combine. Carrot plants were collected by hand and roots and tops were separated in the field using a knife or a spade. Sample sizes were generally in accordance with appendix V of the FAO manual: 1.0–2.6 kg spinach leaves from at least 12 plants, 1.1–3.5 kg wheat forage from at least 12 plants, 1.0–1.5 kg wheat grain and 0.57–1.7 kg wheat straw from at least 12 plants, 2.0–4.0 kg carrot roots and 1.0–2.0 kg carrot tops from at least 12 plants. Sample size for mature spinach leaves from the 366 day plant back interval in trial 01 is rather small (0.30 kg), but since this sample was retrieved from at least 50 plants this is considered acceptable. Samples were stored frozen within 1–5 hrs after harvest at –18 °C for a maximum period of 10 months until analysis. The storage period of 10 months is covered by the storage stability studies on parent, SYN546039 and SYN546206 in commodities with high water content, high starch content and wheat straw. During shipment, temperatures were out of range for short periods of less than 6 hrs. Since the samples remained frozen, this is considered to have no impact on the study.

Soil samples have not been analysed for parent or its soil metabolites (confirmed, Syngenta, 2014b). Wheat forage, grain and straw from the 30 and 60 day plant back intervals were analysed for parent and SYN546039 by HPLC-MS-MS method GRM042.03A with matrix matched standards and minor modifications for parent. Parent compound was analysed directly in the initial acetonitrile/water extract. This modification (A) was shown to have no impact on the validity of the analysis. All other crop samples were analysed for parent, SYN546039 and SYN546206 using HPLC-MS-MS method GRM042.08A with matrix matched standards. Methods GRM042.03A and GRM042.08A are considered valid for the purpose of this study (commodity type and concentration level of the analytes). Average concurrent recoveries were within 70–120% for each analyte and matrix and control samples had residues < 0.01 mg/kg, showing adequate performance at the time of analysis of the samples.

No residues of parent, SYN546039 or SYN546206 were detected at or above the LOQ of 0.01 mg/kg in any of the treated samples, except in treated wheat straw:

- In trial S10-00262-02 for wheat planted 28 days after application, parent was found at 0.014 mg/kg in wheat straw (DAT 125)
- In trial S10-00262-02 for wheat planted 60 days after application, parent was found at 0.012 mg/kg in wheat straw (DAT 152).

Study 3

Four field crop rotation trials were carried out in the USA during 2009, 2010 and 2011 [Mäyer, 2012, A17056B_50013]. Plots with target crops of peanuts or soya beans were treated with three foliar applications at 14 day intervals at a nominal rate of 0.100 kg ai/ha using an EC formulation. All applications incorporated a crop-oil concentrate or non-ionic surfactant adjuvant in the spray. The peanut and soya bean target crops were harvested and removed from the field according to agronomical practices. Experimental conditions are indicated Table 22. In trial E10-9681 (Seven Springs, NC), spinach, radish, and wheat were planted into the treated soil at 30 days after the last application. Wheat grain and straw were harvested at DAT 228, wheat hay at DAT 182, wheat forage at DAT 91, roots/tops at DAT 157 and leafy crops at DAT 158. In trials E11-9678 (Elko, SC), C18-9679 (Richland, IA) and E13-9680 (Madil, TX), lettuce or spinach, radish or turnip and wheat were planted into the treated soil at 180 days after the last application. For trials E11-9678, C18-9679, E13-9680 wheat grain and straw were harvested at DALT 289/275/278, wheat forage and hay at DAT 231/233/231, roots/tops at DAT 231/231/278 and leafy vegetables at DAT 231/231/300. Wheat forage and wheat hay were collected from 12 separate areas of the plot. Wheat grain, wheat straw, radish top, radish root and leafy vegetable samples were collected from at least 12 plants and/or 12 separate areas of the plot at normal crop maturity. Samples were at least 2 kg (roots) or at least 1 kg (tuber tops, leafy vegetables, wheat forage and wheat grains) or at least 0.5 kg (wheat straw and wheat hay). Exceptions are 0.6–1.0 kg for tuber tops and 0.26–0.28 kg for wheat forage in trial E10-9681 (30 day PBI), 1.1–1.5 kg for 40 tubers in trial E11-9678 (180 day PBI), 0.8–0.9 kg for tuber tops in trial (E13-

9680) [Syngenta, 2014b]. In trial E10-9681, plant growth was retarded because of abnormally cold weather and therefore it was not possible to collect at least 1 kg wheat forage from the plot. Two replicate field samples were taken from each plot. Adhering soil was removed from radish roots by light brushing. Wheat hay was harvested on the same date as wheat forage and was dried for 2 days in a shelter (E10-9681), 2 days in the field (E11-9678), 6 days in the field (E13-9680) or 10 days in a shed (C18-9679) [Syngenta, 2014b].

Samples were stored at -20°C for a maximum period of 24 months until analysis [Syngenta, 2014b]. The storage period of 24 months is covered by the storage stability studies on parent, SYN546039 and SYN546206 in commodities with high water content, high starch content and wheat straw.

Soil samples have not been analysed for parent or its soil metabolites (confirmed, Syngenta, 2014b). Crop samples from the 180 day plant-back interval plots were analysed for parent and SYN546039 by HPLC-MS-MS method GRM042.03A. Parent compound was cleaned-up together with the SYN546039 hydrolysate as in the original method. Method GRM042.03A is considered valid for the purpose of this study (commodity type and concentration level of the analytes). Samples were re-analysed for SYN546206 by modification A of HPLC-MS-MS method GRM042.08A. Crop samples from 30 day plant-back interval plots were analysed for parent, SYN546039 and SYN546206 using modification A of HPLC-MS-MS method GRM042.08A. Modification A of method GRM042.08A is considered insufficiently validated for the determination of parent, SYN546039 and SYN546206 in radish roots and tops (30 day PBI samples) and in addition insufficiently validated for the determination of SYN546206 in lettuce, radish roots & tops and turnip roots & tops (180 day PBI samples) (see analytical section). Average concurrent recoveries for a limited number of samples were within 70–120% for each analyte and matrix and control samples had residues < 0.01 mg/kg, showing adequate performance at the time of analysis of the samples

No residues of parent, SYN546039 or SYN546206 were detected at or above the LOQ of 0.01 mg/kg in any of the treated samples, except in wheat:

- In trial E10-9681 with soya bean as primary crop and wheat planted 30 days after application, parent and SYN546039 were found at an average concentration of 0.022 mg/kg (range 0.012–0.033 mg/kg) and 0.021 mg/kg (range 0.015–0.028 mg/kg), respectively, in wheat forage (DALT 91). Two field samples were taken and each sample was extracted twice.
- In trial E10-9681 with soya bean as primary crop and wheat planted 30 days after application, SYN546039 was found at an average concentration of 0.016 mg/kg (range 0.016–0.017 mg/kg) in wheat straw (DAT 228). Two replicate field samples were taken and each sample was extracted twice.

Note: Since residues were found at the 30 day plant back interval, the 30 day PBI trial is considered the most critical trial (spinach, radish and wheat). The wheat forage samples of the 30 day PBI trial are considered not representative, because wheat growth is retarded because of abnormally cold weather and consequently it was not possible to collect a sufficient (representative) amount of sample from the plot. In addition, modification A of method GRM042.08A is considered insufficiently validated for the determination of parent, SYN546039 and SYN546206 in radish roots and tops (30 day PBI samples).

Table 22 Experimental conditions for field rotational crop studies

Trial & Location	Application; last appl date	Soil type	pH	% om	CEC (meq/100 g)	PBI	Rotational crops
S10-00261-01; Dollern, Germany	1 × 0.20 kg ai/ha to bare soil 29 Mar 2010	sandy loam	6.5	1.4	ns	30, 60, 365 ^b	Spinach (var Tornado) Short-cycle carrots (var Laguna F1) Spring wheat (var Chasin)
S10-00261-02; Banbury,	1 × 0.20 kg ai/ha to bare soil	clay loam	6.7	5.1	ns	28, 61,	Spinach (var Renegade)

Trial & Location	Application; last appl date	Soil type	pH	% om	CEC (meq/100 g)	PBI	Rotational crops
Oxfordshire, UK	17 Mar 2010					355 ^b	Short-cycle carrots (var Napoli) Spring wheat (var Belvoir)
S10-00262-01; Barry d'Islemade, Tarn et Garonne, France	1× 0.20 kg ai/ha to bare soil 20 Apr 2010	sandy clay loam	8.4	8	ns	32, 69, 366 ^b	Spinach (var Lagos F1 or Island) Short-cycle carrots (var Napoli F1 or Mokum F1) Spring wheat (var Printidor)
S10-00262-02; Castagnito, Piedmont, Italy	1× 0.20 kg ai/ha to bare soil 19 March 2010	sandy loam	7.9	1.4	ns	28, 60, 357 ^b	Spinach (var America or Viridis Olter) Short-cycle carrots (var Nantese di Chioggia 2 or Nantes 2) Spring wheat (var Valbona)
E10-9681; Seven Springs, NC, USA	3× 0.10 kg ai/ha to soya beans ^a at BBCH 75–89; 15 Oct 2010	Loamy Sand	6.4	0.7	6.5	30	Spinach (var Baker) Radish (var Cherriette) Wheat (var Coker 9804)
E11-9678; Elko, SC, USA	3× 0.10 kg ai/ha to peanuts at BBCH 73–83 ^c ; 24 Aug 2009	Loamy Sand	6.8	1.1	3.1	180	Lettuce (var Butter Crunch) Radish (var Early Scarlet Globe) Wheat (var Steele)
C18-9679; Richland, IA, USA	3× 0.10 kg ai/ha to soya beans ^a at BBCH 79 ^c ; 29 Sept 2009	Silt loam	6.7	4.5	18	180	Spinach (var Bloomsdale) Radish (var Champion) Spring wheat (var Briggs)
E13-9680; Madill, TX, USA	3× 0.10 kg ai/ha to peanuts at BBCH 71–85 ^c ; 2 Oct 2009	Sandy Loam	7.9	0.8	18	180	Spinach (var Spargo F1) Turnip (Scarlet Queen) Spring wheat (var Glenn)

^a Syngenta, 2014b: soya bean is the target crop

^b The 355–365 day rotation crops were planted on the same plot as the 28–32 day plots, after the first rotation crops were removed

^c Primary crops were harvested and removed before rotational crops were planted

Environmental fate in water/sediment systems

No data submitted.

RESIDUE ANALYSIS

The Meeting received information on enforcement/monitoring methods for the determination of benzovindiflupyr and its metabolites in plant and animal commodities. In addition the Meeting received information on analytical methods for the determination of benzovindiflupyr and its metabolites as used in the various study reports (supervised residue trials, storage stability studies, processing studies, feeding studies). The analytical methods have been evaluated according to the guidance provided by OECD (Series on Pesticides number 39) as indicated on page 25 of the FAO manual 2009.

Validation results are required for every commodity submitted for MRL-setting: at least one full validation for a commodity within the five defined crop groups (high acid content, high water content, high oil content, high protein content, high starch content) and a reduced validation for every other commodity within a certain crop group. Where validation results do not meet the criteria given below, this is indicated.

When the analytical method is validated according to a full validation scheme, it means that:

- at least five recovery experiments per level were conducted on at least two levels (LOQ and $10\times$ LOQ) and average recovery per level was shown to be between 70–120% and the relative standard deviation (RSD_r or CV) per level was shown to be $< 20\%$
- at least two control samples were analysed and were shown to be below $0.3\times$ LOQ
- the calibration was conducted with at least five single points or at least three duplicate points and was shown to be linear (either standards in solvent or matrix matched standards).

When the analytical method is validated according to a reduced validation scheme, it means that:

- a full validation is available for a crop in the same crop group (high acid content, high water content, high oil content, high protein content, high starch content)
- at least three recovery experiments per level were conducted on at least two levels (LOQ and $10\times$ LOQ) and the average recovery per level was shown to be between 70–120% and the relative standard deviation (RSD_r or CV) per level was $< 20\%$
- at least two control samples were analysed and shown to be below $0.3\times$ LOQ
- the calibration was conducted with at least five single points or at least three duplicate points and was shown to be linear (only relevant for matrix matched standards; standards in solvent are already covered by full validation).

Analytical methods for enforcement in plant and animal commodities

FDA PAM Multi-Residue Method

The suitability of the FDA Pesticide Analytical Manual (PAM-I) Multi-Residue Method (MRM) to analyse for benzovindiflupyr and its three metabolites (SYN546039, SYN545720 and SYN546206) in non-fatty and fatty foods was tested using the FDA MRM Testing Protocol [Barker and Ballard, 2012, SYN546039_50003]. The FDA MRM Testing Protocol [FDA, 1999] consists of several protocols: one protocol for determining if the compounds are naturally fluorescent (protocol A); one protocol for methylation of acidic analytes, in case the native volatility is insufficient for GC determination (protocol B); two protocols for determining GC characteristics of chemicals (protocol C and G); three protocols by which chemicals can be evaluated through the FDA multi-residue testing program (protocols D, E and F); Based on its molecular structure, benzovindiflupyr and its metabolites, were taken through protocols A, B, C, D and F.

Protocol A (PAM I, Section 401, module DL2) demonstrated that benzovindiflupyr, SYN546039 or SYN546206 had no detector response in a HPLC system with fluorescence detection, even with 10,000 ng on column. Signals were reported for SYN545720 but did not provide enough response to warrant further investigation (greater than 10,000 ng required 50% FSD). Further work on this protocol was terminated.

Protocol B showed that compound SYN545720 could be methylated. However, 150 ng of SYN545720-methyl ester could not be detected with GC-ECD (module DG1). Further work on this protocol was terminated due to poor sensitivity of the methyl ester.

Protocol C experiments demonstrated that benzovindiflupyr SYN546039 and SYN546206 were found to be suitable for detection by GC-ECD using module DG10. None of the modules tested

(GC-ECD modules DG1, DG7, DG10 or GC-NPD module DG17) gave a suitable response for SYN545720. For compounds which can only be determined by ECD, the extract must be cleaned up by Florisil chromatography before determination.

Protocol D (for non-ionic chemicals in non-fatty foods) showed that benzovindiflupyr and its metabolites SYN546039 and SYN546206 could not be recovered through the section 301 C1 Florisil clean-up elution procedure. Further testing through Protocol D was terminated.

Protocol F (for moderately non-polar chemicals in fatty foods) showed that only benzovindiflupyr was recovered through either the C1 or C2 Florisil clean-up elution procedure. Recovery was 60% (50% ethyl ether eluent) for C1 and 106% (eluent 3) for C2 clean-up procedures. When corn oil was fortified with parent at 0.05 and 0.50 mg/kg and extracted using Section 304 E3 conditions, recovery was poor. Recoveries through the Section 304 E3 + C1 procedure averaged 32% and 57% for the 0.05 and 0.50 mg/kg fortified samples, respectively. Recoveries through the Section 304 E3 + C2 procedure averaged 56% and 58% for the 0.05 and 0.50 mg/kg fortified sample, respectively.

Conclusion: Data generated from testing of benzovindiflupyr and its metabolites SYN546039, SYN545720 and SYN546206 through Protocols A, B, C, D and F indicate that none of the compounds of interest are successful candidates for enforcement by the FDA PAM I multi-residue methods for non-fatty or fatty foods.

QuEChERS method

In the published multi-residue QuEChERS method [ECS, 2009] homogenised samples (5.0 g) were mixed with acetonitrile/water (1:1, v/v, plant matrices) or acetonitrile (animal matrices). Fat samples were heated to approximately 40 °C before mixing with acetonitrile. Thereafter, magnesium sulfate (to bind water), sodium chloride (to salt out the analyte), trisodium citrate dihydrate and disodium hydrogen citrate sesquihydrate (slightly acidic buffer) were added. After mixing, the sample was centrifuged. The upper acetonitrile phase was cleaned-up by a dispersive solid phase extraction purification step using PSA (primary and secondary amine exchange material) and magnesium sulfate. The mixture was centrifuged and the upper clear acetonitrile phase was concentrated using a stream of air. The final extract was diluted with water/acetonitrile (9/1, v/v, or 1/4, v/v). Samples were analysed by HPLC-MS-MS at their appropriate mass transitions.

The determination of parent using QuEChERS method was validated according to the full validation scheme in oranges (whole fruit), wheat (grain), lettuce (head), oilseed rape (seed) and coffee bean (roasted) [Winter and Armann, 2012, SYN545192_10193]. HPLC-MS-MS mass transitions m/z 396 $\rightarrow$ 368 and m/z 396 $\rightarrow$ 91 were used for quantification or confirmation; no preference could be seen in the results. The reported LOQ was 0.01 mg/kg for each commodity and each analyte. Average recoveries at 0.01 mg/kg and 0.1 mg/kg of parent were within 70–120% limits and RSDs were within 20%. Linearity of detector response (coefficient of determination $R^2 > 0.99$) was observed in the range of 0.25–50 ng/mL standards using five or six matrix-matched standards. This range covers at least 0.3–12LOQ in the samples. Significant matrix effects ($> 20\%$) were found for all matrices and therefore matrix-matched standards were used for quantification for all matrices. No interfering peaks $> 0.3\text{LOQ}$ (i.e. 0.003 mg/kg) were detected in any of the control extracts.

The determination of parent by the QuEChERS method was independently validated according to the full validation scheme on lettuce (head), oilseed rape (seed) and coffee bean (roasted) [Watson, 2012a, SYN545192_10172]. The validation was performed for both MS-MS transitions. The reported LOQ was 0.01 mg/kg for each commodity and each analyte. Average recoveries at 0.01 mg/kg and 0.1 mg/kg of parent were within 70–120% limits and RSDs were within 20%. Linearity of detector response (coefficient of determination $R^2 > 0.999$) was observed in the range of 0.25–50 ng/mL standards using six matrix-matched standards. This range is equivalent to 0.25–25LOQ in the samples. Significant matrix effects ($> 20\%$) were observed for oilseed rape seed and coffee bean matrix for both transitions and therefore matrix-matched standards were used for quantification for all matrices. No interfering peaks $> 0.3\text{LOQ}$ (i.e. 0.003 mg/kg) were detected in any of the control extracts.

The determination of parent using the published multi-residue QuEChERS method (EN 15662:2009) was validated according to the full validation scheme in bovine meat, liver, kidney, fat, milk and chicken eggs [Class and Göcer, 2011, SYN545192_10150]. HPLC-MS-MS mass transition m/z 396 $\rightarrow$ 91 was used for quantification; m/z 396 $\rightarrow$ 368 was used for confirmation. The reported LOQ was 0.01 mg/kg for each commodity and each analyte. Average recoveries at 0.01 mg/kg and 0.1 mg/kg of parent were within 70–120% limits and RSDs were within 20%. Linearity of detector response (correlation coefficient $r > 0.99$ for $1/\times$ weighted regression) was observed in the range of 0.05–5 ng/mL (fat) or 0.10–10 ng/mL (other matrices) using seven standards in solvent. This range is equivalent to 0.20–20LOQ in the samples [Syngenta, 2014c]. Significant matrix effects ($> 20\%$) were not observed and standards in solvent were used for calibration. No interfering peaks > 0.3 LOQ (i.e. 0.003 mg/kg) were detected in any of the control extracts.

The determination of parent using the published multi-residue QuEChERS method (EN 15662:2009) was independently validated according to the full validation scheme in bovine liver, fat, milk and chicken eggs [Watson, 2012b, SYN545192_10169]. HPLC-MS-MS mass transition m/z 396 $\rightarrow$ 91 was used for quantification; m/z 396 $\rightarrow$ 368 was used for confirmation. The reported LOQ was 0.01 mg/kg for each commodity and each analyte. Average recoveries at 0.01 mg/kg and 0.1 mg/kg of parent were within 70–120% limits and RSDs were within 20%. Linearity of detector response (coefficient of determination $R^2 > 0.99$ for $1/\times$ weighted regression) was observed in the range of 0.05–5 ng/mL (fat) or 0.10–10 ng/mL (other matrices) using seven standards in solvent. This is equivalent to 0.2–20LOQ in the samples. Significant matrix effects ($> 20\%$) were not observed and standards in solvent were used for calibration. No interfering peaks > 0.3 LOQ (i.e. 0.003 mg/kg) were detected in any of the control extracts.

Conclusion: The multi-residue QuEChERS method is considered valid for use as enforcement method in plant commodities with high acid content (oranges), high water content (lettuce), high starch content (wheat grain), high oil content (oilseed rape, coffee beans) and animal commodities (meat, liver, kidney, fat, milk and eggs) with an LOQ of 0.01 mg/kg for benzovindiflupyr (parent).

Analytical methods used in study reports in plant commodities

HPLC-MS-MS method GRM042.03A

HPLC-MS-MS method GRM042.03A, version 4 August 2011, [Braid and Lin, 2011b, SYN545192_10118] is intended for use as pre-registration method for the determination of parent, and SYN546039 (free and conjugates) in plant commodities. The original method or its modification was used in field rotational crop studies and storage stability studies.

High water content commodities (10 g) were extracted by homogenisation in 80/20 (v/v) acetonitrile/water. Dry matrices (5 g, e.g. grains, hay, straw) were pre-soaked with 25 mL water for 30 min prior to homogenisation. An aliquot of the extracts was evaporated at 50 °C under a stream of air or nitrogen to remove the acetonitrile and to concentrate the sample. The remaining aqueous extract was acidified with 1 M HCl and partitioned with iso-hexane to separate parent off. The iso-hexane fraction was evaporated to dryness and taken up in 50/50 v/v acetonitrile/water for direct determination of parent by HPLC-MS-MS. Alternatively the parent fraction (in iso-hexane) may be stored refrigerated and then combined with the post-hydrolysis extract for sample clean-up if required. The remaining aqueous acidic fraction was diluted to 0.5 M HCl and heated at 100 °C for 6 hours to hydrolyse conjugates of SYN546039. If necessary, the parent fraction in iso-hexane was diluted with methanol and evaporated to remove the iso-hexane and to concentrate the sample. The hydrolysate (together with the parent fraction if necessary) was cleaned-up using an SPE cartridge. SYN546039 (and parent if included at this stage) was eluted with 50/50 (v/v) acetonitrile/water. Following dilution, samples were analysed by HPLC-MS/MS. The primary transition monitored for parent was m/z 396 $\rightarrow$ 91 with confirmatory transition m/z 396 $\rightarrow$ 368. For SYN546039 the primary transition monitored was m/z 412 $\rightarrow$ 91 with confirmatory transition m/z 412 $\rightarrow$ 340. The reported LOQ is 0.01 mg/kg for both parent and SYN546039. Samples with expected residues > 0.1 mg/kg need to be diluted before analysis.

The analysis conditions as specified in the method description need to be followed carefully. Since parent compound degrades under the hydrolysis conditions used to hydrolyse conjugates of SYN546039 into their free form, parent is separated from the sample before hydrolysis. Change in hydrolysis conditions may result in either incomplete deconjugation or degradation of SYN546039 (free). Parent compound has been observed to adsorb to glass surfaces in solutions with < 50% organic solvent. These considerations have been dealt with in the described analytical method.

HPLC-MS-MS Method GRM042.03A was validated [Lin, 2012, SYN545192_50049] according to the full validation scheme for apples, grapes, wheat commodities (forage, grain, hay and flour), spinach, peanuts, coffee beans, carrot roots, orange juice and sugarcane. The validation was performed for the quantification transition ions as well as for the confirmation transition ions. Fortifications of parent compound were performed before extraction and parent compound was quantified after the SPE clean-up stage for all commodities. Fortifications of SYN546039 were performed after the acid hydrolysis step. The reported LOQ was 0.01 mg/kg for each commodity and each analyte. Average recoveries at 0.01 mg/kg and 0.1 mg/kg of parent or SYN546039 (free) were within 70–120% limits and RSDs were within 20%. For wheat forage and wheat hay recoveries at 0.01 mg/kg and 0.5 mg/kg were also within these ranges. Linearity of detector response (coefficient of correlation $r > 0.99$) was observed in the range 0.02–0.5 ng/mL using 5–6 single standards. Equivalent concentrations in the samples were not stated. The magnitude of the matrix effect was considered not to be significant (< 20% suppression or enhancement) with the exception of coffee beans. Matrix matched standards were used for coffee beans; standards in solvent were used for all other matrices. No interfering peaks > 0.3LOQ (i.e. 0.003 mg/kg) were detected in any of the control extracts.

Reduced validation data were available for the determination of parent and SYN546039 (free) in wheat (forage, hay, grain, straw), lettuce, spinach, turnips (roots, tops) and radish (roots, tops) [Mäyer, 2012, A17056B_50013]. Parent was analysed after SPE clean-up. Fortifications of both parent and SYN546039 were performed prior to extraction. Average recoveries at 0.01 mg/kg ($n=3$) and 0.1 mg/kg ($n=1$) were within 70–120% limits and RSDs were within 20%. Linearity of detector response (coefficient of correlation $r > 0.99$ for $1/\times$ weighted regression) was observed in the range 0.1–2 ng/mL or 0.02–1 ng/mL using five single matrix matched standards. No interfering peaks > 0.005 mg/kg) were detected in any of the control extracts.

Reduced validation data were available for the determination of parent and SYN546039 (free) in wheat straw and carrot (roots, tops) [Ellis, 2012b/c, A17056B_10004, A17056B_10005]. Parent was analysed directly in the initial acetonitrile/water extract. Average recoveries at 0.01 mg/kg ($n=3$), 0.05 mg/kg ($n=2$) and 0.1 mg/kg ($n=1$) were within 70–120% limits and RSDs were within 20% at 0.01 mg/kg. Linearity of detector response (coefficient of correlation $r > 0.999$) was observed in the range 0.02–4 ng/mL using five single matrix matched standards. No interfering peaks > 0.3LOQ (i.e. 0.003 mg/kg) were detected in any of the control extracts.

Modification A of HPLC-MS-MS method GRM042.03A was used for quantification of parent and SYN546039 in storage stability study 1 [Watson, 2013, SYN545192_10137]. Parent and SYN546039 were determined directly in the acetonitrile/water (80/20) extract without further clean-up. The hydrolysis step was not included for the purpose of this study. Validation data were available for spinach, orange, wheat grain, wheat straw and potato at the fortification level used in the storage stability study. Parent or SYN546039 (free) were added before extraction as a single analyte per sample. Average recoveries at 0.2 mg/kg of parent or SYN546039 (free) were within 70–120% limits and RSDs were within 20% ($n=12$). Linearity of detector response (coefficient of correlation $r > 0.99$) was observed in the range 0.02–10 ng/mL using 5–6 single matrix matched standards. No interfering peaks > 0.3LOQ (i.e. 0.003 mg/kg) were detected in any of the control extracts.

Modification B of HPLC-MS-MS method GRM042.03A was used for quantification of parent and SYN546039 in storage stability study 3 [Hagan and Bertrand, 2012, SYN545192_50177]. Samples were extracted with acetonitrile/water (80:20, v/v). An aliquot of the extracts was evaporated at 50 °C under a stream of air or nitrogen to remove the acetonitrile and to concentrate the sample. The remaining aqueous extract was acidified with 1 M HCl and cleaned-up using an SPE cartridge. Parent and SYN546039 were eluted with acetonitrile/water (60:40, v/v). The hydrolysis step was not

included for the purpose of this study. Validation data were available for maize flour and maize meal, raisins, dried apple and apple juice at the fortification level used in the storage stability study. Parent or SYN546039 (free) were added before extraction as a single analyte per sample. Average recoveries at 0.2 mg/kg of parent or SYN546039 (free) were within 70–120% limits and RSDs were within 20% (n=8). Linearity of detector response (coefficient of correlation $r > 0.99$, $1/\times$ weighted regression) was observed in the range 0.02–1 ng/mL using seven single standards in solvent. No interfering peaks (> 0.02 ng/mL standard signal) were detected in any of the control extracts.

Conclusion:

- HPLC-MS-MS method GRM042.03A is considered valid for the determination of parent and SYN546039 (free) in the range 0.01–0.1 mg/kg for apples, grapes, wheat (forage, grain, hay, straw, flour), spinach, lettuce, peanuts, coffee beans, carrot (roots and leaves), turnip (roots and leaves), radish (roots and leaves), orange juice and sugarcane. The valid range is extended to 0.5 mg/kg for wheat forage and wheat hay.
- The original method indicates to fortify the sample with the free form of SYN546039 after the acid hydrolysis procedure, and thereby only validates the SPE clean-up part. However, reduced validation data are available, where SYN546039 was added before extraction and therefore, the full analytical procedure including extraction, liquid-liquid partition and hydrolysis was validated for the free form of SYN546039.
- Efficiency of extraction and hydrolysis for SYN546039 including its conjugates has been verified in a radio-validation study using method GRM042.04A, where the extraction and hydrolysis procedures are identical to those in method GRM042.03A. Based on this study, HPLC-MS-MS method GRM042.03A is considered sufficiently validated for the determination of the free and conjugated forms of SYN546039.
- Modification A of HPLC-MS-MS method GRM042.03A is considered valid for the determination of parent and SYN546039 (free) at the level of 0.2 mg/kg for spinach, orange, wheat grain, wheat straw and potato for the purpose of storage stability studies only.
- Modification B of HPLC-MS-MS method GRM042.03A is considered valid for the determination of parent and SYN546039 (free) at the level of 0.2 mg/kg for spinach, orange, maize flour and maize meal, raisins, dried apple and apple juice for the purpose of storage stability studies only.

HPLC-MS-MS methods GRM042.04A and POPIT MET.134 rev 01

HPLC-MS-MS Method GRM042.04A, version 4 August 2011, [Braid and Lin, 2011c, SYN545192_10119] is intended for use as pre-registration method for the determination of parent, SYN546039 (free and conjugates) and SYN545720 (free and conjugates) in soya bean commodities. The original method or its modification was used in supervised residue trials on soya beans, processing studies on soya beans and storage stability studies.

In the original method, soya bean forage (10 g) was extracted by homogenisation in 80/20 v/v acetonitrile/water. Soya bean hay (5 g) was pre-soaked with 25 mL water for 30 min prior to homogenisation. Soya bean seeds (10 g) were extracted by homogenisation with 50/50 v/v acetonitrile/water. Soya bean oil (10 g) was dissolved in 50 mL n-hexane and residues were extracted by partition into 80/20 v/v acetonitrile/water. At this stage, an aliquot of the extract may be diluted with 50/50 acetonitrile/water for direct determination of parent by HPLC-MS-MS (soya bean oil). For further clean-up, an aliquot of the extract was evaporated at 50 °C under a stream of air or nitrogen to remove the acetonitrile and to concentrate the sample. The remaining aqueous extract was acidified with 1 M HCl and partitioned with iso-hexane to separate parent off. The iso-hexane fraction was evaporated to dryness and taken up in 50/50 v/v acetonitrile/water for direct determination of parent by HPLC-MS-MS (soya bean forage, hay and seed). Alternatively the parent fraction (in iso-hexane) may be stored refrigerated and then combined with the post-hydrolysis extract for sample clean-up if required. The remaining aqueous acidic fraction was diluted to 0.5 M HCl and heated at 100 °C for 6

hours to hydrolyse SYN546039 conjugates in all crop commodities and SYN545720 conjugates in soya bean seed. If necessary, the parent fraction in iso-hexane was diluted with methanol and evaporated to remove the iso-hexane and to concentrate the sample. The hydrolysate (together with the parent fraction if necessary) was cleaned-up on a SPE cartridge. SYN545720 was eluted with 50/50 v/v methanol/water and SYN546039 (and parent if included at this stage) were eluted with 50/50 v/v acetonitrile/water. Following dilution, samples were analysed separately by HPLC-MS/MS. The primary transition monitored for parent was m/z 396→91 with confirmatory transition m/z 396→368. For SYN546039 the primary transition monitored was m/z 412→91 with confirmatory transition m/z 412→340. The primary transition monitored for SYN545720 is m/z 161→141 with confirmatory transition m/z 161→66. Samples with expected residues > 0.1 mg/kg need to be diluted before analysis.

The analysis conditions as specified in the method description need to be followed carefully. Since parent compound degrades under the hydrolysis conditions used to hydrolyse conjugates of SYN546039 and SYN545720 into their free form, parent is separated from the sample before hydrolysis. Change in hydrolysis conditions may result in either incomplete deconjugation or degradation of SYN546039 (free). Parent compound has been observed to adsorb to glass surfaces in solutions with < 50% organic solvent. These considerations have been dealt with in the described analytical method.

The original HPLC-MS-MS Method GRM042.04A was validated [Lin, 2012, SYN545192_50049] according to the full validation scheme for soya bean commodities (forage, oil, seed and milk). For soy milk, the extraction scheme as for forage was used [Syngenta, 2014b]. The validation was performed for the quantification transition ions as well as for the confirmation transition ions. Fortifications of parent compound were performed before extraction and parent compound was quantified after the acetonitrile/water extraction stage for soya bean oil, after the iso-hexane liquid-liquid partition stage for soya bean seeds, and after the SPE clean-up stage for soya bean forage and soya bean milk. Fortifications of SYN546039 and SYN545720 were performed after the acid hydrolysis step. The reported LOQ was 0.01 mg/kg for each commodity and each analyte. Average recoveries at 0.01 mg/kg and 0.1 mg/kg of parent or SYN546039 (free) or SYN545720 (free) were within 70–120% limits and RSDs were within 20%. For soya bean forage recoveries at 0.01 mg/kg and 0.5 mg/kg were also within these ranges. Linearity of detector response (coefficient of correlation $r > 0.99$, no weighing) was observed in the range 0.02–0.5 ng/mL for parent and SYN546039 using six standards. Linearity of detector response (coefficient of correlation $r > 0.99$, no weighing) was observed in the range 0.05–0.5 ng/mL for SYN545720 using five standards. Equivalent concentrations in the samples were not stated. The magnitude of the matrix effect was considered not to be significant (< 20% suppression or enhancement), with the exception of SYN546039 in soya bean seed and SYN545720 in soya bean forage. Standards in solvent were used for soya bean oil and soya bean milk; matrix matched standards were used for soya bean seeds and soya bean forage. No interfering peaks > 0.3LOQ (i.e. 0.003 mg/kg) were detected in any of the control extracts.

HPLC-MS-MS Method POPIT MET.134, Rev01, version 10 January 2012, [Volpi, 2012, SYN545192_10195] is a modification of HPLC-MS-MS method GRM042.04A and is intended for analysis of soya bean seeds only. Rev 01 is identical to the original version of 23 May 2011, except for some textual changes. The method was used in the Brazilian supervised residue trials on soya bean seeds. Extraction was conducted as described for GRM042.04A, except that 5 g soya bean seeds were taken instead of 10 g and extraction volumes were adapted accordingly. Parent was directly analysed after iso-hexane liquid-liquid partition without further clean-up. The hydrolysate, containing SYN545720 and SYN546039, was cleaned-up on a SPE cartridge. SYN545720 and SYN546039 were eluted with acetonitrile instead of 50/50 v/v methanol/water or 50/50 v/v acetonitrile/water, respectively. Following dilution, samples were analysed by HPLC-MS/MS using different transition ions. The primary transition monitored for parent was m/z 396→91 with confirmatory transition m/z 396→131. For SYN546039 the primary transition monitored was m/z 412→340 with confirmatory transition m/z 412→91. The primary transition monitored for SYN545720 is m/z 161→141 with confirmatory transition m/z 161→66.

HPLC-MS-MS Method POPIT MET.134 rev01 was validated [Volpi, 2012, SYN545192_10195] according to the full validation scheme for soya bean seeds. The validation was performed only for the quantification transition ions. Fortifications of parent compound were performed before extraction and parent compound was quantified after the iso-hexane liquid-liquid partition stage. Fortifications of SYN546039 and SYN545720 were performed after the acid hydrolysis step. The reported LOQ was 0.01 mg/kg for each commodity and each analyte. Average recoveries at 0.01 mg/kg and 0.1 mg/kg of parent or SYN546039 (free) or SYN545720 (free) were within 70–120% limits and RSDs were within 20%. Linearity of detector response (coefficient of determination $R^2 > 0.99$) was observed in the range of 0.5–16 ng/mL for parent and 0.25–8.0 ng/mL for SYN545039 and SYN545720 using six duplicate standards. This is equivalent to 0.5–16LOQ in the samples. Standards for parent were prepared in solvent; standards for SYN545039 and SYN545720 were prepared in matrix. No interfering peaks $> 0.3\text{LOQ}$ (i.e. 0.003 mg/kg) were detected in any of the control extracts.

Modification A of HPLC-MS-MS method GRM042.04A was used for quantification of parent and SYN546039 in storage stability study 1 [Watson, 2013, SYN545192_10137]. Dry broad bean seeds (5 g) were pre-soaked in water prior to extraction. Parent and SYN546039 were determined directly in the acetonitrile/water (80/20) extract from dry broad beans without further clean-up. Dry soya bean extracts required further clean-up. Parent was determined in dry soya bean extracts after the iso-hexane partition step, while SYN546039 was determined after SPE clean-up. The hydrolysis step to free the conjugated residues of SYN546039 was not included for the purpose of the study. Parent or SYN546039 (free) were added before extraction as a single analyte per sample. Additional validation data were available for dry soya beans and dry broad beans at the fortification level used in the storage stability study. Average recoveries at 0.2 mg/kg of parent or SYN546039 (free) were within 70–120% limits and RSDs were within 20% ($n=12$). Linearity of detector response (coefficient of correlation $r > 0.999$) was observed in the range 0.02–10 ng/mL using 5–6 single matrix matched standards. No interfering peaks $> 0.3\text{LOQ}$ (i.e. 0.003 mg/kg) were detected in any of the control extracts.

Modification B of HPLC-MS-MS method GRM042.04A was used for quantification of parent and SYN546039 in storage stability Study 3 [Hagan and Bertrand, 2012, SYN545192_50177]. Soya bean flour and soya bean milk were extracted with 50/50 v/v acetonitrile/water. Soya bean oil and maize oil were dissolved in 50 mL n-hexane and residues were extracted by partition into 80/20 v/v acetonitrile/water. For further clean-up, an aliquot of the extract was evaporated at 50 °C under a stream of air or nitrogen to remove the acetonitrile and to concentrate the sample. The remaining aqueous extract was acidified with 1 M HCl and cleaned up by SPE. SYN545720 was eluted with 50/50 v/v methanol/ water and parent and SYN546039 were eluted with 50/50 v/v acetonitrile/water. The hydrolysis step to free the conjugated residues of SYN546039 was not included for the purpose of the study. Parent or SYN546039 (free) or SYN545720 were added before extraction as a single analyte per sample. SYN546720 was only added to soya bean matrices. Validation data were available for maize oil, soya bean flour, soya bean milk and soya bean oil at the fortification level used in the storage stability study. Average recoveries at 0.2 mg/kg of parent or SYN546039 (free) or SYN545720 were within 70–120% limits and RSDs were within 20% ($n=8$). Linearity of detector response (coefficient of correlation $r > 0.99$, $1/\times$ weighted regression) was observed in the range 0.02–1 ng/mL using seven single standards in solvent. No interfering peaks (> 0.02 ng/mL standard signal) were detected in any of the control extracts.

Modification C of HPLC-MS-MS method GRM042.04A was used for quantification of parent, SYN546039 and SYN545720 in a soya bean processing study [Ellis, 2012a, A17056D_10002]. Due to small sample sizes, volumes and weights for extraction were reduced. Soya bean oil was dissolved in n-hexane and partitioned into acetonitrile/water (80/20, v/v), as described in the original method. The extract was diluted with acetonitrile/water (25/75, v/v) prior to quantification of parent (soya bean oil). All other soya bean commodities were extracted with 50/50 acetonitrile/water as described in the original method for seeds. In these commodities, parent was analysed after iso-hexane liquid-liquid partition without SPE clean-up step. As in the original method validation, the free forms of SYN546039 and SYN545720 were added after the hydrolysis step to

verify the recovery. Matrix matched standards were used for quantification of all three analytes for all matrices. For soya bean meal, fat soy flour, pollard, refined oil, crude oil and aspirated grain fractions the secondary transition m/z 396 to 368 was used for quantification of parent, since the primary transition gave interferences. For pollard and fat soy flour the secondary transition of m/z 412 to 340 was used for quantification of SYN546039, since the primary transition gave interferences.

Modification C of HPLC-MS-MS method GRM042.04A was validated with very limited data [Ellis, 2012a, A17056D_10002]. Limited validation data were available in the range 0.01–1.0 mg/kg for soya bean seeds and aspirated grain fractions, 0.01–0.5 mg/kg for soya bean hulls, soy oil, soya bean meal, fat soy flour, pollard, okara, soy milk, tofu, soy sauce and miso. Fortifications of parent compound were performed before extraction and parent compound was quantified after the acetonitrile/water extraction stage for soya bean oil and after the iso-hexane liquid-liquid partition stage for all other soya bean commodities. Individual recoveries were between 72–116% for each matrix and analyte (range 0.01–1.0 mg/kg, $n=1-2$ per level, 3–4 levels per matrix). These limited method recoveries confirm the validity of the method under the modified conditions and for the processed soya bean commodities. Linearity of detector response (coefficient of correlation $r > 0.99$) was observed in the range 0.2–4 ng/mL using five single matrix matched standards. No interfering peaks (> 0.01 mg/kg) were detected in any of the control extracts.

The extraction and hydrolysis procedure used in method GRM042.04A was radio-validated using samples of soya bean hay and soya bean seed from the pyrazole labelled metabolism study [Oddy, 2012, SYN545192_10102]. An aliquot of the homogenised soya bean hay sample (5 g) was soaked in 25 mL water for 30 min and subsequently extracted with acetonitrile/water (80:20 v/v). An aliquot of the homogenised soya bean seed sample (10 g) was extracted twice with acetonitrile/water (1:1 v/v). Following centrifugation aliquots of the supernatants of each sample were analysed by LSC to determine the efficiency of extraction. A separate aliquot of the supernatants was evaporated to remove the acetonitrile and to concentrate the extract, acidified and partitioned (3 $\times$) with iso-hexane. The aqueous phase was adjusted to 0.5 M HCl and heated at 100 °C for 6 hrs. Aliquots of the hexane phase and the hydrolysate were analysed by LSC and TLC to determine levels of parent and the metabolites SYN546039 (free and conjugates) and SYN545720 (free and conjugates).

Results are shown in Table 23. Analysis of soya bean hay and soya bean seed extracts by LSC showed that at least 94% of the total radioactive residue could be extracted by method GRM042.04A. Good agreement was obtained between the levels of parent, SYN546039 (free including conjugates) and SYN546720 (free including conjugates) found after extraction (and hydrolysis) as performed in the metabolism study and as performed in method GRM042.04A. The slightly higher levels of parent and SYN546720 in soya bean hay and seeds obtained by method GRM042.04A could be the result of a different extraction and hydrolysis scheme used in the metabolism study.

Table 23 Radiovalidation of extraction procedures used in analytical method GRM042.04A

Commodity	Components	Metabolism Study		Method GRM042.04A		Trueness
		%TRR	Residue mg/kg eq	%TRR	Residue mg/kg eq	
Soya bean hay	TRR extracted	98 ^a	12	100 ^b	13	103%
	parent	67	8.4	68 ^b	8.5	99%
	SYN546039 incl conjugates	12 ^c	1.5	14 ^c	1.7	112%
Soya bean seed	TRR extracted	98 ^a	0.099	94 ^b	0.095	96%
	parent	15	0.015	18 ^b	0.018	124%
	SYN545720 incl conjugates	47 ^d	0.048	57 ^c	0.058	121%

^a In the metabolism study primary extraction of soya bean hay and seeds was by hexane (3 $\times$), diethyl ether (2 $\times$), acetonitrile (2 $\times$), acetonitrile/water 4:1 v/v (2 $\times$ –7 $\times$), acetonitrile/water 3:7 v/v (1 $\times$ –2 $\times$), water (1 $\times$), and acetonitrile (1 $\times$).

^b In method GRM042.04A primary extraction was by acetonitrile/water 4:1 v/v (1 $\times$) for hay and acetonitrile/water 1:1 v/v (2 $\times$) for seeds. After concentration and acidification of the extract, parent was partitioned into iso-hexane.

^c In the metabolism study for hay only the acetonitrile/water extracts (26.0% TRR) were subjected to 3 hydrolysis procedures, of which bovine rumen fluid (pH 7, 24 hrs at 39 °C) was the most effective.

^d In the metabolism study for seeds only the acetonitrile/water extracts (85.0% TRR) were subjected to hydrolysis by

bovine rumen fluid (pH 7, 24 hrs at 39 °C). The resulting hydrolysate was partitioned between diethyl ether and water. The resulting aqueous phase (29.8% TRR) was subjected to hydrolysis by 0.5 M HCl for 6 hrs at 100 °C.

^e In GRM042.04A the aqueous phase after liquid-liquid partition was subjected to 0.5 M HCl for 6 hrs at 100 °C.

Conclusion:

- Analytical method GRM042.04A and its modifications have three possible moments to analyse parent: just after acetonitrile/water extraction, just after iso-hexane partition or after SPE clean-up. Validation of the method therefore needs to mention the conditions for the parent analysis.
- Analytical method GRM042.04A and its modifications indicate that fortifications of SYN546039 and SYN545720 need to be conducted after the acid hydrolysis procedure. In this way, the full analytical procedure is not validated for the free forms of SYN546039 and SYN545720, because loss or degradation during extraction, liquid-liquid partition and hydrolysis remains unnoticed. It is recommended to include a validation for the whole procedure in case method compliance needs to be shown during actual sample analysis.
- Analytical method GRM042.04A and its modifications use a hydrolysis procedure to cleave the SYN546039 and SYN545720 conjugates. The extraction efficiency of the free forms and the conjugates, the efficacy of the hydrolysis procedure for the conjugates and the stability of the free forms during hydrolysis is validated by a separate radio-validation experiment in soya bean seeds and soya bean hay. The recovery of any SYN546039 or SYN545720 released after hydrolysis is validated by the fortifications of SYN546039 or SYN546720 after the acid hydrolysis procedure.
- The original HPLC-MS-MS method GRM042.04A is considered valid for the determination of parent, SYN546039 (free and conjugated) and SYN545720 (free and conjugated) in the range 0.01–0.1 mg/kg for soya bean seeds, soya bean oil, soya bean milk and 0.01–0.5 mg/kg for soya bean forage. The validity for the parent compound extends only to quantification after the acetonitrile/water extraction stage for soya bean oil, after iso-hexane partition for soya bean seeds and after SPE clean-up for soya bean milk and soya bean forage.
- HPLC-MS-MS method POPIT MET134 Rev 01 is considered valid for the determination of parent, SYN546039 (free and conjugated) and SYN545720 (free and conjugated) in the range 0.01–0.1 mg/kg for soya bean seed. The validity for the parent compound extends only to quantification after iso-hexane partition for soya bean seeds.
- Modification A of HPLC-MS-MS method GRM042.04A is considered valid for the determination of parent and SYN546039 (free) at the level of 0.2 mg/kg for dry soya beans and dry broad beans for the purpose of storage stability studies only.
- Modification B of HPLC-MS-MS method GRM042.04A is considered valid for the determination of parent and SYN546039 (free) at the level of 0.2 mg/kg for maize oil, soya bean flour, soya bean milk and soya bean oil for the purpose of storage stability studies only.
- Modification C of HPLC-MS-MS method GRM042.04A is considered valid for the determination of parent, SYN546039 (free and conjugated) and SYN545720 (free and conjugated) in the range 0.01–1.0 mg/kg for soya bean seeds and aspirated grain fractions, 0.01–0.5 mg/kg for soya bean hulls, soy oil, soya bean meal, fat soy flour, pollard, okara, soy milk, tofu, soy sauce and miso. The validity for the parent compound extends only to quantification after the acetonitrile/water extraction stage for soya bean oil and after the iso-hexane liquid-liquid partition stage for all other soya bean commodities.

HPLC-MS-MS method GRM042.08A

HPLC-MS-MS Method GRM042.08A, version 26 June 2012, [Braid, 2012, SYN545192_10216] is intended for use as pre-registration method for the determination of parent (free), SYN546039 (free

and conjugates) and SYN546206 (free) in plant commodities. The original method or its modification was used in field rotational crop studies and storage stability studies.

High water content crops (10 g) were extracted by homogenisation in 80/20 (v/v) acetonitrile/water. Dry sample matrices (5 g, e.g. grains, straw, hay) were pre-soaked with water (25 mL) for 30 min prior to homogenisation. For the determination of parent and SYN546206 (free), diluted aliquots of the filtered extracts were analysed directly by HPLC-MS-MS. For the determination of SYN546039, aliquots of the extracts were evaporated under a stream of air or nitrogen to remove the acetonitrile, acidified with 1 M HCl and partitioned with 75/25 (v/v) iso-hexane/diethyl ether to separate parent and SYN546206 off. The iso-hexane/diethyl ether fraction was back-partitioned with water to recover the small amount of conjugated SYN546039. The water fraction was then combined with the aqueous acidic fraction. The aqueous acidic fraction was heated at 100 °C for 6 hrs to hydrolyse conjugates of SYN546039. The hydrolysate was cleaned up by SPE and SYN546039 was eluted with 65/35 (v/v) acetonitrile/ water. Following dilution, samples were analysed by HPLC-MS-MS. The primary transition monitored for parent was m/z 396→91 with confirmatory transition m/z 396→368. For SYN546039 the primary transition monitored was m/z 412→91 with confirmatory transition m/z 412→340. The primary transition monitored for SYN546206 is m/z 382→342 with confirmatory transition m/z 382→362. Samples with expected residues > 0.1 mg/kg need to be diluted before analysis.

The analysis conditions as specified in the method description need to be followed carefully. Since parent compound degrades under the hydrolysis conditions used to hydrolyse conjugates of SYN546039 into their free form, parent is separated from the sample before hydrolysis. This step will also remove residues of SYN546206 as these are not conjugated. The metabolite SYN546039 is mainly conjugated in rotational crops and to release the free metabolite, an acid hydrolysis step is required. Change in hydrolysis conditions may result in either incomplete deconjugation or degradation of SYN546039 (free). Parent compound has been observed to adsorb to glass surfaces in solutions with < 50% organic solvent and therefore storage conditions need to be chosen carefully. These considerations have been dealt with in the described analytical method.

HPLC-MS-MS Method GRM042.08A was validated [Watson, 2012c, SYN545192_10203] according to the full validation scheme for spinach, carrot roots and wheat commodities (grain, straw, forage). The validation was performed for the quantification transition ions as well as for the confirmation transition ions. Samples were fortified with a mixture of SYN545192, SYN546039 and SYN546206 standard before extraction. The reported LOQ was 0.01 mg/kg for each commodity and each analyte. Average recoveries at 0.01 mg/kg and 0.1 mg/kg of parent or SYN546039 (free) or SYN546206 (free) were within 70–120% limits and RSDs were within 20%. For wheat straw and wheat forage recoveries at 0.01 mg/kg and 0.5 mg/kg were also within these ranges. Linearity of detector response (coefficient of determination $R^2 > 0.99$) was observed in the range 0.1–4 ng/mL using six single standards. This range is equivalent to 0.25–10× LOQ in the samples. Samples which were fortified at 10× LOQ were diluted by a factor of two and samples which were fortified at 50× LOQ were diluted by a factor of 10 to fall within the linear range. The magnitude of the matrix effect was considered to be significant ($\geq 20\%$ suppression or enhancement). Matrix matched standards were used for each matrix/analyte combination. No interfering peaks > 0.3LOQ (i.e. 0.003 mg/kg) were detected in any of the control extracts.

Additional validation data for the determination of SYN546206 were available for spinach, wheat grain, wheat straw and potato at the fortification level used in the storage stability study [Watson, 2013, SYN545192_10137]. SYN546206 was added before extraction as a single analyte per sample. Average recoveries at 0.2 mg/kg of SYN546206 were within 70–120% limits and RSDs were within 20% (n=12). Linearity of detector response (coefficient of correlation $r > 0.999$) was observed in the range 0.02–10 ng/mL using six single matrix matched standards. No interfering peaks > 0.3LOQ (i.e. 0.003 mg/kg) were detected in any of the control extracts.

Reduced validation data were available for the determination of parent, SYN546206 (free) and SYN546039 (free) in carrot (roots and tops) [Ellis, 2012b/c, A17056B_10004, A17056B_10005]. Average recoveries at 0.01 mg/kg (n=3), 0.05 mg/kg (n=2) and 0.1 mg/kg (n=1) were within 70–

120% limits and RSDs were within 20% at 0.01 mg/kg. Linearity of detector response (coefficient of correlation $r > 0.999$) was observed in the range 0.02–4 ng/mL using five single matrix matched standards. No interfering peaks $> 0.3\text{LOQ}$ (i.e. 0.003 mg/kg) were detected in any of the control extracts.

Modification A of HPLC-MS-MS method GRM042.08A was used in the US field rotational crop studies [Mayer, 2012, A17056B_50013]. Parent and SYN546206 were analysed after liquid-liquid partition and SPE clean-up. An aliquot of the acetonitrile/water extracts was evaporated under a stream of nitrogen to remove the acetonitrile. The remaining aqueous extract was acidified with 1 M HCl and partitioned with 75:25 iso-hexane/diethyl ether to separate parent and SYN546206 (organic phase) from SYN546039 (acidic aqueous phase). The iso-hexane/diethyl ether fraction was back-partitioned with water. The organic fraction was evaporated to dryness and taken up in 50/50 v/v acetonitrile/water. The aqueous fractions were combined, diluted to 0.5 M HCl, and heated at 100 °C for 6 hours to hydrolyse conjugates of SYN546039. The post-hydrolysis extract containing the SYN546039 residues was combined with the fraction containing the parent and SYN546206 residues and cleaned-up on SPE using acetonitrile/water (50/50, v/v) as eluent. The eluent was analysed by HPLC-MS-MS.

Only limited validation data were available for modification A for the determination of parent, SYN546206 (free) and SYN546039 (free) in lettuce, spinach, wheat commodities (forage, hay, straw and grain), turnip (roots, tops), radish (roots and tops) [Mayer, 2012, A17056B_50013]. The original method determines parent and SYN546206 directly in the initial extract, while the modification determines parent and SYN546206 after liquid-liquid partition and an SPE clean-up step, which may introduce losses of both analytes. Individual recoveries at 0.01 mg/kg ($n=1$) and 0.1 mg/kg ($n=1$) for each of the commodities were within 70–120% limits. Linearity of detector response (coefficient of correlation $r > 0.999$) was observed in the range 0.02–4 ng/mL using five single matrix matched standards. No interfering peaks > 0.005 mg/kg) were detected in any of the control extracts.

The extraction procedure used in method GRM042.08A was radio-validated using samples of wheat hay and straw from the pyrazole labelled confined rotational crop study [Roohi and Adams, 2012, SYN545192_10246]. An aliquot of the homogenised wheat hay and straw sample (5 g) was soaked in 25 mL water for 30 min and subsequently extracted with acetonitrile/water (80:20 v/v). Following centrifugation aliquots of the supernatants of each sample were analysed by LSC and TLC

Results are shown in Table 24. Analysis of wheat hay and wheat straw extracts showed that the efficiency of extraction for the residue analytical method was at least 71% for parent and 81% for SYN546206 (free). Efficiency of extraction of SYN546039 from wheat hay and wheat straw was not verified and efficiency of hydrolysis to extract the SYN546049 conjugates from wheat hay and wheat straw was not verified either.

Table 24 Radiovalidation of primary extraction procedures used in analytical method GRM042.08A

Commodity	Components	Confined rotational crop study ^a 90 day PBI		Method GRM042.08A ^b		Efficiency %TRR method: confined study
		%TRR	Residue mg/k g eq	%TRR	Residue mg/k g eq	
Wheat hay	TRR ^c extract	100 ° 88	0.34 ° 0.30	100 ° 68	0.34 ° 0.23	– 77%
	parent	25	0.085	18	0.061	71%
	SYN546039	1.9—total 1.9—free ND—conj	0.006—total 0.006—free ND—conj	not verified	not verified	–
	SYN546206	14—total 11—free 2.6—conj	0.048—total 0.039—free 0.009—conj	– 9.2—free –	– 0.031—free –	– 81—free –
Wheat straw	TRR ^c extract	100 ° 87	0.33 ° 0.29	100 ° 66	0.33 ° 0.22	– 76%
	parent	18	0.059	14	0.047	81%

Commodity	Components	Confined rotational crop study ^a 90 day PBI		Method GRM042.08A ^b		Efficiency %TRR method: confined study
		%TRR	Residue mg/k g eq	%TRR	Residue mg/k g eq	
	SYN546039	5.8—total 1.9—free 3.9—conj	0.019—total 0.006—free 0.013—conj	not verified	not verified	
	SYN546206	15—total 14—free 1.3—conj	0.050—total 0.046—free 0.004—conj	— 14—free —	— 0.045—free —	— 99—free —

^a In the confined rotational crop study primary extraction of wheat hay and straw was by acetonitrile (1×), acetonitrile/water (4:1, v/v) (2–3×), acetonitrile/water (3:7, v/v) (1×), water (1×), acetonitrile (1×). Extracts were combined, concentrated, re-suspended in pH 2 water and partitioned with hexane/diethyl ether (1:3 v/v). The organic fraction contained the free metabolites. The aqueous fraction was subjected to hydrolysis by bovine rumen fluid (pH 7, 24 hrs at 39 °C, under nitrogen). This fraction contained the conjugates.

^b In method GRM042.08A primary extraction was by acetonitrile/water 4:1 v/v (1×).

Conclusion:

- Efficiency of extraction is at least 71% for parent and 81% for SYN546206 (free) as shown by a radio-validation study in wheat hay and wheat straw.
- Efficiency of extraction and hydrolysis for SYN546039 conjugates has been confirmed in a radio-validation study using method GRM042.04A, where the extraction and hydrolysis procedures are identical to those in method GRM042.08A.
- HPLC-MS-MS method GRM042.08A is considered valid for the determination of parent, SYN546039 (free/conjugated) and SYN546206 (free) in the range 0.01–0.1 mg/kg for spinach, carrot (roots, leaves) and wheat grain and 0.01–0.5 mg/kg for wheat straw and wheat forage.
- HPLC-MS-MS method GRM042.08A is considered valid for the determination of SYN546206 (free) at a single level of 0.2 mg/kg in spinach, wheat grain, wheat straw and potato tuber for the purpose of a storage stability study.
- Modification A of HPLC-MS-MS method GRM042.08A is considered valid for the determination of parent, SYN546206 (free) and SYN546039 (free/conjugated) in the range 0.01–0.1 mg/kg for spinach and wheat commodities (forage, hay, straw and grain), since it could be shown that extension of the original method with liquid-liquid partition and an SPE clean-up step did not introduce losses compared to the original method.
- Modification A of HPLC-MS-MS method GRM042.08A (or the original method) is considered insufficiently validated for the determination of parent, SYN546206 (free) and SYN546039 (free/conjugated) in lettuce, turnip (roots, tops) and radish (roots, tops) since only one recovery per level (0.01 and 0.1 mg/kg) is available for each commodity.

HPLC-MS-MS method GRM023.03A

HPLC-MS-MS method GRM023.03A, version 11 February 2010, [Hargreaves, 2010, SYN508210_10287] is intended for use as pre-registration method for the determination of SYN508272 (CSCC210616, free and conjugated) and SYN545720 (CSCD465008, free only) and seven other related chemicals in plant commodities. The original method or its modification was used in a storage stability study.

High water samples and grains (10 g) were extracted by homogenisation in 80/20 (v/v) acetonitrile/water. Straw and hay samples (5 g) were pre-soaked with 25 mL water for 30 min prior to homogenisation. Soya bean hay was extracted twice with 80/20 acetonitrile/water and extracts were combined. For the determination of SYN508272 (CSCC210616), separate aliquots of the initial extracts (equivalent to 0.2 g sample) were evaporated to remove the acetonitrile. The aliquots were

buffered to pH 5 with 0.2 M sodium acetate buffer and hydrolysed using driselase® at 37 °C (6–20 hrs) and cleaned-up through an SPE procedure using Oasis TM HLB cartridges. SYN508272 was eluted with acetonitrile. The acetonitrile was evaporated to dryness and the residue re-dissolved in water. For the determination of SYN545720 (CSCD465008), separate aliquots of the initial extracts (equivalent to 0.2 g) were evaporated to remove the acetonitrile and partitioned with iso-hexane. The aqueous phase was then acidified to pH 1 with 1 M HCl to ensure that SYN545720 is retained on the SPE cartridge and taken through an SPE clean-up procedure, using Oasis TM HLB cartridges. SYN545720 was eluted with 50/50 v/v water/acetonitrile. The eluate was evaporated to remove the acetonitrile and then diluted with water. SYN508272 and SYN545720 were analysed separately by HPLC-MS-MS using different solvent and ionisation systems. The primary transition monitored for SYN508272 was m/z 176→136 with confirmatory transition m/z 176→156. For SYN545720 the primary transition monitored was m/z 161→141 with confirmatory transition m/z 161→66. Samples with expected residues > 0.5 mg/kg need to be diluted before analysis.

The analysis conditions as specified in the method description need to be followed carefully. It is important that all the acetonitrile is removed from the sample prior to enzyme hydrolysis, since any remaining acetonitrile may adversely affect the efficiency of the hydrolysis. Change in enzyme hydrolysis conditions may result in either incomplete deconjugation or degradation of free metabolites. These considerations have been dealt with in the described analytical method.

HPLC-MS-MS Method GRM023.03A was validated [Klimmek and Gizler, 2009, SYN524464_11238] according to the full validation scheme for SYN508272 (CSCC210616) in wheat (grain, straw, whole plant), carrot roots, spinach, potatoes, whole oranges, oilseed rape seeds, dry lentils, tomatoes and maize kernels, using the driselase hydrolysis step as indicated the analytical method. Further the method was validated according to the full validation scheme for SYN545720 (CSCD465008) in whole oranges and dry soya bean seeds without addition of a pectinase hydrolysis step and for SYN545720 (CSCD465008) in potatoes, whole oranges, oil seed rape seeds, lentils, tomatoes, maize kernels and dry soya bean seeds with addition of a pectinase hydrolysis step (see note below). The validation was performed for the quantification transition ions as well as for the confirmation transition ions. Samples were fortified with a mixture of SYN508272 and SYN545720 (and seven other related chemicals) before extraction. The reported LOQ was 0.01 mg/kg for each commodity and each analyte. Average recoveries at 0.01 mg/kg and 0.1 mg/kg of SYN508272 and SYN545720 were within 70–120% limits and RSDs were within 20%. Linearity of detector response (determination coefficient $R^2 > 0.99$) was observed in the range 0.05–20 ng/mL using 6–8 single standards. This range is equivalent to 0.25–12× LOQ in the samples. Significant enhancement or suppression ($> \pm 10\%$) of detector response was observed for some of the matrices tested. Matrix matched standards were used to compensate for these effects observed in method validation. For the determination of SYN508272 (CSCC210616), matrix matched standards were used for wheat grain, wheat straw, orange, oilseed rape seed, lentils, tomatoes, maize kernels and soya bean seeds. For the determination of SYN545720 (CSCD465008), matrix matched standards were used in orange, oilseed rape seed, lentils, tomatoes, maize kernels and soya bean seeds. No interfering peaks $> 0.3\text{LOQ}$ (i.e. 0.003 mg/kg) were detected in any of the control extracts. The viability of individual batches of driselase® to cleave sugar conjugates was investigated by hydrolysing 2-Nitrophenyl β -D-glucopyranoside into 2-nitrophenol, causing a colour change from colourless to yellow/green.

Note: SYN545720 (CSCD465008) residues in crops may be present as conjugates and therefore a pectinase hydrolysis step was included during validation of the analytical method. For the analysis of SYN545720 (free + conjugated) aliquots (equivalent to 0.2 g) were evaporated to remove acetonitrile then buffered to pH 5 with sodium acetate buffer then hydrolysed using pectinase at 37 °C (16-20 hrs). Thereafter, the solution was partitioned with hexane and the aqueous phase was acidified to pH 1 with 1 M HCl and taken through a SPE clean-up procedure using Oasis TM HLB cartridges. SYN545720 was eluted with 50:50 v/v water:acetonitrile. The column eluates were evaporated to remove the acetonitrile and then diluted with water. During this method validation it was demonstrated in oranges and soya bean seed matrix, that the removal/addition of the pectinase hydrolysis step had no adverse effect on the recovery of free SYN545720. The analytical method as used in the storage stability studies did not include the pectinase step.

Additional validation data are available for the determination of SYN508272 and SYN545720 in whole oranges, dry soya bean seeds and dry broad beans [Klimmek *et al*, 2011, SYN524464_11446]. The analytical method included an enzyme hydrolysis step (driselase for SYN508272 and pectinase for SYN465008) as described in the validation report [Klimmek and Gizler, 2009, SYN524464_11238]. Samples were fortified separately with SYN508272 or SYN545720 before extraction. Average recoveries at 0.2 mg/kg of SYN508272 and SYN545720 were within 70–120% limits and RSDs were within 20%.

Conclusion:

- HPLC-MS-MS method GRM023.03A is considered valid for the determination of SYN508272 (free) in the range 0.01–0.1 mg/kg for wheat (grain, straw, whole plant), carrot roots, spinach, potatoes, whole oranges, oilseed rape seeds, dry lentils, tomatoes, maize kernels for the full analytical method including the driselase hydrolysis step.
- HPLC-MS-MS method GRM023.03A is considered valid for the determination of SYN545720 (free) in the range 0.01–0.1 mg/kg in potatoes, whole oranges, oil seed rape seeds, lentils, tomatoes, maize kernels and dry soya bean seeds. For whole oranges and dry soya bean seeds it has been shown that inclusion of a pectinase step does not have an effect on the recovery of the free SYN545720.
- HPLC-MS-MS method GRM023.03A is considered valid for the determination of SYN508272 (free) or SYN545720 (free) at the level of 0.2 mg/kg for whole oranges, dry soya beans and dry broad beans for the purpose of storage stability studies only.
- HPLC-MS-MS method GRM023.03A is considered not valid for the determination of conjugated SYN545720 and conjugated SYN508272, since the efficacy of the hydrolysis procedure has not been verified. However, since the method was only used in a storage stability study (free metabolites), this will have no impact on the results.

Analytical methods used in study reports in animal commodities

HPLC-MS-MS method GRM042.06A and Method 1887 version 2.0

HPLC-MS-MS Method GRM042.06A, version 27 January 2012, [Harris and Braid, 2012, SYN545192_10182] is identical to Method 1887 rev 2.0, version 22 September 2011, [Ward and Harris, 2011, SYN545192_10157]. The method is intended for use as pre-registration method for the determination of parent (free), SYN546039 (free and conjugates) and SYN546422 (free and conjugates) in animal commodities. A modification of the method was used in a cow feeding study.

Meat, liver, kidney, milk, and eggs (10 g) were homogenised with acetonitrile:water (80:20, v/v). Fat (10 g) was dissolved into n-hexane before liquid-liquid partitioning into acetonitrile:water (80:20, v/v). For the determination of parent, diluted aliquots of the extract were analysed directly by HPLC-MS-MS. For determination of SYN546039 and SYN546422, an aliquot of the extract was diluted with water prior to removal of the acetonitrile by evaporation. The remaining extract was then buffered with 0.4 M sodium acetate and conjugates of SYN546039 and SYN546422 were hydrolysed with β -glucuronidase by incubation at 37 °C and pH 5 for a minimum of 6 hours. After hydrolysis, the sample was diluted with acetonitrile:water (50:50, v/v), centrifuged and analysed by HPLC-MS-MS. The primary transition monitored for parent was m/z 396 $\rightarrow$ 91 with confirmatory transition m/z 396 $\rightarrow$ 368. For SYN546039 the primary transition monitored was m/z 412 $\rightarrow$ 91 with confirmatory transition m/z 412 $\rightarrow$ 340. The primary transition monitored for SYN546422 is m/z 430 $\rightarrow$ 364 with confirmatory transition m/z 430 $\rightarrow$ 394. Samples with expected residues > 0.1 mg/kg need to be diluted before analysis.

The analysis conditions as specified in the method description need to be followed carefully. To release the free metabolites of SYN546039 and SYN546422 a hydrolysis step is required. It is important that hydrolysis conditions are not changed to ensure efficient hydrolysis. The viability of the β -glucuronidase enzyme needs to be checked prior to use by hydrolysing phenolphthalein

glucuronide. Parent compound has been observed to adsorb to glass surfaces in solutions with < 50% organic solvent. These considerations have been dealt with in the described analytical method.

HPLC-MS-MS Analytical Method 1887 version 2.0 [Ward and Harris, 2011, SYN545192_10157] including the hydrolysis step was validated according to the full validation scheme for bovine meat, liver, kidney, fat, milk, and chicken eggs. The validation was performed for the quantification transition ions as well as for the confirmation transition ions. Samples were fortified with a mixture of SYN545192, SYN546039 and SYN546422 standard before extraction. The reported LOQ was 0.01 mg/kg for each commodity and each analyte. Average recoveries at 0.01 mg/kg and 0.1 mg/kg of parent or SYN546039 (free) or SYN546422 (free) were within 70–120% limits and RSDs were within 20%. Linearity of detector response (coefficient of determination $R^2 > 0.99$ for $1/\times$ weighted regression) was observed in the range of 0.15–10 ng/mL for parent and 0.06–4 ng/mL for SYN546039 and SYN546422 using six single standards. This range is equivalent to 0.3–20 $\times$ LOQ in the samples. The magnitude of the matrix effect was considered to be significant ($\geq 20\%$ suppression or enhancement) for parent, SYN546039 or SYN546422 in meat, liver, kidney or eggs. Matrix-matched calibration standards were used for all analytes in meat, liver and kidney and for SYN546039 and SYN546422 in eggs. Solvent standards were used for all analytes in fat and milk and for SYN545192 in eggs. No interfering peaks $> 0.3\text{LOQ}$ (i.e. 0.003 mg/kg) were detected in any of the control extracts. The viability of the β -glucuronidase enzyme was checked visually before use by observing a colour change from colourless to pink after hydrolysis of phenolphthalein glucuronide to phenolphthalein.

Modification A of HPLC-MS-MS method 1887 Version 2.0 was used in the cow feeding study and the storage stability study in animal commodities [Ward and Vance, 2012, SYN545192_10188]. During analysis of bovine tissues, a chromatographic peak was found that interfered with the analysis of SYN546039. The extraction and work-up procedure were unchanged; modifications were made to the HPLC-MS-MS system only. Samples were fortified with a mixture of parent, SYN546039 and SYN546422 standard before extraction. Additional validation data were available for eggs, whole milk, skimmed milk, cream, bovine muscle, liver, kidney and fat. Average recoveries at 0.01–0.2 mg/kg (milk, muscle and liver) or 0.01–0.1 mg/kg (skimmed milk, cream, kidney and fat) or 0.2 mg/kg (eggs) were within 70–120% limits and RSDs were within 20% ($n=2$ –16 per level). Linearity of detector response (coefficient of correlation $r > 0.99$, weighting factor $1/\times$) was observed in the range 0.15–10 ng/mL (parent) or 0.06–4.0 ng/mL (metabolites) using seven single solvent standards or matrix matched standards. No interfering peaks $> 0.3\text{LOQ}$ (i.e. 0.003 mg/kg) were detected in any of the control extracts.

The extraction and hydrolysis procedure used in method GRM042.06A was radio-validated using samples of milk, liver and muscle from the pyrazole labelled metabolism study in lactating goats [Green, 2012, SYN545192_10184] and samples of egg yolk from the pyrazole labelled metabolism study in laying hens [Lowrie and Kadow, 2012, SYN545192_10170]. An aliquot of milk (50 g), liver (25 g), muscle (25 g) and egg yolk (25 g) was extracted once with acetonitrile/water (80:20 v/v). An aliquot of the liver extract was evaporated to remove the acetonitrile. The remaining extract was then buffered with 0.4 M sodium acetate and conjugates were hydrolysed with β -glucuronidase by incubation at 37 °C and pH 5 for a minimum of 6 hours. After hydrolysis, the enzyme was denatured by addition of acetonitrile. Aliquots of the milk, liver, muscle and egg extracts and the liver hydrolysate were analysed by LSC and 2D-TLC.

Results are shown in Table 25. Good agreement was obtained between the levels of parent, SYN546039 (free including conjugates) and SYN546422 (free including conjugates) found after extraction (and hydrolysis) as performed in the metabolism study and as performed in method GRM042.06A.

Table 25 Radiovalidation of extraction procedures used in analytical method GRM042.06A

Commodity	Components	Metabolism Study ^a		Method GRM042.06A ^b		Trueness
		%TRR	Residue mg/kg eq	%TRR	Residue mg/kg eq	%TRR method: metabolism
Milk	TRR	100	0.034	100	0.038	–
	extract	100	0.034	96	0.036	96%
	PES	NA	NA	4.1	0.002	–
	parent	7.3	0.002	10	0.004	142%
	SYN546039	22	0.008	25	0.010	112%
	SYN546422	20	0.007	21	0.008	106%
Liver	TRR	100	0.70	100	0.71	–
	extract (hydrolysate)	78	0.54	73 (71)	0.52 (0.51)	94%
	PES	22	0.16	27	0.19	–
	parent	10	0.071	11	0.079	109%
	SYN546039 total	38	0.26	38	0.27	101%
	free	3.3	0.023	3.6	0.026	–
	conjugates	35	0.24	35	0.25	–
	SYN546422 total	6.0	0.042	4.0	0.029	67%
	free	0.4	0.003	ND	ND	–
	conjugates	5.6	0.039	4.0	0.029	–
Muscle	TRR	100	0.032	100	0.034	–
	extract	96	0.031	95	0.032	99%
	PES	3.7	0.001	4.9	0.002	–
	parent	25	0.008	28	0.009	113%
	SYN546039	39	0.012	42	0.014	107%
	SYN546422	8.9	0.003	8.8	0.003	99%
Egg yolk	TRR	100	0.18	100	0.18	–
	extract	83	0.15	75	0.13	90%
	PES	17	0.030	33	0.058	–
	parent	14	0.024	15	0.026	110%
	SYN546039	12	0.021	13	0.023	110%
	SYN546422	not verified	not verified	not verified	not verified	–

^a In the metabolism study liver and muscle were primary extracted sequentially with acetonitrile (1×), acetonitrile/water 4:1 v/v (1–4×), acetonitrile/water 3:7 v/v (1–2×), water (1×) and acetonitrile (1×). Liver extracts were subject to β -glucuronidase (pH 5, 37 °C, 18 hrs) and subsequently partitioned with ethyl acetate. Milk was primary extracted with dichloromethane (3×), ethyl acetate (2×) and acetone (1×). Egg yolk was primary extracted with dichloromethane (1×), acetonitrile (1×), acetonitrile/water 4:1 v/v (2–3×), acetonitrile/water 3:7 v/v (1–4×), water (1×) and acetonitrile (1×).

^b In method GRM042.06A primary extraction was by acetonitrile/water 4:1 v/v (1×). Extracts were then subjected to β -glucuronidase (pH 5, 37 °C, 6 hrs). In this experiment only liver extracts were subjected to β -glucuronidase; in the actual method, all matrices were β -glucuronidase.

Conclusion:

- The efficacy of the extraction procedure of parent and SYN546039 (free) is confirmed by a separate radio-validation experiment in muscle, liver, milk and eggs, while the efficacy of the extraction procedure of SYN546422 (free) is confirmed in muscle, liver and milk.
- The efficacy of the extraction and hydrolysis procedure of SYN546039 conjugates or SYN546422 conjugates is confirmed by a separate radio-validation experiment in bovine liver.
- HPLC-MS-MS method GRM042.06A or Analytical Method 1887 version 2.0 is considered valid for the determination of parent (free), SYN546039 (free/conjugated) and SYN546422 (free/conjugated) in the range 0.01–0.1 mg/kg for meat, liver, kidney, fat, milk and eggs.
- Modification A of HPLC-MS-MS method GRM042.06A is considered valid for the determination of parent (free), SYN546039 (free/conjugated) and SYN546422 (free/conjugated) in the range 0.01–0.2 mg/kg (milk, muscle, liver) or 0.01–0.1 mg/kg (skimmed milk, cream, kidney and fat) or 0.2 mg/kg (eggs).

Analytical methods used in study reports in soil and water

The Meeting received analytical methods descriptions and validations for the determination of parent and its metabolites in soil and water. Since soil or water samples were not analysed in the field rotational crop studies, analytical methods for soil and water were not summarized.

Stability of pesticide residues in stored analytical samples

The Meeting received information on storage stability of parent, SYN546039, SYN546206 and SYN545720 in raw and processed plant commodities and of parent, SYN546039 and SYN546422 in animal commodities.

Study 1

Storage stability was investigated by spiking various homogenised plant commodities with 0.20 mg/kg parent or SYN546039 or SYN546206 (one analyte per sample) [Watson, 2013, SYN545192_10137]. Samples were stored for 22–24 months at –18 °C and were analysed in duplicate at various intervals. Modification A of HPLC-MS-MS method GRM042.03A was used for quantification of parent and SYN546039 in spinach, orange, wheat grain, wheat straw and potato. Modification A of HPLC-MS-MS method GRM042.04A was used for quantification of parent and SYN546039 in dry soya bean seeds and dry broad bean seeds. HPLC-MS-MS method GRM042.08A was used for quantification of SYN546206 in spinach, wheat grain, wheat straw and potato. Methods GRM042.03A, GRM04.04A and GRM042.08A are considered valid for the purpose of this study (commodity type and concentration level of the analytes). Average concurrent recoveries were within 70–120% for each analyte and matrix and control samples had residues < 0.3LOQ (i.e. < 0.003 mg/kg), showing adequate performance at the time of analysis of the samples.

Storage stability results (not corrected for concurrent recovery) and concurrent recoveries are shown in Table 26 and 27. Residues of parent and SYN546039 (free) are stable for at least 24 months in crop commodities representative of the high water (spinach), high acid (orange), high starch (wheat grain and potato), high protein (dry broad bean seed), high oil (soya bean seed) commodity groups as well as in wheat straw when stored at or below –18 °C. Residues of SYN546206 are stable for at least 22 months in crop commodities representative of the high water (spinach) and high starch (wheat grain, potato) commodity groups as well as in wheat straw when stored at or below -18 °C.

Table 26 Storage stability of 0.20 mg/kg parent or SYN546039 in crop commodities stored at -18 °C

Matrix	Storage period (days)	parent mg/kg	parent Mean % remaining	parent Mean concurrent recovery (%)	SYN546039 mg/kg	SYN546039 Mean % remaining	SYN546039 Mean concurrent recovery (%)
spinach	0	0.20	100 ^a	109	0.20	100 ^a	102
	91	0.20	101	98	0.22	111	107
	186	0.18	94	101	0.19	98	94
	368	0.20	101	97	0.20	99	98
	550	0.20	102	104	0.19	97	104
	734	0.22	110	98	0.21	105	97
orange	0	0.21	100 ^a	101	0.19	100 ^a	100
	91	0.20	96	97	0.20	102	91
	186	0.19	94	101	0.19	99	98
	368	0.19	94	102	0.19	100	99
	550	0.20	98	103	0.21	106	105
	734	0.20	99	100	0.21	106	99
wheat grain	0	0.20	100 ^a	104	0.21	100 ^a	107
	91	0.19	94	103	0.16	77	78
	186	0.20	98	101	0.20	99	105
	368	0.20	100	103	0.20	98	103
	549	0.20	99	106	0.20	96	106
	733	0.20	101	106	0.22	106	103

Matrix	Storage period (days)	parent mg/kg	parent Mean % remaining	parent Mean concurrent recovery (%)	SYN546039 mg/kg	SYN546039 Mean % remaining	SYN546039 Mean concurrent recovery (%)
wheat straw	0	0.19	100 ^a	93	0.19	100 ^a	96
	91	0.19	103	92	0.15	80	74
	186	0.20	106	95	0.18	95	95
	368	0.22	115	98	0.20	103	100
	549	0.22	115	101	0.20	104	98
	733	0.23	120	105	0.21	112	100
potato	0	0.19	100 ^a	106	0.20	100 ^a	99
	91	0.19	100	99	0.19	94	99
	186	0.18	96	100	0.19	98	104
	368	0.19	99	99	0.19	95	97
	550	0.20	103	102	0.20	100	105
	734	0.20	107	102	0.21	104	103
dry soya bean seed	0	0.19	100 ^a	100	0.20	100 ^a	108
	91	0.19	100	101	0.19	97	95
	184	0.15	81	83	0.17	87	93
	366	0.18	96	84	0.21	106	103
	548	0.20	105	106	0.20	102	105
	730	0.17	89	82	0.18	90	97
dry broad bean seed	0	0.20	100 ^a	100	0.19	100 ^a	99
	91	0.20	101	107	0.20	106	99
	184	0.21	105	107	0.20	105	97
	366	0.21	105	99	0.19	102	98
	548	0.21	105	102	0.21	108	102
	730	0.22	108	104	0.21	110	102

^a The % remaining was set at 100% at time zero. If 0.16 mg/kg was found at time zero, instead of the expected 0.20 mg/kg, the 0.16 mg/kg was set at 100%, to see the development of this residue level in time.

Table 27 Storage stability of 0.20 mg/kg SYN546206 in crop commodities stored at -18 °C

Matrix	Nominal storage period (months)	SYN546206 mg/kg	SYN546206 Mean % remaining	SYN546206 Mean concurrent recovery (%)
spinach	0	0.18	100 ^a	94
	93	0.19	110	100
	183	0.20	113	101
	366	0.18	103	93
	554	0.18	102	99
	672	0.21	120	106
wheat grain	0	0.19	100 ^a	97
	93	0.19	98	101
	183	0.18	93	100
	366	0.18	92	101
	554	0.18	90	99
	672	0.20	102	107
wheat straw	0	0.19	100 ^a	107
	92	0.21	107	107
	182	0.20	102	95
	365	0.18	93	100
	553	0.18	94	95
	671	0.21	110	107
potato	0	0.19	100 ^a	98
	92	0.19	101	98
	182	0.19	98	99
	365	0.19	96	99

Matrix	Nominal storage period (months)	SYN546206 mg/kg	SYN546206 Mean % remaining	SYN546206 Mean concurrent recovery (%)
	553	0.19	97	102
	671	0.21	110	106

^a The % remaining was set at 100% at time zero. If 0.16 mg/kg was found at time zero, instead of the expected 0.20 mg/kg, the 0.16 mg/kg was set at 100%, to see the development of this residue level in time.

Study 2

Storage stability was investigated by spiking various homogenised plant commodities with 0.20 mg/kg SYN508272 and SYN545720 [Klimmek *et al*, 2011, SYN524464_11446]. Samples were stored for 24 months at -18 °C and were analysed in duplicate at various intervals using HPLC-MS-MS method GRM023.03A. The analytical method included an enzyme hydrolysis step (driselase for SYN508272 and pectinase for SYN465008) as described in the validation report of the method [Klimmek and Gizler, 2009, SYN524464_11238]. Method GRM023.03A is considered valid for the purpose of this study (commodity type and concentration level of the analytes). Average concurrent recoveries were within 70–120% for each analyte and matrix and control samples had residues < 0.3LOQ (i.e. < 0.003 mg/kg), showing adequate performance at the time of analysis of the samples.

Storage stability results (not corrected for concurrent recovery) and concurrent recoveries are shown in Table 28. Residues of SYN508272 (free) and SYN545720 (free) are stable for at least 24 months in crop commodities representative of the high oil (soya bean seeds), high acid (orange) and high protein (dry broad bean seeds) commodity groups when stored at or below -18 °C.

Note: The study report describes storage stability for a set of seven related chemicals. SYN508272 (CSCC210616) and SYN545720 (CSCD465008) were added as a single compound to separate samples. The other related chemicals were added to other samples. Addition of these chemicals did therefore not affect the storage stability results of SYN508272 or SYN545720.

Table 28 Storage stability of 0.20 mg/kg SYN545720 or SYN508272 in crop commodities stored frozen at -18 °C

Matrix	Storage period (days)	SYN545720 mg/kg	mean % remaining	Mean procedural recovery (%)	SYN508272 mg/kg	mean % remaining	Mean procedural recovery (%)
orange	0–1	0.21	100 ^a		0.15	100 ^a	
	90	0.20	95	96	0.17	113	81
	181	0.19	91	85	0.18	118	98
	365	0.20	96	100	0.20	132	96
	540–559	0.19	92	74	0.17	113	90
dry soya bean seed	735	0.18	87	90	0.21	135	102
	0–4	0.17	100 ^a		0.21	100 ^a	
	90	0.16	93	94	0.17	81	84
	183	0.20	113	92	0.20	95	102
	369	0.21	122	103	0.19	90	102
	540	0.14	80	78	0.18	85	81
	743	0.17	98	86	0.18	89	86
	0–5	0.16	100 ^a		0.18	100 ^a	
	89	0.20	124	95	0.17	95	88
	180	0.17	106	94	0.21	114	102
dry broad bean seed	198	–	–	–	0.24	132	118
	364	0.20	123	92	0.20	109	105

Matrix	Storage period (days)	SYN545720 mg/kg	mean % remaining	Mean procedural recovery (%)	SYN508272 mg/kg	mean % remaining	Mean procedural recovery (%)
	539	0.16	101	101	0.18	97	86
	734	0.18	112	88	0.21	115	109

^a The % remaining was set at 100% at time zero. If 0.16 mg/kg was found at time zero, instead of the expected 0.20 mg/kg, the 0.16 mg/kg was set at 100%, to see the development of this residue level in time.

Study 3

Storage stability was investigated by spiking homogenised processed plant commodities with 0.20 mg/kg parent or SYN546039 or SYN545720 (one analyte per sample) [Hagan and Bertrand, 2012/2013, SYN545192_50177, SYN545192_50544]. The SYN545720 compound was only added to processed soya bean commodities. Samples were stored for 24 months at –10 °C and were analysed in duplicate at various intervals. Modification B of HPLC-MS-MS method GRM042.03A was used for the quantification of parent and SYN546039 in maize flour and maize meal, raisins, dried apple and apple juice. Modification B of HPLC-MS-MS method GRM042.04A was used for the quantification of parent, SYN546039 and SYN545720 in maize oil, soya bean flour, soya bean milk and soya bean oil. Methods GRM042.03 and GRM042.04A are considered valid for the purpose of this study (commodity type and concentration level of the analytes). Average concurrent recoveries were within 70–120% for each analyte and matrix. Levels in control samples were < 0.04 mg/kg, but since the fortification levels are 0.2 mg/kg, this is considered to have no impact on the study results. Therefore, performance is considered adequate at the time of analysis of the samples.

Storage stability results (not corrected for concurrent recovery) and concurrent recoveries are shown in Tables 29 and 30. Residues of parent, SYN546039 (free) and SYN545720 (free) are stable for at least 24 months in crop commodities various processed crop commodities (flour, meal, oil, soymilk, dried fruit, fruit juice) when stored at or below –10 °C.

Table 29 Storage stability of 0.20 mg/kg parent or SYN546039 in processed crop commodities stored at -10 °C

Matrix	Storage period (days)	parent mg/kg	parent Mean % remaining	parent Mean concurrent recovery (%)	SYN546039 mg/kg	SYN546039 Mean % remaining	SYN546039 Mean concurrent recovery (%)
Maize flour	0	0.23	100 ^a	103	0.22	100 ^a	104
	93	0.23	100	119	0.21	93	110
	179	0.20	85	101	0.20	89	104
	274	0.17	72	98	0.17	77	91
	375	0.22	93	110	0.15	66	83
	554	0.21	89	102	0.19	84	92
	731	0.23	98	113	0.21	93	103
Maize meal	0	0.22	100 ^a	109	0.21	100 ^a	101
	93	0.23	105	114	0.20	96	109
	179	0.19	86	99	0.18	89	93
	274	0.16	76	94	0.16	76	88
	362	0.18	81	104	0.15	71	80
	555	0.21	96	102	0.18	87	101
	731	0.17	79	103	0.20	97	101
Maize refined oil	0	0.23	100 ^a	113	0.22	100 ^a	111
	93	0.18	79	100	0.19	86	104
	181	0.19	81	101	0.17	76	92
	275	0.16	67	78	0.16	69	81
	366	0.21	89	93	0.15	68	77
	554	0.20	88	116	0.22	96	99
	734	0.17	72	79	0.18	81	99

Matrix	Storage period (days)	parent mg/kg	parent Mean % remaining	parent Mean concurrent recovery (%)	SYN546039 mg/kg	SYN546039 Mean % remaining	SYN546039 Mean concurrent recovery (%)
Soya bean flour	0	0.21	100 ^a	102	0.21	100 ^a	91
	94	0.22	107	93	0.19	90	84
	181	0.16	76	80	0.17	81	81
	277	0.18	83	81	0.17	83	75
	371	0.21	98	100	0.18	88	86
	554	0.21	100	99	0.22	105	107
	732	0.19	89	86	0.16	77	79
Soya bean milk	0	0.20	100 ^a	92	0.20	100 ^a	110
	92	0.19	96	95	0.19	93	95
	180	0.18	92	98	0.18	86	102
	273	0.17	86	84	0.16	79	97
	364	0.22	109	104	0.17	85	80
	558	0.18	93	83	0.20	95	102
	730	0.20	101	97	0.19	94	99
Soya bean crude oil	0	0.19	100 ^a	89	0.17	100 ^a	92
	95	0.20	106	98	0.16	96	87
	181	0.17	90	97	0.17	100	90
	277	0.19	97	104	0.17	102	86
	372	0.22	114	112	0.16	96	82
	558	0.18	96	92	0.16	97	101
	731	0.22	114	101	0.19	111	99
Grape raisins	0	0.21	100 ^a	116	0.23	100 ^a	118
	93	0.22	104	99	0.22	94	119
	181	0.21	98	113	0.21	92	112
	273	0.20	93	116	0.20	85	108
	361	0.24	115	128	0.21	88	119
	560	0.23	107	115	0.21	90	111
	734	0.24	112	127	0.23	100	118
Apple dried	0	0.22	100 ^a	107	0.23	100 ^a	111
	97	0.23	102	114	0.23	101	120
	180	0.22	96	105	0.23	98	116
	272	0.19	85	114	0.18	80	107
	365	0.23	102	125	0.17	73	90
	564	0.21	92	114	0.22	93	120
	739	0.23	102	119	0.23	99	114
Apple juice	0 ^b	–	–	–	–	–	–
	97	0.22	110	120	0.23	117	113
	180	0.19	96	103	0.20	100	100
	272	0.15	76	88	0.16	80	99
	365	0.18	90	112	0.14	71	86
	557	0.20	98	105	0.19	93	103
	734	0.22	112	111	0.21	103	107

^a The % remaining was set at 100% at time zero. If 0.16 mg/kg was found at time zero, instead of the expected 0.20 mg/kg, the 0.16 mg/kg was set at 100%, to see the development of this residue level in time.

^b Zero day sample is missing, % remaining based on nominal fortification level of 0.20 mg/kg

Table 30 Storage stability of 0.20 mg/kg SYN545720 in crop commodities stored at -10 °C

Matrix	Storage period (days)	SYN545720 mg/kg	SYN545720 mean % remaining	Mean concurrent recovery (%)
soya bean flour	0	0.18	100 ^a	89
	94	0.22	122	95
	181	0.15	84	86

Matrix	Storage period (days)	SYN545720 mg/kg	SYN545720 mean % remaining	Mean concurrent recovery (%)
	277	0.17	93	85
	371	0.18	100	87
	554	0.18	100	86
	732	0.16	89	81
soya bean milk	0	0.15	100 ^a	78
	92	0.19	127	96
	180	0.18	122	94
	273	0.16	104	73
	364	0.18	120	101
	558	0.16	103	102
	730	0.20	129	93
soya bean crude oil	0	0.19	100 ^a	104
	95	0.17	89	92
	181	0.16	85	95
	277	0.16	84	80
	372	0.19	100	109
	558	0.20	106	104
	731	0.20	106	89

^a The % remaining was set at 100% at time zero. If 0.16 mg/kg was found at time zero, instead of the expected 0.20 mg/kg, the 0.16 mg/kg was set at 100%, to see the development of this residue level in time.

Study 4

Storage stability was investigated by spiking various animal commodities with 0.20 mg/kg parent, SYN546039 or SYN546422 (one analyte per sample) [Ward and Vance, 2012, SYN545192_10188]. Samples for concurrent recoveries were spiked with a mixture of these three analytes. Samples were stored for 56–78 days at –20 °C and were analysed in duplicate using modification A of HPLC-MS-MS method 1887 (see method GRM042.06). The modification of Method 1887 is considered valid for the purpose of this study (commodity type and concentration level of the analytes). Average concurrent recoveries were within 70–120% for each analyte and matrix and control samples had residues < 0.3LOQ (i.e. < 0.003 mg/kg), showing adequate performance at the time of analysis of the samples.

Storage stability results (not corrected for concurrent recovery) and concurrent recoveries are shown in Table 31. Residues of SYN545192, SYN546039 and SYN546422 are stable in milk stored frozen for at least 62 days, in eggs for at least 56 days, in liver for at least 78 days and in muscle for at least 76 days. According to OECD Guideline 506 storage stability in kidney and fat may be extrapolated from the other animal tissues.

Table 31 Storage stability of 0.20 mg/kg parent, SYN546039 and SYN546422 in animal commodities stored at –20 °C

Analyte	Matrix	Storage period (days)	Mean residue (mg/kg)	Mean % remaining	Mean concurrent recovery (%)
parent	milk	0	0.18	100 ^a	89
		62	0.21	119	94
SYN546039	milk	0	0.22	100 ^a	111
		62	0.19	86	88
SYN546422	milk	0	0.24	100 ^a	119
		62	0.19	77	83
parent	egg	0	0.20	100 ^a	100
		56	0.22	108	109
SYN546039	egg	0	0.19	100 ^a	94
		56	0.22	115	104
SYN546422	egg	0	0.18	100 ^a	90
		56	0.19	103	98
parent	muscle	0	0.20	100 ^a	100
		76	0.21	105	109

Analyte	Matrix	Storage period (days)	Mean residue (mg/kg)	Mean % remaining	Mean concurrent recovery (%)
SYN546039	muscle	0	0.19	100 ^a	96
		76	0.17	88	86
SYN546422	muscle	0	0.19	100 ^a	93
		76	0.16	83	85
parent	liver	0	0.17	100 ^a	87
		56	0.21	118	100
		78	0.21	118	110
SYN546039	liver	0	0.21	100 ^a	106
		56	0.19	87	102
		78	0.19	87	93
SYN546422	liver	0	0.19	100 ^a	97
		56	0.17	85	98
		78	0.16	80	94

^a The % remaining was set at 100% at time zero. If 0.16 mg/kg was found at time zero, instead of the expected 0.20 mg/kg, the 0.16 mg/kg was set at 100%, to see the development of this residue level in time.

USE PATTERN

Benzovindiflupyr is a broad-spectrum fungicide belonging to the chemical class of pyrazole carboxamides (like bixafen, fluxapyroxad, furametpyr, isopyrazam, penflufen, penthiopyrad, sedaxane). Benzovindiflupyr acts as an inhibitor of the fungal complex II respiratory chain, where it inhibits the succinate dehydrogenase enzyme (succinate dehydrogenase inhibitor, SDHI fungicide) by blocking the ubiquinone-binding sites in the mitochondrial complex. Benzovindiflupyr is a broad-spectrum fungicide, especially used for its effect against rust. [Label information and Mayer, 2012, A17056B_50013].

Benzovindiflupyr is a registered fungicide in soya beans only and only in Paraguay (Table 32). The original registered labels were submitted in the original language as well as in its English translation.

Benzovindiflupyr registrations are intended in the USA in 2014 for pome fruit, blueberries, grapes, fruiting vegetables (including cucurbits and sweet corn), root and tuber vegetables, pulses (dry beans, dry peas and soya beans), cereals (barley, maize and wheat), oilseeds (cottonseed; peanuts and rapeseed), sugarcane and coffee beans. At the time of this evaluation, the respective labels were not authorised and consequently the residue trials were not submitted.

Table 32 Registered pre-harvest uses of benzovindiflupyr

Crop	Country	Form (g ai/kg)	Application				PHI, days
			Method	Rate kg ai/ha	Spray volume	Number (interval in days)	
Soya bean	Paraguay	WG 150 ^{a, b}	Foliar spray	3 × 0.045	100–200 L/ha	3 (14)	21

^a water dispersible granule containing 150 g/kg benzovindiflupyr and 300 g/kg azoxystrobin.

^b The product must be used with a paraffinic mineral oil, used as an adjuvant. The recommendation is to add the adjuvant to the water volume together with the formulated product for benzovindiflupyr [Syngenta, 2014b].

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on supervised residue trials of benzovindiflupyr for soya beans only. Application rates and spray concentrations have been rounded to two significant figures; residues at around the LOQ or 0.01 mg/kg have been rounded to one figure. Residue data are recorded unadjusted for percentage recoveries or for residue values in control samples unless otherwise stated. Unquantifiable residues are shown as below the reported LOQ (e.g. < 0.01 mg/kg). Where multiple samples were taken from a single plot or where multiple analyses were conducted on a single sample, the average value is reported. Where results from separate plots with distinguishing characteristics such as different formulations, crop varieties or treatment schedules were reported, results are listed

separately for each plot, but only one value is selected for MRL derivation. Residues from the trials conducted according to critical GAP have been used for the estimation of maximum residue levels, STMR and HR values. Those results are underlined.

Soya beans

Supervised residue field trials were conducted on soya beans in the 2010–2011 season in Brazil [Casallanova, 2011a/b/c, A17961A_10022; A17056F_10022; A18126B_10032]. Soy plants were treated with an EC formulation containing 150 g ai/L benzovindiflupyr alone (report M11063) or 50 g ai/L benzovindiflupyr in combination with 100 g ai/L azoxystrobin (report M11075) or with a WG formulation containing 150 g ai/kg benzovindiflupyr in combination with 300 g ai/kg azoxystrobin (report M11086). Small plots of soya beans (60–90 m²) were treated as indicated in Table 33 using boom sprayers with a spray volume of 200 L/ha. All treatments included Nimbus adjuvant at 600 mL/ha.

Soya bean plants were harvested at maturity at three time points after the final application with growth stages BBCH 85–97, 86–97 and 87–97. Soya bean plants were threshed immediately to separate the seeds and the rest of plant. Samples were collected from at least 12 different points along the plot reaching at least 2 kg of soya bean seeds after threshing. Sample sizes were in accordance with appendix V of the FAO manual. All samples were stored frozen for 15–73 days at –20 °C. This storage period is covered by the storage stability studies on parent, SYN546039 and SYN545720 in commodities with high oil content.

Samples were analysed for parent and its metabolites SYN546039 and SYN545720 using HPLC-MS-MS method POPIT MET.134 (see GRM042.04A). The analytical method is considered valid in the range 0.01–0.1 mg/kg for the determination of parent, free and/or conjugated SYN546039 and free and/or conjugated SYN545720 in dry soya bean seed. Average concurrent recoveries were within 70–120% for each analyte and control samples had residues < LOQ (i.e. < 0.01 mg/kg), showing adequate performance at the time of analysis of the samples.

Residues of benzovindiflupyr and its metabolites SYN546039 and SYN545720 from the trials are summarised in Table 33.

Table 33 Residues in soya bean seeds after foliar spray applications in the field

SOYA BEAN SEEDS Location, year, (Variety) Soil type	Form	No (Interv al days)	kg ai/ha	kg ai/hL	GS, date (last appl)	DAT	parent mg/kg	SYN 54603 9 mg/kg	SYN 54572 0 mg/kg	Report; Trial; (remarks)
Holambra, SP, Brazil, 2010–2011, (CD 214 RR) Soil: Sa/Si/Cl= 52.0/12.5/35.5	EC ^a	3 (19,14)	0.030 0.030 0.030	0.015 0.015 0.015	BBCH 76–78, 30-Mar	21 28 35	< 0.01 0.02 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11063; M11063- DMO1 I
Holambra, SP, Brazil, 2010–2011, (CD 214 RR) Soil: Sa/Si/Cl= 52.0/12.5/35.5	EC ^a	3 (19,14)	0.045 0.045 0.045	0.022 0.022 0.022	BBCH 76–78; 30-Mar	21 28 35	< 0.01 < 0.01 0.02	< 0.01 < 0.01 < 0.01	< 0.01 0.01 0.01	M11063; M11063- DMO1 c
Holambra, SP, Brazil, 2010–2011, (CD214 RR) Soil: Sa/Si/Cl= 52.0/12.5/35.5	EC ^{a, b}	3 (19,14)	0.030 0.030 0.030	0.015 0.015 0.015	BBCH 76–78, 30-Mar	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11075; M11075- DMO3 c
Holambra, SP, Brazil, 2010–2011, (CD214 RR)	EC ^{a, b}	3 (19,14)	0.050 0.050 0.050	0.025 0.025 0.025	BBCH 76–78, 30-Mar	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11075; M11075- DMO3

SOYA BEAN SEEDS Location, year, (Variety) Soil type	Form	No (Interv al days)	kg ai/ha	kg ai/hL	GS, date (last appl)	DAT	parent mg/kg	SYN 54603 9 mg/kg	SYN 54572 0 mg/kg	Report; Trial; (remarks)
Soil: Sa/Si/Cl= 52.0/12.5/35.5										c
Holambra, SP, Brazil, 2010–2011, (CD214 RR) Soil: Sa/Si/Cl= 52.0/12.5/35.5	WG ^a , _b	3 (19,14)	0.030 0.030 0.030	0.015 0.015 0.015	BBCH 76–78, 30-Mar	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11086; M11086- DMO1 c
Holambra, SP, Brazil, 2010–2011, (CD214 RR) Soil: Sa/Si/Cl= 52.0/12.5/35.5	WG ^a , _b	3 (19,14)	0.045 0.045 0.045	0.022 0.022 0.022	BBCH 76–78, 30-Mar	21 28 35	0.03 < 0.01 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11086; M11086- DMO1 c
Bandeirantes, PR, Brazil, 2010–2011, (BMX Potencia RR) Soil: Sa/Si/Cl= 2.0/18.0/80.0	EC ^a	3 (22,14)	0.030 0.030 0.030	0.015 0.015 0.015	BBCH 79, 1-Mar	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11063; M11063- DMO2
Bandeirantes, PR, Brazil, 2010–2011, (BMX Potencia RR) Soil: Sa/Si/Cl= 2.0/18.0/80.0	EC ^a	3 (22,14)	0.045 0.045 0.045	0.022 0.022 0.022	BBCH 79, 1-Mar	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11063; M11063- DMO2
Bandeirantes, PR, Brazil, 2010–2011, (BMX Potencia RR) Soil: Sa/Si/Cl= 2.0/18.0/80.0	EC ^{a, b}	3 (22,14)	0.030 0.030 0.030	0.015 0.015 0.015	BBCH 79, 1-Mar	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11075; M11075- DMO2
Bandeirantes, PR, Brazil, 2010–2011, (BMX Potencia RR) Soil: Sa/Si/Cl= 2.0/18.0/80.0	EC ^{a, b}	3 (22,14)	0.050 0.050 0.050	0.025 0.025 0.025	BBCH 79, 1-Mar	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11075; M11075- DMO2
Bandeirantes, PR, Brazil, 2010–2011, (BMX Potencia RR) Soil: Sa/Si/Cl= 2.0/18.0/80.0	WG ^a , _b	3 (22,14)	0.030 0.030 0.030	0.015 0.015 0.015	BBCH 79, 1-Mar	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11086; M11086- DMO2
Bandeirantes, PR, Brazil, 2010–2011, (BMX Potencia RR) Soil: Sa/Si/Cl= 2.0/18.0/80.0	WG ^a , _b	3 (22,14)	0.045 0.045 0.045	0.022 0.022 0.022	BBCH 79, 1-Mar	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11086; M11086- DMO2
Uberlândia, MG, Brazil, 2010–2011, (SYN 9070 RR)	EC ^a	3 (22,14)	0.030 0.030 0.030	0.015 0.015 0.015	BBCH 80, 9-Mar	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11063; M11063- JJB1

SOYA BEAN SEEDS Location, year, (Variety) Soil type	Form	No (Interv al days)	kg ai/ha	kg ai/hL	GS, date (last appl)	DAT	parent mg/kg	SYN 54603 9 mg/kg	SYN 54572 0 mg/kg	Report; Trial; (remarks)
Soil: Sa/Si/Cl= 15.1/15.5/69.3										
Uberlândia, MG, Brazil, 2010–2011, (SYN 9070 RR) Soil: Sa/Si/Cl= 15.1/15.5/69.3	EC ^a	3 (22,14)	0.045 0.045 0.045	0.022 0.022 0.022	BBCH 80, 9-Mar	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11063; M11063- JJB1
Uberlândia, MG, Brazil, 2010–2011, (SYN 9070 RR) Soil: Sa/Si/Cl= 15.1/15.5/69.3	EC ^{a, b}	3 (20,14)	0.030 0.030 0.030	0.015 0.015 0.015	BBCH 80, 9-Mar	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11075; M11075- JJB1
Uberlândia, MG, Brazil, 2010–2011, (SYN 9070 RR) Soil: Sa/Si/Cl= 15.1/15.5/69.3	EC ^{a, b}	3 (20,14)	0.050 0.050 0.050	0.025 0.025 0.025	BBCH 80, 9-Mar	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11075; M11075- JJB1
Uberlândia, MG, Brazil, 2010–2011, (SYN 9070 RR) Soil: Sa/Si/Cl= 15.1/15.5/69.3	WG ^{a, b}	3 (22,14)	0.030 0.030 0.030	0.015 0.015 0.015	BBCH 80, 9-Mar	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11086; M11086- JJB1
Uberlândia, MG, Brazil, 2010–2011, (SYN 9070 RR) Soil: Sa/Si/Cl= 15.1/15.5/69.3	WG ^{a, b}	3 (22,14)	0.045 0.045 0.045	0.022 0.022 0.022	BBCH 80, 9-Mar	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11086; M11086- JJB1
Engenheiro Coelho, SP, Brazil, 2010–2011, (Vallosa) Soil: Sa/Si/Cl= 58.0/8.5/33.5	EC ^a	3 (59,14)	0.030 0.030 0.030	0.015 0.015 0.015	BBCH 73–74, 25-Apr	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11063; M11063- AMA
Engenheiro Coelho, SP, Brazil, 2010–2011 (Valosa) Soil: Sa/Si/Cl= 58.0/8.5/33.5	EC ^a	3 (59,14)	0.045 0.045 0.045	0.022 0.022 0.022	BBCH 73–74, 25-Apr	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11063; M11063- AMA
Engenheiro Coelho, SP, Brazil, 2010–2011, (Valiosa) Soil: Sa/Si/Cl= 44.0/13.8/42.2	EC ^{a, b}	3 (59,14)	0.030 0.030 0.030	0.015 0.015 0.015	BBCH 73–74, 25-Apr	14 21 28	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11075; M11075- AMA
Engenheiro Coelho, SP, Brazil, 2010–2011, (Valiosa) Soil: Sa/Si/Cl= 44.0/13.8/42.2	EC ^{a, b}	3 (59,14)	0.050 0.050 0.050	0.025 0.025 0.025	BBCH 73–74, 25-Apr	14 21 28	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11075; M11075- AMA

SOYA BEAN SEEDS Location, year, (Variety) Soil type	Form	No (Interv al days)	kg ai/ha	kg ai/hL	GS, date (last appl)	DAT	parent mg/kg	SYN 54603 9 mg/kg	SYN 54572 0 mg/kg	Report; Trial; (remarks)
Engenheiro Coelho, SP, Brazil, 2010–2011 (Valiosa) Soil: Sa/Si/Cl= 44.0/13.8/42.2	WG ^a , _b	3 (59,14)	0.030 0.030 0.030	0.015 0.015 0.015	BBCH 73–74, 25-Apr	14 21 28	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11086; M11086- AMA
Engenheiro Coelho, SP, Brazil, 2010–2011, (Valiosa) Soil: Sa/Si/Cl= 44.0/13.8/42.2	WG ^a , _b	3 (59,14)	0.045 0.045 0.045	0.022 0.022 0.022	BBCH 73–74, 25-Apr	14 21 28	< 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11086; M11086- AMA
Rio Verde, GO, Brazil, 2010–2011, (Valiosa) Soil: Sa/Si/Cl= 42.5/8.8/48.7	EC ^a , _b	3 (23,14)	0.030 0.030 0.030	0.015 0.015 0.015	BBCH 79, 24-Febr	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11075; M11075- JJB2 _c
Rio Verde, GO, Brazil, 2010–2011, (Valiosa) Soil: Sa/Si/Cl= 42.5/8.8/48.7	EC ^a , _b	3 (23,14)	0.050 0.050 0.050	0.025 0.025 0.025	BBCH 79, 24-Febr	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11075; M11075- JJB2 _c
Rio Verde, GO, Brazil, 2010–2011, (Valiosa) Soil: Sa/Si/Cl= 42.5/8.8/48.7	WG ^a , _b	3 (23,14)	0.030 0.030 0.030	0.015 0.015 0.015	BBCH 79, 24-Febr	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11086; M11086- JJB2 _c
Rio Verde, GO, Brazil 2010–2011 (Valiosa) Soil: Sa/Si/Cl= 42.5/8.8/48.7	WG ^a , _b	3 (23,14)	0.045 0.045 0.045	0.022 0.022 0.022	BBCH 79, 24-Febr	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11086; M11086- JJB2 _c
Rondonópolis, MT, Brazil, 2010–2011, (TMG 132) Soil: Sa/Si/Cl= 61.0/0.1/39.0	EC ^a , _b	3 (23,14)	0.030 0.030 0.030	0.015 0.015 0.015	BBCH 79, 24- Febr	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11075; M11075- JJB3 _c
Rondonópolis MT, Brazil, 2010–2011, (TMG 132) Soil: Sa/Si/Cl= 61.0/0.1/39.0	EC ^a , _b	3 (23,14)	0.050 0.050 0.050	0.025 0.025 0.025	BBCH 79, 24-Febr	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11075; M11075- JJB3
Rondonópolis, MT, Brazil, 2010–2011 (TMG 132) Soil: Sa/Si/Cl= 61.0/0.1/39.0	WG ^a , _b	3 (23,14)	0.030 0.030 0.030	0.015 0.015 0.015	BBCH 79, 24- Febr	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11086; M11086- JJB3 _c
Rondonópolis, MT, Brazil, 2010–2011, (TMG 132)	WG ^a , _b	3 (23,14)	0.045 0.045 0.045	0.022 0.022 0.022	BBCH 79, 24-Febr	21 28 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	M11086; M11086- JJB3

SOYA BEAN SEEDS Location, year, (Variety) Soil type	Form	No (Interv al days)	kg ai/ha	kg ai/hL	GS, date (last appl)	DAT	parent mg/kg	SYN 54603 9 mg/kg	SYN 54572 0 mg/kg	Report; Trial; (remarks)
Soil: Sa/Si/Cl= 61.0/0.1/39.0										^c

Brazilian States: SP Sao Paulo, GO Goiás, MG Minas Gerais, PA Parana

^a 600 mL/ha of Nimbus adjuvant added to the spray solution. Nimbus is a paraffinic mineral oil, used as an adjuvant [Syngenta, 2014b]

^b The formulation also contained azoxystrobin. The treatment of 0.030/0.045/0.050 kg ai/ha for benzovindiflupyr resulted in 0.060/0.090/0.100 kg ai/ha for azoxystrobin. All benzovindiflupyr treatments were preceded by a single treatment with azoxystrobin at 0.050 kg ai/ha.

^c Rainfall within 24 hrs after the last application: 16 mm in in Holambra, 10 mm in Rio Verde and 0.4 mm in Rondonopolis. Given the high residues found in Holambra, trials can be selected for MRL derivation.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

No data submitted.

In processing

The Meeting received information on the nature of residues under conditions simulating pasteurisation, baking/brewing/boiling and sterilisation. In addition the Meeting received processing studies on soya beans.

Study 1

The behaviour of benzovindiflupyr was studied under conditions simulating pasteurisation, baking/brewing/boiling and sterilisation [Simmonds, 2011, SYN545192_10162]. Duplicate solutions of 1.00–1.01 mg/L [pyrazole-5-¹⁴C]-benzovindiflupyr were prepared in 0.1 M sterile citrate buffers at pH 4, 5 and 6 in glass tubes. The buffer solutions were incubated in the dark for 20 minutes at 88–90 °C (pH 4), 60 minutes at 98–100 °C (pH 5) or 20 minutes at 120–121 °C (pH 6) and 0.95–1.00 bar. Control samples from each pH group were incubated in the dark at room temperature. The buffer solution was then removed from each tube and the tubes rinsed with acetonitrile to recover the radioactivity adsorbed to the glassware. The buffer and rinse solutions were combined and analysed by LSC on the day of incubation. All samples were analysed by HPLC with radio-detection and representative samples were also analysed by TLC with radio-detection to confirm the identity of benzovindiflupyr parent. Parent was used as reference standard for both analyses.

The pH of the solutions did not change throughout the course of each experiment. The mean recovery of radioactivity applied to the test systems ranged from 98.4 to 100.7% for hydrolysed samples and from 97.7 to 101.8% for control sample. Table 34 shows radioactivity in the treated samples and control samples comprised only parent (no hydrolysis products were observed).

Table 34 Recovery and identification of radioactivity in 1 mg/L [¹⁴C]benzovindiflupyr solutions under pasteurisation, baking/brewing/boiling and sterilisation simulating conditions

Conditions	post incubation (%TAR)	parent (%TRR)
pH 4 (90 °C, 20 min) (pasteurisation)	99/101	100/100
pH 5 (100 °C, 60 min) (baking/brewing/boiling)	99/100	100/100
pH 6 (120 °C, 20 min) (sterilisation)	99/98	100/100

Study 2

Two supervised residue field trials with soya beans were conducted in Greece in 2010 [Ellis, 2012a, A17056D_10002]. Small plots of soya beans (360 m²) were treated with an EC formulation containing 150 g ai/L benzovindiflupyr alone. Four foliar applications were made, the first two at 0.074–0.075 kg ai/ha and the second two at an exaggerated rate of 0.369–0.372 kg ai/ha, each with a spray interval of 7 days. The applications were made with a backpack sprayer at a spray volume of 591–598 L/ha (no adjuvant). The last application was made at growth stage BBCH 82 (19–20 October 2010). Soya bean plants were harvested at normal commercial harvest (BBCH 89), 14 days after the last application. Plants were harvested and threshed using a combine and then separated further using an air stream to remove lighter parts and heavy parts were removed by hand. The seeds (50 kg per trial) were transported to the processing facility (Melissohori, Greece) at ambient temperature (< 5 hrs). The soya bean seeds were processed into refined oil, fat soy flour, tofu and soy sauce using standardised procedures designed to simulate relevant industrial processes. All weight fractions have been calculated for the initial weight of sub-sample processed in the relevant step for trial 01 and 02 respectively.

Refined oil processing

Soya bean seeds (26.140/25.460 kg) were cleaned by air cleaning, resulting in cleaned soya bean seeds (25.907/25.267 kg) and aspirated grain fractions (0.233/0.193 kg). The cleaned soya bean seeds were then dried until the optimal moisture content was reached. Afterwards the conditioned soya bean seeds were ground and sieved, resulting in soya bean hulls (2.393/2.300 kg) and dehulled soya bean seeds (22.054/20.626 kg). Subsequently the dehulled soya bean seeds were conditioned and flaked. The flakes were then extracted in two steps. Firstly n-hexane was added, heated up to around 60 °C and circulated for about 2 hours and then the solvent-oil mixture was distilled to separate oil and solvent. A rotary evaporator was used to remove any remaining solvent from the oil. The resulting fractions were crude oil (5.109/4.941 kg) and wet soya bean meal (17.936/17.168 kg). The wet soya bean meal was toasted at 120 °C to inactivate trypsin inhibitors. The toasted soya bean meal was conditioned to obtain an optimal moisture content of 15%. After conditioning the meal was milled, resulting in milled conditioned soya bean meal (20.033/19.216 kg). The crude oil was refined in several steps. For hydration, the crude oil was heated up to 60–70 °C and 10% of water was added. The mixture was continuously stirred for 45 min at 85–90 °C. After that the mixture was left for phase separation and the watery phase was removed. The remaining oily phase was heated up to 85 °C and 1% of phosphorus acid was added. The mixture was continuously stirred for 45 min at 85 °C. After that, 10% water was added and the mixture was left at 85 °C for phase separation. The watery phase was removed. For neutralisation the oily phase was heated up to 90 °C while continuously stirred. After that sodium hydroxide was added and the mixture was left for about 20 min. Then 10% of water was added and after 5 min the stirrer was switched off. After separation of phases the soapstock was removed. For washing, the remaining oily phase was heated up to 90 °C while stirred. After that, 10% of water was added and the mixture was left for 20 min for phase separation. The watery phase was removed. For drying, the remaining oily phase was heated up to 95 °C while stirred and citric acid was added. Drying was done using a vacuum until no more water remained in the oil. For bleaching, the oil was heated up to 95 °C while stirred. The oil was bleached for 35 min after addition of 1% of podsol. After that, the oil was filtered to remove the podsol. For deodorisation the oil was heated up and steam was transferred through the oil when it reached 160 °C. The oil was heated up further, until it reached 240 °C and then stayed at this temperature for about 20 min. The steam supply was stopped after cooling down to 160 °C. Drying of the oil was performed until a temperature ≤ 80 °C was reached. The resulting fraction is the refined oil (2.606/2.452 kg).

Fat soy flour processing

Soya bean seeds (5.000/5.000 kg) were cleaned by air cleaning, resulting in cleaned soya bean seeds (4.945/4.967 kg) and aspirated grain fractions (0.055/0.033 kg). Afterwards the soya bean seeds were milled, resulting in pollard (1.058/1.174 kg) and dehulled soya bean seeds (3.875/3.769 kg). Dehulled soya bean seeds were milled to get fat soy flour (3.440/3.385 kg).

Tofu processing

Soya bean seeds (5.000/5.000 kg) were cleaned by air cleaning, resulting in cleaned soya bean seeds (4.934/4.955 kg) and aspirated grain fractions (0.066/0.045 kg). The cleaned soya bean seeds were mixed with 2.5 fold rate of water for 12 hours swelling time. Afterwards mixture was separated to steep water and swelled soya bean seeds. The swelled soya bean seeds were mixed with 5 fold tap water and cooked for 10 min with continuous stirring. After cooking, the suspension was squeezed to get okara (9.792/9.627 kg) and soy milk (48.384/48.445 kg). The soy milk was heated up and at 85 °C calcium sulfate was added. The mixture was pressed to get raw tofu. The raw tofu was pasteurised at 82–87 °C for 5 min. After cooling down the resulting product was pasteurised tofu (3.664/3.497 kg).

Soy sauce processing

Soya bean seeds (5.000/5.000 kg) were cleaned by air cleaning, resulting in cleaned soya bean seeds (4.946/4.967 kg) and aspirated grain fractions (0.054/0.033 kg). The cleaned soya bean seeds were mixed with 6.25 fold water for 12 hours swelling time. Afterwards soya bean seeds were cooked for 85 min until they were soft. The cooked soya bean seeds were mixed with wheat flour (type 405), wheat pollards, *Aspergillus oryzae* and water. All the ingredients were kneaded to a loaf and matured for 5 days to get koji. Mixed salt and water (brine) was added to koji and a mash (moromi) was created. After one day a pH value of 4.5–5.0 was reached with lactic acid. One day later *Saccharomyces rouxii* was added to the mash. The moromi underwent a fermentation process which took around 3 months at 33 °C. After the maturing process, the moromi was pressed, resulting in raw soy sauce (12.730/12.030 kg) and miso (9.355/10.636 kg). The raw soy sauce was filtered and pasteurised to get pasteurized soy sauce (12.730/12.030 kg).

Soya bean seeds and processed fractions were stored at –18 °C for a maximum of 12 months prior to analysis. The storage period of 12 months is covered by the storage stability studies on parent, SYN546039 and SYN545720 in commodities with high oil content, soya bean flour, soya bean milk and soya bean crude oil.

Samples were analysed for parent, SYN546039 and SYN545720 using modification C of HPLC-MS-MS method GRM042.04A using matrix matched standards. Modification C of HPLC-MS-MS method GRM042.04A is considered valid for the determination of parent, free and/or conjugated SYN546039 and free and/or conjugated SYN545720 in the range 0.01–1.0 mg/kg for soya bean seeds and aspirated grain fractions, 0.01–0.5 mg/kg for soya bean hulls, soy oil, soya bean meal, fat soy flour, pollard, okara, soy milk, tofu, soy sauce and miso. Soya bean seeds were analysed immediately before processing. Results were not corrected for control levels (< 0.01 mg/kg for each analyte and matrix). Results were not corrected for individual concurrent method recoveries (72–116% for each analyte and matrix). Residue results and processing factors are shown in Table 35.

Table 35 Residues and processing factors in soya bean seeds and processed soy commodities

Trial, Location, year, (variety), dose rate, interval, DALT	Soya bean commodities	parent, mg/kg	SYN546039	SYN545720	PF parent
S10-02879-01, Kolchiko, Thessaloniki, Greece, 2010, (variety M10), 0.074+0.074+0.371+0.372 kg ai/ha, interval 7 days, DALT=14 Soil: sandy loam	Soya bean seeds (RAC)	0.025	< 0.01	< 0.01	–
	aspirated grain fractions	0.29	0.017	< 0.01	11
	soya bean hulls	0.26	< 0.01	< 0.01	10
	soy oil, crude	0.024	< 0.01	< 0.01	0.96
	soy oil, refined	0.017	< 0.01	< 0.01	0.68
	milled soya bean meal (15% moisture)	< 0.01	< 0.01	< 0.01	< 0.40
	Soya bean seeds (RAC)	0.022	< 0.01	< 0.01	–
	aspirated grain fractions	0.17	0.017	0.010	7.7
	fat soy flour	< 0.01	< 0.01	< 0.01	< 0.44
	pollard	0.11	< 0.01	< 0.01	4.8
	Soya bean seeds (RAC)	0.022	< 0.01	< 0.01	–
	aspirated grain fractions	0.32	0.022	< 0.01	14
	okara	< 0.01	< 0.01	< 0.01	< 0.44
	soy milk	< 0.01	< 0.01	< 0.01	< 0.44

Trial, Location, year, (variety), dose rate, interval, DALT	Soya bean commodities	parent, mg/kg	SYN546039	SYN545720	PF parent
	tofu, pasteurised	0.013	< 0.01	< 0.01	0.58
	Soya bean seeds (RAC)	0.028	< 0.01	< 0.01	–
	aspirated grain fractions	0.21	0.022	< 0.01	7.6
	soy sauce, pasteurised	< 0.01	< 0.01	< 0.01	< 0.36
	miso pasteurized	< 0.01	< 0.01	< 0.01	< 0.36
S10-02879-02, Galarinos, Chalkidiki, Greece, 2007, (variety M10), 0.075+0.075+0.370+0.369 kg ai/ha, interval 7 days, DALT=14 Soil: clay loam	Soya bean seeds (RAC)	0.026	< 0.01	< 0.01	–
	aspirated grain fractions	0.22	0.022	< 0.01	8.3
	soya bean hulls	0.28	0.010	< 0.01	11
	soy oil, crude	0.020	< 0.01	< 0.01	0.77
	soy oil, refined	0.017	< 0.01	< 0.01	0.65
	milled soya bean meal (15% moisture)	< 0.01	< 0.01	< 0.01	< 0.38
	Soya bean seeds (RAC)	0.029	< 0.01	< 0.01	–
	aspirated grain fractions	0.23	0.016	< 0.01	7.9
	fat soy flour	< 0.01	< 0.01	< 0.01	< 0.34
	pollard	0.10	< 0.01	< 0.01	3.6
	Soya bean seeds (RAC)	0.031	< 0.01	< 0.01	–
	aspirated grain fractions	0.23	0.012	< 0.01	7.4
	okara	< 0.01	< 0.01	< 0.01	< 0.32
	soy milk	< 0.01	< 0.01	< 0.01	< 0.32
	tofu, pasteurised	0.016	< 0.01	< 0.01	0.52
	Soya bean seeds (RAC)	0.030	< 0.01	< 0.01	–
	aspirated grain fractions	0.28	0.019	< 0.01	9.6
	soy sauce, pasteurised	< 0.01	< 0.01	< 0.01	< 0.34
	miso, pasteurized	< 0.01	< 0.01	< 0.01	< 0.34

Processing studies summary

Calculated processing factors for soya beans are summarized in Table 36.

Table 36 Overview of processing factors

Soya bean commodities	PF parent	PF median or best estimate
Soya bean seeds (RAC)	–	
aspirated grain fractions	7.4, 7.6, 7.7, 7.9, 8.3, 9.6, 11, 14	8.1
soya bean hulls	10, 11	10
soy oil, crude	0.77, 0.96	0.86
soy oil, refined	0.65, 0.68	0.66
soya bean meal, dried	< 0.38, < 0.40	< 0.4
soy fat flour	< 0.34, < 0.44	< 0.4
pollard	3.6, 4.8	4.2
okara	< 0.32, < 0.44	< 0.4
soy milk	< 0.32, < 0.44	< 0.4
tofu, pasteurised	0.52, 0.58	0.55
soy sauce, pasteurised	< 0.34, < 0.36	< 0.4
miso, pasteurised	< 0.34, < 0.36	< 0.4

Residues in the edible portion of food commodities

No data submitted.

Residues in animal commodities

Direct animal treatments

Not applicable.

Farm animal feeding studies

The Meeting received information on feeding studies in dairy cows.

A residue feeding study in lactating Holstein/Friesian dairy cows was conducted in the UK in 2011 [Ward and Vance, 2012, SYN545192_10188]. Benzovindiflupyr was administered orally in gelatine capsules once daily to three groups of three cows for 28 consecutive days. Two control cows received gelatine dosing capsules containing no active substance. Experimental conditions are indicated in Table 37. Milk samples were collected twice daily throughout acclimation and feeding periods (days –1, 1, 2, 3, 5, 7, 10, 14, 17, 21, 24 and 28) and PM and AM milk was combined to produce a 24 hr bulk milk sample. Average daily milk yields ranged from 7.5–16 kg/animal. Skimmed milk and cream were produced from milk collected at day 21 and 28 using a centrifugal cream separator. Animal 4 (1× dose group) was killed on welfare grounds on day 14 due to an underlying health condition unrelated to on-study procedures. All remaining treated cows and one cow from the control group were killed approximately 22–24 hours following the final dose. Samples of liver, kidney, muscle (diaphragm, loin and hind leg) and fat (perirenal, mesenteric and subcutaneous) were collected from all animals. Milk and tissue samples were stored at –20 °C until analysed. The maximum frozen storage intervals prior to analysis were 18 days for milk, 15 days for skimmed milk, 24 days for cream, 20 days for liver, 23 days for kidney, 34 days for muscle and 57 days for fat.

Samples were analysed for parent, SYN546039 (including conjugates) and SYN546422 (including conjugates) using modification A of HPLC-MS-MS method 1887 (see method GRM042.06) and using matrix-matched standards. Method 1886 version 2.0 is considered valid for the purpose of this study (commodity type and concentration level of the analytes). Residues of parent, SYN546039 or SYN546422 were not found in any of the control samples (each < 0.01 mg/kg). Mean concurrent recoveries for all analytes and all tissues were within 70–120%, showing adequate performance at the time of sample analysis.

Analytical results in tissue samples are shown in Table 38. Samples from the 1× and 5× dose groups were only analysed when residues in one or more samples from the next highest dose rate contained residues ≥ 0.01 mg/kg (LOQ). Residues of parent and SYN546039 (free plus conjugated) were highest in liver. Residues in other tissues decreased in the order kidney > fat > muscle for both parent and SYN546039. Residues of SYN546422 (free plus conjugated) were not found (< 0.01 mg/kg) in any of the tissues.

No residues (< 0.01 mg/kg, each analyte) were found in whole milk at any of the treatment days (10× dose rate samples analysed only). No residues (< 0.01 mg/kg, each analyte) were found in skimmed milk in the day 21 and day 28 samples (10× dose rate samples analysed only). Residues in cream for the 21 and 28 day samples are shown in Table 39.

Table 37 Experimental conditions of the cow feeding study

Cow number	Mean individual and group bodyweight over the feeding period (kg)	Mean daily dry feed intake (kg feed/animal)	Actual mean dose (ppm ai in feed)	Actual mean dose (mg ai/animal/day)	Actual mean dose (mg/kg bw/day)
1, 2	676, 648 (group 662)	14	0 0× dose group	0	0
3, 4, 5	734, 596, 613 (group 648)	15	3.5 1× dose group	53	0.082
6, 7, 8	623, 679, 674 (group 658)	17	16 5× dose group	281	0.43
9, 10, 11	610, 694, 679 (group 661)	16	32 10× dose group	529	0.80

Table 38 Benzovindiflupyr related residues in tissues for 1×, 5× and 10× dose groups

Sample	Dose rate (ppm feed)	Cow	parent (mg/kg)	SYN546039 (mg/kg)	SYN546422 (mg/kg)	Parent Mean (mg/kg)	Parent Max (mg/kg)
Hind Leg Muscle	3.5 (1× dose)	3	NA	NA	NA	NA	NA
		4	NS	NS	NS		
		5	NA	NA	NA		
	16 (5× dose)	6	< 0.01	< 0.01	NA	< 0.01	< 0.01
		7	< 0.01	< 0.01	NA		
		8	< 0.01	< 0.01	NA		
	32 (10× dose)	9	0.01	0.01	< 0.01	0.010	0.01
		10	< 0.01	< 0.01	< 0.01		
		11	< 0.01	< 0.01	< 0.01		
Loin muscle	3.5 (1× dose)	3	NA	NA	NA	NA	NA
		4	NS	NS	NS		
		5	NA	NA	NA		
	16 (5× dose)	6	NA	< 0.01	NA	NA	NA
		7	NA	< 0.01	NA		
		8	NA	< 0.01	NA		
	32 (10× dose)	9	< 0.01	0.01	< 0.01	< 0.01	< 0.01
		10	< 0.01	< 0.01	< 0.01		
		11	< 0.01	< 0.01	< 0.01		
Diaphragm muscle	3.5 (1× dose)	3	NA	< 0.01	NA	NA	NA
		4	NS	NS	NS		
		5	NA	< 0.01	NA		
	16 (5× dose)	6	< 0.01	< 0.01	NA	< 0.01	< 0.01
		7	< 0.01	< 0.01	NA		
		8	< 0.01	< 0.01	NA		
	32 (10× dose)	9	0.02	0.02	< 0.01	0.017	0.02
		10	0.02	< 0.01	< 0.01		
		11	< 0.01	< 0.01	< 0.01		
Liver	3.5 (1× dose)	3	< 0.01	< 0.01	NA	< 0.01	< 0.01
		4	NS	NS	NS		
		5	< 0.01	< 0.01	NA		
	16 (5× dose)	6	0.07	0.04	NA	0.037	0.07
		7	0.02	0.03	NA		
		8	0.02	0.04	NA		
	32 (10× dose)	9	0.07	0.21	< 0.01	0.047	0.07
		10	0.04	0.08	< 0.01		
		11	0.03	0.05	< 0.01		
kidney	3.5 (1× dose)	3	< 0.01	< 0.01	NA	< 0.01	< 0.01
		4	NS	NS	NS		
		5	< 0.01	< 0.01	NA		
	16 (5× dose)	6	< 0.01	< 0.01	NA	0.010	0.01
		7	< 0.01	< 0.01	NA		
		8	0.01	< 0.01	NA		
	32 (10× dose)	9	0.02	0.03	< 0.01	0.013	0.02
		10	0.01	0.02	< 0.01		
		11	< 0.01	0.01	< 0.01		
subcutaneous fat	3.5 (1× dose)	3	< 0.01	< 0.01	NA	< 0.01	< 0.01
		4	NS	NS	NS		
		5	< 0.01	< 0.01	NA		
	16 (5× dose)	6	< 0.01	< 0.01	NA	< 0.01	< 0.01
		7	< 0.01	0.01	NA		
		8	< 0.01	< 0.01	NA		
	32 (10X dose)	9	0.02	0.04	< 0.01	0.017	0.02
		10	0.02	0.01	< 0.01		
		11	< 0.01	< 0.01	< 0.01		
perirenal fat	3.5 (1× dose)	3	< 0.01	< 0.01	NA	< 0.01	< 0.01
		4	NS	NS	NS		
		5	< 0.01	< 0.01	NA		
	16	6	< 0.01	0.02	NA	0.013	0.02

Sample	Dose rate (ppm feed)	Cow	parent (mg/kg)	SYN546039 (mg/kg)	SYN546422 (mg/kg)	Parent Mean (mg/kg)	Parent Max (mg/kg)
	(5× dose)	7	0.02	0.02	NA		
		8	< 0.01	< 0.01	NA		
	32	9	0.03	0.04	< 0.01	0.023	0.03
	(10× dose)	10	0.03	0.02	< 0.01		
		11	< 0.01	< 0.01	< 0.01		
mesenterial fat	3.5	3	< 0.01	< 0.01	NA	< 0.01	< 0.01
	(1× dose)	4	NS	NS	NS		
		5	< 0.01	< 0.01	NA		
	16	6	< 0.01	0.01	NA	0.010	0.01
	(5× dose)	7	0.01	0.01	NA		
		8	< 0.01	< 0.01	NA		
	32	9	0.03	0.03	< 0.01	0.023	0.03
	(10× dose)	10	0.03	0.02	< 0.01		
		11	< 0.01	< 0.01	< 0.01		

NA—not analysed, no residue > LOQ in next highest dose group

NS—no sample (animal killed on welfare grounds on day 14)

Table 39 Benzovindiflupyr related residues in cream for 1×, 5× and 10× dose groups

Dose rate ppm feed	Sampling	Cow	parent mg/kg	SYN546039 (including conjugates) mg/kg	SYN546422 (including conjugates) mg/kg
3.5 (1× dose)	Day 21	3	< 0.01	< 0.01	NA
		4	NS	NS	NS
		5	< 0.01	< 0.01	NA
	Day 28	3	< 0.01	< 0.01	NA
		4	NS	NS	NS
		5	< 0.01	< 0.01	NA
	Average		< 0.01	< 0.01	NA
	Highest residue		< 0.01	< 0.01	NA
16 (5× dose)	Day 21	6	0.01	< 0.01	NA
		7	0.01	< 0.01	NA
		8	< 0.01	< 0.01	NA
	Day 28	6	0.01	< 0.01	NA
		7	0.01	< 0.01	NA
		8	< 0.01	< 0.01	NA
	average		0.01	< 0.01	NA
	highest residue		0.01	< 0.01	NA
32 (10× dose)	Day 21	9	0.03	0.02	< 0.01
		10	0.02	0.01	< 0.01
		11	0.01	< 0.01	< 0.01
	Day 28	9	0.03	0.02	< 0.01
		10	0.03	0.01	< 0.01
		11	0.01	< 0.01	< 0.01
	average		0.02	0.013	< 0.01
	highest residue		0.03	0.02	< 0.01

NA = not analysed, no residue > LOQ in next highest dose group

NS = no sample (animal killed on welfare grounds on day 14)

Residues in food in commerce or at consumption

Data no longer required.

National Residue Definition

No information available.

APPRAISAL

Benzovindiflupyr was scheduled for residue evaluation as a new compound by the 2014 JMPR at the 45th session of the CCPR (2013). The toxicological review was conducted in 2013, which established an ADI of 0–0.05 mg/kg bw and an ARfD of 0.1 mg/kg bw. Additional toxicological data were provided for the metabolites SYN546039 and SYN545720. Benzovindiflupyr was defined by the WHO panel as the only toxicologically significant compound in animals, plants and the environment.

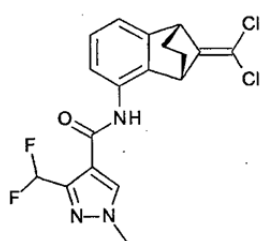
Benzovindiflupyr is a broad-spectrum fungicide belonging to the chemical class of pyrazole carboxamides. Benzovindiflupyr acts as an inhibitor of the fungal complex II respiratory chain, where it inhibits the succinate dehydrogenase enzyme (succinate dehydrogenase inhibitor, SDHI) by blocking the ubiquinone-binding sites in the mitochondrial complex.

The Meeting received information from the manufacturer on identity, metabolism, storage stability, residue analysis, use pattern, residues resulting from supervised trials on soya beans, fate of residue during processing, and livestock feeding studies.

Chemical name:

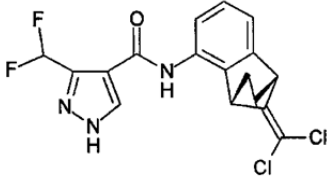
Benzovindiflupyr: N-[(1R,4S)-9-(dichloromethylene)-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1-methylpyrazole-4-carboxamide (IUPAC).

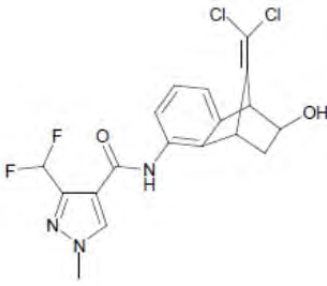
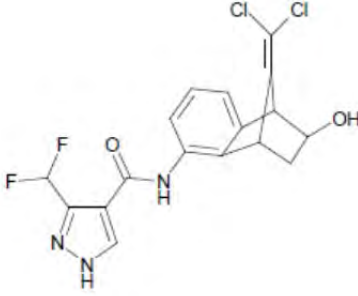
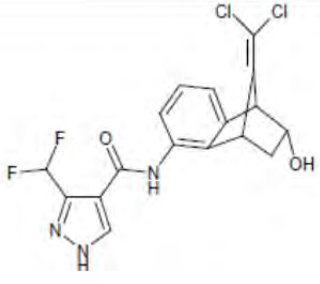
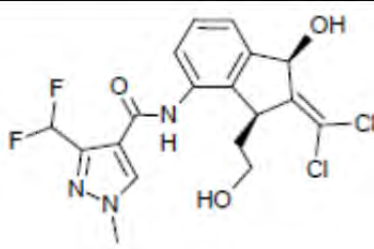
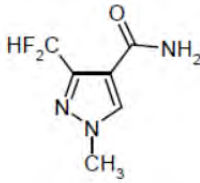
Structural formula:

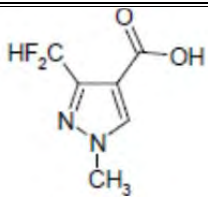
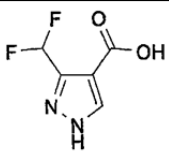


Benzovindiflupyr contains chiral centres at both the bridgehead carbon atoms potentially resulting in four stereoisomeric forms. However, the bicyclic ring is a rigid structure, and therefore only two stereoisomers exist (an enantiomeric pair). Technical benzovindiflupyr consists of a racemic mixture of two enantiomers SYN546526 and SYN546527, at a ratio of 50:50. SYN546526 represents N-[(1R,4S)-9-(dichloromethylene)-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1-methylpyrazole-4-carboxamide. SYN546527 represents N-[(1S,4R)-9-(dichloromethylene)-1,2,3,4-tetrahydro-1,4-methanonaphthalen-5-yl]-3-(difluoromethyl)-1-methylpyrazole-4-carboxamide. The enantiomers were unresolved using the chromatographic solvent systems in the reports. Both enantiomers were measured within the same single chromatographic peak and they were collectively reported as one concentration. Both enantiomers are fungicidally active. No toxicological studies were performed on the individual enantiomers.

Metabolites referred to in the appraisal by codes:

SYN546206 (N-demethyl-BVFP)	 N-demethyl-benzovindiflupyr

SYN546039 (BVFP-OH)	 hydroxy-benzovindiflupyr
SYN546041 (N-demethyl-BVFP-OH)	 N-demethyl-hydroxy-benzovindiflupyr
SYN546042 (N-demethyl-BVFP-OH)	 N-demethyl-hydroxy-benzovindiflupyr
SYN546422	 N- [(1SR,3RS)-2-(dichloromethylene)-1-hydroxy-3-(2-hydroxy-ethyl)-indan-4-yl]-3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxamide
SYN508272	 3-difluoromethyl-1-methyl-1H-pyrazole-4-carboxylic acid amide

NOA449410	 3-difluoromethyl-1-methyl-1H-pyrazole-4-carboxylic acid
SYN545720	 3-difluoromethyl-1H-pyrazole-4-carboxylic acid

Animal metabolism

The Meeting received results of metabolism studies in laboratory animals, lactating goats and laying hens.

Metabolism in laboratory animals was summarized and evaluated by the WHO panel of the JMPR in 2013. In absorption, distribution, metabolism and excretion (ADME) studies, overnight-fasted animals showed clinical signs at doses that were non-toxic to fed animals. Therefore, most of the ADME studies were performed in fed animals. Absorption of benzovindiflupyr was approximately 80% at the low dose (1 mg/kg bw) and showed saturation at the higher dose (approximately 60% absorption at 40 mg/kg bw). In low-dose bile duct cannulated animals, 4% of the administered dose was found in urine, 17% in faeces and 69–76% in bile; in high-dose animals, 9% of the administered dose was found in urine, 32% in faeces and 47–57% in bile. At both dose levels, 86–97% of the administered dose was excreted within 48 hours after administration. The major route of excretion was by bile. For tissues, the elimination half-lives were in the range of 40–316 hours. Highest residues were identified in the liver, kidney, adrenals, thyroid and heart. After repeated daily dosing, levels of radioactivity in tissues appeared to have reached steady-state concentrations after 14 days. The predominant metabolic pathway for benzovindiflupyr is N-demethylation, phenyl and/or bicyclo hydroxylation and opening of the bicyclo system. Additionally, subsequent formation of glucuronic acid or sulfate conjugates was observed. The amide bond of benzovindiflupyr is preserved.

One lactating goat per radiolabel was dosed orally once daily for 7 consecutive days with a gelatin capsule containing [¹⁴C-phenyl]-benzovindiflupyr or [¹⁴C-pyrazole]-benzovindiflupyr. The equivalent actual mean daily doses in the dry feed were 41 or 32 ppm for the phenyl or pyrazole label, respectively. Goats were sacrificed 12 hours after the last dose. Total recovered radioactivity amounted to 90% and 86% of the administered dose for the phenyl and pyrazole radiolabelled forms, respectively. The majority of the radioactivity was recovered in faeces (79%/73%, phenyl/pyrazole). The remainder of the dose was recovered in urine (4.5%/5.2%, phenyl/pyrazole) and GI tract contents (6.8%/7.2%), while only low levels were found in milk and tissues (<0.5% in total).

The highest radioactivity concentrations were found in liver (1.3/0.70 mg/kg eq) and kidney (0.28/0.18 mg/kg eq), followed by fat (0.098/0.070 mg/kg eq) and muscle (0.070/0.032 mg/kg eq). Total radioactive residues in milk reached a plateau concentration of approximately 0.046 mg/kg eq following 96 hours dosing for the phenyl label and 0.035 mg/kg eq following 72 hours dosing for the pyrazole label..

Following solvent extraction, residue extractabilities were 78–89% TRR for liver and ≥ 94% TRR for milk and all other tissues. Liver and kidney extracts were treated with β-glucuronidase to cleave glucuronide and sulphate conjugates.

Parent was identified in milk and all goat tissues at levels of 5.5–13% TRR in milk, kidney and liver, 24–25% in muscle and 41–44% TRR in fat. Conjugated metabolites formed a significant

part of the extracted residue in liver and kidney. The most significant metabolites (including conjugates) identified in all tissues and milk were the mono-hydroxylated metabolite SYN546039 (22–50% TRR) and metabolite SYN546422 (16–25% TRR in milk and kidney, 1.5–8.9% TRR in other tissues). Levels of SYN546039 in milk and tissues (except fat) or levels of SYN546422 in milk and kidney were higher than those of the parent compound. Other metabolites (including conjugates) were found at levels below 10% TRR. Post-extraction solids from liver (11–22% TRR) were shown to be associated with protein. Protease treatment of the post-extraction solids resulted in a mixture of highly polar metabolites.

Five laying hens per radiolabel were dosed orally once daily for 14 consecutive days with a gelatin capsule containing [¹⁴C-phenyl]-benzovindiflupyr or [¹⁴C-pyrazole]-benzovindiflupyr. The equivalent actual mean daily doses in the dry feed were 16–20 or 17–20 ppm for the phenyl or pyrazole label, respectively. Hens were sacrificed 12 hours after the last dose. Total recovered radioactivity amounted to 88% and 93% of the administered dose for the phenyl and pyrazole radiolabelled forms, respectively. The majority of the radioactivity was recovered in excreta (88%/92%, phenyl/pyrazole), while only low levels were found in eggs and tissues (<0.2% in total).

The highest radioactivity concentrations were found in liver (0.19/0.25 mg/kg eq), followed by fat (0.033/0.045 mg/kg eq) and muscle (0.025/0.036 mg/kg eq). Total radioactive residues in egg yolks achieved a plateau concentration of 0.17–0.18 mg/kg eq after 168–240 hrs of dosing. Total radioactive residues in egg whites achieved a plateau concentration of 0.03–0.04 mg/kg eq after 120–168 hrs of dosing.

Following solvent extraction, residue extractabilities were ≥83% TRR for egg yolk and egg white, 68–73% TRR for skin with fat, 48–49% TRR for liver and 24–37% TRR for muscle. Liver extracts were treated with β-glucuronidase to cleave glucuronide and sulphate conjugates.

Parent was identified at levels of 0.2–3.3% TRR in liver and muscle, 11–14% in eggs and 38–42% TRR in skin with fat. Benzovindiflupyr was extensively metabolised, resulting in low levels of various metabolites in tissues (<6.0% TRR each). The most significant metabolites in eggs were the mono-hydroxylated metabolites SYN546039 (12–22% TRR in eggs, 1.3–5.1% TRR in tissues) and the mono-hydroxylated demethylated metabolites SYN546041 (10.6–12.5% TRR) and SYN546042 (6.6–12.2% TRR). Levels of SYN546039, SYN546041 and SYN546042 in eggs were in the same order of magnitude as those of the parent compound. Post-extraction solids from liver (51–52% TRR) and muscle (63–76% TRR) were shown to be associated with protein. Protease treatment of the post-extraction solids resulted in a mixture of highly polar metabolites.

In summary, metabolism observed in lactating goats and laying hens arose via hydroxylation on the alicyclic ring to form SYN546039 (all tissues, milk, eggs). In hens metabolism proceeded through N-demethylation to form SYN546041 and SYN546042 isomers (mainly in eggs), while in ruminants metabolism proceeded through an alternative pathway consisting of oxidative opening of the alicyclic ring to form SYN546422 (mainly in milk, kidney). Several other minor metabolites arose in livestock by cleavage between the pyrazole and phenyl rings and/or conjugation as glucuronide or sulphate compounds. Further metabolism involves association with proteins.

The major compounds identified in goat, hen tissues, milk or eggs are: parent, SYN546039, SYN546422, SYN546041 and SYN546042 and their conjugates. Parent and SYN546039 and its conjugates comprise a significant part of residue in tissues, milk and eggs. Significant additional contributions are found for metabolites SYN546041 and SYN546042 in eggs and SYN546422 and its conjugates in milk and goat kidney.

In general, metabolism between goat, hen and rat is similar, with a few exceptions. Formation of SYN546422 is only found in goat. Opening of the bicyclo system as such is found in rat and metabolite SYN546422 is postulated as a plausible rat intermediate between the open bicyclo rat metabolites SYN546634 and SYN546707 (the demethylated form of SYN546422). Cleavage between the pyrazole and phenyl rings to form SYN508272 is found in the pyrazole labelled studies in hens and goats, while in the rat studies this bond is preserved.

Plant metabolism

The Meeting received plant metabolism studies for benzovindiflupyr after foliar application on fruits (tomatoes), cereals (wheat) and pulses/oilseeds (soya beans).

The metabolism of ^{14}C -phenyl-benzovindiflupyr or ^{14}C -pyrazole-benzovindiflupyr in indoor grown tomatoes was studied following four foliar applications at 0.13–0.14 kg ai/ha at weekly intervals. Total radioactive residues (TRR) in mature tomato fruits at DAT = 1 and 14 were 0.047 and 0.092 mg/kg eq for the phenyl label and 0.18 and 0.15 mg/kg eq for the pyrazole label, respectively. A high proportion of the residue remained on the surface of the fruit (65–79% TRR). The residues in or on the fruit could be extracted by acetonitrile/water (> 99% TRR). The principal component of the residue was the parent compound (91–95% TRR). A number of metabolites were detected, none reaching >0.5% TRR.

The metabolism of ^{14}C -phenyl-benzovindiflupyr or ^{14}C -pyrazole-benzovindiflupyr in indoor grown wheat was studied following two foliar applications at 0.14 kg ai/ha at a 35 day interval. Residue levels in wheat forage harvested 9 days after the first application and residue levels in wheat hay, wheat straw and wheat grains harvested 10, 40 and 41 days after the second application were 3.0, 4.9, 8.1, 0.12 mg/kg eq for the phenyl label and 2.1, 6.4, 9.0, 0.092 mg/kg eq for the pyrazole label, respectively. The major part of the residues (> 97% TRR) could be extracted with acetonitrile/water. The principal component of the residue was the parent compound: 84–87% TRR in grain, 81–84% TRR in straw and 89–103% in wheat forage and hay. A number of metabolites were detected, none reaching >5.0% TRR. Several of these metabolites were present in the free and conjugated form.

The metabolism of ^{14}C -phenyl-benzovindiflupyr or ^{14}C -pyrazole-benzovindiflupyr in indoor grown soya beans was studied following two foliar applications at 0.12–0.13 kg ai/ha at a 22 day interval. Residue levels in soya bean forage harvested 11 days after the first application and soya bean hay and soya bean seeds harvested at 13 and 30 days after the second application were 3.4, 14 and 0.029 mg/kg eq for the phenyl label and 4.1, 13 and 0.10 mg/kg eq for the pyrazole label, respectively. The major part of the residues (> 89% TRR) could be extracted with solvents. Parent was identified at levels of 15–31% TRR in soya bean seeds, 67–72% TRR in soya bean hay and 83–85% TRR in soya bean forage. The most significant metabolite in soya bean seeds was the cleavage product SYN545720 (47% TRR). The major part of the SYN545720 metabolite was present in the conjugated form (30% TRR), particularly as an aspartic acid conjugate or monosaccharide conjugate. The most significant metabolite in soya bean forage and hay was the mono-hydroxylated metabolite SYN546039 (9.2–12% TRR). The major part of the SYN546039 metabolite was present in the conjugated form (8.5–12% TRR), particularly as a malonyl glycoside or glycoside conjugate. A number of other metabolites were detected, none reaching >14% TRR. One of these metabolites was NOA449410, a plant specific metabolite, which was not detected in rat. NOA449410 was present at very low levels: < 1.2% TRR or 0.012 mg/kg eq) in soya forage, 0.039 mg/kg eq in soya hay or 0.0012 mg/kg eq in soya seeds.

In summary, the degree of metabolism in crops after foliar application varied. In fruits and cereals parent compound represented the principal part of the residue in tomato fruits, wheat grain, wheat straw and wheat forage and hay. Metabolism proceeded further in pulses/oilseeds with parent representing 15–31% TRR in soya bean seeds, 67–72% TRR in soya bean hay and 83–85% TRR in soya bean forage. Metabolism observed in pulses/oilseeds arose via hydroxylation on the alicyclic ring to form SYN546039). Metabolism proceeded through N-demethylation and cleavage between the pyrazole and phenyl rings to form SYN545720.

In general, metabolism between plants and rat is similar, except for the cleavage between the pyrazole and phenyl rings. In rat this bond is preserved. The cleavage product SYN508272 is found in the pyrazole labelled studies in hens and goats. Cleavage products NOA449410 and SYN545720 are not found in rat nor in livestock.

Environmental fate in soil

The Meeting received information on photolysis in water and on soil and information on rotational crops.

All plant metabolism studies have been conducted indoors. Since the interval between the first application and harvest for the investigated commodities is long (29–111 days) and the residue is a surface residue, photolysis may form a major route of degradation. Since indoor grown plants are not subjected to the full spectrum of sunlight, degradation on field grown plants may show a different behaviour. Photolysis studies in water or on soil show a DT₅₀ of 44 or 144–244 days, respectively, confirming the potential for photolysis. Photolysis in water after 15 days and on soil after 30 days demonstrated the formation of low levels of SYN546039, SYN508272, NOA449410, and SYN545720 (0.6–8.5% TAR), indicating the absence of an alternative degradation pathway under outdoor conditions. In addition, preliminary experimental work on photolysis on leaf surfaces grown under greenhouse, under artificial sunlight and outdoor conditions for 7 days, demonstrated very minor degradation in all three test conditions while the resulting minor degradates were qualitatively similar. The Meeting concluded that the indoor plant metabolism studies are considered acceptable for deriving a residue definition for plant commodities.

Metabolism of ¹⁴C-phenyl-benzovindiflupyr or ¹⁴C-pyrazole-benzovindiflupyr was investigated in confined rotational crops following a single bare soil treatment. A sandy loam soil was treated at a rate of 0.53–0.54 kg ai/ha under indoor conditions. Rotational crops (lettuce, wheat and turnip) were sown at 30, 90 and 300 day plant back intervals (PBI). Total radioactivity in rotational crops ranged from 0.003–0.77 mg/kg eq at 30 day PBI, 0.002–0.34 mg/kg eq at 90 day PBI and 0.007–0.29 mg/kg eq at 300 day PBI. Total radioactivity levels above 0.05 mg/kg eq were found in wheat forage, hay and straw at all plant back intervals.

Wheat grain had very low residues (< 0.01–0.014 mg/kg eq) and most extracts were not amenable to chromatographic examination. In turnip roots, parent was the principal component and ranged from 81–90% TRR at the 30 day plant back interval, 69–72% TRR at the 90 day plant back interval and 64–71% at the 300 day plant back interval. In the leafy parts of crops (lettuce, turnip leaves, wheat forage, hay, straw) parent compound was the principal component of the residue at the 30 day PBI. Parent ranged from 14–37% TRR at the 30 day PBI, 13–35% TRR at the 90 day PBI and 6.5–29% TRR at the 300 day PBI. At the 90 and 300 day PBI, significant metabolites were the cleavage products NOA449410 and SYN545720, together accounting for 53–73% TRR, 34–46% TRR and 24–26% TRR in immature lettuce, mature lettuce and wheat forage respectively. NOA449410 was mainly present in the conjugated form whereas SYN545720 was present in the free and conjugated form. Other metabolites were generally present at low levels (each < 10% TRR). In wheat forage, wheat hay and wheat straw, SYN546206 and SYN546039 were more significant, frequently occurring at > 10% TRR and occasionally approaching 20% TRR each (including conjugates). SYN546206 was mainly present in the free form; SYN546039 was mainly present in the conjugated form.

From these data the Meeting concluded that benzovindiflupyr can be taken up from the soil under confined conditions even after long plant back intervals (300–366 days). Metabolites found in confined rotational crops (SYN546206, SYN546039, NOA449410 and SYN545720) are identical to those observed in primary crops and may have arisen from photolysis, degradation in and uptake from soil as well as from metabolism within the crop itself.

In two field rotational crop studies at four different locations in the EU benzovindiflupyr was applied onto bare soil at a single application of 0.20 kg ai/ha. Rotational crops (spinach, wheat, carrots) were sown 28–32, 60–69 or 355–366 days after application.

In another field rotational crop study at four different locations in the USA benzovindiflupyr was applied as foliar application to soya beans or peanuts at 3x 0.10 kg ai/ha with 14 day intervals. The last application was at BBCH 71–89 of the target crops. The soya bean and peanut target crops were harvested and removed from the field. Rotational crops (spinach or lettuce, radish or turnip, wheat) were sown 30 or 180 days after application.

No residues > 0.01 mg/kg of parent benzovindiflupyr or metabolites SYN546206 or SYN546039 (including conjugates) were found in any of the harvested commodities at any of the rotations, except in wheat forage and wheat straw from the 28–30 or 60 day plant back intervals. Parent and SYN546039 (including conjugates) were found in wheat forage and wheat straw at levels up to 0.012–0.022 mg/kg. Metabolites NOA449410 and SYN545720 were not analysed.

The dose rates as used in the field rotational crop studies (1x0.20 kg ai/ha or 3x0.10 kg ai/ha) are higher than those used in the actual supervised residue trials submitted (3x 0.045 kg ai/ha for soya beans). Based on the current uses, the Meeting concluded that no residues are expected in rotational crops. Should additional uses be developed in future, rotational crop studies may need to be re-evaluated.

Methods of Analysis

The Meeting received description and validation data for analytical methods for the determination of benzovindiflupyr related residues in plant and animal commodities.

The existing multi-residue method QuEChERS was submitted as enforcement/monitoring method for the determination of parent compound in plant and animal commodities. Plant commodities were extracted with acetonitrile/water (1:1, v/v), Samples were cleaned-up by SPE prior to quantification by HPLC-MS/MS. The Meeting considers validation sufficient for plant commodities with high acid content, high water content, high starch content, high oil content and all animal commodities (meat, liver, kidney, fat, milk and eggs). The LOQ was 0.01 mg/kg for parent compound in each matrix.

Several other HPLC-MS/MS methods were submitted for the determination of parent and its metabolites SYN546206 (free), SYN545720 (including conjugates) and/or SYN546039 (including conjugates) in plant material. Crop commodities were extracted with acetonitrile/water (80/20). Parent and SYN546206 were separated off by liquid-liquid partition and the remaining extract was treated with acid at pH 2 for 6 hrs at 100 °C to cleave the SYN545720 and/or SYN546039 conjugates. Extraction efficiency for acetonitrile/water was at least 71% for parent and 81% for SYN546206 (free) as shown by a radio-validation study in wheat hay and wheat straw. Efficiency of extraction and hydrolysis was >100% for SYN546039 (including conjugates) and SYN545720 (including conjugates) as shown by a radio-validation study in soya bean hay and soya bean seed. Most analytical methods were considered fit for purpose with LOQs of 0.01 mg/kg for individual analytes.

Another HPLC-MS/MS method was submitted for the determination of parent and its metabolites SYN546039 (including conjugates) and SYN546422 (including conjugates) in milk, eggs or animal tissues. Animal commodities were extracted with acetonitrile/water (80/20). Parent was determined directly in the primary extract, while the remaining extract was treated with beta-glucuronidase at pH 5 for 6 hrs at 37 °C to cleave the SYN546039 and/or SYN546422 conjugates. Extraction efficiency for acetonitrile/water was >100 % for parent, free SYN546039 and free SYN546422 as shown by a radio-validation study in milk, muscle, and egg yolk. Efficiency of extraction and hydrolysis was >100% for SYN546039 (including conjugates) and at least 67% for SYN546422 (including conjugates) as shown by a radio-validation study in liver. The analytical method was considered fit for purpose with LOQs of 0.01 mg/kg for individual analytes.

Stability of pesticide residues in stored analytical samples

The Meeting received information on the storage stability of parent, SYN546039, SYN546206 and SYN545720 in raw and processed plant commodities and of parent, SYN546039 and SYN546422 in animal commodities.

Storage stability studies showed that benzovindiflupyr and metabolite SYN546039 (free) were stable for at least 24 months at -18°C in crop commodities representative of the high water, high acid, high starch, high protein and high oil commodity groups as well as in wheat straw. Metabolite SYN546206 (free) was stable for at least 22 months at -18 °C in crop commodities representative of the high water and high starch commodity groups as well as in wheat straw. Metabolites SYN545720

(free) and SYN508272 (free) were stable for at least 24 months at -18 °C in crop commodities representative of the high oil, high acid and high protein commodity groups.

Benzovindiflupyr and metabolites SYN546039 (free) and SYN545720 (free) were stable for at least 24 months at -10 °C in various processed commodities: flour (maize, soya), meal (maize), oil (maize, soya), soymilk, dried fruits (grape, apple) and fruit juice (apple).

Benzovindiflupyr and metabolites SYN546039 (free) and SYN546422 (free) were stable at -20 °C for at least 56–62 days in milk and eggs and at least 76–78 days in liver and muscle. According to OECD Guideline 506 storage stability in kidney and fat may be extrapolated from the other animal tissues, so The Meeting concluded that residues in kidney and fat are likely to be stable for at least 76 days.

Definition of the residue

The major compounds identified in goat, hen tissues, milk or eggs are: parent, SYN546039, SYN546422, SYN546041 and SYN546042 and their conjugates. Parent was identified at levels of 38–44% TRR (0.012–0.040 mg/kg) in goat fat and hen skin with fat, 24–25% (0.008–0.017 mg/kg) in goat muscle and 0.2–14% TRR (<0.001–0.14 mg/kg) in milk, eggs, and all other tissues. The mono-hydroxylated metabolite SYN546039 and its conjugates was identified at levels of 22–50% TRR (0.008–0.64 mg/kg eq) in milk and goat tissues, 12–22% TRR (0.008–0.022 mg/kg eq) in eggs and <6.0% TRR in hen tissues. Metabolite SYN546422 and its conjugates was only found in goat and was identified at levels of 16–25% TRR (0.010–0.052 mg/kg eq) in milk and goat kidney and 1.5–8.9% TRR (0.001–0.056 mg/kg eq) in other goat tissues. The mono-hydroxylated demethylated metabolite isomers SYN546041 and SYN546042 were identified at levels of 11–12% TRR (0.003–0.020 mg/kg eq) and 6.6–12% TRR (0.002–0.020 mg/kg eq) in eggs, respectively, <5.0% TRR in hen tissues and <10% TRR in goat tissues and milk.

Benzovindiflupyr parent is found in every animal commodity, although the levels in milk, eggs, edible offal and hen muscle are low. To be able to detect benzovindiflupyr related residues, metabolites SYN546039, SYN546422 (goat only), SYN546041 (eggs only) and SYN546042 (eggs only) could be included in the residue definition for enforcement/monitoring. Since a significant part of these metabolites is present as conjugates, a hydrolysis procedure is required to be able to measure these metabolites. Including the metabolites in the residue definition means the residue is unlikely to be measured by a multi-residue method. Since benzovindiflupyr itself can be measured by a multi-residue method and use of a multi-residue method is encouraged, the Meeting decided to define the residue for enforcement/monitoring as parent only.

The log K_{ow} for benzovindiflupyr is 4.3. The goat metabolism study and the goat feeding study did not show a clear partition of the parent compound into the fat tissues, although in the high dose cow feeding study, parent was found in cream and not in the corresponding whole milk. In a hen metabolism study, the partitioning of the parent compound into the fatty tissues is more pronounced: highest levels of benzovindiflupyr are found in egg yolks (0.022–0.024 mg/kg) and skin with fat (0.012–0.019 mg/kg). Since benzovindiflupyr has a preference for fat in the poultry tissues as well as in high dose milk, the Meeting considers the residue fat soluble.

Apart from benzovindiflupyr, metabolites found at significant levels in livestock commodities were: SYN546039, SYN546041, SYN546042, SYN546422 and their conjugates. The toxicity of SYN546039, SYN546041, SYN546042 and SYN546422 is considered to be covered by toxicity studies on benzovindiflupyr since each of the free metabolites was actually found or agreed to be a possible intermediate in the rat. N-demethylation is regarded as neutral for toxicological potency while hydroxylation generally lowers toxicity. The JMPR 2013 received additional toxicological data for the mono-hydroxylated metabolite SYN546039, showing that this compound is at least 10 fold less toxic than parent. For metabolites SYN546041, SYN546042 and SYN546422 a read across to SYN546039 toxicity studies seems justified based on the close structural similarity and this suggests that they are also at least 10 fold less toxic than the parent.. Therefore none of these metabolites is considered relevant for the residue definition for dietary risk assessment. The Meeting decided to define the residue for dietary risk assessment as parent only.

In primary crops, parent compound represented the principal part of the residue in most crop commodities: 91–95% TRR (0.004–0.16 mg/kg) in tomato fruits, 84–87% TRR (0.077–0.10 mg/kg) in wheat grain, 81–84% TRR (6.6–7.6 mg/kg) in wheat straw and 89–103% (2.1–5.9 mg/kg) in wheat forage and hay, 67–72% TRR (8.7–10 mg/kg) in soya bean hay and 83–85% TRR (2.8–3.5 mg/kg) in soya bean forage. Metabolism proceeded further in pulses/oilseeds with parent representing 15–31% TRR (0.009–0.015 mg/kg) in soya bean seeds. A significant metabolite in soya bean seeds was SYN545720 (47% TRR (0.047 mg/kg eq) including conjugates). A significant metabolite in soya forage and hay was SYN546039 (9.2–12% TRR (0.38–1.6 mg/kg eq) including conjugates).

In rotational crops parent was the principal component in root commodities (64–90% TRR, 0.008–0.023 mg/kg) and a significant component (6.5–37% TRR, 0.001–0.085 mg/kg) in the leafy parts of crops (lettuce, turnip leaves, wheat forage, hay, straw). Significant metabolites were the cleavage products NOA449410 and SYN545720, together accounting for 34–73% TRR (0.009–0.014 mg/kg eq) and 24–26% TRR (0.023–0.026 mg/kg eq) in lettuce and wheat forage, respectively. In wheat forage, wheat hay and wheat straw, SYN546206 and SYN546039 were frequently occurring at > 10% TRR each, and occasionally approaching 20% TRR each (0.006–0.12 mg/kg eq, including conjugates).

Benzovindiflupyr is found in every primary crop commodity, although the levels in soya bean seeds are low. Benzovindiflupyr is found in every rotational crop commodity, except cereal grains, although levels in leafy crop parts vary. To be able to detect benzovindiflupyr related residues metabolites SYN545720 and NOA449410 could be included in the residue definition for enforcement/monitoring. Since a significant part of these metabolites is present as conjugates, a hydrolysis procedure is required to be able to measure them. Including the metabolites in the residue definition means the residue is unlikely to be measured by a multi-residue method. Furthermore, SYN545720 and NOA449410 can also arise in plant commodities as a result of treatment with other pyrazole fungicides like bixafen, fluxapyroxad, isopyrazam and sedaxane. Since SYN545720 and NOA449410 cannot be seen as a marker for benzovindiflupyr, the Meeting decided to define the residue for enforcement/monitoring as parent only.

Apart from benzovindiflupyr, metabolites found at significant levels in plant commodities were: SYN546206, SYN546039, NOA449410, SYN545720 and their conjugates. The toxicity of SYN546206 and SYN546039 is considered to be covered by toxicity studies on benzovindiflupyr since each of the free metabolites was actually found in the rat. The cleavage products NOA449410 and SYN545720 are not found in rat. N-demethylation is regarded as neutral for toxicological potency, while hydroxylation generally lowers toxicity. The JMPR 2013 received additional toxicological data for the mono-hydroxylated metabolite SYN546039, showing that this compound is at least 10 fold less toxic than parent. Toxicity studies for NOA449410 (from sedaxane studies) and SYN545720 (from isopyrazam and sedaxane studies) showed that the toxicity of these metabolites is probably 100–1000 fold less toxic than parent. SYN546206 is the only compound which might be relevant for the residue definition for dietary risk assessment, since the toxicological potency might be similar to the parent. Metabolite SYN546206 is only found in feed commodities (wheat forage, wheat hay, wheat straw) in the confined rotational crop studies and its presence could not be confirmed in the field rotational crops studies. The Meeting decided to define the residue for dietary risk assessment as parent only.

The Meeting recommended the following residue definition for benzovindiflupyr:

Definition of the residue for compliance with the MRL and for dietary risk assessment for plant and animal commodities: *benzovindiflupyr*.

The Meeting considers the residue fat soluble.

Results of supervised residue trials on crops

Soya beans (dry)

Field trials involving soya beans were performed in Brazil.

Critical GAP for soya beans in Paraguay is for three foliar applications without adjuvant at 0.045 kg ai/ha at 14 day intervals with a PHI of 21 days. Trials from Brazil (3x 0.045 kg ai/ha, interval 19–59 and 14 days, PHI 21–28 days, adjuvant added) matched this GAP. For each plot location, replicate trials were conducted with two-three different formulations. Only the highest residue was selected from these trials. For trials, where residues at longer PHIs (28 – 35 days) were higher than residues at PHI 21 days, the highest residue was selected. Benzovindiflupyr residues were: <0.01, <0.01, <0.01, <0.01, 0.01, 0.03 mg/kg (n=6).

The Meeting estimated a maximum residue level of 0.05 mg/kg on soya beans (dry). The Meeting estimated an STMR of 0.01 mg/kg.

Fate of residues during processing

Information on the fate of residues during processing showed that benzovindiflupyr is stable (100% recovery) under standard conditions simulating pasteurisation, baking/brewing/boiling and sterilisation.

Processing studies were undertaken for soya beans. Processing factors based on the residue for parent only are listed in the table below. Using the STMR_{RAC} obtained from benzovindiflupyr use, the Meeting estimated STMR-Ps for processed commodities to be used in the livestock dietary burden calculations and/or dietary intake calculations.

Commodity	Processing factors Residue: parent only	Processing factor (PF) (median or best estimate)	STMR-P = STMR _{RAC} x PF (mg/kg)
soya aspirated grain fractions	7.4, 7.6, 7.7, <u>7.9</u> , <u>8.3</u> , 9.6, 11, 14	8.1	0.081
soya bean hulls	<u>10</u> , <u>11</u>	10	0.10
soya oil, crude	<u>0.77</u> , <u>0.96</u>	0.86	0.0086
soya oil, refined	<u>0.65</u> , <u>0.68</u>	0.66	0.0066
soya meal, dried	<0.38, <0.40	<0.4	0.004
soya fat flour	<0.34, <0.44	<0.4	0.004
soya pollard	<u>3.6</u> , <u>4.8</u>	4.2	0.042
soya okara	<0.32, <0.44	<0.4	0.004
soya milk	<0.32, <0.44	<0.4	0.004
soya tofu, pasteurised	<u>0.52</u> , <u>0.58</u>	0.55	0.0055
soya sauce, pasteurised	<0.34, <0.36	<0.4	0.004
soya miso, pasteurised	<0.34, <0.36	<0.4	0.004

Residues in animal commodities

The Meeting estimated the dietary burden of benzovindiflupyr residues on the basis of the livestock diets listed in the FAO manual appendix IX (OECD feedstuff table). For bulk commodities like soya beans, calculation from STMR provides the levels in feed suitable for estimating maximum residue levels as well as STMR values for animal commodities. Commodities used in the dietary burden calculation are soya beans and soya bean processed commodities. Supervised residue trials on soya bean forage and fodder were not available, whereby the dietary burden for livestock might be underestimated.

Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are provided in Annex 6. A mean and maximum dietary burden for livestock, based on benzovindiflupyr use, is shown in the table below.

Livestock dietary burden for benzovindiflupyr residues, expressed as ppm of dry matter diet

	US	EU	AU	JP	overall	
	Max and mean	Max and mean	Max and mean	Max and mean	Max and mean	
beef cattle	0.017	0.013	0.0022	0.011	0.017	^a
dairy cattle	0.0016	0.013	0.0029	0.0069	0.013	^b

	US	EU	AU	JP	overall	
	Max and mean	Max and mean	Max and mean	Max and mean	Max and mean	
poultry broiler	0.0033	0.015	0.0081	0.0015	0.015	^c
poultry layer	0.0033	0.0081	0.0081	0.0013	0.0081	^d

^a Highest mean and maximum dietary burden suitable for maximum residue level and STMR estimates for mammalian meat

^b Highest mean and maximum dietary burden suitable for maximum residue level and STMR estimates for milk

^c Highest mean and maximum dietary burden suitable for maximum residue level and STMR estimates for poultry meat

^d Highest mean and maximum dietary burden suitable for maximum residue level and STMR estimates for eggs

The Meeting received a feeding study on lactating cows.

Three groups of three lactating Holstein cows were dosed once daily via capsules at levels of 3.5, 16 and 32 ppm parent compound in dry weight feed for 28 consecutive days. Two control cows received a placebo. Milk was collected throughout the study and tissues were collected on day 28 within 22–24 hrs after the last dose. No parent residues >0.01 mg/kg were found in whole milk, cream, muscle, liver, kidney or fat at the 3.5 ppm dose level.

The dietary burden for beef and dairy cattle of 0.017 and 0.013 ppm, respectively, is 200 times lower than the lowest dose administered in the cow feeding study (3.5 ppm). Therefore, no parent residues > 0.01 mg/kg are expected in milk, cream and cattle tissues.

No feeding study is available for poultry. In a metabolism study laying hens were dosed at 16–20 ppm parent compound in the dry feed for 14 consecutive days. Parent residues were: 0.024 mg/kg in egg yolks, 0.0037 mg/kg in egg whites, 0.019 mg/kg in fat, in 0.00050 mg/kg in liver and 0.0012 mg/kg in muscle. The dietary burden for broiler and layer poultry of 0.015 and 0.0081 ppm, respectively, is 1000 times lower than the dose administered in the hen metabolism study (16–20 ppm). Therefore, no parent residues > 0.01 mg/kg are expected in eggs, egg yolks and hen tissues.

The Meeting estimated maximum residue levels of 0.01* mg/kg in milk, eggs and all animal commodities. The Meetings estimated an STMR and HR of 0 mg/kg in milk, eggs and all animal commodities. The residue in animal commodities is considered fat soluble.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits

Definition of the residue for compliance with the MRL and for dietary risk assessment for plant and animal commodities: *benzovindiflupyr*.

The Meeting considers the residue fat soluble.

Summary of recommendations

CCN	Commodity name	MRL mg/kg	STMR or STMR-P mg/kg	HR or HR-P mg/kg	Comment
VD 0541	soya bean (dry)	0.05	0.01	-	-
MM 0095	Meat (from mammals other than marine mammals)	0.01*	0	0	-
MO 0105	Edible offal (Mammalian)	0.01*	0	0	-
MF 0100	Mammalian fats (except milk fats)	0.01*	0	0	-
ML 0106	Milks	0.01*	0	0	-
PM 0110	Poultry meat	0.01*	0	0	-
PF 0111	Poultry fats	0.01*	0	0	-
PO 0111	Poultry, Edible offal of	0.01*	0	0	-
PE 0112	Eggs	0.01*	0	0	-

CCN	Commodity name	MRL mg/kg	STMR or STMR-P mg/kg	HR or HR-P mg/kg	Comment
-	soya aspirated grain fractions	-	0.081	-	feed stuff

CCN	Commodity name	MRL mg/kg	STMR or STMR-P mg/kg	HR or HR-P mg/kg	Comment
-	soya bean hulls	-	0.10	-	feed stuff
OC 0541	soya oil, crude	-	0.0086	-	-
OR 0541	soya oil, refined	-	0.0066	-	-
-	soya meal, dried	-	0.004	-	feed stuff
-	soya fat flour	-	0.004	-	-
-	soya pollard	-	0.042	-	feed stuff
-	soya okara	-	0.004	-	feed stuff
-	soya milk	-	0.004	-	-
-	soya tofu, pasteurised	-	0.0055	-	-
-	soya sauce, pasteurised	-	0.004	-	-
-	soya miso, pasteurised	-	0.004	-	-

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDI) for benzovindiflupyr was calculated from recommendations for STMRs for raw and processed commodities in combination with consumption data for corresponding food commodities. The results are shown in Annex 3.

The IEDI of the 17 GEMS/Food cluster diets, based on the estimated STMRs represented 0% of the maximum ADI of 0.05 mg/kg bw, expressed as benzovindiflupyr.

The Meeting concluded that the long-term intake of residues of benzovindiflupyr from uses considered by the Meeting is unlikely to present a public health concern.

Short-term intake

The International Estimated Short Term Intake (IESTI) for benzovindiflupyr was calculated from recommendations for STMRs for raw and processed commodities in combination with consumption data for corresponding food commodities. The results are shown in Annex 4.

The IESTI for the diets submitted to the JMPR represented 0% of the ARfD (0.1 mg/kg bw, expressed as benzovindiflupyr). The Meeting concluded that the short-term intake of residues of benzovindiflupyr from uses considered by the Meeting is unlikely to present a public health concern.

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BUPROFEZIN (173)

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APPRAISAL

Buprofezin was evaluated by JMPR in 1991 for the first time and then in 1995 and 1999. It was also reviewed under the Periodic Re-evaluation Programme in 2008 for toxicity and residues followed by residue evaluations in 2009 and 2012; and for consideration of a concern form from the USA in 2013¹.

The 2012 Meeting received information on supervised trials on coffee conducted in Brazil and the USA as well as coffee processing. These trials were conducted in accordance with GAP in the USA for coffee while employed different processing methods for producing green beans from coffee berries: wet method in Hawaii, USA; and dry method in Brazil. Due to the apparent difference in residues from different processing methods and the lack of information on processing practices for coffee berries used in the USA, the 2013 Meeting could not estimate a maximum residue level.

The current Meeting received another concern form from the USA stating that since the processing of coffee berries to green coffee followed the local practices of countries where the trials were conducted, a maximum residue levels should be estimated for coffee beans and roasted coffee beans.

During the current Meeting, new information became available about the processing practices in Hawaii, USA. According to the information, both the wet and dry methods are used for processing coffee berries to green beans. At the present time, mostly the wet method is used while the dry method is used in the small farms and some recently established large coffee farms. Based on the information, the Meeting concluded that the trials in the USA and Brazil complied with GAP in the use of pesticides for coffee and the processing practices used in the USA.

The Meeting reviewed the data provided to the 2012 Meeting. GAP in the USA for coffee allows four foliar applications (minimum interval of 14 days) at a rate of 1.12 kg ai/hL with a PHI of 0 days. The previous Meeting selected the following residue concentrations from the trials matching the above GAP:

From the trials in the USA (4): 0.08, 0.12, 0.155 and 0.24 mg/kg; and

From the trials in Brazil (3): 0.055 and 0.075 (2) mg/kg.

The combined residues were: 0.055, 0.075, 0.075, 0.08, 0.12, 0.155 and 0.24 mg/kg.

The Meeting estimated a maximum residue level and an STMR of 0.4 mg/kg and 0.08 mg/kg, respectively, for coffee beans.

Based on the STMR for coffee beans and the processing factor of 0.32 from coffee beans to roasted coffee beans and < 0.2 to freeze-dried coffee calculated in 2012 (2012 Evaluation), the Meeting estimated STMR-Ps of 0.0256 mg/kg and 0.016 mg/kg, respectively, for roasted coffee beans and freeze-dried coffee.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for compliance with MRLs and for estimation of dietary intake (Plant commodities and animal commodities): *buprofezin*

¹ 2012 Report and Evaluation; and 2013 Report (FAO)

Commodity		Recommended MRL, mg/kg		STMR/STMR-P	HR/HR-P
CCN	Name	New	Previous	mg/kg	mg/kg
SB 0716	Coffee beans	0.4	-	0.08	-
SM 0716	Coffee beans, roasted			0.0256	-
	Freeze-dried coffee			0.016	-

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Dietary Intakes (IEDIs) of buprofezin were calculated for the 17 GEMS/Food cluster diets using STMRs and STMRPs estimated by the 2008, 2009, 2012 and current Meetings (Annex 3). The ADI is 0-0.009 mg/kg bw and the calculated IEDIs were 3–40% of the maximum ADI. The Meeting concluded that the long-term intake of residues of buprofezin resulting from the uses considered by the 2008, 2009, 2012 and current JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short-Term Intakes (IESTI) of buprofezin were calculated for coffee beans and their processed products commodities using STMRs/STMR-Ps estimated by the current Meeting (see Annex 4). The ARfD is 0.5 mg/kg and the calculated IESTIs were 0–0 % of the ARfD for the general population and for children. The Meeting concluded that the short-term intake of residues of buprofezin, when used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

CHLORANTRANILIPROLE (230)

First draft was prepared by Dr Paul Humphrey, Australian Pesticides and Veterinary Medicines Authority, Canberra, Australia

EXPLANATION

Chlorantraniliprole is a novel insecticide belonging to the class of selective ryanodine receptor agonists and was evaluated for the first time by JMPR in 2008 (T, R). It was also evaluated in 2010 and 2013 for additional MRLs. At the Forty-fifth Session of the CCPR (2013), chlorantraniliprole was listed for residue evaluation for additional maximum residue levels by the 2014 JMPR.

The Meeting received information on registered use patterns, supervised residue trials and fate of residues in processing.

The 2008 JMPR established an ADI and AfRD for chlorantraniliprole of 0–2 mg/kg bw/day and “not required” respectively. A residue definition of *chlorantraniliprole* was established for both compliance and dietary risk assessment in both plant and animal commodities.

USE PATTERN

Information on registered uses made available to this meeting is shown in Table 1.

Table 1 Registered uses of chlorantraniliprole on citrus fruit, green bulb vegetables, pulses (mung beans, chickpeas and soya beans), cereal grains, peanuts and herbs

Crop	Country	Formulation	Type	Application		Rate [g ai/ ha]	Concentration [g ai/ 100 L]	Season Max. [g ai/ha/year] or (no. per crop)	PHI [days]
		g ai/L or [g ai/kg]		Method	Timing [Interval —days]				
Citrus Fruits									
Citrus	Republic of South Africa	200	SC	Foliar	42	297.5	3.5	(2)	7
Bulb Vegetables									
Bulb vegetables ^a	USA	200	SC	Foliar	7	50–73	–	224 g ai/ha/ crop 672 g ai/ha/ year (4)	1
Pulses									
Chickpea, mung beans and soya beans	Australia	[350]	WG ^e	Foliar	7	24.5	–	(2)	14 (harvest grazing)
Bengal gram	India	200	SC	Foliar	–	25	5	(2)	11
Soya bean, dry	Japan	50	OD	Foliar	–	12.5–37.5	1.25	(2)	7
Soya bean	USA	200	SC	Foliar	3	50–73	–	224 g ai/ha/ year (4)	1
Cereal Grains									
Cereal grains: Crop Group 15 ^b (except corn and wild rice) and cereal for forage Crop Group 16 (except corn and wild rice)	Canada	200	SC	Foliar	7	25–75	–	225 g ai/ha/ season (3)	1
Cereal grains except corn and rice ^c	USA	200	SC	Foliar	7	50–73	–	224 g ai/ha/ year (4)	14

Crop	Country	Formulation		Application				PHI [days]		
		g ai/L or [g ai/kg]	Type	Method	Timing [Interval —days]	Rate [g ai/ ha]	Concentration [g ai/ 100 L]		Season Max. [g ai/ha/year] or (no. per crop)	
Oilseeds										
Peanut	USA	200	SC	Foliar	3	50–73	–	224 g ai/ha/ year (4)	1	
Herbs										
Herbs subgroup d	USA	200	SC	Foliar	3	50–73	–	224 g ai/ha/ crop 897 g ai/ha/ year (4)	1	

^a Chive, fresh leaves; Chive; Chinese fresh leaves; Daylily, bulb; Elegans, hosta; Fritillaria, bulb; Fritillaria, leaves; Garlic, bulb; Garlic, great-headed bulb; Garlic, serpent, bulb; Kurrat, Lady's leek; Leek; Leek, wild; Lily, bulb; Onion, Beltsville bunching; Onion, bulb; Onion, Chinese, bulb; Onion, fresh; Onion, green; Onion macrostem; Onion, pearl; Onion, potato, bulb; Onion, tree tops; Shallot, bulb; Shallot fresh leaves; Cultivars, varieties and/or hybrids of these.

^b Barley; Buckwheat; Millet, Pearl; Millet, Proso; Oats; Rye; Sorghum; Teosinte; Triticale; Wheat.

^c Cereal Grains except Corn and Rice Including Barley; Buckwheat; Pearl Millet; Proso Millet; Oats; Rye; Sorghum (milo); Sorghum spp. grain sorghum Sudan grass (seed crop) and hybrids of these grown for its seed; Teosinte; Triticale; Wheat.

^d Including Angelica; balm basil; borage; burnet; camomile; catnip; chervil (dried); chive, Chinese; clary; coriander (leaf); costmary; culantro (leaf); curry (leaf); dillweed; horehound; hyssop; lavender; lemongrass; lovage (leaf); marigold; marjoram; nasturtium; parsley (dried); pennyroyal; rosemary; rue; sage; savory; summer and winter; sweet bay; tansy; tarragon; thyme; wintergreen; woodruff and wormwood.

^e Use a non-ionic surfactant/ wetting agent at 125 g active/ 100 L.

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised trials for the uses of chlorantraniliprole on citrus fruits (oranges and mandarins), bulb vegetables (green onions), pulses (chickpeas, mung beans and soya beans), cereals (barley, sorghum and wheat) and oilseeds (peanuts).

Trials were well documented with laboratory and field reports. The former included method validation including recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of sample storage were also provided. Concurrent storage stability data was provided for the green onion trials, confirming sample stability over the trial storage period (24 months). Sufficient storage stability data for a range of crop matrices has also been evaluated by previous Meetings. Applications were generally made using backpack sprayers although occasionally tractor mounted sprayers were used. Samples were collected and stored frozen immediately or soon after sampling. Although trials included control plots, no control data are recorded in the Tables because, unless noted, no residues in control samples exceeded the LOQ. When residues were observed in the control samples they are shown as *c* followed by the residues observed in the control sample. Residues are unadjusted for recoveries. In some trials, samples were taken just before the final application and then again on the same day after the spray had dried. In the data tables the notation for these sampling times is '–0' and '0' respectively.

Residues from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels and dietary intake assessment. If a higher residue level was observed at a longer PHI than the GAP, the higher value has been used in MRL setting and dietary intake assessment. For replicate samples (from the same plot), the mean value (calculated on unrounded individual values) was used for maximum residue level estimation and dietary intake assessment. For two or more analyses of the same sample, the mean value was used for maximum residue level estimation and dietary intake assessment, with the individual results given in brackets. For multiple trials on a crop from the same location, the result from the trial yielding the highest residue was utilised for maximum residue level estimation and dietary intake assessment. In this case the trials are separated by a dotted line.

Group	Commodity	Country	Table No.
FC Citrus Fruits	Oranges	RSA	2
	Mandarins/ tangelos	RSA	2
VA Bulb Vegetables	Green onion (fresh and dried)	Canada,	3
VD Pulses	Chickpeas	Australia	4
	Bengal gram (chickpea)	India	5
	Mung beans	Australia	6
	Soya beans	Australia	7
	Barley	USA	8
GC Cereal Grains	Sorghum	USA	9
	Wheat	USA	10
	Peanuts	USA	11
SO Oilseeds	Chickpeas trash and forage	Australia	12
Animal Feeds	Bengal gram (chickpea) pods	India	13
	Mung beans trash and forage	Australia	14
	Soya beans trash and forage	Australia	15
	Barley hay and straw	USA	16
	Sorghum forage and stover	USA	17
	Wheat forage, hay and straw	USA	18
Processed commodities	Wheat and processed	USA	19

The results of these supervised trials are shown in the following tables:

Citrus fruits

Supervised trials were carried out on citrus fruit [eight trials (four orange, four mandarins)—Table 2] in the Republic of South Africa (RSA) during the 2010 growing season (van Zyl 2010, 2418/D924). Two foliar applications of a 200 g ai/L SC formulation were made with ground equipment at 3.5 g ai/100 L in 4200–13100 L/ha, resulting in applications of 147–459 g ai/ha. The second application was made 29–31 days after the first and 21 days before harvest. Two field sample replicates were harvested. Residues of chlorantraniliprole in peel and flesh were quantitated by GC-ECD method DuPont 13291. Acceptable concurrent recovery data were obtained.

Note: The RSA citrus data from the 2010 JMPR Meeting have also been tabulated below.

Table 2 Residues from the foliar application of chlorantraniliprole to citrus fruits in the RSA

Country Year (Variety)	Application						DAT	Chlorantraniliprole (mg/kg)				Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	g ai/100 L	Water (L/ha)	Growth Stage (Last Application)		Peel	Flesh	Flesh Mean	Whole Fruit	
GAP, RSA (citrus)	2	42		3.5			7					
RSA LA Visagie, Nelspruit, Mpumalanga Province 2010 Orange—Valencia Midnight	2	31	355	3.5	10100	21 days before	0 before	0.57	ND	ND	0.11	Van Zyl
			—	—	—	harvest		0.57	ND			2010,
							0 after	0.74	0.07	0.07	0.22	2418/D924,
								0.77	0.07			Trial 1
							3	0.96	0.04	0.05	0.23	
								1.0	0.05			
							7	0.76	0.03	0.04	0.20	
RSA LA Visagie,	2	29	450	3.5	12900	21 days before	0 before	0.61	0.06	0.05	0.17	Van Zyl
			459	3.5	13100	harvest		0.49	0.04			2010,

Country Year (Variety)	Application						DAT	Chlorantraniliprole (mg/kg)				Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	g ai/100 L	Water (L/ha)	Growth Stage (Last Application)						
Nelspruit, Mpumalanga Province 2010 Orange—Valencia Late							0 after	1.2	0.10	0.09	0.38	2418/D924, Trial 2
								1.3	0.08			
							3	1.2	0.09	0.10	0.33	
								0.97	0.11			
							7	0.82	0.06	<u>0.06</u>	<u>0.24</u>	
								0.78	0.06			
RSA Addo Sundays River, Eastern Cape Province 2010 Orange—Autumn Gold	2	29	296	3.5	8400	21 days before	0 before	0.33	0.03	0.03	0.09	Van Zyl
			296	3.5	8500	harvest		0.29	0.03			2010,
							0 after	0.60	0.05	0.05	0.17	2418/D924,
								0.51	0.05			Trial 5
							3	0.51	0.05	0.05	0.15	
								0.49	0.05			
							7	0.49	0.06	<u>0.05</u>	<u>0.14</u>	
								0.45	0.04			
RSA Addo Sundays River, Eastern Cape Province 2010 Navel Orange— Lane Late	2	29	296	3.5	8400	21 days before	0 before	0.19	0.03	0.03	0.06	Van Zyl
			295	3.5	8400	harvest		0.18	0.02			2010,
							0 after	0.40	0.04	0.04	0.13	2418/D924,
								0.51	0.03			Trial 6
							3	0.50	0.04	0.05	0.14	
								0.44	0.05			
							7	0.54	0.05	<u>0.05</u>	<u>0.15</u>	
								0.52	0.04			
RSA Letsitele, Mopani, Tzaneen, Limpopo Province 2010 Mandarin— Clemengold	2	30	290	3.5	8300	21 days before	0 before	0.39	0.02	0.03	0.12	Van Zyl
			290	3.5	8300	harvest		0.37	0.03			2010,
							0 after	1.4	0.08	0.09	0.42	2418/D924,
								1.4	0.10			Trial 3
							3	1.6	0.07	0.08	0.40	
								1.4	0.09			
							7	1.0	0.08	<u>0.08</u>	<u>0.30</u>	
								0.96	0.08			
RSA Letsitele, Mopani, Tzaneen, Limpopo Province 2010 Clementine Mandarin —Nardocott	2	30	316	3.5	9000	21 days before	0 before	0.40	0.02	0.02	0.11	Van Zyl
			316	3.5	9000	harvest		0.41	0.02			2010,
							0 after	0.80	0.07	0.07	0.26	2418/D924,
								0.90	0.07			Trial 4
							3	0.92	0.05	0.06	0.24	
								0.79	0.07			
							7	0.85	0.07	<u>0.06</u>	<u>0.22</u>	
								0.74	0.05			
RSA Drankenstein	2	30	147	3.5	4200	21 days before	0 before	0.18	ND	ND	0.05	Van Zyl
			216	3.5	6200	harvest		0.17	ND			2010,

[illegible]

Country Year (Variety)	Application						DAT	Chlorantraniliprole (mg/kg)				Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	g ai/100 L	Water (L/ha)	Growth Stage (Last Application)						
RSA Addo, Eastern Cape Province 2009 Tangelo—Nova Tangelo	2	30	293	3.5	8400		0 before	0.36	0.03		0.10	2418-D80
			295	3.5	8400		0 after	0.87	0.06		0.26	
							3	0.91	0.08		0.31	(2010
							7	0.72	0.04		0.22	JMPR)
							14	0.81	0.05		0.25	
							21	0.68	0.05		0.20	
RSA Addo, Eastern Cape Province 2009 Mandarin—Nules Clementine	2	30	295	3.5	8400		0 before	0.31	0.06		0.10	2418-D80
			295	3.5	8400		0 after	0.77	0.07		0.24	
							3	0.72	0.07		0.23	(2010
							7	0.57	0.06		0.18	JMPR)
							14	0.60	0.07		0.18	
							21	0.62	0.04		0.16	
RSA Paarl, Western Cape Province 2009 Mandarin— Satsuma	2	30	295	3.5	8400		0 before	0.53	0.02		0.16	2418-D80
			295	3.5	8400		0 after	0.75	0.05		0.24	
							3	0.79	0.04		0.24	(2010
							7	0.91	0.03		0.32	JMPR)
							14	0.89	0.05		0.27	
							21	1.1	0.07		0.35	
RSA Wellington, Western Cape Province 2009 Mandarin— Nules	2	30	247	3.5	7100		0 before	0.25	ND		0.07	2418-D80
			247	3.5	7100		0 after	0.47	0.04		0.16	
							3	0.48	0.03		0.16	(2010
							7	0.41	0.03		0.13	JMPR)
							14	0.44	0.03		0.13	
							21	0.41	0.06		0.14	

LOQ = 0.02 mg/kg for flesh and 0.05 mg/kg for peel

DAT = Days After Treatment

Bulb Vegetables

Supervised trials were carried out on green onions (five trials—Table 3) in Canada and the USA during the 2009–2010 growing seasons (Dorschner 2012b, A10204). Two foliar applications of a 200 g ai/L SC formulation were made at 110–118 g ai/ha. Applications were made to plots using spray volumes of 215–421 L/ha with ground equipment. Two field sample replicates were harvested from each plot for fresh green onions and also for dried green onions (i.e., four samples from each plot). Residues of chlorantraniliprole in fresh and dried whole plant were quantitated using a method similar to LC-MS/MS method DuPont 13294. Acceptable concurrent recovery data were obtained.

Table 3 Residues from the foliar application of chlorantraniliprole to green onions in the USA and Canada

Country Year (Variety)	Application					Matrix	DAT	Chlorantraniliprole (mg/kg)		Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage			Individual	Mean	
GAP, USA (bulb vegetables)	4	7	73 (224/ crop)				1			
USA	2	3	113	412	Mature	Whole plant,	1	0.70	0.65	Dorschner
Salinas, California			113	421	Mature	fresh		0.61		2012b,
2009			226			Whole plant,	1	6.0	6.3	IR-4 A10204,
White spear						dried		6.6		CA 99
USA	2	3	111	215	Bulbing	Whole plant,	1	0.37	0.41	Dorschner
Holtville, California			112	224	Mature	fresh		0.44		2012b,
2010			223			Whole plant,	1	1.1	1.0	IR-4 A10204,
Tri-5503						dried		0.90		CA100
USA	2	3	113	281	Mature	Whole plant,	1	0.84	0.79	Dorschner
Salisbury, Maryland			113	281	Mature	fresh		0.73		2012b,
2009			226			Whole plant,	1	2.7	2.7	IR-4 A10204,
Evergreen hardy white						dried		2.7		MD01
Canada	2	3	118	318	Mature	Whole plant,	1	1.5	1.5	Dorschner
Harrow, Ontario			110	290	Mature	fresh		1.5		2012b,
2009			228			Whole plant,	1	11	11	IR-4 A10204,
Emerald Isle						dried		11		ON09
Canada	2	3	112	393	5 true leaves	Whole plant,	1	0.70	0.72	Dorschner
St Sur Richelieu,			113	402	Mature	fresh		0.74		2012b,
Quebec			225			Whole plant,	1	1.5	1.5	IR-4 A10204,
2009						dried		1.5		QC05
Parade										

LOQ = 0.01 mg/kg

DAT = Days After Treatment

Pulses

Supervised trials were carried out on chickpeas (three trials—Table 4) in Australia during the 2011 growing season (Litzow 2013, DuPont-33763). Two foliar applications of a 350 g ai/kg formulation were made at 24.5–24.9 or 48.7–49.7 g ai/ha. A non-ionic surfactant was added. Applications were made to plots using spray volumes of 77–93 L/ha with ground equipment. Residues of chlorantraniliprole were quantitated by LC-MS/MS using Method DuPont 13294. Acceptable concurrent recovery data were obtained.

Residues in chickpea trash and forage are shown in Table 12.

Table 4 Residues from the foliar application of chlorantraniliprole to chickpeas in Australia

Country Year (Variety)	Application					Matrix	DAT	Chlorantraniliprole (mg/kg)	Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage				
GAP, Australia (chickpea)	2	7	24.5				14		
Australia	2	7	24.5	78	BBCH 79/81	Seed	14	< 0.01	Litzow 2013,
Condamine Plains,			24.5	80	BBCH 88/93				DuPont- 33763,
Queensland,	2	7	48.7	77	BBCH 79/81	Seed	14	< 0.01	Trial 110807
2011			48.7	79	BBCH 88/93				
Hatrick									
Australia	2	7	24.5	91	BBCH 82	Seed	5	0.045	Litzow 2013,
Bellata, New South			24.5	92	BBCH 85		7	< 0.01	DuPont- 33763,
Wales							14	0.015	Trial 110808
2011	2	7	49.7	93	BBCH 82	Seed	5	0.37	
Hatrick			49.7	92	BBCH 85		7	0.065	
							14	0.064	
Australia	2	7	24.9	93	BBCH 86/91	Seed	14	0.025	Litzow 2013,
Narrabri,			24.9	90	BBCH 87/94				DuPont- 33763,
New South Wales	2	7	49.0	91	BBCH 86/91	Seed	14	0.14	Trial 110809
2011			49.0	90	BBCH 87/94				
(Hatrick)									

LOQ = 0.01 mg/kg

DAT = Days After Treatment

Supervised trials were carried out on Bengal gram (chickpeas) (four trials—Table 5) in India during the 2009–2010 growing seasons (Piriyadarsini 2010, 1004574). Two foliar applications of a 200 g ai/L SC formulation were made at 25 or 50 g ai/ha. Applications were made to plots using spray volumes of 400–500 L/ha with ground equipment. Residues of chlorantraniliprole were quantitated by GC-ECD using a method based on DuPont 13294. Acceptable concurrent recovery data were obtained.

Residues in Bengal gram (chickpeas) pods are shown in Table 13.

Table 5 Residues from the foliar application of chlorantraniliprole to Bengal gram (chickpeas) in India

Country Year (Variety)	Application					Matrix	DAT	Chlorantraniliprole (mg/kg)	Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage				
GAP, India (chickpea)	2		25				11		
India	2	16	25	400	76 days after planting	Seed	20	< 0.03	Piriyadarsini
Jabalpur			25	400	Pod development				2010,
2010	2	16	50	400	76 days after planting	Seed	20	< 0.03	1004574,
JG-130			50	400	Pod development				Trial 1
India	2	34	25	500	25 days after planting	Seed	18	< 0.03	Piriyadarsini

Country Year (Variety)	Application					Matrix	DAT	Chlorantraniliprole (mg/kg)	Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage				
Raichur			25	500	Maturity				2010,
2009–2010	2	34	50	500	25 days after planting	Seed	18	< 0.03	1004574,
JG-11			50	500	Maturity				Trial 2
India	2	17	25	500	53 days after planting	Seed	23	< 0.03	Piriyadarsini
Andhra Pradesh			25	500	Pod formation				2010,
2009–2010	2	17	50	500	53 days after planting	Seed	23	< 0.03	1004574,
JG-11			50	500	Pod formation				Trial 3
India	2	10	25	500	83 days after planting	Seed	11	< 0.03	Piriyadarsini
West Bengal			25	500	Pod formation				2010,
2010	2	10	50	500	83 days after planting	Seed	11	< 0.03	1004574,
Anuradha			50	500	Pod formation				Trial 4

LOQ = 0.03 mg/kg

DAT = Days After Treatment

Supervised trials were carried out on mung beans (three trials—Table 6) in Australia during the 2012 growing season (Litzow 2013, DuPont-33763). Two foliar applications of a 350 g ai/kg formulation were made at 24.5 or 49–49.4 g ai/ha. A non-ionic surfactant was added. Applications were made to plots using spray volumes of 81–110 L/ha with ground equipment. In one trial application was made once at BBCH 65/71 and forage samples were taken. Residues of chlorantraniliprole were quantitated by LC-MS/MS using Method DuPont 13294. Acceptable concurrent recovery data were obtained.

Residues in mung bean trash and forage are shown in Table 14.

Table 6 Residues from the foliar application of chlorantraniliprole to mung beans in Australia

Country Year (Variety)	Application					Matrix	DAT	Chlorantraniliprole (mg/kg)	Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage				
GAP, Australia (mung bean)	2	7	24.5				14		
Australia	2	7	24.5	110	BBCH 81/84	Seed	–7 (0 DAT1)	0.18	Litzow 2013,
Wellcamp,			24.5	105	BBCH 84/87		–0	0.084	DuPont- 33763,
Queensland							0	0.42 <i>c</i> 0.01	Trial 110810
Crystal							3	0.30 <i>c</i> 0.037	
2012							7	0.49 <i>c</i> 0.015	
							14	0.26 <i>c</i> 0.051	
	2	7	49.4	110	BBCH 81/84	Seed	–7 (0 DAT1)	0.34	
			49.4	106	BBCH 84/87		–0	0.42	
							0	0.80 <i>c</i> 0.01	
							3	0.47 <i>c</i> 0.037	
							7	0.49, 0.72 (mean	

Country Year (Variety)	Application					Matrix	DAT	Chlorantraniliprole (mg/kg)	Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage				
								0.61) <i>c</i> 0.015	
							14	0.33 <i>c</i> 0.051	
Australia	2	7	24.5	82	BBCH 83/91	Seed	14	0.12, 0.063 (mean 0.092)	Litzow 2013,
Nandi, Queensland			24.5	81	BBCH 88/93			<i>c</i> 0.02, < 0.01	DuPont- 33763,
2012	2	7	49	82	BBCH 83/91	Seed	14	0.42, 0.17 (mean 0.30)	Trial 110811
Crystal			49	81	BBCH 88/93			<i>c</i> 0.02, < 0.01	
Australia	2	7	24.5	94	BBCH 84/92	Seed	14	0.17	Litzow 2013,
Bellata, New South			24.5	91	BBCH 87/94				DuPont- 33763,
Wales	2	7	49	93	BBCH 84/92	Seed	14	0.32	Trial 110812
2012			49	92	BBCH 87/94				
Crystal									

LOQ = 0.01 mg/kg

DAT = Days After Treatment

DAT 1 = Days After Treatment 1

Supervised trials were carried out on soya beans (four trials—Table 7) in Australia during the 2012 growing season (Litzow 2013, DuPont-33763). Two foliar applications of a 350 g ai/kg formulation were made at 24.2–24.5 or 49–49.4 g ai/ha. A non-ionic surfactant was added. Applications were made to plots using spray volumes of 82–106 L/ha with ground equipment. In two trials application was made once at BBCH 51/55 or 71/75 and forage samples were taken. Residues of chlorantraniliprole were quantitated by LC-MS/MS using Method DuPont 13294. Acceptable concurrent recovery data were obtained.

Residues in soya bean trash and forage are shown in Table 15.

Table 7 Residues from the foliar application of chlorantraniliprole to soya beans in Australia

Country Year (Variety)	Application					Matrix	DAT	Chlorantraniliprole (mg/kg)	Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage				
GAP, Australia (soya bean)	2	7	24.5				14		
Australia	2	6	24.5	106	BBCH 85/93	Seed	–6 (0 DAT1)	< 0.01	Litzow 2013,
Condamine Plains,			24.5	100	BBCH 86/93		–0	< 0.01	DuPont- 33763,
Queensland							0	< 0.01	Trial 110813
2012							3	0.021	
Fraser							7	< 0.01	
							14	<u>0.029</u>	
	2	6	49	106	BBCH 85/93	Seed	–6 (0 DAT1)	0.050	
			49	100	BBCH 86/93		–0	0.020	

Country Year (Variety)	Application					Matrix	DAT	Chlorantraniliprole (mg/kg)		Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage			Individual	Mean	
							0	0.040		
							3	0.056		
							7	0.022		
							14	0.022		
Australia	2	7	24.5	94	BBCH 83	Seed	14	< 0.01		Litzow 2013,
Forest Hill,			24.5	82	BBCH 87					DuPont- 33763,
Queensland	2	7	49	94	BBCH 83	Seed	14	0.013		Trial 110814
2012			49	82	BBCH 87					
Rose										
Australia	2	7	24.5	107	BBCH 83	Seed	14	< 0.01		Litzow 2013,
Murwillumbah,			24.5	106	BBCH 87					DuPont- 33763,
New South Wales	2	7	49.4	107	BBCH 83	Seed	14	< 0.01		Trial 110815
2012			49.4	106	BBCH 87					
A6785										
Australia	2	6	24.2	94	BBCH 75/98	Seed	14	< 0.01		Litzow 2013,
Narrabri,			24.2	89	BBCH 80/98					DuPont- 33763,
New South Wales	2	6	49.4	94	BBCH 75/98	Seed	14	0.027		Trial 110816
2012			49.4	93	BBCH 80/98					
Bunya										

LOQ = 0.01 mg/kg

DAT = Days After Treatment

DAT 1 = Days After Treatment 1

Cereal grains

Supervised trials were carried out on barley (three trials—Table 8) in the USA during the 2009 growing season (Dorschner 2012a, IR-4 10204, also submitted to JMPR 2013). Two foliar applications of a 200 g ai/L SC formulation were made 7 days apart at 112–117 g ai/ha. Applications were made to plots using spray volumes of 115–210 L/ha with ground equipment. Replicate samples were taken from each plot. Residues of chlorantraniliprole were quantitated by LC-MS/MS using a method based on DuPont 13294. Acceptable concurrent recovery data were obtained.

Residues in barley hay and straw are shown in Table 16.

Table 8 Residues from the foliar application of chlorantraniliprole to barley in the USA

Country Year (Variety)	Application					Matrix	DAT	Chlorantraniliprole (mg/kg)		Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage			Individual	Mean	
GAP, Canada (cereal grains)	3	7	75 (225/season)				1			
USA	2	7	117	116	Kernel hard	Grain	1	1.8	2.0	Dorschner 2012a,
Velva, North Dakota			115	115	Harvest			2.2		IR-4 10204,

Country Year (Variety)	Application					Matrix	DAT	Chlorantraniliprole (mg/kg)		Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage			Individual	Mean	
					ripe					
2009			232							09-ND01
Tradition										
USA	2	7	114	208	Mature grain	Grain	1	1.7	1.9	Dorschner 2012a,
Aurora, South Dakota			114	210	still hard			2.2		IR-4 10204,
2009			228		dough					09-SD07
Lacey					Mature					
USA	2	7	112	187	Drying down	Grain	1	1.9	1.9	Dorschner 2012a
Kimberley, Idaho			112	187	Drying down			1.9		IR-4 10204,
2009			224							09-ID14
Camas Spring										

LOQ = 0.01 mg/kg

DAT = Days After Treatment

Supervised trials were carried out on sorghum (three trials-Table 9) in the USA during the 2009 growing season (Dorschner 2012a, IR-4 10204, also submitted to JMPR 2013). Two foliar applications of a 200 g ai/L SC formulation were made 7 or 30 days apart at 111–114 g ai/ha. Applications were made to plots using spray volumes of 139–255 L/ha with ground equipment. Replicate samples were taken from each plot. Residues of chlorantraniliprole were quantitated by LC-MS/MS using a method based on DuPont 13294. Acceptable concurrent recovery data were obtained.

Residues in sorghum forage and stover are shown in Table 17.

Table 9 Residues from the foliar application of chlorantraniliprole to sorghum in the USA

Country Year (Variety)	Application					Matrix	DAT	Chlorantraniliprole (mg/kg)		Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage			Individual	Mean	
GAP, Canada (cereal grains)	3	7	75 (225/season)				1			
USA	2	30 ^a	112	139	Senesced via	Grain	1	1.2	1.2	Dorschner 2012a,
Fargo,			112	139	frost			1.1		IR-4 10204,
North Dakota			224		Ripe					09-ND02
2009										
LM 5001										
USA	2	7	112	172	Seeding	Grain	1	1.5	1.5	Dorschner 2012a,
Las Cruces,			114	194	Mature grain			1.5		IR-4 10204,
New Mexico			226							09-NM13
2009										
DK 28E										
USA	2	7	113	250	Hard dough	Grain	1	0.83	0.79	Dorschner 2012a,
Las Cruces,			111	255	to mature			0.74		IR-4 10204,

DAT = Days After Treatment

Residues in wheat forage, hay and straw are shown in Table 18. Processed fraction samples for analysis were generated from trial ND03 (Table 19).

[illegible]

LOQ = 0.01 mg/kg
DAT = Days After Treatment

Supervised trials were carried out on peanuts (six trials—Table 11) in the USA during the 2011 growing season. Two foliar applications of a 200 g ai/L SC formulation were made at 111–115 g ai/ha with an adjuvant added. Applications were made to plots using spray volumes of 117–234 L/ha with ground equipment. Peanut samples (one replicate per untreated plot and two replicates per treated plot) were collected at maturity, 1 day after the second application.

Table 11 Residues from the foliar application of chlorantraniliprole to peanuts in the USA

[illegible]

Country Year (Variety)	Application					Matrix	DAT	Chlorantraniliprole (mg/kg)		Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage			Individual	Mean	
USA	2	6	115	187	Mature	Nutmeat	1	0.022	0.034	Rice 2012,
Levelland, Texas			113	187	Mature			0.046		DuPont-31666,
2011			228							Trial 6
Tamnut OL06										

LOQ = 0.01 mg/kg

DAT = Days After Treatment

Animal Feeds

Note: Animal feed residues are expressed on a wet weight or 'as received' basis unless stated otherwise.

Table 12 Residues from the foliar application of chlorantraniliprole to chickpeas in Australia

Country Year (Variety)	Application					Matrix	DAT	Moisture Content (%)	Chlorantraniliprole (mg/kg)	Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage					
GAP, Australia (chickpea)	2	7	24.5				14			
Australia	2	7	24.5	78	BBCH 79/81	Trash	14	38.5	0.26 <i>c 0.01</i>	Litzow 2013,
Condamine Plains,			24.5	80	BBCH 88/93					DuPont-33763,
Queensland,	2	7	48.7	77	BBCH 79/81	Trash	14	25.3	0.82 <i>c 0.01</i>	Trial 110807
2011			48.7	79	BBCH 88/93					
Hatrick										
Australia	2	7	24.5	91	BBCH 82	Trash	5	49.1	1.8	Litzow
Bellata,			24.5	92	BBCH 85		7	31.5	1.1	2013,
New South							14	17.4	0.48	DuPont-
Wales						Forage	-7 (0 DAT1)	68.6	3.6	33763,
2011							-0	62.7	0.33	Trial
Hatrick							0	56.9	2.3	110808
	2	7	49.7	93	BBCH 82	Trash	5	52.6	4.6	
			49.7	92	BBCH 85		7	36.7	5.4	
							14	31.7	2.0	
						Forage	-7 (0 DAT1)	66.2	5.3	
							-0	61.3	2.7	
							0	63.1	7.2	
Australia	2	7	24.9	93	BBCH 86/91	Trash	14	13.3	0.27	Litzow 2013,
Narrabri,			24.9	90	BBCH 87/94					DuPont-33763,
New South Wales	2	7	49.0	91	BBCH 86/91	Trash	14	13.4	0.53	Trial 110809
2011			49.0	90	BBCH					

Country Year (Variety)	Application					Matrix	DAT	Moisture Content (%)	Chlorantraniliprole (mg/kg)	Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage					
					87/94					
Hatrick										

LOQ = 0.01 mg/kg for forage and 0.05 mg/kg for trash

Results for forage and trash are expressed on a dry weight basis

DAT = Days After Treatment

Table 13 Residues from the foliar application of chlorantraniliprole to Bengal gram (chickpeas) in India

Country Year (Variety)	Application					Matrix	DAT	Chlorantraniliprole (mg/kg)	Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage				
GAP, India (chickpea)	2		25				11		
India	2	16	25	400	76 days after planting	Pods	20	< 0.03	Piriyadarsini
Jabalpur			25	400	Pod development				2010,
2010	2	16	50	400	76 days after planting	Pods	20	< 0.03	1004574,
JG-130			50	400	Pod development				Trial 1
India	2	34	25	500	25 days after planting	Pods	18	< 0.03	Piriyadarsini
Raichur			25	500	Maturity				2010,
2009–2010	2	34	50	500	25 days after planting	Pods	18	< 0.03	1004574,
JG-11			50	500	Maturity				Trial 2
India	2	17	25	500	53 days after planting	Pods	23	< 0.03	Piriyadarsini
Andhra Pradesh			25	500	Pod formation				2010,
2009–2010	2	17	50	500	53 days after planting	Pods	23	< 0.03	1004574,
JG-11			50	500	Pod formation				Trial 3
India	2	10	25	500	83 days after planting	Pods	11	< 0.03	Piriyadarsini
West Bengal			25	500	Pod formation				2010,
2010	2	10	50	500	83 days after planting	Pods	11	< 0.03	1004574,
Anuradha			50	500	Pod formation				Trial 4

LOQ = 0.03 mg/kg

DAT = Days After Treatment

Table 14 Residues from the foliar application of chlorantraniliprole to mung beans in Australia

Country Year (Variety)	Application					Matrix	DAT	Moisture Content (%)	Chlorantraniliprole (mg/kg)	Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage					
GAP, Australia (mung bean)	2	7	24.5				14			
Australia	2	7	24.5	110	BBCH	Trash	-7 (0 DAT1)	69.0	3.1	Litzow

Country Year (Variety)	Application					Matrix	DAT	Moisture Content (%)	Chlorantraniliprole (mg/kg)	Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage					
Wellcamp,					81/84		–0	53.9	4.5	2013,
Queensland			24.5	105	BBCH		0	61.9	7.4	DuPont-
2012					84/87		3	63.0	6.4	33763,
Crystal							7	65.2	10	Trial
							14	61.0	3.8 <i>c 0.13</i>	110810
	1	–	24.5	83	BBCH	Forage	14	74.4	3.5	
					65/71					
	2	7	49.4	110	BBCH	Trash	–7 (0 DAT1)	69.2	10	
					81/84		–0	61.2	7.0	
			49.4	106	BBCH		0	65.5	14	
					84/87		3	63.2	12	
							7	64.5	15, 14 (mean 15)	
							14	62.9	8.3 <i>c 0.13</i>	
Australia Nandi,	2	7	24.5	82	BBCH 83/91	Trash	14	56.9, 57.2	2.7, 2.0 (mean 2.3) <i>c 0.081</i>	Litzow 2013,
Queensland 2012			24.5	81	BBCH 88/93					DuPont- 33763,
Crystal	2	7	49	82	BBCH 83/91	Trash	14	68.6, 58.6	3.3, 3.3 (mean 3.3) <i>c 0.081</i>	Trial 110811
			49	81	BBCH 88/93					
Australia Bellata,	2	7	24.5	94	BBCH 84/92	Trash	14	62.1	3.3	Litzow 2013,
New South Wales			24.5	91	BBCH 87/94					DuPont- 33763,
2012 Crystal	2	7	49	93	BBCH 84/92	Trash	14	57.7	11	Trial 110812
			49	92	BBCH 87/94					

LOQ = 0.01 mg/kg for forage and 0.05 mg/kg for trash

Results for forage and trash are expressed on a dry weight basis

Results in parentheses are for reserve samples

DAT = Days After Treatment

Table 15 Residues from the foliar application of chlorantraniliprole to soya beans in Australia

Country Year (Variety)	Application					Matrix	DAT	Moisture Content (%)	Chlorantraniliprole (mg/kg)	Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage					
GAP, Australia (soya bean)	2	7	24.5				14			
Australia	2	6	24.5	106	BBCH	Trash	–6 (0 DAT1)	28.5	1.1	Litzow
Condamine					85/93		–0	23.8	0.55	2013,
Plains,			24.5	100	BBCH		0	23.4	1.8	DuPont-
Queensland					86/93		3	35.6	1.1	33763,
2012							7	18.7	1.3	Trial

Country Year (Variety)	Application					Matrix	DAT	Moisture Content (%)	Chlorantraniliprole (mg/kg)	Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage					
Fraser							14	20.2 18.6	0.74, 1.7 (mean 1.2)	110813
	1	—	24.5	83	BBCH	Forage	14	74.4	0.68	
					51/55					
	2	6	49	106	BBCH	Trash	—6 (0 DAT1)	36.5	2.5	
					85/93		—0	28.2	1.6	
			49	100	BBCH		0	38.2	3.8	
					86/93		3	37.9	2.8	
							7	26.5	2.6	
							14	24.9, 26.3)	2.6, 2.7 (mean 2.7)	
Australia Forest Hill,	2	7	24.5	94	BBCH 83	Trash	14	17.7	0.68	Litzow 2013,
Queensland 2012			24.5	82	BBCH 87					DuPont- 33763,
Rose	1	—	24.5	83	BBCH	Forage	14	76.3	0.69	Trial
					71/75					110814
	2	7	49	94	BBCH 83	Trash	14	17.6	1.7	
			49	82	BBCH 87					
Australia	2	7	24.5	107	BBCH 83	Trash	14	35.5	0.36	Litzow
Murwillumbah,			24.5	106	BBCH 87					2013,
New South Wales	2	7	49.4	107	BBCH 83	Trash	14	44.4	0.90	DuPont-
2012			49.4	106	BBCH 87					33763,
A6785										Trial
										110815
Australia Narrabri,	2	7	24.2	94	BBCH 75/98	Trash	14	9.7	0.96	Litzow 2013,
New South Wales 2012			24.2	89	BBCH 80/98					DuPont- 33763,
Bunya	2	7	49.4	94	BBCH 75/98	Trash	14	9.5	1.8	Trial 110816
			49.4	93	BBCH 80/98					

LOQ = 0.01 mg/kg for forage and 0.05 mg/kg for trash

Results for forage and trash are expressed on a dry weight basis

Results in parentheses are for reserve samples

DAT = Days After Treatment

DAT 1 = Days After Treatment 1

Table 16 Residues from the foliar application of chlorantraniliprole to barley in the USA

Country Year (Variety)	Application					Matrix	DAT	Chlorantraniliprole (mg/kg)		Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage			Individual	Mean	
GAP, Canada (cereal grains)	3	7	75 (225/season)				1			
USA	2	8	115	115	Flag leaf	Hay	1	9.2	9.2	Dorschner 2012a,
Velva, North Dakota			<u>113</u>	113	Head			9.3		IR-4 10204,
			228		emergence					09-ND01
2009					complete					
Tradition	2	7	117	116	Kernel hard	Straw	1	13	14	
			<u>115</u>	115	Harvest			15		
			232		ripe					
USA	2	7	113	205	Milk	Hay	1	5.5	5.5	Dorschner 2012a,
Aurora,			<u>121</u>	217	Soft dough			5.5		IR-4 10204,
South Dakota			234							
2009	2	7	114	208	Mature grain	Straw	1	3.3	3.6	09-SD07
Lacey			<u>114</u>	210	still hard			3.8		
			228		dough					
					Mature					
USA	2	7	110	184	Milk	Hay	1	9.5	11	Dorschner 2012a,
Kimberley, Idaho			<u>111</u>	186	Milk to			12		IR-4 10204,
2009			221		soft dough					09-ID14
Camas Spring	2	7	112	187	Drying down	Straw	1	12	12	
			<u>112</u>	187	Drying down			12		
			224							

LOQ = 0.01 mg/kg

DAT = Days After Treatment

Table 17 Residues from the foliar application of chlorantraniliprole to sorghum in the USA

Country Year (Variety)	Application					Matrix	DAT	Chlorantraniliprole (mg/kg)		Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage			Individual	Mean	
GAP, Canada (cereal grains)	3	7	75 (225/season)				1			
USA	2	7	112	139	Early dough	Forage	1	3.0	2.7	Dorschner 2012a,
Fargo, North Dakota			<u>112</u>	139	Soft dough			2.4		IR-4 10204,
			224							
2009	2	30 ^a	112	139	Senesced via	Stover	1	3.3	3.4	09-ND02
LM 5001			<u>112</u>	139	frost			3.6		
			224		Ripe					

Country Year (Variety)	Application					Matrix	DAT	Chlorantraniliprole (mg/kg)		Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage			Individual	Mean	
USA	2	7	111	140	Milk,	Forage	1	4.7	4.1	Dorschner 2012a,
Las Cruces,			<u>114</u>	170	soft dough			3.4		IR-4 10204,
New Mexico			225		Soft dough					09-NM13
2009	2	7	112	172	Seeding	Stover	1	6.9	5.9	
DK 28E			<u>114</u>	194	Mature grain			4.9		
			226							
USA	2	7	122	242	Early milk	Forage	1	3.2	3.4	Dorschner 2012a,
Las Cruces,			<u>111</u>	235	Soft to			3.5		IR-4 10204,
New Mexico			235		hard dough					09-NM19
2009	2	7	113	250	Hard dough	Stover	1	4.8	4.1	
M3838			<u>111</u>	255	to mature			3.4		
			224		Mature grain					

LOQ = 0.01 mg/kg

^a Second application made 30 days after first application because of prolonged wet and cold weather

DAT = Days After Treatment

Table 18 Residues from the foliar application of chlorantraniliprole to wheat in the USA

Country Year (Variety)	Application					Matrix	DAT	Chlorantraniliprole (mg/kg)		Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage			Individual	Mean	
GAP, Canada (cereal grains)	3	7	75 (225/season)				1			
USA	2	7	113	140	Beginning	Forage	1	4.2	4.3	Dorschner 2012a,
Fargo,			<u>113</u>	140	anthesis			4.3		IR-4 10204,
North Dakota			226		Late anthesis	Hay	1	8.6	9.5	09-ND04
2009								10		
Glenn	2	7	113	140	Hard dough	Straw	1	5.4	4.7	
			<u>112</u>	139	Ripe wheat			4.0		
			225							
USA	2	8	115	115	Flag leaf	Forage	1	4.0	4.4	Dorschner 2012a,
Velva,			<u>114</u>	114	Heading			4.8		IR-4 10204,
North Dakota			229			Hay	1	9.2	8.6	09-ND05
2009								8.0		
Faller	2	7	118	117	Kernel hard	Straw	1	15	15	
			<u>115</u>	115	Ripe for			15		
			233		cutting					

Country Year (Variety)	Application					Matrix	DAT	Chlorantraniliprole (mg/kg)		Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage			Individual	Mean	
USA	2	7	113	203	Boot to	Forage	1	5.0	4.3	Dorschner 2012a,
Aurora, South			115	204	flowering			3.7		IR-4 10204,
Dakota			228		Flowering	Hay	1	11	11	09-SD08
2009								12		
Briggs Hard Red	2	7	120	211	Mature	Straw	1	6.5	6.4	
			106	193	Mature			6.3		
			226							
USA	2	6	115	191	Early boot	Forage	1	4.6	4.6	Dorschner 2012a,
Las Cruces,			114	209	Late boot			4.6		IR-4 10204,
New Mexico			229			Hay	1	11	11	09-NM18
2010								10		
El Dorado	2	7	117	232	Hard dough,	Straw	1	4.2	4.5	
			118	252	mature			4.8		
			235		Mature grain					

LOQ = 0.01 mg/kg

DAT = Days After Treatment

FATE OF RESIDUES IN STORAGE AND PROCESSING

Residues after processing

Processing studies are necessary according to the uses and the residues of chlorantraniliprole on raw agricultural commodities. The fate of chlorantraniliprole residues during processing of raw agricultural commodities was investigated in wheat.

As a measure for the transfer of residues into processed products, a processing factor (PF) was used, defined as:

$$\text{PF} = \frac{\text{Residue in processed products (mg/kg)}}{\text{Residue in raw agricultural commodity (mg/kg)}}$$

Wheat

The effect of processing (laboratory scale) on residues of chlorantraniliprole in wheat was investigated in a trial conducted in the USA in the 2009 growing season (Dorschner 2012a, IR-4 10204). Wheat with incurred residues was obtained where plants were sprayed twice at 114 and 112 g ai/ha. Applications were made in 122 or 124 L/ha using ground equipment. Wheat bulk RAC samples were harvested 1 day after the second application.

Bulk wheat grain samples were processed according to simulated commercial procedures into the following samples: aspirated grain fractions, germ, middlings, flour, shorts and bran.

The moisture content of grain was determined and the grain dried in an oven set at 43–57 °C until the moisture content was 10–13%. The grain was transferred to the dust generation room and moved through the conveyor system for 120 minutes to remove the grain dust with an aspirator. Aspirated grain was cleaned with different sized screens to remove broken grain and foreign material. Cleaning yielded grain dust, screenings and cleaned grain. The aspirated grain fraction (grain dust) sample was collected and placed in frozen storage.

Cleaned grain was further processed into germ, middlings, flour, shorts and bran. For wheat germ, a portion of the cleaned grain was conditioned with water for 1 to 1.5 hours to adjust the moisture content to approximately 16%. The conditioned grain was passed through a mill and sifted with three different sized sieves. Material passing through all three sieves was aspirated to remove bran from the germ. The germ with endosperm was passed through a reduction mill and sifted to separate the germ from the endosperm. The germ was aspirated again to remove remaining bran and milled and sieved again to remove remaining endosperm. The germ fraction sample was collected and placed in frozen storage.

The remaining cleaned grain was mixed with water for at least 15 minutes and moisture conditioned according to the physical property of the wheat. The conditioned grain was passed through a mill, broken with three break rolls and sifted through two different sized screens. Break flour passed through the smaller screen and middlings passed through the larger screen. Coarse bran did not pass through either screen. A middlings fraction sample was collected and placed in frozen storage.

The remaining middlings were poured into the reductions system, passed through two reduction rolls and sifted. Reduction flour passed through the sifter, while shorts did not. The break flour and reduction flour were poured into an agitator and mixed for 15 minutes. The recombined flour fraction sample was collected and placed in frozen storage. The coarse bran was placed in a bran finisher and conveyed over a screen. Shorts passed through the screen and bran passed over the screen. The shorts from the bran finisher were added to the shorts from the reduction system to produce the shorts fraction sample, which was placed in frozen storage. The bran fraction sample was collected and placed in frozen storage.

Residues of chlorantraniliprole in wheat RAC and processed commodities were determined by LC/MS/MS using Method 13294. Acceptable concurrent recovery data were obtained for all wheat commodities.

Table 19 Residues of chlorantraniliprole in wheat and processed commodities

Country Year (Variety)	Application					Matrix	DAT	Chlorantraniliprole (mg/kg)		Processing Factor	Author, Study No., Trial No.
	No	Interval (Days)	g ai/ha	Water (L/ha)	Growth Stage			Individual	Mean		
GAP, USA (wheat)	3		75 (225/season)				1				
USA	2	7	114	124	Hard dough	Grain	1	0.22, 0.23	0.23		Dorschner
Fargo, North Dakota 2009			<u>112</u>	122	Ripe wheat	AGF		7.3, 7.9	7.6	33	2012a,
			226			Germ		0.26, 0.26	0.26	1.13	IR-4
						Middlings		0.064, 0.066	0.065	0.28	10204,
Alsen						Flour		0.088, 0.088	0.088	0.38	09-ND03
						Shorts		0.18, 0.14	0.16	0.7	
						Bran		0.24, 0.24	0.24	1.04	

AGF = Aspirated Grain Fractions

DAT = Days After Treatment

APPRAISAL

Chlorantraniliprole was first evaluated for residues and toxicological aspects by the 2008 JMPR. The 2008 JMPR established an ADI for chlorantraniliprole of 0-2 mg/kg bw and concluded that an ARfD was unnecessary. It was also evaluated in 2010 and 2013 for additional maximum residue levels. At the Forty-fifth Session of the CCPR (2013), chlorantraniliprole was listed for consideration of further additional maximum residue levels by the 2014 JMPR.

The Meeting received information on registered use patterns, supervised residue trials and fate of residues in processing. Product labels were available from Australia, Canada, India, the Republic of South Africa and the United States of America.

The residue definition for compliance with MRL and for dietary intake for plant and animal commodities is chlorantraniliprole. The residue is considered fat soluble.

Methods of analysis

Residue trial samples were analysed using LC-MS/MS methods based on those previously evaluated by the JMPR in 2008.

Stability of pesticide residues in stored analytical samples

Samples from the submitted studies were stored for periods less than the period of stability demonstrated in studies provided to the 2008 Meeting. Since the storage stability data from the 2008 JMPR cover a diverse range of crops and demonstrated stability of chlorantraniliprole for up to 2 years, it is considered that these data should be sufficient to cover the storage stability of all commodities in this submission.

The Meeting noted that concurrent storage stability data provided with the green onion residue trials also demonstrated stability of chlorantraniliprole residues over 24 months (the period for which the samples were stored) in fresh and dried green onions.

Results of supervised residue trials on crops

The Meeting received supervised trial data for application of chlorantraniliprole on oranges, mandarins, green onions (fresh and dried), chickpeas, mung beans and soya beans, barley, grain sorghum, wheat and peanuts.

Citrus Fruits

Residue trials were conducted in citrus fruits in the Republic of South Africa (RSA) in 2010 according to the critical GAP in the RSA (up to 2 applications at 3.5 g ai/ 100L, and a 7 day PHI).

Four trials were conducted in oranges and four trials in mandarins. In one orange trial the rate of the second application was not known, so data from this trial were not considered for estimation of a maximum residue level and STMR.

The Meeting noted that the RSA GAP is for the citrus fruit group and that a group maximum residue level of 0.5 mg/kg for chlorantraniliprole in citrus fruits was estimated at the 2010 JMPR Meeting based on 2009 South African trials in oranges (4) and mandarins/ tangelos (4). An STMR of 0.07 mg/kg was estimated.

The new citrus data were combined with the 2009 data to give a larger data set on which to base an estimation of the maximum residue level and STMR.

The ranked order of residues in oranges (whole fruit) from supervised trials in the RSA in 2009 and 2010 according to GAP was: **0.14**, 0.15, **0.15**, 0.22, 0.22, **0.24** and 0.27 mg/kg (*new data in bold italics*).

The ranked order of residues in mandarins and tangelos (whole fruit) from supervised trials in the RSA in 2009 and 2010 according to GAP was: **0.11**, 0.14, **0.15**, 0.18, 0.22, 0.25, **0.30** and 0.35 mg/kg (*new data in bold italics*).

The Meeting noted that the RSA GAP is for the citrus group and considered a group maximum residue level. To consider a group maximum residue level, residues across individual crops should not differ by more than 5×median. The Meeting noted that the median of the oranges and mandarins/ tangelos differed by less than 5-fold (only a 1.1-fold difference).

In deciding whether to combine the datasets for oranges and mandarins/ tangelos for use in the statistical calculator or to only utilise the data from the commodity with the highest residues, the Meeting recognised the similarity of the datasets (Mann-Whitney U-Test). Therefore the Meeting decided to combine the data from oranges and mandarins/ tangelos in order to estimate a maximum residue level for citrus fruit.

The ranked order of residues in oranges and mandarins/ tangelos (whole fruit) from supervised trials in the RSA in 2009 and 2010 according to GAP was: 0.11, 0.14 (2), 0.15 (3), 0.18, 0.22 (3), 0.24, 0.25, 0.27, 0.30 and 0.35 mg/kg.

The ranked order of residues in oranges and mandarins/ tangelos (edible portion - flesh) from supervised trials in the RSA in 2009 and 2010 according to GAP was: 0.02, 0.04, 0.05 (4), 0.06 (3), 0.07 (2), 0.08 (2), 0.09 and 0.11 mg/kg.

The Meeting estimated a maximum residue level of 0.7 mg/kg for residues of chlorantraniliprole in citrus fruits, together with an STMR of 0.06 mg/kg (based on the edible portion data). The Meeting estimated a median residue for whole citrus fruit of 0.22 mg/kg for use in processing calculations.

The Meeting withdrew its previous recommendation of 0.5 mg/kg for chlorantraniliprole in citrus fruits.

Bulb Vegetables – green onion

The GAP for bulb vegetables in the USA is for up to 4 applications at a maximum rate of 73 g ai/ha, or a maximum of 224 g ai/ ha, with a 7 day retreatment interval and a PHI of 1 day.

Residue trials were conducted in green onions in the USA (3 trials) and Canada (2 trials) in which two applications of chlorantraniliprole were made at 110 – 118 g ai/ ha (223-228 g ai/ ha per crop) with a 3 day retreatment interval and a PHI of 1 day.

The Meeting did not estimate a maximum residue level as the trials were not in accordance with the GAP.

Pulses – chickpeas

The critical GAP in India is 2 applications at 25 g ai/ ha and an 11-day PHI.

Four residue trials were conducted in chickpea (Bengal gram) in India in which two foliar applications of chlorantraniliprole were made at 25 or 50 g ai/ha. The PHI was 11 - 23 days.

Only one trial matches the Indian GAP. The observed residues were < 0.03 mg/kg.

The Meeting determined that a single trial was insufficient for estimation of a maximum residue level.

Three residue trials were conducted in chickpeas in Australia according to the GAP in Australia (2 applications at 24.5 g ai/ ha, 7 day retreatment interval and a 14 day PHI).

The ranked order of residues from supervised trials in Australia according to GAP was:

< 0.01, 0.015 and 0.025 mg/kg.

The Meeting decided that the number of trials available was not adequate to estimate a maximum residue level for chickpeas (dry).

Pulses – mung beans

Residue trials were conducted in mung beans in Australia according to the critical GAP in Australia (2 applications at 24.5 g ai/ ha, 7 day retreatment interval and a 14 day PHI).

The ranked order of residues from supervised trials in Australia according to GAP was: 0.092, 0.17 and 0.26 mg/kg.

The Meeting concluded that the number of trials available was not adequate to estimate a maximum residue level for mung beans (dry).

Pulses – soya beans

The GAP for soya beans in Australia is 2 applications at 24.5 g ai/ ha, 7-day retreatment interval, and a 14 day PHI.

Residue trials were conducted in soya beans in Australia.

The ranked order of residues from supervised trials in Australia according to GAP was:

≤ 0.01 (3) and 0.029 mg/kg.

Residue trials conducted in soya beans in Japan which were considered at the time of the 2010 JMPR showed that residues in dry soya beans were < 0.01 (2) mg/kg after 3 applications at 25 g ai/ ha at 7 day intervals and with a 14 day PHI. These trials match the Australian GAP, with the exception of three rather than two applications being made. However, the Meeting noted that the additional application had no effect on the residues in the Japanese trials, which were below the LOQ.

The Australian and Japanese soya bean data were combined and the ranked order of residues from supervised trials in Australia and Japan according to Australian GAP was:

≤ 0.01 (5) and 0.029 mg/kg.

The Meeting estimated a maximum residue level and an STMR value of 0.05 and 0.01 mg/kg respectively for chlorantraniliprole in soya beans.

Cereals

The Codex MRL for chlorantraniliprole in cereal grains is 0.02 mg/kg following the recommendation of the 2008 JMPR based on rotational crop data. An STMR of 0.01 mg/kg was estimated.

A study conducted on cereals in the USA in 2009 - 2010 (three trials in barley and sorghum and five in wheat) was submitted to the 2013 JMPR. As the compound was not registered in the USA for these crops, no estimations of maximum residue levels or STMRs were made. The study has been resubmitted, with relevant registered label use patterns in the USA and Canada for cereal grains except corn and wild rice, and is evaluated here against the critical Canadian GAP.

The GAP for cereals in Canada is 3×75 g ai/ha applications, with a 7-day retreatment interval and a 1-day PHI.

However, the submitted cereal trials were conducted with 2×111 -117 g ai/ha applications (RTI 7 days, PHI 1 day). The Meeting therefore did not estimate maximum residue levels for cereal grains as the trials were not conducted in accordance with the GAP.

Oilseeds – peanuts

The GAP in the USA is up to 4 applications at a rate of 73g ai/ha (or a maximum of 224 g ai/ha/ year) with a 3 day retreatment interval and a PHI of 1 day.

Six residues trials were conducted in peanuts in the USA in which two applications of chlorantraniliprole were made at 111–115 g ai/ ha (total application rate of 224–228 g ai/ha) with a 5-6 day retreatment interval and a PHI of 1 day.

A maximum residue level and STMR were not estimated as the trials were not conducted in accordance with the GAP.

Animal feeds

The Meeting received supervised trials data for chickpea, mung bean and soya bean forage, barley hay and straw, grain sorghum forage and stover and wheat forage, hay and straw.

Pulse forage

The GAP in Australia for chickpea, mung bean and soya bean is 2×24.5 g ai/ha applications with a 14-day grazing PHI.

Data for mung bean, chickpea and soya bean forage is available from the Australian trials, but does not match GAP as only one application was made, while in the chickpea trials, forage was not sampled at the correct PHI. The Meeting therefore did not estimate median and highest residues for pulse forages.

Cereals forages and fodders

Residue data for sorghum and wheat forage, barley and wheat hay, wheat straw and sorghum stover were received. The Meeting determined that the trials did not match the Canadian GAP, and maximum residue levels and median and highest residues were not estimated.

Fate of residues during processing

The Meeting received a processing study for wheat. STMR-P values were estimated for wheat grain processed commodities using the cereal grains STMR value of 0.01 mg/kg estimated by the 2008 Meeting based on rotational crop data (see table below).

A processing study for orange processing into juice was considering by the 2010 Meeting (see table below).

Processing Factors for Chlorantraniliprole from the Processing of Raw Agricultural Commodities (RACs)

RAC	Processed Commodity	Best Estimate Processing Factor	RAC MRL	RAC STMR	Processed Commodity STMR-P/median residue
Wheat	Aspirated Grain Fractions	33	0.02	0.01	0.34
	Bran	1.04			0.011
	Flour	0.38			0.004
	Middlings	0.28			0.003
	Shorts	0.7			0.007
	Germ	1.13			0.011
Oranges	Juice	0.17	0.7	0.22	0.037

Animal commodities

The Meeting recalculated the livestock dietary based on the uses considered by the current Meeting and by the 2008, 2010 and 2013 Meetings on the basis of diets listed in the FAO Manual Appendix IX (OECD Feedstuff Table).

The maximum dietary burdens are 36.1 ppm for beef cattle and 29.0 ppm for dairy cattle, while the mean dietary burdens are 17.4 ppm for beef cattle and 13.6 ppm for dairy cattle. These values have changed only marginally from those calculated by the 2013 Meeting (beef cattle maximum/mean of 31.7/15.7 ppm, and dairy cattle maximum/mean of 26.8/13.1 ppm). The maximum and mean dietary burdens for poultry were unchanged from those previously calculated.

The Meeting confirmed its previous recommendations for maximum residue levels and STMR values for meat from mammals other than marine mammals, milks, edible offal (mammalian), poultry meat, poultry, edible offal of, and eggs.

The Meeting noted that maximum residue levels have not previously been estimated for mammalian fats and poultry fats.

The Meeting noted that the 2010 Meeting estimated a maximum residue level of 0.2 mg/kg for meat (from mammals other than marine mammals) of 0.2 mg/kg (fat), together with STMR values of 0.049 mg/kg in fat and 0.009 mg/kg in muscle. The dietary burden has not changed significantly since. The Meeting estimated a maximum residue level of 0.2 mg/kg for mammalian fats (except milk fats), together with an STMR of 0.049 mg/kg.

The Meeting noted that the 2013 Meeting estimated a maximum residue level of 0.01* mg/kg (fat) and an STMR of 0 for poultry meat. The dietary burden has not changed significantly. The Meeting estimated a maximum residue level of 0.01* mg/kg for poultry fats, together with an STMR of 0.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI assessment.

Definition of the residue (for compliance with the MRL and for estimation of dietary intake) for plant and animal commodities: *chlorantraniliprole*

The residue is fat soluble

The Meeting estimated the maximum residue levels and STMR values shown below.

Commodity		MRL, mg/kg		STMR or STMR-P, mg/kg
CCN	Name	New	Previous	
FC 0001	Citrus Fruits	0.7	0.5	0.06
	Citrus fruit juice	-	-	0.037
MF 0100	Mammalian fats (except milk fats)	0.2	-	0.049
PF 0111	Poultry fats	0.01*	-	0
VD0541	Soya bean (dry)	0.05	-	0.01
CF 0654	Wheat bran, processed	-	-	0.011
CF 1211	Wheat flour	-	-	0.004
CF 1210	Wheat germ	-	-	0.012

Commodity		MRL, mg/kg	Highest residue, mg/kg	Median residue, mg/kg	Comments
CCN	Name				
-	Wheat aspirated grain fractions	-	-	0.34	Feedstuff
-	Wheat middlings	-	-	0.003	Feedstuff
-	Wheat shorts	-	-	0.007	Feedstuff

DIETARY RISK ASSESSMENT

Long-term intake

The evaluation of chlorantraniliprole has resulted in recommendations for MRLs and STMRs for raw and processed commodities. The International Estimated Daily Intakes for the 17 GEMS/Food cluster diets, based on STMRs estimated by this Meeting and the 2008, 2010 and 2013 Meetings were in the range 0-1 % of the maximum ADI of 2 mg/kg bw (Annex 3).

The Meeting concluded that the long-term intake of residues of chlorantraniliprole from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The 2008 JMPR decided that an ARfD was unnecessary and concluded that the short-term intake of residues of chlorantraniliprole is unlikely to present a public health concern.

REFERENCES

Code	Authors	Year	Title, Institute, Report reference
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IR-4 A10204	Dorschner, K	2012b	Chlorantraniliprole: Magnitude of the residue on green onion (fresh and dried) and dill seed. IR-4 Project, Princeton, New Jersey, USA. DuPont Report No IR-4 A10204. Unpublished.
DuPont-33763	Litzow, D	2013	Determination of chlorantraniliprole residues in pulse crops following foliar application of Altacor, Australia, 2011/2012. Agrisearch Services Pty Ltd, Orange, New South Wales, Australia. DuPont Report No DuPont-33763. Unpublished.
1004574	Piriyadarsini, JR	2010	Studies on the residues of chlorantraniliprole 20% SC w/v in Bengal gram (<i>Cicer arietinum</i> L) and Soil (Multi-Location Study). International Institute of Biotechnology and Toxicology (IIBAT). Dupont Report No. II BAT 1004574. Unpublished.
DuPont-31666	Rice, F	2012	Magnitude of chlorantraniliprole residues in peanuts following foliar application with chlorantraniliprole (DPX-E2Y45) 20SC [200 g/L (w/v); 18.4% (w/w)], U.S., 2011. ABC Laboratories, Inc., Columbia, Missouri, USA. DuPont Study No DuPont-31666. Unpublished.
2418/D924	Van Zyl, PFC	2010	Chlorantraniliprole residue study on citrus commodities conducted in the RSA during the 2009/2010 citrus-growing season for support in the acquisition of an EU import tolerance. Du Pont de Nemours International, Centurion, Republic of South Africa. DuPont Report No 2418/D924. Unpublished.

CLOTHIANIDIN (238)

First draft prepared by Ms T. van der Velde-Koerts, Centre for Nutrition, Prevention and Health Services (VPZ), National Institute for Public Health and the Environment (RIVM), The Netherlands

APPRAISAL

Clothianidin is reconsidered by the Meeting because new data for thiamethoxam became available and clothianidin is a metabolite (sometimes referred to as CGA322704) of thiamethoxam.

Clothianidin was evaluated for toxicology and residues as a new compound in 2010, resulting in a number of MRL recommendations. Additional residue data were evaluated in 2011 and 2012. The residue definition for clothianidin in plant commodities for enforcement and dietary risk assessment is clothianidin.

The 2010 Meeting established an acceptable daily intake (ADI) of 0–0.1 mg/kg bw per day and established the acute reference dose (ARfD) as 0.6 mg/kg bw for clothianidin. Clothianidin residues can also arise from thiamethoxam use (metabolite CGA322704). Therefore, both compounds need to be considered if residue data in support of new uses are submitted. The residue definition in plant commodities for enforcement is thiamethoxam, while the residue definition for dietary risk assessment is thiamethoxam and the metabolite CGA322704 (clothianidin), considered separately.

At the 2014 JMPR Meeting residue data were submitted to support the use of thiamethoxam on use avocado, mango, beans, mint, hops, and persimmon.

The Meeting received analytical methods and supervised trials data for the use of thiamethoxam on avocado, mango, beans, mint, hops and persimmon as well as processing data on mango and mint. The resulting CGA322704 data relevant for maximum residue level recommendations of clothianidin are summarized below.

Combination of residues from clothianidin use and thiamethoxam use

Clothianidin residues may arise from use of clothianidin as well as from use of thiamethoxam (metabolite CGA322704).

The Meeting considered it unlikely that both pesticides are used on the same crop and therefore the maximum estimated residue levels, the maximum STMR, and the maximum HR of each use is taken as recommendation.

Summary of residue data of clothianidin following use of thiamethoxam in relation to clothianidin use reviewed by the 2014 Meeting.

CCN	Commodity name	Origin of use	MRL mg/kg	STMR or STMR-P mg/kg	HR or HR-P mg/kg
FI 0326	Avocado	thiamethoxam	0.03 T	0.01 T	0.02 T
FI 0345	Mango	thiamethoxam	0.04 T	0.02 T	0.02 T
DH 1100	Hops	thiamethoxam	0.07 T	0.026 T	0.028 T
HH 0738 ^e	Mints	thiamethoxam	0.3 T	0.11 T	0.12 T
VP 0061	Beans with pods	thiamethoxam	0.2 T	0.07 T	0.10 T
	Bean forage	thiamethoxam		0.075 T	0.11 T

T = Thiamethoxam use

Fate of residues during processing

Processing studies were undertaken for the use of thiamethoxam on mango and mint (no known use of clothianidin). Processing factors based on the residue for the metabolite clothianidin are listed in the table below. Using the STMR_{RAC} obtained from the thiamethoxam use, the Meeting estimated STMR-Ps for processed commodities to be used in dietary intake calculations.

Commodity	PFs	PF (median or best estimate)	STMR-P = STMR _{RAC} x PF (mg/kg)	HR-P = HR _{RAC} x PF (mg/kg)
	Metabolite CGA322704 (clothianidin) (STMR _{pulp+peel} = 0.02 mg/kg, HR _{pulp+peel} = 0.02 mg/kg)			
Mango, dried flesh	5.67, 8.40, 7.00, 4.00	6.3	0.13	0.13
Mint, oil	< 0.22, < 0.19	< 0.20	n.a.	n.a.

Livestock dietary burden

The additional data on residue levels of clothianidin in bean forage from thiamethoxam use (JMPR 2014) warranted new dietary burden calculations for clothianidin.

The Meeting estimated the dietary burden of clothianidin on the basis of the livestock diets listed in the FAO manual appendix IX (OECD feedstuff table) using the OECD_Feed_Calculator_V1_4. Calculation from highest residue, STMR (some bulk commodities) and STMR-P values provides the levels in feed suitable for estimating MRLs, while calculation from STMR and STMR-P values from feed is suitable for estimating STMR values for animal commodities. The 2014 JMPR Meeting recalculated the livestock dietary burden of clothianidin through clothianidin and thiamethoxam use based on the uses presented by the 2010 Meeting and including the residue values for fresh bean forage from the 2014 JMPR Meeting on thiamethoxam.

The new dietary burdens calculations of clothianidin for beef cattle, dairy cattle, broilers and laying poultry are provided in Annex 6. The calculations were made according to the livestock diets from US/CAN, EU, Australia and Japan in the OECD Feed Table 2009.

Livestock dietary burden, clothianidin, ppm of dry matter

	US	EU	AU	JP	overall	
	Max	Max	Max	Max	max	
Beef cattle	0.30	0.80	1.1	0.027	1.1	^a
Dairy cattle	0.28	0.61	0.87	0.051	0.87	^b
Poultry – broiler	0.050	0.21	0.094	0.022	0.21	
Poultry – layer	0.050	0.26	0.094	0.018	0.26	^c
	Mean	Mean	Mean	Mean	Mean	
Beef cattle	0.088	0.17	0.75	0.024	0.75	^a
Dairy cattle	0.12	0.17	0.61	0.033	0.61	^b
Poultry – broiler	0.050	0.040	0.094	0.017	0.094	
Poultry – layer	0.050	0.070	0.094	0.018	0.094	^c

^a Highest mean and maximum beef or dairy cattle dietary burden suitable for maximum residue level and STMR estimates for mammalian meat.

^b Highest mean and maximum dairy cattle dietary burden suitable for maximum residue level and STMR estimates for milk.

^c Highest mean and maximum poultry dietary burden suitable for maximum residue level and STMR estimates for poultry meat and eggs.

Animal commodities maximum residue level estimation

Clothianidin residues in animal commodities from both thiamethoxam (metabolite CGA322704) and clothianidin use are considered below.

Cattle-clothianidin residues from thiamethoxam use

The residues of clothianidin were evaluated in the same way as described for thiamethoxam (see appraisal 2010) for maximum residue level estimation, the highest residue in the tissues and milk were calculated by interpolating the maximum dietary burden (6.1 ppm) between the relevant feeding levels (6-20 ppm) from the dairy cow feeding study and using the highest tissue concentrations based on the residue definition for enforcement from individual animals within those feeding groups and using the mean milk concentration from those feeding groups (see table below).

The STMR values for the tissues and milk were calculated by interpolating the mean dietary burden (2.4 ppm) between the relevant feeding levels (2-6 ppm) from the dairy cow feeding study and using the mean tissue and milk concentrations based on the residue definition for dietary risk assessment from those feeding groups (see table below).

	Feed level (ppm) for milk residues	Total residues in milk (mg/kg)	Feed level (ppm) for tissue residues	Total residue (mg/kg) in			
				muscle	Liver	kidney	fat
Maximum residue level – beef or dairy cattle							
Feeding study	6, 20	0.013, 0.043	6, 20	< 0.01, < 0.01	0.14, 0.38	< 0.01, 0.04	< 0.01, < 0.01
Dietary burden and residue estimate	6.1	0.013	6.1	< 0.01	0.14	< 0.01	< 0.01
STMR – beef or dairy cattle							
Feeding study	2, 6	0.005, 0.013 ^a	2, 6	< 0.01, < 0.01	0.049, 0.14	< 0.01, < 0.01	< 0.01, < 0.01
Dietary burden and residue estimate	2.4	0.0060	2.4	< 0.01	0.058	< 0.01	< 0.01

^a Calculated mean (day 3-26), data retrieved from JMPR 2010 evaluation

Cattle-clothianidin residues from clothianidin use

In a feeding study where lactating cows were dosed with clothianidin at up to 2.6 ppm dry feed, no clothianidin was found in tissues (< 0.02 mg/kg). Therefore, no residues are to be expected in tissues at the mean and maximum calculated dietary burden of 0.75 and 1.1 ppm based on the new clothianidin dietary burden calculation.

For milk MRL estimation, the highest residues in the milk resulting from dietary burden based on clothianidin were calculated by interpolating the maximum dietary burden for dairy cattle (0.87 ppm) between the relevant feeding levels (0.8 and 2.6 ppm) from the dairy cow feeding study and using the mean milk concentration from those feeding groups.

For milk STMR estimation, the median residues in the milk resulting from dietary burden were calculated by interpolating the mean dietary burden for dairy cattle (0.61 ppm) between the relevant feeding levels (0.27 and 0.80 ppm) from the dairy cow feeding study and using the mean milk concentration from those feeding groups.

	Feed level (ppm) for milk residues	Total residues in milk (mg/kg)
Maximum residue level – dairy cattle		
Feeding study	0.80, 2.6	0.0020, 0.012
Dietary burden and residue estimate	0.87	0.002
STMR – dairy cattle		
Feeding study	0.27, 0.80	< 0.0020, < 0.0020
Dietary burden and residue estimate	0.61	< 0.0020

Cattle- data combined from clothianidin and thiamethoxam use

Based on the combined data, the clothianidin data from the thiamethoxam cattle feeding studies were used to support the estimation of maximum residue levels, STMR and HR values for mammalian meat and milk.

The Meeting confirmed the previous maximum residue level recommendations for clothianidin in milk, liver, muscle, fat and edible offal (except liver). The Meeting also confirmed the previous recommended STMR and HR values for meat, muscle, fat, and edible offal (except liver).

The Meeting estimated a new STMR for clothianidin in milk of 0.006 mg/kg and recommended to withdraw the previous recommendation of 0.002 mg/kg. The Meeting estimated a new STMR and HR value for clothianidin in liver of 0.058 and 0.14 mg/kg, respectively and recommended to withdraw the previous recommendations of 0.034 and 0.10 mg/kg, respectively.

Poultry-clothianidin

The new maximum and mean dietary burdens of clothianidin through clothianidin and thiamethoxam use for poultry are marginally lower or unchanged. No new estimations are required. The Meeting agreed that the previous recommendations were confirmed.

RECOMMENDATIONS

The 2011 Meeting recommended the following residue definition for clothianidin (through thiamethoxam and clothianidin use):

Definition of the residue for compliance with the MRL and for estimation of dietary intake for plant and animal commodities: *clothianidin*.

The residue is not fat soluble.

CCN	Commodity Name	CLOTHIANIDIN Maximum residue level recommendations (mg/kg)		STMR or STMR-P (mg/kg)		HR or HR-P (mg/kg)	
		New	previous	New	previous	new	previous
FI 0326	Avocado	0.03 T		0.01 T		0.02 T	
VP 0061	Beans, except broad bean and soya bean	0.2 T		0.07 T		0.10 T	
DH 1100	Hops, dry	0.07 T		0.026 T		0.028 T	
FI 0345	Mango	0.04 T		0.02 T		0.02 T	
HH 0738 ^a	Mints	0.3 T		0.11 T		0.12 T	
-	Mango pulp			0.02 T		0.02 T	
-	Mango dried			0.13 T		0.13 T	
MM 0095	Meat (from mammals other than marine mammals)		0.02* C,t		0.02		0.02
MF 0100	Mammalian fats (except milk fats)		0.02* C,t		0.02		0.02
MO 0105	Edible offal (mammalian) (except liver)		0.02* C,t		0.02		0.02
MO 0099	Liver of cattle, goats, pigs and sheep		0.2* T,c	0.058 T,c	0.034 ^a	0.14 T,c	0.10 ^a
ML 0106	Milk		0.02*	0.006 T,c	0.002		

T = based on thiamethoxam use; T,c or C,t = combined clothianidin and thiamethoxam use

^a Not reported in Annex 1 of clothianidin report 2010, retrieved from 2010 appraisal and based on Thiamethoxam use.

CCN	Commodity Name	CLOTHIANIDIN Maximum residue level recommendations mg/kg	Median residue mg/kg	Highest residue mg/kg
AL	Bean forage	n.a.	0.075	0.11

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDI) of for clothianidin was calculated from recommendations for STMRS for raw and processed commodities in combination with consumption data for corresponding food commodities. The results are shown in Annex 3.

The IEDI of clothianidin in the 17 food cluster diets, based on the estimated STMRS was in the range 1–3% of the maximum ADI of 0.1 mg/kg bw. The Meeting concluded that the long-term intake of residues of clothianidin arising from uses of thiamethoxam and clothianidin considered by the 2010, 2011, 2012 and the present Meeting is unlikely to present a public health concern.

Short-term intake

The International Estimated Short Term Intake (IESTI) for clothianidin was calculated from recommendations for STMRS and HRs for raw and processed commodities in combination with consumption data for corresponding food commodities. The results are shown in Annex 4.

The IESTI for the general population represented 0% of the ARfD (0.6 mg/kg bw) and the IESTI for children represented 0–0% of the ARfD. The Meeting concluded that the short-term intake of residues of clothianidin, from uses of thiamethoxam considered by the present Meeting, is unlikely to present a public health concern.

CYFLUMETOFEN (273)

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Tokyo, Japan*

EXPLANATION

Cyflumetofen (consisting of isomers) is a bridged diphenyl acaricide (miticide) for control of *Tetranychus* sp. (Two spotted spider mite, Kanzawa spider mite, Desert spider mite) and influences the mitochondrial electron transport chain by inhibiting the complex II substance in the cell. Cyflumetofen does not have translaminar or systemic activity.

Cyflumetofen was first registered in Japan in 2007. Henceforth, cyflumetofen has been registered in a number of countries. It is used in a variety of crops, including citrus fruits, pome fruits, stone fruits, berry fruits, vegetables, dried herbs, and teas. It was included in the Codex Priority List in 2013 by the 45th CCPR as a new compound for evaluation by the current JMPR.

The Meeting received information on physical and chemical properties, animal and plant metabolism, environmental fate, analytical methods, storage stability, use patterns, supervised trials, and processing. Cyflumetofen is reviewed by the JMPR for the first time.

IDENTITY

ISO common name: Cyflumetofen
 Chemical name
 IUPAC: 2-methoxyethyl (RS)-2-(4-*tert*-butylphenyl)-2-cyano-3-oxo-3-(α,α,α -trifluoro-*o*-tolyl)propionate
 CAS: 2-methoxyethyl α -cyano- α -[4-(1,1-dimethylethyl)phenyl]- β -oxo-2-(trifluoromethyl)benzenepropanoate

CAS Registry No.: 400882-07-7

CIPAC No.: 821

Structural formula:



Molecular formula: C₂₄H₂₄F₃NO₄

Molecular weight: 447.45

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

Property	Results	Reference
Appearance	White solids at 21.5 °C	Iijima K, 2001a [OTSA-0029(EN)-FR]
Odour:	Odourless at 21.5 °C	Iijima K, 2001c [OTSA-0030(EN)-FR]

Hydrolysis	Half-life at 25 and 40 °C 25 °C 40 °C pH 4 222 h 70 h pH 7 5 h 3 h pH 9 12 m not calculable	Knight L, 2004a [OTSA-0076]
Photolysis	After 4 and 2 hours of irradiation with xenon arc light (38 W/m ² , 300–400 nm) respectively at 25 °C (0.01 mg/L). DT ₅₀ : pH 4 buffer Natural water Eq. summer, 40 °N lat: 5.00 h 54 h (including hydrolysis) Eq. Tokyo spring, 35 °N lat: 10.6 h 5.36 h In non-irradiated aqueous pH 4 buffer solution, 93.4% of AR recovered as the parent after 4 h, while in non-irradiated natural water, degradation was significant (38.6% of AR recovered as the parent after 2 hours). Since degradation also occurred in non-irradiated samples, a significant proportion of the degradation in natural water could be attributed to hydrolysis.	Knight L, 2004a, [OTSA-0078]

Technical material

Property	Results	Reference
Appearance:	Yellow solid at room temperature	Baltussen E, 2006a [OTSA-0327-FR]
Odour	No characteristic odour at room temperature	Baltussen E, 2006a [OTSA-0327-FR]
Relative density:	1.229 g/mL at 20 °C (pycnometer comparable method)	Iijima K, 2003a [OTSA-0031(EN)-FR]
Melting point:	779–81.7 °C	Iijima K, 2002a [OTSA-0032(EN)-FR]
Boiling point:	293–297 °C at atmospheric pressure (no decomposition prior to boiling)	Iijima K, 2002a [OTSA-0032(EN)-FR] Iijima K, 2002c [OTSA-0039(EN)-FR]
Temperature decomposition of	In the range of 320–446 °C	Iijima K, 2002a [OTSA-0032(EN)-FR]
Vapour pressure:	< 5.9 × 10 ⁻⁶ Pa at 25 °C 3.1 ± 0.23 × 10 ⁻⁵ Pa at 50 °C (Extrapolation from measurements at 150 °C to 190 °C) < 1.3 × 10 ⁻⁵ Pa (1 × 10 ⁻⁷ mmHg) (25 °C) (Triplicate test system using gas saturation method)	Saka M, 2002 [OTSA-0049(EN)-FR]
Volatility, Henry's Law Constant at 20 °C	< 9.4 × 10 ⁻² Pa m ³ /mol (Calculation by dividing vapour pressure by water solubility)	Cardinaals JM, 2006a [OTSA-0331-FR]
Octanol-water partition coefficient at (log P _{ow})	4.3 at 25 °C No significant effect of pH (4–10) on log P _{ow} .	Iijima K, 2004 [OTSA-0036(EN)-FR]
Solubility in water	In pure water	Iijima K, 2002e

Property	Results	Reference
	28 µg/L at 20 °C (pH 7) No effect of PH expected	[OTSA-0034(EN)-FR]
Solubility in solvents at 20 °C:	mg/mL solvent Acetone > 500 Dichloromethane > 500 Ethyl acetate > 500 n-Hexane 5.16 (in solution) Methanol 98.7 (in solution) Toluene > 500 Deionized water 2.15 0.215 pH 5 Buffer 36.0 3.69 pH 7 Buffer 479 68.1 pH 9 Buffer 518 76.5 ^a corrected for solution density	Iijima K, 2003c [OTSA-0035(EN)-FR]
Hydrolysis:	DT ₅₀ at 25 °C pH 4 7.7 d pH 5 6.0 d pH 7 9.8 h pH 9 10.3 m Five major metabolites (> 10% of TAR) were formed	Nakamura H, 2004a [OTSA-0156(EN)-FR]
Dissociation constant (pKa)	-4.2	Cardinaals JM, 2006b [OTSA-0330-FR]
Photolysis	The aquatic photolysis was determined after 2 days of irradiation with xenon arc light (20 W/m ² , 290–400 nm) at 25 °C. In natural water a significant proportion of the degradation may be attributed to hydrolysis since non-irradiated solution showed extensive degradation DT ₅₀ in pH 5 buffer: Irradiated: 1.28 h (including hydrolysis) Non irradiated: 134 h DT ₅₀ in natural water: Irradiated: 1.07 h (including hydrolysis) Non irradiated: 3.40 h	Ohyama K, 2004a [OTSA-0157(EN)-FR]
Formulations:	Suspension concentrate (flowable concentrate) (SC) formulations containing 200 g ai/L available	

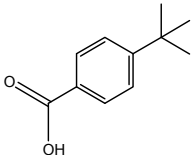
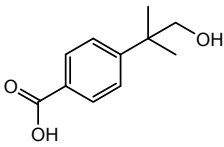
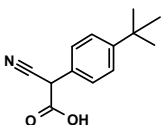
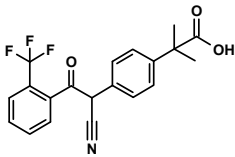
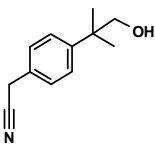
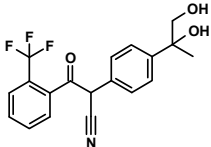
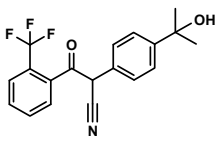
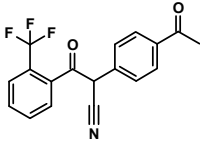
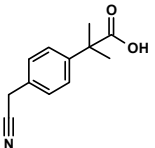
No FAO specification has been developed for cyflumetofen.

METABOLISM AND ENVIRONMENTAL FATE

The following links code numbers and structure or description of the compounds appearing in the various metabolism and environmental fate studies.

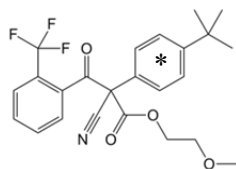
Table 1 Structure of compounds appearing in metabolism and environmental fate studies

Code (MW)	IUPAC name	Structure	Metabolite or degradate found in
Cyflumetofen (447.45)	2-methoxyethyl (RS)-2-(4- <i>tert</i> -butylphenyl)-2-cyano-3-oxo-3-(α,α,α -trifluoro- <i>o</i> -tolyl)propionate		Goat Satsuma mandarin Apple Eggplant
B-1 (190.12)	2-(trifluoromethyl)benzoic acid		Rat Goat Satsuma mandarin Apple Eggplant Soil
B-3 (189.13) Syn: M9210I005	2-(trifluoromethyl)benzamide		Soil
AB-1 (345.36) Syn: M9210I003	(RS)-2-(4- <i>tert</i> -butylphenyl)-3-oxo-3-[2-(trifluoromethyl)phenyl]propanenitrile		Rat Goat Hydrolysis in buffer Hydrolysis simulating processing Soil
AB-6 (465.45)	2-methoxyethyl-2-((R,S)-4- <i>tert</i> -butylphenyl)-3-oxo-3-({2-(trifluoromethyl)phenyl}carbonyl)amino)propanoate		Satsuma mandarin Apple Eggplant
AB-7 (447.45)	2-methoxyethyl-((R,S)-4- <i>tert</i> -butyl-2-({2-(trifluoromethyl)phenyl}carbonyl)phenyl (cyano)acetate		Satsuma mandarin Apple Eggplant
A-1 (275.35) Syn: M9210I008	4-methoxyethyl (RS)-4- <i>tert</i> -butylphenyl(cyano)acetate		Soil
A-2 (173.26) Syn: M9210I001	4- <i>tert</i> -butylphenyl-acetonitrile		Goat Soil

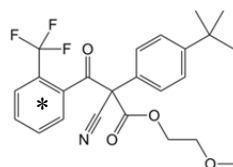
Code (MW)	IUPAC name	Structure	Metabolite or degradate found in
A-12 (178.23) Syn: M9210I002	4- <i>tert</i> -butylbenzoic acid		Goat Soil
A-20 (173.26) Syn: M9210I019	2-(4-carboxyphenyl)-2-methyl-1-propanol		Rat Goat
M9210I014 (217.67)	2-cyano-2-(4- <i>tert</i> -butylphenyl)acetic acid		Goat
M9210I021 (375.35)	(2 <i>RS</i>)-2-methyl-2-{4-[2-(2-trifluoromethylphenyl)-1-cyano-2-oxoethyl]phenyl}-propionic acid		Goat
M9210I023 (189.26)	4-(1-hydroxy-2-methyl-2-propyl) benzonitrile		Goat
M9210I029 (363.33)	(2 <i>RS</i>)-2-{4-[(2 <i>RS</i>)-1,2-dihydroxy-2-propyl]phenyl}-3-(2-trifluorophenyl)-3-oxopropionitrile		Goat
M9210I030 (347.34)	(2 <i>RS</i>)-2-[4-(2-hydroxy-2-propyl)phenyl]-3-(2-trifluorophenyl)-3-oxopropionitrile		Goat
M9210I031 (331.30)	(2 <i>RS</i>)-2-(4-acetylphenyl)-3-(2-trifluorophenyl)-3-oxopropionitrile		Goat
M9210I032 (203.24)	2-(4-cyanomethylphenyl)-2-methylpropionic acid		Goat

Code (MW)	IUPAC name	Structure	Metabolite or degradate found in
M9210I033 (235.28)	<i>N</i> -(4- <i>tert</i> -butylphenylcarbonyl)-aminoacetic acid		Goat
M9210I040 (251.28)	<i>N</i> -[4-(1-hydroxy-2-methyl-2-propyl)phenylcarbonyl]-aminoacetic acid		Goat
M9210I042/ M9210I044 (isomers) (521.49)	α -[2-(4- <i>tert</i> -butylphenyl)-2-cyano-1-(2-trifluoromethylphenyl)ethenyl]glucoside		Goat
M9210I043 (537.47)	α -{2-cyano-1-(2-trifluoromethylphenyl)-2-[4-(1-hydroxy-2-methyl-2-propyl)phenyl]ethenyl}glucoside		Goat

The metabolism and distribution of cyflumetofen in animals and plants and the fate of cyflumetofen in the environment were investigated using the following ^{14}C -radiolabelled compounds.



[*t*-butylphenyl-ring- $\text{U-}^{14}\text{C}$]-cyflumetofen
("butylphenyl-label")



[benzoyl-ring- $\text{U-}^{14}\text{C}$]-cyflumetofen
("benzoyl-label")

Animal metabolism

The Meeting received information on the results of studies on lactating goats which were fed radio-labelled cyflumetofen.

Metabolism studies on laboratory animals including rats were reviewed in the framework of toxicological evaluation by the current JMPR and the relevant information is summarized below.

Rat

Highest residues were found in the liver followed by the kidney regardless of sex, dose and label position and time point of measurement. Cyflumetofen was extensively metabolized: in total 18 metabolites were identified. B-1, 2-trifluoromethylbenzoic acid was a major metabolite in the rat metabolism (occurring up to 28% of the applied dose). The predominant metabolic pathway for cyflumetofen involves cleavage of the *tert*-butylphenyl and trifluorotolyl moieties. Major reactions on the *tert*-butylphenyl ring are cleavage of the methoxyethyl group, hydroxylation at the butyl group, decarboxylation and glucuronidation at the butyl group. Major reactions on the trifluorotolyl-ring are glutathione conjugation at the carboxyl group and further changes of the glutathione group to mercapturic acid or thiolactic acid. In addition, hydroxylation and

oxidation reactions at the butyl group and cleavage of the carboxylic ester moiety is observed on the parent structure.

Lactating goats (Sowka, et al, 2011a; and Fabian & Landsiedel, 2011a)

Two lactating goats were given orally by gavage radio-labelled cyflumetofen for 12 (benzoyl-label) or 10 (butylphenyl-label) consecutive days. The mean actual dose of benzoyl-label for each of the two goats (Goat 1 and 2) was 0.27 and 0.30 mg/kg bw corresponding to 14.9 and 12.1 ppm in feed. The mean actual dose of butylphenyl-label for each of the two goats (Goat 3 and 4) was 0.43 and 0.48 mg/kg bw corresponding to 12.8 and 11.8 ppm in feed. Milk samples were collected twice daily. The goats were sacrificed 18–24 h after the last dose and tissue/organ samples were collected.

The radioactivity in urine, faeces, blood, stomach content, gut content, gut/stomach, individual milk samples (two samples per day), cage wash, cream and skim milk was determined by liquid scintillation counting (LSC). After transport of the samples to the test facility, subsamples of urine, faeces, and milk of both animals dosed with the benzoyl-label and both animals dosed with the butylphenyl-label were combined to generate label-specific pooled samples. The tissues/organs of both animals (benzoyl- and butylphenyl-label separately) were combined and homogenized. Bile was also pooled. The pooled samples and homogenized tissues were measured with LSC (solid samples after oxidative combustion of small aliquots). All samples were stored at -18 °C or below.

No extraction was necessary for urine and bile prior to HPLC analysis. Subsamples of the pooled milk and faeces samples, and homogenized tissue samples were extracted with acetonitrile and water, or only acetonitrile. The results of the solvent extractions (LSC of the combined extracts) are referred to as extracted radioactive residues (ERR). The residual radioactive residues after solvent extraction (RRR) were determined by combustion analysis. The solvent extracts with sufficient concentrations of radioactive residues were analysed by reversed-phase HPLC with gradient elution and radio-detection, generally using two chromatographic methods.

Identification of metabolites was mainly based on fractionation of urine of both labels and bile of the butylphenyl-label, followed by HPLC-MS, GC-MS and/or NMR analysis of the isolated fractions. Thereafter, the MS samples were used as reference items for co-chromatography experiments to achieve peak assignment.

The residues after solvent extraction of liver (benzoyl- and butylphenyl-label) and kidney (benzoyl-label) were further incubated with protease, whereby significant portions of radioactivity were released. The protease solubilizates of liver (both labels) were analysed by HPLC.

During the study period, no behavioural or physical abnormalities were observed. At sacrifice, macroscopic examinations revealed no abnormalities.

Daily samples of milk, urine, and faeces obtained for twelve and ten consecutive days were measured for total radioactive residues for each animal. The results in Table 2 indicate that the radioactive residue was rapidly and extensively excreted.

Until sacrifice, the radioactive residues excreted via urine amounted to 29.9% and 33.6% (Goat 1 and 2 of the benzoyl-label) and 38.4% and 45.3% (Goat 3 and 4 of the butylphenyl-label) of the total radioactivity administered. The residues excreted via faeces accounted for 49.0% and 43.9% (Goat 1 and 2) and 50.3% and 42.6% (Goat 3 and 4) of the dose. The recovered radioactivity from urine and faeces together with from cage wash accounted for 79.4 and 78.5% of the total administered radioactivity for Goat 1 and Goat 2, respectively. Similarly, the recovered radioactivity from urine and faeces together with from cage wash accounted for 89.6, and 89.1% of the total administered radioactivity for Goat 3 and 4, respectively.

In milk, only low proportions of the administered dose were found, 0.10% and 0.14% (benzoyl-label, Goat 1 and 2) and 0.04% and 0.03% (butylphenyl-label, Goat 3 and Goat 4).

For both labels, only low portions of the administered dose were retained in edible tissues and organs. The total radioactive residues of the benzoyl- and butylphenyl-label in edible tissues/organs and milk were as follows: 0.019 mg/kg and 0.008 mg/kg in milk; 0.404 mg/kg and 0.287 mg/kg in liver, 0.191 mg/kg and 0.167 mg/kg in kidney, 0.010 mg/kg and 0.005 mg/kg in leg muscle, 0.010 mg/kg and 0.004 mg/kg in back muscle, 0.014 mg/kg and 0.018 mg/kg in abdominal fat and 0.014 mg/kg and 0.015 mg/kg in renal fat. In milk, muscle and fat tissues, only low residue levels were found. The rate of extracted radioactivity was high, above 78% TRR, except for milk from goats dosed with the benzoyl-label (51% TRR) and liver

($\geq 65\%$ TRR). In milk; however, the radioactive residues after solvent extraction were below 0.010 mg/kg, and from liver additional radioactivity was released upon protease treatment.

For further analysis, samples from Goat 1 and Goat 2 were pooled (benzoyl-label) and Goat 3 and Goat 4 (butylphenyl-label) were pooled to generate pooled samples for milk (Days 5-12 and Days 1-10), urine (Days 1-12 and Days 1-10), and homogenized faeces (Days 1-12 and Days 1-10). Similarly for each label, samples of liver, bile, kidney, muscle tissues and fat tissues were used to generate one pooled sample per matrix and label. These samples were LSC measured, resulting in the TRR. Additionally, milk and tissue samples were extracted and the TRR was calculated as the sum of the ERR and the residue after solvent extraction (TRR calculated). The TRR calculated was used for all further calculations.

The total radioactive residues in goat milk, tissue, urine and faeces are provided in Table 2.

Table 2a Material balance after repeated oral dose of radiolabelled cyflumetofen to goats, including distribution of residues, and total radioactive residues (TRRs) in milk, tissues, urine and faeces

Matrix	Benzoyl-label				
	TRR measured ^a mg eq/kg		% of Dose		TRR calculated ^b mg eq/kg
	Goat 1	Goat 2	Goat 1	Goat 2	Goats 1 & 2
Urine Subtotal Day 1–12			29.873	33.608	
Urine, pooled sample Day 1–12	4.084				^c
Faeces Subtotal Day 1–12			48.965	43.906	
Faeces, pooled sample Day 1–12 ^e	1.357				1.252
Milk; Application day 1			0.003	0.007	^c
Milk; Application day 2			0.006	0.007	^c
Milk; Application day 3			0.006	0.008	^c
Milk; Application day 4			0.008	0.012	^c
Milk; Application day 5			0.009	0.014	^c
Milk; Application day 6			0.012	0.020	^c
Milk; Application day 7			0.013	0.012	^c
Milk; Application day 8			0.011	0.013	^c
Milk; Application day 9			0.008	0.012	^c
Milk; Application day 10			0.008	0.011	^c
Milk; Application day 11			0.008	0.009	^c
Milk; Application day 12			0.008	0.015	^c
Milk Subtotal			0.101	0.141	
Milk: pooled sample Day 5–12	0.019				0.018
Liver	0.404	0.404	0.184	0.154	0.390
Blood	0.039	0.014	0.047	0.018	^c
Kidney	0.191	0.191	0.017	0.016	0.190
Kidney fat	0.014	0.014	0.001	0.002	0.015
Abdominal fat ^f	0.014	0.014	0.001	0.002	0.015
Leg muscle	0.010	0.010	0.020	0.019	0.010
Back muscle ^f	0.010	0.010	0.004	0.003	0.009
Bile	1.426	1.426	0.008	0.019	^c
Gut/Stomach	0.066	0.061	0.337	0.276	^c
Stomach contents	0.271	0.104	4.667	1.190	^c
Gut contents	1.556	1.535	4.122	5.974	^c
Urine in bladder	^d	3.219	^d	0.026	^c
Organs/Tissues Subtotal			9.408	7.699	
Cage wash	–	–	0.546	0.961	
Total Recovery			88.894	86.318	

^a For liver, bile, kidney, fat and muscle samples, the values determined from pool samples are given and were converted to % dose values for the individual animals.

^b TRR calculated from pooled samples as the sum of ERR and residue after solvent extract (used for calculation of metabolite concentrations)

^c Not calculated

^d Not sampled

^e Faeces was homogenized with water prior LSC (approximately 4 to 8 parts water per part faeces)

^f Abdominal fat referred as subcutaneous fat in the in-life report, back muscle referred as flank muscle

Table 2b Material balance after repeated oral dose of radiolabelled cyflumetofen to goats, including distribution of residues, and total radioactive residues (TRRs) in milk, tissues, urine and faeces

Matrix	Butylphenyl-label				
	TRR measured mg eq/kg ^a		% of Dose		TRR calculated ^b mg eq/kg
	Goat 3	Goat 4	Goat 3	Goat 4	Goats 3 /4
Urine Subtotal Day 1–12			38.437	45.272	
Urine, pooled sample Day 1–12	6.079				^c
Faeces Subtotal Day 1–12			50.280	42.639	
Faeces, pooled sample Day 1–12 ^e	1.304				1.290
Milk; Application day 1			0.003	0.002	^c
Milk; Application day 2			0.003	0.002	^c
Milk; Application day 3			0.003	0.003	^c
Milk; Application day 4			0.003	0.003	^c
Milk; Application day 5			0.004	0.003	^c
Milk; Application day 6			0.003	0.004	^c
Milk; Application day 7			0.004	0.003	^c
Milk; Application day 8			0.004	0.003	^c
Milk; Application day 9			0.005	0.002	^c
Milk; Application day 10			0.004	0.002	^c
Milk Subtotal			0.036	0.026	
Milk: pooled sample Day 5–10	0.008				0.007
Liver	0.287	0.287	0.118	0.121	0.282
Kidney	0.167	0.167	0.012	0.013	0.163
Kidney fat	0.015	^d	0.000	^d	^d
Abdominal fat ^f	0.018	0.018	0.002	0.001	0.020
Leg muscle	0.005	0.005	0.010	0.012	0.004
Back muscle ^f	0.004	0.004	0.010	0.002	0.003
Blood	0.015	0.024	0.010	0.023	^c
Bile	3.164	3.164	0.022	0.050	^c
Gut/Stomach	0.056	0.039	0.176	0.136	^c
Gut contents	0.762	0.654	2.080	0.772	^c
Stomach contents	0.220	0.279	1.770	3.182	^c
Urine in bladder	3.295	^d	0.042	^d	^c
Organs / Tissues Subtotal			4.252	4.312	
Cage wash	-	-	0.921	1.192	
Total Recovery			93.917	95.442	

^a For liver, bile, kidney, fat and muscle samples, the values determined from pool samples are given and were converted to % dose values for the individual animals.

^b TRR calculated from pooled samples as the sum of ERR and residue after solvent extract (used for calculation of metabolite concentrations)

^c Not calculated

^d Not sampled

^e Faeces was homogenized with water prior LSC (approximately 4 to 8 parts water per part faeces)

^f Abdominal fat referred as subcutaneous fat in the in-life report, back muscle referred as flank muscle

Recovered radioactivity from goat matrices were characterized and the results are shown in Tables 3 (benzoyl-label) and 4 (butylphenyl-label).

In urine of the goats given benzoyl-label, the main component B-1 accounted for 1.92 mg/kg or 47.0% TRR. Metabolite M9210I021 was the second most abundant component (0.61 mg/kg or 15.0% TRR). Metabolites M9210I029, M9210I030 and AB-1 were less abundant and accounted for up to 8.9% TRR.

In urine of the goats dosed with butylphenyl-label, metabolite M9210I014 was the main component and accounted for 1.26 mg/kg or 20.7% TRR. Metabolites M9210I021 and M9210I040 were the second most abundant components and accounted for 0.644 mg/kg or 11.0% TRR and 0.612 mg/kg and 10.1% TRR, respectively. Other identified metabolites were M9210I019, M9210I023, M9210I032, M9210I029, M9210I033, M9210I030, A-12, A-2 and AB-1 (each below or equal to 0.316 mg/kg or 5.2% TRR).

The extracted radioactive residues (ERR) in pooled samples of faeces of the goat administered with benzoyl-label (Days 1-12) and butylphenyl-label (Days 1-10) amounted to 78.7% and 95.5% of the TRR, respectively. In the faeces acetonitrile extract of the benzoyl-label metabolite B-1 was the main component

with 0.801 mg/kg or 63.9% TRR. The parent compound BAS 9210 I was also identified, although at significantly lower amounts, and accounted for 0.092 mg/kg or 7.4% TRR. In the faeces acetonitrile extract of the butylphenyl-label, the unchanged parent compound was the only component identified accounting for 0.588 mg/kg or 45.6% TRR.

The ERR in pooled samples of milk from the goats dosed with the benzoyl-label (Days 5-12) and butylphenyl-label (Days 5-10) amounted to 0.009 mg/kg or 51.0% TRR and 0.007 mg/kg or 93.5% TRR, respectively. In the acetonitrile extract of milk from the benzoyl-label dose, metabolite B-1 was the only component identified and accounted for 0.001 mg/kg or 4.5% TRR. In the solvent-extracted residues of milk from the butylphenyl-label dose, metabolite M9210I033 was the main component and accounted for 0.002 mg/kg or 30.0% TRR. The other identified metabolites, M9210I023, M9210I029 and M9210I030, were significantly less abundant and accounted for up to 7.8% of the TRR, less than 10% TRR.

In bile from the both experiments, the label-unspecific metabolites M9210I042, M9210I044 and M9210I043 were identified from 12.1% to 22.3% of the TRR. In bile from the benzoyl-label dose, B-1 was additionally identified accounting for 0.319 mg/kg or 22.4% TRR.

The ERR in pooled sample of liver from the benzoyl- and butylphenyl-label doses amounted to 0.276 mg/kg or 70.8% TRR and 0.182 mg/kg or 64.6% TRR. The residues after solvent extraction of liver (from benzoyl-label and butylphenyl-label) were further incubated with protease, whereby 14.0% and 17.4% of the TRR were released additionally. In the solvent extracted residues of liver from the benzoyl-label dose, metabolite B-1 was the main component accounting for 0.125 mg/kg or 32.0% of the TRR. M9210I042 accounted for 6.7% of the TRR. All components characterized in the ERR from the benzoyl-label dose by HPLC were below or equal to 3.3% TRR. Metabolite B-1 was also identified in the protease solubilize (0.010 mg/kg or 2.5% TRR). In the solvent extracted residues of liver from the butylphenyl-label dose, M9210I042 accounted for 0.023 mg/kg or 8.3% TRR and M9210I043 for 0.014 mg/kg and 5.1% TRR. All components characterized in the ERR of the butylphenyl-label dose by HPLC were below or equal to 3.1% TRR. The characterized components in the protease solubilize of the butylphenyl-label dose were each below or equal to 4.6% TRR.

The ERR in samples of kidney from the benzoyl- and butylphenyl-label doses amounted to 0.161 mg/kg or 85.1% TRR and 0.155 mg/kg or 95.5% TRR. The residue after solvent extraction of kidney (benzoyl-label) was further incubated with protease, whereby 9.4% of the TRR were released additionally. In the solvent extracted residues of kidney of the benzoyl-label dose, metabolite B-1 was the most abundant component with 0.102 mg/kg or 53.9% TRR. All components characterized in the ERR of the benzoyl-label dose by HPLC were below or equal to 5.3% TRR. In the acetonitrile extract of kidney of the butylphenyl-label dose, metabolite M9210I023 accounted for 0.017 mg/kg or 10.2% TRR. Other metabolites identified were M9210I032, M9210I033, M9210I030, M9210I014 and A-2, which were present at up to 6.7% of the TRR. All components characterized in the ERR of the butylphenyl-label by HPLC were below or equal to 8.0% TRR.

The ERR in samples of leg muscle from the benzoyl- and butylphenyl-label dose amounted to 0.008 mg/kg or 84.3% TRR and 0.004 mg/kg or 92.6% TRR. In samples of back muscle the ERR was 0.007 mg/kg or 78.4% and 0.003 mg/kg or 90.4%, respectively. In the acetonitrile extracts of leg muscle and back muscle of the benzoyl-label dose, metabolite B-1 was the main component accounting for 0.005 mg/kg or 50.5% TRR and 0.004 mg/kg or 46.5% TRR, respectively. Due to low residue levels, the muscle tissue extracts of the butylphenyl-label were not analysed by HPLC.

The ERR in samples of abdominal fat of the benzoyl- and butylphenyl-label dose amounted to 0.014 mg/kg or 93.2% TRR and 0.019 mg/kg or 96.3% TRR. In samples of kidney fat (benzoyl-label) the ERR was 0.014 mg/kg or 93.0%. In the acetonitrile extracts of abdominal and kidney fat of the benzoyl-label dose, metabolite B-1 was also the main component (0.006 mg/kg or 40.2% TRR and 0.006 mg/kg or 41.0% TRR), but additionally the parent compound was identified (0.003 mg/kg or 19.6% TRR and 0.003 mg/kg or 21.0% TRR). In the acetonitrile extract of abdominal fat of the butylphenyl-label dose, metabolite A-2 was the main component accounting for 0.008 mg/kg or 40.2% TRR.

The results of the above metabolism studies indicate that residue levels of Cyflumetofen and its metabolites were relatively low in edible tissues and milk except liver and kidney.

After repeated oral doses of benzoyl-label, metabolite B-1 was the predominant radioactive residue present in all of edible tissues tested and milk as well as excreta. It is the only metabolite present above 0.01 mg eq/kg and > 10% TRR in edible tissues or milk: in liver (0.125 mg eq/kg, 32.0% TRR) and kidney

(0.102 mg eq/kg, 53.9% TRR). M9210I042 was found at 0.026 mg eq/kg but 6.7% TRR. Cyflumetofen was found only in fat, among edible tissues, at 0.003 mg eq/kg, around 20%TRR. There were no identified metabolites in edible tissues or milk.

After repeated oral dose of butylphenyl-label, only M9210I023 was identified above 0.01 mg eq/kg and > 10% TRR in kidney (0.017 mg eq/kg, 10.2% TRR). M9210I043 and M9210I042 were found in liver at 0.014 mg eq/kg (5.1% TRR) and 0.023 mg eq/kg (8.3% TRR) respectively. M9210I033 was present in kidney at 0.011 mg eq/kg but 6.7% TRR. There were some other minor metabolites identified in edible tissues and milk but all were < 0.01 mg eq/kg. Cyflumetofen was not found in any edible tissues or milk.

From the metabolism studies, the metabolic pathway of cyflumetofen in lactating goats is speculated as:

- hydrolysis of the formic acid ester followed by hydrolytic cleavage of the trifluoromethylbenzoyl moiety resulting in metabolite B-1 (2-trifluoromethylbenzoic acid) and the labile metabolite M9210I014
- decarboxylation of metabolite M9210I014 resulting in metabolite A-2
- hydrolysis of the formic acid ester followed by decarboxylation yielding metabolite AB-1
- hydrolytic cleavage of the trifluoromethylbenzoyl moiety of metabolite AB-1 also leads to metabolite A-2
- glucuronidation of AB-1 to form M9210I042 and M9210I044
- hydroxylation yields M9210I043.

Oxidation, decarboxylation and hydroxylation of the t-butyl side chain seem to occur. Loss of nitrile from A-2 or its further metabolites also occur. Some conjugation of the carboxyl group of metabolites with the amino acid glycine, yielding conjugated metabolites also may occur.

Table 3 Extraction of radioactivity in milk, tissues, and faeces from goats given radioactive cyflumetofen

	TRR	Acetonitrile Extract ^a		Water extract ^a		ERR ^b		RRR ^c		TRR
Matrix	(measured)									(calculated)
	mg eq/kg	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Benzoyl-label										
Milk ^d	0.019	0.009	51.0	n.a.	n.a.	0.009	51.0	0.009	49.0	0.018
Liver	0.404	0.233	59.8	0.045	11.4	0.276	70.8	0.114	29.2	0.390
Kidney	0.191	0.146	77.0	0.016	8.5	0.161	85.1	0.028	14.9	0.190
Abdominal fat	0.014	0.011	73.0	0.003	19.5	0.014	93.2	0.001	6.8	0.015
Kidney fat	0.014	0.011	74.7	0.003	18.3	0.014	93.0	0.001	7.0	0.015
Leg muscle	0.010	0.007	74.1	0.001	10.3	0.008	84.3	0.001	15.7	0.010
Back muscle	0.010	0.007	72.5	0.001	7.7	0.007	78.4	0.002	21.6	0.009
Faeces ^e	1.357	0.986	78.7	n.a.	n.a.	0.986	78.7	0.266	21.3	1.252
Butylphenyl-label										
Milk ^d	0.008	0.007	92.7	n.a.	n.a.	0.007	93.5	< 0.001	6.5	0.007
Liver	0.287	0.156	55.5	0.028	10.0	0.182	64.6	0.100	35.4	0.282
Kidney	0.167	0.132	81.2	0.023	13.9	0.155	95.5	0.007	4.5	0.163
Leg muscle	0.005	0.004	85.9	< 0.001	6.5	0.004	92.6	< 0.001	7.4	0.004
Back muscle	0.004	0.003	84.0	< 0.001	11.6	0.003	90.4	< 0.001	9.6	0.003
Abdominal fat	0.018	0.017	82.9	0.002	11.3	0.019	96.3	0.001	3.7	0.020
Faeces ^e	1.304	1.205	93.4	0.033	2.6	1.232	95.5	0.058	4.5	1.290

^a TRR of pool samples^b ERR = Extractable Radioactive Residue (calculated from individual samples)^c RRR = Residual Radioactive Residue (residue after solvent extraction)^d Pooled samples of milk (Day 5-12 for the benzoyl-label or 5-10 for the butylphenyl-label), extracted with 3 × acetonitrile^e Pooled faeces sample (Day 1-12 for the benzoyl-label and 1-10 for the butylphenyl-label). Sample diluted with water.

Table 4(1) Characterization of radioactive residues in goat milk, liver, kidney, muscle, fat, urine, faeces and bile from goats dosed with benzoyl-label cyflumetofen

Metabolite/Fraction	Milk ^a		Liver		Kidney		Muscle ^b		Fat ^c		Urine ^d		Faeces ^a		Bile ^d	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Parent									0.003 0.003	19.6 21.0			0.092	7.4		
B-1	0.001	4.5	0.125	32.0	0.102	53.9	0.005 0.004	50.5 46.5	0.006 0.006	40.2 21.0	1.918	47.0	0.801	63.9	0.319	22.4
M9210I043															0.200	14.0
M9210I029											0.364	8.9				
M9210I030											0.142	3.5				
M9210I021											0.611	15.0				
M9210I042			0.026	6.7											0.194	13.6
M9210I044															0.172	12.1
AB-1											0.047	1.2				
Total Identified (ERR)	0.001	4.5	0.151	38.7	0.102	53.9	0.005 0.004	50.5 46.5	0.009 0.009	59.8 62.0	3.082	75.5	0.893	71.3	0.885	62.1
Total Characterized by HPLC (ERR)	0.004	19.6	0.070	17.9	0.024	12.6	0.002 0.002	17.2 19.8	0.001 0.002	10.0 10.8	1.002	24.5	0.093	7.4	0.541	37.9
RRR after solvent extraction	0.009	49.0	0.114	29.2	0.028	14.9	0.001 0.002	15.7 21.6	0.001 0.001	6.8 7.0	e	e	0.266	21.3	e	e
Total Characterized	0.007	41.2	0.163	41.7	0.063	33.2	0.003 0.002	28.4 27.5	0.005 0.005	31.7 31.0	1.002	24.5	0.093	7.4	0.541	37.9
Total Identified and/or Characterized	0.008	45.7	0.313	80.4	0.165	87.1	0.008 0.007	78.9 74.0	0.013 0.014	91.5 93.0	4.084	100.0	0.986	78.7	1.426	100.0
Grand total [%]	0.017	94.7	0.361	92.6	0.173	91.4	0.009 0.009	94.6 95.6	0.014 0.015	98.3 100.0	4.084	100.0	1.252	100.0	1.426	100.0

^a Analysis after extraction with acetonitrile.^b Individual values for leg (top) and back (bottom) muscle are reported.^c Individual values for abdominal (top) and kidney fat (bottom) are reported.^d Analysed after direct injection.^e Not applied.

Cyflumetofen

Table 4(2) Characterization of radioactive residues in goat milk, liver, kidney, muscle, fat, urine, faeces and bile from goats dosed with butylphenyl-label cyflumetofen

Metabolite/Fraction	Milk ^a		Liver		Kidney		Fat ^b		Urine ^c		Faeces		Bile ^c	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Parent											0.588	45.6		
M9210I040									0.612	10.1				
M9210I019									0.214	3.5				
M9210I023	0.0005	6.7			0.017	10.2			0.312	5.3				
M9210I032					0.005	3.2			0.165	2.7				
M9210I043			0.014	5.1									0.705	22.3
M9210I029	0.0005	6.8							0.316	5.2				
M9210I033	0.0021	30.0			0.011	6.7			0.306	5.0				
M9210I030	0.0006	7.8			0.007	4.1			0.137	2.3				
M9210I021									0.664	11.0				
M9210I042			0.023	8.3									0.592	18.7
A-12									0.221	3.7				
M9210I014					0.007	4.2			1.257	20.7				
M9210I044													0.462	14.6
A-2					0.009	5.3	0.008	40.2	0.082	1.3				
AB-1									0.027	0.4				
Total Identified (ERR)	0.0036	51.3	0.038	13.4	0.055	33.7	0.008	40.2	4.320	71.1	0.588	45.6	1.759	55.6
Total Characterized by HPLC (ERR)	0.0026	36.3	0.103	36.8	0.068	41.5	0.006	28.6	1.759	28.9	0.605	46.9	1.406	44.4
RRR after solvent extraction	0.0005	6.5	0.100	35.4	0.007	4.5	0.001	3.7	^d	^d	0.058	4.5	^d	^d
Total Characterized	0.0029	40.6	0.176	62.5	0.093	56.9	0.010	48.0	1.759	28.9	0.638	49.4	1.406	44.4
Total Identified and/or Characterized	0.0065	91.8	0.213	75.8	0.147	90.6	0.018	88.2	6.079	100.0	1.226	95.0	3.164	100.0
Grand total [%]	0.0070	98.4	0.254	90.1	0.155	95.1	0.018	91.9	6.079	100.0	1.283	99.5	3.164	100.0

^a Analysis after extraction with acetonitrile^b Abdominal fat sample^c HLC analysed by direct injection^d Not applied

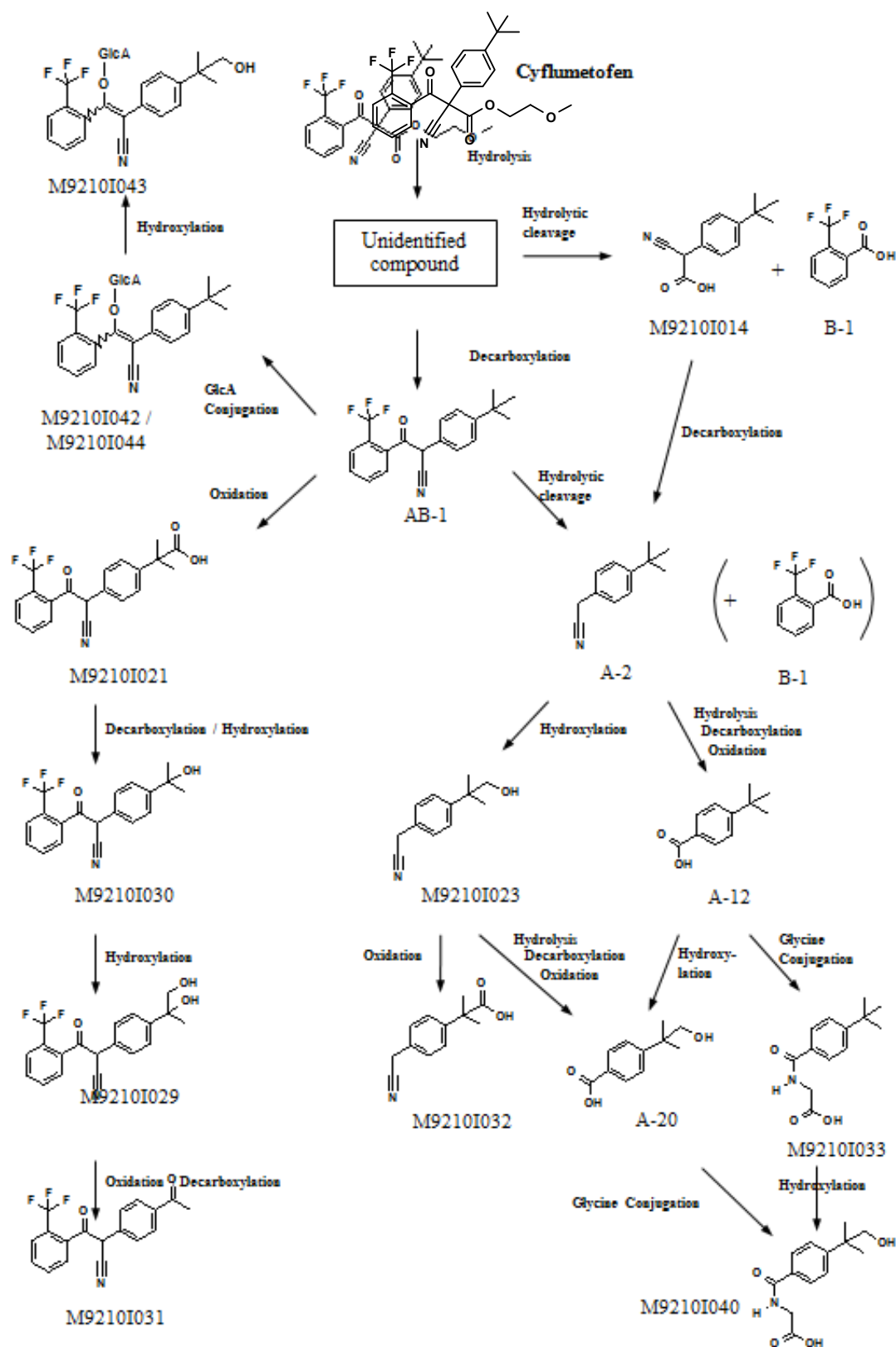


Figure 1 Proposed metabolic pathway of cyflumetofen in goats

Plant metabolism

The Meeting received information on the fate of cyflumetofen in Satsuma mandarin, apple and eggplant.

Satsuma mandarin (Hayashi, 2004a)

Two individual Satsuma mandarin trees were sprayed with formulated butylphenyl-label or benzoyl-label at an equivalent application rate of 0.600 kg ai/ha. The concentration of the application solutions was 16 mg cyflumetofen in 0.080 L blank formulation. The concentration of the blank formulation was 159 mg formulation in 0.199 L of water. The amount of spray solution per tree was 0.0369 L. The growth stage of the Satsuma mandarin fruit was just before fruit maturation. Satsuma mandarin trees, each tree separately, were kept in climate controlled greenhouses. The trees were irrigated and fertilizer (N-P-K-Mg) was added as needed. No other pesticides were used. The achieved application rate was 89.2 to 92.9% of the target.

For both treatments with the two radio-labelled cyflumetofen, fruit samples were taken at 1, 7 and 30 DAT and leaf samples at 1, 7 and 14 DAT. Four fruits were sampled at random per sampling point and pooled per two fruits. Twenty leaves were randomly sampled per sampling points and pooled per 10 leaves. Untreated fruit and leaf samples were taken at the final harvest point. Fruit and leaf samples were immediately surface rinsed with acetonitrile.

Total radioactivity in the rinse was determined by LSC. The surface rinse was concentrated by evaporation to dryness, and then redissolved prior to analysis by HPLC. The rinsed fruit was separated into peel and pulp fractions. Surface washed leaf, peel and pulp samples were homogenized with dry ice. Homogenates were extracted twice with acetonitrile:water (8:2 v/v). The extracts, after filtration, were combined and radioactivity was determined by LSC. Extracts were further concentrated (volume reduction by rotary-evaporation) prior to analysis by HPLC. Solid residues were dried and radioactivity was determined by combustion/LSC. Total radioactivity was determined as the sum of surface rinse, extracts and solid residues.

Identification was achieved by HPLC and TLC, and comparison with reference standards. Confirmation was also achieved by LC-MS/MS analysis of isolated fractions. HPLC recoveries were determined and were always > 86%. The procedural recovery of the extraction method was 96% for cyflumetofen and 100.4% for total radioactivity, demonstrating the applicability of the employed methods. Storage stability was demonstrated over the experimental period.

The results for distribution and identification of radioactivity in Satsuma mandarin fruit and leaves are presented in the following tables.

Total radioactive residues (TRR) in fruit were 0.58–0.62 mg eq/kg on 1 DAT and 0.57–0.57 mg eq/kg on 30 DAT. Distribution of radioactivity for butylphenyl- or benzoyl-label applications were similar. The majority of the radioactivity was found in the surface rinse: 95–96% of the TRR on 1 DAT and 88–89% of the TRR on 30 DAT. In peel, 4.3–4.8% of the TRR was recovered on 1 DAT, increasing to 10.8–11.5% of the TRR on 30 DAT (9.1–10.1% of the TRR extracted from peel on 30 DAT). Radioactive residues in flesh were very low ($\leq 0.6\%$ of the TRR).

TRR in leaves were 33–35 mg eq/kg on 1 DAT and 28–38 mg eq/kg on 14 DAT. Distribution of radioactivity for butylphenyl- or benzoyl-label applications was similar. The majority of the radioactivity was found in the surface rinse: 95–97% of the TRR on 1 DAT and 87–94% of the TRR on 30 DAT. Leaf residues were 3.4–4.9% of the TRR on 1 DAT, increasing to 5.6–12.9% of the TRR on 30 DAT (4.9–12.4% of the TRR extracted from leaves on 14 DAT).

Cyflumetofen was by far the predominant identified fraction in fruit (sum of rinse and peel extracts): 88–90% of the TRR on 1 DAT decreasing to 44–54% of the TRR on 30 DAT. Other identified metabolites were AB-6, AB-7, A-12 and B-1 (all $\leq 1.6\%$ TRR on 1 DAT and maximum 8.6, 8.5, 4.4 and 11.2% of the TRR, respectively on 30 DAT). It is gradually metabolized over time to produce metabolites B-1, AB-6 and AB-7. However, even 30 DAT, the parent accounted for about 50% TRR.

Cyflumetofen was also the predominant identified fraction in leaves (sum of rinse and extracts): 89–90% of the TRR on 1 DAT decreasing to 73-81% of the TRR on 30 DAT. Other identified metabolites were AB-6, AB-7, A-12 and B-1 (all $\leq 4.8\%$ of the TRR on 1 DAT and maximum 4.2, 1.5, 3.6 and 9.1% of the TRR, respectively on 30 DAT).

Table 5 Distribution of radioactivity in Satsuma mandarin fruit and leaf after treatment with radio-labelled cyflumetofen

Sample	Day-1		Day-7		Day-30 (fruit) / Day-14 (leaves)	
	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR
Butylphenyl-label						
Fruit						
Surface rinse	0.5539	95.62	0.4173	93.00	0.5067	88.77
Peel residues	0.0239	4.32	0.0307	6.85	0.0622	10.86
Extracted	0.0207	3.72	0.0256	5.70	0.0523	9.12
Unextracted	0.0032	0.60	0.0051	1.15	0.0099	1.73
Flesh residues	0.0004	0.06	0.0007	0.15	0.0020	0.37
Extracted	0.0004	0.05	0.0006	0.13	0.0018	0.33
Unextracted	0.0000	0.01	0.0001	0.02	0.002	0.04
Total radioactive residues	0.5783	100.00	0.4487	1000.00	0.5709	100.00
Leaves						
Surface rinse	34.8679	96.60	29.5027	93.54	28.3494	94.44
Leaf residues	1.2257	3.40	2.0377	6.46	1.6639	5.56
Extracted	1.1090	3.08	1.8256	5.79	1.4737	4.92
Unextracted	0.1167	0.32	0.2121	0.67	0.1901	0.64
Total radioactive residues	36.0936	100.00	31.5404	100.00	30.0132	100.00
Sample	Day-1		Day-7		Day-30 (fruit) / Day-14 (leaves)	
	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR
Benzoyl-label						
Fruit						
Surface rinse	0.5868	95.02	0.3826	91.40	0.5041	87.87
Peel residues	0.0295	4.84	0.0349	8.31	0.0662	11.53
Extracted	0.0268	4.32	0.0316	7.54	0.0581	10.11
Unextracted	0.0027	0.52	0.0032	0.77	0.0081	1.42
Flesh residues	0.0010	0.14	0.0012	0.29	0.0034	0.60
Extracted	0.0009	0.14	0.0011	0.27	0.0033	0.58
Unextracted	0.0000	0.00	0.0001	0.01	0.0001	0.02
Total radioactive residues	0.6173	100.00	0.4187	100.00	0.5737	100.00
Leaves						
Surface rinse	33.3370	95.09	30.7705	91.23	37.6579	87.14
Leaf residues	1.7188	4.91	2.9824	8.77	5.4724	12.86
Extracted	1.6259	4.65	2.8039	8.23	5.2696	12.37
Unextracted	0.0929	0.27	0.1785	0.53	0.2028	0.49
Total radioactive residues	35.0558	100.00	33.7529	100.00	43.1303	100.00

Table 6 Identification of radioactive residues in Satsuma mandarin surface rinse and peel extracts

Identified compound	Surface rinse		Peel extract		Total	
	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR
Butylphenyl-label						
Day 1						
A-12	0.0040	0.71	0.0062	1.17	0.0102	1.88
AB-6	0.0053	0.92	0.0008	0.16	0.0061	1.07
AB-7	0.0034	0.59	0.0005	0.09	0.0039	0.69
Parent	0.5174	89.25	0.0037	0.58	0.5211	89.83
Unidentified peaks total	0.0238	4.15	0.0095	1.72	0.0333	5.88
Total	0.5539	95.62	0.0207	3.72	0.5746	99.35
Day 7						
A-12	0.0047	1.04	0.0076	1.69	0.0122	2.73
AB-6	0.0084	1.88	0.0010	0.23	0.0095	2.11
AB-7	0.0037	1.50	0.0006	0.13	0.0073	1.63
Parent	0.3674	81.88	0.0033	0.74	0.3708	82.62
Unidentified peaks total	0.0331	6.70	0.0131	2.91	0.0431	9.61
Total	0.4173	93.00	0.0256	5.70	0.4429	98.70
Day 30						
A-12	0.0132	2.32	0.0119	2.04	0.0250	4.36
AB-6	0.0392	6.95	0.0032	0.57	0.0424	7.51
AB-7	0.0389	6.93	0.0015	0.26	0.0404	7.19
Parent	0.3064	53.26	0.0043	0.77	0.3107	54.03
Unidentified peaks total	0.109	19.31	0.0314	5.48	0.1405	24.81
Total	0.5067	88.77	0.0523	9.12	0.5590	97.90
Benzoyl-label						
Day 1						
B-1	0.0097	1.55	0.0185	3.07	0.0282	4.66
AB-6	0.0055	0.89	0.0006	0.10	0.0061	0.99
AB-7	0.0026	0.45	0.0002	0.03	0.0028	0.48
Parent	0.5434	87.94	0.0032	0.46	0.5466	88.40
Unidentified peaks total	0.0256	4.19	0.0043	0.66	0.0299	4.81
Total	0.5868	95.02	0.0268	4.32	0.6136	99.34
Day 7						
B-1	0.0070	1.67	0.0206	4.91	0.0276	6.58
AB-6	0.0093	2.22	0.0010	0.23	0.0103	2.45
AB-7	0.0077	1.84	0.0005	0.12	0.0082	1.97
Parent	0.3282	78.40	0.0020	0.49	0.3303	78.89
Unidentified peaks total	0.0304	7.27	0.0075	1.79	0.0378	9.05
Total	0.3826	91.40	0.0316	7.54	0.4142	98.94
Day 30						
B-1	0.0248	4.32	0.0394	6.86	0.0642	11.17
AB-6	0.0474	8.26	0.0020	0.35	0.0494	8.61
AB-7	0.0481	8.37	0.0007	0.12	0.0488	8.49
Parent	0.2491	43.48	0.0021	0.37	0.2512	43.85
Unidentified peaks total	0.1347	23.44	0.0139	2.41	0.1486	25.87
Total	0.5041	87.87	0.0581	10.11	0.5622	97.99

Data are the mean of duplicate experiments.

Apple (Dohn, 2004a)

One apple tree grown within a commercial apple orchard in California, USA, was sprayed with formulated butylphenyl- or benzoyl-label at an equivalent application rate of 0.600 kg ai/ha.

Application took place in October 2002. The concentration of the application solutions was approximately 225 (butylphenyl-label) and 211 mg/L (benzoyl-label) cyflumetofen in blank formulation. The achieved application rate was 115 to 120% of the target. Storage stability was demonstrated over the experimental period. At the time of application, fruits were near maturity. The plots were (drip) irrigated and fertilizer (N-P-K and Ca) was added as needed. Other pesticides were used prior to application of cyflumetofen, which does not interfere with cyflumetofen. Fruit samples were taken at 1, 7 and 30 DAT (for both labels and untreated plot). Leaf samples were taken at 7 and 30 DAT (also for both labels and untreated plot).

Fruit and leaf samples were immediately surface rinsed two times with acetonitrile. Total radioactivity in the rinse was determined by LSC. The surface rinse was concentrated prior to analysis by HPLC/TLC. The rinsed fruit and leaves were homogenized with dry ice and liquid nitrogen. Homogenates were extracted twice with acetonitrile and twice with acetonitrile:water (1:1 v/v). The extracts after centrifugation were combined and radioactivity was determined by LSC. Extracts were further concentrated prior to analysis by HPLC/TLC. Solid residues were dried and radioactivity was determined by combustion/LSC. Total radioactivity was determined as the sum of surface rinse, extracts and solid residues.

Identification was achieved by HPLC and TLC, and comparison with reference standards. Some extracts were further characterized by reversed phase solid phase extraction and LSC/HPLC analysis of eluates.

Total radioactive residues (TRR) in apple fruit were 0.100–0.113 mg eq/kg on 1 DAT and 0.079–0.057 mg eq/kg on 30 DAT. Results for butylphenyl- and benzoyl-label were similar. For the butylphenyl- and benzoyl-label, the majority of the radioactivity was found in the surface rinse: 95 and 96% of the TRR, respectively on 1 DAT and 71 and 67% of the TRR, respectively on 30 DAT. Extracted radioactivity was not determined on 1 DAT due to low levels of radioactivity, and was 15 and 9.6% of the TRR on 7 DAT for butylphenyl- and benzoyl-label respectively and 22 and 28% of the TRR on 30 DAT, respectively. Unextracted radioactivity was low: 5.3–7.6% of the TRR on 30 DAT.

TRR in apple leaves were 6.1–7.3 mg eq/kg on 7 DAT and 4.9–9.5 mg eq/kg on 30 DAT. Surface rinse removed less radioactivity from leaves for the benzoyl-label compared to the butylphenyl-label. For the butylphenyl- and benzoyl-label, the majority of the radioactivity was found in the surface rinse: 91 and 87% of the TRR, respectively on 7 DAT and 82 and 72% of the TRR, respectively on 30 DAT. Extracted radioactivity was 13 and 21% of the TRR on 30 DAT for butylphenyl- and benzoyl-label respectively. Unextracted radioactivity was low: 4.9–6.7% of the TRR on 30 DAT.

Cyflumetofen was the predominant identified compound in apple fruit (sum of rinse and extracts): 89–95% of the TRR on 1 DAT decreasing to 53–65% of the TRR on 30 DAT. Other identified metabolites were AB-6, AB-7 and B-1 (all $\leq 5\%$ of the TRR on 1 DAT and maximum 5.3, 6.3 and 1.8% of the TRR, respectively on 30 DAT).

Cyflumetofen was also the predominant identified compound in apple leaves (sum of rinse and extracts): 77–85% of the TRR on 7 DAT and 44–60% of the TRR on 30 DAT. Other identified metabolites were AB-6, AB-7 and B-1 at maximum 8.6, 5.6 and 4.8% of the TRR, respectively on 30 DAT.

The results for apple fruit and leaves (distribution and identification of radioactivity for both labels) are presented in the following tables.

Table 7 Distribution of radioactivity in apple fruit and leaf after treatment with radio-labelled cyflumetofen

DAT	Surface rinse		Extracted		Unextracted			Total
	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg
Butylphenyl-label								
Apple fruit								
Day 1	95.0	0.095	Np	np	5.0	0.005	100	0.10
Day 7	82.1	0.064	15.4	0.012	2.6	0.002	100	0.078
Day 30	70.9	0.056	21.5	0.017	7.6	0.006	100	0.079
Leaves								
Day 7	90.8	5.535	7.9	0.479	1.4	0.085	100	6.099
Day 30	82.0	4.046	13.1	0.644	4.9	0.242	100	4.932
Benzoyl-label								
Apple fruit								
Day 1	95.6	0.108	Np	np	4.4	0.005	100	0.113
Day 7	89.2	0.149	9.6	0.016	1.2	0.002	100	0.167
Day 30	66.7	0.038	28.1	0.016	5.3	0.003	100	0.057
Leaves								
Day 7	86.8	6.306	11.6	0.843	1.6	0.117	100	7.266
Day 30	72.0	6.884	21.3	2.039	6.7	0.641	100	9.564

Table 8 Identification of radioactive residues in apple fruit surface rinse and extracts

Identified compound	Surface rinse		Extract		Total	
	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR
Butylphenyl-label						
Day 1						
AB-6	0.005	5.0	Not extracted due to low radioactive residues: < 0.01 mg eq/kg		0.005	5.0
AB-7						
Parent	0.061	61.0			0.061	61.0
Breakdown of parent	0.028	28.0			0.028	28.0
¹⁴ C in Extract (mg eq/kg)	0.095				0.095	
Day 7						
AB-6	0.001	1.3			0.001	1.3
AB-7						
Parent	0.059	75.6	0.002	2.6	0.061	78.2
Minor	0.004	5.1	0.003	3.8	0.007	8.9
Unknown			0.006	7.7	0.006	7.7
¹⁴ C in Extract (mg eq/kg)	0.064		0.012		0.076	
Day 30						
AB-6	0.003	3.8	0.001	1.3	0.004	5.1
AB-7	0.002	2.5	0.003	3.8	0.005	6.3
Parent	0.038	48.1	0.004	5.1	0.042	53.2
Minor	0.013	16.5	0.009	11.4	0.022	27.9
¹⁴ C in Extract (mg eq/kg)	0.056		0.016		0.072	
Benzoyl-label						
Day 1						
AB-6			Not extracted due to low radioactive residues: < 0.01 mg eq/kg			
AB-7						
Parent	0.066	58.4			0.066	58.4
Breakdown of parent	0.041	36.3			0.041	36.3
¹⁴ C in Extract (mg eq/kg)	0.108				0.108	

Identified compound	Surface rinse		Extract		Total	
	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR
Day 7						
B-1	0.002	1.2			0.002	1.2
AB-6						
AB-7	0.003	1.8	0.006	3.6	0.009	5.4
Parent	0.137	82.0	0.003	1.8	0.14	83.8
Minor	0.006	3.6	0.006	3.6	0.012	7.2
¹⁴ C in Extract (mg eq/kg)	0.149		0.016		0.165	
Day 30						
B-1			0.001	1.8	0.001	1.8
AB-6	0.001	1.8	0.002	3.5	0.003	5.3
AB-7	0.001	1.8	0.002	3.5	0.003	5.3
Parent	0.029	50.9	0.008	14.0	0.037	64.9
Minor	0.001	1.8	0.003	5.3	0.004	7.1
Unknown	0.005	8.8			0.005	8.8
¹⁴ C in Extract (mg eq/kg)	0.038		0.016		0.054	

“Minor” refers to ¹⁴C peaks in the chromatogram with a concentration < 0.005 mg eq/kg in the extract. This refers to a total of all minor components.

Table 9 Identification of radioactive residues in apple leaf surface rinse and extracts

Identified compound	Surface rinse		Extract		Total	
	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR
Butylphenyl-label						
Day 7						
AB-6	0.133	2.2	0.031	0.5	0.164	2.7
AB-7	0.188	3.1	0.032	0.5	0.22	3.6
Parent	4.904	80.4	0.272	4.5	5.176	84.9
Minor	0.310	5.1	0.144	2.4	0.454	7.5
Unknown						
¹⁴ C in Extract (mg eq/kg)	5.535		0.479		6.014	
Day 30						
AB-6	0.267	5.4	0.067	1.4	0.334	6.8
AB-7	0.210	4.3	0.028	0.6	0.238	4.9
Parent	2.881	58.4	0.086	1.7	2.967	60.1
Minor	0.688	13.9			0.688	13.9
¹⁴ C in Extract (mg eq/kg)	4.046		0.644		4.69	
Benzoyl-label						
Day 7						
B-1	0.120	1.7	0.140	1.9	0.26	3.6
AB-6	0.290	4.0	0.050	0.7	0.34	4.7
AB-7	0.265	3.6	0.044	0.6	0.309	4.2
Parent	5.278	72.6	0.333	4.6	5.611	77.2
Minor	0.353	4.9			0.353	4.9
¹⁴ C in Extract (mg eq/kg)	6.306		0.843		7.149	
Day 30						
B-1	0.248	2.6	0.208	2.2	0.456	4.8
AB-6	0.688	7.2	0.139	1.5	0.827	8.7
AB-7	0.454	4.7	0.077	0.8	0.531	5.5
Parent	3.979	41.6	0.208	2.2	4.187	43.8
Minor	0.317	3.3	0.812	8.5	1.129	11.8
Unknown	1.198 (4 peaks)	12.5	0.595 (2 peaks)	6.2	1.793	18.7
¹⁴ C in Extract (mg eq/kg)	6.884		2.039		8.923	

“Minor” refers to ¹⁴C peaks in the chromatogram with a concentration < 0.200 mg eq/kg in the extract. This refers to a

total of all minor components.

Eggplant (Dohn, 2004b)

Eggplants grown on two plots (1.1–1.5 m²) located in California and containing six to eight eggplants were sprayed with formulated butylphenyl- or benzoyl-label cyflumetofen at an equivalent application rate of 0.600 kg ai/ha. Application took place on August 2002. The concentration of the application solutions was approximately 140 (butylphenyl-label) and 220 mg/L (benzoyl-label) cyflumetofen in blank formulation. The achieved application rate was 98 to 114% of the target. Storage stability was demonstrated over the experimental period. The growth stage of the eggplant fruit was from just pollinated to mature. The eggplants were grown outdoors. The plots were (drip) irrigated and fertilizer (N-P-K-Ca-S) was added as needed. Other pesticides were used which do not interfere with cyflumetofen. Fruit samples were taken at 1, 7 and 14 DAT (for both labels and untreated plot). Leaf samples were taken at 14 DAT (also for both labels and untreated plot).

Fruit and leaf samples were immediately surface rinsed two times with acetonitrile. Total radioactivity in the rinse was determined by LSC. The surface rinse was concentrated, if necessary, prior to analysis by HPLC. The rinsed fruit and leaves were homogenized with dry ice. Homogenates were extracted twice with acetonitrile and twice with acetonitrile:water (1:1 v/v). The extracts, after centrifugation, were combined and radioactivity was determined by LSC. Extracts were further concentrated, if necessary by volume reduction by rotary-evaporation, prior to analysis by HPLC. Solid residues were dried and radioactivity was determined by combustion/LSC. Total radioactivity was determined as the sum of surface rinse, extracts and solid residues.

Identification was achieved by HPLC and 2D-TLC, and comparison with reference standards. Some degradates (U4) were identified by LC-MS/MS analysis of isolated fractions or were subjected to acid hydrolysis (U1 and U2) with 1 N HCl at 80 °C to release acid labile conjugates.

Total radioactive residues (TRR) in eggplant fruit were 0.323–0.488 mg eq/kg on 1 DAT and 0.315–0.413 mg eq/kg on 14 DAT. Surface rinse removed less radioactivity from fruit for the benzoyl-label compared to the butylphenyl-label. For the butylphenyl- and benzoyl-label, the majority of the radioactivity was found in the surface rinse: 92 and 86% of the RR, respectively on 1 DAT and 81 and 56% of the TRR, respectively on 14 DAT. Extracted radioactivity was 7.1 and 13% TRR on 1 DAT for butylphenyl- and benzoyl-label respectively and 15 and 41% TRR on 14 DAT, respectively. Unextracted radioactivity was low: 2.7–4.1% of the TRR on 14 DAT.

TRR in eggplant leaves were 17–23 mg eq/kg on 14 DAT. Surface rinse removed less radioactivity from leaves for the benzoyl-label compared to the butylphenyl-label. For the butylphenyl- and benzoyl-label, the majority of the radioactivity was found in the surface rinse: 83 and 69% of the TRR, respectively on 14 DAT. Extracted radioactivity was 14 and 27% of the TRR on 14 DAT for butylphenyl- and benzoyl-label respectively. Unextracted radioactivity was low: 2.6–4.7% of the TRR on 14 DAT.

Cyflumetofen was the predominant identified compound in eggplant fruit (sum of rinse and extracts): 91–95% of the TRR on 1 DAT decreasing to 42–62% of the TRR on 14 DAT. Other identified metabolites were AB-6, AB-7, B-1, U4 and U1 and U2 (acid labile conjugates of B-1) (all ≤ 2.5% TRR on 1 DAT and maximum 3.4, 3.6, 14.8, 1.2, 16.2 and 6.3% of the TRR, respectively on 14 DAT).

Cyflumetofen was also the predominant identified compound in eggplant leaves (sum of rinse and extracts): 47–58% of the TRR on 14 DAT. Other identified metabolites were AB-6, AB-7, B-1, U4 (structure proposed) and U1 (conjugate of B-1) and U2 (conjugate of B-1) at 8.1, 5.7, 4.6, 4.3, 4.1 and 1.4% of the TRR, respectively on 14 DAT).

The results for eggplant fruit and leaves (distribution and identification of radioactivity for both labels) are presented in the following tables.

Table 10 Distribution of radioactivity in eggplant fruit and leaf after treatment with radio-labelled cyflumetofen

DAT	Surface Rinse		Extracted		Unextracted		Total	
	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg
Butylphenyl-label								
Eggplant fruit								
Day 1	92.0	0.297	7.1	0.023	0.9	0.003	100	0.323
Day 7	86.1	0.323	11.5	0.043	2.4	0.009	100	0.375
Day 14	81.3	0.256	14.6	0.046	4.1	0.013	100	0.315
Leaves								
Day 14	83.4	19.144	14.1	3.232	2.6	0.592	100	22.968
Benzoyl-label								
Eggplant fruit								
Day 1	86.5	0.422	12.7	0.062	0.8	0.004	100	0.488
Day 7	79.2	0.442	19.4	0.108	1.4	0.008	100	0.558
Day 14	56.4	0.233	40.9	0.169	2.7	0.011	100	0.413
Leaves								
Day 14	68.7	12.001	26.6	4.644	4.7	0.818	100	17.463

Table 11 Identification of radioactive residues in eggplant fruit surface rinse and extracts

Identified compound	Surface rinse		Extract		Total	
	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR
Butylphenyl-label						
Day 1						
Parent	0.294	91.0	0.013	4.0	0.307	95
Minor	0.003	0.9	0.010	3.1	0.013	4
¹⁴ C in Extract (mg eq/kg)	0.297		0.023			
Day 7						
AB-6	0.033	8.8			0.033	8.8
AB-7	0.018	4.8			0.018	4.8
Parent	0.240	64.0	0.011	2.9	0.251	66.9
U4(tentatively identified)	0.022	5.9			0.022	5.9
Minor	0.010	2.7	0.013	3.5	0.023	6.2
Unknown			0.019 (3 peaks)	5.1	0.019	5.1
¹⁴ C in Extract (mg eq/kg)	0.323		0.043			
Day 14						
AB-6	0.016	5.1			0.016	5.1
AB-7	0.016	5.1			0.016	5.1
Parent	0.188	59.7	0.008	2.5	0.196	62.2
U4(tentatively identified)	0.011	3.5			0.011	3.5
Minor	0.025	7.9			0.025	7.9
Unknown			0.019 (2 peaks)	6.0	0.016	6.0
¹⁴ C in Extract (mg eq/kg)	0.256		0.046			
Benzoyl-label						
Day 1						
B-1			0.012	2.5	0.012	2.5
Parent	0.408	83.6	0.037	7.6	0.445	91.2
Minor	0.014	2.9	0.008	1.6	0.022	4.5
Unknown			0.005	1.0	0.005	1.0

Identified compound	Surface rinse		Extract		Total	
	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR
¹⁴ C in Extract (mg eq/kg)	0.422		0.062			
Day 7						
B-1	0.011	2.0	0.048	8.6	0.059	10.6
AB-6	0.017	3.0			0.017	3
AB-7	0.016	2.9			0.016	2.9
Parent	0.387	69.4	0.007	1.3	0.394	70.7
U1 (conjugate of B-1)			0.034	6.1	0.034	6.1
U2 (conjugate of B-1)			0.011	2.0	0.011	2
Minor	0.007	1.3			0.007	1.3
Unknown	0.005	0.9			0.005	0.9
¹⁴ C in Extract (mg eq/kg)	0.442		0.108			
Day 14						
B-1			0.061	14.8	0.061	14.8
AB-6	0.014	3.4			0.014	3.4
AB-7	0.015	3.6			0.015	3.6
Parent	0.173	41.9	0.002	0.5	0.175	42.4
U4(tentatively identified)	0.005	1.2			0.005	1.2
U2 (conjugate of B-1)			0.026	6.3	0.026	6.3
U1 (conjugate of B-1)			0.067	16.2	0.067	16.2
Minor	0.020	4.8	0.014	3.4	0.034	8.2
Unknown	0.005	1.2			0.061	14.8
¹⁴ C in Extract (mg eq/kg)	0.233		0.169			

“Minor” refers to ¹⁴C peaks in the chromatogram with a concentration < 0.005 mg eq/kg in the extract. This refers to a total of all minor components.

Table 12 Identification of radioactive residues in eggplant leaf surface rinse and extracts

Identified compound	Surface rinse		Extract		Total	
	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR
Butylphenyl-label						
Day 14						
AB-6	2.068	9.0	0.278	1.2	2.346	10.2
AB-7	1.455	6.3	0.110	0.5	1.565	6.8
Parent	12.061	52.5	1.173	5.1	13.234	57.6
U4(tentatively identified)	0.861	3.7			0.861	3.7
U3	0.823	3.6			0.823	3.6
Minor	0.249	1.1	0.931	4.1	1.18	5.2
Unknown	1.436 (3 peaks)	6.2	0.740	3.2	2.176	9.4
¹⁴ C in Extract (mg eq/kg)	19.144		3.232			
Benzoyl-label						
Day 14						
B-1	0.240	1.4	0.571	3.3	0.811	4.7
AB-6	0.996	5.7	0.418	2.4	1.414	8.1
AB-7	0.876	5.0	0.121	0.7	0.997	5.7
Parent	6.937	39.7	1.333	7.6	8.27	47.3
U4(tentatively identified)	0.480	2.7	0.279	1.6	0.759	4.3
U3	0.504	2.9			0.504	2.9
U2 (conjugate of B-1)			0.251	1.4	0.251	1.4
U1 (conjugate of B-1)			0.715	4.1	0.715	4.1
Minor	0.516	3.0			0.516	3

Identified compound	Surface rinse		Extract		Total	
	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR
Unknown	1.452 (4 peaks)	8.3			1.452	8.3
¹⁴ C in Extract (mg eq/kg)	12.001		4.644			

“Minor” refers to ¹⁴C peaks in the chromatogram with a concentration < 0.200 mg eq/kg in the extract. This refers to a total of all minor components.

Proposed metabolic pathway of cyflumetofen in plants

In the metabolism studies on Satsuma mandarin, apple and eggplant, radioactive residues after treatment were at significantly higher concentrations in surface rinse of the respective fruits than their extracts. In all plants, cyflumetofen was the predominant residue. Metabolite B-1 was found in the fruit extract of apple (30 DAT) and eggplant (14 DAT) at 11.2% and 14.8% of TRR corresponding to 0.064 and 0.061 mg eq/kg respectively. No other individual components accounted for > 10% TRR. At smaller amounts, metabolites AB-6 and AB-7 were often identified in fruits.

Considering the results of the plant metabolism studies, the metabolic pathway of cyflumetofen in plants was speculated as follows:

- Cyflumetofen is photodegraded to AB-7, hydrolysed to B-1, or its nitrile is hydrolysed to amide followed by acyl migration to produce AB-6.
- The side chain of AB-7 is hydrolysed and oxidized to produce U4 (identified only in eggplant).
- B-1 conjugates to produce U1 and U2 (found only in eggplant).
- B-1 may also be produced from AB-6 and AB-7. A-12 (found only in Satsuma mandarin) may be derived directly from cyflumetofen or through AB-6

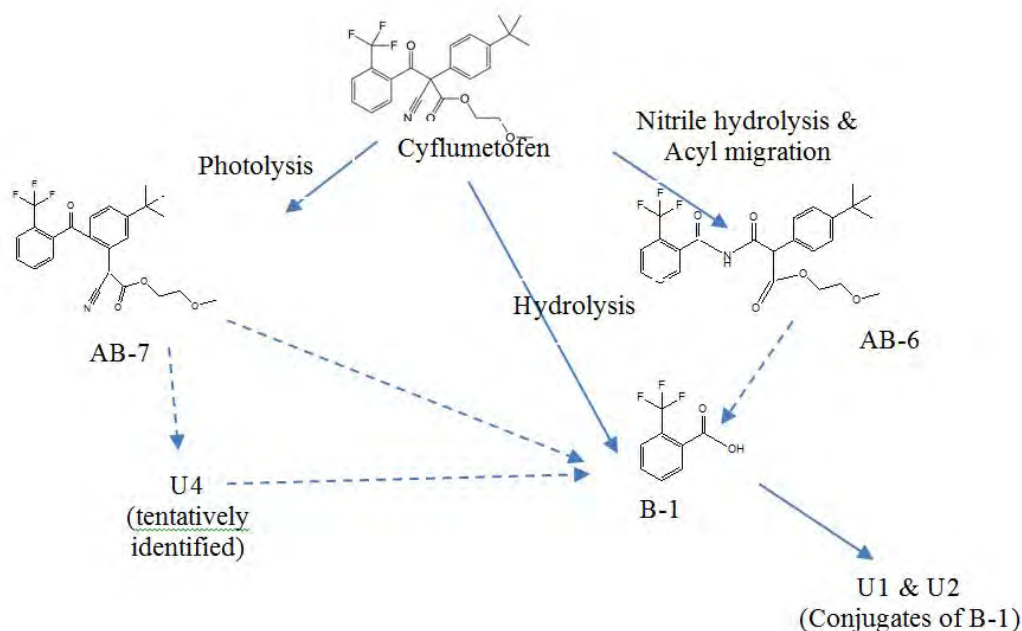


Figure 2 Proposed metabolic pathway of cyflumetofen in plants

Environmental fate

The Meeting received extensive information on fate and behaviour in soil, hydrolysis in aquatic systems, and residues in succeeding crops. Since cyflumetofen is applied as foliar spray on trees (citrus fruits, pome fruits, grapes and tree nuts), strawberry, tomato and eggplant, studies on hydrolysis rate and products (some included in the Physical and Chemical Properties Section), photolysis on plant surface (conditional: No information was available) and rotational crops were relevant to the current review ("Submission and evaluation of pesticide residues data for the estimation of maximum residue levels in food and feed", FAO, 2009).

Aerobic soil metabolism

Studies on aerobic soil metabolism studies on cyflumetofen and its degradates were provided.

Study 1_(Knight, 2004a)

Butylphenyl- or benzoyl-label cyflumetofen was incubated for 181 days in a sandy loam soil in the dark at 25 °C under aerobic conditions at a concentration of 0.93 mg/kg dw. Moisture was maintained throughout the incubation period and volatile radioactivity was trapped (PUF, ethyl digol, KOH). Soil biomass was 3.08% at the start of the experiment and 0.6% after 181 days (study end), indicating sufficiently viable conditions (at least during the first 100 days, as is also indicated by the continuous increase of [¹⁴C]CO₂).

Single soil portions of each label treatment were sampled at 0, 1, 3, 6, 10, 14, 30, 59, 90, 121 and 181 days. Samples were extracted with 100 mL acetonitrile (2×), followed by extraction with 100 mL acetonitrile:water (3:1 v/v) (2×) and finally reflux for 6 hours in acetonitrile:water (3:1 v/v). Polyurethane foam plugs were extracted with acetonitrile. Post-extraction solids were fractionated into fulvic and humic acids and humin. Extracts were subjected to chromatographic analysis by HPLC-RAM and TLC. Metabolite identification was obtained by comparison with reference substances of metabolites.

Mass balances were between 89.6 and 102.6% of applied radioactivity (AR) for both labels. Unextracted radioactivity was at maximum 43.4% AR (14 d) and 37.8% AR on day 90. Volatile radioactivity consisted only of CO₂ and was 25.7 and 34.0% AR on day 90 for butylphenyl- and benzoyl-label, respectively (27.9 and 39.3% AR at study end).

Cyflumetofen degraded with a DT₅₀ of 2.76 days and a DT₉₀ of 9.16 days by linear regression analysis. Degradation could be described by first-order kinetics (r² 0.952). Significant metabolites (exceeding 10% AR or 2× 5% at two consecutive time points) were AB-1 (max 8.3% AR, day 59), B-1 (max 22.9% AR, day 6) and SHL9 (max 7.9%, day 10).

Table 13 Soil properties of Wolston sandy loam

Property	Value
Origin	Wolston, UK
Texture (USDA)	Sandy loam
Sand (% , USDA)	70.8
Silt (% , USDA)	16.8
Clay (% , USDA)	12.4
pH-water	6.5
pH-KCl	6.5
pH-CaCl ₂	5.8
% OM	3.3
% OC	1.9
CEC (meq/100g)	15.3
water content at pF2 (% , w/w)	22.3
MWHC (% , w/w)	52.4

Table 14 Distribution of radioactivity after aerobic incubation at 25 °C of sandy loam soil treated with butylphenyl- or benzoyl-label cyflumetofen at 0.93 mg/kg

Time d	% AR						
	Extracted radioactivity	Unextracted radioactivity				Volatile radioactivity	Mass balance
		fulvic acids	humic acids	humins	total		
Butylphenyl-label							
0	98.2	nd	nd	nd	2.8	na	101.0
1	87.5	2.1	3.7	4.2	10.0	< 0.1	97.5
3	58.5	5.7	11.7	10.4	27.8	6.6	92.9
6	46.3	6.7	13.9	12.0	32.5	14.2	93.0
10	44.4	7.3	13.3	12.8	33.4	11.8	89.6
14	34.9	8.4	16.0	19.1	43.4	16.4	94.7
30	32.9	7.0	14.2	19.6	40.8	19.3	93.0
59	34.5	7.1	19.4	15.8	42.3	24.6	101.4
90	28.3	6.9	17.2	13.7	37.8	25.7	91.8
121	29.0	7.2	15.4	14.4	36.9	31.2	97.1
181	29.9	7.9	15.9	14.1	37.9	27.9	95.4
Benzoyl-label							
0	98.3	nd	nd	nd	4.3	na	102.6
1	94.2	4.6	0.7	1.4	6.7	0.7	101.6
3	79.1	12.2	2.1	3.8	18.0	3.6	100.7
6	67.2	15.0	3.0	6.2	24.2	7.7	99.1
10	55.7	16.4	4.5	7.8	28.7	12.6	97.0
14	44.4	18.5	5.6	10.3	34.3	20.8	99.5
30	36.8	15.0	4.9	11.2	31.2	27.1	95.1
59	34.5	16.3	5.7	12.9	34.9	28.6	98.0
90	29.4	14.6	5.7	10.3	30.6	34.0	94.0
121	30.0	13.9	5.4	9.6	29.0	36.7	95.7
181	27.1	14.8	4.9	11.0	30.7	39.3	97.1

Nd = Not determined

na = Not applicable

Table 15 Identification of radioactivity after aerobic incubation at 25 °C of sandy loam soil treated with butylphenyl- or benzoyl-label at 0.93 mg/kg

Component	% AR										
	0 d	1 d	3 d	6 d	10 d	14 d	30 d	59 d	90 d	121 d	181 d
Butylphenyl-label											
Polar comp.	nd	nd	0.8	1.0	1.2	1.1	0.5	1.1	1.5	1.2	1.4
SHL1	nd	nd	1.2	1.1	0.8	0.8	1.3	0.9	1.0	1.1	1.5
SHL3	nd	0.9	6.2	2.5	2.0	2.1	1.6	0.9	1.4	1.6	1.7
SHL4	nd	1.4	2.6	2.7	2.8	2.5	1.6	1.8	1.9	1.8	2.0
AB-1	nd	2.4	3.3	4.4	6.9	6.4	5.5	8.3	6.5	6.0	3.8
SHL6	0.9	nd	0.9	2.1	1.2	0.7	0.9	0.6	0.5	1.4	1.9
Parent	96.8	76.6	27.1	15.1	9.8	6.2	4.6	3.6	2.2	1.9	1.3
SHL7	nd	nd	1.7	1.1	0.2	0.6	0.7	1.4	1.2	1.7	1.3
SHL8	nd	nd	1.0	2.4	3.7	1.4	1.4	2.9	1.6	1.2	1.7
AB-1 dimer	nd	2.0	4.6	4.4	5.8	4.6	4.8	5.0	3.4	2.8	3.6
AB-1 (cis)	nd	1.8	6.4	5.7	1.2	5.1	2.0	3.2	5.8	2.7	3.2
AB-1 (trans)	nd				5.1		3.2	3.4		4.1	4.7
Others	0.5	2.5	2.7	3.7	3.6	3.4	4.7	1.2	1.2	1.5	1.8
Benzoyl-label											
Polar comp.	nd	0.5	1.7	1.9	2.3	3.2	1.8	2.7	1.5	2.2	1.6
B-1	nd	7.6	18.7	22.9	18.3	8.4	3.6	4.3	2.3	3.2	2.7
SHL3	nd	nd	0.3	0.4	0.3	0.3	0.4	nd	nd	nd	nd
SHL4	nd	0.2	1.4	0.9	0.4	0.8	0.5	1.9	0.7	0.8	1.2
AB-1	nd	0.7	4.0	4.8	5.6	3.6	7.8	5.2	7.0	6.5	5.1
SHL6	nd	0.4	0.8	0.9	1.4	2.0	1.3	1.8	1.1	1.1	nd
Parent	98.0	80.6	38.8	17.5	7.1	6.3	5.0	4.4	2.6	2.2	1.8
SHL7	nd	nd	1.1	1.9	0.7	1.3	0.7	0.6	0.4	1.9	1.6
SHL8	nd	nd	1.7	1.8	1.3	1.6	1.1	2.2	1.9	0.9	2.2

Component	% AR										
	0 d	1 d	3 d	6 d	10 d	14 d	30 d	59 d	90 d	121 d	181 d
AB-1 dimer	nd	1.4	4.6	5.0	7.9	7.1	5.8	3.8	4.4	3.5	3.3
AB-1 (cis)	nd	1.7	4.2	6.0	1.0	6.5	1.1	4.3	1.4	1.2	1.2
AB-1 (trans)	nd				7.1		4.6		4.1	4.1	4.1
Others	0.3	1.1	1.7	3.1	2.3	3.2	2.9	3.2	2.1	2.4	2.4

nd = Not detected

Table 16 Distribution of radioactivity after sterile incubation at 25 °C of sandy loam soil treated with butylphenyl- or benzoyl-label at 0.93 mg/kg

Time, d	% AR			
	Extracted	Unextracted	Volatiles	Mass balance
Butylphenyl-label				
0	103.5	< 0.1	na	103.5
1	100.7	0.8	< 0.1	101.5
7	93.0	8.4	< 0.1	101.4
30	61.0	42.7	4.1	107.8
Benzoyl-label				
0	100.2	< 0.1	na	100.2
1	99.0	0.7	< 0.1	99.7
7	94.6	5.3	< 0.1	99.9
30	83.6	19.7	< 0.1	103.3

Na = Not applicable

Table 17 Identification of radioactivity after sterile incubation at 25 °C of sandy loam soil treated with butylphenyl or benzoyl-label at 0.93 mg/kg

	% AR							
	Butylphenyl-label				Benzoyl-label			
	0 d	1 d	7 d	30 d	0 d	1 d	7d	30 d
Polar comp.	nd	nd	nd	7.3	nd	nd	0.4	5.3
SHL1	nd	nd	nd	nd	na	na	na	na
B-1	na	na	na	na	nd	1.8	12.0	55.1
SHL3	nd	nd	0.8	7.7	nd	nd	nd	nd
SHL4	nd	0.4	0.7	1.9	nd	nd	nd	nd
AB-1	nd	0.6	2.7	6.5	nd	0.5	2.6	3.0
Parent	95.4	97.0	85.0	30.8	97.7	95.1	77.9	17.0
SHL7	nd	nd	0.9	1.8	nd	nd	nd	1.1
SHL8	nd	nd	nd	0.9	nd	nd	0.3	nd
AB-1 dimer	nd	nd	0.4	0.4	nd	nd	nd	nd
AB-1 (cis/trans)	nd	nd	nd	0.7	nd	nd	nd	nd
Others	4.6	2.0	2.5	3.1	2.3	1.6	1.4	2.2

Na = Not applicable

nd = Not detected

Study 2 (Willems, 2008a)

Basing on the above study results, DT_{50} and DT_{90} for the degradation of cyflumetofen in soil were calculated following FOCUS guidance of EU. Optimization was by the Marquardt method using ordinary least squares weighting.

Cyflumetofen degrades in Wolston sandy loam soil (at 25 °C) with a half-life of 1.81–2.29 days (mean 2.05 d, persistence endpoint). Degradation was described by DFOP kinetics. For modelling a mean $DegT_{50}$ of 2.38 days was calculated (correction for soil moisture not included).

Study 3 (Ta and Strobush, 2011a)

The metabolism of cyflumetofen in four US soils under aerobic conditions was investigated using benzoyl- and butylphenyl-label. Four soils of different textures (loamy sand and loam soils) collected from different locations representing various geographical regions in the USA were used. Cyflumetofen was applied at the rate of 0.8 mg ai/kg soil, equivalent to 200 g ai/ha.

Soil equivalent to 50 g of dry weight was weighed into 250 mL wide-mouth polypropylene centrifuge bottles. The soils were maintained aerobically by connecting to an air flow-through system in the dark at 20 ± 2 °C for 120 days. The soil moistures were adjusted to approximately 40–60% of the maximum water holding capacity (MWHC) prior to application of test material to the soil and maintained throughout the incubation period by adding HPLC water if necessary. Moisturized CO₂-free air was passed over the soil surface and air leaving the system was passed through 1 N NaOH solution to trap any CO₂ produced. Duplicate samples were analysed at 0, 1, 3, 7, 16, 29, 58, and 120 days after treatment (DAT). The soil samples were extracted once with acetonitrile, followed by 70% acetonitrile in water twice, and trifluoroacetic acid was added to bring the extract solution to a concentration of 0.1% acid to prevent any hydrolysis that may occur. The solvent extracts were pooled, concentrated and analysed by HPLC. Identification of parent and its transformation products was performed by retention matching with reference standards by HPLC and confirmation by LC/MS and/or NMR. The amounts of CO₂ and organic volatiles produced were determined at each sampling time (excluding 0 DAT). The traps were assayed directly by adding aliquots of the trapping solutions into liquid scintillation fluid and counting by liquid scintillation counting.

Table 18 Soil properties

Geographic Location	CA, USA	IN, USA	NJ, USA	WI, USA
Soil series	Corralitos	Crosby	Penn	Burkhardt
Classification	Mixed, thermic, Typic Xeropsamments	Fine, mixed, active, mesic Aeric Epiaqualfs	Fine-loamy, mixed, superactive mesic Ultic Hapludalfs	Sandy, mixed, mesic Typic Hapludolls
Texture Class	Loamy Sand	Loam	Loam	Loamy Sand
Sand (%)	87	35	29	87
Silt (%)	8	46	44	6
Clay (%)	5	19	27	7
pH (in water)	8.1	6.5	6.9	6.3
Organic Matter (%)	0.49	2.30	2.30	2.70
Organic Carbon % (1)	0.28	1.33	1.33	1.57
Soil Biomass (µgC/ g soil)				
0 DAT	43.2	160.9	139.8	128.5
120 DAF	55.3	127.9	54.2	86.8
Cation Exchange Capacity (CEC, meq/100 g)	6.6	9.0	8.1	8.4
Max Moisture Holding Capacity (%)	18.8	36.6	43.3	28.0
Bulk Density g/cc (disturbed)	1.42	1.04	1.11	1.34

The average total recovery of the radiolabelled material was 95.5% of the total applied radioactivity (TAR) for all samples from four soils with two labels.

California soil:

The total radioactivity recovered from the benzoyl-label ranged from 97.1 to 103.3% of the total applied radioactivity (TAR) with an average of 99.4% TAR. Radioactive residues extracted by solvent decreased from approximately 99.7–100% TAR at 0 day after treatment (DAT) to approximately 67.6–77.4% TAR at 120 DAT. Unextracted radioactive residues increased from approximately 0.3% TAR at 0 DAT to 16–21.6% TAR at 120 DAT. The concentration of the parent compound decreased from approximately 98.1–98.2% TAR at 0 DAT to approximately 3–5% TAR at 120 DAT. There

were three major (> 10% TAR) transformation products identified as B1, B3, and AB1 + AB1 dimer 2. At 120 DAT, the total evolved $^{14}\text{CO}_2$ was 9.9% TAR.

Indiana soil:

The total radioactivity recovered from the benzoyl-label ranged from 87 to 100.6% TAR with an average of 94.3% TAR. Radioactive residues extracted by solvent decreased from approximately 99–100.2% TAR at 0 DAT to approximately 31.5–32.6% TAR at 120 DAT. Unextracted radioactive residues increased from approximately 0.4% TAR at 0 DAT to 37.8–38.0% TAR at 120 DAT. The concentration of the parent compound decreased from approximately 97.6–98.9% TAR at 0 DAT to approximately 1.2–1.9% TAR at 120 DAT. There were 3 major (> 10% TAR) transformation products identified as B1, AB1 + AB1 dimer 1, and AB1 + AB1 dimer 2. At 120 DAT, the total evolved $^{14}\text{CO}_2$ was 17.6% TAR.

New Jersey soil:

The total radioactivity recovered from the benzoyl-label ranged from 90.2 to 101.5% TAR with an average of 95.4% TAR. Radioactive residues extracted by solvent decreased from approximately 100.5–101% TAR at 0 DAT to approximately 37.6–38.7% TAR at 120 DAT. Unextracted radioactive residues increased from approximately 0.5% TAR at 0 DAT to 31.1–32.7% TAR at 120 DAT. The concentration of the parent compound decreased from approximately 98.8–99.3% TAR at 0 DAT to approximately 1.3–2.7% TAR at 120 DAT. There were 3 major (> 10% TAR) transformation products identified as B1, AB1 + AB1 dimer 1, and AB1 + AB1 dimer 2. At 120 DAT, the total evolved $^{14}\text{CO}_2$ was 24.1% TAR.

The total radioactivity recovered from the butylphenyl-label ranged from 90.3 to 100% TAR with an average of 93.7% TAR. Radioactive residues extracted by solvent decreased from approximately 97.6–99.5% TAR at 0 DAT to approximately 33.8–36.2% TAR at 120 DAT. Unextracted radioactive residues increased from approximately 0.5% TAR at 0 DAT to 41.2–43.1% TAR at 120 DAT. The concentration of the parent compound decreased from approximately 94.5–96.6% TAR at 0 DAT to approximately 2.5–2.6% TAR at 120 DAT. There was 1 major (> 10% TAR) transformation product identified as AB1. At 120 DAT, the total evolved $^{14}\text{CO}_2$ was 17.3% TAR.

Wisconsin soil:

The total radioactivity recovered from the benzoyl-label ranged from 88.3 to 100.1% TAR with an average of 95.2% TAR. Radioactive residues extracted by solvent decreased from approximately 98.1–99.9% TAR at 0 DAT to approximately 39.8% TAR at 120 DAT. Unextracted radioactive residues increased from approximately 0.3% TAR at 0 DAT to 31.5–31.6% TAR at 120 DAT. The concentration of the parent compound decreased from approximately 96.8–98.4% TAR at 0 DAT to approximately 2.8–3.2% TAR at 120 DAT. There were four major (> 10% TAR) transformation products identified as B1, AB1, AB1 + AB1 dimer 1, and AB1 + AB1 dimer 2. At 120 DAT, the total evolved $^{14}\text{CO}_2$ was 17.3% TAR.

Table 19 Summary of aerobic soil metabolism study 2 results

Soil	Mass balance (Total recovery)	Degradation of cyflumetofen in 120 days	Half-life/ Dissipation time
CA soil	97.1 to 103.3% TAR	93.2–95.1%	3.51 days ^a
IN soil	87.0 to 100.6% TAR	96.4–97.0%	2.28 days ^b
NJ soil	90.2 to 101.5% TAR	92.0–97.5	2.36 days ^b
WI soil	88.3 to 100.1% TAR	93.6–95.6%	3.41 days ^b

^a First order multi compartment

^b Single first order

Study 4 (Brands, 2011a)

Benzoyl-label was incubated for 120 days in the dark in 100 g dw equivalent each of three soils (Speyer 2.2, loamy sand; Speyer 2.3, sandy loam; and Speyer 6S, clay), which were equilibrated for 20–21 days in the dark, at 20 ± 2 °C under aerobic conditions at a concentration of 0.383 mg/kg (~0.8% acetonitrile) on a dry weight basis. A humidified air stream was passed through the soil containers (2 x 30 minutes/day). The outgoing air was passed through a polyurethane foam plug, a trap containing 2-ethoxyethanol and two traps containing 2M NaOH to trap volatile substances and CO₂. Soil moisture was kept constant at 45% of the MWHC throughout the incubation period. Soil biomass was determined at the start and end of the incubation period by the fumigation/extraction method.

Speyer 2.2, 2.3 and 6S soils, after sieving (2 mm), were used within two months (storage at 4 °C) of field sampling. The properties of these soils are shown in the following Table.

Table 20 Properties of the three soils used in the study.

Property	Speyer 2.2	Speyer 2.3	Speyer 6S
Origin	Hanhofen, Rheinland-Pfalz, Germany	Offenbach, Rhein-land-Pfalz, Germany	Siebelingen, Rhein-land-Pfalz, Germany
Texture (USDA)	Loamy sand	Sandy loam	Clay
Sand (% , USDA)	79.1	61.0	21.9
Silt (% , USDA)	13.5	29.8	36.0
Clay (% , USDA)	7.9	9.2	42.0
pH-water	-	-	-
pH-KCl	-	-	-
pH-CaCl ₂	5.6	6.2	7.0
% OM	4.07	1.76	3.26
% OC	2.36	1.02	1.89
CEC (meq/100g)	11	9	20
MWHC (% , w/w)	48.0	34.9	42.3

Single soil portions and associated traps were sampled at 0, 1, 3, 6 (duplicate sample), 10, 14, 21, 35 (duplicate sample), 58, 90 and 120 days. Soil samples were extracted three times with 100 mL acetonitrile/sodium acetate buffer pH 4 (75:25, v/v). Samples of day 35 were also extracted by n-hexane and acetonitrile-Soxhlet extraction (3 hours). Polyurethane foam plugs were extracted with acetonitrile (2×). Radioactivity in extracts and liquid traps was determined by LSC and in post-extraction solids by combustion/LSC. Soil extracts were fractionated in an organic and aqueous phase. Relevant fractions were subjected to analyses by HPLC-RAM and TLC. Metabolites were identified by comparison with reference substances of metabolites and LC-MS/MS analysis. Radioactivity in the NaOH traps was confirmed as CO₂ by precipitation with Ba(OH)₂.

Soil biomass was 1.1–2.1% of OC at the start and 0.3–2.7% at the end of the experiment. In combination with significant mineralization at the end of the study, sufficiently viable conditions were indicated.

Mass balances were between 87.9 and 109.8% of applied radioactivity (AR). No specific trend was observed and all results were considered acceptable.

The amount of extractable residues decreased from 103.5–104.6% AR to 54.1 (Speyer 2.2), 26.9 (Speyer 2.3) and 62.2% AR (Speyer 6S) at study end (day 120). Unextractables were maximum 32.1 (Speyer 2.2, day 58), 40.1 (Speyer 2.3, day 120) and 38.4% AR (Speyer 6S, day 35). Additional extraction with n-hexane (day 35 sample) released 1.6–3.2% AR radioactivity and with Soxhlet 1.8–5.1% AR.

Volatile radioactivity consisted mostly of CO₂ and was 9.5 (Speyer 2.2), 20.7 (Speyer 2.3) and 1.7% AR (Speyer 6S) at day 120 (study end) (0.8, 16.2 and 1.8% AR, respectively at day 90).

Unextracted radioactivity was at a maximum of 32.1 (Speyer 2.2, day 58), 40.1 (Speyer 2.3, day 120) and 38.4% AR (Speyer 6S, day 35).

Cyflumetofen degraded to < 10% AR after 21 days in Speyer 2.1 soil, to < 10% AR after 10 days in Speyer 6S soil and to ~10% AR at day 120 in Speyer 2.3 soil under aerobic conditions in the dark at 20 °C.

Those metabolites exceeding 10% AR or 2× 5% at two consecutive time points were B-1 (max 63.0% AR, day 90, Speyer 2.2) and B-3 (max 23.0% AR, day 21, Speyer 2.2). No other metabolites exceeding 5% AR were observed in any soil.

Table 21 Distribution of radioactivity after aerobic incubation at 20 °C of Speyer 2.2, 2.3 and 6S soils treated with benzoyl-label at 0.383 mg/kg

Time d	% AR				
	Organic volatiles	CO ₂	Extracted radioactivity	Unextracted radioactivity	Mass balance
Speyer 2.2 Soil					
0	na	Na	103.7	2.6	106.3
1	0.0	0.0	90.2	5.4	95.6
3	0.0	0.1	94.1	9.1	103.3
6	0.0	0.1	82.5	14.1	96.7
6	0.0	0.0	69.1	18.7	87.9
10	0.0	1.3	79.2	20.4	101.0
14	0.1	1.4	76.6	16.1	94.2
21	0.0	1.2	76.8	18.1	96.2
35	0.3	5.1	67.3	21.2	93.9
35	0.1	0.7	72.6	22.5	95.9
58	0.4	1.1	72.5	32.1	106.1
90	0.2	0.8	77.2	18.3	96.6
120	1.4	9.5	54.1	30.1	95.0
Speyer 2.3 Soil					
0	na	na	104.6	0.9	105.5
1	0.0	0.1	81.7	7.4	89.1
3	0.1	0.5	78.4	19.3	98.4
6	0.1	0.8	66.7	22.4	90.0
6	0.1	0.3	75.7	24.2	100.4
10	0.1	2.8	68.6	27.8	99.2
14	0.1	3.2	71.4	19.4	94.1
21	0.1	3.5	71.2	20.8	95.8
35	0.2	4.4	71.1	24.8	100.4
35	0.1	7.0	61.0	25.1	93.2
58	0.2	11.4	41.5	38.3	91.4
90	0.1	16.2	41.4	33.3	91.0
120	0.1	20.7	26.9	40.1	87.8
Speyer 6S soil					
0	na	na	103.5	6.3	109.8
1	0.0	0.0	80.6	21.4	102.0
3	0.0	0.0	75.6	26.0	101.6
6	0.1	0.4	67.2	33.4	101.1
6	0.0	0.3	70.5	30.7	101.5
10	0.2	1.0	61.7	34.6	97.5
14	0.1	0.7	57.0	32.1	89.9
21	0.1	0.4	64.9	29.1	94.4
35	0.1	0.0	55.1	37.4	92.6
35	0.1	0.0	59.6	38.4	98.1
58	0.2	1.2	66.3	37.1	104.8
90	0.4	1.8	60.6	32.1	94.9
120	0.4	1.7	62.2	33.0	97.4

na: not applicable

Table 22 Identification of radioactivity after aerobic incubation at 20 °C of Speyer 2.2, Speyer 2.3 and Speyer 6S soils treated with Benzoyl-label at 0.383 mg/kg

Time d	% AR			
	Cyflumetofen	B-1 ^b	B-3 ^b	Others ^a
Speyer 2.2 Soil				
0	97.5	3.0	0.0	0.0
1	94.1	3.8	0.0	0.0
3	75.7	8.9	1.8	4.0
6	31.6	35.7	4.9	5.9
6	29.6	34.3	14.2	0.0
10	22.4	35.4	16.1	3.5
14	17.4	47.1	12.4	0.0
21	6.7	55.2	23.0	0.0
35	14.7	35.3	5.6	12.9
35	7.1	48.7	13.8	6.5
58	14.7	39.9	9.9	1.6
90	2.6	63.0	8.3	6.2
120	10.2	33.8	5.2	7.1
Speyer 2.3				
0	96.2	7.9	0.0	0.0
1	80.3	13.5	0.0	0.0
3	54.4	22.3	0.0	0.0
6	23.4	34.3	12.7	2.1
6	26.4	37.2	3.6	6.9
10	22.3	36.6	4.7	5.6
14	20.9	36.1	2.1	8.2
21	14.7	42.8	3.2	8.9
35	15.2	43.2	3.5	9.0
35	14.6	36.3	1.9	8.4
58	12.5	12.6	1.0	15.4
90	11.2	16.4	0.0	15.0
120	10.6	2.6	0.0	15.7
Speyer 6S				
0	95.1	4.3	0.0	0.0
1	70.1	13.3	0.0	0.0
3	40.8	23.2	0.0	9.4
6	14.9	32.4	2.0	8.4
6	21.8	36.4	3.2	14.1
10	6.4	42.3	2.3	7.8
14	4.8	46.2	3.1	5.9
21	5.9	39.4	4.8	7.9
35	3.4	42.3	1.1	7.3
35	3.0	45.5	2.5	5.9
58	5.0	52.8	1.3	7.5
90	4.8	42.4	0.0	11.5
120	5.4	43.0	0.0	11.6

^a individual fractions <5% AR^b confirmed by co-chromatography with reference standard (HPLC, TLC (only B-1), LC-MS/MS)

DT₅₀ and DT₉₀ values for calculated by fitting single first order (SFO), first order multi-compartment (FOMC) and double first order in parallel (DFOP) models to the data and the best fit was obtained by DFOP model (based on visual examination, r^2 and χ^2). Calculated DT₅₀ and DT₉₀ values are shown in the following Table.

Table 23 Half-lives for degradation of cyflumetofen at 20 °C under aerobic conditions (double first order in parallel was assumed)

Soil	M ₀ %	g	k ₁ days ⁻¹	k ₂ days ⁻¹	DT ₅₀ days	DT ₉₀ days	r ²	χ ² (err)
Speyer 2.2	105 ± 6.0	0.91 ± 0.03	0.197 ± 0.030	0	4.0	21	0.960	15.3
Speyer 2.3	98.8 ± 3.1	0.83 ± 0.03	0.303 ± 0.035	0.004 ±	3.0	130	0.988	7.3

Soil	M ₀ %	g	k ₁ days ⁻¹	k ₂ days ⁻¹	DT ₅₀ days	DT ₉₀ days	r ²	χ ² (err)
				0.003				
Speyer 6S	95.0 ± 1.7	0.95 ± 0.01	0.312 ± 0.014	0	2.4	9.2	0.996	3.5

Study 5 (Willems, 2008a)

The degradation of cyflumetofen in three soils (Speyer 2.2, 2.3 and 6S) was studied by Brands in 2008 (NOTOX report 478338; 7.2.1/01), in which benzoyl-label was spiked to these soils and incubated for 120 days at 20 °C in the dark. Using the degradation results, DT₅₀ and DT₉₀ were calculated for cyflumetofen in accordance with FOCUS guidance ((SANCO/10058/2005, vs 2.0, June 2006). Optimization was by the Marquardt method using ordinary least squares weighting.

Table 24 Cyflumetofen degradation in Speyer 2.2, 2.3 and 6S soils

Time d	Cyflumetofen (% of applied)		
	Speyer 2.2	Speyer 2.3	Speyer 6S
0	100.1 ^a	97.1 ^b	101.4 ^c
1	94.1	80.3	70.1
3	75.7	54.4	40.8
6	31.6	23.4	14.9
6	29.6	26.4	21.8
10	22.4	22.3	6.4
14	17.4	20.9	4.8
21	6.7	14.7	5.9
35	14.7	15.2	3.4
35	7.1	14.6	3.0
58	14.7	12.5	5.0
90	2.6	11.2	4.8
120	10.2	10.6	5.4

^a including 2.6% unextracted residues

^b including 0.9% unextracted residues

^c including 6.3% unextracted residues

According to FOCUS guidance, first, single first order (SFO) and first order multi-compartment (FOMC) kinetics were run. Time zero results for cyflumetofen were adjusted for unextracted residues but not for metabolites because the radioactivity present as metabolites was similar to the amount of impurities in the spike solutions. The possibility for elimination of data points or constraining of M₀ was investigated, and when FOMC still resulted in a better fit, also dual first order in parallel (DFOP) kinetics were fitted to the data. DegT₅₀ and DegT₉₀ values for persistence were taken from the best fit model. For modelling, the SFO fit was first evaluated and when not acceptable, FOMC or DFOP were also assessed. In case FOMC or DFOP was suited for modelling, the DegT₅₀ for modelling was calculated as DegT₉₀/3.32.

Persistence and modelling endpoints for the degradation of cyflumetofen in soil were calculated and are shown in the following Table.

Table 25 Persistence and modelling endpoints for degradation of OK-5101 in Speyer soils

Soil	Persistence			Modelling
	DT ₅₀ at 20 °C days	DT ₉₀ at 20 °C days	model	DT ₅₀ at 20 °C ^a Days
Speyer 2.2	4.33	23.10	DFOP	6.70
Speyer 2.3	3.13	134.10	DFOP	13.30
Speyer 6S	2.20	8.40	DFOP	2.95

^a not including moisture correction.

Study 6 (Brands & Corral, 2011b)

The degradation of B-1 in soil was investigated in three soils (the same soils as in Study 4). A 50 g (dw) portion each of three soils (Speyer 2.2, loamy sand; Speyer 2.3, sandy loam; and Speyer 6S, clay), which were sieved (2 mm), stored at 4 °C and used within 4 weeks of field sampling, was equilibrated for 4 days in the dark at a moisture content of ~40% of the MWHC at 20 °C. Following equilibration, soil portions were treated with B-1 in acetonitrile/water 1/9 (v/v) at a rate of 0.25 mg/kg dw (~0.16% acetonitrile). The test substance was gently mixed in the soil. Incubation took place in the dark at 20±2 °C under aerobic conditions for 120 days. Soil moisture was kept constant at ~40% of the MWHC throughout the incubation period.

Soil biomass was determined at the start and end of the incubation period by the fumigation/extraction method. Single soil portions were sampled at 0, 1, 4 (duplicate sample), 10, 28 (duplicate sample), 70 and 120 days. Soil samples were extracted with 250 mL acetonitrile/water (10/90 v/v) for 30 minutes. The samples were filtered over a 0.2 µm filter prior to analysis by UPLC-MS/MS.

Biomass was > 1% of OC throughout the study. Actual moisture contents (dw basis) throughout the study were 20% (Speyer 2.2), 14% (Speyer 2.3) and 16% (Speyer 6S), equivalent to ~39% of the MWHC.

Table 26 Degradation of B-1 during aerobic incubation at 20 °C in soil treated with B-1 at 0.25 mg/kg

Time d	B-1 (% of applied)		
	Speyer 2.2	Speyer 2.3	Speyer 6S
0	102	105	100
1	93	101	95
4	72	91	95
4	68	93	93
10	34	70	94
28	0.5*	28	56
28	0.5*	33	63
70	na	16	16
120	na	1.3	20

na: not applicable

* set at ½ LOQ

DT₅₀ and DT₉₀ values for B-1 were obtained by fitting single first order (SFO) kinetics to the data. The results are given in the following Table. The FOMC model was also verified and gave similar results as the SFO model, but optimization errors were much larger compared to the SFO model. Because there was also no biphasic pattern visible, the SFO model was selected as best fit model.

Table 27 Half-lives for degradation of B-1 at 20 °C under aerobic conditions based on SFO kinetics ^a

Soil	M ₀ %	K day ⁻¹	DT ₅₀ days	DT ₉₀ Days	r ²	χ ² (err)
Speyer 2.2	104 ± 2.8	0.110 ± 0.008	6.3	21.0	0.993	4.6
Speyer 2.3	106 ± 2.7	0.041 ± 0.003	16.7	55.5	0.988	5.3
Speyer 6S	102 ± 3.7	0.019 ± 0.002	36.3	121	0.959	7.2

^a not corrected for moisture content.

B-1 degraded in three soils under aerobic conditions in the dark at 20 °C with half-lives of 6.3, 16.7 and 36.3 days and DT₉₀ values of 21.0, 55.5 and 121 days. These values are not normalized for moisture. Degradation was described by SFO kinetics.

Study 7 (Corral & Brands, 2010c)

AB-1 was studied by incubating for 120 days in three soils (Speyer 2.1, sand; Speyer 2.2, loamy sand; and Speyer 6S, clay) in the dark at 20 °C under aerobic conditions at a concentration of 0.04 mg/kg

dw. Portions of 50 g dw equivalent soil were equilibrated for 11 days in the dark at a moisture content of ~40% of the MWHC at 20 °C. Following equilibration, soil portions were treated with AB-1 in acetonitrile at a rate of 0.04 mg/kg dw. The test substance was gently mixed in the soil. Incubation took place in the dark at 20±2 °C under aerobic conditions for 120 days. Soil moisture was kept constant at ~40% of the MWHC throughout the incubation period. Soil biomass was determined at the start and end of the incubation period by the fumigation/extraction method.

Single soil portions were sampled at 0, 0.25 (duplicate sample), 1, 2 (duplicate sample), 4, 29, 72 and 120 days. Soil samples were extracted with 400 mL acetonitrile/water (50/50 v/v) containing 0.05 or 0.1% (v/v) ammonium solution for 30 minutes. The samples were filtered over a 0.2 µm filter prior to analysis by UPLC-MS/MS (fully validated, see OTSA-0368).

Biomass was >1% of OC throughout the study. Actual moisture contents (dw basis) throughout the study were 13% (Speyer 2.1), 20% (Speyer 2.2) and 16% (Speyer 6S), equivalent to 39-40% of the MWHC

Table 28 Soil properties of Speyer 2.1, 2.2 and 6S soils

Property	Speyer 2.1	Speyer 2.2	Speyer 6S
Origin	Dudenhofen, Rheinland-Pfalz, Germany	Hanhofen, Rheinland-Pfalz, Germany	Siebelingen, Rheinland-Pfalz, Germany
Texture (USDA)	Sand	Loamy sand	Clay
Sand (% , USDA)	88.1	81.2	21.7
Silt (% , USDA)	8.5	12.1	35.7
Clay (% , USDA)	3.4	6.7	42.6
pH-water	-	-	-
pH-KCl	-	-	-
pH-CaCl ₂	5.1	5.4	7.2
% OM	1.52	4.0	3.1
% OC	0.88	2.29	1.79
CEC (meq/100g)	4	10	20
water content at pF2 (% , w/w)	-	-	-
MWHC (% , w/w)	33.3	49.7	40.1

Material balance was between 85 and 123% for these soils. The degradation of AB-1 over time in these soils is shown in the following table.

Table 29 Degradation of AB-1 during aerobic incubation at 20 °C in soil treated with AB-1 at 0.04 mg/kg

Time d	AB-1 (% of applied)		
	Speyer 2.1	Speyer 2.2	Speyer 6S
0	95	88	90
0.25	18	18	22
0.25	18	19	21
1	14	13	11
2	15	14	9
2	16	14	10
4	12	12	10
29	12	11	8
72	11	9	6
120	8	6	3

DT₅₀ and DT₉₀ values for AB-1 were obtained by fitting single first order (SFO), first-order multi-compartment (FOMC), dual first order in parallel (DFOP) and hockey-stick (HS) kinetics to the data. As there was a clear bi-phasic pattern observed, the HS and DFOP models described the results the best (and equally good). According to FOCUS guidance, the HS model is not used for obtaining endpoints for persistence, therefore the DFOP model was selected for use in exposure assessment. The calculated results are shown in the following table.

AB-1 degraded in three soils under aerobic conditions in the dark at 20 °C with half-lives of 0.07, 0.08 and 0.11 days (not corrected for moisture). Degradation was described by DFOP kinetics.

Table 30 Half-lives for degradation of AB-1 at 20 °C under aerobic conditions based on DFOP kinetics ^a

Soil	M ₀ %	k ₁ , k ₂ and g	DT ₅₀ days	DT ₉₀ Days	r ²	χ ² (err)
Speyer 2.1	95.0 ± 1.2	k ₁ = 12.40 ± 1.13 k ₂ = 0.005 ± 0.001 g = 0.847 ± 0.007	0.07	90.8	0.998	3.9
Speyer 2.2	88.0 ± 0.7	k ₁ = 10.75 ± 0.50 k ₂ = 0.006 ± 0.001 g = 0.846 ± 0.004	0.08	69.3	0.999	2.5
Speyer 6S	90.0 ± 0.7	k ₁ = 7.81 ± 0.22 k ₂ = 0.009 ± 0.001 g = 0.885 ± 0.004	0.11	15.7	0.999	2.7

^a not corrected for moisture

Photodegradation on soil surface (van Noorloos, 2007a and 2007b)

Photodegradation of cyflymetofen on soil surface was investigated using butylphenyl- and benzoyl-label cyflumetofen.

Butylphenyl- or benzoyl-label was applied uniformly over the surface of thin soil layers at a rate of 401 g ai/ha or 391–395 g ai/ha, respectively. The soil layers were irradiated (300–400 nm: 66.5 Watt/m²) for 15 days (equivalent to 33–38 days natural sunlight at 40 °N, summer) at 18–23 °C or for 14 days (equivalent to 35–36 days natural sunlight at 40 °N, summer) at 19–22 °C, respectively, with artificial light (Xenon lamp) simulating the sunlight spectrum. Control samples were incubated in the dark at approximately 20 °C. Duplicate samples were taken after 0, 1, 4, 7, 11 and 15 days of irradiation/incubation. Soil layers were extracted with (75/25 (v/v)) acetonitrile/sodium acetate buffer pH 4. Following fractionation into an organic and aqueous phase the organic phase was analysed by reversed phase HPLC-RAM and selected samples also by TLC. The aqueous phase was not further analysed due to low levels of radioactivity. Polyurethane foam plugs were extracted by acetonitrile and soda lime granules were dissolved in 2 M HCl and released CO₂ was trapped in 2 M NaOH. Radioactivity in liquid fractions was determined by LSC and in residual soil by combustion/LSC. Identification of radioactive fractions was obtained by co-chromatography with reference standards (HPLC and TLC).

Table 31 Soil properties of Speyer 3A loam

Property	Value
Origin	Altlußheim, Baden-Württemberg, Germany
Texture	Loam
Sand (%)	46.7
Silt (%)	35.5
Clay (%)	17.7
pH-water	-
pH-KCl	-
pH-CaCl ₂	7.2
% OM	3.7
% OC	2.17
CEC (meq/100g)	19
water content at pF2 (% w/w)	-
MWHC (% w/w)	48.4

Mass balances

Butylphenyl-label: Mass balances were between 81.4 and 105.4% of applied radioactivity (AR) for the irradiated samples and between 87.2 and 102.0% AR for the dark control samples. Unextracted

radioactivity was 44.6–45.6% AR (irradiated, study end) and 45.0% AR (dark control, study end, mean). Volatile radioactivity was at the maximum 2.5% AR in any sample.

Benzoyl-label: Mass balances were between 96.4 and 104.7% of applied radioactivity (AR) for the irradiated samples and dark control samples. Unextractables were approximately 24% AR (irradiated, study end) and approximately 39% AR (dark control, study end). Volatile radioactivity was insignificant ($\leq 0.5\%$ AR).

Cyflumetofen dissipation

Butylphenyl-label: Cyflumetofen dissipated with a single first order DT_{50} of 1 and 0.95 days in irradiated soil and dark controls, respectively. No metabolites exceeded 10% AR. Specific photolytic metabolites were not observed. All metabolite fractions observed in irradiated soil were also present in similar amounts in dark controls.

Benzoyl-label: Cyflumetofen dissipated with a single first order DT_{50} of 1.4 days in irradiated soil and dark controls, respectively. One significant metabolite, B-1, exceeded 10% AR in both the irradiated and dark soil. Specific significant ($> 10\%$ AR) photolytic metabolites were not observed.

Table 32 Distribution of radioactivity after irradiation of soil layers treated with butylphenyl- or benzoyl-label

Time d	Time sunlight equivalent ^b	% AR					
		PUF	CO ₂	residue in soda lime	extracted ^a	unextracted	mass balance
Butylphenyl-label							
0.0	0.0	Na	na	Na	92.2	6.5	98.6
	0.0	Na	Nas	Na	98.8	6.6	105.4
0.7	1.6	0.0	0.0	0.0	77.9	22.0	99.9
	1.8	0.0	0.0	0.0	79.7	20.1	99.8
3.8	8.2	0.0	1.6	0.1	46.0	37.7	85.4
	9.5	0.0	0.1	0.0	50.5	41.6	92.2
6.8	15.4	0.3	0.0	0.4	53.5	33.0	87.2
	17.7	0.0	1.3	0.7	34.4	44.9	81.4
10.8	24.5	0.0	0.7	0.7	39.2	44.0	84.7
	28.3	0.0	0.6	0.8	45.2	47.7	94.4
14.7	32.6	0.1	1.8	1.7	37.4	45.6	86.7
	37.6	0.0	2.5	1.6	41.9	44.6	90.6
Benzoyl-label							
0.0	0.0	na	Na	Na	97.1	6.6	103.7
	0.0	na	Na	Na	98.5	5.3	103.8
0.9	2.2	0.0	0.0	0.0	82.3	19.0	101.4
	2.3	0.0	0.0	0.0	79.6	25.0	104.7
2.8	7.2	0.0	0.0	0.0	79.4	21.0	100.5
	7.4	0.0	0.1	0.0	78.1	19.7	97.9
5.8	14.8	0.0	0.4	0.1	74.0	25.5	100.1
	14.6	0.0	0.3	0.1	76.0	23.1	99.4
9.8	25.5	0.0	0.4	0.1	72.6	25.1	98.2
	24.1	0.0	0.5	0.3	75.0	23.3	99.1
13.8	35.0	0.0	0.3	0.0	73.5	22.8	96.6
	35.9	0.0	0.3	0.2	71.8	24.5	96.8

Na = Not applicable

^a 0.9 to 4.2% AR remained in aqueous phase after partitioning (butylphenyl-label) and 0.8 to 21.6% AR remained (benzoyl-label) increasing trend with time.

^b summer, 40 °N

Table 33 Distribution of radioactivity in dark controls treated with butylphenyl- and benzoyl-label

Time d	% AR					
	PUF	CO ₂	residue in soda lime	extracted ^a	Unextracted	mass balance
Butylphenyl-label						
0.0	na	Na	na	95.5	6.5	102.0
0.7	0.0	0.0	0.0	73.4	27.7	101.1
3.8	0.1	0.0	0.0	49.8	42.2	92.1
6.8	0.0	0.2	0.0	47.5	44.1	91.8
10.8	0.0	0.6	0.0	47.5	39.1	87.2
14.7	0.0	0.5	0.3	46.2	45.0	92.1
Benzoyl-label						
0.0	na	na	na	97.8	5.9	103.8
0.9	0.0	0.0	0.0	74.8	21.6	96.4
2.8	0.0	0.0	0.0	61.3	37.2	98.6
5.8	0.0	0.0	0.0	56.4	40.0	96.4
9.8	0.0	0.0	0.0	59.8	38.2	98.0
13.8	0.0	0.0	0.0	58.7	38.7	97.5

na = Not applicable

^a 0.9 to 3.8% AR remained in aqueous phase after partitioning (butylphenyl-label) and 0.8 to 19.4% AR remained (benzoyl-label) increasing trend with time.

Table 34a Identification of radioactivity in irradiated soil and dark controls treated with butylphenyl-label

Time d	Time natural sunlight d	% AR						
		Parent 14.5–14.8 min	Met 2 6.8-6.9 min	Met 9 ^b 17.1–17.4 min	Met 10 19.1–19.5 min	Met 11 ^c 20.8–21.1 min	Met 12 ^d 21.4–21.7 min	Sum of others ^a
Butylphenyl-label								
0.0	0.0	47.9	0.3	0.5	0.1	0.0	0.0	1.8
	0.0	79.7	0.4	0.6	0.2	0.0	0.0	2.6
0.7	1.6	53.0	4.7	1.6	1.5	1.2	1.0	6.7
	1.8	59.4	1.7	3.6	2.2	1.3	1.4	3.9
3.8	8.2	16.3	2.9	2.3	2.6	2.9	2.6	6.7
	9.5	12.1	17.7	2.7	2.6	2.8	2.3	3.5
6.8	15.4	11.5	9.5	9.6	7.8	2.5	2.1	3.3
	17.7	4.5	2.1	2.5	1.3	1.4	1.1	14.8
10.8	24.5	4.8	5.1	3.6	3.9	3.2	2.8	10.4
	28.3	6.5	8.8	6.4	4.8	2.5	2.2	3.6
14.7	32.6	3.2	3.2	2.9	4.4	4.2	4.8	8.5
	37.6	4.4	8.9	2.4	3.6	4.6	3.9	6.6
0.7	Dark	48.4	1.4	2.9	2.1	1.0	0.9	3.0
	Dark	60.0	1.7	3.5	2.6	1.5	1.2	1.1
3.8	Dark	13.7	4.5	3.9	4.9	4.3	3.7	8.5
	Dark	14.6	4.4	4.5	5.4	4.8	3.9	8.7
6.8	Dark	7.7	4.5	3.6	4.5	4.6	3.5	8.8
	Dark	8.5	4.6	3.9	5.0	5.2	3.9	9.5
10.8	Dark	8.6	3.2	5.6	6.4	6.2	5.2	8.5
	Dark	3.2	5.7	4.0	4.4	3.0	3.0	6.9
14.7	Dark	5.0	1.1	7.4	7.0	6.1	4.5	7.3
	Dark	5.9	3.8	5.0	6.0	5.4	4.5	9.2

Table 34b Identification of radioactivity in irradiated soil and dark controls treated with benzoyl-label

Time in Suntest (d)	Time natural sunlight (days)	% AR			
		Parent 14.3–14.7 min	Met 4 4.3–4.5 min ^{e, f}	Met 9 13.7–13.9 min ^g	Sum of other metabolites
Benzoyl-label					
0.0	0.0	91.2	2.4	0.0	0.0
	0.0	91.8			

Time in Suntest (d)	Time natural sunlight (days)	% AR			
		Parent 14.3–14.7 min	Met 4 4.3–4.5 min ^{e, f}	Met 9 13.7–13.9 min ^g	Sum of other metabolites
Benzoyl-label					
0.9	2.2	46.3	17.6	5.2	4.8
	2.3	45.8			
2.8	7.2	29.9	31.7	3.4	11.1 ^h
	7.4	23.5			
5.8	14.8	9.1	47.6	2.5	9.4 ^h
	14.6	13.4			
9.8	25.5	6.6	39.4	5.7	14.0 ^h
	24.1	3.6			
13.8	35.0	4.8	32.7	4.6	13.5 ^h
	35.9	3.0			
0.9	dark	57.1	16.0	0.0	2.8
	dark	62.4			
2.8	dark	18.2	30.9	0.0	9.1 ⁱ
	dark	17.8			
5.8	dark	7.9	32.4	0.0	7.5 ⁱ
	dark	9.4			
9.8	dark	8.0	36.1	0.0	7.2 ⁱ
	dark	8.7			
13.8	dark	5.7	37.7	0.0	7.7 ⁱ
	dark	11.5			

^a Each individual peak, < 5% of applied

^b Match with AB-11 based on retention time

^c Match with AB-12 based on retention time

^d Match with AB-13 based on retention time

^e Sum of activity in concentrated extract and aqueous residue

^f Match with B-1 based on retention time

^g Match with AB-8 based on retention time

^h Each individual peak < 5% of applied

ⁱ Each individual peak < 5% of applied, except one metabolite (Met 11) with retention time 19.0–19.2 min ($\leq 5.9\%$ of applied)

Single first order DT₅₀ and DT₉₀ values (in natural sunlight equivalent days) for irradiated soil and dark controls are presented in Tables 23–29. The results indicated there is no difference between the DT₅₀s in irradiated soils and dark controls.

Table 35 Half-lives for degradation of cyflumetofen in irradiated soil and dark controls

System	M ₀ %	K days ⁻¹	DT ₅₀ days	DT ₉₀ days	r ²	χ^2	Number of data points
Butylphenyl-label							
irradiated soil	99.1 ± 4.5	0.673 ± 0.101	1.0	3.4	0.972	13.37	12
dark control	99.3 ± 5.1	0.729 ± 0.123	0.95	3.2	0.965	14.95	12
Benzoyl-label							
Irradiated soil	86.7 ± 4.5	0.480 ± 0.063	1.4	4.8	0.960	15.4	12
Dark control	91.8 ± 4.0	0.502 ± 0.056	1.4	4.6	0.970	12.9	12

In conclusion, photolytic degradation on soil surfaces is not expected to play a role in the overall fate of cyflumetofen residues in soil, regardless of radio-labelled cyflumetofen used.

Hydrolytic degradation in aquatic systems (Nakamura, 2004a)

Portions of 2.5 mL butylphenyl- or benzoyl-label cyflumetofen in acetonitrile were added to 250 mL sterilized, deoxygenated buffer solutions of pH 4, 5, 7 and 9 at a concentration of 0.1 mg/L. The fortified buffer solutions were incubated in the dark at 25 ± 1 °C for 30 days (pH 4, 5 and 7), 10 days (pH 7, benzoyl-label) or 1 day (pH 9). Sterility and pH were checked at the beginning and end of the

incubation period. Samples were taken at various time points. Immediately after sampling, the pH 7 and pH 9 samples were acidified (acetic acid) to slow hydrolysis. Samples were partitioned into an organic phase (acetonitrile) and aqueous phase by C₁₈- SPE. Radioactivity was determined by LSC and the organic phase was subjected to chromatographic analysis (HPLC-RAM, TLC, LC-MS). The aqueous phase for the final sampling point and selected sampling points of benzoyl-label incubations was acidified (HCl) and subjected to PLS-2 SPE. Radioactivity in organic and aqueous phases was determined by LSC. The resulting organic phase (acetonitrile) was subjected to analysis using HPLC-RAM. Metabolite identification was done by comparison of retention times of HPLC, running factors (TLC) and MS spectra (LC-MS ion-trap) with reference standards of metabolites (A1, B1, AB-1, A-18). Metabolites for which no references were available were identified by LC-MS (ion-trap). Hydrolytic half-lives for cyflumetofen were calculated by linear regression using first order kinetics.

The mean total recovery for butylphenyl-label was 94.2 to 103.5% of applied radioactivity and that for benzoyl-label was 97.6 to 102.2%.

Degradation of cyflumetofen and its identified degradates at pH 4–9 at 25 °C are shown in the following Tables. Hydrolysis was fastest at pH 9 and slower in lower pH.

The degradation products of hydrolysis exceeding 10% of the applied radioactivity were A-1 (max 14.44% AR at pH 5–7), A-2 (max 44.12% AR at pH 5–7), A-18 (max 36.22% AR at pH 5–7), AB-1 (max 44.51% AR at pH 5–7) and B-1 (max 53.17% AR at pH 5–7). Other unknown fractions were all ≤ 7.76% AR, with the exception of BU14: 11.39% AR (pH 5, benzoyl-label). This fraction was identical to AU14 (pH 5, butylphenyl-label). When taking the average of the butylphenyl- and benzoyl-label incubations at pH 5, also this fraction was < 10% AR at all time points.

Table 36 Distribution of radioactivity after hydrolysis of butylphenyl- and benzoyl-label cyflumetofen at pH 4 at 25 °C

Time, d	Butylphenyl-label, % of applied			Benzoyl-label, % of applied		
	Organic	Aqueous	Mass balance	Organic	Aqueous	Mass balance
0	97.66	nd	97.66	99.05	1.52	100.57
1	98.15	nd	98.15	92.04	6.13	98.17
3	97.36	nd	97.36	87.01	14.11	101.12
7	99.04	nd	99.04	73.46	25.11	98.56
14	99.55	nd	99.55	61.61	38.55	100.16
21	99.07	nd	99.07	54.25	43.99	98.24
30	98.24	nd	98.24	51.76	48.40	100.16

nd = Not detected

Table 37 Distribution of radioactivity after hydrolysis of butylphenyl- and benzoyl-label cyflumetofen at pH 5 at 25 °C

Time, d	Butylphenyl-label, % of applied			Benzoyl-label, % of applied		
	Organic	Aqueous	Mass balance	Organic	Aqueous	Mass balance
0	94.15	nd	94.15	100.76	1.39	102.15
1	97.39	nd	97.39	90.49	7.13	97.62
3	100.53	nd	100.83	82.13	17.57	99.70
7	99.82	0.76	100.59	68.79	31.48	100.27
14	98.53	1.07	99.60	57.35	43.54	100.89
21	96.29	1.16	97.46	49.52	49.61	99.14
30	96.32	1.22	97.54	47.70	51.86	99.56

Nd = Not detected

Table 38 Distribution of radioactivity after hydrolysis of butylphenyl- and benzoyl-label cyflumetofen at pH 7 at 25 °C

Time, d	Butylphenyl-label, % of applied			Benzoyl-label, % of applied		
	Organic	Aqueous	Mass balance	Organic	Aqueous	Mass balance
0	98.92	nd	98.92	98.06	1.85	99.91
0.08 (2 h)	99.26	nd	99.26	92.39	8.90	101.29

Time, d	Butylphenyl-label, % of applied			Benzoyl-label, % of applied		
	Organic	Aqueous	Mass balance	Organic	Aqueous	Mass balance
0.33 (8 h)	98.92	nd	98.92	74.96	25.34	100.29
1	98.22	0.90	99.12	55.66	44.80	100.46
2	101.78	0.79	102.57	47.80	52.43	100.22
5	101.00	0.87	101.87	47.18	53.11	100.29
10	100.49	nd	100.49	48.03	52.85	100.88
30	100.83	nd	100.83	–	–	–

nd = Not detected

Table 39 Distribution of radioactivity after hydrolysis of butylphenyl- and benzoyl-label cyflumetofen at pH 9 at 25 °C

Time, min	Butylphenyl-label, % of applied			Benzoyl-label, % of applied		
	Organic	Aqueous	Mass balance	Organic	Aqueous	Mass balance
0	101.26	nd	101.26	97.86	1.98	99.84
5	100.19	nd	100.19	79.79	20.79	100.58
15	100.56	nd	100.56	62.37	38.46	100.83
30	100.02	nd	100.02	55.61	45.79	101.40
45	101.25	nd	101.25	55.81	45.01	100.81
90	101.70	nd	101.70	52.57	48.05	100.62
1440	102.45	1.02	103.47	50.29	50.31	100.61

Nd = Not detected

Table 40 Identification of radioactivity after hydrolysis of butylphenyl-label or benzoyl-label cyflumetofen at pH 4 at 25 °C

Time, d	% of applied								
	Parent	A-1	A-2	A-18	AB-1	AU16	UNK 1	UNK 2	UNK 3
Butylphenyl-label									
0	93.79	Nd		Nd	1.97	nd	nd	nd	Nd
1	90.73	4.63		Nd	Nd	nd	nd	nd	Nd
3	71.89	12.79	Nd	Nd	11.90	nd	nd	nd	Nd
7	48.46	20.78	2.46	2.55	19.51	2.79	nd	nd	Nd
14	26.93	24.89	8.67	6.10	22.23	6.56	2.36	nd	Nd
21	14.87	26.94	8.60	10.28	30.92	4.90	nd	nd	Nd
30	6.26	21.03	14.55	12.63	34.80	5.53	nd	1.15	1.47

Nd = Not detected

Time, d	% of applied						
	Parent	B-1	AB-1	UNK 1 (BU16)	UNK 2	UNK 3	UNK 4
Benzoyl-label							
0	97.43	1.52	Nd	Nd	nd	nd	nd
1	88.62	6.13	2.56	Nd	nd	nd	nd
3	73.69	14.11	12.49	Nd	nd	nd	nd
7	48.95	25.90	13.62	4.78	2.50	1.87	nd
14	24.61	40.56	22.59	6.25	3.12	nd	2.30
21	15.18	44.80	29.80	7.76	nd	nd	nd
30	6.50	48.40	34.17	6.39	3.53	nd	nd

Nd = Not detected

Table 41 Identification of radioactivity after hydrolysis of butylphenyl-label or benzoyl-label cyflumetofen at pH 5 at 25 °C

Time, d	% of applied										
	Parent	A-1	A-2	A-18	AB-1	AU16	AU17	UNK 1 (AU14)	UNK 2	UNK 3	UNK 4
Butylphenyl-label											
0	94.15	nd		nd	nd	Nd	nd	nd	nd	nd	nd

Time, d	% of applied										
	Parent	A-1	A-2	A-18	AB-1	AU16	AU17	UNK 1 (AU14)	UNK 2	UNK 3	UNK 4
1	85.38	6.00		nd	3.73	nd	nd	nd	nd	nd	nd
3	71.78	9.89	1.35	nd	9.03	2.15	3.08	nd	nd	nd	nd
7	45.34	10.02	6.38	4.35	16.19	5.67	5.86	4.06	nd	nd	nd
14	18.91	5.73	7.62	8.04	23.67	11.97	13.60	4.16	nd	nd	4.83
21	8.71	0.89	14.12	8.63	18.72	15.19	17.62	3.71	1.31	1.92	5.46
30	5.15	0.51	12.41	7.37	19.93	15.78	21.09	3.82	nd	2.81	7.46

Nd = Not detected

AU14 = BU14 (pH5)

Time, d	% of applied								
	Parent	B-1	AB-1	BU17	UNK 1 (BU14)	UNK 2	UNK 3	UNK 4	UNK 5
Benzoyl-label									
0	99.99	1.39	nd	nd	nd	nd	nd	nd	nd
1	82.89	8.23	3.14	nd	3.36	nd	nd	nd	nd
3	68.76	17.57	8.60	1.64	3.14	nd	nd	nd	nd
7	43.02	31.48	8.89	3.09	7.05	2.64	nd	2.55	nd
14	19.14	43.54	20.61	4.87	11.39	nd	nd	nd	nd
21	8.70	49.61	15.83	7.50	3.23	2.93	2.36	5.98	3.00
30	2.85	52.62	23.35	9.55	5.35	2.51	nd	nd	3.33

Nd = Not detected

BU14 = AU14 (pH5)

Table 42 Identification of radioactivity after hydrolysis of butylphenyl-label or benzoyl-label cyflumetofen at pH 7 at 25 °C

Time, h	% of applied						
	Parent	A-1	A-2	A-18	AB-1	UNK 1 (AU16)	Others
Butylphenyl-label							
0	97.95	nd		nd	nd	nd	nd
2	82.29	7.48		nd	3.33	1.75	nd
8	53.31	14.44	3.62	6.53	12.91	4.01	2.48
24	19.02	9.85	15.70	17.94	29.58	3.95	nd
48	6.32	1.95	14.64	32.91	36.44	4.91	1.99
120	Nd	0.22	19.12	36.22	44.51	nd	nd
240	Nd	24.30		31.98	41.61	2.60	nd
720	Nd	44.12		10.85	36.96	5.03	nd

Nd = Not detected

Time, h	% of applied				
	Parent	B-1	AB-1	UNK 1 (BU16)	Others
Benzoyl-label					
0	98.06	1.85	nd	nd	Nd
2	86.97	8.90	2.38	nd	Nd
8	55.81	25.34	15.91	2.39	Nd
24	16.83	44.80	30.59	5.57	Nd
48	3.29	53.17	38.37	2.80	Nd
120	Nd	53.11	40.00	3.02	2.54
240	Nd	52.85	41.18	1.92	1.92

nd: not detected

Table 43 Identification of radioactivity after hydrolysis of butylphenyl-label or benzoyl-label cyflumetofen at pH 9 at 25 °C

Time, min	% of applied						
	Parent	A-1	A-2	A-18	AB-1	UNK 1 (AU16)	Others
Butylphenyl-label							
0	101.26	Nd		nd	nd	nd	nd
5	64.94	17.48	nd	2.01	13.29	nd	nd
15	26.87	28.27	3.66	7.22	26.02	4.13	3.15
30	16.46	24.77	6.16	11.74	35.99	2.73	nd
45	9.02	27.90	nd	20.98	40.27	3.08	nd
90	Nd	3.70	15.05	33.66	39.60	4.16	2.87
1440	Nd	nd	6.17	48.80	44.75	nd	nd

Nd = Not detected

Time, min	% of applied				
	Parent	B-1	AB-1	Others	UNK 1
Benzoyl-label					
0	97.86	1.98	nd	nd	nd
5	63.14	20.79	16.65	nd	nd
15	25.88	38.46	33.11	nd	nd
30	10.08	47.39	43.16	nd	nd
45	11.53	46.75	34.48	1.67	3.69
90	5.58	48.05	41.85	3.07	nd
1440	Nd	50.31	45.68	nd	nd

Nd = Not detected

Table 44 Kinetic parameters for hydrolysis of cyflumetofen in aqueous buffer solutions of pH 4, 5, 7 and 9 at 25 °C

Test	DT ₅₀	DT ₉₀	first order equation	r ²
pH4				
Butylphenyl-label	7.7 d	25.6 d	lnC = -0.09008t + ln(95.0235)	0.9991
Benzoyl-label	7.7 d	25.6 d	lnC = -0.08980t + ln(94.9601)	0.9977
Mean	7.7 d	25.6 d		
pH 5				
Butylphenyl-label	6.0 d	20.0 d	lnC = -0.11504t + ln(97.4473)	0.9988
Benzoyl-label	6.0 d	20.0 d	lnC = -0.11496t + ln(96.5132)	0.9994
Mean	6.0 d	20.0 d		
pH 7				
Butylphenyl-label	10.3 h	34.1 h	lnC = -0.06751 + ln(94.9138)	0.9984
Benzoyl-label	9.4 h	31.1 h	lnC = -0.07395t + ln(99.7485)	0.9997
Mean	9.8 h	32.6 h		
pH9				
Butylphenyl-label	11.5 min	38.0 min	lnC = -0.06053t + ln(88.0009)	0.9428
Benzoyl-label	9.1 min	30.4 min	lnC = -0.07583t + ln(91.9483)	0.9918
Mean	10.3 min	34.2 min		

Metabolism and Distribution in Succeeding Crops

Confined Rotational Crop Study (Grosshans et al., 2011a)

A confined rotational crop study was conducted with benzoyl- and butylphenyl-label cyflumetofen. The active substance was applied to bare sandy loam soil in plastic containers (40 cm × 60 cm) at an application rate of 1 × 400 g ai/ha using an automatic spray track system. After the soil aging periods, ploughing was simulated by mixing the treated and untreated soil layers using a spade before replanting. The nature and the quantities of the radioactive residues were investigated in lettuce (immature and mature), white radish (top and root) and spring wheat (forage, hay, straw, chaff and

grain) after plant back intervals (PBI) of 30, 120 and 365 days. They are cultivated under natural climatic conditions but protected from rain. Plant samples were harvested at maturity, and additional immature lettuce samples as well as spring wheat forage samples (in part dried to hay) were taken 26 to 39 days and 47 to 62 days after planting or sowing, respectively. Soil samples were taken after application, after ploughing and after harvest of the mature crops for each plant back interval.

Mature and immature lettuce leaves were sampled with the roots remaining in the soil. Ripe white radishes were pulled from the soil and separated into the root and the remaining green parts (top). Immature green plants of spring wheat were sampled (wheat forage) and partly dried to wheat hay. Furthermore, ears of mature wheat and straw were cut off with scissors. Afterwards, ears were separated into chaff and grain using a thresher. For the plant back intervals of 120 and 356 DAT (benzoyl-label), chaff and straw samples were combined. For the butylphenyl-label, chaff and straw samples were generally combined. Soil samples were taken after application into petri dishes, after the individual plant back intervals and after harvest of the mature crops.

All samples were stored in a freezer at -18 °C or below, immediately after they were taken and until they were transferred to the laboratory. The storage conditions remained constant until analysis started and during the whole period of the study. Lettuce, radish and wheat forage and hay samples were homogenized with dry ice using a planetary mill or a blender. Samples of wheat hay, wheat straw and wheat straw + chaff were also frozen in liquid nitrogen and generally homogenized using a meat grinder. Individual hay samples were frozen and coarsely cut prior to homogenization with the meat grinder or homogenized with the blender. Samples of wheat grain and chaff were frozen in liquid nitrogen or dry ice and homogenized using a centrifugal grinding mill or with the blender (grain 365 PBI).

The radioactive residues in homogenized plant or soil samples were determined by oxidative combustion of small aliquots using LSC. Weighed subsamples of homogenized plant material were extracted three times with a sufficient volume of acetonitrile using a Polytron blender and the radioactivity was determined (LSC). Subsamples of spring wheat hay, straw and chaff were soaked in water before extraction. The residues were further extracted with appropriate volumes of water (two times). The water extracts were also radioassayed. The results of the acetonitrile and the water extraction were summed and referred to as extractable radioactive residues (ERR). The residue after solvent extraction (acetonitrile and water) of each sample was dried at room temperature under a fume hood, homogenized and aliquots were combusted for the determination of the residual radioactive residue (RRR). The total radioactive residues (TRR) were the result of the combustion analyses or the calculated sum of ERR and RRR values.

The residual radioactive residues of spring wheat hay, straw, chaff and grain (plant back interval of 30 days in the case of the benzoyl-label, plant back intervals of 120 and 365 days in the case of the butylphenyl-label) were further characterized using sequential solubilisation procedures. In the case of the benzoyl-label, the first solubilisation step with aqueous ammonia solution was also applied for the other rotational crop matrices. The residues from spring wheat hay, straw, chaff and grain after ammonia solubilisation (both labels) were subjected to two consecutive solubilisation steps applying the following hydrolysing enzymes: macerozyme/cellulase and amylase/amyloglucosidase. In the case of the benzoyl-label (30 PBI), the incubations with enzymes were followed by two non-selective solubilisation steps with hydrochloric acid.

The nature of the radioactive residues in the acetonitrile and aqueous extracts (with a sufficient level of radioactivity) as well as solubilizates (from RRR matrices, only spring wheat grain 30 PBI, benzoyl-label) of individual matrices of the different crops and labels was investigated using different HPLC methods with radio-detection. Peak assignment and identification of degradates in extracts was done by comparison of the retention times and the elution profiles/degradate patterns of extracts with reference items in at least two different HPLC methods. Further information for the identification of degradates in the case of the benzoyl-label was obtained from GC-MS or LC-MS analyses. In addition, ¹⁴C-labelled reference items were combined with selected extracts for co-chromatography to identify the degradates unequivocally.

Following the spray application of cyflumetofen to soil at an application rate of 1×0.40 kg ai/ha, the total radioactive residues (TRR) were measured. They were significantly higher after application of the benzoyl-label compared to the butylphenyl-label for all rotational crop matrices, except for spring wheat grain for the plant back intervals of 120 and 365 days.

The residue concentrations in lettuce (immature and mature samples) did not exceed 0.057 mg eq/kg or 0.018 mg eq/kg for all plant back intervals for the benzoyl- and the butylphenyl-label, respectively.

The TRR in white radish root in the case of the benzoyl-label decreased from 0.027 to 0.008 mg eq/kg with increasing soil aging period, whereas in white radish top, the residue levels were slightly higher (decreasing from 0.137 to 0.036 mg eq/kg). In the case of the butylphenyl-label, all residue concentrations in white radish were below 0.010 mg eq/kg.

In spring wheat, the highest residue levels were measured in chaff (benzoyl-label, 30 PBI: 1.271 mg eq/kg), straw (benzoyl-label, 30 PBI: 0.484 mg eq/kg), straw + chaff (0.259 and 0.058 mg eq/kg for the benzoyl-label, 120 and 365 PBI; 0.027 to 0.107 mg eq/kg for the butylphenyl-label) and hay (decreasing from 0.636 to 0.053 mg eq/kg for the benzoyl-label and ranging from 0.016 to 0.086 mg eq/kg for the butylphenyl-label). The total radioactive residues in wheat forage decreased from 0.133 to 0.004 mg eq/kg in the case of the benzoyl-label and were around 0.010 mg eq/kg in the case of the butylphenyl-label. The total radioactive residues in wheat grain accounted for 0.021 to 0.166 mg eq/kg for the benzoyl-label and < 0.010 to 0.063 mg eq/kg for the butylphenyl-label.

The residue concentrations in the top soil layer after aging and ploughing stayed largely stable (plant back intervals 30, 120 and 365 PBI) for the benzoyl- (0.054 to 0.064 mg eq/kg) and butylphenyl-label (0.062 to 0.075 mg eq/kg). The residue levels in soil after harvest of the mature crops also remained more or less stable for both labels, ranging from 0.020 to 0.089 mg eq/kg (benzoyl-label) and from 0.033 to 0.090 mg eq/kg for the butylphenyl-label.

Table 45 Total radioactive residues in rotational crop matrices following cultivation in soil treated with benzoyl-label cyflumetofen after plant back intervals of 30, 120 and 365 days

Crop parts	Combustion value mg eq/kg	TRR calculated mg eq/kg ^a
Plant back interval: 30 DAT		
Immature lettuce (35 DAP ^b)	0.059	0.057
Mature lettuce (48 DAP)	0.056	0.057
White radish root (70 DAP)	0.032	0.027
White radish top (70 DAP)	0.150	0.137
Spring wheat forage (54 DAP)	0.148	0.133
Spring wheat hay (54 DAP)	0.704	0.636
Spring wheat straw (103 DAP)	0.450	0.484
Spring wheat chaff (103 DAP)	1.249	1.271
Spring wheat grain (103 DAP)	0.170	0.166
Plant back interval: 120 DAT		
Immature lettuce (36 DAP)	0.031	0.040
Mature lettuce (50 DAP)	0.022	0.021
White radish root (65 DAP)	0.012	0.025
White radish top (65 DAP)	0.070	0.066
Spring wheat forage (58 DAP)	0.058	0.058
Spring wheat hay (58 DAP)	0.307	0.316
Spring wheat straw + chaff (107 DAP)	0.243	0.259
Spring wheat grain (107 DAP)	0.048	0.045
Plant back interval: 365 DAT		
Immature lettuce (31 DAP)	0.041	0.038
Mature lettuce (51 DAP)	0.021	0.018
White radish root (71 DAP)	0.007	0.008
White radish top (71 DAP)	0.044	0.036

Crop parts	Combustion value mg eq/kg	TRR calculated mg eq/kg ^a
Spring wheat forage (62 DAP)	0.010	0.004
Spring wheat hay (62 DAP)	0.065	0.053
Spring wheat straw + chaff (114 DAP)	0.056	0.058
Spring wheat grain (114 DAP)	0.020	0.021

^a Sum of ERR (acetonitrile extract and water extract) and RRR (extraction residue)

^b Days after planting

Table 46 Total radioactive residues in rotational crop matrices following cultivation in soil treated with butylphenyl-label cyflumetofen after plant back intervals of 30, 120 and 365 days

Crop parts	Combustion value mg eq/kg	TRR calculated mg eq/kg ^a
Plant back interval: 30 DAT		
Immature lettuce (26 DAP ^b)	0.021	0.018
Mature lettuce (53 DAP)	0.006	n. d.
White radish root (74 DAP)	0.001	n. d.
White radish top (74 DAP)	0.005	n. d.
Spring wheat forage (60 DAP)	0.006	n. d.
Spring wheat hay (60 DAP)	0.022	0.016
Spring wheat straw + chaff (102 DAP)	0.028	0.027
Spring wheat grain (102 DAP)	0.008	n. d.
Plant back interval: 120 DAT		
Immature lettuce (27 DAP)	0.006	n. d.
Mature lettuce (48 DAP)	0.004	n. d.
White radish root (69 DAP)	0.004	n. d.
White radish top (69 DAP)	0.009	n. d.
Spring wheat forage (47 DAP)	0.015	0.013
Spring wheat hay (47 DAP)	0.081	0.086
Spring wheat straw + chaff (111 DAP)	0.107	0.107
Spring wheat grain (111 DAP)	0.058	0.063
Plant back interval: 365 DAT		
Immature lettuce (39 DAP)	0.004	n. d.
Mature lettuce (55 DAP)	0.003	n. d.
Mature lettuce small-sized (55 DAP)	0.003	n. d.
White radish root (75 DAP)	0.002	n. d.
White radish top (75 DAP)	0.005	n. d.
Spring wheat forage (50 DAP)	0.006	n. d.
Spring wheat hay (50 DAP)	0.020	0.017
Spring wheat straw + chaff (98 DAP)	0.030	0.027
Spring wheat grain (98 DAP)	0.030	0.031

^a Sum of ERR (acetonitrile extract and water extract) and RRR (extraction residue)

^b Days after planting

n. d. = not determined (due to the low residue concentrations, no extraction was performed)

Table 47 Total radioactive residues in soil samples following treatment with benzoyl-label cyflumetofen and plant back intervals of 30, 120 and 365 days

Soil samples (Days After Treatment DAT)	TRR mg eq/kg determined by direct combustion
After application (petri dishes)	
(0 DAT)	3.854
Plant back interval: 30 DAT	
After Ploughing	
(30 DAT)	0.065
After harvest of mature crops	
Lettuce (78 DAT)	0.042
White radish (100 DAT)	0.056
Spring wheat (133 DAT)	0.078
Plant back interval: 120 DAT	

Soil samples (Days After Treatment DAT)	TRR mg eq/kg determined by direct combustion
After Ploughing	
(120 DAT)	0.064
After harvest of mature crops	
Lettuce (170 DAT)	0.020
White radish (185 DAT)	0.042
Spring wheat (227 DAT)	0.089
Plant back interval: 365 DAT	
After Ploughing	
(365 DAT)	0.054
After harvest of mature crops	
Lettuce (416 DAT)	0.067
White radish (436 DAT)	0.063
Spring wheat (479 DAT)	0.052

Table 48 Total radioactive residues in soil samples following treatment with butylphenyl-label cyflumetofen and plant back intervals of 30, 120 and 365 days

Soil samples (Days After Treatment DAT)	TRR mg eq/kg determined by direct combustion
After application (petri dishes)	
(0 DAT)	4.580
Plant back interval: 30 DAT	
After Ploughing	
(30 DAT)	0.075
After harvest of mature crops	
Lettuce (83 DAT)	0.083
White radish (104 DAT)	0.045
Spring wheat (132 DAT)	0.085
Plant back interval: 120 DAT	
After Ploughing	
(120 DAT)	0.070
After harvest of mature crops	
Lettuce (168 DAT)	0.033
White radish (189 DAT)	0.071
Spring wheat (231 DAT)	0.090
Plant back interval: 365 DAT	
After Ploughing	
(365 DAT)	0.062
After harvest of mature crops	
Lettuce (420 DAT)	0.066
Lettuce small-sized (420 DAT)	0.069
White radish (440 DAT)	0.035
Spring wheat (463 DAT)	0.066

The extractability of the radioactive residues with acetonitrile and water in the case of the benzoyl-label was equal to or above 75% of the TRR for all rotational crop matrices, except for wheat forage (365 PBI: 66.1% TRR) and wheat grain (30 and 365 PBI: 45.9% to 66.7% TRR). In the case of the butylphenyl-label, the extractability of the radioactive residues was notably lower, accounting for 57.9% TRR for immature lettuce (30 PBI), 35.8% to 65.7% TRR for wheat forage, hay and straw + chaff and 18.8% to 20.4% TRR for wheat grain. For both labels, the main portions of the radioactive residues were generally extracted with acetonitrile, except for spring wheat grain where only low portions were extracted with acetonitrile.

Liquid/liquid partition of the acetonitrile extracts of immature lettuce, spring wheat hay and spring wheat straw + chaff between ethyl acetate and water (performed in the case of the butylphenyl-label only) yielded the main portions of the residues (22.6% to 46.1% TRR) in the organosoluble fraction and minor portions (11.7% to 20.1% TRR) in the water phase.

In the case of the benzoyl-label, the radioactive residues in the extracts of all rotational crop matrices mainly consisted of a medium polar component which was identified as trifluoroacetic acid (38.6% to 105.3% TRR, ranging from 0.004 to 1.076 mg eq/kg). In addition, in spring wheat grain 30 PBI, the main degradate trifluoroacetic acid was accompanied by minor amounts of the degradate B-1 (< 0.001 mg eq/kg or 0.2% TRR in the acetonitrile extract) and a peak representing one or several non-polar components, possibly including the unchanged parent molecule (present in both extracts, 0.006 mg eq/kg or 4.1% TRR in sum). The degradates, trifluoroacetic acid and B-1, are only detectable with the benzoyl-label.

In the case of the butylphenyl-label, a series of label-specific degradates was observed in the samples prepared from the extracts, all in minor concentrations (each below 0.010 mg eq/kg, only one non-polar derivative in immature lettuce (30 PBI, 11.6% TRR) and one medium polar component in wheat hay (120 PBI, 10.2% TRR) were above 10% of the TRR. The main portion of the extracted and analysed radioactive residues in all matrices except wheat grain was composed of medium polar derivatives of cyflumetofen (up to 39.4% TRR in sum per matrix, up to twenty peaks per sample). These medium polar degradates eluted in the same range of polarity as the degradates in the goat urine sample, used as a reference item. Therefore, some of the medium polar degradates formed during metabolism of cyflumetofen in the lactating goat after hydrolytic cleavage of the parent molecule (e.g. hydroxylated derivatives of degradate A-2) or similar components may also be present in rotational crops. Besides the medium polar components, up to seven polar peaks were detected in the extractable radioactive residues (representing up to 14.9% TRR in sum per matrix), one non-polar derivative was found in immature lettuce (30 PBI, 11.6% TRR), and eleven non-polar peaks were observed in spring wheat straw and chaff (30 PBI, 6.8% TRR in sum). In the case of wheat grain (120 PBI), the radioactive residues in the water extract mainly consisted of polar components (4.8% TRR in sum), accompanied by minor amounts of medium polar components (0.5% TRR in sum). Since the absolute concentration of these minor degradates was < 0.01 mg eq/kg, no further characterization was deemed necessary.

The residual radioactive residues after solvent extraction of the matrices sampled after the plant back interval of 30 days ranged from a minimum of 11.0% TRR (white radish top) to a maximum of 54.1% TRR (spring wheat grain) for the benzoyl-label. Therefore, the residual radioactive residues from these matrices (30 PBI) were further characterized by treatment with an aqueous ammonia solution which solubilized portions of 1.5% to 2.4% TRR (except for wheat grain, where 15.1% TRR were solubilized). In the cases of spring wheat hay, straw, chaff and grain, the solubilisation with ammonia was followed by incubation with enzymes (macerozyme/cellulase and amylase/amyloglucosidase) and by homogenization or reflux with hydrochloric acid. The most effective solubilisation steps for most of the wheat matrices were the non-selective reflux with hydrochloric acid which released up to 7.3% TRR (spring wheat hay 30 PBI), followed by incubation with macerozyme/cellulase. In the case of wheat grain (30 PBI), treatment with macerozyme/cellulase solubilized 17.4% TRR, ammonia treatment released 15.1% TRR and incubation with amylases/amyloglucosidase was also quite effective and released 7.7% TRR. HPLC analysis of the ammonia solubilizate of wheat grain 30 DAT showed a degradate pattern similar to that of the water extract.

In the case of the butylphenyl-label, notable amounts of the radioactive residues of wheat hay, wheat straw + chaff and wheat grain (120 PBI and 365 PBI, 39.1% to 81.2% TRR) were not extracted with acetonitrile and water. The residual radioactive residues after solvent extraction of these matrices were therefore further characterized using a sequential solubilisation procedure including treatment with aqueous ammonia solution, macerozyme/cellulase and amylase/amyloglucosidase. The most effective solubilisation step was incubation with macerozyme/cellulase (releasing up to 20.4% TRR, wheat grain 365 PBI), except for wheat straw + chaff (365 PBI), where ammonia treatment was slightly more effective (solubilizing 6.5% TRR).

The solubilized residues had possibly been associated with or embedded/incorporated in insoluble plant material (e.g. proteins, cell wall polymers and starch).

Table 49 Extraction of radioactive residues in rotational crop matrices following their cultivation in soil treated with benzoyl-label cyflumetofen after plant back intervals of 30, 120 and 365 days

Crop parts	Distribution of radioactive residues								
	TRR ^a	Acetonitrile extract		Water extract		ERR ^a		RRR ^a	
	mg eq/kg	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Plant back interval: 30 DAT									
Immature lettuce	0.057	0.048	83.6	0.002	2.7	0.049	86.3	0.008	13.7
Mature lettuce	0.057	0.046	79.4	0.002	4.3	0.048	83.7	0.009	16.3
White radish root	0.027	0.016	58.5	0.005	19.0	0.021	77.5	0.006	22.5
White radish root	0.036	0.022	61.6	0.005	14.3	0.027	75.9	0.009	24.1
White radish top	0.137	0.115	83.5	0.008	5.5	0.122	89.0	0.015	11.0
Spring wheat forage	0.133	0.099	74.5	0.007	5.3	0.106	79.8	0.027	20.2
Spring wheat hay	0.636	0.421	66.2	0.057	8.9	0.478	75.0	0.159	25.0
Spring wheat straw	0.484	0.362	74.8	0.029	6.1	0.391	80.9	0.093	19.1
Spring wheat chaff	1.271	1.027	80.8	0.054	4.2	1.081	85.0	0.190	15.0
Spring wheat grain	0.166	0.007	4.5	0.069	41.4	0.076	45.9	0.090	54.1
Spring wheat grain	0.156	0.009	5.5	0.072	46.2	0.080	51.7	0.075	48.3
Plant back interval: 120 DAT									
Immature lettuce	0.040	0.039	97.8	< 0.001	0.9	0.039	98.8	< 0.001	1.2
Mature lettuce	0.021	0.020	96.8	< 0.001	1.3	0.021	98.1	< 0.001	1.9
White radish root	0.025	0.024	94.2	0.001	2.4	0.024	96.6	0.001	3.4
White radish top	0.066	0.063	96.3	0.001	2.2	0.065	98.6	0.001	1.4
Spring wheat forage	0.058	0.054	93.7	0.002	4.0	0.057	97.7	0.001	2.3
Spring wheat hay	0.316	0.283	89.4	0.023	7.2	0.306	96.6	0.011	3.4
Spring wheat straw + chaff	0.259	0.243	93.9	0.010	3.9	0.253	97.7	0.006	2.3
Spring wheat grain	0.045	0.006	13.0	0.032	71.6	0.038	84.7	0.007	15.3
Plant back interval: 365 DAT									
Immature lettuce	0.038	0.036	95.2	0.001	2.2	0.037	97.4	0.001	2.6
Mature lettuce	0.018	0.017	93.4	< 0.001	2.6	0.017	96.0	0.001	4.0
White radish root	0.008	0.007	93.1	< 0.001	3.2	0.008	96.3	< 0.001	3.7
White radish top	0.036	0.034	94.4	0.001	2.8	0.035	97.2	0.001	2.8
Spring wheat forage	0.004	0.002	53.5	0.001	12.5	0.003	66.1	0.002	33.9
Spring wheat hay	0.053	0.038	70.8	0.005	9.7	0.043	80.5	0.010	19.5
Spring wheat straw + chaff	0.058	0.046	80.4	0.003	5.9	0.050	86.3	0.008	13.7
Spring wheat grain	0.021	0.003	12.0	0.012	54.7	0.014	66.7	0.007	33.3

^a ERR = Extractable Radioactive Residue (acetonitrile extract and water extract), RRR = Residual (non-extractable) Radioactive Residue, TRR = sum of ERR and RRR

Table 50 Extraction of radioactive residues in rotational crop matrices following their cultivation in soil treated with butylphenyl-label cyflumetofen after plant back intervals of 30, 120 and 365 days

Crop parts	Distribution of radioactive residues								
	TRR ^a	Acetonitrile extract		Water extract		ERR ^a		RRR ^a	
	mg eq/kg	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Plant back interval: 30 DAT									
Immature lettuce	0.0179	0.0087	48.3	0.0017	9.6	0.0104	57.9	0.0076	42.1
Spring wheat hay	0.0159	0.0070	44.1	0.0011	7.0	0.0081	51.1	0.0078	48.9
Spring wheat straw + chaff	0.0270	0.0158	58.8	0.0019	7.0	0.0177	65.7	0.0092	34.3
Plant back interval: 120 DAT									
Spring wheat forage	0.0133	0.0041	30.6	0.0020	15.2	0.0061	45.8	0.0072	54.2

Crop parts	Distribution of radioactive residues								
	TRR ^a	Acetonitrile extract		Water extract		ERR ^a		RRR ^a	
	mg eq/kg	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Spring wheat hay	0.0860	0.0415	48.2	0.0053	6.1	0.0468	54.4	0.0393	45.6
Spring wheat straw + chaff	0.1067	0.0524	49.1	0.0126	11.8	0.0650	60.9	0.0417	39.1
Spring wheat grain	0.0630	0.0003	0.5	0.0115	18.3	0.0119	18.8	0.0511	81.2
Plant back interval: 365 DAT									
Spring wheat hay	0.0172	0.0050	28.8	0.0012	7.1	0.0062	35.8	0.0110	64.2
Spring wheat straw + chaff	0.0272	0.0093	34.1	0.0023	8.5	0.0116	42.6	0.0156	57.4
Spring wheat grain	0.0311	0.0009	2.9	0.0054	17.5	0.0063	20.4	0.0248	79.6

^a ERR = Extractable Radioactive Residue (acetonitrile extract and water extract), RRR = Residual (non-extractable) Radioactive Residue after solvent extraction, TRR = sum of ERR and RRR

Table 51 Identified components in rotational crop matrices following their cultivation in soil treated with benzoyl-label cyflumetofen after a plant back interval of 30 days

Component	Crop parts									
	Immature lettuce		Mature lettuce		White radish root		White radish top			
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Plant back interval: 30 DAT										
Trifluoroacetic acid	0.050	87.8	0.043	75.2	0.014	38.6	0.106	77.4		
Trifluoroacetic acid corrected ^a	0.075 mg/kg		0.065 mg/kg		0.021 mg/kg		0.159 mg/kg			
Sum of identified components	0.050	87.8	0.043	75.2	0.014	38.6	0.106	77.4		
Component	Crop parts									
	Spring wheat forage		Spring wheat hay		Spring wheat straw		Spring wheat chaff		Spring wheat grain ^b	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Plant back interval: 30 DAT										
Trifluoroacetic acid	0.100	75.2	0.427	67.1	0.332	68.7	1.076	84.6	0.066	42.1
Trifluoroacetic acid Corrected ^a	0.150 mg/kg		0.641 mg/kg		0.498 mg/kg		1.614 mg/kg		0.099 mg/kg	
B-1	n. d	n. d.	n. d.	n. d.	n. d.	n. d.	n. d.	n. d.	< 0.001	0.2
Sum of identified components	0.100	75.2	0.427	67.1	0.332	68.7	1.076	84.6	0.066	42.2

^a Corrected mg/kg values referring to the calculation of the radioactivity and molecular mass of the degradate Trifluoroacetic Acid

^b In the case of spring wheat grain, the sum of identified components in the extractable and residual radioactive residues (ERR + RRR) is given; the degradate Trifluoroacetic Acid was identified in the extracts (0.057 mg/kg or 36.3% TRR in sum) and in the ammonia solubilizate (0.010 mg eq/kg or 5.7% TRR)
n. d. = not detected

Table 52 Identified components in rotational crop matrices following their cultivation in soil treated with benzoyl-label cyflumetofen after a plant back interval of 120 days

Component	Crop parts							
	Immature lettuce		Mature lettuce		White radish root		White radish top	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Plant back interval: 120 DAT								
Trifluoroacetic acid	0.042	105.3	0.015	72.0	0.013	53.1	0.060	91.0
Trifluoroacetic acid Corrected ^a	0.063 mg/kg		0.023 mg/kg		0.020 mg/kg		0.090 mg/kg	
Sum of identified components	0.042	105.3	0.015	72.0	0.013	53.1	0.060	91.0

Component	Crop parts							
	Immature lettuce		Mature lettuce		White radish root		White radish top	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Component	Crop parts							
	Spring wheat forage		Spring wheat hay		Spring wheat straw + chaff		Spring wheat grain	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Plant back interval: 120 DAT								
Trifluoroacetic Acid	0.056	96.9	0.252	79.5	0.246	94.9	0.030	66.6
Trifluoroacetic Acid Corrected ^a	0.084 mg/kg		0.377 mg/kg		0.369 mg/kg		0.045 mg/kg	
Sum of identified components	0.056	96.9	0.252	79.5	0.246	94.9	0.030	66.6

^a Corrected mg/kg values referring to the calculation of the radioactivity and molecular mass of the degradate Trifluoroacetic Acid

Table 53 Identified components in rotational crop matrices following their cultivation in soil treated with benzoyl-label cyflumetofen after a plant back interval of 365 days

Component	Crop parts							
	Immature lettuce		Mature lettuce		White radish root		White radish top	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Plant back interval: 365 DAT								
Trifluoroacetic Acid	0.038	100.5	0.014	78.3	0.004	44.8	0.037	103.4
Trifluoroacetic Acid Corrected ^a	0.058 mg/kg		0.021 mg/kg		0.005 mg/kg		0.056 mg/kg	
Sum of identified components	0.038	100.5	0.014	78.3	0.004	44.8	0.037	103.4
Component	Crop parts							
	Spring wheat forage		Spring wheat hay		Spring wheat straw + chaff		Spring wheat grain	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Plant back interval: 365 DAT								
Trifluoroacetic Acid	n. d.	n. d.	0.027	51.1	0.027	46.6	0.011	52.6
Trifluoroacetic Acid Corrected ^a	n. d.		0.041 mg/kg		0.040 mg/kg		0.017 mg/kg	
Sum of identified components	n. d.	n. d.	0.027	51.1	0.027	46.6	0.011	52.6

^a corrected mg/kg values referring to the calculation of the radioactivity and molecular mass of the degradate Trifluoroacetic acid
n. d. = not detected

Table 54 Characterized radioactive residues (ERR ^a+RRR ^a) from rotational crop matrices following their cultivation in soil treated with butylphenyl-label cyflumetofen after a plant back interval of 30, 120 and 365 days

Characterized Radioactive Residues	Crop parts									
	Immature lettuce		Spring wheat forage		Spring wheat hay		Spring wheat straw + chaff		Spring wheat grain	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Plant back interval: 30 DAT										
Characterized from ERR by HPLC analysis	0.0079	44.3	no extraction		0.0087	54.3	0.0142	52.6	no extraction	
Number of HPLC peaks	up to 7 peaks				up to 19 peaks		up to 31 peaks			
Maximum HPLC peak	0.0021	11.6 ^b			0.0011	7.2	0.0018	6.7		

Characterized Radioactive Residues	Crop parts									
	Immature lettuce		Spring wheat forage		Spring wheat hay		Spring wheat straw + chaff		Spring wheat grain	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Characterized from ERR by Extraction / Work-up	0.0021	11.9			0.0014	9.0	0.0034	12.8		
Characterized from RRR by Solubilization	not applied				not applied		not applied			
Total Characterized	0.0101	56.2			0.0101	63.3	0.0176	65.4		
Plant back interval: 120 DAT										
Characterized from ERR by HPLC analysis	no extraction		not applied		0.0266	30.9	0.0443	41.5	0.0033	5.3
Number of HPLC peaks					2 x 2 peaks		up to 19 peaks		7 peaks	
Maximum HPLC peak					0.0088	10.2 ^c	0.0095	8.9	0.0013	2.1
Characterized from ERR by Extraction / Work-up			0.0061	45.8	0.0085	9.9	0.0044	4.2	0.0058	9.2
Characterized from RRR by Solubilization			not applied		0.0138	16.0	0.0107	10.0	0.0261	41.5
Total Characterized			0.0061	45.8	0.0489	56.8 ^d	0.0594	55.6 ^e	0.0353	56.0
Plant back interval: 365 DAT										
Characterized from ERR by HPLC analysis	no extraction		no extraction		not applied		not applied		not applied	
Characterized from ERR by Extraction / Work-up					0.0062	35.8	0.0116	42.6	0.0063	20.4
Characterized from RRR by Solubilization					0.0033	19.1	0.0040	14.8	0.0130	41.7
Total Characterized					0.0095	55.0	0.0156	57.4	0.0193	62.1

^a ERR = Extractable Radioactive Residues (acetonitrile extract and water extract),

RRR = Residual Radioactive Residues (after solvent extraction)

^b The second most abundant HPLC peak represented 0.0018 mg eq/kg or 9.8% TRR;

^c The second and third most abundant HPLC peaks both represented 0.0062 mg eq/kg or 7.2% TRR

^d Calculated losses of 0.0117 mg eq/kg or 13.6% TRR in sum occurred during work-up of the acetonitrile extract

^e Calculated losses of 0.0151 mg eq/kg or 14.2% TRR in sum occurred during work-up of the acetonitrile extract

Table 55 Characterization of residual radioactive residues (RRR) after solvent extraction of rotational crop matrices following their cultivation in soil treated with benzoyl-label cyflumetofen after a plant back interval of 30 days

Fraction / Supernatant	Crop matrix									
	Immature lettuce		Mature lettuce		White radish root		White radish top			
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR		
Plant back interval: 30 DAT										
RRR	0.008	13.7	0.009	16.3	0.006	22.5	0.015	11.0		
Ammonia solubilizate	0.001	1.7	0.001	1.5	0.001	2.4	0.002	1.5		
Sum of solubilized radioactive residues	0.001	1.7	0.001	1.5	0.001	2.4	0.002	1.5		
Final residue	0.006	10.5	0.004	6.5	0.005	20.0	0.012	9.0		
Procedural recovery	89.1%		49.1%		99.6%		95.9%			
Fraction / Supernatant	Crop matrix									
	Spring wheat forage		Spring wheat hay		Spring wheat straw		Spring wheat chaff		Spring wheat grain	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Plant back interval: 30 DAT										
RRR	0.027	20.2	0.159	25.0	0.093	19.1	0.190	15.0	0.090	54.1
Ammonia solubilizate	0.002	1.8	0.014	2.1	0.008	1.8	0.020	1.6	0.025	15.1
Ammonia residue	0.024	17.9	0.148	23.2	0.104	21.5	0.170	13.4	0.064	38.6

Fraction / Supernatant	Crop matrix									
	Immature lettuce		Mature lettuce			White radish root			White radish top	
	mg eq/kg	% TRR	mg eq/kg	% TRR		mg eq/kg	% TRR		mg eq/kg	% TRR
Macerozyme/cellulase solubilizate	n. a.	n. a.	0.024	3.8	0.012	2.4	0.023	1.8	0.029	17.4
Amylase/amyloglucosidase solubilizate	n. a.	n. a.	0.011	1.7	0.003	0.7	0.013	1.0	0.013	7.7
Amylase/amyloglucosidase residue	n. a.	n. a.	0.105	16.5	0.082	16.9	0.128	10.1	0.022	13.2
1 N HCl homogenization	n. a.	n. a.	0.003	0.4	0.001	0.2	0.002	0.2	0.002	1.2
6 N HCl reflux	n. a.	n. a.	0.047	7.3	0.026	5.3	0.045	3.5	0.012	7.0
Sum of solubilized radioactive residues	0.002	1.8	0.098	15.4	0.050	10.4	0.103	8.1	0.080	48.4
Final residue	0.024	17.9	0.036	5.7	0.040	8.3	0.063	4.9	0.006	3.9
Procedural recovery ^a	97.3%		84.5%			97.9%			87.0%	

^a Recovery calculated as (Sum of solubilized radioactive residues + Final residue) mg eq/kg $\times$ 100 / RRR mg eq/kg
n. a. = not applied

Table 56 Characterization of residual radioactive residues (RRR) after solvent extraction of rotational crop matrices following their cultivation in soil treated with butylphenyl-label cyflumetofen after plant back intervals of 120 and 365 days

Fraction / Supernatant	Crop matrix					
	Spring wheat hay		Spring wheat straw + chaff		Spring wheat grain	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Plant back interval: 120 DAT						
RRR	0.0393	45.6	0.0417	39.1	0.0511	81.2
Ammonia solubilizate	0.0025	2.9	0.0033	3.0	0.0058	9.2
Macerozyme/cellulase solubilizate	0.0076	8.8	0.0045	4.2	0.0122	19.4
Amylase/amyloglucosidase solubilizate	0.0037	4.3	0.0029	2.7	0.0081	12.9
Sum of solubilized radioactive residues	0.0138	16.0	0.0107	10.0	0.0261	41.5
Final residue	0.0203	23.6	0.0279	26.2	0.0216	34.2
Procedural recovery ^a	86.7%		92.6%		93.3%	
Plant back interval: 365 DAT						
RRR	0.0110	64.2	0.0156	57.4	0.0248	79.6
Ammonia solubilizate	0.0009	5.1	0.0018	6.5	0.0022	6.9
Macerozyme/cellulase solubilizate	0.0015	8.5	0.0013	4.7	0.0063	20.4
Amylase/amyloglucosidase solubilizate	0.0009	5.5	0.0010	3.6	0.0045	14.4
Sum of solubilized radioactive residues	0.0033	19.1	0.0040	14.8	0.0130	41.7
Final residue	0.0073	42.4	0.0118	43.2	0.0119	38.4
Procedural recovery	95.9%		101.2%		100.6%	

^a Recovery calculated as (Sum of solubilized radioactive residues + Final residue) mg eq/kg $\times$ 100 / RRR mg eq/kg

With regard to the degradate patterns described above for the two labels, the degradation pathway of cyflumetofen in rotational crops can be proposed as follows: Hydrolytic cleavage of the parent molecule results in the formation of the degradate B-1, which is only detectable with the benzoyl-label, and one or several butylphenyl-label-specific intermediates which were not identified. The predominant degradate in the case of the benzoyl-label, Trifluoroacetic Acid, can be derived from the parent compound or the degradate B-1. The butylphenyl-label-specific cleavage products (such as the goat degradate A-2 which is formed by hydrolysis of the formic acid ester, decarboxylation and

hydrolytic detachment of the trifluoromethyl benzoyl moiety) are further transformed to a series of minor (< 0.01 mg eq/kg), mainly medium polar degradates.

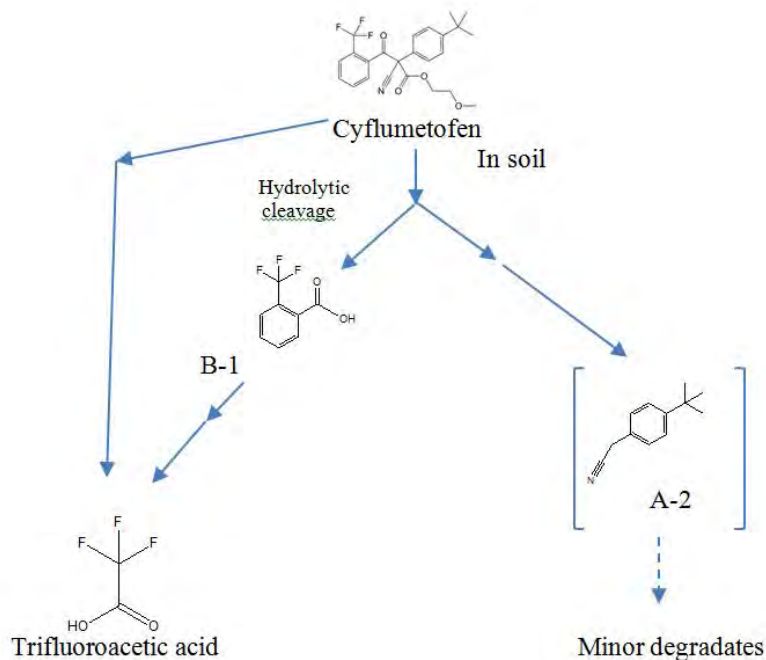


Figure 3 Proposed metabolic pathway in rotational crops (lettuce, radish and wheat)

RESIDUE ANALYSIS

Analytical methods

The Meeting received descriptions and validation data (including concurrent recovery data) for analytical methods for residues of cyflumetofen and its metabolites.

Analytical methods for plant matrices

The descriptions of the analytical methods used in supervised residue trials and for enforcement are shown below.

Method D1003

Analyte: Cyflumetofen and metabolites B-1, AB-6 and AB-7

Matrix: Dry bean, dried grapes (raisins), orange (fruits, juice and oil), rice (grain, straw), soybean seed, tomato

LOQ: 0.01 mg/kg for each analyte in all matrices (defined as the lowest fortification level successfully tested)

Description: Crop materials Residues are extracted from plant matrices by shaking with acetonitrile followed by acetonitrile:water (75:25, v/v). An aliquot of the extract is centrifuged and, after removal of acetonitrile, acidified with formic acid. The residues are partitioned with a mixture of ethyl acetate: cyclohexane (75:25, v/v). An aliquot of the organic phase is concentrated in the presence of 0.1% aqueous formic acid, and re-dissolved in 0.1% formic acid in acetonitrile for determination with LC-MS/MS. Oil Residues are extracted from oil by shaking with a mixture of acetonitrile:water (75:25, v/v) and hexane. The hexane phase is removed and an aliquot of the aqueous acetonitrile phase is subjected to clean-up by partition with hexane or ethyl acetate:cyclohexane (75:25, v/v) followed by final determination as the above.

Juice Residues are extracted from juice by shaking with a mixture of acetonitrile:water (75:25, v/v). An aliquot of the aqueous acetonitrile extract is subject to clean-up and for final determination as the above.

LC-MS/MS condition

For data collection: Less concentrated final sample was used for analysis with highly sensitive LC-MS/MS instrument. From the acetonitrile extract of crop materials, acetonitrile is removed by concentration rather than centrifugation.

For enforcement: Larger amount of aliquot from the extract for sample clean-up to increase the residue concentration.

Reference: Carter *et al.*, 2011a

Validation of D1003 for data collection

Validation of Method D1003 for collecting data on the concentrations of the parent, B-1, AB-6 and AB-7 was conducted using the sample size of 0.1 g for crop materials, and 5 g for oil and orange juice.

Table 57 Recovery results obtained for the analysis of plant matrices and some of their processed foods with Method D1003 for data collection

Matrix	Analyte	No.	Fortification Level (mg/kg)	Primary transition ^a			Secondary transition 2 ^b		
				Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)
Orange	Cyflumetofen	5	0.01	91–106	97	6	91–96	93	3
		5	1.0	96–104	100	3	83–108	97	10
		10	Overall	91–106	99	5	83–108	95	7
	B-1	5	0.01	74–85	80	7	81–87	84	3
		5	1.0	71–75	73	2	72–78	75	3
		10	Overall	71–85	77	7	72–87	80	6
	AB-6	5	0.01	92–96	95	2	86–98	94	5
		5	1.0	92–106	99	6	85–100	94	7
		10	Overall	92–106	97	5	85–100	94	6
	AB-7	5	0.01	92–99	95	3	87–103	95	7
		5	1.0	88–99	93	4	86–99	95	5
		10	Overall	88–99	94	4	86–103	95	6
Tomato	Cyflumetofen	5	0.01	78–124	94	20	84–133	100	20
		5	1.0	80–92	84	6	77–92	84	7
		10	Overall	78–124	89	16	77–133	92	17
	B-1	5	0.01	71–84	77	8	73–81	76	5
		5	1.0	75–97	82	11	70–93	81	12
		10	Overall	71–97	79	10	70–93	78	9
	AB-6	5	0.01	80–107	92	11	87–108	94	9
		5	1.0	79–90	84	5	82–88	86	3
		10	Overall	79–107	88	9	82–108	90	8
	AB-7	5	0.01	74–98	85	12	77–103	89	11
		5	1.0	71–83	78	7	83–92	87	4
		10	Overall	71–98	82	11	77–103	88	8

Matrix	Analyte	No.	Fortification Level (mg/kg)	Primary transition ^a			Secondary transition 2 ^b		
				Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)
Bean, dry	Cyflumetofen	5	0.01	100–114	108	5	104–110	107	2
		5	1.0	98–119	110	8	92–117	106	9
		10	Overall	98–119	109	7	92–117	106	6
	B-1	5	0.01	70–76	72	3	79–94	86	8
		5	1.0	71–95	80	12	74–84	81	5
		10	Overall	70–95	76	11	74–94	83	7
	AB-6	5	0.01	100–116	110	6	103–119	111	7
		5	1.0	87–102	95	7	80–110	95	12
		10	Overall	87–116	103	10	80–119	103	12
	AB-7	5	0.01	99–118	108	7	99–119	111	7
		5	1.0	92–106	99	7	89–105	99	7
		10	Overall	92–118	103	8	89–119	105	9
Soybean	Cyflumetofen	5	0.01	80–93	86	6	90–109	97	8
		5	1.0	101–114	108	5	96–119	108	8
		10	Overall	80–114	97	13	90–119	102	10
(seed)	B-1	5	0.01	68–73	71	3	70–76	73	4
		5	1.0	88–104	99	7	87–102	96	6
		10	Overall	68–104	85	19	70–102	85	15
	AB-6	5	0.01	106–120	113	6	90–123	107	15
		5	1.0	98–114	109	6	92–108	102	7
		10	Overall	98–120	110	6	90–123	104	11
	AB-7	5	0.01	93–118	108	11	102–120	109	7
		5	1.0	99–108	103	3	92–113	105	9
		10	Overall	93–118	106	8	92–120	107	8
Rice	Cyflumetofen	5	0.01	73–98	88	12	74–102	96	13
		5	1.0	104–116	111	4	100–115	106	6
		10	Overall	73–116	100	14	74–115	101	11
(grain)	B-1	5	0.01	70–111	91	16	79–118	107	15
		5	1.0	93–111	98	8	92–109	99	6
		10	Overall	70–111	95	12	79–118	103	12
	AB-6	5	0.01	74–125	107	19	71–124	102	19
		4	1.0	105–107	106	0.8	96–121	111	8
		9	Overall	74–125	107	13	71–124	107	14
	AB-7	5	0.01	70–113	101	18	71–115	104	18
		5	1.0	99–115	106	6	102–112	107	4
		10	Overall	70–115	103	13	71–115	106	12
Orange juice	Cyflumetofen	5	0.01	78–89	85	5	78–98	88	10
		5	1.0	77–93	87	7	81–95	91	6
		10	Overall	77–93	86	6	78–98	89	8
	B-1	5	0.01	72–97	83	11	90–108	97	7
		5	1.0	68–81	74	6	71–87	78	8
		10	Overall	68–97	79	11	71–108	88	14
	AB-6	5	0.01	79–88	83	4	75–83	78	5
		5	1.0	83–92	87	5	81–99	87	9
		10	Overall	79–92	85	5	75–99	83	9
	AB-7	5	0.01	84–92	88	4	78–93	86	8
		5	1.0	78–90	83	6	81–90	85	4
		10	Overall	78–92	86	6	78–93	86	6
Orange oil	Cyflumetofen	5	0.01	70–75	73	3	71–81	78	5
		5	1.0	66–80	74	8	70–85	77	10
		10	Overall	66–80	74	6	70–85	77	7
	B-1	5	0.01	74–104	91	13	100–117	108	6
		5	1.0	71–117	99	18	85–112	99	12
		10	Overall	71–117	95	16	85–117	103	10
	AB-6	5	0.01	93–100	97	3	86–107	98	8
		5	1.0	77–98	88	9	80–101	93	9
		10	Overall	77–100	92	8	80–107	95	8
	AB-7	5	0.01	70–89	81	9	79–107	88	14
		5	1.0	75–93	81	9	70–89	79	10
		10	Overall	70–93	81	8	70–107	84	13

Matrix	Analyte	No.	Fortification Level (mg/kg)	Primary transition ^a			Secondary transition 2 ^b		
				Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)
Dried grape	Cyflumetofen	5	0.01	102–123	109	8	94–117	104	9
		5	1.0	96–127	114	10	92–131	113	15
		10	Overall	96–127	111	9	92–131	109	12
	B-1	5	0.01	75–83	78	5	71–81	75	5
		5	1.0	72–83	78	6	71–89	80	9
		10	Overall	72–83	78	5	71–89	78	8
	AB-6	5	0.01	101–119	110	7	102–114	109	5
		5	1.0	101–131	116	9	100–134	115	12
		10	Overall	101–131	113	8	100–134	112	9
	AB-7	5	0.01	100–114	109	6	106–114	111	3
		5	1.0	96–136	112	14	94–141	114	16
		10	Overall	96–136	110	10	96–141	113	11
Rice straw	Cyflumetofen	5	0.01	97–111	104	5	97–118	104	8
		5	1.0	104–118	111	5	97–118	107	7
		10	Overall	97–118	107	6	97–118	105	7
	B-1	5	0.01	71–91	79	10	68–73	71	3
		5	1.0	71–84	77	7	70–76	73	4
		10	Overall	71–94	78	8	68–76	72	4
	AB-6	5	0.01	97–114	104	6	100–109	105	3
		5	1.0	90–101	97	5	95–107	98	5
		10	Overall	90–114	101	6	95–109	102	5
	AB-7	5	0.01	98–109	101	4	100–107	102	3
		5	1.0	85–99	93	7	86–99	92	6
		10	Overall	85–109	97	7	86–107	97	7

^a Primary transition used for quantification:

Cyflumetofen: m/z 448.2 → 173.0 (ionization mode, positive)

B-1: m/z 189.1 → 69.0 (ionization mode, negative)

AB-6: m/z 466.4 → 173.0 (ionization mode, positive)

AB-7: m/z 448.2 → 344.2 (ionization mode, positive)

^b Secondary transition used for confirmation:

Cyflumetofen: m/z 448.2 → 145.1

B-1: m/z 189.1 → 145.1

AB-6: m/z 466.4 → 145.0

AB-7: m/z 448.2 → 372.2

The method showed good linearity in the range of 0.035–10.0 µg/kg. No known interference was observed from crop components or from reagents, solvents and glassware used, except in rice straw which may show matrix interference for the B-1 secondary ion. As the matrix interference residue was greater than 20% of the LOQ, alternative chromatography was developed for the secondary ion for validation of the method for this analyte.

The mean recoveries for all matrices tested and at all fortification levels ranged between 71 and 116% with relative standard deviations between 2 and 20%. However, some individual recovery values were higher than 120%.

Validation of Method D1003 for enforcement

Validation of Method D1003, as an enforcement method, was conducted using the sample size of 5 g for all matrices tested. In this validation, the parent compound and B-1 metabolite were determined.

Table 58 Recovery results obtained for the analysis of plant matrices and some of their processed foods with Method D1003 for enforcement

Matrix	Analyte	Fortification Level (mg/kg)	No.	Primary transition ^a			Secondary transition 2 ^b		
				Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)
Tomato	Cyflumetofen	0.01	5	87–103	94	7	84–104	95	10
		1.0	5	90–100	93	5	84–111	93	11
		Overall	10	87–103	93	6	84–111	94	10

Matrix	Analyte	Fortification Level (mg/kg)	No.	Primary transition ^a			Secondary transition 2 ^b		
				Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)
	B-1	0.01	5	78–90	84	5	71–97	78	14
		1.0	5	76–102	87	13	68–92	81	12
		Overall	10	76–102	86	9	68–97	80	12
Soybean	Cyflumetofen	0.01	5	82–103	92	9	74–108	94	13
		1.0	5	89–111	98	8	88–107	97	7
		Overall	10	82–111	95	9	74–108	96	10
(seed)	B-1	0.01	5	76–93	83	9	77–106	93	15
		1.0	5	80–102	91	10	80–109	88	14
		Overall	10	76–102	87	11	77–109	90	14
Rice	Cyflumetofen	0.01	5	77–109	88	15	79–100	87	10
		1.0	5	89–106	98	7	96–106	101	4
		Overall	10	77–109	93	12	79–106	94	11
(grain)	B-1	0.01	6	84–100	89	7	78–119	98	19
		1.0	5	87–107	95	8	76–105	86	15
		Overall	10	84–107	92	8	76–119	92	18
Bean, dry	Cyflumetofen	0.01	6	83–110	95	10	90–111	96	9
		1.0	5	97–108	103	5	92–106	101	6
		Overall	10	83–110	98	8	90–111	99	8
	B-1	0.01	6	85–106	96	8	89–125	104	12
		1.0	5	90–99	93	5	85–119	105	13
		Overall	10	85–106	95	6	85–125	104	12
Orange	Cyflumetofen	0.01	5	79–90	84	5	73–84	78	5
		1.0	5	80–88	84	4	79–95	86	7
		Overall	10	79–90	84	4	73–95	82	8
	B-1	0.01	5	73–105	87	14	90–115	100	10
		1.0	5	74–85	80	6	91–121	101	12
		Overall	10	73–105	84	12	90–121	101	10
Orange juice	Cyflumetofen	0.01	5	85–111	96	11	77–93	86	7
		1.0	5	70–101	80	15	71–92	76	12
		Overall	10	70–111	88	15	71–93	81	11
	B-1	0.01	5	70–96	77	14	77–101	89	10
		1.0	5	72–90	81	9	74–100	89	12
		Overall	10	70–96	79	11	74–101	89	10
Orange oil	Cyflumetofen	0.01	5	66–72	70	4	68–76	72	5
		1.0	5	82–96	87	7	86–101	90	7
		Overall	10	66–96	79	12	68–101	81	13
	B-1	0.01	5	84–105	96	9	81–107	97	11
		1.0	5	80–103	90	9	60–99	84	20
		Overall	10	80–105	93	9	60–107	90	17
Rice straw	Cyflumetofen	0.01	6	77–96	88	7	83–90	87	3
		1.0	5	94–104	99	4	92–106	101	6
		Overall	10	77–104	93	9	83–106	93	9
	B-1	0.01	6	79–116	90	16	79–118	92	16
		1.0	5	78–103	91	12	86–100	94	7
		Overall	10	78–116	90	14	79–118	93	12

^a Primary transition used for quantification:

Cyflumetofen: m/z 448.2 → 173.0 (ionization mode, positive)

B-1: m/z 189.1 → 69.0 (ionization mode, negative)

^b Secondary transition used for confirmation:

Cyflumetofen: m/z 448.2 → 145.1

B-1: m/z 189.1 → 145.1

The method showed good linearity in the range of 0.14–15.0 µg/kg. No known interference was observed from crop components or from reagents, solvents and glassware used, except in rice straw as described above.

The mean recoveries for all matrices tested and at all fortification levels ranged between 70 and 105%, within the acceptable range, with relative standard deviations between 3 and 19%. However, some individual analysis recoveries were slightly higher than 120% and some were lower than 70%.

Independent laboratory validation of Method D1003 (Marin, 2011a)

An independent laboratory validation of Method D1003 was conducted for the determination of cyflumetofen and metabolite B-1 in orange, wheat (grain and hay) and canola seed. Fortification levels used are 0.01 (LOQ) and 1.0 mg/kg.

Some modifications were made to the procedure of Method D1003 as follows:

Analyte: Cyflumetofen and metabolite B-1

LOQ: 0.01 mg/kg for each analyte in all matrices

Description: Residues are extracted from plant matrices by shaking with acetonitrile followed by acetonitrile:water (50:50, v/v) and centrifugation. An aliquot (2 mL) of the extract is transferred to a glass vessel containing aqueous formic acid. The extract is concentrated under a gentle flow of nitrogen and then partitioned with ethyl acetate: cyclohexane (75:25, v/v). After mixing, 2 g of sodium chloride is added and the mixture is partitioned for 5 minutes. After centrifugation, an aliquot (7 mL) of the extract is transferred to a glass tube containing 0.2 mL of aqueous formic acid. The extract is concentrated under a gentle flow of nitrogen and the volume is adjusted to 2.5 mL with acetonitrile containing formic acid. Residues are determined with LC-MS/MS.

Sample size used in this validation was 5 g.

Table 59 Recovery results obtained for the analysis of plant matrices with Method D1003 in independent laboratory validation

Matrix	Analyte	Fortification Level (mg/kg)	No.	Primary transition ^a			Secondary transition 2 ^b		
				Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)
Orange	Cyflumetofen	0.01	5	67–80	73	7	65–94	74	16
		1.0	5	84–92	89	3	91–103	96	5
		Overall	10	67–92	81	12	65–103	85	16
	B-1	0.01	5	77–87	82	6	76–84	79	4
		1.0	5	78–83	80	3	75–84	80	5
		Overall	10	77–87	81	5	75–84	80	4
Tomato	Cyflumetofen	0.01	5	61–85	74	12	60–90	72	17
		1.0	5	74–77	75	1	75–85	78	5
		Overall	10	61–85	75	8	60–90	75	12
	B-1	0.01	5	60–86	76	13	58–86	71	14
		1.0	5	77–81	79	3	75–79	78	3
		Overall	10	60–86	78	9	58–86	75	11
Wheat grain	Cyflumetofen	0.01	5	76–108	88	14	78–104	89	12
		1.0	5	84–95	90	4	85–106	92	10
		Overall	10	76–108	89	10	78–106	90	10
	B-1	0.01	5	81–86	83	2	78–85	80	4
		1.0	5	74–81	77	4	72–83	78	5
		Overall	10	74–86	80	5	72–85	79	4
Canola seed	Cyflumetofen	0.01	5	71–86	81	7	64–87	75	15
		1.0	5	88–102	94	6	92–111	99	8
		Overall	10	71–102	87	10	64–111	87	18
	B-1	0.01	5	73–75	74	1	69–74	72	3
		1.0	5	85–92	87	3	83–89	85	2
		Overall	10	73–92	81	9	69–89	79	9
Wheat hay	Cyflumetofen	0.01 ^c	5	84–91	88	5	80–98	87	8
		1.0 ^c	5	90–96	94	2	86–94	90	3
		1.0	5	93–101	97	4	78–91	83	6
		Overall	10	84–101	93	5	78–98	87	7
	B-1	0.01 ^c	5	85–109	95	12	45–59	51	12
		1.0 ^c	5	64–68	66	3	64–71	67	4
		1.0	5	75–81	78	3	75–81	77	3
		Overall	10	64–109	80	18	45–81	65	17

^a Primary transition used for quantification:

Cyflumetofen: m/z 448.2 → 173.0 (ionization mode, positive)

B-1: m/z 189.1 $\rightarrow$ 69.0 (ionization mode, negative)

^b Secondary transition used for confirmation:

Cyflumetofen: m/z 448.2 $\rightarrow$ 145.0

B-1: m/z 189.1 $\rightarrow$ 145.0

^c Analysed by modified method.

The method showed good linearity in the range of 0.35–10.0 $\mu\text{g/kg}$. No known interference was observed from crop components or from reagents, solvents and glassware used, except in wheat hay.

The mean recoveries for all matrices tested and at all fortification levels ranged between 71 and 97%, within the acceptable range, with relative standard deviations between 1 and 19% with the exceptions of B-1 in wheat hay in which even the mean recoveries at 0.01 and 1 mg/kg were lower than 70%. Some individual analysis recoveries for matrices tested were lower than 70%.

Analytical methods for animal matrices

The descriptions of the analytical methods related to the current review are shown below.

All of these methods showed good linearity with a correlation coefficient ≥ 0.99 . There were no interferences at the analyte concentration higher than 30% of LOQ. Additional information on method performance is described later.

Method D1202

Analyte: Cyflumetofen and Metabolite B-1

Matrix: Bovine liver, meat and milk; and poultry eggs

LOQ: 0.001 mg/kg for each analyte in bovine milk; and 0.01 mg/kg for each analyte in other matrices.

Description: Bovine Liver and Poultry Eggs: Residues are extracted 5 g of sample by shaking with acetonitrile followed by acetonitrile-water (50:50, v/v). A salt mixture of magnesium sulfate (400 mg), sodium chloride (100 mg), sodium citrate sesquihydrate (50 mg), and sodium citrate dihydrate (100 mg) is added to an aliquot of the sample vortexed and centrifuged. The process is repeated with another salt mixture of magnesium sulfate (75 mg) and CUPSA (12.5 mg). An aliquot of the extract is concentrated to remove the acetonitrile to the aqueous phase in the presence of 20% formic acid in water. The extract is re-acidified and saturated with sodium chloride and partitioned with a mixture of ethyl acetate-cyclohexane (75:25, v/v). An aliquot of the organic phase is concentrated in the presence of 0.1% formic acid in water, and re-dissolved in 0.1% formic acid in acetonitrile for LC-MS/MS determination.

Bovine Meat: Residues are extracted from 5 g of sample by shaking with acetonitrile followed by acetonitrile-water (50:50, v/v). An aliquot of the extract is concentrated to remove the acetonitrile to the aqueous phase in the presence of 0.1% formic acid in water. The extract is saturated with sodium chloride and partitioned with a mixture of ethyl acetate-cyclohexane (75:25, v/v). An aliquot of the organic phase is concentrated in the presence of 0.1% formic acid in water, and re-dissolved in 0.1% formic acid in acetonitrile for LC-MS/MS determination.

Bovine Milk: Residues are extracted from 5 g or mL of sample by shaking with acetonitrile. An aliquot of the extract is concentrated to remove the acetonitrile to the aqueous phase in the presence of 20% formic acid in water. The extract is saturated with sodium chloride and partitioned with a mixture of ethyl acetate-cyclohexane (75:25, v/v). Aliquots of the organic phase are concentrated in the presence of 0.1% formic acid in water, and re-dissolved in 0.1% formic acid in acetonitrile. A salt mixture of magnesium sulfate (400 mg), sodium chloride (100 mg), sodium citrate sesquihydrate (50 mg), and sodium citrate dihydrate (100 mg) is added and vortexed and centrifuged. Another salt mixture of magnesium sulfate (75 mg) and CUPSA

(12.5 mg) mixture is then added and vortexed and centrifuged. An aliquot is taken for LC-MS/MS determination.

Reference: Sweeney and Nejad, 1998a

An independent laboratory validation of Method D1202 was conducted for the determination of cyflumetofen and metabolite B-1 in bovine liver, meat and milk and poultry eggs at fortification levels of the LOQ and $100 \times \text{LOQ}$: i.e., 0.001 and 0.10 mg/kg for bovine milk; and 0.01 and 1.0 mg/kg for other matrices.

Table 60 Recovery results obtained during independent laboratory validation of Method D1202 for bovine liver, meat and milk and poultry eggs.

Matrix	Analyte	Fortification level (mg/kg)	No.	Primary transition ^a			Secondary transition ^b		
				Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)
Bovine Liver	Parent	0.01 ^c	5	62–108	91	19	64–100	89	16
		1.0 ^c	5	108–124	115	5	107–123	115	5
		Overall	10	62–124	103	17	64–123	102	17
	B-1	0.01	5	74–81	78	4	71–81	78	5
		1.0	5	81–91	85	5	81–90	84	4
		Overall	10	74–91	81	6	71–90	81	6
Bovine Meat	Parent	0.01	5	69–82	77	7	71–84	77	7
		1.0	5	84–99	90	8	85–102	92	9
		Overall	10	69–99	84	11	71–102	85	12
	B-1	0.01	5	76–88	83	6	78–93	86	6
		1.0	5	70–85	77	8	71–86	77	8
		Overall	10	70–88	80	8	71–93	82	9
Bovine Milk	Parent	0.001	5	76–84	79	4	77–83	80	3
		0.10	5	103–112	107	3	104–112	107	3
		Overall	10	76–112	93	16	77–112	93	16
	B-1	0.001 ^c	5	74–90	83	8	74–86	81	6
		0.10 ^c	5	93–101	97	3	92–99	97	3
		Overall	10	74–101	90	10	74–99	89	10
Poultry Eggs	Parent	0.01 ^c	5	64–68	65	3	62–71	66	6
		1.0 ^c	5	60–69	67	6	59–69	66	6
		Overall	10	60–69	66	4	59–71	66	6
	B-1	0.01	5	77–89	86	6	84–103	95	8
		1.0	5	88–95	91	3	88–97	90	4
		Overall	10	77–95	88	5	84–103	93	6

^a Primary transition used for quantification:

Cyflumetofen: m/z 448.2 → 173.2 (ionization mode, positive)

B-1: m/z 189.0 → 69.1 (ionization mode, negative)

^b Secondary transition used for confirmation:

Cyflumetofen: m/z 448.2 → 145.1

B-1: m/z 189.1 → 145.1

^c Results obtained using matrix matched standards.

The method showed good linearity in the range of 0.20–10.0 µg/kg. No known interference was observed from animal components or from reagents, solvents and glassware used.

The mean recoveries for bovine matrices tested and at all fortification levels ranged between 77 and 115% with RSDs between 3 and 19%. The mean recoveries of the parent in poultry eggs ranged between 65–67% with the standard deviations between 3–6% while those for B-1 ranged between 86–95% with the standard deviations between 3–8%.

Stability of pesticide residues in stored analytical samples

The Meeting received information on freezer storage stability of cyflumetofen and three of its metabolites, B-1, AB-6 and AB-7 in various plant matrices, such as orange (fruit, juice and oil), apple (fruit and juice), leaf lettuce, kidney bean (dry), radish root, wheat grain and almond nutmeat (Gorden, 2011a and 2013a).

Homogenized samples (5 g) of each matrix were fortified separately with standard solutions containing Cyflumetofen and metabolites B-1, or AB-6 or AB-7 at a level of 0.1 mg/kg for each analyte and stored at –20 to –10 °C for up to 743–910 days (24–30 months).

After each specified period, a portion of sample was analysed with Method D1003 with the LOQ at 0.01 mg/kg. At the same time, a sample was freshly spiked at 0.1 mg/kg and analysed for concurrent procedural recovery.

Table 61 summarizes the storage periods, mean percent remaining and mean concurrent recovery at each time point.

Table 61 Stability of cyflumetofen and its metabolites B-1, AB-6 and AB-7 in frozen plant matrices fortified at 0.1 mg/kg

Matrix	Storage period (month)	Cyflumetofen		B-1		AB-6		AB-7	
		% remaining	Procedural recovery	% remaining	Procedural recovery	% remaining	Procedural recovery	% remaining	Procedural recovery
Almond	0	85	—	68	82	81	88	103	95
	0.2	86	85	—	—	—	—	—	—
	1	89	94	—	—	—	—	97	95
	2	—	—	—	—	93	88	—	—
	3	106	100	64	82	—	—	73	86
	5	—	—	—	—	85	88	—	—
	6	84	90	70	76	—	—	94	98
	9, 8	87	94	—	—	99	103	92	102
	10	—	—	67	83	—	—	—	—
	11	77	80	—	—	—	—	—	—
	12	—	—	—	—	106	126	91	82
	13	—	—	78	85	—	—	—	—
	18	85	91	—	—	—	—	—	—
	19	—	—	—	—	—	—	98	93
	21	—	—	—	—	77	89	—	—
	23	—	—	78	94	—	—	—	—
	25	79	107	—	—	—	—	—	—
	26	—	—	—	—	—	—	99	118
	28	—	—	—	—	82	115	—	—
	30	—	—	74	101	—	—	—	—
Apple Fruit	0	83	—	77	81	80	89	88	86
	0.2	67	83	—	—	—	—	—	—
	1	76	85	—	—	—	—	79	86
	2	—	—	—	—	81	89	—	—
	3	76	100	76	81	—	—	60	78
	5	—	—	—	—	79	85	—	—
	6	74	77	78	80	—	—	—	—
	7	—	—	—	—	—	—	77	82
	9	106	93	—	—	81	69	—	—
	10	—	—	66	105	—	—	87	80
	11	77	85	—	—	—	—	—	—
	12	—	—	—	—	102	109	71	92
	13	—	—	69	95	—	—	—	—
	18	70	75	—	—	—	—	—	—
	19	—	—	—	—	—	—	67	77
	21	—	—	—	—	59	75	—	—
	22	—	—	66	79	—	—	—	—
	25	79	77	—	—	—	—	—	—
	26	—	—	—	—	—	—	80	82
	28	—	—	—	—	69	88	—	—
	29	—	—	58	77	—	—	—	—
Apple Juice	0	92	—	83	87	94	96	90	96
	0.2	92	92	—	—	—	—	—	—
	1	83	101	—	—	—	—	90	96
	2	—	—	—	—	94	96	—	—
	3	102	99	69	87	—	—	79	101

Cyflumetofen

Matrix	Storage period (month)	Cyflumetofen		B-1		AB-6		AB-7	
		% remaining	Procedural recovery	% remaining	Procedural recovery	% remaining	Procedural recovery	% remaining	Procedural recovery
	5	—	—	—	—	104	93	—	—
	6	105	91	79	84	—	—	—	—
	7	—	—	—	—	—	—	96	94
	9	106	98	—	—	90	89	103	91
	10	—	—	74	81	—	—	—	—
	11	104	90	—	—	—	—	—	—
	12	—	—	—	—	106	102	94	92
	13	—	—	83	94	—	—	—	—
	18	79	79	—	—	—	—	—	—
	19	—	—	—	—	—	—	92	91
	21	—	—	—	—	80	91	—	—
	22	—	—	93	94	—	—	—	—
	25	85	74	—	—	—	—	—	—
	26	—	—	—	—	—	—	87	78
	29	—	—	—	—	65	83	—	—
	30	—	—	51	78	—	—	—	—
Kidney bean	0	99	—	70	78	90	94	101	98
	0.2	90	99	—	—	—	—	—	—
	1	98	97	—	—	—	—	99	98
	2	—	—	—	—	—	—	—	—
	3	101	109	68	78	100	94	87	106
	5	—	—	—	—	91	90	—	—
	6	77	89	79	82	—	—	106	96
	9	72	99	—	—	98	90	101	102
	10	—	—	74	77	—	—	—	—
	11	63	92	—	—	—	—	—	—
	12	—	—	—	—	120	115	96	98
	13	—	—	66	76	—	—	—	—
	18	55	84	—	—	—	—	—	—
	19	—	—	—	—	—	—	99	92
	21	—	—	—	—	83	93	—	—
	23	—	—	85	93	—	—	—	—
	25	37	93	—	—	—	—	—	—
	26	—	—	—	—	—	—	91	99
	28	—	—	—	—	91	110	—	—
	30	—	—	77	106	—	—	—	—
Lettuce	0	93	—	60	65	84	78	80	86
	0.2	80	93	—	—	—	—	—	—
	1	80	89	—	—	—	—	74	86
	2	—	—	—	—	83	78	—	—
	3	82	99	58	65	—	—	—	—
	4	—	—	—	—	—	—	57	81
	5	—	—	—	—	80	88	—	—
	6	53	93	71	84	—	—	—	—
	7	—	—	—	—	—	—	65	90
	9	70	103	—	—	80	78	—	—
	10	—	—	58	103	—	—	82	102
	11	56	85	—	—	—	—	—	—
	12	—	—	—	—	84	109	67	92
	13	—	—	77	81	—	—	—	—
	18	46	86	—	—	—	—	—	—
	19	—	—	—	—	—	—	59	83
	21	—	—	—	—	68	84	—	—
	22	—	—	41	83	—	—	—	—
	26	—	—	—	—	—	—	50	91
	28	46	86	—	—	59	91	—	—
	29	—	—	88	91	—	—	—	—
Orange Fruit	0	86	—	66	75	83	83	92	81
	0.2	69	86	—	—	—	—	—	—

Matrix	Storage period (month)	Cyflumetofen		B-1		AB-6		AB-7	
		% remaining	Procedural recovery	% remaining	Procedural recovery	% remaining	Procedural recovery	% remaining	Procedural recovery
	1	75	93	—	—	—	—	83	81
	2	—	—	—	—	77	83	—	—
	3	86	86	59	75	—	—	65	82
	5	—	—	—	—	97	79	—	—
	6	82	83	81	69	—	—	—	—
	7	—	—	—	—	—	—	77	82
	9	106	93	—	—	81	68	—	—
	10	—	—	60	98	—	—	85	89
	11	80	85	—	—	—	—	—	—
	12	—	—	—	—	104	103	81	89
	13	—	—	77	83	—	—	—	—
	18	83	80	—	—	—	—	—	—
	19	—	—	—	—	—	—	103	81
	21	—	—	—	—	70	80	—	—
	22	—	—	66	88	—	—	—	—
	24	76	77	—	—	—	—	—	—
	26	—	—	—	—	—	—	56	81
	28	—	—	—	—	54	77	—	—
	29	—	—	76	85	—	—	—	—
Orange Juice	0	93	—	82	90	87	99	93	97
	0.2	82	93	—	—	—	—	—	—
	1	92	87	—	—	—	—	92	97
	2	—	—	—	—	91	99	—	—
	3	106	97	79	90	—	—	82	100
	5	—	—	—	—	104	107	—	—
	6	98	97	80	85	—	—	—	—
	7	—	—	—	—	—	—	96	98
	9	104	83	—	—	93	100	97	107
	10	—	—	75	81	—	—	—	—
	11	102	90	—	—	—	—	—	—
	12	—	—	—	—	88	101	89	97
	13	—	—	89	82	—	—	—	—
	18	78	86	—	—	—	—	—	—
	19	—	—	—	—	—	—	91	98
	21	—	—	—	—	80	97	—	—
	22	—	—	83	100	—	—	—	—
	25	93	93	—	—	—	—	—	—
	26	—	—	—	—	—	—	74	91
	29	—	—	—	—	63	90	—	—
	30	—	—	103	90	—	—	—	—
Orange Oil	0	81	—	84	88	88	87	97	92
	0.2	75	81	—	—	—	—	—	—
	1	62	84	—	—	—	—	97	92
	2	—	—	—	—	79	87	—	—
	3	92	84	68	88	—	—	—	—
	4	—	—	—	—	—	—	76	91
	5	—	—	—	—	111	103	—	—
	6	99	72	65	92	—	—	93	91
	9	81	72	—	—	103	104	104	92
	10	—	—	56	100	—	—	—	—
	11	82	72	—	—	98	86	—	—
	12	—	—	41	85	—	—	93	93
	18	79	80	—	—	—	—	—	—
	19	—	—	—	—	—	—	84	83
	21	—	—	—	—	85	96	—	—
	22	—	—	46	103	—	—	—	—
	25	97	83	—	—	—	—	—	—
	26	—	—	—	—	—	—	95	69
	29	—	—	—	—	99	87	—	—

Matrix	Storage period (month)	Cyflumetofen		B-1		AB-6		AB-7	
		% remaining	Procedural recovery	% remaining	Procedural recovery	% remaining	Procedural recovery	% remaining	Procedural recovery
	30	–	–	56	115	–	–	–	–
Radish	0	102	–	68	80	88	82	83	81
Root	0.2	82	102	–	–	–	–	–	–
	1	83	95	–	–	–	–	69	81
	2	–	–	–	–	72	82	–	–
	3	86	101	67	80	–	–	–	–
	4	–	–	–	–	–	–	50	79
	5	–	–	–	–	84	88	–	–
	6	63	82	79	79	–	–	–	–
	7	–	–	–	–	–	–	60	79
	9	59	87	–	–	84	69	–	–
	10	–	–	67	89	–	–	62	99
	11	65	88	–	–	–	–	–	–
	12	–	–	–	–	82	102	43	94
	13	–	–	76	81	–	–	–	–
	18	55	75	–	–	–	–	–	–
	19	–	–	–	–	–	–	44	76
	21	–	–	–	–	77	78	–	–
	22	–	–	60	79	–	–	–	–
	24	60	84	–	–	–	–	–	–
	26	–	–	–	–	–	–	47	88
	28	–	–	–	–	74	94	–	–
	29	–	–	50	70	–	–	–	–
Wheat	0	102	–	86	86	102	97	94	90
Grain	0.2	102	102	–	–	–	–	–	–
	1	94	93	–	–	–	–	87	90
	2	–	–	–	–	95	97	–	–
	3	104	100	75	86	–	–	–	–
	4	–	–	–	–	–	–	72	88
	5	–	–	–	–	–	–	–	–
	6	81	85	75	88	–	–	86	96
	9	124	106	–	–	96	94	77	103
	10	–	–	68	79	–	–	–	–
	11	89	92	–	–	–	–	–	–
	12	–	–	–	–	115	120	80	103
	13	–	–	86	86	–	–	–	–
	18	80	88	–	–	–	–	–	–
	19	–	–	–	–	–	–	76	99
	21	–	–	–	–	93	94	–	–
	23	–	–	82	90	–	–	–	–
	25	72	96	–	–	–	–	–	–
	26	–	–	–	–	–	–	56	96
	29	–	–	–	–	80	103	–	–
	30	–	–	67	86	–	–	–	–

The results of the storage stability showed high uncertainty associated with the analytical method used.

Cyflumetofen was stable (> 70% remaining) when stored frozen at the temperature of –20– –10 °C for the longest time tested (24 or 25 months) in almond, apple fruit, apple juice, orange fruit, orange juice, orange oil and wheat grain. However, it showed stability only up to 9 months in lettuce and 3 months in radish root.

Metabolite B-1 was stable frozen up to: 30 months, the longest time tested, in almond, kidney bean, lettuce, orange fruit, orange juice and wheat grain, 22 months in apple fruit and apple juice, and 6 months in orange oil.

Metabolite AB-7 was stable frozen up to: the longest period tested (26 months) in almond, apple fruit, apple juice, kidney bean, orange juice, and orange oil, 19 months in orange fruit and wheat grain, 12 months in lettuce, and 1 month in radish root.

Cyflumetofen is used either alone or in combination with other active substances and provides knock-down and residual control of tetranychid mites shown in the following table. It is highly active contact miticides on egg, nymph and adult stage of these mites.

<i>Bryobia rubrioculus</i>	<i>Panonychus ulmi</i>
<i>Eotetranychus willamettei</i>	<i>Petrobia lateens</i>
<i>Eotetranychus yumensis</i>	<i>Tetranychus cinnabarinus</i>
<i>Eutetranychus banksi</i>	<i>Tetranychus mcdanieli</i>
<i>Oligonychus pratensis</i>	<i>Tetranychus pacificus</i>
<i>Oligonychus ununguis</i>	<i>Tetranychus turkestani</i>
<i>Panonychus citri</i>	<i>Tetranychus urticae</i>

For the current review, a US label was provided which indicates that thorough and uniform spray coverage of foliage, with direct contact to target pest, is required for effective control and therefore sufficient volume of water is necessary to ensure thorough coverage of foliage.

Commodity	Country	Formulation Type and g/kg or g/L	F or, G	Application				Application rate per treatment		PHI, days	
				Method	Max rate per season, kg ai/ha	Min interval, days	No. of applications	Timing	Rate, kg ai/ha		Min water vol., L/ha
Citrus fruits											
Citrus fruits	USA	SC 200	F	Foliar spray	0.40	14	2	At first sign of infestation	0.19–0.20	935	7
Citrus fruits	Brazil	SC 200	F	Foliar spray	–	–	1		0.08 kg ai/hL	–	7
Pome fruits											
Pome fruits	USA	SC 200	F	Foliar spray	0.40	14	2	At first sign of infestation	0.19–0.20	935	7
Berries and other small fruits											
Grapes	USA	SC 200	F	Foliar spray	0.40	14	2	At first sign of infestation	0.19–0.20	468	14
Strawberry	USA	SC 200	F	Foliar spray	0.40	14	2	At first sign of infestation	0.19–0.20	468	1
Fruiting vegetables, other than Cucurbits											
Tomato	USA	SC 200	F	Foliar spray	0.40	14	2	At first sign of infestation	0.19–0.20	468	3
Eggplant	Japan	SC 200	F G	Foliar spray	–	–	2	At first sign of infestation	0.02 kg ai/hL	(1000–3500)	1
Tree nuts											

Commodity	Country	Formulation Type and g/kg or g/L	F or, G ^a	Application					Application rate per treatment		PHI, days ^b
				Method	Max rate per season, kg ai/ha	Min interval, days	No. ^c	Timing	Rate, kg ai/ha	Min water vol., L/ha	
Tree nuts	USA	SC 200	F	Foliar spray	0.40	14	2	At first sign of infestation	0.19–0.20	935	7

^a F= outdoor or field use; G= glasshouse

^b PHI= Pre-harvest interval

^c Do not make more than 1 application before using an effective miticide with a different mode of action

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received residue data from supervised field trials conducted on citrus fruits (orange in Brazil and the USA, grapefruit and lemon in the USA), pome fruits (apple in Italy and the USA and pear in the USA); grapes (in the USA); strawberry (in the USA); fruiting vegetables, other than Cucurbits (tomato in the USA and eggplant in Japan; and tree nuts (in the USA).

Application rates and residue concentrations were reported as Cyflumetofen. Residue concentrations are recorded unadjusted for recoveries or for residue values in control samples. Where multiple samples were taken from a single plot, individual results are reported, and the calculated mean concentration is put in parentheses and used for estimation of maximum residue level. Where trials were conducted in the same location, with the same or similar varieties, same or similar formulations, and same equipment, and at the same or similar timing, they are not regarded as independent and only one result from these trials was chosen for the estimation of a maximum residue level. All the trials, except those on eggplant in Japan, were conducted outdoor using 20% SC formulations except one trial conducted in Italy on apple (25% SC). Residue concentrations are expressed in parent equivalents.

Residues from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels and they are underlined.

The results of supervised trials are summarized in the following tables:

Crop group	Commodities	Table no
Citrus fruits	Orange, grapefruit, lemon	Table 64
Pome fruits	Apple, pear	Table 65
Berries and other small fruits	Grapes	Table 66
	Strawberry	Table 67
Fructing vegetables, other than Cucurbits	Tomato	Table 68
	Eggplant	Table 69
Tree nuts	Almond, pecan	Table 70
By-products, used for animal feeding purposes, derived from fruits and vegetable processing	Almond hulls	Table 71

Citrus fruits (orange, grapefruit and lemon)

During the growing season of 2009 and 2010, 23 supervised residue trials were conducted on citrus fruits (orange, grapefruit and lemon) in Arizona (one trial), California (eight trials), Florida (12 trials) and Texas (two trials) in the USA. Three (two in CA and one in FL) of these trials were decline trials. At each test location, one untreated and one treated plot were established. The treated plot received two broadcast foliar applications of cyflumetofen (20% SC formulation) each at 0.193–0.207 kg ai/ha, with a 14-day (+/- one day) re-treatment interval (RTI), except one trial that had a 12-day RTI. The applications were made with ground equipment using 280–2818 L/ha of water. Commercially mature fruits of orange, grapefruit or lemon were collected 7 days after treatment (DAT). In the three decline trials, citrus samples were collected 0, 1, 3, 7, 14 and 21 DAT.

All samples were analysed for residues of cyflumetofen and its metabolites B-1, AB-6 and AB-7 using Method D1003 (with modifications), which quantifies residues by LC-MS/MS. Mean recoveries for all analytes were within the acceptable range of 70–120%. A recovery of 66% was obtained for B-1 on lemon fortified at the 0.01 mg/kg level. Several recovery values exceeded 120%. The LOD and LOQ for all analytes were 0.002 and 0.01 mg/kg, respectively. For B-1, when its concentration is expressed in parent equivalents, the LOQ will be 0.02 mg/kg.

In Brazil, during the growing season of 2007, four trials were conducted on oranges to determine the magnitude of the residues of cyflumetofen after treatment with 20% SC formulation of cyflumetofen. The formulation was applied two times at an application rate of 0.160 kg ai/ha and a spray volume of 2000 L/ha. In two of the four trials, the samples were taken immediately after the last application, 1, 7, 14 and 21. In the other two trials the samples were collected 7 days after the last application.

The residues of cyflumetofen were determined by LC-MS/MS. The LOQ was 0.05 mg/kg. The mean recoveries at 0.05 mg/kg spiked level were within the acceptable range of 70–120%.

Table 64 Results of supervised residue trials on citrus fruits (orange, grapefruit and lemon) conducted in Brazil and the USA.

CROP Country, Year Location variety Trial No.	Application				Portion analysed	DAT	Residues (mg eq/kg) ^a				Author Report Year Study No. DocID.
	Method	No.	Rate kg ai/ha	Spray volume L/ha			Parent	B-1	AB-6	AB-7	
US GAP		2	0.2	Min. 935		7					(Interval: 14 days)
ORANGE											
USA, 2009 Orange, FL Hamlin R090446	Tractor mounted, PTO- Driven Airblast	2	0.2	1 × 1658 1 × 1640	Fruit	7	0.087 0.068 (0.078)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656
USA, 2009 Volusia, FL Hamlin R090447	Tractor mounted, PTO- Driven Airblast	2	0.2	1 × 1648 1 × 1655	Fruit	7	0.095 0.080 (0.088)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656
USA, 2009 Seminole, FL Navel R090448	Tractor mounted, PTO- Driven Airblast	2	0.2	1 × 699 1 × 706	Fruit	7	0.073 0.159 (0.116)	< 0.02 < 0.02	< 0.01 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656
USA, 2009 Polk, FL Hamlin R090449	SS Airblast	2	0.2	1 × 808 1 × 800	Fruit	7	0.102 0.099 (0.101)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656
USA, 2010 Osceola, FL Valencia R090450	SS Airblast	2	0.2	1 × 747 1 × 718	Fruit	7	0.107 0.109 (0.108)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656
USA, 2009 Lake, FL Hamlin R090451	SS Airblast	2	0.2	1 × 2232 1 × 2027	Fruit	7	0.046 0.075 (0.061)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656
USA, 2009 Hobe Sound Martin, FL Hamlin R090452	Airblast	2	0.2	2 × 616	Fruit	7	0.107 0.084 (0.096)	< 0.02 < 0.02	0.013 0.014	0.012 0.015	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656

CROP Country, Year Location variety Trial No.	Application				Portion analysed	DAT	Residues (mg eq/kg) ^a				Author Report Year Study No. DocID.
	Method	No.	Rate kg ai/ha	Spray volume L/ha			Parent	B-1	AB-6	AB-7	
USA, 2009 Hobe Sound Martin, FL Pineapple R090453	Airblast	2	0.2	1 × 2049 1 × 1984	Fruit	7	0.062 0.076 (0.069)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656
USA Willacy, TX N-33 Navel R090454	FMC DP 50 Airblast Sprayer 9SR-77	2	0.2	1 × 585 1 × 579	Fruit	7	0.010 0.010 (0.010)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656
USA, 2009 Tulare, CA Atwood Navel R090455	Broadcast	2	0.2	1 × 2109 1 × 2083	Fruit	0	0.055 0.061 (0.058)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt
						1	0.055 0.049 (0.052)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	350843 2012/7003656
						3	0.044 0.053 (0.0485)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
						7	0.038 0.031 (0.0345)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
						14	0.024 0.025 (0.0245)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
						21	0.015 0.014 (0.0145)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
USA, 2009 San Luis Obispo, CA Olinda Valencia R090456	Tractor- mounted, PTO- Driven Airblast	2	0.2	1 × 611 1 × 606	Fruit	7	0.016 0.011 (0.014)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656
USA, 2009 Kern, CA Navel R090457	Broadcast	2	0.2	1 × 1193 1 × 2152	Fruit	7	0.067 0.059 (0.063)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656
GAP in Brazil		1	0.08 kg ai/hL	—		7					
Brazil, 2007	(n.r.)	2	0.2	2000	Fruit	0	0.3	—	—	—	G. Casadei de Baptista 2007
			Spray concentration not reported			1	0.3	—	—	—	n.r.
						3	0.2	—	—	—	OTSA-0484-FR
						7	0.08	—	—	—	
						14	0.06	—	—	—	
Brazil, 2007	(n.r.)	2	0.2	2000	Fruit	7	< 0.05	—	—	—	G. Casadei de Baptista 2007 n.r.
			Spray concentration not reported								OTSA-0508-FR
Brazil, 2007	(n.r.)	2	0.2	2000	Fruit	0	0.1	—	—	—	G. Casadei de Baptista 2007
			Spray concentration not reported			1	0.1	—	—	—	n.r.
						3	0.08	—	—	—	OTSA-0511-FR
						7	0.06	—	—	—	
						14	< 0.05	—	—	—	
Brazil, 2007	(n.r.)	2	0.2	2000	Fruit	7	< 0.05	—	—	—	G. Casadei de Baptista 2007 n.r.
			Spray concentration not reported								OTSA-0513-FR
GRAPEFRUIT											
US GAP		2	0.2	Min. 935		7					(Interval: 14 days)

CROP Country, Year Location variety Trial No.	Application				Portion analysedd	DAT	Residues (mg eq/kg) ^a				Author Report Year Study No. DocID.
	Method	No.	Rate kg ai/ha	Spray volume L/ha			Parent	B-1	AB-6	AB-7	
USA, 2009 Brevard, FL White Marsh R090458	Airblast Sprayer	2	0.2	1 × 700 1 × 712	Fruit	0	0.191 0.084 (0.1375)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt
						1	0.101 0.057 (0.079)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	350843 2012/7003656
						3	0.097 0.043 (0.070)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
						7	0.046 0.031 (0.0385)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
						14	0.059 0.023 (0.041)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
						21	0.038 0.008 (0.023)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
USA, 2009 Lake, FL White R090459	SS Airblast	2	0.2	1 × 1798 1 × 1588	Fruit	7	0.067 0.077 (0.072)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656
USA, 2009 Polk, FL Ruby Red R090460	Broadcast	2	0.2	1 × 820 1 × 777	Fruit	7	0.039 0.040 (0.0395)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656
USA, 2009 Willacy, TX Rio Red R090461	FMC DP 50 Airblast Spayer (SR- 77)	2	0.2	1 × 2495 1 × 2467	Fruit	7	< 0.01 < 0.01 (<u>< 0.01</u>)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656
USA, 2009 Porterville Tulare, CA Mellogold R090462	Tractor- mounted, PTO- Driven Airblast	2	0.2	1 × 724 1 × 765	Fruit	7	0.036 0.038 (0.037)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656
USA, 2009 Strathmore Tulare, CA Mellogold R090463	Tractor- mounted, PTO- Driven Airblast	2	0.2	1 × 2253 1 × 2294	Fruit	7	0.027 0.019 (0.023)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656
LEMON											
USA, 2009 St. Lucie, FL Bearss R090464	Airblast Sprayer	2	0.2	1 × 664 1 × 649	Fruit	7	< 0.01 < 0.01 (<u>< 0.01</u>)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656
USA, 2009 Tulare, CA Pyrar R090465	Tractor- mounted, PTO- Driven Airblast	2	0.2	1 × 2203 1 × 2063	Fruit	0	0.122 0.113 (0.1175)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt
						1	0.086 0.112 (0.099)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	350843 2012/7003656
						3	0.106 0.100 (0.103)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
						7	0.077 0.086 (0.0815)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	

CROP Country, Year Location variety Trial No.	Application				Portion analysedd	DAT	Residues (mg eq/kg) ^a				Author Report Year Study No. DocID.
	Method	No.	Rate kg ai/ha	Spray volume L/ha			Parent	B-1	AB-6	AB-7	
						14	0.049 0.052 (0.0505)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
						21	0.031 0.041 (0.036)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
USA, 2009 Kern, CA Lisbon R090466	Tractor-mounted, PTO-Driven Airblast	2	0.2	1 × 714 1 × 742	Fruit	7	0.129 0.141 (0.135)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656
USA, 2010 Yuma, AZ Lisbon R090467	D-4015-03 Airblast	2	0.2	1 × 2818 1 × 2801	Fruit	7	0.021 0.021 (0.021)	< 0.02 < 0.02	< 0.01 < 0.01	0.010 0.011	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656
USA, 2010 San Luis Obispo, CA Eureka R090468	Tractor-mounted, PTO-Driven Airblast	2	0.2	1 × 280 1 × 299	Fruit	7	0.021 0.019 (0.020)	< 0.02 < 0.02	0.01 < 0.01	< 0.01 < 0.01	2011 / D.R. Hattermann, L.E. Crawford, S. Holt 350843 2012/7003656

^a Metabolite residues expressed in parent equivalents.

n.r. = Not reported

Pome fruits (apple and pear)

During the years 2009 and 2010, 12 trials were conducted on apple in the USA (two trials in New York, one in Pennsylvania, one in Georgia, two in Wisconsin, one in Utah, one in California, two in Idaho, one in Oregon and one in Washington). At each test location, two broadcast foliar applications of 200 g/L SC formulation of cyflumetofen were made to apple trees, at a target application rate of 0.200 kg ai/ha, with a 13–15 day retreatment interval. The applications were made to plots at each site using either concentrate (478–857 L/ha of water, Treatment 2) or dilute (1129–2274 L/ha of water, Treatment 3) spray volumes with ground equipment (airblast sprayers). At each location, apple samples (fruit, one sample per untreated plot and duplicate samples per treated plot) were harvested at a 6 to 7 day preharvest interval (PHI). At one trial in New York (trial no. R090479) treated samples were collected at 1, 3, 8, 10, and 15 days after the last application, to evaluate residue decline. The residues of cyflumetofen and metabolites B-1, AB-6 and AB-7 were quantitated by Method D1003 (LC-MS/MS). Acceptable concurrent recovery data were obtained for each analyte. The LOQ was 0.01 mg/kg for each analyte. Apparent residues of cyflumetofen, B 1, AB 6 and AB 7 were < LOQ in/on all untreated samples.

During the growing season 2006, one trial was conducted in Italy in which apple trees were treated with two applications of 250 g/L SC formulation of cyflumetofen at either 0.1 kg ai/ha, 0.2 kg ai/ha, 0.3 kg ai/ha or 0.4 kg ai/ha, diluted with water immediately prior to application to a good coverage spray volume. Samples of apple fruit from the untreated and treated plots were taken immediately after the last application, 3, 7, 14 and 21 days after last application. The LOQ for cyflumetofen in apple was set at 0.05 mg/kg. Procedural recoveries run currently with test specimen at levels of 0.05 mg/kg and 5.0 mg/kg gave an overall mean recovery of 93% and 85% respectively.

During the years 2009 and 2010, five trials were conducted on pear in the USA (one trial in New York, one in California, one in Oregon and two in Washington). At each test location, two broadcast foliar applications of 200 g/L SC formulation of cyflumetofen were made to pear trees, at a target application rate of 0.200 kg as/ha, with a 13–15 day retreatment interval. The applications were made to plots at each site using either concentrate (565–831 L/ha of water) or dilute (1111–1996 L/ha of water) spray volumes with ground equipment (airblast sprayers). At each location, pear samples

(fruit, one sample per untreated plot and duplicate samples per treated plot) were harvested at a 7 to 8 day pre-harvest interval (PHI). At one trial in Washington, treated samples were collected at 1, 3, 7, 10, and 14 days after the last application, to evaluate residue decline. The residues of cyflumetofen and metabolites B-I, AB-6 and AB-7 were quantitated using Method D1003. Acceptable concurrent recovery data were obtained for each analyte. The LOQ was 0.01 mg/kg for each analyte. Apparent residues of cyflumetofen, B 1, AB 6 and AB 7 were < LOQ in/on all untreated pear samples.

Table 65 Results of supervised residue trials on pome fruits (apple and pear) conducted in Italy and the USA

CROP Country, Year Location variety Trial No.	Application				Portion analysed	DAT	Residues (mg eq/kg) ^a				Author Report Year Study No. DocID.
	Method	No.	Rate kg ai/ha	Spray volume L/ha			Parent	B-1	AB-6	AB-7	
US GAP		2	0.2	Min. 935		7					(Interval: 14 days)
APPLE											
USA, 2009 Wayne, NY	Broad-cast foliar	2	0.2	1 × 562 1 × 571	Fruit	1	0.182 0.175	< 0.02 < 0.02	0.011 < 0.01	< 0.01 < 0.01	M. T. White 2011
Romes RCN R090479						3	0.196 0.125	< 0.02 < 0.02	< 0.01 0.011	< 0.01 < 0.01	375302 2011/7000143
						8	0.248 0.122 (0.185)	< 0.02 < 0.02	0.016 0.015	0.013 0.011	
						10	0.068 0.095	< 0.02 < 0.02	0.011 0.013	< 0.01 < 0.01	
						15	0.104 0.073	< 0.02 < 0.02	0.012 0.010	< 0.01 < 0.01	
		2	0.2	1 × 1129 1 × 1133	Fruit	1	0.121 0.122	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
						3	0.079 0.065	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
						8	0.065 0.055 (0.060)	< 0.02 < 0.02	0.011 0.010	< 0.01 < 0.01	
						10	0.051 0.071 (0.061)	< 0.02 < 0.02	0.011 0.011	< 0.01 < 0.01	
						15	0.041 0.046	< 0.02 < 0.02	< 0.01 0.010	< 0.01 < 0.01	
USA, 2009 Wayne, NY	Broad-cast foliar	2	0.2	1 × 609 1 × 610	Fruit	7	0.101 0.091 (0.096)	< 0.02 < 0.02	< 0.01 0.010	< 0.01 < 0.01	M. T. White 2011
Romes RCN R090480		2	0.2	1 × 2091 1 × 2097	Fruit	7	0.067 0.058 (0.0625)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	375302 2011/7000143
USA, 2009 Berks, PA	Broad-cast foliar	2	0.2	1 × 563 1 × 564	Fruit	6	0.160 0.238 (0.199)	< 0.02 < 0.02	< 0.01 0.010	0.011 0.012	M. T. White 2011
Starkrimson RCN R090481		2	0.2	1 × 2243 1 × 2249	Fruit	6	0.119 0.091 (0.105)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	375302 2011/7000143
USA, 2009 Fannin, GA	Broad-cast foliar	2	0.2	1 × 691 1 × 613	Fruit	6	0.214 0.139 (0.1765)	< 0.02 < 0.02	0.012 0.012	0.013 0.011	M. T. White 2011
Yate RCN R090482		2	0.2	1 × 1594 1 × 1398	Fruit	6	0.082 0.108 (0.095)	< 0.02 < 0.02	< 0.01 < 0.01	0.011 < 0.01	375302 2011/7000143
USA, 2009 Lenawee, MI	Broad-cast foliar	2	0.2	1 × 823 1 × 806	Fruit	7	0.168 0.153 (0.1605)	< 0.02 < 0.02	0.018 0.017	0.013 0.012	M. T. White 2011

CROP Country, Year Location variety Trial No.	Application				Portion analysed	DAT	Residues (mg eq/kg) ^a				Author Report Year Study No. DocID.
	Method	No.	Rate kg ai/ha	Spray volume L/ha			Parent	B-1	AB-6	AB-7	
Delicious RCN R090483		2	0.2	1 × 1778 1 × 1757	Fruit	7	0.080 0.137 (0.1085)	< 0.02 < 0.02	< 0.01 0.010	< 0.01 < 0.01	375302 2011/7000143
USA, 2009 Pepin, WI (Connel Red)	Broad-cast foliar	2	0.2	1 × 478 1 × 483	Fruit	6	0.130 0.122 (0.126)	< 0.02 < 0.02	0.012 0.015	< 0.01 0.013	M. T. White 2011
RCN R090484		2	0.2	1 × 1927 1 × 1941	Fruit	6	0.027 0.045 (0.036)	< 0.02 < 0.02	< 0.01 0.011	< 0.01 < 0.01	375302 2011/7000143
USA, 2009 Cache, UT	Broad-cast foliar	2	0.2	1 × 707 1 × 707	Fruit	7	0.042 0.042 (0.042)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	M. T. White 2011
Red Delicious RCN R090485		2	0.2	1 × 1598 1 × 1659	Fruit	7	0.061 0.059 (0.060)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	375302 2011/7000143
USA, 2009 Madera, CA	Broad-cast foliar	2	0.2	1 × 747 1 × 751	Fruit	7	0.033 0.047 (0.040)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	M. T. White 2011
Fuji RCN R090486		2	0.2	1 × 1888 1 × 1858	Fruit	7	0.032 0.024 (0.028)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	375302 2011/7000143
USA, 2009 Bingham, ID	Broad-cast foliar	2	0.2)	1 × 576 1 × 555	Fruit	7	0.022 < 0.01 (0.0135)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	M. T. White 2011
McIntosh RCN R090487		2	0.2	1 × 1423 1 × 1335	Fruit	7	0.011 0.018 (0.0145)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	375302 2011/7000143
USA, 2009 Bingham, ID	Broad-cast foliar	2	0.2	1 × 574 1 × 552	Fruit	7	0.078 0.040	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	M. T. White 2011
Golden Delicious RCN R090488		2	0.2	1 × 1439 1 × 1385	Fruit	7	0.077 0.122 (0.0995)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	375302 2011/7000143
USA, 2010 Hood River, OR	Broad-cast foliar	2	0.2	1 × 857 1 × 822	Fruit	7	0.072 0.067 (0.0695)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	M. T. White 2011
Honey Crisp RCN R090489		2	0.2	1 × 2274 1 × 2235	Fruit	7	0.054 0.052 (0.053)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	375302 2011/7000143
USA, 2010 Grant, WA	Broad-cast foliar	2	0.2	1 × 615 1 × 611	Fruit	7	0.209 0.177 (0.193)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	M. T. White 2011
Red Delicious RCN R090490		2	0.2	1 × 1413 1 × 1424	Fruit	7	0.123 0.143 (0.133)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	375302 2011/7000143
Italy, 2006	Mist blower	2	0.1	1 × 1241	Fruit	0	< 0.05	—	—	—	n.r.
Bologna	sprayer			1 × 1132		3	< 0.05	—	—	—	2007
Double red						7	< 0.05	—	—	—	AF/10943/OT
AF/10943/OT/						14	< 0.009	—	—	—	OTSA-0301-
1						21	< 0.009	—	—	—	FR
		2	0.2	1 × 1193	Fruit	0	0.0998	—	—	—	
				1 × 1175		3	0.0780	—	—	—	
						7	0.0596	—	—	—	
						14	< 0.05	—	—	—	
						21	< 0.05	—	—	—	
		2	0.3	1 × 1197	Fruit	0	0.149	—	—	—	
				1 × 1158		3	0.111	—	—	—	

CROP Country, Year Location variety Trial No.	Application				Portion analysed	DAT	Residues (mg eq/kg) ^a				Author Report Year Study No. DocID.
	Method	No.	Rate kg ai/ha	Spray volume L/ha			Parent	B-1	AB-6	AB-7	
						7	< 0.05	—	—	—	
						14	< 0.05	—	—	—	
						21	< 0.05	—	—	—	
		2	0.4	1 × 1197	Fruit	0	0.151	—	—	—	
				1 × 1153		3	0.0914	—	—	—	
						7	0.0501	—	—	—	
						14	0.0522	—	—	—	
						21	< 0.05	—	—	—	
PEAR											
USA, 2009 Wayne, NY	Broad-cast foliar	2	0.2	1 × 565 1 × 567	Fruit	8	0.127 0.125	< 0.02 < 0.02	0.038 0.027	0.036 0.028	M. T. White 2011
Bosc RCN R090491		2	0.2	1 × 1111 1 × 1118	Fruit	8	0.121 0.265 (0.1995)	< 0.02 < 0.02 < 0.02	0.034 0.032	0.034 0.035	375302 2011/7000143
USA, 2009 Tulare, CA	Broad-cast foliar	2	0.2	1 × 819 1 × 831	Fruit	7	0.045 0.038 (0.0415)	< 0.02 < 0.02	< 0.01 < 0.01	0.011 < 0.01	M. T. White 2011
Olympic RCN R090493		2	0.2	1 × 1808 1 × 1831	Fruit	7	0.064 0.087 (0.0755)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 0.012	375302 2011/7000143
USA, 2009 Hood River, OR	Broad-cast foliar	2	0.2	1 × 805 1 × 765	Fruit	7	0.032 0.039 (0.0355)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	M. T. White 2011
Starkrismson RCN R090494		2	0.2	1 × 1729 1 × 1996	Fruit	7	0.061 0.065 (0.063)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	375302 2011/7000143
USA, 2010 Grant, WA	Broad-cast foliar	2	0.2	1 × 607 1 × 610	Fruit	1	0.217 0.226	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	M. T. White 2011
Concord RCN R090495						3	0.206 0.203	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	375302 2011/7000143
						7	0.125 0.106 (0.1155)	< 0.02 < 0.02	0.014 < 0.01	0.012 < 0.01	
						10	0.086 0.116	< 0.02 < 0.02	0.010 0.013	< 0.01 0.012	
						14	0.055 0.079	< 0.02 < 0.02	< 0.01 0.014	< 0.01 0.011	
		2	0.2	1 × 1398 1 × 1401	Fruit	1	0.215 0.231	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
						3	0.231 0.170	< 0.02 < 0.02	0.012 0.010	0.011 < 0.01	
						7	0.125 0.114 (0.1195)	< 0.02 < 0.02	0.012 0.013	0.011 0.012	
						10	0.081 0.100	< 0.02 < 0.02	0.012 0.012	< 0.01 < 0.01	
						14	0.063 0.077	< 0.02 < 0.02	0.011 0.012	< 0.01 < 0.01	
USA, 2010 Grant, WA	Broad-cast foliar	2	0.2	1 × 610 1 × 608	Fruit	7	0.089 0.124 (0.1065)	< 0.02 < 0.02	0.013 0.016	0.011 0.014	M. T. White 2011
Bartlett RCN R090496		2	0.2	1 × 1403 1 × 1396	Fruit	7	0.085 0.125 (0.105)	< 0.02 < 0.02	0.010 0.011	< 0.01 < 0.01	375302 2011/7000143

^a Metabolite residues expressed in parent equivalents.

n.r.= Not reported

*Berries and Other Small Fruits**Grapes*

During the 2009 growing season, a total of 12 trials were conducted on grapes in the USA (two trials in New York, eight in California, one in Washington, and one in Oregon. One in California was a decline trial. In all trials, two broadcast foliar applications of 200 g/L SC formulation of cyflumetofen at 0.193–0.206 kg ai/ha were made with a 14–15 re-treatment interval. The applications were made with ground equipment using either a dilute or concentrated spray volume. Grape Samples were collected at 14 DAT. In the decline trial, the samples were collected at 0, 3, 7, 14, 21 and 28 DAT.

Samples of grape were analysed for residues of cyflumetofen and metabolites B-1, AB-6 and AB-7 using Method D1003. Acceptable concurrent recovery data for grape commodities between 69% and 111% were obtained for each analyte. The LOQ was 0.01 mg/kg for each analyte.

Table 66 Results of supervised residue trials on Grapes conducted in the USA

CROP Country, Year Location variety Trial No.	Application				Portion analysed	DAT	Residues (mg eq/kg) ^a				Author Report Year Study No. DocID.
	Method	No.	Rate kg ai/ha	Spray volume L/ha			Parent	B-1	AB-6	AB-7	
US GAP		2	0.2	Min. 468		14					(Interval: 14 days)
USA, 2009 Seneca, NY Catawba R090511	broadcast foliar application	2	0.20	1 × 2313 1 × 2354	Fruit	14	0.252 0.290 (0.271)	0.064 0.061 (0.0625)	< 0.01 < 0.01	< 0.01 < 0.01	D. R. Hattermann, T. Lott, 2011 375306 2011/7003995
USA, 2009 Yates, NY Vidal Blanc R090512	broadcast foliar application	2	0.20	1 × 466 1 × 473	Fruit	14	0.406 0.439 (0.4225)	0.040 0.045 (0.0425)	< 0.01 < 0.01	< 0.01 < 0.01	D. R. Hattermann, T. Lott, 2011 375306 2011/7003995
USA, 2009 Kern, CA Thompson Seedless R090513	broadcast foliar application	2	0.20	1 × 721 1 × 703	Fruit	14	0.073 0.124 (0.0985)	0.024 0.024 (0.024)	< 0.01 < 0.01	< 0.01 < 0.01	D. R. Hattermann, T. Lott, 2011 375306 2011/7003995
USA, 2009 Kingsberg Fresno, CA Crimson R090514	broadcast foliar application	2	0.20	1 × 1884 1 × 1889	Fruit	14	0.175 0.151 (0.163)	0.035 0.040 (0.0375)	0.013 0.016	0.013 0.015	D. R. Hattermann, T. Lott, 2011 375306 2011/7003995
USA, 2009 San Luis Obispo, CA Syrah R090515	broadcast foliar application	2	0.20	1 × 466 1 × 448	Fruit	14	0.014 0.031 (0.0225)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	D. R. Hattermann, T. Lott, 2011 375306 2011/7003995
USA, 2009 Monterey, CA Syrah R090516	broadcast foliar application	2	0.20 0.20	1 × 628 1 × 602	Fruit	14	0.102 0.085 (0.0935)	0.024 0.024 (0.024)	0.011 < 0.01	< 0.01 < 0.01	D. R. Hattermann, T. Lott, 2011 375306 2011/7003995
USA, 2009 Kerman Fresno, CA Thompson Seedless R090517	broadcast foliar application	2	0.20 0.20	1 × 1876 1 × 1886	Fruit	14	0.125 0.117 (0.121)	0.071 0.061 (0.066)	0.013 0.013	0.012 0.011	D. R. Hattermann, T. Lott, 2011 375306 2011/7003995
USA, 2009 Kerman Fresno, CA Thompson Seedless R090518	broadcast foliar application	2	0.20 0.20	1 × 704 1 × 694	Fruit	14	0.126 0.121 (0.1235)	0.040 0.038 (0.039)	0.013 0.016	0.014 0.016	D. R. Hattermann, T. Lott, 2011 375306 2011/7003995

CROP Country, Year Location variety Trial No.	Application				Portion analysed	DAT d	Residues (mg eq/kg) ^a				Author Report Year Study No. DocID.
	Method	No.	Rate kg ai/ha	Spray volume L/ha			Parent	B-1	AB-6	AB-7	
USA, 2009 Plainview Tulare, CA Crimson R090519	broadcast foliar application	1+ 1	0.20 0.19	1 × 1968 1 × 2112	Fruit	0	0.312 0.349	0.024 0.028	< 0.01 < 0.01	< 0.01 < 0.01	D. R. Hattermann, T. Lott, 2011 375306 2011/7003995
						3	0.289 0.367	0.028 0.028	< 0.01 < 0.01	< 0.01 < 0.01	
						7	0.246 0.285	0.024 0.026	< 0.01 < 0.01	< 0.01 < 0.01	
						14	0.179 0.199 (0.189)	0.026 0.033 (0.0295)	< 0.01 < 0.01	< 0.01 < 0.01	
						21	0.096 0.181	0.045 0.040	< 0.01 < 0.01	< 0.01 0.012	
						28	0.122 0.104	0.031 0.033	< 0.01 < 0.01	0.010 < 0.01	
USA, 2009 Earlimart Tulare, CA Crimson R090520	broadcast foliar application	2	0.20 (0.20)	1 × 1880 1 × 1890	Fruit	14	0.225 ^b 0.223 ^b (0.224)	0.024 0.024 (0.024)	< 0.01 < 0.01	0.012 ^a 0.011 ^a	D. R. Hattermann, T. Lott, 2011 375306 2011/7003995
USA, 2009 Yakima, CA Riesling R090521	broadcast foliar application	1+ 1	0.21 0.20	1 × 888 1 × 859	Fruit	14	0.271 0.177 (0.224)	0.024 0.024 (0.024)	< 0.01 < 0.01	< 0.01 < 0.01	D. R. Hattermann, T. Lott, 2011 375306 2011/7003995
USA, 2009 Benton, OR Pinot R090522	broadcast foliar application	1+ 1	1 × 0.19 1 × 0.20	1 × 2612 1 × 2542	Fruit	14	0.154 0.142 (0.148)	0.085 0.075 (0.080)	0.034 0.027	0.021 0.019	D. R. Hattermann, T. Lott, 2011 375306 2011/7003995

^a Metabolite residues expressed in parent equivalents.

^b Results of multiple analyses of individual field samples.

Strawberry

During the growing season 2009–2010, a total of eight field trials on strawberries were conducted in the USA (one trial in New York, one in Georgia, one in Florida one in Wisconsin, three in California, and one in Oregon). Each trial location consisted of one untreated and one treated plot. Each treated plot received two broadcast foliar applications of 200 g/L SC formulation of Cyflumetofen at 0.196–0.206 kg ai/ha, with a retreatment interval targeting 14 ± 1 days. The actual retreatment interval was 13–15 days, with the exception of one site in California (trial no. R090501) where a 7 day retreatment interval was used. The applications were made using ground equipment in 225–460 L/ha of water spray volumes. Strawberry samples were harvested at a 1-day preharvest interval (PHI). At one trial in California (trial no. R090502), additional treated samples were collected at 0, 3, 7 and 10 days after the last application, in addition to the targeted 1-day PHI, to evaluate residue decline.

Samples of strawberry were analysed for residues of cyflumetofen and metabolites B-1, AB-6 and AB-7 using Method D1003. Acceptable concurrent recovery data for strawberry commodities were obtained for each analyte. The LOQ was 0.01 mg/kg for each analyte. Apparent residues of cyflumetofen, B-1, AB-6 and AB-7 were < LOQ in/on all samples of untreated strawberries.

Table 67 Results of supervised residue trials on Strawberry conducted in the USA.

CROP Country, Year Location variety Trial No.	Application				Portion analysed	DAT d	Residues (mg eq/kg) ^a				Author Report Year Study No. DocID.
	Method	No.	Rate kg ai/ha	Spray volume L/ha			Parent	B-1	AB-6	AB-7	
US GAP		2	0.2	Min. 468		1					(Interval: 14 days)
USA, 2010 Dane, WI L'Amour R090497	Broad-cast foliar	2	0.2	1 × 245 1 × 250	Berry	1	0.04 0.04 (0.04)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	M. White, M. Saha 2011 375304 2010/7014999
USA, 2010 Tift, GA Camarosa R090498	Broad-cast foliar	2	0.2	1 × 237 1 × 225	Berry	1	0.11 0.09 (0.10)	0.031 0.026 (0.0285)	< 0.01 < 0.01	< 0.01 < 0.01	M. White, M. Saha 2011 375304 2010/7014999
USA, 2009 Washington, OR Albion R090499	Broad-cast foliar	2	0.2	2 × 235	Berry	1	0.23 0.18 (0.205)	0.032 0.029 (0.0305)	< 0.01 < 0.01	< 0.01 < 0.01	M. White, M. Saha 2011 375304 2010/7014999
USA, 2010 Yates, NY Honeoye R090500	Broad-cast foliar	2	0.2	1 × 455 1 × 460	Berry	1	0.15 0.18 (0.165)	0.028 0.032 (0.030)	< 0.01 < 0.01	< 0.01 < 0.01	M. White, M. Saha 2011 375304 2010/7014999
USA, 2010 Tulare, CA Diamante R090501	Broad-cast foliar	2	0.2	1 × 306 1 × 300	Berry	1	0.21 0.25 (0.23)	< 0.02 0.104 (0.052)	< 0.01 < 0.01	< 0.01 < 0.01	M. White, M. Saha 2011 375304 2010/7014999
USA, 2009 Santa Barbara, CA Albion R090502	Broad-cast foliar	2	0.2	2 × 374	Berry	0	0.16 0.21	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	M. White, M. Saha 2011
						1	0.11 0.14 (0.125)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	375304 2010/7014999
						3	0.16 0.11 (0.135)	0.028 < 0.02 (0.024)	< 0.01 < 0.01	< 0.01 < 0.01	
						7	0.15 0.11	0.026 0.027	< 0.01 < 0.01	< 0.01 < 0.01	
						10	0.10 0.09	0.025 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
USA, 2010 Madera, CA San Andreas R090503	Broad-cast foliar	2	0.2	1 × 284 1 × 279	Berry	1	0.16 0.12 (0.14)	0.025 0.025	< 0.01 < 0.01	< 0.01 < 0.01	M. White, M. Saha 2011 375304 2010/7014999
USA, 2010 Lake, FL Camarosa R090504	Broad-cast foliar	2	0.2	1 × 371 1 × 370	Berry	1	0.29 0.44 (0.365)	0.037 0.035 (0.036)	< 0.01 < 0.01	< 0.01 < 0.01	M. White, M. Saha 2011 375304 2010/7014999

^a Metabolite residues expressed in parent equivalents.

Fruiting vegetables, other than Cucurbits (Tomato, eggplant)

During the growing season 2009–2010, a total of 16 field trials were conducted on tomatoes in the USA (one trial in New York, one in Georgia, two in Florida, one in Kansas, 10 in California and one in Arizona). Two of these trials were decline trials (one trial in NY and the other in CA). At each test location, one untreated and one treated plot were established. The treated plot received two broadcast foliar applications of 200 g/L CS formulation of cyflumetofen at 0.196–0.211 kg ai/ha/application, with a 14-day (+/– one day) re-treatment interval. The applications were made with ground equipment using 115–472 L/ha of water. Commercially mature, tomato samples were collected 3 days after treatment (DAT).

All samples were analysed for residues of cyflumetofen, and its metabolites, B-1, AB-6, and AB-7, using Method D1003. Mean recoveries for all analytes were within the acceptable range of 70–120%. Three individual recoveries were in the range of 65–69%. The LOD and LOQ for all analytes were 0.002 and 0.01 mg/kg, respectively.

Table 68 Results of supervised residue trials on Tomato conducted in the USA.

CROP Country, Year Location variety Trial No.	Application				Portion analysed	DAT d	Residues (mg eq/kg) ^a				Author Report Year Study No. DocID.
	Method	No.	Rate kg ai/ha	Spray volume L/ha			Parent	B-1	AB-6	AB-7	
US GAP		2	0.2	Min. 468		3					(Interval: 14 days)
USA, 2009 Wayne, NY Basket Vee R090531	Backpack hand- boom	2	0.2	2 × 234	Fruit	0	0.23 0.14	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	L.E. Crawford 2011 375305 2011/7002631
						1	0.04 0.03	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
						3	< 0.01 0.05 (0.03)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
						7	0.02 0.11 (0.064)	< 0.02 0.04 (0.03)	< 0.01 < 0.01	< 0.01 < 0.01	
						14	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
						21	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
USA, 2009 Tift, GA Mountain Fresh R090532	Backpack hand- boom	2	0.2	1 × 345 1 × 352	Fruit	3	0.03 0.04 (0.035)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	L.E. Crawford 2011 375305 2011/7002631
USA, 2009 Jackson, FL BHN602 R090533	Backpack hand- boom	2	0.2	2 × 347	Fruit	3	0.02 0.06 (0.04)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	L.E. Crawford 2011 375305 2011/7002631
USA, 2009 Seminole, FL Better Boy R090534	Backpack hand- boom	2	2 × 0.2	1 × 284 1 × 280	Fruit	3	0.03 0.02 (0.025)	0.05 0.02 (0.035)	< 0.01 < 0.01	< 0.01 < 0.01	L.E. Crawford 2011 375305 2011/7002631
USA, 2009 Barber, KS Early Girl R090535	Backpack hand- boom	2	0.2	1 × 115 1 × 149	Fruit	3	0.07 0.07 (0.07)	0.02 0.02 (0.02)	< 0.01 < 0.01	< 0.01 < 0.01	L.E. Crawford 2011 375305 2011/7002631
USA, 2009 Fresno, CA 2401 R090536	Backpack hand- boom	2	0.2	1 × 467 1 × 472	Fruit	3	0.01 0.02 (0.015)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	L.E. Crawford 2011 375305 2011/7002631
USA, 2009 Merced, CA Heinz 2780 R090537	Backpack hand- boom	2	0.2	2 × 466	Fruit	3	0.06 0.07 (0.065)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	L.E. Crawford 2011 375305 2011/7002631
USA, 2010 Kerman Fresno, CA Sun 6366 R090538	Backpack hand- boom	2	0.2	1 × 189 1 × 187	Fruit	3	0.04 0.03 (0.035)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	L.E. Crawford 2011 375305 2011/7002631
USA, 2010 Kwerman Fresno, CA APT 410 R090539	Backpack hand- boom	2	0.2	1 × 189 1 × 187	Fruit	3	0.04 0.05 (0.045)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	L.E. Crawford 2011 375305 2011/7002631
USA, 2010 Porterville	Backpack hand- boom	2	0.2	1 × 339 1 × 335	Fruit	0	0.06 0.05	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	L.E. Crawford 2011

CROP Country, Year Location variety Trial No.	Application				Portion analysed	DAT d	Residues (mg eq/kg) ^a				Author Report Year Study No. DocID.
	Method	No.	Rate kg ai/ha	Spray volume L/ha			Parent	B-1	AB-6	AB-7	
Tular, CA Tom Whopper R090540						1	0.02 0.03	0.02 0.02	< 0.01 < 0.01	< 0.01 < 0.01	375305 2011/7002631
						3	0.02 0.02 (0.02)	0.05 0.02 (0.035)	< 0.01 < 0.01	< 0.01 < 0.01	
						7	0.02 0.01	0.07 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
						14	< 0.01 0.01	0.07 0.09	< 0.01 < 0.01	< 0.01 < 0.01	
						21	< 0.01 < 0.01	0.02 0.05	< 0.01 < 0.01	< 0.01 < 0.01	
USA, 2010 Porterville Tular, CA ACE 55 VF (AS) R090541	Backpack hand- boom	2	0.2	1 × 337 1 × 335	Fruit	3	0.05 0.07 (0.06)	< 0.02 0.02	< 0.01 < 0.01	< 0.01 < 0.01	L.E. Crawford 2011 375305 2011/7002631
USA, 2009 Tular, CA Naoni Cherry (CT) R090542	Tractor mounted sprayer	2	0.2	1 × 363 1 × 383	Fruit	3	0.12 0.12 (0.12)	0.07 0.05 (0.06)	< 0.01 < 0.01	< 0.01 < 0.01	L.E. Crawford 2011 375305 2011/7002631
USA, 2009 King City Monterey, CA 8004 R090543	Backpack hand- boom	2	0.2	1 × 274 1 × 283	Fruit	3	0.03 0.04 (0.035)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	L.E. Crawford 2011 375305 2011/7002631
USA, 2009 San Ardo Monterey, CA 8004 R090544	Backpack hand- boom	2	0.2	1 × 288 1 × 281	Fruit	3	0.10 0.08 (0.09)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	L.E. Crawford 2011 375305 2011/7002631
USA, 2009 Tehama, CA APT 410 R090545	Backpack hand- boom	2	0.2	2 × 187	Fruit	3	0.16 0.14 (0.15)	< 0.02 < 0.02	0.02 0.01	0.03 0.02	L.E. Crawford 2011 375305 2011/7002631
USA, 2010 Yuma, AZ Montain Fresh R090546	Backpack hand- boom	2	0.2	1 × 230 1 × 229	Fruit	3	0.01 0.01 (0.01)	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	L.E. Crawford 2011 375305 2011/7002631

^a Metabolite residues expressed in parent equivalents.

During the growing season 2004, two trials were conducted on eggplants in greenhouses in Japan to determine the magnitude of the residues of cyflumetofen after treatment with 20% SC formulation of cyflumetofen. The formulation was applied two times at an application rate of 0.400 kg ai/ha and a spray volume of 2000 L/ha. The samples were taken at 1, 3, 7 and 21 days after the last application. The residues of cyflumetofen were determined by LC-UV detection. The LOQ was 0.05 mg/kg.

Table 69 Results of supervised residue trials on Eggplant conducted in Japan

CROP Country, Year Location variety Trial No.	Application				Portion analysed	DAT d	Residues (mg eq/kg) ^a				Author Report Year Study No. DocID.
	Method	No.	Rate kg ai/ha	Spray volume L/ha			Parent	B-1	AB-6	AB-7	
GAP in Japan		2	0.02 kg ai/hL	(1000– 3500)		1					

CROP Country, Year Location variety Trial No.	Application				Portion analysed	DAT	Residues (mg eq/kg) ^a				Author Report Year Study No. DocID.
	Method	No.	Rate kg ai/ha	Spray volume L/ha			Parent	B-1	AB-6	AB-7	
Japan, 2004 Kochi	n.r.	2	0.40	2 × 2000	Fruit (calyx removed)	1	0.35 0.32 (0.335)	0.06 0.05	< 0.05 < 0.05	< 0.05 < 0.05	n.r. 2004
Ryuma n.r.						3	0.28 0.24	0.06 0.06	< 0.05 < 0.05	< 0.05 < 0.05	Saku 16-P-2-48 OTSA-0203 (2)-FR
(greenhouse)						7	0.06 0.06	0.09 0.09	< 0.05 < 0.05	< 0.05 < 0.05	
						21	< 0.05 < 0.05	0.13 0.12	< 0.05 < 0.05	< 0.05 < 0.05	
Japan, 2004 Miyazaki	n.r.	2	0.40	2 × 2000	Fruit (calyx removed)	1	0.46 0.45 (0.455)	0.43 0.42	< 0.05 < 0.05	< 0.05 < 0.05	n.r. 2004
Kokuyo n.r.						3	0.37 0.36	0.50 0.49	< 0.05 < 0.05	< 0.05 < 0.05	Saku 16-P-2-48 OTSA-0203 (2)-FR
(greenhouse)						7	0.06 0.06	0.58 0.58	< 0.05 < 0.05	< 0.05 < 0.05	
						21	< 0.05 < 0.05	0.26 0.25	< 0.05 < 0.05	< 0.05 < 0.05	

^a Metabolite residues expressed in parent equivalents.

n.r. = Not reported

Tree nuts (Almond and pecan)

During the growing season 2009, five trials were conducted on almonds in California in the USA. Each trial consisted of one untreated control and one treated plot except for sites that where both dilute and concentrate applications in which case there were two treated plots and one non-treated plot. For the concentrate plots, the treated plot received two air-blast foliar broadcast applications of 200 g/L SC formulation of cyflumetofen at 0.195–0.202 kg ai/ha, with a 14 day re-treatment interval. The applications were made with ground equipment using 469–656 L/ha of water. For the dilute plots, the treated plot received two airblast foliar broadcast applications of 200 g/L SC formulation of cyflumetofen at 0.197–0.200 kg ai/ha/, with a 13–14 day re-treatment interval. The applications were made with ground equipment using 1219–1875 L/ha of water. Almond samples were harvested 7 days after the last application for all trials except the decline trial (R090478). For the decline trial, samples were collected 0, 1, 3, 7, 14, and 21 days after the last application.

All samples were analysed for residues of cyflumetofen, and its metabolites, B-1, AB-6, and AB-7, using Method D1003. Mean recoveries for all analytes were within the acceptable range of 70–120%. The LOD and LOQ for all analytes were 0.002 and 0.01 mg/kg, respectively.

During the growing season 2009, five trials were conducted on pecans in the USA (two trials in Georgia, one in Louisiana, one in Texas and one in Oklahoma). Each trial consisted of one untreated control and one treated plot except for sites that where both dilute and concentrate applications in which case there were two treated plots and one non-treated plot. For the concentrate plots, the treated plot received two air-blast foliar broadcast applications of 200 g/L SC formulation of cyflumetofen at 0.195–0.204 kg ai/ha, with a 13–14 day re-treatment interval. The applications were made with ground equipment using 540–794 L/ha of water. For the dilute plots, the treated plot received two airblast foliar broadcast applications of 200 g/L SC formulation of Cyflumetofen at 0.198–0.205 kg ai/ha, with a 13–14 day re-treatment interval. The applications were made with ground equipment using 1104–2376 L/ha of water. Pecan samples were harvested 7 or 8 days after the last application.

All samples were analysed for residues of cyflumetofen, and its metabolites, B-1, AB-6, and AB-7, using Method D1003. Mean recoveries for all analytes were within the acceptable range of 70–120%. The LOD and LOQ for all analytes were 0.002 and 0.01 mg/kg, respectively.

Table 70 Results of supervised residue trials on Almonds and Pecans conducted in the USA

CROP Country, Year Location variety Trial No.	Application				Portion analysed	DAT d	Residues (mg eq/kg) ^a				Author Report Year Study No. DocID.
	Method	No.	Rate kg ai/ha	Spray volume L/ha			Parent	B-1	AB-6	AB-7	
US GAP		2	0.2	Min. 935		7					(Interval: 14 days)
ALMOND											
USA, 2009 Fresno, CA Non-Pariel R090474	Airblast foliar broadcast	2	0.20	1 × 1848 1 × 1875	Nutmeat	7	< 0.01 < 0.01 (<u>< 0.01</u>)	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	L. E. Crawford 2011 375303 2011/7003996
USA, 2009 Kern, CA Price R090475	Airblast foliar broadcast	2	0.20	1 × 656 1 × 610	Nutmeat	7	< 0.01 < 0.01 (<u>< 0.01</u>)	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	L. E. Crawford 2011 375303 2011/7003996
USA, 2009 Tulare, CA Carmel R090476	Airblast foliar broadcast	2	0.20	1 × 1813 1 × 1675	Nutmeat	7	< 0.01 < 0.01 (<u>< 0.01</u>)	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	L. E. Crawford 2011 375303 2011/7003996
USA, 2009 Glenn, CA Non-Pariel R090477	Airblast foliar broadcast	2	0.20	1 × 469 1 × 473	Nutmeat	7	< 0.01 < 0.01 (<u>< 0.01</u>)	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	L. E. Crawford 2011 375303 2011/7003996
USA, 2009 Tulare, CA	Airblast foliar	2	0.20	1 × 1219 1 × 1476	Nutmeat	0	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	L. E. Crawford 2011
Monterey R090478	broadcast					1	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	375303 2011/7003996
						3	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
						7	< 0.01 < 0.01 (<u>< 0.01</u>)	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	
						14	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
						21	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
PECAN											
USA, 2009 Tift, GA Sumner R090469	Airblast foliar broadcast	2	0.20	1 × 2081 1 × 1999	Nutmeat	7	< 0.01 < 0.01 (<u>< 0.01</u>)	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	L. E. Crawford 2011 375303 2011/7003996
USA, 2009 Irwin, GA Sumner R090470	Airblast foliar broadcast	2	0.20	1 × 667 1 × 648	Nutmeat	7	< 0.01 < 0.01 (<u>0.01</u>)	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	L. E. Crawford 2011 375303 2011/7003996
USA, 2009 Rapides Parish, LA	Airblast foliar broadcast	2	0.20	1 × 1909 1 × 1821	Nutmeat	7	< 0.01 < 0.01 (<u>< 0.01</u>)	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	L. E. Crawford 2011 375303 2011/7003996
Creek R090471				1 × 743 1 × 794	Nutmeat	7	< 0.01 < 0.01 (<u>< 0.01</u>)	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	
USA, 2009 Jackson, OK	Airblast foliar broadcast	2	0.20	1 × 2376 1 × 2312	Nutmeat	8	< 0.01 < 0.01 (<u>< 0.01</u>)	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	L. E. Crawford 2011 375303 2011/7003996
Cheyenne R090472				1 × 740 1 × 731	Nutmeat	8	< 0.01 < 0.01 (<u>< 0.01</u>)	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	
USA, 2009 Frio, TX Wichita R090473	Airblast foliar broadcast	2	0.20	1 × 1159 1 × 1104	Nutmeat	7	< 0.01 < 0.01 (<u>< 0.01</u>)	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	L. E. Crawford 2011 375303 2011/7003996
				1 × 558 1 × 540	Nutmeat	7	< 0.01 < 0.01 (<u>< 0.01</u>)	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	

^a Metabolite residues expressed in parent equivalents.

Animal feeds

Almond hulls

Table 71 Results of supervised residue trials on Almond hulls conducted in the USA

CROP Country, Year Location variety Trial No.	Application				Portion analysed	DAT d	Residues (mg/kg) ^a				Author Report Year Study No. DocID.
	Method	No.	Rate kg ai/ha	Spray volume L/ha			Parent	B-1	AB-6	AB-7	
US GAP			0.2	Min. 935		7					(Interval: 14 days)
ALMOND											
USA, 2009 Fresno, CA Non-Pariel R090474	Airblast foliar broadcast	2	0.20	1 × 1848 1 × 1875	Hull	7	2.046 1.696 (1.871)	0.294 0.191 (0.242)	0.227 0.189	0.137 0.116	L. E. Crawford 2011 375303 2011/7003996
USA, 2009 Kern, CA Price R090475	Airblast foliar broadcast	2	0.20	1 × 656 1 × 610	Hull	7	0.522 0.545 (0.534)	0.092 0.094 (0.093)	0.074 0.076	0.021 0.022	L. E. Crawford 2011 375303 2011/7003996
USA, 2009 Tulare, CA Carmel R090476	Airblast foliar broadcast	2	0.20	1 × 1813 1 × 1675	Hull	7	0.840 0.887 (0.864)	0.106 0.115 (0.111)	0.102 0.096	0.040 0.041	L. E. Crawford 2011 375303 2011/7003996
USA, 2009 Glenn, CA Non-Pariel R090477	Airblast foliar broadcast	2	0.20	1 × 469 1 × 473	Hull	7	0.339 0.364 (0.352)	0.028 0.026 (0.027)	0.073 0.073	0.025 0.025	L. E. Crawford 2011 375303 2011/7003996
USA, 2009 Tulare, CA Monterey R090478	Airblast foliar broadcast	2	0.20	1 × 1219 1 × 1476	Hull	0	0.737 0.885	0.045 0.035	0.104 0.098	0.027 0.026	L. E. Crawford 2011 375303 2011/7003996
						1	0.758 0.829	0.045 0.049	0.107 0.097	0.031 0.028	
						3	0.464 0.481	0.040 0.045	0.101 0.098	0.029 0.027	
						7	0.628 0.437 (0.533)	0.028 0.028	0.101 0.103	0.027 0.030	
						14	0.305 0.349	0.033 0.038	0.098 0.105	0.025 0.027	
						21	0.598 0.520 (0.559)	0.125 0.099 (0.112)	0.104 0.102	0.046 0.038	

^a Metabolite residues expressed in parent equivalents.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

The Meeting received information on the fate of residues of Cyflumetofen during hydrolysis and processing of oranges, apples, grapes and tomato reflecting the relevant processing procedures and household preparations.

Hydrolysis (Hassing, J, 2013a)

To simulate the degradation of cyflumetofen during pasteurization, hydrolysis of cyflumetofen was tested using benzoyl-ring-U-¹⁴C labelled and t-butylpheny-ring-U-¹⁴C labelled cyflumetofen in aqueous buffer solutions at pH 4, 5 and 6 under reflux at 90 °C for 20 min. Due to the rapid degradation of cyflumetofen under the light, each test procedure was done in the dark.

Also for simulating baking, brewing and boiling, the test was conducted similarly but under reflux at 100 °C for 60 minutes. For sterilization, the test was conducted at about 120 °C in an autoclave for 20 minutes.

Aliquots were taken right before starting a test and at the end of the test for LSC-measurements after cooling of the solution. Additionally, aliquots for HPLC-analysis were taken before and at the end of a test. Because of the photolytic and hydrolytic instability of the test item and its metabolites, no work-up for an eventual confirmation by MS-analysis could be performed. Direct MS-analysis was not possible due to the low concentration of the test item especially metabolites in the samples.

The following table demonstrates the results of hydrolysis tests using the radioactive Cyflumetofen labelled at different positions.

Table 72 Effect of hydrolysis on Cyflumetofen simulating pasteurization, baking, brewing and boiling, and sterilization

Simulated process	Hydrolysis condition			% TAR				
	pH	Temp °C	Time, min	Parent	B-1	AB-1	Others	Sum
[Benzoyl-ring-U- ¹⁴ C]-cyflumetofen								
Pasteurization	4	90	20	70.9	23.2	4.2	nd	98.3
Baking, brewing and boiling	5	100	60	17.9	58.7	31.7	nd	108.2
Sterilization	6	120	20	Nd	75.3	38.8	1.0	115.1
Simulated process	Hydrolysis condition			% TAR				
	pH	Temp °C	Time, min	Parent	A-2	AB-1	Others	Sum
[t-Butylphenyl-ring-U- ¹⁴ C]-cyflumetofen								
Pasteurization	4	90	20	69.3	14.3	Nd	15.4	99.1
Baking, brewing and boiling	5	100	60	5.0	52.9	39.9	4.3	102.2
Sterilization	6	120	20	Nd	44.4	49.1	1.8	95.2

nd =not detected

No loss of radioactivity of the both radio-labelled cyflumetofen occurred during heating in buffers of pH 4, 5 and 9 at 90–120 °C for 20 min or 60 min.

At the pH 4/90 °C condition, benzoyl-ring labelled cyflumetofen was degraded to 70.9% after 20 min. The major degradate was metabolite B-1 (23.2% TAR) followed by AB-1 (4.2% TAR). At the pH 5/100 °C condition, benzoyl-ring labelled cyflumetofen was degraded to 17.9% after 20 min. The major metabolites were B-1 (58.7% TAR) and AB-1 (31.7% TAR). At the pH 6/120 °C condition, benzoyl-ring labelled cyflumetofen was completely degraded to B-1 (75.3% TAR) and AB-1 (38.8% TAR) after 60 min.

At pH 4/90 °C, t-butylphenyl-ring labelled cyflumetofen was degraded to 68.3% TAR. The main metabolite was A-2 (14.3% TAR) with no other metabolite no more than 5% TAR. At pH 5/100 °C, t-butylphenyl-ring labelled cyflumetofen was degraded to 5.0%. The main metabolites were A-2 (52.9% TAR) and AB-1 (39.9% TAR). At pH 6/120 °C, t-butylphenyl-ring labelled cyflumetofen was completely degraded to A-2 (44.4% TAR) and AB-1 (49.1% TAR).

These results indicate that the higher the temperature of heating, the more cyflumetofen degrades and heating splits the Cyflumetofen into t-butylphenyl ring and benzoyl ring.

Oranges (Hattermann & Cowen, 2011c)

In two trials conducted in Florida and California in the USA during the 2009 growing season, 200 g/L SC formulation of cyflumetofen was applied as two foliar broadcast sprays to oranges at a target rate of 1.0 kg ai/ha (5 times GAP rate), with a 14-day re-treatment interval. The actual rate in the trials was 1.00 to 1.02 kg ai/ha, with a 14–15-day re-treatment interval. One trial had the application made in a concentrated spray volume and one in a dilute spray volume of 703 and 2457 L/ha of water,

respectively, using ground equipment. Orange samples were harvested 7 days after the last application and sent for processing into processed commodities.

Two bulk samples were sent for processing, one treated and one untreated. These bulk orange samples were separately processed (untreated first) using simulated commercial processing procedures for citrus production to generate the required fractions of unwashed fruit, peel, wet pomace, dried pulp, meal, molasses, marmalade, fresh juice and citrus oil with slight variation to the commercial methodology.

The residues of cyflumetofen, B-1, AB-6, and AB-7 in orange processed commodity samples were quantitated using Method D1003. Acceptable concurrent recovery data for orange commodities were obtained for each analyte. The LOQ was 0.01 mg/kg each for parent and the metabolites B-1, AB-6, and AB-7 in/on processed orange samples. The LOD was set at 20% of the LOQ, or 0.002 mg/kg each for parent cyflumetofen and the metabolites B-1, AB-6, and AB-7. The residue levels detected in the treated specimens and processed fractions as well as the calculated processing factors are presented in the following tables. The concentrations of metabolites were expressed in cyflumetofen equivalents.

Table 73 Residues of cyflumetofen and its metabolites in processed commodities of orange

Trial ID County, State of USA /Year	Commodity	Residues, mg eq/kg ^a			Processing factor	
		Parent	B-1	Parent + B-1 ^b	Parent	Parent + B-1
R090554 Seminole,	Orange RAC	0.664	0.028	0.692	–	–
FL / 2009	Wet Pomace	0.169	0.104	0.273	0.255	0.395
	Peel	1.899	0.078	1.977	2.86	2.86
	Dried Pulp	0.388	0.275	0.663	0.584	0.958
	Juice	< 0.01	< 0.02	< 0.03	< 0.02	< 0.04
	Oil	90.72	1.339	92.06	137	133
	Meal	0.283	0.275	0.558	0.426	0.806
	Molasses	< 0.01	0.339	< 0.349	< 0.02	< 0.504
	Marmalade	0.017	0.024	0.041	0.026	0.059
R090555 Kern, CA /	Orange RAC	0.134	0.024	0.158	–	–
2009	Wet Pomace	0.031	0.024	0.055	0.23	0.348
	Peel	0.398	0.024	0.422	2.97	2.67
	Dried Pulp	0.059	0.061	0.120	0.44	0.759
	Juice	< 0.01	0.024	< 0.034	< 0.08	< 0.22
	Oil	13.65	0.250	13.90	102	88.0
	Meal	0.061	0.061	0.122	0.46	0.77
	Molasses	< 0.01	0.042	< 0.052	< 0.08	< 0.33
	Marmalade	< 0.01	0.024	< 0.034	< 0.08	< 0.22

Trial ID County, State of USA /Year	Commodity	Residues, mg eq/kg ^a	
		AB-6	AB-7
R090554 Seminole,	Orange RAC	0.079	0.022
FL / 2009	Wet Pomace	0.008	0.012
	Peel	0.122	0.077
	Dried Pulp	0.072	0.021
	Juice	0.061	< 0.02
	Oil	2.675	3.160
	Meal	0.077	0.020
	Molasses	0.058	< 0.01
	Marmalade	0.048	0.011
R090555 Kern, CA /	Orange RAC	0.062	0.013
2009	Wet Pomace	0.005	< 0.02
	Peel	0.076	0.025

Trial ID County, State of USA /Year	Commodity	Residues, mg eq/kg ^a	
		AB-6	AB-7
	Dried Pulp	0.050	< 0.02
	Juice	0.061	< 0.02
	Oil	0.413	0.600
	Meal	0.057	< 0.02
	Molasses	0.055	< 0.02
	Marmalade	0.057	< 0.02

^a Expressed in cyflumetofen equivalents.

^b As the concentration of B-1 is sometimes higher than that of the parent, for the calculation of sum and processing factor, the LOQ value was used when the concentration is below the LOQ.

The results from these trials show that after two applications of cyflumetofen at 5× the maximum GAP rate, residues of cyflumetofen or its metabolites (B-1, AB-6 and AB-7) in the treated bulk orange RAC samples ranged from 0.013 to 0.664 mg/kg. Residues of cyflumetofen and its metabolites in processed commodities wet pomace, peel, dried pulp, juice, oil, meal, molasses and marmalade ranged from < 0.01 mg/kg (LOQ) to 2.675 mg/kg in all matrices but oil. The residues in oil from the CA test site ranged from 0.600 mg/kg for metabolite AB-6 to 90.716 mg/kg for cyflumetofen.

A comparison of the total residues of cyflumetofen and all three metabolites combined in the processed RAC with those in each of the processed matrix samples showed that the total residue concentrated in peel and oil only (2.2× to 2.7× and 64× to 123×, respectively). Residue in all the other processed matrices did not concentrate and was diluted to concentrations less than the RAC samples collected before processing.

Apples (White, 2011b)

In two trials conducted in NY and PA during the 2009 growing season, 200 g/L SC formulation of cyflumetofen was applied as two airblast foliar sprays to apples at a rate of 0.996–1.027 kg ai/ha (5× the maximum GAP rate), with a 14 day retreatment interval. The first application was made approximately 3 weeks (21 days) prior to the harvest of fruit, when fruit were ripening (growth stage BBCH 79 to BBCH 81). The applications were made in 606–872 L/ha of water using ground equipment. Apple bulk RAC samples were harvested 7 days after the last application. The two bulk apple RAC samples were separately processed using simulated commercial processing procedures into wet pomace and juice. In addition, apple sauce, canned apples, and dried apples were collected for analysis.

The residues of cyflumetofen and metabolites B-1, AB-6 and AB-7 in apple RAC and processed commodity samples were quantitated using Method D1003. Acceptable concurrent recovery data for apple commodities were obtained for each analyte. The LOQ was 0.01 mg/kg for each analyte. Apparent residues of cyflumetofen, B-1, AB-6 and AB-7 were < LOQ in/on all untreated apple RAC and processed commodities.

Table 74 Residues of cyflumetofen and its metabolites in processed commodities of apple.

Trial ID County, State of USA /Year	Commodity	Residues (mg/kg) ^a			Processing Factor	
		Parent	B-1	Parent + B-1 ^b	Parent	Parent + B-1
RCN R090509	Apple RAC	0.584	< 0.02	0.604	–	–
Wayne, NY	Wet pomace	0.547	< 0.02	0.567	0.937	0.939
/ 2009	Applesauce	1.700	0.554	2.254	2.91	3.73
	Juice	0.115	< 0.02	0.135	0.197	0.224
	Dried apples	0.482	0.047	0.529	0.825	0.876
	Canned apples	0.102	< 0.02	0.122	0.175	0.202
RCN R090510	Apple RAC	0.451	< 0.02	0.471	–	–
LeHigh, PA	Wet pomace	0.719	< 0.02	0.739	1.59	1.57
/ 2009	Applesauce	1.146	0.342	1.488	2.54	3.16
	Juice	0.121	< 0.02	0.141	0.268	0.299

Trial ID County, State of USA /Year	Commodity	Residues (mg/kg) ^a			Processing Factor	
		Parent	B-1	Parent + B-1 ^b	Parent	Parent + B-1
	Dried apples	0.077	< 0.02	0.097	0.17	0.21
	Canned apples	0.016	< 0.02	0.036	0.035	0.076

Trial ID County, State of USA /Year	Commodity	Residues (mg/kg) ^a	
		AB-6	AB-7
RCN R090509	Apple RAC	0.013	0.010
Wayne, NY	Wet pomace	0.022	0.016
/ 2009	Applesauce	0.055	< 0.01
	Juice	< 0.01	< 0.01
	Dried apples	0.010	< 0.01
	Canned apples	< 0.01	< 0.01
RCN R090510	Apple RAC	0.010	0.010
LeHigh, PA	Wet pomace	0.015	0.013
/ 2009	Applesauce	0.039	< 0.01
	Juice	< 0.01	< 0.01
	Dried apples	< 0.01	< 0.01
	Canned apples	< 0.01	< 0.01

^a Expressed in cyflumetofen equivalents.

^b As the concentration of B-1 is sometimes higher than that of the parent, for the calculation of sum and processing factor, the LOQ value was used when the concentration is below the LOQ.

The results from these trials show that after two applications of cyflumetofen targeting 2.0 kg as/ha/season, mean parent cyflumetofen residues in the treated apple RAC samples collected 7 days after the last application were 0.58 mg/kg and 0.45 mg/kg. For the individual analytes, expressed in parent equivalents, residues of the metabolites B-1, AB-6, and AB-7 were < 0.02 mg/kg, 0.01 mg/kg, and 0.01 mg/kg, respectively, in the treated apple RAC samples collected 7 days after the last application.

A comparison of the residues in the RAC with those in each processed fraction indicate that parent cyflumetofen residues concentrate in apple sauce (2.5–2.9×, average processing factor 2.7×), but do not concentrate appreciably in any other apple processed commodities, wet pomace, juice, dried apples, or canned apples.

Grapes (Hattermann & Lott, 2011b)

In four trials conducted in NY, CA and OR during the 2009 growing season, 200 g/L SC formulation of cyflumetofen was applied as two foliar broadcast sprays to grape at a target rate of 1.0 kg ai/ha (5× the maximum GAP rate), with a 14-day re-treatment interval (13-day interval at one CA site). The actual application rates were 1.99 to 2.02 kg ai/ha. The applications were made in 465–2600 L/ha of water using ground equipment. Two trials had product applied in a dilute application (935.4–3741.6 L/ha) and two in a concentrate spray volume (187.1–935.4 L/ha range). Grape samples were harvested 14 days after the last application except for one CA trial which was harvested at 15-day after the final application. One treated and one non-treated bulk sample was processed for each of four trials. Dried grapes, dried grape stems, juice, wine (young), wet pomace, must, yeast and grape stems were collected for analysis. The study was planned to include two trials, each performed on red and white grape varieties. However, an exception to protocol occurred when a white grape variety was used at the King County, CA trial instead of red. The white grapes from this trial were processed using the red grape procedure for wine, such that there were two trials processed with red and two with white grape procedures.

The residues of cyflumetofen, B-1, AB-6, and AB-7 in grape RAC and processed commodity samples were quantitated using Method D1003. Acceptable concurrent recovery data for grape commodities were obtained for each analyte. The LOQ was 0.01 mg/kg each for parent cyflumetofen and the metabolites B-1, AB-6, and AB-7 in/on grape RAC and processed commodity samples. The

LOD was set at 20% of the LOQ, or 0.002 mg/kg each for parent cyflumetofen and the metabolites B-1, AB-6 and AB-7.

Table 75 Residues of cyflumetofen and its metabolites in processed commodities of grapes

Trial ID County, State of USA /Year	Commodity	Residues (mg/kg) ^a			Processing Factor	
		Parent	B-1	Parent + B-1 ^b	Parent	Parent + B-1
RCN R090550	Grape RAC	1.71	0.07	1.78	–	–
Yates, NY	Dried grape	7.94	0.33	8.27	4.64	4.65
/ 2009	Dried grape Stem	23.88, 18.96, 17.63	0.56	20.72	11.8	11.6
	Juice	0.11	0.07	0.18	0.064	0.10
	Wine (Young)	< 0.01	0.07	< 0.08	< 0.006	0.04
	Wet Pomace	5.80	0.14	5.94	3.39	3.34
	Must	0.03	0.05	0.08	0.02	0.04
	Yeast	2.34	1.37	3.71	1.37	2.08
	Grape Stems	4.21	0.21	4.42	2.46	2.48
RCN R090551	Grape RAC	0.34	< 0.02	0.36	–	–
Tulare, CA	Dried grape	0.22	0.09	0.31	0.65	0.86
/ 2009	Dried grape Stem	3.07, 1.51, 1.73	0.38	2.48	6.16	6.89
	Juice	0.08	< 0.02	0.10	0.2	0.28
	Wine (Young)	< 0.01	0.02	< 0.03	0.029	0.083
	Wet Pomace	0.37	0.02	0.39	1.1	1.1
	Must	0.14	< 0.02	0.16	0.412	0.44
	Yeast	2.23	0.73	2.96	6.56	8.22
	Grape Stems	0.34	0.05	0.39	1.0	1.1
RCN R090552	Grape RAC	0.57	0.02	0.59	–	–
Kings, CA	Dried grape	0.53	0.09	0.62	0.93	1.1
/ 2009	Dried grape Stem	14.79, 6.63, 6.90	1.01	10.45	16.4	17.7
	Juice	0.14	0.05	0.19	0.25	0.32
	Wine (Young)	0.02	0.07	0.09	0.04	0.2
	Wet Pomace	1.72	0.07	1.79	3.02	3.03
	Must	0.30	0.05	0.35	0.53	0.59
	Yeast	0.31	0.54	0.85	0.54	1.4
	Grape Stems	1.72	0.14	1.86	3.02	3.15
RCN R090553	Grape RAC	1.37	0.12	1.49	–	–
Benton,	Dried grape	2.57	1.11	3.68	1.88	2.47
OR / 2009	Dried grape Stem	13.46, 10.08, 8.33	1.20	11.82	7.75	7.93
	Juice	0.15	0.09	0.24	0.11	0.16
	Wine (Young)	0.06	0.19	0.25	0.04	0.17
	Wet Pomace	5.78	0.14	5.92	4.22	3.97
	Must	0.25	0.07	0.32	0.18	0.22
	Yeast	14.63	0.99	15.62	10.7	10.5
	Grape Stems	3.23	0.33	3.56	2.36	2.39

Trial ID County, State of USA /Year	Commodity	Residues (mg/kg) ^a	
		AB-6	AB-7
RCN R090550	Grape RAC	0.03	0.02
Yates, NY	Dried grape	0.11	0.07
/ 2009	Dried grape Stem	0.19	0.12
	Juice	< 0.01	< 0.01
	Wine (Young)	< 0.01	< 0.01
	Wet Pomace	0.04	0.03
	Must	< 0.01	< 0.01
	Yeast	0.01	< 0.01

Trial ID County, State of USA /Year	Commodity	Residues (mg/kg) ^a	
		AB-6	AB-7
	Grape Stems	0.03	0.01
RCN R090551	Grape RAC	0.02	0.02
Tulare, CA	Dried grape	0.02	0.02
/ 2009	Dried grape Stem	0.07	0.10
	Juice	< 0.01	< 0.01
	Wine (Young)	< 0.01	< 0.01
	Wet Pomace	0.02	0.03
	Must	< 0.01	< 0.01
	Yeast	0.07	0.11
	Grape Stems	0.02	0.02
RCN R090552	Grape RAC	< 0.01	< 0.01
Kings, CA	Dried grape	< 0.01	< 0.01
/ 2009	Dried grape Stem	0.17	0.10
	Juice	< 0.01	< 0.01
	Wine (Young)	< 0.01	< 0.01
	Wet Pomace	0.02	0.02
	Must	< 0.01	< 0.01
	Yeast	0.01	0.01
	Grape Stems	0.02	0.02
RCN R090553	Grape RAC	0.15	0.12
Benton,	Dried grape	0.29	0.19
OR / 2009	Dried grape Stem	0.42	0.30
	Juice	< 0.01	< 0.01
	Wine (Young)	< 0.01	< 0.01
	Wet Pomace	0.55	0.42
	Must	< 0.01	< 0.01
	Yeast	0.11	0.10
	Grape Stems	0.10	0.08

^a Expressed in cyflumetofen equivalents.

^b As the concentration of B-1 is sometimes higher than that of the parent, for the calculation of sum and processing factor, the LOQ value was used when the concentration is below the LOQ.

The results from these trials show that after two applications of cyflumetofen, targeting 1.0 kg ai/ha per application, residues of cyflumetofen, B-1, AB-6 or AB-7 (expressed in parent equivalents) in the treated grape RAC samples (whole fruit) collected 14–15 days after the last application ranged from < 0.01–1.71 mg/kg for cyflumetofen or its metabolites.

RAC grape samples showed measurable levels of parent cyflumetofen and the three metabolites. A comparison of the residues in the treated grape RAC samples with those in each processed fraction indicated that combined residues of cyflumetofen and its metabolites concentrated in dried grape at 2.37 and 4.62× at the OR and NY test sites, respectively, and in dried grape stem (6.63–17.6×), wet pomace (1.10–3.91×), yeast (1.43–8.99×), and grape stems (1.08–3.11×) at all four test sites. The compound did not concentrate in any of the other processed grape matrices and was actually diluted in these matrices (i.e. concentration factors less than or equal to 1×).

Tomato (Crawford, 2011b)

In two trials conducted in NY and CA during the 2009 growing season, 200 g/L SC formulation of cyflumetofen was applied as two foliar broadcast sprays to tomato at a rate of 1.00–1.01 kg ai/ha, with a 14-day retreatment interval, resulting in a total exaggerated rate (5×) of 2.01–2.02 kg ai/ha. The applications were made in 187–360 L/ha using ground equipment, and an adjuvant was added to the spray mixture for all applications. Tomato samples were harvested 3 days after the last application. A total of two separate treated bulk samples were processed for a total of two processing tests. The treated bulk tomato RAC samples were separately processed using simulated commercial processing procedures into paste and puree. In addition, canned tomatoes, peeled tomatoes, tomato juice, tomato peels, wash water, washed tomatoes, and wet pomace were collected for analysis.

The residues of cyflumetofen and metabolites B-1, AB-6, and AB-7 in tomato processed commodity samples were quantitated using Method D1003. Acceptable concurrent recovery data for tomato commodities were obtained for each analyte. The LOQ was 0.01 mg/kg each for parent and the metabolites B-1, AB-6, and AB-7 in/on tomato processed commodity samples. The LOD was set at 20% of the LOQ, or 0.002 mg/kg each for parent cyflumetofen and the metabolites B-1, AB-6, and AB-7.

Table 76 Residues of cyflumetofen and its metabolites in processed commodities of tomato

Trial ID County, State of USA /Year	Commodity	Residues (mg/kg) ^a			Processing Factor	
		Parent	B-1	Parent + B-1 ^b	Parent	Parent + B-1
RCN R090548	Tomato RAC	0.24	0.05	0.29	—	—
Wayne, NY / 2009	Washed Tomatoes	0.26	0.05	0.31	1.1	1.1
	Peeled Tomatoes	< 0.01	< 0.02	< 0.03	< 0.04	< 0.1
	Canned Tomatoes	< 0.01	0.02	< 0.03	< 0.04	< 0.1
	Puree	0.21	0.02	0.23	0.88	0.79
	Paste	0.09	0.54	0.63	0.4	2.2
	Wet Pomace	0.32	0.08	0.40	1.3	1.4
	Tomato Peels	4.05	0.08	4.13	16.9	14.2
	Wash Water	0.69	0.09	0.78	2.9	2.7
	Tomato Juice	0.04	0.07	0.11	0.2	0.38
	Tomato RAC	0.17	0.02	0.19	—	—
Kings, CA / 2009	Washed Tomatoes	0.11	0.02	0.13	0.65	0.68
	Peeled Tomatoes	< 0.01	< 0.02	< 0.03	< 0.06	< 0.2
	Canned Tomatoes	0.03	0.02	0.05	0.2	0.3
	Puree	0.05	< 0.02	0.07	0.3	0.4
	Paste	0.04	0.19	0.23	0.2	1.2
	Wet Pomace	0.93	0.02	0.95	5.5	5.0
	Tomato Peels	1.52	0.02	1.54	8.94	8.10
	Wash Water	0.02	< 0.02	0.04	0.1	0.2
	Tomato Juice	< 0.01	0.02	< 0.03	< 0.06	< 0.2
	Tomato RAC	0.17	0.02	0.19	—	—

Trial ID County, State of USA /Year	Commodity	Residues (mg/kg) ^a	
		AB-6	AB-7
RCN R090548	Tomato RAC	0.05	0.08
Wayne, NY / 2009	Washed Tomatoes	0.06	0.11
	Peeled Tomatoes	< 0.01	< 0.01
	Canned Tomatoes	< 0.01	< 0.01
	Puree	0.05	0.08
	Paste	0.07	0.17
	Wet Pomace	0.06	0.11
	Tomato Peels	0.73	1.46
	Wash Water	0.01	0.01
	Tomato Juice	< 0.01	0.01
	Tomato RAC	< 0.01	0.01
Kings, CA / 2009	Washed Tomatoes	< 0.01	0.01
	Peeled Tomatoes	< 0.01	< 0.01
	Canned Tomatoes	< 0.01	< 0.01
	Puree	< 0.01	< 0.01
	Paste	< 0.01	< 0.01
	Wet Pomace	0.03	0.05
	Tomato Peels	0.06	0.10
	Wash Water	< 0.01	< 0.01
	Tomato Juice	< 0.01	< 0.01
	Tomato RAC	< 0.01	0.01

^a Expressed in cyflumetofen equivalents.

^b As the concentration of B-1 is sometimes higher than that of the parent, for the calculation of sum and processing factor, the LOQ value was used when the concentration is below the LOQ.

The results from these trials show that after two applications of cyflumetofen targeting 1.0 kg ai/ha to each field trial to obtain 5× the maximum GAP per-season rate of 0.400 kg ai/ha, residues of cyflumetofen (metabolites B-1, AB-6 and AB-7 expressed in parent equivalents) in the treated tomato RAC samples (whole fruit) sampled 3 days PHI were < LOQ to 0.24 mg/kg. For the processed commodity samples, parent residues in the treated fractions from fruit sampled 3 days PHI were < LOQ to 4.05 mg/kg; residues of the metabolites B-1, AB-6, and AB-7 ranged from < LOQ to 1.46 mg/kg.

A comparison of the residues in the RAC tomato with those in each processed fraction indicated that combined residues of cyflumetofen concentrated in wash water (1.9×) at the New York site only and in paste (1.2 to 2.1×), wet pomace (1.3 to 4.2×) and tomato peel (8.1 to 15.0×) at both test sites. The compound did not concentrate in any other processed tomato fractions.

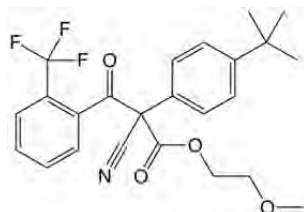
RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

No information on farm animal feeding studies were received by the current Meeting.

APPRAISAL

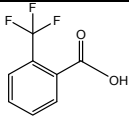
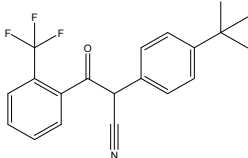
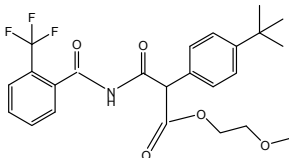
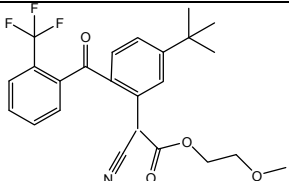
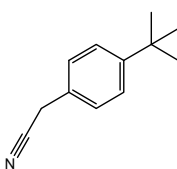
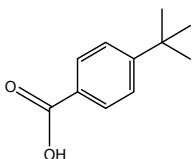
Cyflumetofen (consisting of RS isomers) is a bridged diphenyl acaricide (miticide) for control of *Tetranychus* sp. and influences the mitochondrial electron transport chain by inhibiting the complex II substance in the cell. It has been registered in a number of countries.



The Meeting received information on physical and chemical properties, animal and plant metabolism, environmental fate, analytical methods, storage stability, use patterns, supervised trials, and processing. Cyflumetofen was scheduled by the Forty-fifth Session of the CCPR for review by the 2014 JMPR for the first time.

In this Appraisal, the following abbreviated names were used for referred metabolites.

Code (MW)	IUPAC name	Structure	Metabolite or degradate found in
Cyflumetofen (447.45)	2-methoxyethyl (RS)-2-(4-tert-butylphenyl)-2-cyano-3-oxo-3-(α,α,α -trifluoro- <i>o</i> -tolyl)propionate		Goat Satsuma mandarin Apple Eggplant
B-1 (190.12)	2-(trifluoromethyl)benzoic acid		Rat

			Goat Satsuma mandarin Apple Eggplant Soil
AB-1 (345.36) Syn: M9210I003	(RS)-2-(4-tert-butylphenyl)-3-oxo-3-[2-(trifluoromethyl)phenyl]propanenitrile		Rat Goat Hydrolysis in buffer Hydrolysis simulating processing Soil
AB-6 (465.45)	2-methoxyethyl-2-((R,S)-4-tert-butylphenyl)-3-oxo-3-({[2-(trifluoromethyl)phenyl]carbonyl}amino)propanoate		Satsuma mandarin Apple Eggplant
AB-7 (447.45)	2-methoxyethyl-((R,S)-4-tert-butyl-2-{{[2-(trifluoromethyl)phenyl]carbonyl}phenyl}(cyano)acetate		Satsuma mandarin Apple Eggplant
A-2 (173.26) Syn: M9210I001	4-tert-butylphenyl-acetonitrile		Goat Soil
A-12 (178.23) Syn: M9210I002	4-tert-butylbenzoic acid		Goat Soil

Animal metabolism

The Meeting received information on the fate of orally-dosed cyflumetofen in lactating goats.

In metabolism studies, total radioactive residues are expressed in mg eq./kg cyflumetofen equivalents unless otherwise stated.

Metabolism of cyflumetofen in rat

Metabolism studies on laboratory animals including rats were reviewed in the framework of toxicological evaluation by the current JMPR.

In a rat metabolism study, highest residues were found in the liver followed by kidney regardless of sex, dose and label position and time point of measurement. Cyflumetofen was extensively metabolized. B-1, 2-trifluoromethylbenzoic acid, was the major metabolite (occurring up to 28% of the applied dose). The predominant metabolic pathway for cyflumetofen involves cleavage of the tert-butylphenyl and trifluorotolyl moieties. Major reactions on the tert-butylphenyl ring are cleavage of the methoxyethyl group, hydroxylation at the butyl group, decarboxylation and glucuronidation at the butyl group. Major reactions on the trifluorotolyl-ring are glutathione conjugation at the carboxyl group and further changes of the glutathione group to mercapturic acid or thiolactic acid. In addition, hydroxylation and oxidation reactions at the butyl group and cleavage of the carboxylic ester moiety are observed on the parent molecule.

Metabolism of cyflumetofen in lactating goat

Two lactating goats were orally administered [benzoyl-ring- U - ^{14}C]-cyflumetofen (hereafter abbreviated as benzoyl-label) for 12 consecutive days at 0.43 or 0.48 mg/kg bw (13 or 12 ppm in feed). Other two goats were orally administered [t-butylphenyl-ring- U - ^{14}C]-cyflumetofen (hereafter abbreviated as butylphenyl-label) for 10 consecutive days at 0.27 or 0.30 mg/kg bw (15 or 12 ppm in feed). The goats were sacrificed 18–24 hours after the last dose.

After radio-labelled cyflumetofen was administered orally to lactating goats, it was excreted in faeces and urine (total > 78% excreted during the testing periods) and only a small portion accounting for < 0.3% of the administered radioactivity (AR) remained in body tissues/organs. Radioactive residues were the highest in liver (0.29–0.40 mg eq./kg) followed by kidney (0.17–0.19 mg eq./kg) but low in fat (0.028–0.033 mg eq./kg) and muscle (0.009–0.020 mg eq./kg). Milk of each day contained $\leq 0.02\%$ AR and milk collected throughout the study period contained, in total, 0.008–0.19 mg eq./kg (0.03–0.14% AR).

The parent compound, cyflumetofen, was found only in fat but at low concentration of 0.003 mg/kg accounting for 20–21% of the total radioactive residues (TRR). The predominant metabolite in all tissues/organs and milk was B-1 at around 0.1 mg eq./kg in liver (32% TRR) and kidney (54% TRR), and lower than 0.01 mg eq./kg in muscle (46–51% TRR), fat (21–40% TRR) and milk (4.5% TRR).

The metabolism of cyflumetofen in goat involves extensive hydrolysis of formic acid ester and trifluoromethylbenzoyl moiety, decarboxylation, conjugation, hydroxylation and oxidation. In principle, excretion and distribution of cyflumetofen and its metabolism in goat are similar to those in rats with some differences in metabolites identified.

Plant metabolism

Satsuma mandarin

When Satsuma mandarin trees (grown outdoor) were sprayed with benzoyl- or butylphenyl-label cyflumetofen at a rate approximating 0.60 kg ai/ha (3 $\times$ GAP rate of the USA), and Satsuma mandarin fruit samples were collected 1, 7 and 30 days after the treatment, the majority of the radioactivity was recovered from the acetonitrile surface rinse of fruits: 95–96% TRR on 1DAT; 91–93% TRR on 7DAT; and 88–89% on 30 DAT.

The predominant radioactive residue on/in mandarin fruits was cyflumetofen at 0.52–0.55 mg/kg (88–90% TRR) on 1DAT, decreasing to 0.33–0.37 mg/kg (79–83% TRR) on 7 DAT and further to 0.25–0.31 mg/kg (44–54% TRR) on 30 DAT.

Other than the parent, metabolites AB-6, AB-7, A-12 and B-1 were formed. B-1 increased over time from 0.028 mg eq./kg (4.7% TRR) on 1 DAT to 0.064 mg eq./kg (11% TRR) on 30 DAT. None of AB-6, AB-7 and A-12 exceeded 10% TRR. A number of unknown peaks were observed but none of them exceeded 10% of TRR.

The radioactive residues on/in leaves showed similar profile as those on/in fruits with the majority (>87% TRR) of radioactive residues found in the surface rinse, although the TRR was >50

times that on/in fruits. A majority of radioactive residues remaining in leaves were extracted. Cyflumetofen was also the predominant identified fraction in leaves: >73% TRR in rinse and extract together. Four metabolites were identified but none exceeded 10% TRR.

Apple

When an apple tree (grown outdoor) was sprayed with benzoyl- or butylphenyl-label cyflumetofen at a rate approximating 0.60 kg ai/ha (3X GAP rate of the USA), and apple fruit samples were collected 1, 7 and 30 days after the treatment, the majority of the radioactivity was recovered from the acetonitrile surface rinse of fruits: 95–96% TRR on 1 DAT, 82–89% TRR on 7 DAT; and 67–71% TRR on 30 DAT.

The predominant radioactive residue on/in apple fruits was cyflumetofen at 0.061–0.066 mg/kg (58–61% TRR) in rinse while pulp extract contained too little radioactivity for further analysis on 1DAT, 0.061–0.14 mg/kg (78–84% TRR) on 7 DAT and decreased to 0.037–0.042 mg/kg (53–65% TRR) on 30 DAT.

There were minor metabolites identified. However, none of them exceeded 5% TRR.

The radioactive residues on/in leaves showed similar profile as those on/in fruits with the majority (>72% TRR) of radioactive residues found in the surface rinse, although the TRR was >50 times that on/in fruits. Cyflumetofen was also the predominant identified fraction in leaves: >44% TRR in rinse and extract together. No metabolites exceeded 10% TRR.

Eggplant

When eggplants (grown outdoor) were sprayed with benzoyl- or butylphenyl-label cyflumetofen at a rate approximating 0.60 kg ai/ha (3X GAP rate of the USA), and eggplant samples were collected 1, 7 and 14 days after the treatment, the majority of the radioactivity was also recovered from the acetonitrile surface rinse of fruits: 87–92% TRR on 1 DAT, 79–86% TRR on 7 DAT; and 56–81% TRR on 30 DAT.

The predominant radioactive residue on/in eggplant fruits was also cyflumetofen accounting for 0.31–0.44 mg/kg (91–95% TRR) on 1 DAT, decreased to 0.25–0.39 mg/kg (67–71% TRR) on 7 DAT and then to 0.18–0.20 mg/kg (42–62% TRR) on 14 DAT.

Metabolites B-1, AB-6 and AB-7 were identified from the fruit rinse and/or extracts. B-1 was found at 0.059 mg eq./kg (11% TRR) on 7 DAT and at 0.061 mg eq./kg (15% TRR) on 14 DAT on/in fruits. AB-6 and AB-7 did not exceed 10% TRR.

The tentatively identified U1/U2 (likely to be acid labile conjugates of B-1) and U4 were also found in 7 DAT and 14 DAT fruits but not in 1 DAT fruits. U1 was present at 0.067 mg eq./kg (16% TRR) on 14 DAT but <10% TRR on 1 DAT and 7 DAT. No other metabolites exceeded 10% TRR. U1 and U2 were found only in the fruit extracts but not in rinse, indicating that they were formed in the fruits.

The radioactive residues on/in leaves showed similar profile as those on/in fruits with the majority (>69% TRR) of radioactive residues found in the surface rinse, although the TRR was >50 times that on/in fruits. Cyflumetofen was also the predominant identified fraction in leaves: >47% TRR in rinse and extract together. No metabolites exceeded 10% TRR.

Summary of plant metabolism

The metabolism of cyflumetofen in these crops involves hydrolysis, acyl migration, oxidation and conjugation (U1 and U2 are the conjugate of B-1).

The plant metabolism studies on Satsuma mandarin, apple and eggplant showed similar pattern. Applied radioactively was mostly recovered from surface rinse with decreasing trend over time. Fruit flesh contained far less radioactivity. The predominant radioactive residue was cyflumetofen accounting for >42% TRR at all time points. The next important radioactive residue was

B-1 but at the maximum 15% TRR (around 0.06 mg eq./kg). B-1 may be present in fruits in conjugated forms.

Environmental fate

Aerobic soil metabolism

The studies on aerobic soil degradation of cyflumetofen indicate that cyflumetofen sprayed on soil was rapidly and completely degraded with DT50 around 1.8–4.3 days in various soils at 20–25°C. Additionally, aerobic soil degradation of B-1 and AB-1 was studied resulting in DT50 of 6–36 days and 0.07–0.11 days respectively at 20°C.

Photolysis on soil surface

Photolysis of cyflumetofen on soil was found to be insignificant as the degradation rates of cyflumetofen with and without light irradiation were not significantly different.

Hydrolysis in aquatic system

Cyflumetofen is susceptible to hydrolysis and was hydrolysed faster at higher pH in aqueous buffer solutions at 25°C. DT50 was calculated using first order kinetics to be 7.7 days at pH 4, 6.0 days at pH 5, 9.8 h at pH 7 and 10.3 min at pH 9.

At slightly acidic to neutral pH, a number of degradates exceeded 10% of the applied dose: A-1 (max. 14% at 8 h), A-2 (max. 44% at 720 h), A-18 (max. 36% at 120 h), B-1 (max. 53% at 48 h) and AB-1 (max. 45%, at 120 h).

Residues in succeeding crops

A confined rotational crop study was conducted to examine the nature and level of residues of cyflumetofen in three succeeding crops (white radish, lettuce and wheat) using benzoyl- and butylphenyl-label cyflumetofen. A single application of radio-labelled cyflumetofen was made on bare soil in plastic containers at a rate of 0.40 kg ai/ha (2x GAP rate of the USA). After plant back interval (PBI) of 30, 120 and 365 days, lettuce, white radish and spring wheat were sown into the treated soil.

Following the application of cyflumetofen to soil, uptake of radioactivity from soil into rotational crops was observed, in particular, with benzoyl-label (up to 1.25 mg eq./kg in wheat chaff of 30 PBI but much lower in edible portions with the maximum of 0.17 mg eq./kg in wheat grain of 30 PBI). With butylphenyl-label, uptake of radioactivity into rotational crops were lower (maximum of 0.11 mg eq./kg in wheat straw+chaff of 120 PBI). Uptake was, in general, less with longer plant back interval.

The only major radioactive residue identified from benzoyl-label treatment was trifluoroacetic acid in all crops (39–100% TRR). B-1, specific to this label, was detected as a minor component.

With the butylphenyl-label, a series of label-specific metabolites were observed in the extracts but all of them were less than 0.01 mg eq./kg.

No parent compound was detected from any of the crop extracts.

No significant residues of cyflumetofen, other than trifluoroacetic acid, were expected to be found in rotational crops on the basis of aerobic soil degradation studies and confined succeeding crop study.

Methods of analysis

Analytical methods for determination of residues of cyflumetofen and its metabolites B-1, AB-6 and AB-7 were developed for a wide range of matrices of plant and animal origin.

In general, the method for data generation and enforcement for plant matrices employ extraction by shaking with acetonitrile and then a mixture of acetonitrile:water (75:25, v/v) or only with acetonitrile:water, clean-up by partitioning with a mixture of ethyl acetate:cyclohexane (75:25, v/v) and determination of the analytes using LC-MS/MS.

The method for plant matrices was validated for cyflumetofen, B-1, AB-6 and AB-7 resulting in acceptable recoveries and relative standard deviations (RSDs) with the LOQ of 0.01 mg/kg for various plant matrices.

The analytical method developed for cyflumetofen and B-1 in animal matrices was similar to the one for plant matrices but employs acetonitrile:water (50:50, v/v) for extraction instead of acetonitrile:water (75:25, v/v) and different clean-up procedure. This method was validated for cyflumetofen and B-1 resulting in acceptable recoveries and RSDs with the LOQ of 0.01 mg/kg for bovine liver, and meat and 0.001 mg/kg for bovine milk. The mean recoveries for cyflumetofen in poultry eggs were low at 65–67% (RSD, 3–6%) while those for B-1 were acceptable between 86–95%.

A number of scientific papers report the validation of the QuEChERS multi-residue method with GC-MS/MS for cyflumetofen in various plant commodities with the LOQ around 0.01 mg/kg.

Stability of pesticide residues in stored analytical samples

The stability of cyflumetofen, B-1, AB-6 and AB-7 in homogenates of almond, apple (fruit, juice) kidney bean, lettuce, orange (fruit, juice, oil), radish and wheat grain at -20 to -10 °C was tested at a spike level (each analyte separately spiked) of 0.1 mg/kg for 743–910 days (24–30 months).

Cyflumetofen was stable when stored frozen for 25 months, the longest storage time tested, in almond, apple fruit, apple juice, orange fruit (24 months), orange juice, orange oil and wheat grain. However, it was stable only up to 9 months in kidney bean and lettuce, and 3 months in radish root.

B-1 was stable when stored frozen for 30 months, the longest storage time tested, in almond, kidney bean, lettuce, orange fruit, orange juice and wheat grain. It was stable up to 22 months in apple fruit, apple juice and radish root, and 6 months in orange oil.

AB-6 was stable when stored frozen for 28 months, the longest storage time tested, in almond, apple fruit, kidney bean, orange juice, orange oil, radish root, and wheat grain. It was stable up to 21 months in apple juice, lettuce and orange fruit.

AB-7 was stable when stored frozen for 26 months, the longest storage time tested, in almond, apple fruit, apple juice, kidney bean, orange juice, and orange oil. It is stable up to 19 months in orange fruit and wheat grain, 12 months in lettuce, but one month in radish root.

Definition of the residue

In goat metabolism studies, radioactive residues were highest in liver followed by kidney. Far less radioactive residues were found in fat, muscle and milk.

When benzoyl-label cyflumetofen was administered, metabolite B-1 (2-trifluoromethylbenzoic acid) was the predominant residue in all edible tissues/organs (21–54% TRR) and milk (4.5% TRR). B-1 was also found in rat metabolism and is considered to be no more toxic than the parent and, therefore, toxicologically covered by the ADI for parent compound. Cyflumetofen was detected only in fat at 0.003 mg/kg (20% TRR). No other metabolites were detected above 10% TRR.

When butylphenyl-label cyflumetofen was administered, cyflumetofen was not detected in any of edible tissues/organs or in milk. None of all the identified metabolites exceeded 0.01 mg eq./kg and 10% TRR.

There is a validated LC-MS/MS method for cyflumetofen and B-1 in bovine matrices. The Meeting considered that both cyflumetofen and B-1 were suitable residues for enforcement of MRLs and for estimating dietary intake.

LogP_{ow} of cyflumetofen is 4.3 at 25 °C and cyflumetofen was found only in the fat at < 0.01 mg/kg in the goat metabolism study. B-1 is a carboxylic acid and was present at 0.13 mg eq./kg in liver and 0.10 mg eq./kg in kidney but < 0.01 mg eq./kg in fat. The Meeting considered that, over all, residues (cyflumetofen and B-1) were not fat soluble.

In the plant metabolism studies on Satsuma mandarin, apple and eggplant, cyflumetofen was the predominant residue and accounted for > 42% TRR in the fruits of these crops at any time points. The Meeting considered that for enforcement of MRLs, cyflumetofen was a suitable residue.

The next important radioactive residue was B-1 but at the maximum 15% TRR (around 0.06 mg eq./kg). In the supervised residue trials, the concentrations of B-1 were mostly 1/10–1/2 of those of cyflumetofen. Conjugated forms of B-1 (U1 and U2) were found in the eggplant metabolism at slightly higher concentrations (in total) than B-1 itself but they were not found in the studies on Satsuma mandarin or apple. A validated LC-MS/MS method is available and used in the trials for quantification of cyflumetofen and B-1 but it determines the free form of B-1.

The Meeting considered that, for calculating dietary intake from commodities of plant origin, cyflumetofen and B-1 were suitable residues.

Based on the above, the Meeting recommended the following residue definition for plant and animal commodities:

Definition of the residue for plant commodities (for compliance with the MRL): *Cyflumetofen*.

Definition of the residue for plant commodities (for estimation of dietary intake): *Sum of cyflumetofen and 2-trifluoromethylbenzoic acid, expressed as cyflumetofen*.

Definition of the residue for animal commodities (for compliance with the MRL and estimation of dietary intake): *Sum of cyflumetofen and 2-trifluoromethylbenzoic acid, expressed as cyflumetofen*.

Residue is not fat-soluble.

Results of supervised residue trials on crops

The Meeting received supervised trial data for cyflumetofen on citrus fruits, pome fruits, grapes, strawberry, tomato, eggplant and tree nuts conducted outdoor (except trials on eggplant in Japan) using foliar spray of 20% SC formulation of cyflumetofen (except in a trial on apple in Italy).

Residues were expressed in cyflumetofen equivalents. The analytical results for B-1 were converted to cyflumetofen equivalents by multiplying the analytical results for B-1 with a factor of 2.35 based on the molecular weights of cyflumetofen (447.45) and B-1 (190.12).

For the estimation of a sum of cyflumetofen and B-1, where B-1 was below the LOQ, it was regarded as 0.02 mg eq./kg (LOQ of 0.01 mg/kg for B-1 is converted to 0.02 mg eq./kg) as B-1 was sometimes present at concentrations comparable to those of parent in trials.

Although in a number of trials conducted in the USA the water volume was smaller than the minimum water volume on the label, the Meeting agreed to use the results of these trials in estimating maximum residue levels as the purpose of the minimum requirement was for better efficacy and not for safety, and residues from lower water volume trials were not always lower than those from higher water volume trials.

Citrus fruits

A total of 23 supervised trials were conducted on orange (12), grapefruit (6) and lemon (5) in the USA in 2009 and 2010. GAP in the USA for citrus fruits allows 2 applications at a maximum rate of 0.2 kg ai/ha (at least 935 L/ha) with a PHI of 7 days.

Residues of cyflumetofen from 11 independent orange trials matching GAP for citrus fruits in the USA were: 0.01, 0.01, 0.04, 0.06, 0.06, 0.08, 0.09, 0.10, 0.10, 0.11 and 0.12 mg/kg (median 0.08 mg/kg).

The information on four trials conducted on orange in Brazil in 2007 was also provided but they did not match the GAP in Brazil (one application at a spray concentration of 0.08 kg ai/hL).

Residues of cyflumetofen from six grapefruit trials matching GAP for citrus fruits in the USA were: < 0.01, 0.02, 0.04, 0.04, 0.04 and 0.07 mg/kg (median 0.04 mg/kg).

Residues of cyflumetofen from five lemon trials matching GAP for citrus fruits in the USA were: < 0.01, 0.02, 0.02, 0.08 and 0.14 mg/kg (median 0.02 mg/kg).

As the GAP in the USA is established for the group of citrus fruits, the median values from the trials conducted in the USA on these three commodities were not different more than 5-fold, and Kruskal-Wallis test indicated that the residues of orange, grapefruits and lemon were not statistically different, the Meeting decided to estimate a group maximum residue level. The residues of orange, grapefruits and lemon were combined for estimating a maximum residue level for estimating a maximum residue level for citrus fruits (n=22): < 0.01, < 0.01, 0.01, 0.01, 0.02, 0.02, 0.02, 0.04, 0.04, 0.04, 0.04, 0.06, 0.06, 0.07, 0.08, 0.08, 0.09, 0.10, 0.10, 0.11, 0.12 and 0.14 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg for citrus fruit.

For the estimation of STMR, the sum of cyflumetofen and B-1 was calculated: < 0.03, < 0.03, 0.03, 0.03, 0.04, 0.04, 0.04, 0.06, 0.06, 0.06, 0.06, 0.08, 0.08, 0.09, 0.10, 0.10, 0.11, 0.12, 0.12, 0.13, 0.14 and 0.16 mg eq./kg.

The Meeting estimated an STMR of 0.07 mg/kg expressed as cyflumetofen.

Pome fruits

A total of 17 supervised trials were conducted in the USA in 2009 and 2010 on apples (12) and pear (5). One trial was conducted in Italy in 2006 on apples. The GAP in the USA for pome fruits allows 2 applications at a maximum rate of 0.2 kg ai/ha (at least 935 L/ha) with a PHI of 7 days.

The trial from Italy was provided but did not match any GAP and was not considered.

Residues from 12 apple trials matching US GAP were: 0.01, 0.04, 0.06, 0.07, 0.09, 0.10, 0.13, 0.14, 0.16, 0.18, 0.19 and 0.24 mg/kg (median 0.115 mg/kg).

Residues from five pear trials matching US GAP were: 0.06, 0.08, 0.12, 0.12 and 0.20 mg/kg (median 0.12 mg/kg).

As the GAP in the USA is established for the group of pome fruits, and the median values from the trials conducted in the USA on the two commodities did not differ by more than 5-fold, the Meeting decided to estimate a group maximum residue level.

Man-Whitney test indicated that the residues from apple trials and those from pear trials were not statistically different. The Meeting decided to combine the data for estimating a maximum residue level. Residues were (n=17): 0.01, 0.04, 0.06, 0.06, 0.07, 0.08, 0.09, 0.10, 0.12, 0.12, 0.13, 0.14, 0.16, 0.18, 0.19, 0.20 and 0.24 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg for pome fruits.

The sum of cyflumetofen and B-1 was calculated: 0.03, 0.06, 0.08, 0.08, 0.09, 0.10, 0.11, 0.12, 0.14, 0.14, 0.15, 0.16, 0.18, 0.20, 0.21, 0.22 and 0.26 mg eq./kg.

The Meeting estimated an STMR of 0.14 mg/kg expressed as cyflumetofen.

*Berries and Other Small Fruits**Grapes*

A total of 12 supervised trials were conducted on grapes in the USA in 2009. The GAP in the USA for grapes allows 2 applications at a maximum rate of 0.2 kg ai/ha (at least 468 L/ha) with a PHI of 14 days.

Residues from 11 independent trials matching US GAP were: 0.02, 0.09, 0.10, 0.12, 0.15, 0.16, 0.19, 0.22, 0.22, 0.27 and 0.42 mg/kg.

The Meeting estimated a maximum residue level of 0.6 mg/kg for grapes.

The sum of cyflumetofen and B-1 was calculated: 0.04, 0.12, 0.12, 0.16, 0.20, 0.22, 0.23, 0.25, 0.25, 0.33 and 0.46 mg eq./kg.

The Meeting estimated an STMR of 0.22 mg/kg expressed as cyflumetofen.

Strawberry

A total of eight supervised trials were conducted on strawberry in the USA in 2009 and 2010. The GAP in the USA for strawberry allows 2 applications at a maximum rate of 0.2 kg ai/ha (at least 468 L/ha) with a PHI of 1 day.

Residues from eight trials matching US GAP were: 0.04, 0.10, 0.14, 0.14, 0.16, 0.20, 0.23 and 0.36 mg/kg.

The Meeting estimated a maximum residue level of 0.6 mg/kg for strawberry.

The sum of cyflumetofen and B-1 was calculated: 0.06, 0.13, 0.16, 0.16, 0.20, 0.24, 0.28 and 0.40 mg eq./kg.

The Meeting estimated an STMR of 0.18 mg/kg expressed as cyflumetofen.

*Fruiting Vegetables, Other Than Cucurbits**Tomato*

A total of 16 supervised trials were conducted on tomato in the USA in 2009 and 2010. The GAP in the USA for tomato allows 2 applications at a maximum rate of 0.2 kg ai/ha (at least 468 L/ha) with a PHI of 3 days.

Residues from 14 independent trials matching US GAP were: 0.01, 0.02, 0.02, 0.04, 0.04, 0.04, 0.04, 0.06, 0.06, 0.06, 0.07, 0.09, 0.12 and 0.15 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg for tomato.

The sum of cyflumetofen and B-1 was calculated: 0.03, 0.04, 0.06, 0.06, 0.06, 0.06, 0.06, 0.08, 0.08, 0.09, 0.10, 0.11, 0.17 and 0.18 mg eq./kg.

The Meeting estimated an STMR of 0.07 mg/kg expressed as cyflumetofen.

Eggplant

Two supervised trials were conducted on eggplant, green house grown, in Japan in 2004 with the analysis of cyflumetofen and the conjugates of B-1. The current GAP of Japan allows 2 applications at a spray concentration of 0.02 kg ai/hL with a PHI of 1 day. A spray volume of 1000–3500 L can be sprayed per ha. In the trials, spray concentration was 0.02 kg ai/hL and spray volume was 1996 L/ha, matching Japanese GAP. Residues from these trials were: 0.34 and 0.46 mg/kg.

The Meeting concluded that the data were insufficient for estimating a maximum residue level for eggplant.

Tree nuts

Five trials were conducted on almonds and five other trials were conducted on pecans in the USA in 2009. The GAP in the USA for tree nuts allows 2 application at a maximum rate of 0.2 kg ai/ha (in at least 935 L/ha) with a PHI of 7 days.

Residues in almond nutmeat from five trials matching US GAP were: < 0.01 (5) mg/kg.

Residues in pecan nutmeat from five trials matching US GAP were: < 0.01 (5) mg/kg.

As the GAP in the USA is established for the group of tree nuts and as the residues were all < 0.01 mg/kg, the Meeting agreed to estimate a maximum residue level for the tree nuts group at 0.01* mg/kg.

As the B-1 was also below the LOQ in all the trials, and the nutmeat is protected by the hull and not exposed to cyflumetofen foliar spray, the Meeting estimated an STMR to be 0.01 mg/kg expressed as cyflumetofen.

Animal feeds*Almond hulls*

Five trials were conducted on almond in the USA in 2009. The GAP in the USA allows 2 application at a maximum rate of 0.2 kg ai/ha (in at least 935 L/ha) with a PHI of 7 days.

Residues in almond hull from five trials matching US GAP were: 0.35, 0.53, 0.56, 0.86 and 1.87 mg/kg.

The Meeting estimated a maximum residue level of 4 mg/kg.

The sum of cyflumetofen and B-1 was calculated: 0.38, 0.63, 0.67, 0.98 and 2.11 mg/kg. The Meeting estimated a median residue of 0.67 mg/kg.

Fate of residues during processing*High temperature hydrolysis*

To simulate the degradation of cyflumetofen during pasteurization, baking, brewing, boiling and sterilization, the hydrolysis of radio-labelled cyflumetofen was investigated in sterile buffered aqueous solutions.

After incubation at 90 °C (pH 4) for 20 minutes, 100 °C (pH 5) for 60 minutes or 120 °C (pH 6) for 20 minutes, no loss of radioactivity occurred. After heating at 90 °C for 20 minutes, about 69–71% of cyflumetofen remained with 14–23% of the applied radioactivity degraded to B-1. At 100 °C for 60 minutes, only 5–18% of cyflumetofen remained with the formation of B-1 (53–59% AR) and AB-1 (32–40% AR). At 120 °C, cyflumetofen was completely hydrolysed with the formation of B-1 (44–75% AR) and AB-1 (39–49% AR). Cyflumetofen was susceptible to hydrolysis at high temperature.

Processing

The Meeting received information on processing of orange, apple, grape and tomato.

Processing factors calculated for the processed commodities of the above raw agricultural commodities are shown in the table below. STMR-Ps were calculated for processed commodities of orange, apple and grape for which maximum residue levels were estimated.

Processed Orange Product	Cyflumetofen		Cyflumetofen and B-1*		STMR-P or median
	Processing factor	Best estimate	Processing factor	Best estimate	
Orange					0.07
Juice	< 0.02, < 0.08	0.05	< 0.04, < 0.022	0.031	0.0022
Oil	102, 137	120	88.0, 133	111	7.77

Processed Orange Product	Cyflumetofen		Cyflumetofen and B-1*		STMR-P or median
	Processing factor	Best estimate	Processing factor	Best estimate	
Marmalade	0.026, < 0.08	0.026	0.059, < 0.22	0.14	0.0098
Peel	2.86, 2.97	2.92	2.56, 2.67	2.6	0.18
Molasses	< 0.02, < 0.08	< 0.06	< 0.075, < 0.502	0.28	0.020
Dried pulp	0.44, 0.584	0.51	0.759, 0.958	0.86	0.060
Apple					0.14
Applesauce	2.54, 2.91	2.7	3.16, 3.73	3.4	0.48
Juice	0.197, 0.268	0.23	0.224, 0.299	0.26	0.036
Dried apples	0.17, 0.825	0.50	0.21, 0.876	0.54	0.076
Canned apples	0.035, 0.175	0.10	0.076, 0.202	0.14	0.020
Wet pomace	0.937, 1.59	1.3	0.939, 1.57	1.3	0.18
Grape					0.22
Dried grape	0.65, 0.93, 1.88, 4.64	2.0	0.86, 1.1, 2.47, 4.65	2.3	0.506
Juice	0.064, 0.11, 0.2, 0.25	0.16	0.10, 0.16, 0.28, 0.32	0.22	0.0484
Wine (Young)	< 0.006, 0.029, 0.04, 0.04	0.04	0.04, 0.083, 0.17, 0.2	0.12	0.0264
Must	0.02, 0.18, 0.41, 0.53	0.29	0.04, 0.22, 0.44, 0.59	0.32	0.071
Wet Pomace	1.1, 0.02, 3.39, 4.22	2.2	1.1, 3.03, 3.3, 3.97	2.9	0.638
Tomato					0.07
Juice	< 0.06, 0.2	0.2	< 0.2, 0.38	0.38	0.027
Canned Tomato	< 0.04, 0.2	0.2	< 0.1, 0.3	0.3	0.021
Puree	0.3, 0.88	0.59	0.4, 0.79	0.60	0.042
Paste	0.2, 0.4	0.3	1.2, 2.2	1.7	0.12
Wet Pomace	1.3, 5.5	3.4	1.4, 5.0	3.2	0.22

* expressed as cyflumetofen.

As the residue concentration is higher in orange oil than in fresh orange, the Meeting estimated a maximum residue level of 36 mg/kg for citrus oil by multiplying the maximum residue level of citrus fruits (0.3 mg/kg) by 120.

As the residue concentration is higher in dried grapes than in fresh grapes, the Meeting estimated a maximum residue level of 1.5 mg/kg by multiplying the maximum residue level for grapes (0.6 mg/kg) by 2.0.

In processing of foods, heating in aquatic system at high temperature may be employed, and therefore it is likely that AB-1 is present at significant concentrations after processing at slightly acidic to neutral pH (≥ 5). It may also occur from cyflumetofen during the storage of processed foods with high water content at room temperature. However, the Meeting concluded that AB-1 is covered by the ADI for the parent compound.

Residues in animal commodities

Estimation of dietary burden

The maximum and mean dietary burdens were calculated using the median residues of cyflumetofen (sum of cyflumetofen and B-1 expressed as cyflumetofen, to cover the worst case) estimated at the current Meeting on a basis of the OECD Animal Feeding Table.

Summary of livestock dietary burdens (ppm of dry matter diet)

	US-Canada		EU		Australia		Japan	
	Max	Mean	max	Mean	Max	Mean	Max	mean
Beef cattle	0.007	0.007	0.090	0.090	0.934 ^a	0.934 ^b	0	0
Dairy cattle	0.119	0.119	0.052	0.052	0.934 ^a	0.934 ^b	0	0
Broilers	0	0	0	0	0	0	0	0
Layers	0	0	0	0	0	0	0	0

^a Suitable for estimating maximum residue levels for milk, meat, fat and edible offal of cattle.

^b Suitable for estimating STMRs for milk, meat, fat and edible offal of cattle.

Residues in milk and cattle tissues

In the goat metabolism studies, in which goats were administered benzoyl-label cyflumetofen at a dose equivalent to 12.8 ppm in feed for 12 consecutive days and sacrificed one day later, cyflumetofen was found only in fat at 0.003 mg/kg. B-1 were found in liver, kidney, muscle, fat and milk at 0.125, 0.102, 0.005, 0.006 and 0.001 mg eq./kg respectively. The sum of cyflumetofen and B-1 in fat is 0.009 mg/kg as cyflumetofen. Neither cyflumetofen nor metabolites were found above 0.01 mg/kg or 10% TRR when butylphenyl-label cyflumetofen was administered to goats.

These concentrations were multiplied by 0.934/12.8 for estimating STMRs. The Meeting estimated STMRs of 0.010, 0.008, 0, 0 and 0 mg/kg for liver, kidney, meat, fat and milk, respectively.

The Meeting estimated maximum residue levels of 0.02, 0.01*, 0.01* and 0.01* mg/kg expressed as cyflumetofen for edible offal (mammalian), meat (from mammals other than marine mammals), mammalian fats (except milk fat) and milks, respectively.

The dietary burden for poultry was calculated to be 0 ppm of dry matter diet. No metabolism or feeding studies on laying hens were conducted.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for plant commodities (for compliance with the MRL): *Cyflumetofen*.

Definition of the residue for plant commodities (for estimation of dietary intake): *Sum of cyflumetofen and 2-trifluoromethylbenzoic acid, expressed as cyflumetofen*.

Definition of the residue for animal commodities (for compliance with the MRL and estimation of dietary intake): *Sum of cyflumetofen and 2-trifluoromethylbenzoic acid, expressed as cyflumetofen*.

Residue is not fat-soluble.

Commodity		Recommended MRL, mg/kg		STMR/STMR-P mg/kg ^a
CCN	Name	New	Previous	
AM 0660	Almond hulls	4	-	Median: 0.67
FC 0001	Citrus fruit	0.3	-	0.07
	Citrus oil	36		7.77
DF 0269	Dried grape	1.5		0.506
MO 0105	Edible offal (mammalian)	0.02		Liver: 0.010 Kidney: 0.008
FB 0269	Grapes	0.6	-	0.22
MF 0100	Mammalian fats (except milk fat).	0.01*		0
MM 0095	Meat (from mammals other than marine mammals)	0.01*		0
ML 0106	Milks	0.01*		0
FP 0009	Pome fruits	0.4	-	0.14
FB 0275	Strawberry	0.6	-	0.18
VO 0448	Tomato	0.3	-	0.07
TN 0085	Tree nuts	0.01*	-	0.01
	Orange juice			0.0022
	Marmalade			0.098
	Citrus peel			0.18

Commodity		Recommended MRL, mg/kg		STMR/STMR-P
CCN	Name	New	Previous	mg/kg ^a
	Citrus molasses			0.020
	Apple sauce			0.48
	Apple juice			0.036
	Dried apple			0.076
	Canned apple			0.020
	Grape juice			0.048
	Grape wine			0.026
	Grape must			0.071
	Tomato juice			0.027
	Canned tomato			0.021
	Tomato puree			0.042
	Tomato paste			0.12

^a Sum of cyflumetofen and B-1 expressed as cyflumetofen.

For calculating animal dietary burdens.

Commodity		Median residue, mg/kg ^a
CCN	Name	
	Citrus dry pulp	0.060
	Apple wet pomace	0.18
	Grape wet pomace	0.64
	Tomato wet pomace	0.22

Expressed on an “as received” basis.

^a Sum of cyflumetofen and B-1 expressed as cyflumetofen.

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Dietary Intakes (IEDIs) of cyflumetofen were calculated for the 17 GEMS/Food cluster diets using STMRs estimated by the current Meeting (Annex 3 of the 2014 Report). The ADI is 0–0.1 mg/kg bw and the calculated IEDIs were 0–1% of the maximum ADI. The Meeting concluded that the long-term intake of residues of cyflumetofen resulting from the uses considered by the current JMPR is unlikely to present a public health concern.

Short-term intake

The 2014 JMPR decided that an ARfD was unnecessary. The Meeting therefore concluded that the short-term intake of residues of cyflumetofen is unlikely to present a public health concern.

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OTSA-0035(EN)-FR	Iijima, K	2003 c	OK-5101 pure: Solubility study in organic solvents Institute of Environmental Toxicology, Mitsukaido Ibaraki 303-0043, Japan Unpublished
OTSA-0036(EN)-FR	Iijima, K	2004 a	OK-5101 pure: Determination of n-octanol/water partition coefficient Institute of Environmental Toxicology, Mitsukaido Ibaraki 303-0043, Japan Unpublished
OTSA-0076	Knight, L	2004 a	OK-5101—Hydrolysis under laboratory conditions Huntingdon Life Sciences Ltd., Huntingdon Cambridgeshire PE28 4HS, United Kingdom Unpublished
OTSA-0078	Knight, LJL	2004 b	OK-5101—Photodegradation in water and quantum yield of direct photodegradation in water Huntingdon Life Sciences Ltd., Huntingdon Cambridgeshire PE28 4HS, United Kingdom Unpublished
OTSA-0060(EN)-FR	Nakamura, H	2002 a	OK-5101 pure: Determination of IR spectrum Institute of Environmental Toxicology, Mitsukaido Ibaraki 303-0043, Japan Unpublished
OTSA-0156(EN)-FR	Nakamura, H	2004 a	(¹⁴ C)OK-5101 (phenyl-ring-U-and tolyl-ring-U-label): A hydrolysis study Institute of Environmental Toxicology, Mitsukaido Ibaraki 303-0043, Japan Unpublished
OTSA-0301-FR	North, L <i>et al.</i>	2007 a	Final report on project AF/10943/OT—To determine the magnitude of OK-5101 20SC residues at harvest in the raw agricultural commodity apple resulting from sequential overall foliar applications of OK-5101 20 SC, in Italy during 2006 Agrisearch UK Ltd., Melbourne Derbyshire DE73 8AG, United Kingdom Unpublished
OTSA-0203 (2)-FR	Odanaka, Y and Wakazone, Y	2004 a	OK-5101 (Danisaraba)—Pesticide residue analysis on eggplant The Institute of Environmental Toxicology, Joso-shi Ibaraki 303-0043, Japan Unpublished
OTSA-0157(EN)-FR	Ohyama, K	2004 a	(¹⁴ C)OK-5101 (phenyl-ring-U-and tolyl-ring-U-label): A photolysis study in water Institute of Environmental Toxicology, Mitsukaido Ibaraki 303-0043, Japan Unpublished
OTSA-0049(EN)-FR	Saka, M	2002 a	OK-5101 pure: Vapour pressure study Institute of Environmental Toxicology, Mitsukaido Ibaraki 303-0043, Japan Unpublished
2011/1106409	Sowka, N <i>et al.</i>	2011 a	The metabolism of ¹⁴ C-Cyflumetofen in lactating goats BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished

Code	Author(s)	Year	Title, Institute, Report reference
2011/7000143	White, MT	2011 a	Magnitude of Cyflumetofen (BAS 9210 I) in pomefruit following applications of BAS 9210 0I Alliance Pharma Inc., Malvern PA, United States of America Unpublished
2011/7000144	White, MT	2011 b	Magnitude of Cyflumetofen (BAS 9210 I) in apple processed fractions following applications of BAS 9210 0I Alliance Pharma Inc., Malvern PA, United States of America Unpublished
2010/7014999	White, MT and Saha, M	2011 b	Magnitude of Cyflumetofen (BAS 9210 I) in strawberry following applications of BAS 9210 0 I BASF Agricultural Research Center, Research Triangle Park NC, United States of America Unpublished
2008/1098832	Willems, HPL	2008 a	OK-5101—Atkinson calculation Notox BV, s-Hertogenbosch, Netherlands Unpublished

DICHLOBENIL (274)*see also FLUOPICOLIDE (235)**First draft prepared by Dr Samuel Margerison, Australian Pesticides and Veterinary Medicines Authority, Canberra, Australia***EXPLANATION**

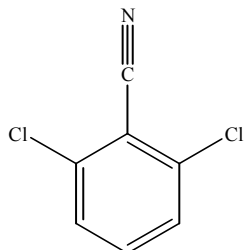
Dichlobenil is a herbicide belonging to the benzonitrile group of chemicals. It is a systemic herbicide, which acts by inhibition of cell wall (cellulose) biosynthesis, thereby inhibiting actively dividing meristems, germination of seeds, and damaging rhizomes. It is used for control of annual and perennial weeds, principally in orchard crops.

At the Forty-fifth session of the CCPR (2013), dichlobenil was scheduled for evaluation as a new compound by the 2014 JMPR.

The Meeting received information on identity and physical and chemical properties, animal and plant metabolism, environmental fate in soil and water, rotational cropping, analytical methods, storage stability, use patterns, supervised residue trials, fate of residues on processing, and animal feeding studies.

IDENTITY

Common name:	Dichlobenil
Chemical names	
IUPAC:	2,6-Dichlorobenzonitrile
CAS:	2,6-Dichlorobenzonitrile
CAS number:	1194-65-6
Synonyms:	Casoron
Structural formula:	



Molecular formula:	C ₇ H ₃ Cl ₂ N
Molecular weight:	172.0

SPECIFICATIONS

Specifications for dichlobenil have not been published by FAO.

PHYSICAL AND CHEMICAL PROPERTIES

Table 1a Physical and chemical properties of dichlobenil

Appearance	Off-white crystalline solid (technical material)
Melting point	143.8–144.3 °C (technical material)
Boiling point	270 °C (101.3 kPa)
Relative density	1.55
Water solubility	25 mg/L (25 °C)

Solubility in organic solvents	Acetone: 86 g/L (20 °C) Cyclohexane: 3.7 g/L (25 °C) Methanol: 17.2 g/L (20 °C) Dichloromethane: 151 g/L (20 °C) Ethyl acetate: 59.3 g/L (20 °C) Xylene: 53 g/L (25 °C)
Hydrolysis	Stable in pH 5, 7 and 9 unsterilized buffers at 22 ± 1 °C, with half-life >150 days (Buisman, 1988)
Aqueous photolysis (sterile pH 7 buffer in sealed ampoules at 25 ± 1 °C)	10 days under spring/summer sunlight at 40°N latitude. Metabolites included 4-chloro-2(3H)benzoxazolone, 2,6-dichlorobenzoic acid, 2,6-dichlorobenzamide, 2-hydroxybenzonitrile, 2,6-dichloro-4-hydroxybenzonitrile, 2-chlorobenzonitrile and various small polar molecules (Boelhouwers <i>et al.</i> , 1989)
Vapour pressure	0.14 Pa (25 °C)
Octanol/water partition coefficient ($\log_{10}P_{OW}$)	2.70 (pH 3, 22 °C)
pH	6.7
Stability	Stable for 3 months at 54 °C
Explosive characteristics	Not explosive
Corrosive characteristics	Not corrosive

Table 1b Physico-chemical properties of 2,6-dichlorobenzamide

Appearance	Crystalline solid
Melting point	195–203 °C
Boiling point	335 °C (estimated)
Density	1.44 g/cm ³
Water solubility	2.7 g/L (20–25 °C)
Vapour pressure	$0.35\text{--}4.35 \times 10^{-3}$ Pa at 25 °C (various estimated values)
Octanol/water partition coefficient ($\log_{10}P_{OW}$)	0.77 (Nakagawa <i>et al.</i> , 1992)
Hydrolysis (5.19–5.26 mg/L ¹⁴ C-(U-phenyl) label in pH 5, 7, and 9 buffers at 25 ± 2 °C in the dark)	No degradation over 33 days (Nag, 2000)
Aqueous photolysis (¹⁴ C-1-phenyl) label in sterile pH 5 buffer at 25 °C irradiated with 40.3°N summer sunlight in a 12 hour light/dark cycle)	Compound was not photolysed or hydrolysed over 31 days (Reynolds, 2000)

FORMULATIONS

GAPs have been reported for two formulations, a capsule suspension (CS) containing 168 g/L dichlobenil, and a granular formulation containing 40 g/kg dichlobenil.

METABOLISM AND ENVIRONMENTAL FATE

The metabolism of dichlobenil has been investigated in grapes, rats, laying hens and lactating goats. Metabolism of the major plant metabolite 2,6-dichlorobenzamide has also been investigated in lactating goats and laying hens.

Metabolism studies for dichlobenil and 2,6-dichlorobenzamide were conducted with compound uniformly labelled with carbon-¹⁴ in the benzyl ring.

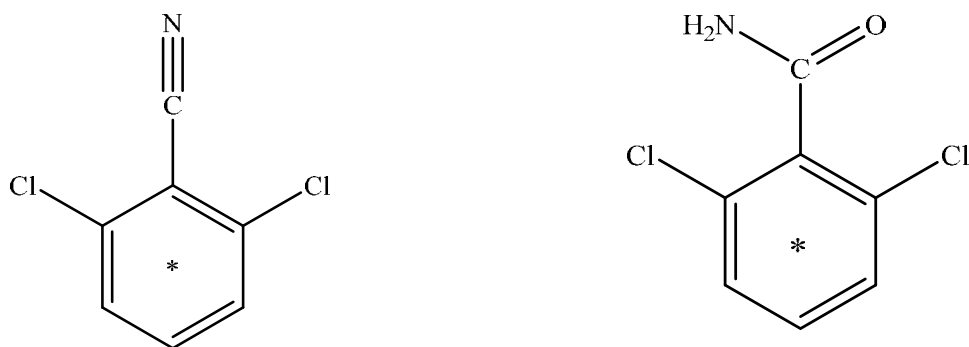
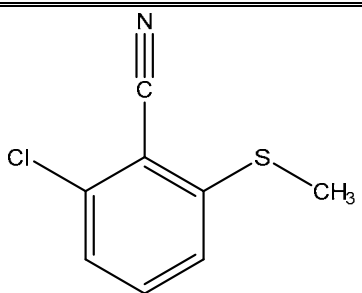
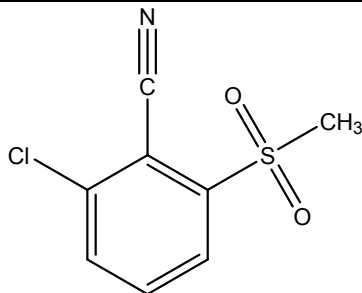
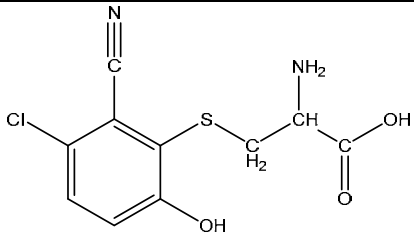
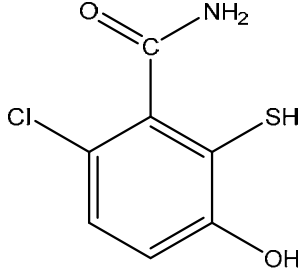
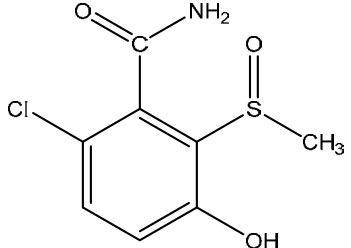
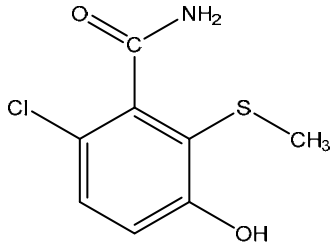


Figure 1: [^{14}C -U-phenyl]-dichlobenil and [^{14}C -U-phenyl]-dichlorobenzamide using in the metabolism studies

Table 2 Metabolites of dichlobenil and 2,6-dichlorobenzamide

Code name, common name	IUPAC name	Structure	Where found
Dichlobenil (parent compound)	2,6-Dichlorobenzonitrile		
BAM	2,6-Dichlorobenzamide		Grapes, apples, soil
BAM-4-OH	2,6-Dichloro-4-hydroxybenzamide		Grapes
	4-Chloro-2(3H)benzoxazolone		Aqueous photolysis

Code name, common name	IUPAC name	Structure	Where found
Methylthio- dichlobenil	6-Chloro-2-methylthiobenzonitrile		Goat, hen (tentative)
Methylsulfonyl- dichlobenil	6-Chloro-2-methylsulfonylbenzonitrile		Goat, hen
Dichlobenil-OH- cysteine	S-(3-Chloro-2-cyano-6-hydroxyphenyl)-cysteine		Goat, hen
BAM-OH-SH	6-Chloro-3-hydroxy-2-thiobenzamide		Goat metabolite of BAM
Methylsulfinyl- BAM-OH	6-Chloro-3-hydroxy-2-methylsulfinylbenzamide		Goat metabolite of BAM
Methylthio-BAM- OH	6-Chloro-3-hydroxy-2-methylthiobenzamide		Goat metabolite of BAM

Animal metabolism

Rats

The metabolism of dichlobenil in rats was investigated and was evaluated by the WHO Panel of the 2014 JMPR.

Rats: dichlobenil

Following oral gavage dosing of rats, dichlobenil was rapidly absorbed: about 89 and 97% of the administered dose in male and female in 7 days, respectively, based on comparison of urinary excretion following oral and intravenous routes of exposure. Oral absorption after repeated exposure is at least 60%, irrespective of dose and sex, based on radiolabel recovered from urine, faeces and cage wash. Liver, kidney and kidney-fat retained the highest concentrations of radiolabel, peaking 1 hour after administration. The elimination of the radioactivity associated with [¹⁴C]dichlobenil following oral administration was rapid, with the majority (about 80%) of the radioactivity being eliminated within 24 hours at the low dose level. Around 60% is excreted in the urine and around 20% in the faeces, irrespective of sex or dose regime, 9 to 10 days after initiation of exposure. Approximately 79% of the administered dose was excreted in the bile in 24 hours in bile duct cannulated rats. Ten metabolites were identified in urine, although no parent compound was identified in urine. Most metabolites were conjugates such as sulphate, glutathione (and derivatives) and glucuronic acid conjugates. In faeces, three major metabolites (each representing 5% of the administered dose or more) were observed as well as parent compound (dichlobenil). The proportion of conjugated metabolites decreases with increasing and multiple doses, while the amount of parent compound in faeces increases. No chemical names were provided for the identified metabolites. Dichlobenil is metabolized in rats via two metabolic pathways: hydroxylation at the 3 or 4 position followed by glucuronidation or sulphation and the second pathway include substitution of one chlorine atom by glutathione.

Lactating goats: dichlobenil

Metabolism of [¹⁴C-U-phenyl]-dichlobenil was investigated in lactating goats (Cameron and Dunsire, 1988 and Ruijten *et al.*, 1988). Three animals were dosed orally twice daily by capsule for three consecutive days at the same dose of 10 mg/kg bw/day (one animal, goat 10P, as a pilot study and the other two, goats 7M and 8M, comprising the main study). Two further goats were dosed with unlabelled material at the same level.

The goats were kept in metabolism cages during the feeding phase of the study. Fresh drinking water and hay were available *ad libitum*, while commercially available diet was provided twice daily at milking. The dose was administered orally twice daily at approximately 0900 and 1700 using gelatin capsules. Actual dose levels were 9.9–10.2 mg/kg bw/day (mean = 10.0 mg/kg bw/day). Feed consumption was not recorded. Based on an estimated dry matter consumption figure of 4% of the body weight per day, these doses would equate to a mean of 250 ppm in feed.

The goats were milked twice daily and milk was sampled. Blood samples were taken prior to first and fifth doses, and at intervals of 30 minutes to 8 hours after each of these doses. A final blood sample was taken just prior to sacrifice. The goats were sacrificed 15 hours after the final dose, and samples of liver, kidney, muscle, fat, gastrointestinal (GI) tract and contents, and the remaining carcass were collected.

Total radioactive residues in liquid samples (urine, cage wash and plasma) were mixed with scintillation cocktail and analysed by liquid scintillation counting (LSC). Prior to combustion and LSC of the captured ¹⁴CO₂, faeces and residual carcass samples were homogenised with water, tissue samples were finely chopped, while the GI tract samples were separated into tissue and contents, with the contents being homogenised in water, and the tissue being minced then homogenised in water with carboxymethylcellulose.

The majority of the administered dose was excreted (39–45% in urine, 24–31% in faeces, 2.9–4.0% in cage wash, and 0.06 to 0.33% in milk). Carcass tissue accounted for 3.2 to 3.4 % of the

administered radioactivity, while 1.1–3.3% remained in GI tract tissue, and 0.4–3.3% GI tract contents.

Table 3 Total radioactive residues in milk after dosing of dichlobenil at 250 ppm

Sampling interval (hours)	Concentration (mg eq./kg)			
	Goat 10P	Goat 7M	Goat 8M	Mean
Predose	0	0	0	0
0-8	0.96	0.95	0.95	0.95
8-24	0.73	0.87	0.76	0.79
24-32	0.85	1.46	1.58	1.30
32-48	0.77	0.90	0.90	0.86
48-56	1.12	1.28	1.48	1.29
56-71	0.91	0.85	0.84	0.87

NA = not applicable.

Total dichlobenil radioactive residues appeared to reach a plateau in milk after 1.5 days of dosing.

Concentrations of ^{14}C -dichlobenil total residue in plasma and whole blood peak around 2-3 hours after each dose.

Table 4 Total radioactive residues in goat tissues after dosing of dichlobenil at 250 ppm

Tissue	Residue (mg eq./kg)			
	Goat 10P	Goat 7M	Goat 8M	Mean
Plasma (15 hours after final dose)	1.04	0.76	0.64	0.81
Whole blood (15 hours after final dose)	0.80	0.49	0.45	0.58
Liver	32.4	25.0	24.7	27.4
Kidney	4.91	3.82	3.47	4.07
Fat	1.77	3.40	1.98	2.38
Muscle	0.58	0.35	0.34	0.42
GI tract tissue	1.08	1.65	1.65	1.46
GI tract contents	6.52	11.5	12.0	10.0
Carcass	1.40	1.18	1.09	1.22

Residues in milk and tissues of goats 7M and 8M from the main study were further characterised. To provide additional material to aid identification, the samples from the goats treated with unlabelled compound were co-processed, except for muscle, for which no unlabelled samples were available. Instead, the muscle from the pilot study goat was used.

The samples were homogenised, with water in the case of liver, kidney and milk, and with blank skim milk in the case of fat and muscle, and then filtered and centrifuged to remove solid material. The samples were then freeze concentrated if necessary to reduce the volume, prior to HPLC with on-line clean-up using a pre-column. Metabolic profiles of the sample extracts were analysed by HPLC with gradient elution (mobile phases were 3 g/L ammonium hydrogen phosphate and methanol) and radiodetection with fraction collection. Isolated fractions were further purified by passing them through the HPLC system a second time, and by thin layer chromatography.

The proportions of residue extracted were relatively low, and variable, at 65–70% of TRR in milk, 65% in liver, 26% in fat, 13% in muscle, and 28% in kidney. It is noted that no further characterisation was carried out for residues not extracted by the initial homogenisation with water or skim milk.

The metabolites were identified using mass spectrometry and ^1H NMR, and the data are presented in tables 5 and 6 below. Radioactivity present at each step of the work up process was reported as a percentage of the TRR. Significant losses were noted in several of the sample work-ups. It is noted that no attempt was made to characterise residues in the post-extraction solids. The metabolic profile was determined at different stages during the extraction and purification processes, with the relative proportions remaining consistent, indicating that losses were non-selective.

Components identified in the milk and tissues of lactating goats given ^{14}C -phenyl dichlobenil were: parent compound, the hydroxylated cysteine conjugate of deschloro dichlobenil, and the subsequent metabolites of the cysteine conjugate (without hydroxylation), 6-chloro-2-methylthiobenzonitrile (tentatively identified on the basis of comparison of retention times with a standard) and 6-chloro-2-methylsulfonylbenzonitrile. Parent compound was only present in significant quantities in fat. The cysteine conjugate was the most significant component of the liver residue, and was also noted in the milk samples, which contained higher proportions of this and other polar metabolites, such as 2-chloro-6-methylsulfonylbenzonitrile. 2-Chloro-6-methylthiobenzonitrile, a cleavage product of the cysteine conjugate, was found in significant proportions in muscle and kidney and the later milk samples, consistent with its position further down the metabolic pathway. The key metabolic pathway for dichlobenil in goats is cysteine conjugation at one of the chlorine positions on the phenyl ring, followed by cleavage and oxidation of the cysteine side chain, together with hydroxylation at the 3-position which may occur in parallel with the cysteine conjugation.

Table 5 Relative amounts of dichlobenil metabolites chromatographed in milk centrifugate (pooled milk of goats 7M and 8M)

Metabolite	0-48 h		48-56 h		56-72 h	
	%TRR	mg eq. /kg	%TRR	mg eq. /kg	%TRR	mg eq. /kg
Dichlobenil	-	-	0.7	0.01	-	-
Unknown 1	12	0.12	3.9	0.05	7.2	0.06
S-(3-Chloro-2-cyano-6-hydroxyphenyl)-cysteine	20	0.20	21	0.29	11	0.09
2-chloro-6-methylsulfonylbenzonitrile (tentative)	38	0.37	20	0.28	14	0.12
2-Chloro-6-methylthiobenzonitrile (tentative)	-	-	20	0.27	33	0.28
Total extracted	70	0.69	65	0.90	65	0.55
TRR	100	0.98	100	1.38	100	0.85

Table 6: Identification of metabolites in tissues

Component	Liver		Fat		Muscle		Kidney	
	%TRR	mg eq./kg	%TRR	mg eq./kg	%TRR	mg eq./kg	%TRR	mg eq./kg
Dichlobenil	-	-	16	0.42	-	-	-	-
S-(3-Chloro-2-cyano-6-hydroxyphenyl)-cysteine	62	15.3	-	-	0.9	0.003	1.1	0.04
2-Chloro-6-methylsulfonylbenzonitrile	3	0.81	10	0.28	-	-	1.4	0.05
Unidentified component	-	-	-	-	1.3	0.005	2.5	0.09
2-Chloro-6-methylthiobenzonitrile: tentative	-	-	-	-	11	0.038	23	0.84
Total extracted	65	16.1	26	0.70	13	0.046	28	1.0
TRR	100	24.9	100	2.69	100	0.35	100	3.65

Laying hens: dichlobenil

Metabolism of ^{14}C -phenyl labelled dichlobenil was investigated in laying hens (Cameron and Dunsire, 1988 and Ruijten *et al*, 1988). Eight birds were dosed orally twice daily by capsule for three consecutive days, with radiolabelled material at 9.8-10.2 mg/kg bw/day (mean = 10.0 mg/kg bw/day). Feed consumption was not recorded. Based on an estimated dry matter consumption figure of 7.5% of the body weight per day, these doses would equate to a mean of 133 ppm in feed.

The hens were kept in metabolism cages during the feeding phase of the study. Fresh drinking water and layer pellets were available *ad libitum*. The dose was administered orally twice daily at approximately 0900 and 1700 using gelatin capsules. .

Eggs, excreta and cage washings were collected twice daily prior to administration of each dose, and 15 hours after the final dose. Blood samples were taken from all birds just prior to sacrifice. Two of the hens additionally had blood samples taken and plasma separated prior to doses 1 and 5, and at 1, 3 and 6 hours after these doses. The other six hens were sacrificed 15 hours after the final dose, and samples of liver, kidney, breast muscle, abdominal fat, gastrointestinal (GI) tract and contents, and the remaining carcass were collected.

Total radioactive residues in liquid samples (cage wash and plasma) were mixed with scintillation cocktail and analysed by liquid scintillation counting (LSC). Prior to combustion and LSC of the captured $^{14}\text{CO}_2$, excreta and residual carcass samples were homogenised with water; and tissue and GI tract samples were finely chopped.

The majority of the administered dose was excreted (64–73%) with 0.17–0.59% found in eggs, 2.8–16% in the GI tract at sacrifice, and 0.9–11% in the carcass. The recoveries of the total administered radioactivity were 74–92%.

Table 7 Total radioactive residues in eggs

Sampling interval (hours)	Concentration (mg eq./kg)						
	Hen 6P	Hen 7P	Hen 1M	Hen 2M	Hen 3M	Hen 4M	Mean
Pre-dose	0	0	0	0	0	0	0
Post-dose 1	0	NS	0	0	0	0	0
Post-dose 3	0.40	0.13	0.25	0.32	0.43	0.48	0.34
Post-dose 5	NS	1.25	1.15	1.30	1.18	1.37	1.25
Post-dose 6	2.11	1.90	1.64	NS	1.83	2.18	1.93

NS = no sample.

Total dichlobenil radioactive residues do not appear to have reached a plateau in eggs after 3 days of dosing. Concentrations of ^{14}C -dichlobenil total residue in hen plasma peak around 3 hours after each dose.

Table 8 Total radioactive residues in hen tissues

Tissue	Residue (mg eq./kg)						
	Hen 6P	Hen 7P	Hen 1M	Hen 2M	Hen 3M	Hen 4M	Mean
Plasma (15 hours after final dose)	2.4	2.5	1.4	1.7	1.0	2.0	1.8
Whole blood (15 hours after final dose)	1.2	1.3	0.68	1.0	0.65	1.3	1.0
Liver	6.4	6.1	6.6	9.2	18.4	8.5	9.2
Kidney	3.6	3.1	3.5	4.5	3.9	5.4	4.0
Fat	34.8	4.1	15.1	19.0	12.0	14.7	16.7
Muscle	0.46	0.29	0.32	0.33	1.07	0.42	0.48
GI tract	8.7	9.3	10.1	22.8	10.3	33.6	15.8
Carcass	1.3	0.89	3.2	3.9	1.8	1.9	2.2

The highest levels of total radioactive residue are found in fat and the GI tract, with significant levels also being observed in the liver.

Residues in eggs and tissues of hens from the study were further characterised. The samples were homogenised, with water in the case of liver, kidney and eggs, and with skim milk in the case of fat and muscle, and then filtered and centrifuged to remove solid material. The samples were then freeze concentrated if necessary to reduce the volume, prior to HPLC with on-line cleanup using a pre-column. Metabolic profiles of the sample extracts were analysed by HPLC with gradient elution (mobile phases were 3 g/L ammonium hydrogen phosphate and methanol) and radio-detection with fraction collection. Isolated fractions were further purified by passing them through the HPLC system a second time, and by thin layer chromatography.

The proportions of residue extracted were variable, at 60% of TRR in eggs, 90% in liver, 15% in fat, 8% in muscle, and 39% in kidney.

The metabolites were identified using mass spectrometry and ^1H NMR, and the data are presented in table 9 below. Significant losses were noted in several of the sample work-ups. It is noted that no attempt was made to characterise residues in the post-extraction solids.

Components identified in the eggs and tissues of laying hens given ^{14}C -phenyl dichlobenil were: parent compound, the hydroxylated cysteine conjugate of deschloro dichlobenil, and the subsequent metabolites of the cysteine conjugate, 6-chloro-2-methylthiobenzonitrile and 6-chloro-2-methylsulfonylbenzonitrile. Parent was the only component identified in the more hydrophobic matrices, fat and eggs, with parent also being found in smaller proportions in the other tissues. A metabolite tentatively identified as 2-chloro-6-methylthiobenzonitrile was the predominant component of the residue in liver and muscle, while kidney showed the greatest number of residue components, and a higher proportion of the more water soluble metabolites, the hydroxylated cysteine conjugate, and its oxidative cleavage product, 2-chloro-6-methylsulfonylbenzonitrile. The key metabolic pathway for dichlobenil in hens is cysteine conjugation at one of the chlorine positions on the phenyl ring, followed by cleavage and oxidation of the cysteine side chain, together with hydroxylation at the 3-position.

Table 9 Identification of metabolites in tissues and eggs

Metabolite	Eggs (day 2)		Liver		Fat		Muscle		Kidney	
	%TRR	mg eq./kg	%TRR	mg eq./kg	%TRR	mg eq./kg	%TRR	mg eq./kg	%TRR	mg eq./kg
Dichlobenil	60	0.22	5.4	0.58	15	2.3	1.6	0.01	9.8	0.43
Unknown 1	-	-	-	-	-	-	-	-	1.6	0.07
Unknown 2	-	-	-	-	-	-	-	-	1.2	0.05
S-(3-Chloro-2-cyano-6-hydroxyphenyl)-cysteine	-	-	-	-	-	-	-	-	17	0.74
2-Chloro-6-methylsulfonylbenzonitrile)	-	-	-	-	-	-	-	-	5.1	0.22
2-Chloro-6-methylthiobenzonitrile: tentative	-	-	85	9.1	-	-	6.4	0.03	4.3	0.18
Total characterised	60	0.22	90	9.6	15	2.3	8	0.04	39	1.7
Total residue	100	0.37	100	10.7	100	15.2	100	0.54	100	4.33

Overview of metabolism of dichlobenil in animals

The major metabolic pathway for dichlobenil in food producing animals is conjugation, substituting cysteine for one of the chlorine atoms accompanied by hydroxylation of the phenyl ring. Cleavage and oxidation of the cysteine side chain of the unhydroxylated conjugate also takes place, giving 6-chloro-2-methylthiobenzonitrile and 6-chloro-2-methylsulfonylbenzonitrile.

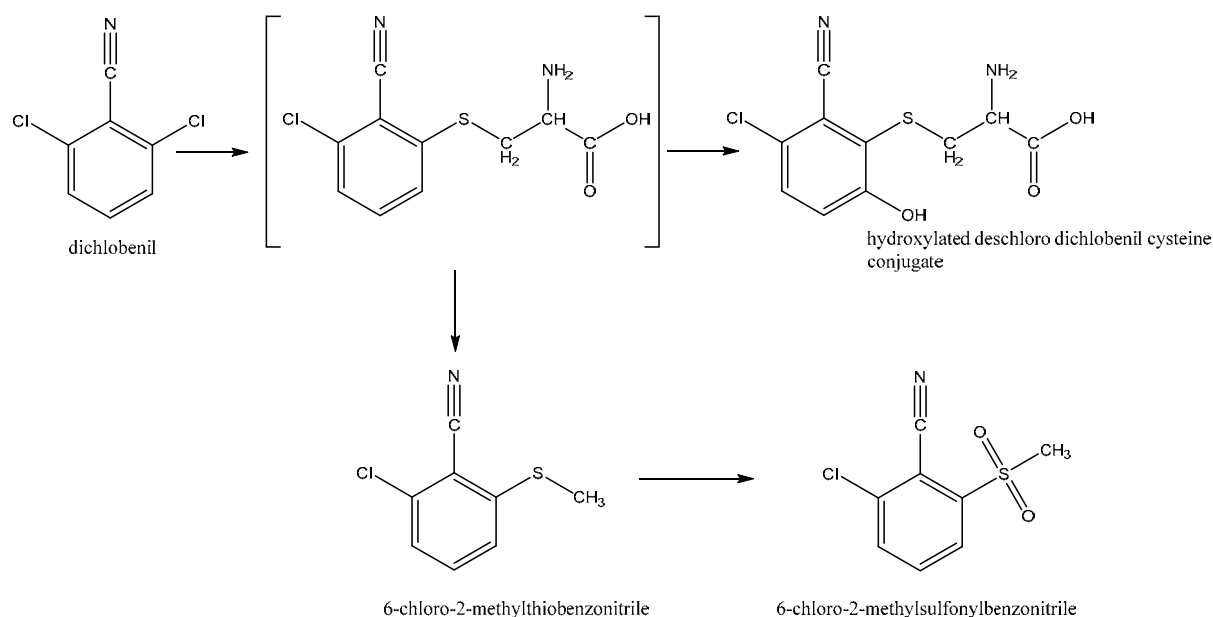


Figure 2: Metabolism of dichlobenil in animals

There are strong similarities to the metabolism of dichlobenil in rats. In rats, hydroxylation at the 3-position also takes place, as well as substitution of one of the chlorine atoms, initially by glutathione, which is subsequently cleaved to give a cysteine moiety. Further cleavage and oxidation of the cysteine substituent to give the methylthio-dichlobenil and methylsulfonyl-dichlobenil also takes place in the rat.

Given that almost the only component of the residue in fruit crops is 2,6-dichlorobenzamide, with no parent being identified, the metabolism of dichlobenil in animals is of secondary importance to the animal metabolism of 2,6-dichlorobenzamide.

Rats: 2,6-Dichlorobenzamide

Metabolism data for 2,6-dichlorobenzamide in rats was not provided to the Meeting.

Lactating goats: 2,6-dichlorobenzamide

Metabolism of ^{14}C -phenyl labelled 2,6-dichlorobenzamide was investigated in lactating goats (Burke, 1994a and Ruijten *et al*, 1994a). Dairy goats were dosed with ^{14}C -2,6-dichlorobenzamide daily at 13 ppm (0.32 mg/kg bw/day) for five days or 252 ppm (7.2 mg/kg bw/day) for three days.

The goats were kept in metabolism cages during the feeding phase of the study. Fresh drinking water was available *ad libitum*, while measured quantities of feed (commercially available diet, shredded molasses beet pulp, and hay) were provided each morning and afternoon, with feed consumption being monitored. The dose was administered orally using gelatin capsules. No overt signs of toxicity were observed during the study, and the dosing did not have any adverse effects on the weight of the animals, feed intake, or milk production.

Urine and faeces were collected at 24 hour intervals, with milk being collected twice daily and pooled for each 24 hour period. The cages were rinsed after each collection of excreta and the cage wash was collected. The low dose animal was sacrificed approximately 23 hours after the final dose, while the high dose animal was sacrificed around 4 hours following the final dose. Samples of skeletal muscle (composite of fore and hind quarter), fat (composite of omental and perirenal), liver, and kidney were collected. The tissue and faeces samples were macerated and sub-sampled prior to frozen storage, with the liquid samples being appropriately sub-sampled.

Samples of urine, milk and cage wash were analysed for total radioactivity directly by liquid scintillation counting (LSC), with the tissue and faeces samples being combusted prior to capture and LSC of the liberated $^{14}\text{CO}_2$.

In the low dose goat, 62% of the administered dose was recovered in urine, 17% in faeces, 0.3% in milk, and 11% in tissues, with >90% total recovery. For the high dose animal, 48% of the dose was recovered in urine, 18% in faeces, 0.4% in milk and 5% in tissues (>70% total recovery).

Table 10 Total radioactive residues in milk

Sampling interval (hours)	Concentration (mg eq./kg)	
	Low dose animal	High dose animal
0-24	0.033	0.95
24-48	0.045	1.12
48-58	NA	2.88
48-72	0.047	NA
72-96	0.048	NA
96-120	0.045	NA
120-126	0.019	NA

NA = not applicable.

Residues of 2,6-dichlorobenzamide reached a plateau in milk at about day 3 of dosing.

Table 11 Total radioactive residues in goat tissues

Tissue	Residue (mg eq./kg)	
	Low dose animal (12.8 ppm)	High dose animal (252 ppm)
Fat	0.024	2.15
Kidney	1.19	17.9
Liver	9.41	67.6
Muscle	0.25	4.47

Goat tissues and milk were homogenised and uniformly mixed with Lichroprep® 25–40 µm silica, then loaded into a column subjected to online (milk and liver) or off-line (all other matrices) extraction and HPLC analysis using gradient elution with 50 mM ammonium acetate in distilled water adjusted to pH 3 and 50 mM ammonium acetate in methanol, with UV (254 nm) and radio detection with fraction collection. For the off-line extractions, homogenates of muscle and kidney were mixed with the Lichroprep silica, with fat homogenates additionally being mixed with DMSO prior to extraction. An analytical scale of the HPLC method was used for determination of the metabolite profile, with a preparative scale of the same method being used for isolation of the metabolites from the liver of goats from the high dose group. Liver was chosen as it contained the highest amount of total radioactive residue, as well as the greatest number of residue components. The isolated metabolites were further purified using thin layer chromatography and a second HPLC-UV-radio-detection method with fraction collection using 5 g/L ammonium bicarbonate in water and methanol as the mobile phases. Metabolites were identified using a combination of comparison of the HPLC chromatograms with authentic compounds, ¹H NMR, and mass spectrometry.

The extraction efficiencies of the on-line HPLC method for the matrices are presented in Table 12 below, together with the composition of the residue in each matrix.

The column extraction methods were effective, with 92% of TRR extracted from liver, 94% from milk, 103% from kidney, 110% from muscle and 121% from fat. The high figures for muscle and fat were attributed to uncertainties in the measurement of small amounts of radioactivity in large extraction volumes.

2,6-Dichlorobenzamide was present in all matrices and was the largest component of the residue in all matrices except liver. Metabolism was most extensive in liver, with three sulphur containing compounds identified, which appear to be degradates of a cysteine conjugate of 2,6-dichlorobenzamide. The largest component of the residue in liver was 6-chloro-3-hydroxy-2-thiobenzamide, present at 6.6 mg eq./kg, or 70% of the TRR. The dichlobenil-cysteine conjugate metabolites 6-chloro-3-hydroxy-thiobenzamide, 6-chloro-3-hydroxy-2-methylthiobenzamide, and 6-chloro-3-hydroxy-2-methylsulfinylbenzamide were found in varying proportions in all matrices. Small amounts of a metabolite found only in muscle and fat were postulated to be dichlobenil on the

basis of matching chromatographic retention times with a standard. However, this assignment should be regarded as tentative, as no structural confirmation was carried out using mass spectrometry or nuclear magnetic resonance.

Cysteine conjugation, together with hydroxylation at the 3-position, followed by cleavage of the cysteine side chain and oxidation of the sulphur therefore forms the main metabolic pathway for 2,6-dichlorobenzamide in goats.

Table 12 Identification of the residues in matrices of goat dosed with 13 ppm 2,6-dichlorobenzamide

Component	Liver		Kidney		Muscle		Fat		Milk (72 h)	
	%TRR	mg eq./kg	%TRR	mg eq./kg		mg eq./kg		mg eq./kg		mg eq./kg
2,6-Dichlorobenzamide	0.98	0.09	63	0.75	77	0.19	40	0.009	80	0.038
Unknown	2.2	0.21	-	-	2.5	0.006	-	-	-	-
6-Chloro-3-hydroxy-2-thiobenzamide	70	6.6	21	0.25	-	-	1.6	< 0.001	13	0.006
6-Chloro-3-hydroxy-2-methylthiobenzamide	17	1.6	19	0.23	-	-	-	-	-	-
6-Chloro-3-hydroxy-2-methylsulfinylbenzamide	1.7	0.16	-	-	4.4	0.01	1.5	< 0.001	-	-
Dichlobenil (tentative)	-	-	-	-	4.4	0.01	24	0.005	-	-
% Extracted	92	9.6	103	1.2	110	0.28	121	0.029	93	0.045
TOTAL	100	9.41	100	1.19	100	0.25	100	0.024	100	0.048

Laying hens: 2,6-dichlorobenzamide

Metabolism of ^{14}C -phenyl labelled 2,6-dichlorobenzamide was investigated in laying hens (Burke, 1994b and Ruijten *et al.*, 1994b). A group of five hens was dosed with ^{14}C -2,6-dichlorobenzamide daily for 5 days at a target dose of 10 ppm in feed on a dry matter basis. A second group of five birds was given an exaggerated dose at a target level of 200 ppm in feed daily for 3 days to provide additional samples to facilitate identification of residues. Finally, a single untreated control hen was kept in order to provide material for determination of analytical method LODs and LOQs.

The hens were kept in metabolism cages during the feeding phase of the study. Fresh drinking water was available *ad libitum*, while measured quantities of feed (commercially available diet and grit) were provided each morning and afternoon, with feed consumption being monitored. The dose was administered orally using gelatin capsules. Actual dose levels ranged from 11–14 ppm (mean = 12 ppm), or 0.78–0.92 mg/kg bw/day (mean = 0.86 mg/kg bw/day) and 218–249 ppm (mean value = 236 ppm), or 15–19 mg/kg bw/day (mean value = 17 mg/kg bw/day) for the low and high dose birds respectively. No overt signs of toxicity were observed during the study, and the dosing did not have any adverse effects on the weight of the animals, feed intake, or egg production.

Excreta, and cage debris if required, were collected at 24 hour intervals, with eggs being collected twice daily and pooled for each 24 hour period. Whole eggs were pooled for the low dose birds, with yolks and white being pooled separately for the high dose birds. The cages were rinsed after each collection of excreta and the cage wash was collected. The low dose birds were sacrificed approximately 23 hours after the final dose, while the high dose birds were sacrificed around 4 hours following the final dose. Samples of skeletal muscle (composite of breast and thigh), fat (peritoneal), skin with adhering subcutaneous fat, liver, and kidney were collected. The tissue were macerated and sub-sampled prior to frozen storage, while the egg and excreta samples were homogenised and sub-sampled, the excreta samples after addition of a minimum volume of water, then stored frozen.

Samples of excreta, whole eggs, egg yolks, and tissues were analysed for total radioactivity by liquid scintillation counting (LSC), after combustion and capture of the liberated $^{14}\text{CO}_2$. Cage wash and egg whites were added directly to scintillant solutions and LSC was conducted without combustion.

In the low dose hens, 49% of the administered dose was recovered in excreta, 1.5% in cage wash, 3.7% in eggs, and 23% in tissues, with a total recovery of 77%. In the high dose birds, 15% was recovered in excreta, 1.8% in eggs and 30% in tissues (total recovery of 47%).

A good recovery of the total administered radioactive dose was achieved for the low dose birds, while recovery is poor for the high dose group. It is noted that the digestive tract and contents were not analysed, and a significant proportion of the radioactivity could be present there, especially given that sacrifice of the high dose birds took place only 4 hours after the final dose.

Table 13 Total radioactive residues in eggs

Sampling interval (hours)	TRR (mg eq./kg)		
	Low dose birds, whole eggs	High dose birds	
		Egg yolk	Egg white
0-24	0.41	8.35	6.97
24-48	1.01	13.5	10.9
48-52	NA	28.8	21.9
48-72	1.90	NA	NA
72-96	2.09	NA	NA
96-120	2.36	NA	NA

NA = not applicable.

Total radioactive residue concentrations in eggs do not appear to have reached a plateau by the end of the dosing phase, although it is noted that the rate of increase in the whole egg samples from the low dose birds is slowing after three days, indicating that the levels are approaching a plateau.

Table 14 Total radioactive residues in hen tissues

Tissue	Low dose birds, mg eq./kg	High dose birds, mg eq./kg
Fat	1.29	18.0
Skin with attached fat	1.85	25.1
Kidney	5.03	40.4
Liver	8.58	55.3
Muscle	1.93	32.9

Tissues and eggs were homogenised and uniformly mixed with Lichroprep® 25-40 µm silica, then loaded into a pre-column subjected to inline extraction and HPLC analysis using gradient elution with 50 mM ammonium acetate in distilled water adjusted to pH 3 and 50 mM ammonium acetate in methanol, with UV (254 nm) and radio detection with fraction collection. An analytical scale of the HPLC method was used for determination of the metabolite profile, with a preparative scale of the same method being used for isolation of the metabolites from liver from the high dose group of hens, which contained the largest amount of radioactive residue for characterisation. The isolated metabolites were further purified using a second HPLC-UV-radio-detection method with fraction collection using 5 g/L ammonium bicarbonate in water and methanol as the mobile phases. Metabolites were identified using a combination of comparison of the HPLC chromatograms with standard chromatograms, ¹H NMR, and mass spectrometry.

The extraction efficiencies of the on-line HPLC method for the matrices are tabulated below, together with the composition of the residue in each matrix. The column extraction methods were essentially quantitative, with 95% of the TRR in kidney extracted, while >99% was extracted from liver, skin, muscle, fat and eggs.

Table 15 Extraction yields and composition of the radioactive residues from hen matrices (low dose birds)

Matrix	% Recovery by extraction and HPLC	2,6-Dichlorobenzamide		Unknown	
		mg eq./kg	%TRR	mg eq./kg	%TRR
Liver	99.3	8.52	99.3	-	-
Kidney	94.6	4.76	94.6	-	-
Skin	99.4	1.84	99.4	-	-
Muscle	99.6	1.87	96.9	0.053	2.7
Fat	99.6	1.28	99.6	-	-
Egg (96 hours)	99.9	1.97	94.3	0.12	5.6

The only component of the residue identified was unchanged 2,6-dichlorobenzamide, with the exception of muscle and egg, in which a single unidentified component was found, comprising 3% and 6% of the radioactivity respectively. 2,6-Dichlorobenzamide is essentially not metabolised by hens, and is largely excreted unchanged.

Overview of metabolism of 2,6-dichlorobenzamide in animals

2,6-Dichlorobenzamide is metabolised in goats in a similar manner to dichlobenil, with the principal metabolic pathway being conjugation with cysteine (replacing one of the chlorine atoms), accompanied by hydroxylation of an adjacent position on the phenyl ring. Subsequently, the cysteine side chain is cleaved and oxidised, giving residues such as 6-chloro-3-hydroxy-2-methylthiobenzamide, 6-chloro-3-hydroxy-2-thiobenzamide, and 6-chloro-3-hydroxy-2-methylsulfinylbenzamide.

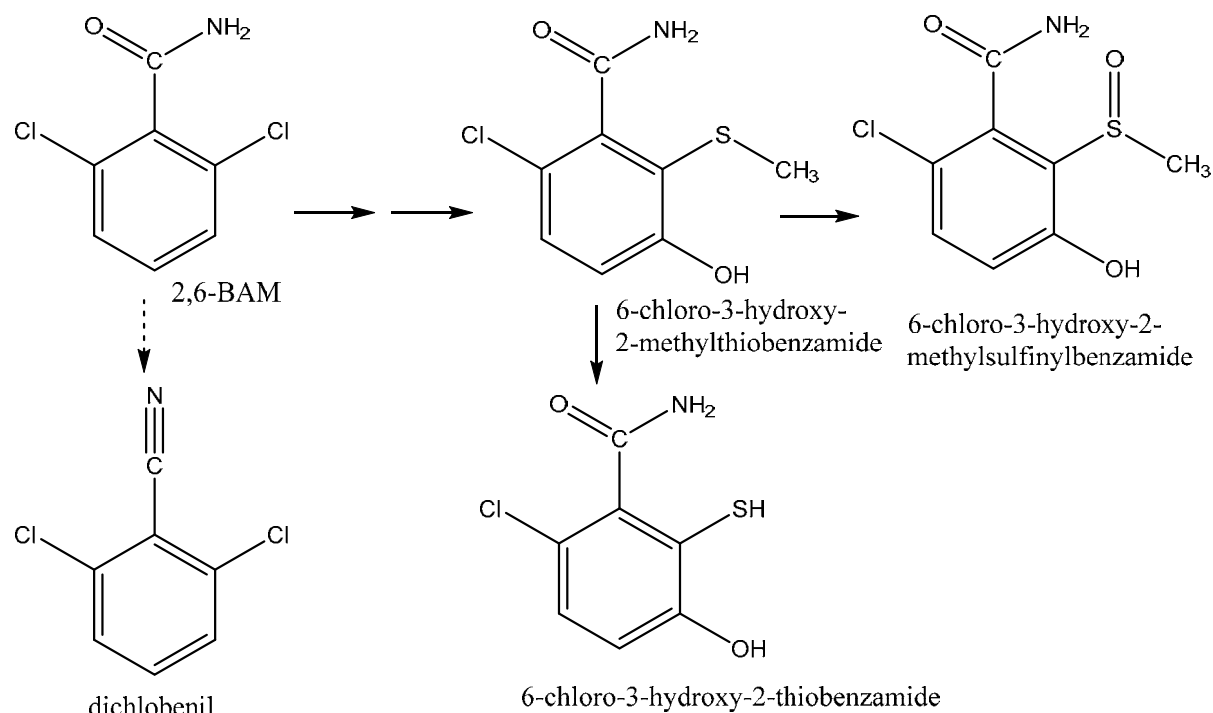


Figure 3 Metabolism of 2,6-dichlorobenzamide in goats

By contrast, 2,6-dichlorobenzamide appears to be excreted largely unchanged by laying hens, with only one other component observed (but not identified) at low levels (3-6% of the TRR) in muscle and eggs.

Plant metabolism

The Meeting received studies on the metabolism of dichlobenil in apples and grapes.

Apples

Small outdoor plots of one apple tree each (Gravenstein variety) were treated with ^{14}C -dichlobenil prepared as a liquid formulation at 6.72 kg ai/ha by a single soil application in mid-February (late dormancy; flowering was noted 20 days after application) (Hubert, 1990b). The total radioactive residues in soil and fruit samples were determined by combustion and liquid scintillation counting (LSC) analysis. Homogenised apple was extracted with methanol. Radioactivity in the post-extraction solids was quantified by combustion and LSC. The methanol extract was concentrated and the resulting solid residue filtered out and rinsed then sonicated with methanol. Residues in the methanol extract were further characterised using separate partitions of portions of the extract with ether and toluene. Radioactivity in the various methanol extracts and ether and toluene partitions were analysed by LSC, with residues in the extracts containing sufficient residue (the ether and toluene partitions) being identified with the aid of reference standards using HPLC-UV with fraction collection and LSC, and TLC.

Table 16 Total radioactive residues in apple and soil samples

DALA	Matrix	TRR (mg eq./kg)
-0	Soil 0-7.6 cm	ND
	Soil 7.6-15.2 cm	ND
0	Soil 0-7.6 cm	1.292
	Soil 7.6-15.2 cm	ND
30	Soil 0-7.6 cm	0.494
	Soil 7.6-15.2 cm	ND
77	Soil 0-7.6 cm	0.486
	Soil 7.6-15.2 cm	0.098
107	Soil 0-7.6 cm	0.746
	Soil 7.6-15.2 cm	0.166
137	Soil 0-7.6 cm	0.601
	Soil 7.6-15.2 cm	0.166
77	Fruit	0.012
107	Fruit	0.028
137	Fruit	0.042

The greater amount of the soil residue was found in the upper sections of the cores, consistent with the volatility of dichlobenil. Total residues in mature fruit were low, at 0.042 mg eq./kg. Most of the residue was extracted with methanol, at 81% of the TRR (0.034 mg eq./kg). Around 86% of the methanol extracted radioactivity partitioned into ether, while around 73% partitioned into toluene.

Table 17 Characterisation and identification of residues in apples collected at normal harvest (using the results from the ether partition)

Component/extract	Residue	
	mg eq./kg	%TRR
2,6-Dichlorobenzamide	0.024	56.5
Unidentified residue components in the ether partition	< 0.01 (0.0055)	13.2
Aqueous partition	< 0.01 (0.0057)	16.7
Unextracted residue	< 0.01 (0.0045)	10.7
Sum of identified and extracted/characterised residues	0.039	97.1
Total radioactive residue	0.042	100

The only metabolite identified in apples was 2,6-dichlorobenzamide, at 0.024 mg eq./kg, or 57% of the TRR. Other unidentified components were present at < 0.01 mg eq./kg.

Grapes

A single soil application of ^{14}C -dichlobenil was made to small plots (two plants per plot) of outdoor grown Emperor variety grape vines in mid-February (late dormancy) using a liquid formulation at 6.72 kg ai/ha (Hubert, 1990a). Soil and fruit samples were frozen and homogenised and the total radioactive residues determined by combustion radio-analysis. Homogenised grape samples were

extracted four times with methanol and the extracts combined. The methanol extract was reduced to an aqueous residue and partitioned with dichloromethane. HPLC and TLC analyses were conducted on the aqueous and organic phases. Further characterisation of the aqueous phase was carried out by acid and enzyme (β -glucosidase) hydrolysis followed by LSC analysis.

Table 18 Total radioactive residues in grape and soil samples

DALA	Matrix	TRR (mg eq./kg)
0	Soil 0-7.6 cm	7.924
	Soil 7.6-15.2 cm	ND
61	Soil 0-7.6 cm	1.838
	Soil 7.6-15.2 cm	0.210
159	Soil 0-7.6 cm	0.160
	Soil 7.6-15.2 cm	0.616
190	Soil 0-7.6 cm	0.389
	Soil 7.6-15.2 cm	0.139
222	Soil 0-7.6 cm	0.507
	Soil 7.6-15.2 cm	0.216
159	Immature fruit	0.322
190	Immature fruit	0.286
222	Mature fruit	0.357, 0.392*

*A second combustion radio-analysis of the mature grapes was conducted at the time of extraction and characterisation of the residues, and the %TRRs are based on this second, higher figure.

Nearly all the residue in mature grapes (99% or 0.39 mg eq./kg) was extractable with methanol, with only 7.1% (0.03 mg eq./kg) remaining in the post-extraction solids (PES). Due to the low residue levels, no further characterisation was conducted for the PES. Around 81-85% of the total residue was found in dichloromethane after the initial neutral partition, with a much smaller amount (1.8%) found in dichloromethane after the second partition under acidic conditions.

Table 19 Characterisation and identification of residues in mature grapes

Component/extract	Residue	
	mg eq./kg	%TRR
2,6-Dichlorobenzamide	0.34	86.7
2,6-Dichloro-4-hydroxybenzamide	< 0.01	2.1
Unidentified residue components	0.06	15.3
Unextracted residue	0.03	7.2
Sum of identified and characterised residues	0.43	109.7
Total radioactive residue	0.39	100

The major component of the residue in grapes was 2,6-dichlorobenzamide, which was found at 0.34 mg eq./kg (86.7% of the TRR), when summing the amounts found in the neutral dichloromethane partition and the amounts tentatively identified in other fractions. A trace amount (< 0.01 mg eq./kg) of 2,6-dichloro-4-hydroxybenzamide was tentatively identified in the aqueous partition after acid hydrolysis of the initial aqueous partition.

Plant metabolism summary

The metabolism of dichlobenil in fruit crops is straightforward, with the only significant transformation being hydrolysis to give 2,6-dichlorobenzamide, which comprised 57% of the TRR in apples and 87% of the TRR in grapes. A very minor component of the residue in grapes was 2,6-dichloro-4-hydroxybenzamide, indicating some hydroxylation of the phenyl ring was taking place. 2,6-Dichlorobenzamide is a significant environmental metabolite, indicating that the transformation of the nitrile to the amide may be taking place in soil, with the 2,6-dichlorobenzamide then being taken up by the roots, rather than the hydrolysis taking place within the plant. However, this cannot be confirmed without further data, such as plant metabolism studies for 2,6-dichlorobenzamide itself.

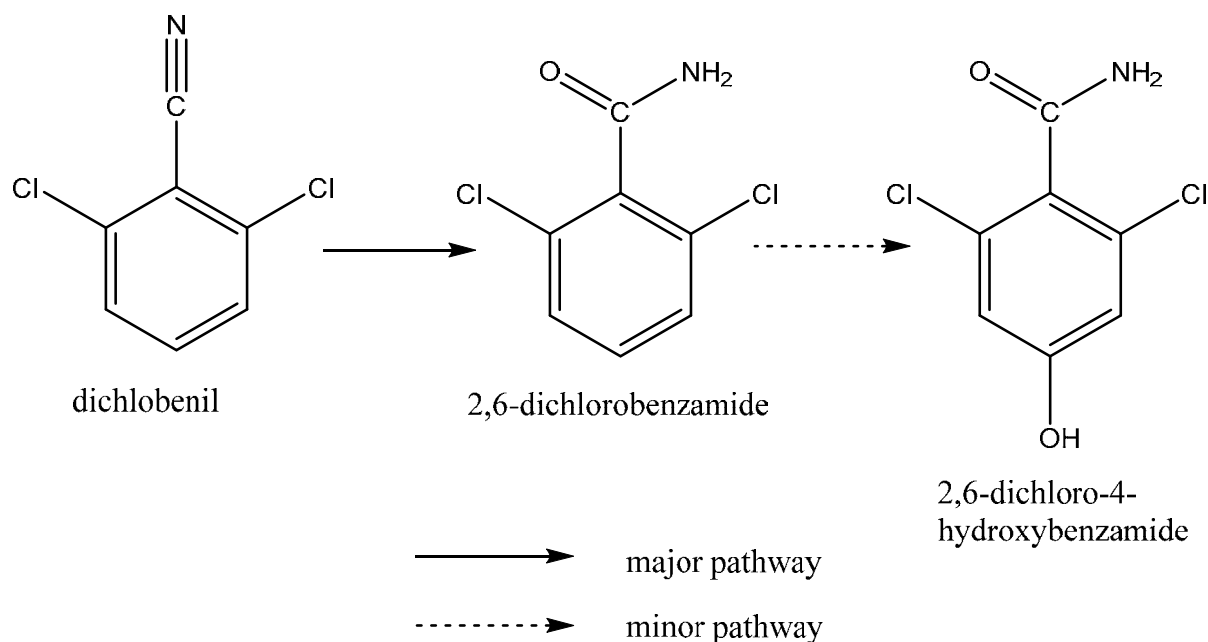


Figure 4 Metabolism of dichlobenil in fruit crops

Environmental fate

Data on aerobic soil metabolism, aqueous photolysis, soil surface photolysis, and aerobic and anaerobic hydrolysis of dichlobenil, and hydrolysis and aqueous photolysis of the soil and plant metabolite 2,6-dichlorobenzamide were supplied.

Based on the environmental data requirements in the 2009 FAO Manual and considering the use pattern of dichlobenil (a soil applied herbicide), the soil metabolism, soil surface photolysis and hydrolysis studies are considered pertinent to the compound. Soil studies are evaluated below, while the results of the hydrolysis studies are summarised in Tables 1a and 1b above.

Aerobic soil metabolism: dichlobenil

Sandy loam soil samples were spiked with ^{14}C -dichlobenil at 2.1 mg/kg (Walstra *et al.*, 1989) and incubated in the dark at 24 °C for intervals up to 50 weeks. The biometer sample flasks were equipped with a trap filled with dilute aqueous potassium hydroxide for capturing CO_2 and other volatiles.

Soil samples were extracted twice by homogenising with methanol and twice by refluxing with methanol. The combined extracts were analysed by LSC and HPLC, with the unextracted residues determined by combustion and LSC. Radioactivity in the KOH traps was determined by LSC both before and after partition with dichloromethane. Some of the dichloromethane partitions were also analysed by HPLC.

The composition of the residues extractable from soil and captured in the traps is tabulated below.

Table 20 Degradation of ^{14}C -Dichlobenil in sandy loam soil under aerobic conditions

Sampling interval (weeks)	Volatiles (%TAR)			Extracted (%TAR)				Bound (%TAR)	Total Recovery (%TAR)
	Total	$^{14}\text{CO}_2$	dichlobenil	Total	dichlobenil	2,6-dichloro-benzamide	Other metabolites		
0	-	-	-	101	101	< 0.1	< 0.1	< 0.2	101
2	14.8	0.1	13.3	80.7	80.3	0.4	< 0.1	2.2	97.8
5	19.0	0.7	18.0	74.2	71.7	0.8	0.5	5.1	98.2
10	39.2	0.7	37.2	52.9	49.8	1.3	< 0.1	5.8	97.8
20	46.0	1.9	41.9	44.4	35.6	7.3	1.2	8.2	98.5

Sampling interval (weeks)	Volatiles (%TAR)			Extracted (%TAR)				Bound (%TAR)	Total Recovery (%TAR)
	Total	¹⁴ CO ₂	dichlobenil	Total	dichlobenil	2,6-dichloro-benzamide	Other metabolites		
30	63.9	1.5	61.0	26.8	20.6	5.2	< 0.6	8.2	98.9
40	62.3	2.7	58.5	26.1	16.2	8.4	0.5	8.0	96.4
50	61.0	2.3	56.8	26.7	11.6	13.1	0.7	10.2	97.9

Over half the applied dichlobenil was observed to dissipate via evaporation due to the high volatility of dichlobenil, mostly as parent compound (up to 61% of the TAR and up to 96% of the volatilised residue), with a small amount ($\leq 2.7\%$ of the TAR) being recovered as ¹⁴CO₂. At 2 weeks incubation, over 99% of the radioactivity extractable from soil was parent compound together with a trace (0.5% of the extractable soil residues) of 2,6-dichlorobenzamide. At 50 weeks, the proportion of dichlobenil had declined to 45% of the soil extractable residues, while 2,6-dichlorobenzamide had risen to 50%. Smaller amounts of the applied radioactivity were bound to the soil (2.2% of the TRR after 2 weeks, rising to 10.2% at 50 weeks).

The calculated DT₅₀ for dichlobenil in sandy loam soil is 13 weeks. Most of the loss of dichlobenil in soil is due to volatilisation rather than metabolism.

A second laboratory study of the aerobic degradation of soil (Nimmo, 1985) considered the fate of phenyl ¹⁴C-dichlobenil in several soil types, with both sterilised and unsterilised samples. ¹⁴C-Phenyl-dichlobenil was uniformly mixed into soil at a target of 1.8 mg/kg. The samples were stored in stoppered Erlenmeyer flasks in the dark at 20 °C. To determine the effect of soil microbes on degradation, some soil samples were sterilised in an autoclave, with the fortification solution being micro-filtered and added to the soil under aseptic conditions. Samples were extracted using ethanol/water, initially at room temperature and then under reflux, and the extracts analysed by LSC. The extracts were isotopically diluted with dichlobenil, 2,6-dichlorobenzamide and 2,6-dichlorobenzoic acid, the components separated by recrystallization then analysed by LSC and TLC.

Degradation of dichlobenil, and formation of the metabolites 2,6-dichlorobenzamide and 2,6-dichlorobenzoic acid is shown in the table below for the unsterilised soil samples. Only trace amounts of 2,6-dichlorobenzoic acid were observed (maximum of 0.4% of applied dose), while 2,6-dichlorobenzamide was a much more significant metabolite at up to 57% of the applied dose. The half lives of dichlobenil in the six soil types tested ranged from 4 to 30 weeks.

Table 21 Degradation of dichlobenil in unsterilized soils (percentages of applied dose)

Incubation time (weeks)	Compound	Light clay (pH 6.3, OM 5.9%)	Clay loam (pH 5.6, OM 1.9%)	Silty clay loam (pH 6.7, OM 3.3%)	Sandy peat (pH 5.1, OM 24.4%)	Sandy clay (pH 7.5, OM 1.3%)	Calcareous clay (pH 7.8, OM 1.2%)
0	Parent	94	101	93	95	97	100
2-3	Parent	84	79	70	86	54	91
6-7	Parent	75	65	33	62	28	71
	BAM	-	-	57	-	-	27
	DCBA	-	-	-	-	-	0.2
12-14	Parent	60	39	21	59	-	46
	BAM	31	-	-	41	-	48
	DCBA	0.4	-	-	0.4	-	-
18	Parent	51	-	13	41	-	28
	BAM	-	-	-	-	-	-
	DCBA	-	-	0.4	-	-	-
26	Parent	43	-	-	31	-	-
36	Parent	-	-	-	-	-	19
52	Parent	29	-	-	21	-	-

BAM = 2,6-Dichlorobenzamide. DCBA = 2,6-dichlorobenzoic acid.

Degradation of dichlobenil in the sterilised soil samples was essentially zero, strongly suggesting that soil microbes were largely responsible for the degradation of dichlobenil.

Table 22 DT₅₀ values (by soil type)

Soil type	DT ₅₀ (weeks)
Light clay	30
Clay loam	11
Silty clay loam	6.2
Sandy peat	24
Sandy clay	3.8
Calcareous clay	14

This study is not considered fully representative of the expected fate of dichlobenil in soil under field conditions as the samples were stored in stoppered flasks, preventing losses of parent by volatilisation which was the major route of dissipation in the first study (Walstra *et al.*, 1989).

Soil surface photolysis: dichlobenil

The photolytic fate of ¹⁴C-phenyl dichlobenil (Kabler and Carpenter, 1990) coated on irradiated soil was studied. Soil samples (sandy loam) were fortified with radio-labelled dichlobenil at a concentration of 10 mg/kg and placed in glass photolysis chambers equipped with water cooled jacketing, traps to capture volatiles, and a constant flow through of CO₂ free water saturated air was maintained. The samples were constantly irradiated using a xenon arc UV lamp at an intensity that resulted in irradiation over a 24-hour period equivalent to a solar day at the spring equinox at 40° N latitude. A number of samples were not irradiated and were wrapped with aluminium foil in order to act as dark controls. Samples of soil were analysed at intervals from 0 to 30 days.

Soil samples were extracted with methanol and the extracts and trapping solutions were analysed using LSC and HPLC. Radioactivity remaining in the extracted soil samples was determined by combustion and LSC.

For the irradiated samples, a DT₅₀ of 50 days was calculated, while for the dark control samples, a much shorter DT₅₀ of 4.7 days was determined.

Given that the calculated DT₅₀ for the irradiated samples were much longer than those for the dark control samples, this study is not considered to be reliable and no conclusion on aqueous photolysis of dichlobenil on the soil surface can be reached.

Aerobic soil metabolism: 2,6-dichlorobenzamide

Studies on metabolism of 2,6-dichlorobenzamide in soil were not provided. However, the EFSA evaluation of dichlobenil reported in-field DT₅₀ values of 73–257 days for 2,6-dichlorobenzamide calculated from field dissipation studies in fluopicolide, of which 2,6-dichlorobenzamide is also a metabolite (European Food Safety Authority, 2010). Most losses were through leaching. DT₅₀ values of 186–261 days were reported for laboratory experiments with 2,6-dichlorobenzamide in Finnish topsoil samples (Pukkila and Kontro, 2014).

Environmental metabolism summary

Dichlobenil is lost from soil in significant quantities by volatilisation, and is also metabolised in soils, mainly to 2,6-dichlorobenzamide. A laboratory DT₅₀ value of 13 weeks was determined for sandy loam soil. Dichlobenil was stable to hydrolysis in buffer solutions.

The key plant and soil metabolite of dichlobenil, 2,6-dichlorobenzamide, was stable to hydrolysis. It is more persistent in soil than dichlobenil, with laboratory DT₅₀ values of up to 261 days.

Field dissipation studies for dichlobenil and 2,6-dichlorobenzamide were not available to the Meeting.

Residues in succeeding crops

Uses considered are only for permanent crops and no studies on residues of dichlobenil in rotational crops were made available to the Meeting.

RESIDUE ANALYSIS

Methods of residue analysis

Details of validated analytical methods were supplied for the determination of dichlobenil in plant and animal matrices.

Plant matrices

A GC-ECD method (method number L 3-53-71) was developed and validated for determination of 2,6-dichlorobenzamide in berry fruits, stone fruits and hazelnuts (Ver Hey, 1990). Samples were homogenised with anhydrous sodium sulphate and filter pulp, then extracted with ethyl acetate. An aliquot of the ethyl acetate extract was filtered, evaporated to dryness, and reconstituted in 1:9 v/v acetone/petroleum ether, and cleaned up by solid phase extraction using neutral alumina. The residue was eluted from the column using 3:17 v/v ethanol/petroleum ether, evaporated to dryness, and reconstituted in ethyl acetate, before analysis by GC-ECD. An additional cleanup by partition between acetonitrile and hexane, with the hexane phase being discarded, was incorporated prior to the solid phase extraction step for hazelnut samples to remove oily contaminants.

Quantification was achieved by linear regression using the external standard method. The method linearity was good, with an r^2 value of 0.9978 over a concentration range 1–100 µg/L, or 0.008–0.8 mg/kg in samples (depending on the dilution factor). The method LOQ is 0.01 mg/kg. Method recoveries and precision were generally good, with all recoveries except for those in hazelnuts and prunes in the range 70–120%, and RSD values all <20%.

Table 23 Recovery of 2,6-dichlorobenzamide from fruit and nut matrices after GC-ECD analysis (Ver Hey, 1990)

Matrix	Fortification (mg/kg)	Recovery (%)
Blackberries	0.01	87.0
	0.05	95.8
	0.50	96.2
Blueberries	0.01	94.9
	0.05	90.4
	0.50	94.1
Cranberries	0.01	100.9
	0.05	90.5
	0.50	93.5
Hazelnuts	0.01	64.3
	0.05	58.6
	0.50	68.7
Plums	0.01	103.2
	0.05	97.8
	0.50	93.9
Peaches	0.01	92.2
	0.05	96.4
	0.50	98.2
Raspberries	0.01	104.0
	0.05	97.7
	0.50	98.0

Table 24 Recovery of 2,6-dichlorobenzamide from fruit and nut matrices after GC-ECD analysis using method L-3-53-71 (recoveries from residue trials)

Matrix	Fortification (mg/kg)	Recovery (%)	Reference
Grapes	0.01	92, 93	Chickering, 1991
	0.03	101, 106	
	0.10	94, 96	
	0.30	97, 98	
	1.0	88, 93	
Grape juice	0.01	97	Ver Hey, 1992d
	0.03	86	
	0.05	88	
	0.50	81	
Grape wet pomace	0.01	106	
	0.03	81	
	0.05	91	
	0.50	89	
Grape dry pomace	0.01	117	
	0.03	93	
	0.05	91	
	0.50	93	
Raisins	0.01	104	
	0.03	84	
	0.05	98	
	0.50	91	
Raisin waste	0.05	101	
	0.10	99	
	0.30	98	
	0.50	96	
Plums	0.01	103	Hamilton, 1993
	0.05	88	
	0.50	91	
Prunes	0.01	63	
	0.03	68	
	0.05	74	
	0.50	82	
Raspberries	0.01	91.9, 94.0	Dykstra, 1991c
	0.10	68.9, 95.8	
	0.50	84.0, 97.7	

A second method (study number 2005-046) using GC-ECD and GC-MS/MS as alternative analytical techniques was validated for analysis of both dichlobenil and 2,6-dichlorobenzamide in grapes (Bacher, 2005). Separate extractions and analyses were conducted for the two analytes.

For determination of dichlobenil, grape samples were macerated with anhydrous sodium sulphate and 1:4 v/v ethyl acetate/hexane, then filtered. The extraction was repeated twice more and the extracts were filtered and combined. To an aliquot of the combined extracts, a small amount of 1% decanol in acetone was added, prior to concentration by rotary evaporation using a water bath at 40 °C. Hexane was added, and the volume was reduced again before addition of a second volume of hexane and further evaporation prior to transfer to a sample tube and making up to volume with hexane. The extract was then cleaned up by solid phase extraction using 5% deactivated neutral alumina. The column was rinsed with hexane, with these eluates being discarded, prior to elution of the residue with 2% acetone in hexane. The eluted residue was transferred to an evaporation flask, and a small amount of 1% decanol in acetone was added before concentration by rotary evaporation at 40 °C, prior to adjustment of the extract to volume with hexane. The cleaned up extract was analysed by GC-MS/MS (detection transition was 171 → 136 for dichlobenil, and 173 → 145 for 2,6-dichlorobenzamide), or GC-ECD.

For determination of 2,6-dichlorobenzamide, samples were macerated with ethyl acetate, anhydrous sodium sulphate, and filter pulp. After centrifuging and filtration, an aliquot of the extract

was evaporated to dryness by rotary evaporation followed by a gentle stream of nitrogen, and reconstituted in 1:9 v/v acetone/petroleum ether. The extract was cleaned up by solid phase extraction with 2% deactivated alumina. After loading the extract onto the column, the flask was rinsed with 1:9 v/v acetone/petroleum ether and the rinses added to the column. The initial eluates were discarded, and the 2,6-dichlorobenzamide residues were eluted with 3:17 v/v ethanol/petroleum ether, evaporated to dryness by rotary evaporation at 40 °C followed by a gentle stream of nitrogen, then re-dissolved in ethyl acetate. The cleaned up extract was analysed by GC-MS/MS or GC-ECD.

The method LOQ is 0.01 mg/kg for both analytes. The method linearity was good, with r^2 values >0.99 for both analytes using GC-MS/MS. Linearity was less good for GC-ECD, although r^2 values were >0.98. Method accuracy and precision in grapes were good, with recoveries all in the range 70–120%, and RSD values all <20%.

Table 25 Recovery of dichlobenil and 2,6-dichlorobenzamide from grapes (method 2005-046)

Analyte	Fortification (mg/kg)	n	Recovery, GC-MS/MS determination (%)		Recovery, GC-ECD determination (%)	
			Range	Mean $\pm$ RSD	Range	Mean $\pm$ RSD
Dichlobenil	0.01	5	86-110	97 $\pm$ 8.9	89-109	100 $\pm$ 8.2
	0.10	5	82-102	91 $\pm$ 9.1	82-98	89 $\pm$ 6.5
2,6-Dichlorobenzamide	0.01	5	106-111	109 $\pm$ 1.9	103-109	106 $\pm$ 2.0
	0.10	5	91-104	98 $\pm$ 5.0	95-105	102 $\pm$ 3.9

Table 26 Method validation recovery data for method 2005-046 when used for determination of dichlobenil and 2,6-dichlorobenzamide in raw grapes and processed grape commodities (Maselli, 1998)

Matrix	Analyte	Fortification level	Recoveries (%)
Method validation recoveries			
Grapes	Dichlobenil	0.02	70, 76, 84, 88, 90, 90 (mean = 83, RSD = 8.5%)
		0.10	85, 86, 86, 90, 92, 93 (mean = 89, RSD = 0.7%)
		0.20	80, 83, 84, 92, 96, 97 (mean = 89, RSD = 2.3%)
	2,6-Dichlorobenzamide	0.003	89, 99, 101, 106, 111, 118 (mean = 104, RSD = 6.2%)
		0.03	87, 95, 97, 98, 98, 102 (mean = 96, RSD = 5.5%)
		0.30	91, 92, 93, 93, 98, 99 (mean = 94, RSD = 1.1%)
Raisins	Dichlobenil	0.02	76, 77, 79, 79, 86, 89 (mean = 81, RSD = 1.9%)
		0.10	65, 70, 73, 75, 75, 75 (mean = 72, RSD = 5.6%)
		0.20	68, 70, 73, 75, 77, 78 (mean = 74, RSD = 3.4%)
	2,6-Dichlorobenzamide	0.03	83, 87, 87, 89, 89, 90 (mean = 88, RSD = 2.6%)
		0.15	95, 101, 101, 103, 103, 105 (mean = 101, RSD = 3.4%)
		0.30	96, 99, 99, 99, 99, 100 (mean = 99, RSD = 1.8%)
Grape juice	Dichlobenil	0.02	81, 81, 86, 86, 87, 89 (mean = 85, RSD = 3.4%)
		0.10	87, 90, 92, 92, 93, 93 (mean = 92, RSD = 2.8)
		0.20	88, 89, 92, 92, 93, 94 (mean = 91, RSD = 2.3%)
	2,6-Dichlorobenzamide	0.003	137, 138, 142, 146, 147, 147 (mean = 143, RSD = 1.9%)
		0.03	107, 108, 109, 112, 112, 113 (mean = 111, RSD = 0.9%)
		0.30	98, 99, 101, 101, 102, 110 (mean = 102, RSD = 1.5%)

Table 27 Concurrent and method validation recovery data for method based on number 2005-046 for determination of dichlobenil and 2,6-dichlorobenzamide in cherries

Matrix	Analyte	Fortification level	Recoveries	Reference
Concurrent fortification recoveries				
Sour cherry	Dichlobenil	0.08	70, 72, 92, 97 (mean = 83, RSD = 15%)	Maselli, 1996
		0.20	82, 95, 97, 101 (mean = 94, RSD = 8.7%)	
	2,6-Dichlorobenzamide	0.02	98, 99, 102, 104 (mean = 101, RSD = 2.1%)	
		0.20	90, 100, 100, 104 (mean = 99, RSD = 5.9%)	
Sweet cherry	Dichlobenil	0.08	77, 81, 84, 88, 90 (mean = 84, RSD = 4.2%)	Gaydosh, 1996
		0.20	81, 85, 87, 88, 91 (mean = 86, RSD = 3.5%)	
	2,6-Dichlorobenzamide	0.02	97, 100, 100, 120 (mean = 104, RSD = 1.7%)	
		0.20	97, 100, 103, 106 (mean = 102, RSD = 3.0%)	
Method validation recoveries				
Sweet cherries	Dichlobenil	0.02	84, 90, 90 (mean = 88, RSD = 3.9%)	Gaydosh, 1996
		0.04	88, 90, 94 (mean = 91, RSD = 3.4%)	
		0.20	82, 87, 89 (mean = 86, RSD = 4.2%)	
	2,6-Dichlorobenzamide	0.003	110, 111, 111 (mean = 111, RSD = 0.5%)	
		0.03	101, 111, 116 (mean = 109, RSD = 7.0%)	
		0.30	97, 97, 113 (mean = 102, RSD = 9.0%)	

Table 28 Concurrent and method validation recoveries for modified method 2005-046 for determination of dichlobenil and 2,6-dichlorobenzamide in berry fruits (Reibach, 2013)

Matrix	Analyte	Fortification level (mg/kg)	Recoveries (%)
Concurrent recoveries			
Raspberry	Dichlobenil	0.01	113, 186*
		0.20	75, 94
	2,6-Dichlorobenzamide	0.01	80, 111
		0.20	75, 90
Blueberry	Dichlobenil	0.01	82, 92
		0.10	88
		0.20	87
	2,6-Dichlorobenzamide	0.01	80, 127, 127
		0.20	48*, 95, 101, 118
Blackberry	Dichlobenil	0.01	76, 112
		0.20	95, 100
	2,6-Dichlorobenzamide	0.01	83, 118, 120
		0.20	81, 95, 102
Method validation recoveries			
Raspberry	Dichlobenil	0.01	99, 104, 106
		0.20	78, 90, 101
	2,6-Dichlorobenzamide	0.01	95, 111, 135
		0.20	77, 96, 103
Blueberry	Dichlobenil	0.01	91, 102, 102
		0.20	74, 81, 82
	2,6-Dichlorobenzamide	0.01	107, 108, 109
		0.20	86, 88, 91
Blackberry	Dichlobenil	0.01	86, 94, 96
		0.20	78, 84, 88
	2,6-Dichlorobenzamide	0.01	75, 79, 89
		0.20	94, 95, 96

*Rejected due to fortification error.

An independent validation of the method was conducted for both metabolites with no significant modifications to the extraction procedures, with analyses using both GC-ECD and GC-MS rather than GC-MS/MS (Wolf, 2006). Good recoveries were achieved in grapes (70–120%), with an LOQ of 0.01 mg/kg being confirmed for both analytes.

Animal matrices

A GC-MS method (96045) was developed and validated for determination of both dichlobenil and 2,6-dichlorobenzamide in animal matrices (Batorewicz, 1997).

Fat samples were extracted twice by homogenisation with hexane. The hexane extracts were then cleaned up by partitioning twice with acetonitrile, with the hexane being discarded and the acetonitrile phases combined and concentrated by rotary evaporation before addition of the internal standard, adjustment to volume and analysis.

Kidney, muscle and liver samples were homogenised with water and extracted twice with ethyl acetate. An aliquot of the combined ethyl acetate supernatants was evaporated to dryness and reconstituted in hexane. The hexane extract was then partitioned twice with acetonitrile, with the hexane being discarded and the combined acetonitrile extracts being washed with hexane. The combined acetonitrile extracts were evaporated to about 1 mL by rotary evaporation at 30–40 °C followed by a gentle stream of nitrogen. The internal standard was added and the sample adjusted to volume.

Milk samples were homogenised and then extracted twice with ethyl acetate. Combined aliquots of the ethyl acetate supernatants were evaporated, then reconstituted in hexane. The extract was then cleaned up by solid phase extraction (deactivated Florisil column). The residue was eluted with 1:1 v/v hexane/ethyl acetate and concentrated. The internal standard was added and the extract adjusted to volume.

Samples were analysed by GC-MS using 4-chlorobenzonitrile as an internal standard. Quantification was achieved by linear regression. The method LOQ is 0.01 mg/kg. Method linearity was good, with r^2 values >0.99 for dichlobenil and 2,6-dichlorobenzamide.

Table 29 Recovery of dichlobenil and 2,6-dichlorobenzamide from animal matrices

Analyte	Matrix	Fortification (mg/kg)	n	Recovery (%)	
				Range	Mean $\pm$ RSD
Dichlobenil m/z = 136, 171	Beef fat	0.01	3	73-87	79 $\pm$ 8.8
		0.05	3	67-74	71 $\pm$ 4.8
		0.10	3	70-74	73 $\pm$ 3.3
	Cattle kidney	0.01	3	77-96	89 $\pm$ 11
		0.05	3	62-79	72 $\pm$ 13
		0.10	3	86-90	88 $\pm$ 2.4
	Cattle liver	0.01	3	90-98	95 $\pm$ 4.2
		0.05	3	90-96	93 $\pm$ 3.0
		0.10	3	89-94	91 $\pm$ 2.6
	Beef muscle	0.01	3	81-89	84 $\pm$ 4.4
		0.05	3	72-84	79 $\pm$ 8.2
		0.10	3	80-82	81 $\pm$ 0.9
	Milk	0.01	3	65-76	72 $\pm$ 8.7
		0.05	3	79-85	82 $\pm$ 3.4
		0.10	3	80-83	82 $\pm$ 1.7
2,6-Dichlorobenzamide m/z = 173, 189	Beef fat	0.01	3	68-81	76 $\pm$ 8.7
		0.05	3	73-83	78 $\pm$ 6.3
		0.10	3	70-84	76 $\pm$ 9.4
	Cattle kidney	0.01	3	111-128	121 $\pm$ 7.2
		0.05	3	97-106	102 $\pm$ 4.6
		0.10	3	103-128	112 $\pm$ 13
	Cattle liver	0.01	3	92-110	98 $\pm$ 10
		0.05	3	75-87	81 $\pm$ 7.8
		0.10	3	111-122	115 $\pm$ 5.1
	Beef muscle	0.01	3	103-105	104 $\pm$ 1.0

Analyte	Matrix	Fortification (mg/kg)	n	Recovery (%)	
				Range	Mean $\pm$ RSD
	Milk	0.05	3	84-100	93 $\pm$ 9.0
		0.10	3	102-121	110 $\pm$ 9.0
		0.01	2	84-108	96
		0.05	3	88-97	92 $\pm$ 5.0
		0.10	3	91-112	104 $\pm$ 11

Multiresidue methods were not provided by the sponsor.

The USA FDA official multiresidue method PAM I has been successfully validated (complete recoveries, i.e. >80%) (US FDA, 1999). For dichlobenil parent compound this involved using Method 302 (E1-E3, DG1-19), corresponding to acetone extraction followed by solvent partition or removal of the aqueous component using diatomaceous earth, and GC analysis with a low temperature column (detector not specified) and Method 304 E1-E3 + C1-C4 + DG1-19 (extraction with acetone followed by solvent partition or removal of the aqueous component followed by solid phase extraction cleanup and GC analysis). Analysis of 2,6-dichlorobenzamide was successful using Method 302 (E1-E3, DG1-19).

EFSA reported good recoveries from unspecified high water content matrices for dichlobenil parent compound using a QuEChERS method (mean = 94%, RSD = 5.3%, n = 12 at 0.05 mg/kg fortification and mean recovery = 97%, RSD = 10%, n = 25 at 0.1 mg/kg fortification) (European Food Safety Authority, 2013).

Stability of pesticide residues in stored analytical samples

Plant matrices

No separate stability study was submitted for residues of dichlobenil or 2,6-dichlorobenzamide in plant matrices. However, stability was verified concurrently with the analyses of the samples in the residue trials.

Table 30 Storage stability data for dichlobenil and 2,6-dichlorobenzamide in sweet cherries at 0.20 mg/kg fortification (Gaydosh, 1996)

Analyte	Storage interval (months)	Stored recovery (%)	Concurrent recovery (%)
Dichlobenil	0	-	90
	0.5	73, 92, 95 (mean = 87, RSD = 14%)	73
	1	99, 105, 113 (mean = 106, RSD = 6.6%)	87
	2	74, 88, 88 (mean = 83, 9.7%)	98
	6 (197 days)	62, 76, 77 (mean = 72, RSD = 12%)	94
2,6-Dichlorobenzamide	0	-	95
	0.5	100, 101, 101 (mean = 101, RSD = 0.6%)	101
	1	93, 94, 95 (mean = 94, RSD = 1.1%)	98
	2	94, 101, 104 (mean = 100, RSD = 5.1%)	102
	8 (246 days)	88, 90, 92 (mean = 90, RSD = 2.2%)	100

The above data for sweet cherries is sufficient to confirm that the samples from the cherry residue trials, which were stored for a maximum of 198 days (dichlobenil analyses), and 251 days (2,6-dichlorobenzamide analyses) before analysis.

Table 31 Concurrent storage stability data for 2,6-dichlorobenzamide in stone fruit and berries (1990s trials)

Matrix	Fortification (mg/kg)	Storage interval for stability samples (days)	Maximum storage interval for trial samples	Stored recovery	Reference
Blackberries	0.01	25-26	35	93, 94	Dykstra, 1991b
	0.10			88, 92	
	0.50			89, 91	
Cranberries	0.01	75-87	89	92, 100	Ver Hey, 1992c
	0.05			78, 88	
	0.50			70, 91	
Grapes	0.10	112	141	93, 93, 95	Chickering, 1991
		119		87, 88, 94	
		119		98, 98, 98	
		133		88, 89, 91	
Grape juice	0.01	0	124-163*	88	Ver Hey, 1992d
		99		96	
	0.03	0		91	
		99		81	
	0.05	0		87	
		99		80	
	0.50	0		88	
		99		82	
	Wet pomace	0		98	
		108		92	
	0.03	0		92	
		108		85	
	0.05	0		90	
		108		91	
	0.50	0		90	
		108		93	
	Dry pomace	0.01		80	
		0.03		96	
		0.05		97	
		0.50		98	
Raisins	0.01	0		93	
		79		75	
	0.03	0		96	
		79		69	
	0.05	0		93	
		79		56	
	0.50	0		94	
		79		56	
Plums	0.01	29	39	76	Ver Hey, 1992b
	0.05			90	
	0.50			97	
Plums	0.01	35	37	88	Hamilton, 1993
	0.05			94	
	0.50			91	
Prunes	0.01	4	35	50	
	0.03			62	
	0.05			59	
	0.50			68	
Peaches	0.01	50-54	60	95, 114, 124, 130	Dykstra, 1991a
	0.10			90, 94, 97, 99	
	0.50			84, 88, 94, 97	
Peaches	0.01	52-59	60	90, 99	Ver Hey, 1992a
	0.05			95, 94	
	0.50			83, 84	
Raspberries	0.01	7-8	49	92, 94	Dykstra, 1991c

Matrix	Fortification (mg/kg)	Storage interval for stability samples (days)	Maximum storage interval for trial samples	Stored recovery	Reference
	0.10	40-41		124, 82	
		7-8		69, 96	
		40-41		88, 90	
	0.50	7-8		84, 98	
		40-41		89, 9e	

*See also the data in Table 32 below for grapes, raisins and juice.

With the exception of prunes, the data listed in Table 31 above is sufficient to verify the stability of 2,6-dichlorobenzamide residues in relevant stone and berry fruit matrices for the period the samples were stored between collection and analysis for the appropriate residue trials.

Table 32 Residue stability data for dichlobenil and 2,6-dichlorobenzamide in raw and processed grape matrices at a 0.10 mg/kg fortification (Maselli, 1998)

Analyte	Matrix	Storage interval (months)	Stored recovery (%)	Concurrent recovery (%)
Dichlobenil	Grapes	0	-	80, 91
		1	88, 90, 94 (mean = 91, RSD = 3.4%)	87, 89
		3	84, 86, 86 (mean = 85, RSD = 1.4%)	81, 86
		6	79, 79, 82 (mean = 80, RSD = 2.2%)	96, 100
		11 (323 days)	78, 85, 85 (mean = 83, RSD = 4.9%)	88, 90
	Raisins	0	-	79, 96
		1	73, 82, 86 (mean = 80, RSD = 8.3%)	80, 84
		3	69, 70, 74 (mean = 71, RSD = 3.7%)	100, 102
		6	55, 57, 60 (mean = 57, RSD = 4.4%)	97, 98
		9 (273 days)	61, 75, 76 (mean = 71, RSD = 12%)	78, 79
	Grape juice	0	-	83, 89
		1	89, 89, 91 (mean = 90, RSD = 1.3%)	81, 82
		3	78, 81, 86 (mean = 82, RSD = 4.9%)	94, 99
		6	68, 79, 85 (mean = 77, RSD = 11%)	96, 97
		11 (322 days)	78, 82, 83 (mean = 81, RSD = 3.3%)	83, 80
2,6-Dichlorobenzamide	Grapes	0	-	100, 101
		1	90, 91, 94 (mean = 92, RSD = 2.3%)	108, 108
		3	88, 89, 93 (mean = 90, RSD = 2.9%)	99, 101
		6	82, 92, 94 (mean = 89, RSD = 7.2%)	103, 103
		11 (345 days)	95, 99, 101 (mean = 98, RSD = 3.1%)	104, 111
	Raisins	0	-	97, 100
		1	105, 110, 112 (mean = 109, RSD = 3.3%)	70, 92
		3	97, 99, 100 (mean = 99, 1.5%)	100, 102
		6	101, 105, 108 (mean = 104, 3.3%)	93, 94

Analyte	Matrix	Storage interval (months)	Stored recovery (%)	Concurrent recovery (%)
			=105, RSD = 3.4%)	
		9 (266 days)	97, 104, 107 (mean = 103, RSD = 5.0%)	100, 110
	Grape juice	0	-	96, 98
		1	101, 101, 102 (mean = 101, RSD = 0.6%)	103, 111
		3	89, 92, 95 (mean = 92, RSD = 3.3%)	102, 107
		6	87, 96, 97 (mean = 93, 5.9%)	105, 107
		11 (343 days)	102, 103, 105 (mean = 103, RSD = 1.5%)	91, 99

The above results show 2,6-dichlorobenzamide residues to be stable for at least 9 months of frozen storage in raisins, and for at least 11 months in raw grapes and grape juice. Residues of dichlobenil parent compound are stable for at least 11 months in raw grapes and grape juice and for 3 months in raisins, with lower recoveries being observed in raisins after 6 months of storage.

Animal matrices

Storage stability data for residues of dichlobenil and 2,6-dichlorobenzamide in cattle matrices (milk, muscle, fat, liver and kidney) was generated concurrently with the cattle feeding study (Batorewicz, 1998). Storage stability for 2,6-dichlorobenzamide residues was acceptable in all matrices over the period for which trial samples were stored, as well as for dichlobenil in muscle, milk and fat. Poor stability was noted for dichlobenil residues in kidney and particularly liver samples. However, based on the plant metabolism studies and field trial data, residues in plant commodities are expected to consist only of 2,6-dichlorobenzamide, with finite residues of dichlobenil not being expected in livestock feed. Further, as the animal metabolism studies for 2,6-dichlorobenzamide show that dichlobenil is not a significant animal metabolite of 2,6-dichlorobenzamide, parent dichlobenil is not expected to be found in animal commodities.

USE PATTERN

Dichlobenil is registered in the USA in stone fruit (cherries, both sweet and sour varieties,) and berries and other small fruits (blueberries, blackberries, cranberries, grapes and raspberries).

Table 33 Registered uses of dichlobenil in the USA relevant to the evaluation

Crop	Country	Application						PHI (days)	Comments
		Form.	Method	Spray conc. (kg ai/hL)	Volume (L/ha)	Rate (kg ai/ha)	No.		
Stone fruits									
Cherries (Sweet and Sour)	USA	CS	Ground based soil surface spray.			2-4	1	-	¹
Cherries	USA	G	Soil surface application followed by shallow incorporation as needed.	-	-	4.8-6.7	1	-	²
Cherries	Canada	G	Broadcast or banded soil application.	-	-	4.4-7	1	-	³
Peaches	Canada	G	Broadcast or banded soil application.	-	-	4.4-7	1	-	³
Plums	Canada	G	Broadcast or banded soil application.	-	-	4.4-7	1	-	³
Berries and other small fruits									
Blueberries	USA	CS	Ground based soil surface spray.			2-4	1	-	⁴
Blueberries	USA	G	Soil surface application	-	-	4.5-6.7	1	-	⁵

Crop	Country	Application						PHI (days)	Comments
		Form.	Method	Spray conc. (kg ai/hL)	Volume (L/ha)	Rate (kg ai/ha)	No.		
			followed by shallow incorporation as needed.						
Blueberries (Highbush)	Canada	G	.	-	-	7-9	1	-	^{6.}
Blackberries	USA	CS	Ground based soil surface spray.			2-4	1	-	^{1.}
Blackberries	USA	G	Soil surface application followed by shallow incorporation as needed.	-	-	4.5	1	-	^{2.}
Cranberries	USA	G	Soil surface application followed by shallow incorporation as needed.	-	-	4.48	1	-	^{7..}
Cranberries	USA	G	Soil surface application followed by shallow incorporation as needed.	-	-	4.5	1	-	^{8.}
Cranberries	Canada	G	Soil surface application followed by shallow incorporation as needed.	-	-	4.4	1	-	^{9.}
Cranberries	Canada (BC only)	G		-	-	3.6-4.4	2	-	^{10.}
Grapes	USA	CS	Ground based soil surface spray.			2-4	1	-	^{4.}
Grapes	USA	G	Soil surface application followed by shallow incorporation as needed.	-	-	4.5-6.7	1	-	^{2.}
Grapes	Canada	G	Soil surface application.	-	-	4.4.-7	1	-	^{11.}
Grapes	Canada	G	Soil application.	-	-	7-9	1	-	^{12.}
Raspberries	USA	CS	Ground based soil surface spray.			2-4	1	-	^{4..}
Raspberries	USA	G	Soil surface application followed by shallow incorporation as needed.	-	-	4.5	1	-	^{2.}
Raspberries	Canada	G		-	-	7	1	-	^{2.}

Comments:

^{1.} Spray prior to weed emergence or when emerged weeds are less than 2 inches tall. Apply from late fall through to early spring.

^{2.} Apply as a soil surface treatment for perennial weeds in late fall (15 November to 15 February). Apply as an incorporated treatment for perennial weeds in late fall or very early spring prior to 1 May. For control of annual weeds, apply in early spring before germination of weeds or after cultivation. Incorporation or irrigation recommended..

^{3.} Apply to well prepared weed free soil in early spring or late fall before annual weed seeds have germinated. Do not apply more than the recommended amount per season. Application in spring should be made well before bud burst.

^{4.} Spray prior to weed emergence or when emerged weeds are less than 2 inches tall. Apply from late fall through to early spring.

^{5.} For control of annual weeds, apply in early spring before germination of weeds or after cultivation. Incorporation or irrigation recommended.

^{6.} Apply in the dormant period (late winter).

^{7..} Apply in early spring while perennial weeds are still dormant and annual weeds have not started to germinated or in late fall after crop has been harvested. Apply pre-budbreak or postharvest. Thoroughly incorporate granules into the surface through watering in.

^{8.} Apply in early spring when perennial weeds are still dormant and annual weeds have not started to germinate or in late fall after the crop has been harvested. Thoroughly incorporate through watering in. Do not apply more than 4.5 kg ai/ha in a 12-month period.

^{9.} Do not make a spring application after application the previous fall.

^{10.} Apply two equal applications in early spring with an interval of 3 to 6 weeks between each application. Use the higher rate only every second year. Do not apply after flowering. Do not make fall applications

^{11.} For seedling weeds apply this rate in early spring or fall.

^{12.} For perennial weeds apply this rate in the fall.

RESIDUES RESULTING FROM SUPERVISED RESIDUE TRIALS

The Meeting received information on dichlobenil supervised field residue trials for stone fruit (peaches, plums and cherries) and berries and other small fruits (blueberries, blackberries, cranberries, grapes and raspberries).

Trials were well documented with laboratory and field reports. The laboratory reports included method validation with determination of recoveries at the method LOQ and one other fortification level. Dates of sample collection and analysis were provided, allowing determination of storage intervals. At all trials, a granular formulation of dichlobenil was applied using either a hand spreader (e.g. shaker can), cyclone spinner applicator, or by a tractor-mounted granular spreader. A single plot was treated at each trial site. Plot areas and sample sizes were adequate. Samples were frozen within 6 hours of collection and stored frozen until analysis, with the exception of plums from the processing study (Hamilton, 1993) where the unprocessed fruit sample was frozen the day after collection. Generally, a single untreated control sample and duplicate or triplicate treated plot samples were collected.

In some studies, only residues of 2,6-dichlorobenzamide have been determined, while for others, both 2,6-dichlorobenzamide and dichlobenil have been tested for. Residues were not found in untreated control samples except where noted. Residues and application rates have generally been rounded to two significant figures. The tabulated results have not been corrected for recovery. When residues were not detected, they are shown as below the LOQ, and the LOQ value was used for maximum residue level determination and dietary intake assessment. Where replicate samples were analysed, results for the individual samples are reported together with the mean result, with the mean results used for maximum residue level estimation and dietary risk assessment.

The residue data is presented in the following tables:

Crop group	Commodity	Table
Stone fruits	Cherries	34
	Peach	35
	Plum	36
Berries and other small fruits	Blueberries	37
	Blackberries	38, 39
	Cranberries	40
	Grapes	41, 42
	Raspberries	43, 44

Stone fruits

Cherries

A series of residue trials was conducted in both sour and sweet varieties of cherries (Maselli, 1996 and Gaydosh, 1996 respectively). A single banded or broadcast soil application of a 4% w/w granular formulation of dichlobenil was made at a target application rate of 6.7 kg ai/ha when the fruit was at mid maturity, followed by irrigation with 0.6-1.3 cm of water (this was not required at some sites due to adequate rainfall being received shortly after application). Mature fruit was harvested at a single interval from each site, approximately 28 days after application.

Samples were analysed for residues of both dichlobenil and 2,6-dichlorobenzamide using a GC-ECD method very similar to study number 2005-046. Residues were determined on the edible portion (minus stones). Concurrent and method validation recoveries for dichlobenil and 2,6-dichlorobenzamide in sweet and sour cherries were within 70–120%. Sour cherry samples were stored frozen for up to 198 and 226 days between collection and analysis of dichlobenil and 2,6-dichlorobenzamide respectively, while sweet cherry samples were stored for up to 184 and 251 days respectively. A concurrent storage stability study was conducted for sweet cherries as part of the residue study, with mean recoveries of 72% for dichlobenil after 197 days of frozen storage, and 90%

for 2,6-dichlorobenzamide after 246 days of frozen storage. The samples are therefore unlikely to have been adversely affected by storage.

Table 34 Residues of dichlobenil in cherries (mature fruit)

Location, Trial no., Year (Variety)	Application				DAT	Residue (mg/kg)		Reference
	Date	Growth stage	Application method	Rate (kg ai/ha)		Dichlobenil	2,6-Dichlorobenzamide	
Hart, MI, USA, JGC-95008, 1995 (Montmorency)	14/06/95	immature fruit 0.95-1.3 cm dia.	banded soil	7.4	28	< 0.02 (< 0.02, < 0.02)	< 0.003 (< 0.003, < 0.003)	Maselli, 1996
Hart, MI, USA, JGC-95013, 1995 (Gold)	14/06/95	immature fruit 1.3 cm dia.	banded soil	7.4	28	< 0.02 (< 0.02, < 0.02)	< 0.003 (< 0.003, < 0.003)	Gaydos h, 1996
Cedar, MI, USA, JGC-95009, 1995 (Montmorency)	18/06/95	immature fruit 0.95-1.3 cm dia.	banded soil	7.4	28	< 0.02 (< 0.02, < 0.02)	< 0.003 (< 0.003, < 0.003)	Maselli, 1996
North Rose, NY, USA, RGC95RES05, 1995 (Mahaleb)	19/06/95	0.95 cm green-yellow fruit	banded soil	7.6	28	< 0.02 (< 0.02, < 0.02)	< 0.003 (< 0.003, < 0.003)	Maselli, 1996
Conklin, MI, USA, JGC-95007, 1995 (Montmorency)	13/06/95	immature fruit 1.3 cm dia.	banded soil	7.4	28	< 0.02 (< 0.02, < 0.02)	< 0.003 (< 0.003, < 0.003)	Maselli, 1996
Conklin, MI, USA, JGC-95012, 1995 (Sam's)	02/06/95	immature fruit 0.95-1.3 cm dia.	banded soil	7.4	28	< 0.02 (< 0.02, < 0.02)	< 0.003 (< 0.003, < 0.003)	Gaydos h, 1996
Dix, IL, USA, JGC-95010, 1995 (Montmorency grafted on Mahaleb)	17/05/95	0.3 cm fruit diameter	broadcast to area under each tree	7.4	28	< 0.02 (< 0.02, < 0.02)	0.004 (0.004, 0.005)	Maselli, 1996
Paw Paw, MI, USA, JGC-95011, 1995 (Montmorency grafted on Mahaleb)	09/06/95	immature fruit 1.3 cm dia.	banded soil	7.4	28	< 0.02 (< 0.02, < 0.02)	< 0.003 (< 0.003, < 0.003)	Maselli, 1996
Perry, UT, USA, RCP-95006, 1995 (Montmorency grafted on Mazzard rootstock)	08/07/95	orange immature fruit	banded soil	7.4	28	< 0.02 (< 0.02, < 0.02)	< 0.003 (< 0.003, < 0.003)	Maselli, 1996
Hillsboro, OR, USA, DNJ-95106, 1995 (Montmorency)	14/06/95	immature green to yellow fruit	soil broadcast	7.4	28	< 0.02 (< 0.02, < 0.02)	< 0.003 (< 0.003, < 0.003)	Maselli, 1996
Manteca, CA, USA, RCP-95002, 1995 (Bing)	21/04/95	mid maturity fruit	banded soil	7.5	28	< 0.02 (< 0.02, < 0.02)	< 0.003 (< 0.003, < 0.003)	Gaydos h, 1996
Hughson, CA, USA, RCP-95003, 1995 (Bing)	12/04/95	small fruit	banded soil	7.6	28	< 0.02 (< 0.02, < 0.02)	0.004 (0.005, < 0.003)	Gaydos h, 1996
Zillah, WA, USA, DNJ-95102, 1995 (Bing)	26/05/95	mid maturity green fruit	banded soil	7.4	28	< 0.02 (< 0.02, < 0.02)	< 0.003 (< 0.003, < 0.003)	Gaydos h, 1996
Buena, WA, USA, DNJ-95103, 1995 (Bing)	26/05/95	mid maturity green fruit	banded soil	7.4	28	< 0.02 (< 0.02, < 0.02)	< 0.003 (< 0.003, < 0.003)	Gaydos h, 1996
Gaston, OR, USA, DNJ-95104, 1995 (Royal Anne)	30/05/95	immature fruit 0.64-1.3 cm dia.	soil broadcast	7.4	28	< 0.02 (< 0.02, < 0.02)	< 0.003 (< 0.003, < 0.003)	Gaydos h, 1996
The Dalles, OR, USA, DNJ-95105, 1995 (Royal Anne)	31/05/95	immature fruit 0.64-0.85 cm dia.	soil broadcast	7.4	28	< 0.02 (< 0.02, < 0.02)	< 0.003 (< 0.003, < 0.003)	Gaydos h, 1996

No residues were found above the LOQ (0.02 mg/kg for dichlobenil and 0.003 mg/kg for 2,6-dichlorobenzamide) in the untreated control samples.

Peaches

Four residue trials were conducted in peaches in South Carolina and California during 1990 and 1991 (Dykstra, 1991a and Ver Hey, 1992a). A single banded or broadcast soil application of dichlobenil was made using a 4% granular formulation at a target application rate of 6.72 kg ai/ha. Mature fruit was sampled at varying intervals (20–68 days) after application.

Peach samples were analysed for 2,6-dichlorobenzamide residues using a GC-ECD method (method number L 3-53-71), evaluated in detail above. It is not clear whether the residues have been calculated on a whole fruit basis (including stones), or only on the edible portion. Concurrent recoveries of 2,6-dichlorobenzamide in peach samples were within 70–120%. Samples were stored frozen for up to 60 days before analysis. Untreated control peach samples were fortified with 2,6-dichlorobenzamide and stored frozen for up to 59 days: mean recoveries after storage were all within the range 70–120%, indicating that the peach samples are unlikely to have been adversely affected by storage.

Table 35 Residues of dichlobenil in peaches (mature fruit)

Location, Trial no., Year (Variety)	Application				DAT	2,6-Dichlorobenzamide residue (mg/kg)	Reference
	Date	Growth stage	Application method	Rate (kg ai/ha)			
Madera, CA, USA, C 201 50 020, 1990 (Springcrest)	05/05/90	5.0-7.5 cm diameter fruit	Banded soil	6.8	20	< 0.01 (< 0.01, < 0.01, < 0.01)	Dykstra, 1991a
Kline, SC, USA, C 201 50 021, 1990 (Winblo)	26/04/90	not stated	soil broadcast	7.1	47	0.01 (0.01, 0.01, < 0.01)	Dykstra, 1991a
Fresno, CA, USA, C 201 50 031, 1991 (Flavorcrest)	22/04/91	2.5 cm diameter fruit	Banded soil	7.7	63	0.023 (0.027, 0.021, 0.02)	Ver Hey, 1992a
Kline, SC, USA, C 201 50 032, 1991 (Winblo)	17/04/91	not stated	soil broadcast	7.4	68	0.037 (0.038, 0.035, 0.039)	Ver Hey, 1992a

No residues were found above the LOQ (0.01 mg/kg) in the untreated control samples.

Plums

Two trials were conducted in plums in Idaho during 1991 and in Michigan during 1992 (Ver Hey, 1992b and Hamilton, 1993). A single soil broadcast or banded application of a 4% granular formulation of dichlobenil was made at a target rate of 6.72 kg ai/ha. Mature fruit was sampled around 4-5 months after treatment.

Plum samples were analysed for 2,6-dichlorobenzamide residues using a GC-ECD analytical method (method number L 3-53-71, evaluated in detail above). For the Michigan trial, residues were determined on the edible portion while for the Idaho trial it is not clear whether the residues have been calculated on a whole fruit basis (including stones), or only on the edible portion. Samples were stored frozen for up to 41 days prior to analysis. Concurrent recoveries of 2,6-dichlorobenzamide from plum samples were within the range 70–120%, as were those in fortified samples stored frozen for up to 35 days, indicating that the plum samples are unlikely to have been adversely affected by storage.

The results from the Parma, Idaho trial are anomalously high when compared with other trial results in stone fruit.

Table 36 Residues of dichlobenil in plums (mature fruit)

Location, Trial no., Year (Variety)	Application				DAT	2,6-Dichlorobenzamide residue (mg/kg)	Reference
	Date	Growth stage	Application method	Rate (kg ai/ha)			
Parma, ID, USA, C 201 50 034, 1991 (Empress)	19/04/91	not stated	soil broadcast	7.9	154	0.39 (0.37, 0.40)	Ver Hey, 1992b
Conklin, MI, USA, C 201 50 048, 1992 (Stanley)	15/04/92	not stated	banded soil	6.7	139	< 0.01 (< 0.01, < 0.01, < 0.01)	Hamilton, 1993

Residues were not found above the LOQ (0.01 mg/kg) in the untreated control samples.

Berries and other small fruits

Blueberries

Four trials in blueberries were reported (Reibach, 2013). A single broadcast soil application of dichlobenil was made using a 4% w/w granular formulation at a target rate of 4.48 kg ai/ha. Mature fruit was sampled, typically around 50–90 days after application.

Samples were analysed for both dichlobenil and 2,6-dichlorobenzamide residues using a GC-ECD method, a modification of the method in study number 2005-046 evaluated above, without the solid phase extraction cleanup. Mean and individual method validation recoveries and mean concurrent recoveries were within the range 70–120%. Samples were stored frozen for up to 90 days prior to analysis.

Table 37 Residues of dichlobenil in blueberries (mature fruit)

Location, Trial no., Year (Variety)	Application			DAT	Residue (mg/kg)	
	Date	Application method	Rate (kg ai/ha)		Dichlobenil	2,6-Dichlorobenzamide
Lehigh, PA, USA, 2012-01-PA, 2012 (Blueray)	07/05/12	soil broadcast	4.3	53	< 0.01 (< 0.01, 0.01)	0.015 (0.016, 0.014)
Bucks, PA/NJ, USA, 2012-02-PA/NJ, 2012 (Bluecrop)	08/05/12	soil broadcast	4.3	51	< 0.01 (< 0.01, 0.01)	< 0.01 (< 0.01, 0.01)
Ottawa, MI, USA, 2012-03-MI, 2012 (Draper)	10/05/12	soil broadcast	4.3	56	< 0.01 (< 0.01, 0.01)	< 0.01 (< 0.01, 0.01)
Washington, OR, USA, 2012-04-OR, 2012 (Bluecrop)	14/05/12	soil broadcast	4.3	58	< 0.01 (< 0.01, 0.01)	< 0.01 (< 0.01, 0.01)

No residues were found in the untreated control samples above the LOQ (0.01 mg/kg for each analyte).

Blackberries

A single residue study in blackberries was conducted in Oregon, USA during 1990 (Dykstra, 1991b). A single broadcast soil application of dichlobenil was made using a granular formulation at a target application rate of 4.48 kg ai/ha. Mature fruit was harvested 3 months after application.

Blackberry samples were analysed for 2,6-dichlorobenzamide using a GC-ECD method (method number L 3-53-71, evaluated in detail above). Mean and individual method validation recoveries in blackberries were within 70–120%. Storage stability recoveries for samples of blackberries fortified with 2,6-dichlorobenzamide and stored frozen for 26 days were within 70–

120%. Samples were stored frozen for up to 35 days between collection and analysis, and are unlikely to have been adversely affected by storage.

A further four trials in blackberries were reported (Reibach, 2013). A single broadcast soil application of dichlobenil was made using a 4% w/w granular formulation at a target rate of 100 pounds per acre (4.48 kg ai/ha). Mature fruit was sampled, typically around 50–90 days after application.

Samples were analysed for both dichlobenil and 2,6-dichlorobenzamide residues using a GC-ECD method, a modification of the method in study number 2005-046 evaluated above, without the solid phase extraction clean-up. Mean and individual method validation recoveries and mean concurrent recoveries in blackberries were within the range 70–120%. Samples were stored frozen for up to 70 days before analysis.

Table 38: Residues of dichlobenil in blackberries, mature fruit (Dykstra, 1991b)

Location, Trial no., Year (Variety)	Application				DA T	2,6-Dichlorobenzamide residue (mg/kg)
	Date	Growth stage	Application method	Rate (kg ai/ha)		
Cornelius, OR, USA, 1990, C 201 50 009 (Marion)	19/04/ 90	not stated	soil broadcast	4.7	89	<u>0.01</u> (0.01, 0.01, 0.01)

Residues were not found at levels above the LOQ (0.01 mg/kg) in the untreated control samples.

Table 39: Residues of dichlobenil in blackberries, mature fruit (Reibach, 2013)

Location, Trial no., Year (Variety)	Application			DAT	Residue (mg/kg)	
	Date	Applicatio n method	Rate (kg ai/ha)		Dichlobenil	2,6- Dichlorobenzam ide
Bucks, PA/NJ, USA, 2012-05-PA/NJ, 2012 (Chester)	08/05/1 2	soil broadcast	4.3	78	< 0.01 (< 0.01, < 0.01)	<u>0.056</u> (0.049, 0.062)
Washington, OR, USA, 2012-06- OR, 2012 (Marion)	14/05/1 2	soil broadcast	4.3	57	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
Washington, OR, USA, 2012-07- OR, 2012 (Chester)	14/05/1 2	soil broadcast	4.3	88	< 0.01 (< 0.01, < 0.01)	<u>0.035</u> (0.035, 0.034)
Washington, OR, USA, 2012-08- OR, 2012 (Evergreen blackberry)	22/06/1 2	soil broadcast	4.3	53	< 0.01 (< 0.01, < 0.01)	<u>0.031</u> (0.029, 0.032)

No residues were found in the untreated control samples above the LOQ (0.01 mg/kg for each analyte).

Cranberries

Two residue studies in cranberries were conducted in Massachusetts and Wisconsin during 1991 (Ver Hey, 1992c). A single broadcast soil application of dichlobenil was made during spring using a granular formulation at a target application rate of 4.48 kg ai/ha. Mature fruit was harvested around 5–6 months after application.

Cranberry samples were analysed for residues of 2,6-dichlorobenzamide using a GC-ECD method (method number L 3-53-71, evaluated in detail above). Mean and individual method validation recoveries in cranberry samples were within the range 70–120%. Samples were stored frozen between collection and analysis for up to 94 days. Mean and individual storage stability recoveries for untreated control samples of cranberries fortified with 2,6-dichlorobenzamide and stored frozen for up to 87 days were within the range 70–120%; the residue trial samples are therefore unlikely to have been adversely affected by storage.

Table 40: Residues of dichlobenil in cranberries, mature fruit (Ver Hey, 1992c)

Location, Trial no., Year (Variety)	Application				DAT	2,6-Dichlorobenzamide residue (mg/kg)
	Date	Growth stage	Application method	Rate (kg ai/ha)		
East Wareham, MA, USA, C 201 50 037, 1991 (not specified)	19/04/91	mid spring	soil broadcast	5.7	158	0.020 (0.018, 0.024, 0.018)
Warrens, WI, USA, C 201 50 038, 1991 (not specified)	19/04/91	mid spring	soil broadcast	4.8	166	0.019 (0.015, 0.023, 0.020)

Residues were not found at levels above the LOQ (0.01 mg/kg) in the untreated control samples.

Grapes

Four trials were conducted in the USA (Chickering and Koch, 1991). A banded soil application was made to grapevines using a granular formulation (40 g/kg dichlobenil) at a target rate of 6.73 kg ai/ha in mid to late spring. Approximately 2.5-3.8 cm of irrigation was applied by sprinkler shortly after application at the Biola and Fresno trial sites. Mature grapes were sampled around 4-5 months after application.

Residues of 2,6-dichlorobenzamide in grapes were determined using a GC-ECD method based on method number L 3-53-71, evaluated in detail above. Individual and mean method validation and concurrent recoveries were within the range 70–120%. Grapes were analysed within 196 days of harvest. Fortified grape samples were stored frozen for 112-133 days, and showed individual and mean recoveries within the range 70–120%. Given that residues of dichlobenil and 2,6-dichlorobenzamide were shown to be stable for 323 and 345 days of frozen storage respectively during the Maselli, 1998 study (see below), the grape samples are unlikely to have been adversely affected by storage.

Additional residue data for grapes is available from the field component of a processing study in grapes (Maselli, 1998). Field trials were conducted at two sites in California during 1996. A single banded soil application of a 4% w/w granular formulation of dichlobenil was made around the end of dormancy at a target application rate of 8.75 kg ai/ha. At least 6.4 mm of water was applied within 48 hours of application, either by rainfall or irrigation if rainfall was insufficient to prevent sublimation of the test material. Mature grapes were sampled around 6 months after application.

Residues of dichlobenil and 2,6-dichlorobenzamide in raw and processed grape samples were determined using a GC-ECD method very similar to method number 2005-046 (evaluated in more detail above). Acceptable individual and mean method validation and concurrent recoveries in the range 70–120% were achieved in grapes, for both dichlobenil and 2,6-dichlorobenzamide. Analyses of dichlobenil were completed within 289 days of sample collection, while those for 2,6-dichlorobenzamide were completed within 124 days. A concurrent storage stability study was conducted, showing recoveries of dichlobenil and 2,6-dichlorobenzamide remaining within the range 70–120% after storage for 323 and 345 days respectively.

Table 41 Residues of dichlobenil in grapes, mature berries (Chickering and Koch, 1991)

Location, Trial no., Year (Variety)	Application				DAT	2,6-Dichlorobenzamide residue (mg/kg)
	Date	Growth stage	Application method	Rate (kg ai/ha)		
Biola, CA, USA, C 201 50 014, 1990 (Thompson Seedless)	30/04/90	15 cm bunch length	Banded soil	7.1	119	0.041 (0.042, 0.037, 0.043)
Fresno, CA, USA, C 201 50 015, 1990 (Thompson Seedless)	30/04/90	15 cm bunch length	Banded soil	6.8	119	0.057 (0.061, 0.047, 0.062)

Location, Trial no., Year (Variety)	Application				DAT	2,6-Dichlorobenzamide residue (mg/kg)
	Date	Growth stage	Application method	Rate (kg ai/ha)		
Madera, CA, USA, C 201 50 016, 1990 (Thompson Seedless)	01/05/90	Shot berry	Banded soil	6.8	120	0.032 (0.031, 0.026, 0.040)
Phelps, NY, USA, C 201 50 017, 1990 (Catawba)	27/04/90	Not stated	Banded soil	6.7	158	0.041 (0.041, 0.053, 0.029)

Except where otherwise noted, residues were <LOQ in untreated control samples.

Table 42: Residues of dichlobenil in grapes, mature berries (Maselli, 1998)

Location, Trial no., Year (Variety)	Application				DAT	Residues (mg/kg)	
	Date	Growth stage	Application method	Rate (kg ai/ha)		Dichlo benil	2,6- Dichlorobenza mide
Madera, CA, USA, CEJ- 96001, 1996 (Thompson seedless)	02/03/96	end of dormancy	Banded spray	8.1	177	< 0.02	0.029
Fresno, CA, USA, CEJ- 96002, 1996 (Thompson seedless)	01/03/96	end of dormancy	Banded spray	8.3	178	< 0.02	0.004

No residues were found in untreated control samples above the LOQ (0.02 mg/kg for dichlobenil, and 0.003 mg/kg for 2,6-dichlorobenzamide).

Raspberries

Two residue trials in raspberries were conducted in Oregon and Washington during 1990 (Dykstra, 1991c). A single broadcast soil application of dichlobenil was made during spring using a granular formulation at a target application rate of 4.48 kg ai/ha.

Raspberry samples were analysed for 2,6-dichlorobenzamide using a GC-ECD method (method number L 3-53-71, evaluated in detail above). Individual and mean method validation recoveries were within the range 70–120%. Samples were analysed within 49 days of collection. Mean storage stability recoveries for untreated control samples of raspberries fortified with 2,6-dichlorobenzamide were within the range 70–120% after 41 days storage. The residue trial samples are therefore unlikely to have been adversely affected by storage.

A further four trials in raspberries were reported (Reibach, 2013). A single broadcast soil application of dichlobenil was made using a 4% w/w granular formulation at a target rate of 4.48 kg ai/ha. Mature fruit was sampled, typically around 50–90 days after application.

Samples were analysed for both dichlobenil and 2,6-dichlorobenzamide residues using a GC-ECD method, a modification of the method in study number 2005-046 evaluated above, without the solid phase extraction cleanup. Mean method validation and quality control recoveries in raspberries were within the range 70–120%. Samples were stored frozen for up to 79 days between collection and analysis.

Table 43 Residues of dichlobenil in raspberries (Dykstra, 1991c)

Location, Trial no., Year (Variety)	Application				Sample	DAT	2,6-Dichlorobenzamide residue (mg/kg)
	Date	Growth stage	Applicatio n method	Rate (kg ai/ha)			
Banks, OR, USA, C 201 150 022, 1990 (Willamette)	20/04/90	mid spring	soil broadcast	4.8	Mature berries	60	< 0.01 (< 0.01, < 0.01, < 0.01)

Location, Trial no., Year (Variety)	Application				Sample	DAT	2,6-Dichlorobenzamide residue (mg/kg)
	Date	Growth stage	Applicatio n method	Rate (kg ai/ha)			
Vancouver, WA, USA, C 201 150 023, 1990 (Willamette)	20/04/90	mid spring	soil broadcast	4.9	Mature berries	60	0.020 (0.021, 0.021, 0.018)

No residues were found at levels above the LOQ (0.01 mg/kg) in the untreated control samples.

Table 44 Residues of dichlobenil in raspberries, mature fruit (Reibach, 2013)

Location, Trial no., Year (Variety)	Application			DAT	Residue (mg/kg)	
	Date	Application method	Rate (kg ai/ha)		Dichlobenil	2,6- Dichlorobenzami de
Lehigh, PA, USA, 2012-09-PA, 2012 (Boyne)	07/05/12	soil broadcast	4.3	44	< 0.01 (< 0.01, < 0.01)	<u>0.067</u> (0.054, 0.080)
Ottawa, MI, USA, 2012-10-MI, 2012 (Nova)	10/05/12	soil broadcast	4.3	50	< 0.01 (< 0.01, < 0.01)	<u>0.021</u> (0.026, 0.016)
Washington, OR, USA, 2012-11-OR, 2012 (Willamette)	14/05/12	soil broadcast	4.3	51	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
Washington, OR, USA, 2012-12-OR, 2012 (Coho)	14/05/12	soil broadcast	4.3	57	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)

No residues were found in the untreated control samples above the LOQ (0.01 mg/kg for each analyte).

FATE OF RESIDUES IN PROCESSING

Plums

A processing trial for plums was conducted in Michigan (Hamilton, 1993). After a single banded soil application during spring of a 4% w/w granular formulation of dichlobenil at a target rate of 6.7 kg ai/ha, mature plums were harvested and shipped unfrozen for processing. Processing commenced the day after harvest. An unwashed raw plum sample was set aside, before processing the remaining plums into prunes using a batch process. Plums were dried to a target moisture content of 18-22% over a period of approximately 24 hours at temperatures from 66-82 °C. Samples were frozen within a few hours of completion of processing.

Frozen whole plums and prune samples were shipped to the laboratory for analysis of 2,6-dichlorobenzamide residues using a GC-ECD method (method number L 3-53-71, evaluated in detail above). The mean concurrent recovery for 2,6-dichlorobenzamide from prunes were within the range of 70–120%, although it is noted that some individual concurrent recovery values and the recoveries after only four days of frozen storage were < 70%. The plum samples showed storage stability recoveries within the 70–120% range after 35 days of storage. Plum and prune samples were analysed 39–40 days after harvest/processing, and prune samples may therefore have been adversely affected by storage.

Table 45 Residues of dichlobenil in plums and prunes

Location, Trial no., Year (Variety)	Application				Sam- ple	D A T	2,6-Dichlorobenzamide residue (mg/kg)	Referenc e
	Date	Growth stage	Application method	Rate (kg ai/ha)				
Conklin, MI, USA, C 201 50 048, 1992 (Stanley)	15/0 4/92	not stated	banded soil	6.7	plu ms	13 9	< 0.01 (< 0.01, < 0.01, < 0.01)	Hamilto n, 1993
					pru nes		0.01 (0.01, 0.01, 0.01)	

No residues were found in untreated control samples above the LOQ (0.01 mg/kg).

Residues of 2,6-dichlorobenzamide were observed to concentrate slightly in plums.

Grapes

Residue trials for generating processing data for grapes were conducted at two sites in California (Maselli, 1998). Plots of grapevines were treated with a single banded spray application at a target rate of 8.8 kg ai/ha of 4G dichlobenil formulation. The application was made just as the dormancy period was finishing. Grapes were sampled at maturity from the treated plots and untreated control areas, with separate samples for analysis of unprocessed grapes, and for processing into juice and raisins being collected. Unprocessed grapes were frozen within 4 hours of collection, while grapes for processing were kept at ambient temperature, with processing into raisins commencing on the day of harvest, and processing into juice 3 days after harvest.

For juice processing, grapes were crushed and destemmed using a commercial crusher-stemmer, and the wet mash was loaded into a hydraulic press for juicing. Juice samples were collected and stored frozen until analysis, while the wet pomace was discarded.

Grapes were processed into raisins by sun drying on paper trays for approximately 3 weeks, with the drying grapes being turned twice. Untreated control grapes were kept well separated from the treated grapes during drying. After drying to approximately 20% of the fresh weight, the stems were removed using simulated commercial equipment, involving first passing over a screen shaker to remove loose impurities such as leaf and stem fragments, then destemming followed by passing over a vacuum unit to remove remaining stem fragments and substandard raisins. The raisins were then passed through a sizing shaker to separate the smaller raisins normally used for baking or breakfast cereals, with the remaining raisins being passed over two further vacuum units to remove any remaining substandard raisins, and C-grade, which are discarded. The combined A- and B-grade raisins were washed and then rehydrated to commercial specification using a 'riffle' washer, followed by cleaning and visual inspection. A sample of raisins was collected and placed in frozen storage prior to analysis.

Residues of dichlobenil and 2,6-dichlorobenzamide in raw and processed grape samples were determined using a GC-ECD method very similar to method number 2005-046 evaluated in more detail above. Acceptable individual and mean method validation and concurrent recoveries in the range 70–120% were achieved in grapes, for both dichlobenil and 2,6-dichlorobenzamide. Analyses of dichlobenil in grapes were completed within 289 days of sample collection, those for raisins were completed within 277 days, and those for grape juice were completed within 277 days. Analyses of 2,6-dichlorobenzamide in grapes were completed within 124 days, those for raisins were completed within 276 days, and those for grape juice were completed within 275 days. A concurrent storage stability study was conducted, showing recoveries of dichlobenil and 2,6-dichlorobenzamide remaining within the range 70–120% after storage for 323 and 345 days respectively for grapes. In raisins, a storage stability trial was carried out over 273 days for dichlobenil, and 266 days for 2,6-dichlorobenzamide, with mean recoveries remaining within 70–120% at the end of storage. In grape juice, fortified samples were stored for 322 days (dichlobenil) and 343 days (2,6-dichlorobenzamide),

with mean recoveries remaining within 70–120%. The storage stability data shows that the raw and processed grape residue trial samples are unlikely to have been adversely affected by storage.

A second processing study was conducted in the USA (Ver Hey, 1992d). At a site in California, a single application of dichlobenil was made using a granular formulation at a target rate of 150 pounds of a 4% w/w formulation per treated acre (6.72 kg ai/ha), with the application made prior to flowering. The vines were irrigated (5.0-7.5 cm) on the day of application. Mature grape samples were collected for processing into juice and raisins.

Processing of grapes into juice and raisins commenced on the day of harvest. Limited information on processing was provided. Processing was conducted using domestic kitchen equipment rather than simulated commercial processes. The grapes were juiced using a food processor, and the juice was decanted. The remaining solids were collected as the wet pomace fraction, and a portion was placed in a cloth bag and the moisture squeezed to generate dry pomace. Raisins were sun dried over 19 days, and separated into raisins and raisin waste (stems and debris). Samples were frozen within 6 hours of collection.

Samples of grape juice, raisins, raisin waste, and wet and dry grape pomace were analysed for residues of 2,6-dichlorobenzamide using a GC-ECD method (method number L 3-53-71, evaluated above). Concurrent and stored recoveries (after 99, 108, 111, 79 and 101 days for juice, wet pomace, dry pomace, raisins and raisin waste respectively) were within the range 70–120%, with the exception of stored recoveries for raisins (mean stored recovery = 64%). Samples were analysed within 124–163 days of harvest of the grapes. Given the storage stability results for this and the Maselli study, with the possible exception of raisins, it is unlikely that the samples have been adversely affected by storage.

Samples were analysed in triplicate. The results are tabulated below. However it is noted that residues were not reported for raw grapes from the field site, so processing factors cannot be calculated.

Table 46: Processing study in grapes (Maselli, 1998)

Location, Trial no., Year (Variety)	Application				Sample	DA T	Residues (mg/kg)	
	Date	Growth stage	Applicatio n method	Rate (kg ai/ha)			Dichlobe nil	2,6-Dichloroben zamide
Madera, CA, USA, CEJ-96001, 1996 (Thompson seedless)	02/03/96	end of dormancy	Banded soil	8.1	Mature berries	177	< 0.02	0.029
					Raisins		< 0.02	0.082
					Juice		< 0.02	0.027
Fresno, CA, USA, CEJ-96002, 1996 (Thompson seedless)	01/03/96	end of dormancy	Banded soil	8.3	Mature berries	178	< 0.02	0.0041
					Raisins		< 0.02	< 0.03
					Juice		< 0.02	0.0059

No residues were found in untreated control samples above the LOQ (0.02 mg/kg for dichlobenil, and 0.003 mg/kg for 2,6-dichlorobenzamide).

Table 47 Processing study in grapes (Ver Hey, 1992d)

Location, Trial no., Year (Variety)	Application				Sample	DAT	2,6-Dichlorobenzamide residues (mg/kg)
	Date	Growth stage	Applicatio n method	Rate (kg ai/ha)			
Fresno, CA, USA, C 201 50 043, 1991 (Thompson seedless)	19/04/91	Pre- flowering	banded soil	7.9	Grapes	118	Not analysed for this site

Location, Trial no., Year (Variety)	Application				Sample	DAT	2,6-Dichlorobenzamide residues (mg/kg)
	Date	Growth stage	Applicatio n method	Rate (kg ai/ha)			
					Juice		0.030 (0.031, 0.031, 0.028)
					Wet pomace		0.057 (0.061, 0.056, 0.055)
					Dry pomace		0.11 (0.085, 0.13, 0.11)
					Raisins		0.010 (0.012, 0.008, 0.009)
					Raisin waste		0.28 (0.28, 0.28, 0.28)

Residues in untreated control samples were all <LOQ (0.01 mg/kg).

Table 48 Processing factors for 2,6-dichlorobenzamide in grape products (Maselli, 1998)

Product	Processing factor
Raisins	<1, 2.8
Juice	0.93, 1.4

Moderate concentration of residues of 2,6-dichlorobenzamide is noted in both grape juice and raisins.

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

Lactating cattle

A feeding study for the major plant metabolite of dichlobenil, 2,6-dichlorobenzamide (BAM), was conducted in lactating cattle (Batorewicz, 1998).

Sixteen lactating Holstein cattle were purchased and placed in quarantine for 7 days followed by a further 14 days of acclimatisation. Twelve cattle aged between 3-6 years and weighing 534-758 kg (mean milk production during the feeding phase was 17.9 L/animal/day, while mean feed intake was 18.9 kg dry matter/animal/day) were selected for the study on day 6 of acclimatisation from the group of sixteen. Three animals were randomly selected for each of the four groups: group I (untreated control), group II (low dose, 1× at a level of 0.6 ppm), group III (mid dose, 3×, at a level of 1.6 ppm), and group IV (high dose, 5×, at a level of 5.3 ppm). BAM was administered to the animals in groups II-IV orally once daily after the morning milking by capsule for 28 days. The control animals received blank capsules at the same time as the treated animals. The cattle were kept in outdoor pens with overhead shelter, with the different feeding groups being separated. *Ad libitum* access was allowed to fresh drinking water and a total mixed ration containing alfalfa hay, corn silage, rolled corn, cotton seed and various seed and grain by-products and mineral supplements. No adverse effects on cattle weight, feed consumption or milk yield were noted during the trial.

Milk samples were collected twice daily, and weighted pooled 200 gram subsamples (PM and the following AM) from selected days were frozen and submitted for analysis. The animals were sacrificed with 6 hours of the final dose and samples of liver, kidney, muscle (thigh and loin composite) and fat (composite of mesenteric and perirenal) were collected for analysis and frozen. Frozen tissue samples were processed by grinding in the presence of liquid nitrogen. The liquid nitrogen was allowed to evaporate in the freezer, and subsamples were collected for submission for analysis (250 grams for fat and 500 grams for other tissue types).

Samples were analysed for both dichlobenil and 2,6-dichlorobenzamide using a GC-MS method (method number 96145, evaluated in detail above). Mean concurrent recoveries in fortified control samples were generally within 70–120%.

Samples were stored frozen for up to 62 days (fat), 66 days (kidney), 70 days (liver), 63 days (muscle), and 66 days (milk) between collection and analysis. Storage stability was checked at fortification level of 0.05 mg/kg for both dichlobenil and 2,6-dichlorobenzamide. Acceptable storage stability was observed for 2,6-dichlorobenzamide in fat (62 days), kidney (67 days), liver (70 days), muscle (63 days), and milk (71 days), and for dichlobenil in muscle (63 days), fat (62 days), kidney (52 days), and milk (71 days). After 67 days storage, the mean stored recovery in kidney had dropped to 62%, although after correction for concurrent recovery, a corrected recovery of 70% was determined. Stability of dichlobenil in liver was poor, with mean recoveries of 43% after 55 days, and 31% after 70 days.

Residues of dichlobenil were not found above the LOQ (0.01 mg/kg) in any of the control or treated milk or tissue samples. Finite residues were found in tissue samples from all treatment groups, and in milk sample from the mid- and high-dose groups.

Table 49 Residues of BAM in milk

Dose level (ppm dry weight in feed)	Study day	2,6-dichlorobenzamide residues (mg/kg)
0.6	0	Not analysed.
	4	
	8	
	15	
	22	
	27	
1.6	0	Not analysed.
	4	
	8	
	15	
	22	
	27	0.008, 0.009, 0.008 (0.008)
5.3	0	< 0.01, < 0.01, < 0.01 (< 0.01)
	4	0.019, 0.013, 0.019 (0.017)
	8	0.014, 0.016, 0.021 (0.017)
	15	0.018, 0.019, 0.031 (0.023)
	22	0.025, 0.029, 0.040 (0.031)
	27	0.016, 0.022, 0.015 (0.018)

Residues of 2,6-dichlorobenzamide were not found above the LOQ (0.01 mg/kg) in the control milk samples.

Table 50 Residues of dichlobenil and 2,6-dichlorobenzamide in tissues

Tissue	Dose level (ppm dry weight in feed)	2,6-dichlorobenzamide residues (mg/kg)
Liver	0	< 0.01
	0.6	0.038, 0.062, 0.059 (0.053)
	1.6	0.054, 0.137, 0.065 (0.085)
	5.3	0.263, 0.024, 0.422 (0.31)
Kidney	0	< 0.01
	0.6	0.008, 0.012, 0.013 (0.011)
	1.6	0.022, 0.037, 0.025 (0.028)
	5.3	0.035, 0.027, 0.046 (0.036)
Muscle	0	< 0.01
	0.6	0.007, 0.010, 0.010 (0.009)
	1.6	0.018, 0.029, 0.021 (0.023)
	5.3	0.060, 0.053, 0.065 (0.059)
Fat	0	< 0.01
	0.6	Not analysed.
	1.6	0.010, 0.010, 0.009 (0.010)
	5.3	0.023, 0.017, 0.020 (0.020)

Residues of 2,6-dichlorobenzamide were not found above the LOQ (0.01 mg/kg) in the control milk samples.

Samples of fat and milk from the 1× treatment group were not analysed, as residues were either not detected or only found at the LOQ in samples from the 3× group. Residues of 2,6-dichlorobenzamide in milk peaked at or around day 22 of dosing. A clear linear relationship between feeding level and tissue residue was observed in liver and muscle ($r^2 > 0.95$), while the relationship was less clear for kidney ($r^2 = 0.61$).

Table 51: Relationship between dosing level and maximum tissue residue

Tissue	Regression equation (forced through origin)	r^2 value
Liver	$y = 0.0804x$	0.9974
Kidney	$y = 0.01x$	0.6008
Muscle	$y = 0.0128x$	0.9651

Table 52: Relationship between dosing level and mean tissue residue

Tissue	Regression equation (forced through origin)	r^2 value
Liver	$y = 0.0584x$	0.993
Kidney	$y = 0.0078x$	0.6108
Muscle	$y = 0.0114x$	0.9855

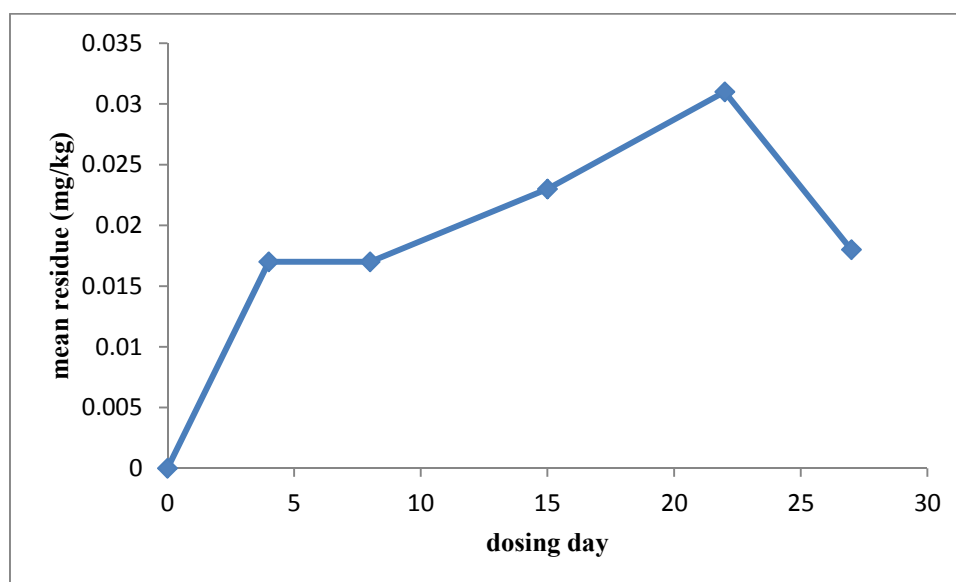


Figure 5 Mean milk residues over time in the 5 ppm feeding group

Poultry

A poultry feeding study was not provided to the Meeting.

APPRAISAL

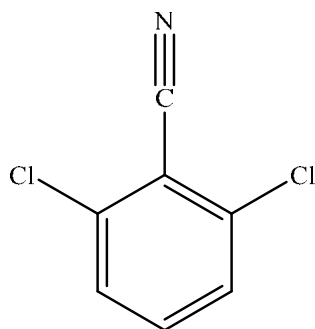
Dichlobenil is a benzonitrile herbicide for use by soil application in fruit crops, including stone fruit, grapes, and other berry crops. Dichlobenil was scheduled at the Forty-fifth Session of the CCPR in 2013 for evaluation as a new compound by the 2014 JMPR. Data was provided on physicochemical properties, metabolism in food producing animals and plants, environmental fates, methods of analysis, stability of residues in stored analytical samples, GAP information, supervised residue trials, processing studies, and animal feeding studies.

The Meeting received information on processing of oranges, strawberries, onions, lettuce head and peas.

Processing factors calculated for the processed commodities for the above raw agricultural commodities, including previously estimated, are shown in the table below. STMP-Ps was calculated for processed commodities of strawberry, onion and peas for which maximum residue levels were estimated.

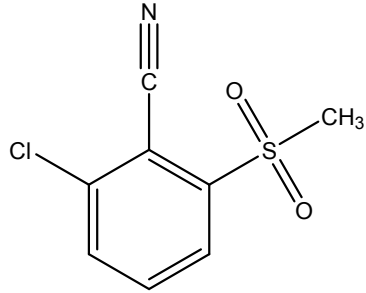
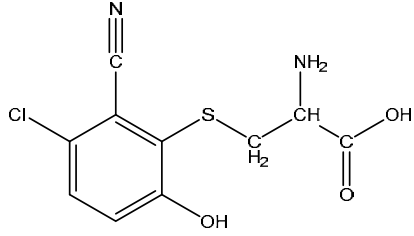
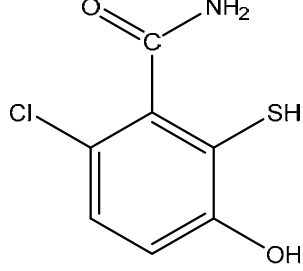
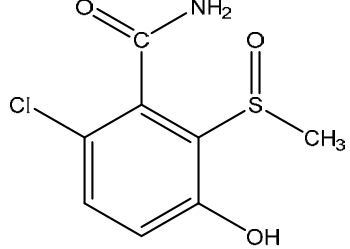
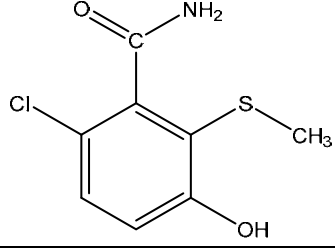
Chemical name and structure

The IUPAC name for dichlobenil is 2,6-dichlorobenzonitrile.



The following metabolites are discussed below:

2,6-Dichlorobenzamide (BAM)	
2,6-Dichloro-4-hydroxybenzamide	
4-Chloro-2(3H)benzoxazolone	
6-Chloro-2-methylthiobenzonitrile	

6-Chloro-2-methylsulfonylbenzonitrile	
S-(3-Chloro-2-cyano-6-hydroxyphenyl)-cysteine	
6-Chloro-3-hydroxy-2-thiobenzamide	
6-Chloro-3-hydroxy-2-methylsulfinylbenzamide	
6-Chloro-3-hydroxy-2-methylthiobenzamide	

Animal metabolism

The Meeting received information on the metabolism of ^{14}C -phenyl labelled dichlobenil in rats, lactating goats, and laying hens, and for the metabolism of ^{14}C -phenyl labelled 2,6-dichlorobenzamide (a significant plant metabolite) in lactating goats and laying hens.

Animal metabolism: dichlobenil

In rats, dichlobenil is metabolized via two metabolic pathways: hydroxylation at the 3 or 4 position followed by glucuronidation or sulphation and the second pathway includes substitution of one chlorine atom by glutathione, followed by cleavage and oxidation of the glutathione moiety to cysteine and ultimately thiol and sulfonyl derivatives.

Lactating goats were dosed orally, twice daily, with ^{14}C -phenyl dichlobenil at 10 mg/kg bw/day for three days. Milk was collected twice daily, with blood samples being collected just prior to

and at various intervals after the first and fifth doses. The goats were sacrificed 15 hours after the final dose.

The majority of the administered dose was excreted (39–45% in urine, 24–31% in faeces, 2.9–4.0% in cage wash, and 0.06 to 0.33% in milk). Carcass tissue accounted for 3.2 to 3.4 % of the administered radioactivity, while 1.1–3.3% remained in GI tract tissue, and 0.4–3.3% GI tract contents.

Levels in milk peaked after about 2 days of dosing, at 1.30 mg eq./kg. At sacrifice, total residues of 27 mg eq./kg were found in liver, 4.1 mg eq./kg in kidney, 2.4 mg eq./kg in fat, and 0.42 mg eq./kg in muscle. The proportions of residue extracted were variable, at 65–70% of TRR in milk, 65% in liver, 26% in fat, 13% in muscle, and 28% in kidney.

In muscle, a metabolite tentatively identified as 2-chloro-6-methylthiobenzonitrile predominated, at 11% TRR, with smaller amounts of S-(3-chloro-2-cyano-6-hydroxyphenyl)-cysteine (0.9% TRR) and an unidentified compound (1.3% TRR).

In fat, parent comprised 60% of the identified residue (16% of TRR), with the other 40% 2-chloro-6-methylsulfonylbenzonitrile (10% TRR).

In milk, S-(3-chloro-2-cyano-6-hydroxyphenyl)-cysteine comprised 11–21% TRR, 2-chloro-6-methylsulfonylbenzonitrile 14–38% of TRR, with 3.9–12% of an unidentified component; parent was only present at $\leq 1\%$.

In liver, the majority of residue was present as S-(3-chloro-2-cyano-6-hydroxyphenyl)-cysteine (95% of that identified, or 62% of TRR), with a small amount of 2-chloro-6-methylsulfonylbenzonitrile (3% of TRR).

In kidney, the largest component was 2-chloro-6-methylthiobenzonitrile (23% of TRR), with smaller amounts (1.1–2.5% of TRR) of S-(3-chloro-2-cyano-6-hydroxyphenyl)-cysteine, 2-chloro-6-methylsulfonylbenzonitrile, and an unidentified component.

Laying hens were dosed orally with ^{14}C -phenyl dichlobenil at 9.8–10.2 mg/kg bw/day for three days. The dose was administered twice daily, eggs were collected twice daily, with blood samples being collected just prior to and at various intervals after the first and fifth doses. The hens were sacrificed 15 hours after the final dose.

The majority of the administered dose was excreted, with a mean of 67% recovered in excreta, 4.2% in cage washings, and 6.1% in the GI tract at sacrifice. Eggs accounted for 0.44% of the dose, with levels not reaching a plateau during the study.

The proportions of residue extracted were variable, at 60% of TRR in eggs, 90% in liver, 15% in fat, 8% in muscle, and 39% in kidney.

In fat and eggs, 100% of the extracted residue was unchanged parent compound (15% and 60% of the TRR in fat and eggs, respectively). In muscle, the largest component was the tentatively identified 2-chloro-6-methylthiobenzonitrile (6.4% of TRR), with parent compound comprising 1.6% of TRR. Similar ratios were observed for liver (2-chloro-6-methylthiobenzonitrile; 85% of TRR and parent; 5.4% of TRR). Kidney contained a number of metabolites, the cysteine conjugate of hydroxylated dichlobenil (17% of TRR), parent (9.8% of TRR), 2-chloro-6-methylsulfonylbenzonitrile (5.1% of TRR), and 2-chloro-6-methylthiobenzonitrile (4.3% of TRR), with small amounts (1.2–1.6% of TRR) of two unidentified components.

Metabolism of dichlobenil in goats and hens was similar, although the degree of metabolism appeared to be higher in goats. The chief metabolic pathway was conjugation with cysteine, replacing one of the chlorine atoms, with or without hydroxylation at an adjacent position on the ring. This was followed by cleavage and oxidation of the cysteine moiety to yield methylthio and methylsulfonyl derivatives.

There are strong similarities to the metabolism of dichlobenil in rats, with hydroxylation and glutathione conjugation followed by cleavage of the glutathione moiety taking place in rats, goats and hens.

The only residue of significance in fruit is 2,6-dichlorobenzamide, with no parent being present. Only uses in fruit crops are under consideration at this stage, therefore the Meeting considers the metabolism of dichlobenil in animals to be of secondary importance to the animal metabolism of 2,6-dichlorobenzamide.

Animal metabolism: 2,6-dichlorobenzamide

A lactating goat was dosed orally with ^{14}C -phenyl 2,6-dichlorobenzamide at 13 ppm in feed daily for five days. Milk was sampled twice daily, and the animals were sacrificed 23 hours after the final dose.

The majority of the dose was excreted. In the low dose animal, 62% of the TAR was recovered in urine, 17% in faeces, 3.4% in cage washings, and 0.26% in milk. TRRs in milk reached a plateau of 0.048 mg eq./kg on day 3 of dosing. Total residues in muscle and fat at sacrifice were 0.25 and 0.024 mg eq./kg respectively, with higher residues in the metabolic organs, 9.4 and 1.2 mg eq./kg in liver and kidney respectively.

High proportions of the residue were extracted from the matrices using column extraction methods: > 90% in all cases.

Except for liver, unchanged 2,6-dichlorobenzamide was the most significant residue component, at 0.75 mg eq./kg in kidney (63% of TRR), 0.19 mg eq./kg in muscle (77% of TRR), 0.009 mg eq./kg in fat (40% of TRR), and 0.038 mg eq./kg (80% of TRR) in 72 hour milk.

In kidney, the cysteine conjugate derivatives 6-chloro-3-hydroxy-2-thiobenzamide and 6-chloro-3-hydroxy-2-methylthiobenzamide were also found at 0.25 mg eq./kg (21% of TRR) and 0.23 mg eq./kg (19% of TRR) respectively.

In liver, the most significant residue component was 6-chloro-3-hydroxy-2-thiobenzamide at 6.6 mg eq./kg (70% of TRR), followed by 6-chloro-3-hydroxy-3-methylthiobenzamide at 1.6 mg eq./kg (17% of TRR). Much smaller amounts of 2,6-dichlorobenzamide and 6-chloro-3-hydroxy-2-methylsulfinylbenzamide (0.09–0.16 mg eq./kg, or 0.98–1.7% of TRR) were also found in liver.

Small amounts of a component postulated as dichlobenil only on the basis of retention time comparison with a standard were found in muscle and fat, at 0.01 mg eq./kg (4.4% of TRR) and 0.005 mg eq./kg (24% of TRR) respectively.

The only component found in milk apart from 2,6-dichlorobenzamide was 6-chloro-3-hydroxy-2-thiobenzamide, at 0.006 mg eq./kg (13% of TRR).

Laying hens were dosed orally with ^{14}C -phenyl 2,6-dichlorobenzamide at a mean level of 12 ppm in feed daily for five days. Eggs were sampled twice daily, and the birds were sacrificed 23 hours after the final dose.

Around half the administered dose for the low dose group was excreted, with 49% recovered from excreta and 1.5% from cage washings. In the low dose hens 3.7% of the dose was recovered in eggs, with levels not having peaked over the five day dosing period, reaching 2.4 mg eq./kg in the final day eggs. Total residues of 8.6 mg eq./kg were found in liver and 5.0 mg eq./kg in kidney. Residues were lower in fat, skin and muscle, at 1.3, 1.9 and 1.9 mg eq./kg.

Extraction was essentially quantitative, with > 95% of the TRR extracted from all matrices.

With the exception of muscle and egg, where a single additional unidentified metabolite was found at < 6% of the TRR, the extracted residue in hen matrices comprised only unchanged 2,6-dichlorobenzamide.

While 2,6-dichlorobenzamide is largely unmetabolized by hens, the metabolism of 2,6-dichlorobenzamide in goats is somewhat similar to that of dichlobenil, with the main pathway being cysteine conjugation replacing one of the chlorine atoms, followed by cleavage and oxidation of the cysteine side chain to yield metabolites such as 6-chloro-3-hydroxy-2-thiobenzamide, 6-chloro-3-hydroxy-2-methylthiobenzamide, and 6-chloro-3-hydroxy-2-methylsulfinylbenzamide.

Plant metabolism

The Meeting received information on the metabolism of ^{14}C -phenyl labelled dichlobenil in apples and grapes.

Single outdoor grown apple trees were treated in mid-February at late dormancy with a soil application of a liquid formulation of ^{14}C -phenyl dichlobenil at 6.7 kg ai/ha, the GAP for most fruit crops. Total radioactive residues were determined in soil samples collected at various intervals, as well as in immature apples collected at 77 and 107 days after soil application, and mature fruit collected at harvest (137 days after application).

Total residues in apples were low, at 0.012, 0.028, and 0.042 mg eq/kg at 77, 107, and 137 (mature harvest) days after application, respectively. The majority of the residue in mature apples, 81% of the TRR, or 0.034 mg eq./kg, could be extracted with methanol. The only residue identified in apples was 2,6-dichlorobenzamide, at 0.024 mg eq./kg, or 57% of the TRR. Unidentified components were all < 0.01 mg eq/kg.

Small plots of outdoor grown grapevines were treated in mid-February at late dormancy with a soil application of a liquid formulation of ^{14}C -phenyl dichlobenil at 6.7 kg ai/ha. Total radioactive residues were determined in soil samples collected at various intervals, as well as in immature fruit collected at 159 and 190 days after soil application, and mature grapes collected at harvest (222 days after application).

The total radioactive residues in grapes were similar at the three sampling intervals, 0.32, 0.29, and 0.39 mg eq./kg at 159, 190 and 222 days respectively. Nearly all the residue in mature grapes collected at harvest could be extracted with methanol (99% of TRR, or 0.39 mg eq./kg). The most significant residue component in grapes was by far 2,6-dichlorobenzamide, at 0.34 mg eq./kg, or 87% of the TRR. A second metabolite, 2,6-dichloro-4-hydroxybenzamide was found at < 0.01 mg eq./kg, or 2% of the TRR, while unidentified components totalled 0.06 mg eq./kg, or 15% of the TRR.

The only significant metabolic pathway for dichlobenil in fruits is hydrolysis to give 2,6-dichlorobenzamide.

Environmental fate

As all residue trials considered by the Meeting involve soil application, in accordance with the FAO Manual, data for aerobic soil metabolism, soil surface photolysis, and hydrolysis were evaluated.

Dichlobenil

In an aerobic metabolism study conducted over 50 weeks at 24 °C in sandy loam soil, dichlobenil was principally lost through volatilization of the unchanged parent compound (57% of TAR after 50 weeks), and also by metabolism to 2,6-dichlorobenzamide (13% TAR), formation of bound residue (10% TAR), and mineralization to $^{14}\text{CO}_2$ (< 3%). The DT_{50} for dichlobenil was 13 weeks.

The Meeting noted that dichlobenil has low solubility in water, 14.6 mg/L at 20 °C. Hydrolysis of dichlobenil was insignificant, with no significant degradation in unsterilized aqueous buffer solutions over 150 days.

2,6-Dichlorobenzamide

The DT_{50} values for 2,6-dichlorobenzamide were up to 261 days (37 weeks) in soil. The Meeting noted that 2,6-dichlorobenzamide has a significantly higher water solubility than dichlobenil (2.7 g/L), and is very stable to hydrolysis.

The Meeting concluded that dichlobenil is principally lost from soil through volatilization of the parent compound, with secondary degradation pathways including metabolism to 2,6-dichlorobenzamide, binding to soil, and mineralization to CO_2 . Dichlobenil is relatively persistent, with a DT_{50} in soil of 13 weeks. Hydrolysis is not a significant pathway for degradation.

Residues in succeeding crops

No information on residues of dichlobenil in following crops was received by the Meeting, however such data is not needed given that only uses in permanent crops are under consideration.

Based on the available soil degradation data for 2,6-dichlorobenzamide in soil, the Meeting considers that there is potential for accumulation of soil residues of 2,6-dichlorobenzamide from application in multiple years. Modelling of multiple year soil accumulation using the DT_{50} value of 261 days for 2,6-dichlorobenzamide as a worst case indicates that residues of 2,6-dichlorobenzamide will reach a steady state level of $1.6\times$ the level resulting from a single application.

Methods of analysis

The Meeting received details of analytical methods for dichlobenil and 2,6-dichlorobenzamide residues in plant and animal matrices.

Analyses of dichlobenil in plant commodities involved extraction with ethyl acetate/hexane in the presence of anhydrous sodium sulphate, with clean-up by solid phase extraction using deactivated neutral alumina as the solid phase. Residues were determined by GC-ECD or GC-MS/MS. The method LOQ is 0.01 or 0.02 mg/kg.

Analyses of 2,6-dichlorobenzamide in plant commodities involved extraction with ethyl acetate in the presence of anhydrous sodium sulphate and filter pulp or diatomaceous earth. The extracts were cleaned up by solid phase extraction (neutral alumina), followed by analysis using GC-ECD or GC-MS/MS. The method LOQ is 0.003–0.03 mg/kg.

Mean recoveries in plant matrices were generally within the acceptable range of 70–120%, with the exception of 2,6-dichlorobenzamide fortified in grape juice at 0.003 mg/kg. However, acceptable recoveries were achieved at fortifications of 0.03 and 0.30 mg/kg.

Residues of dichlobenil and 2,6-dichlorobenzamide in animal commodities were determined using a GC-MS method. Fat samples were homogenized with hexane then partitioned into acetonitrile. Kidney, muscle and liver samples were homogenized with water and extracted with ethyl acetate. Residues were transferred into hexane and partitioned into acetonitrile. Milk samples were extracted with ethyl acetate, and the residues transferred into hexane, which was cleaned up by solid phase extraction (Florisol). An LOQ of 0.01 mg/kg was achieved for both analytes in all matrices.

Mean recoveries in animal matrices were within the acceptable range of 70–120%.

Suitable methods are therefore available for determination of both dichlobenil and 2,6-dichlorobenzamide residues in plant and animal commodities with an LOQ of 0.01 mg/kg for each analyte.

Multiresidue methods were not provided to the Meeting. However, the Meeting noted that the USA FDA multiresidue method PAM I has been successfully validated for dichlobenil and 2,6-dichlorobenzamide. The Meeting also noted successful validations of the QuEChERS method for determination of both dichlobenil and 2,6-dichlorobenzamide residues.

Stability of residues in stored analytical samples

Storage stability data for residues of dichlobenil and 2,6-dichlorobenzamide in plant and animal commodities were generated as part of the residue trial and animal feeding studies.

The data showed that residues of dichlobenil and 2,6-dichlorobenzamide were stable in plant samples for at least the period for which the residue trial and animal feeding study samples were stored between collection and analysis (up to 11 months in the case of grapes and grape juice).

Storage stability of 2,6-dichlorobenzamide residues in animal matrices was acceptable over the storage period in the feeding study (up to 71 days), while dichlobenil residues were stable in muscle, fat and milk. Poor stability was observed in liver and kidney, however based on the available metabolism and feeding data, detectable residues of dichlobenil parent compound are not expected in any animal commodities.

Definition of the residue

Plant metabolism studies were available for dichlobenil in fruit crops. In metabolism studies in apples and grapes where radiolabelled compound was applied to the soil, dichlobenil was metabolised almost exclusively to 2,6-dichlorobenzamide, which comprised 57% of the TRR in mature apples, and 86% in mature grapes. No dichlobenil parent compound was found in either apples or grapes, and only one other component, 2,6-dichloro-4-hydroxybenzamide, was found, albeit at a very low level, < 0.01 mg eq./kg, or 2.1% of the TRR, in grapes only. The JMPR considered that this was not of greater toxicological concern than 2,6-dichlorobenzamide and was covered by the toxicological evaluation of 2,6-dichlorobenzamide and dichlobenil.

It is further noted that in residue trials in stone fruit, grapes, and bush berry crops, where both dichlobenil and 2,6-dichlorobenzamide were both analysed, only 2,6-dichlorobenzamide was found at quantifiable levels.

Given that dichlobenil is almost exclusively metabolised to 2,6-dichlorobenzamide in the edible portions of fruit crops, the Meeting proposes that only 2,6-dichlorobenzamide be included in the residue definition for dichlobenil in plant commodities for both compliance with MRLs and dietary risk assessment.

In considering a residue definition for animal commodities, the Meeting noted that livestock are only likely to be exposed to 2,6-dichlorobenzamide residues through feeding, as parent compound is unlikely to be found in feeds, while 2,6-dichlorobenzamide may be present in finite amounts. Therefore, only the livestock metabolism studies for 2,6-dichlorobenzamide are of significance.

In hens, 2,6-dichlorobenzamide was largely unchanged, being the only residue detected in fat, liver and kidney, and the major residue in muscle and eggs. One unidentified component was detected in muscle and eggs but at low levels (< 6% of the TRR). In goats, unchanged 2,6-dichlorobenzamide was the largest residue component in kidney, muscle, fat, and milk. In liver, 6-chloro-3-hydroxy-2-thiobenzamide was the largest component, with 2,6-dichlorobenzamide only being present at low levels. The JMPR considered that this metabolite and others found in lactating goats were not of greater toxicological concern than 2,6-dichlorobenzamide and were covered by the toxicological evaluation of 2,6-dichlorobenzamide and dichlobenil. Given that 2,6-dichlorobenzamide is not significantly metabolised in poultry, and is the most significant residue in the milk and all tissues except liver of goats dosed with the compound, the Meeting proposes that only 2,6-dichlorobenzamide will be included in the residue definition for dichlobenil in animal commodities for both compliance with MRLs and for dietary risk assessment.

The $\log_{10}P_{ow}$ of 2,6-dichlorobenzamide (0.77) indicates low fat solubility. In the goat and hen metabolism studies, residues of 2,6-dichlorobenzamide were higher in muscle than in fat. Similar observations were made in the cattle feeding study. The Meeting concluded that the residues are not fat soluble.

It is noted that 2,6-dichlorobenzamide is not uniquely a metabolite of dichlobenil, as it is also a metabolite of the fungicide fluopicolide, evaluated by JMPR in 2009. A residue definition of *fluopicolide* was established for compliance with the MRLs for plant and animal commodities, and *fluopicolide* and 2,6-dichlorobenzamide, measured separately, for estimation of dietary intake for plant and animal commodities. Fluopicolide parent compound is the major residue resulting from use of fluopicolide. When 2,6-dichlorobenzamide is present in the absence of fluopicolide, it is most likely to have resulted from use of dichlobenil.

The Meeting considered residues of 2,6-dichlorobenzamide resulting from use of both dichlobenil and fluopicolide for establishment of MRLs and for dietary risk assessment.

The Meeting proposed the following definition of the residue (for compliance with the MRL and for dietary risk assessment, for plant and animal commodities): 2,6-dichlorobenzamide.

The residue is not fat soluble.

Residues of supervised residue trials on crops

The Meeting received supervised trial data for soil application of dichlobenil to stone fruit (peaches, cherries and plums) and berry fruit (grapes, raspberries, blackberries, cranberries and blueberries).

The Meeting noted that residues of 2,6-dichlorobenzamide may arise from use of fluopicolide as well as from use of dichlobenil. Therefore, the Meeting has estimated maximum residue levels and STMR and HR values for 2,6-dichlorobenzamide in crops treated with fluopicolide using the data evaluated by the 2009 Meeting.

Stone fruits***Cherries***

The critical GAP for dichlobenil in cherries is in the USA, where soil application at 6.7 kg ai/ha can be made in late autumn (15 November to 15 February) or early spring (up to 1 May). A harvest withholding period is not specified.

In trials on cherries conducted in the USA, $1 \times 7.4\text{--}7.6$ kg ai/ha soil application was made to cherry trees bearing immature fruit, with mature fruit being harvested 28 days later. Residues of 2,6-dichlorobenzamide were < 0.003 (12), and 0.004 (2) mg/kg.

The Meeting noted that most of the trials were conducted with application significantly later than specified on the label (in June or July rather than prior to 1 May), after fruit set. Insufficient trials matching GAP are available. Therefore, the Meeting did not estimate a maximum residue level for cherries.

Peaches

The GAP for dichlobenil in peaches in Canada is soil application at 7 kg ai/ha in late autumn or early spring, with no more than 7 kg ai/ha per season. A harvest withholding period is not specified.

In trials on peaches conducted in the USA, $1 \times 6.8\text{--}7.7$ kg ai/ha soil application was made, with fruit being harvested 20-68 days later. Residues of 2,6-dichlorobenzamide in peaches were < 0.01 , 0.01, 0.02, and 0.04 mg/kg.

The Meeting determined that there were insufficient data for establishment of a maximum residue level in peaches.

Plums

The GAP for dichlobenil in plums in Canada is soil application at 7 kg ai/ha in late autumn or early spring, with no more than 7 kg ai/ha per season. A harvest withholding period is not specified.

In trials on plums conducted in the USA, $1 \times 6.7\text{--}7.9$ kg ai/ha soil application was made, with fruit being harvested 139-154 days later. Residues of 2,6-dichlorobenzamide in plums were < 0.01 and 0.45 mg/kg.

The Meeting determined that there were insufficient data for establishment of a maximum residue level in plums.

Berries and other small fruits***Blueberries***

The GAP in the USA for blueberries involves soil application at 6.7 kg ai/ha, with application in late autumn (15 November to 15 February) or early spring (up to 1 May). No withholding period is specified.

In trials conducted on blueberries, a single soil application of dichlobenil was made in spring at 4.3 kg ai/ha. Residues of 2,6-dichlorobenzamide in blueberries at normal harvest (at a PHI of 51–58 days) were < 0.01 (3), and 0.015 mg/kg.

The Meeting determined that there were insufficient data for establishment of a maximum residue level in blueberries. Further, the Meeting noted that the trials were not conducted at the label rate, and the results were not amenable to the use of proportional scaling given that application was made at below GAP and the majority of the results were <LOQ.

Caneberries

The critical GAP for blackberries and raspberries in the USA is a soil application at 4.5 kg ai/ha made in late autumn (15 November to 15 February) or early spring (up to May 1). No withholding period is specified.

In trials conducted on blackberries and raspberries in the USA, where a single soil application was made at 4.3–4.9 kg ai/ha during spring (between mid-April and mid-May) or early summer (one trial with a mid-June application), residues of 2,6-dichlorobenzamide in mature blackberries and raspberries at normal harvest (44-89 DAT) were < 0.01 (2), 0.01, 0.02, 0.021, 0.031, 0.035, 0.056, and 0.067 mg/kg.

The Meeting noted that although a number of trials were conducted with application after 1 May, there did not appear to be a correlation between the application timing/interval and the residue level, and considered that all the trials were sufficiently robust to provide a realistic estimate of the residues.

The Meeting noted the potential for accumulation of residues of 2,6-dichlorobenzamide from application in successive years and agreed to scale the above values to account for multiple year applications by the factor of 1.6 $\times$ determined from modelling of the multiple year soil accumulation. The resultant scaled data set was: < 0.016 (2), 0.016, 0.032, 0.034, 0.050, 0.056, 0.090, and 0.11 mg/kg.

Based on the above scaled data set, the Meeting estimated a maximum residue level of 0.2 mg/kg for the subgroup caneberries, along with an STMR of 0.034 mg/kg, and an HR of 0.13 mg/kg (the scaled highest residue from an individual sample).

Cranberries

The GAP for cranberries in the USA is soil application at 4.5 kg ai/ha made in late autumn or early spring (no more than 4.5 kg ai/ha in a 12-month period).

In trials conducted on cranberries in the USA, where a single soil application was made during spring at 4.8-5.7 kg ai/ha, residues of 2,6-dichlorobenzamide in cranberries at normal harvest were 0.02 (2) mg/kg.

The Meeting determined that there were insufficient data for establishment of a maximum residue level in cranberries.

Grapes

GAP for grapes in the USA involves soil application at 6.7 kg ai/ha made in early spring, with a harvest withholding period not specified.

In trials conducted in the USA on grapes, where a single soil application was made between the end of dormancy, and the beginning of fruit set, at a rate of 6.7-8.3 kg ai/ha, residues of 2,6-dichlorobenzamide in grapes at normal harvest were 0.004, 0.029, 0.033, 0.042 (2), and 0.058 mg/kg.

The Meeting determined that six trials was insufficient for estimation of a maximum residue level for grapes.

Residues of 2,6-dichlorobenzamide in grapes resulting from use of fluopicolide (2009 evaluation) were: < 0.01 (28), 0.01 (2), 0.013, 0.014, 0.015, 0.02 (2), 0.026, 0.03, 0.037, and 0.04 mg/kg.

Based on the 2,6-dichlorobenzamide residues arising from use of fluopicolide, the Meeting estimated a maximum residue level of 0.05 mg/kg for grapes, together with an STMR of 0.01 mg/kg and an HR of 0.04 mg/kg.

Crops with a fluopicolide use but no dichlobenil use

Bulb vegetables

Residues of 2,6-dichlorobenzamide in bulb onions arising from use of fluopicolide were < 0.01 mg/kg (7). The Meeting estimated a maximum residue level of 0.01* mg/kg for 2,6-dichlorobenzamide in bulb onions, together with STMR and HR values of 0.01 mg/kg.

Residues of 2,6-dichlorobenzamide in Welsh onions arising from use of fluopicolide were < 0.01 (2), and 0.01 mg/kg. The Meeting estimated a maximum residue level of 0.02 mg/kg for 2,6-dichlorobenzamide in Welsh onions, together with STMR and HR value of 0.01 mg/kg.

Brassica vegetables

In the data set for head cabbage (with wrapper leaves) used by the 2009 Meeting for fluopicolide MRL estimation and dietary risk assessment, residues of 2,6-dichlorobenzamide were < 0.01 (6), and 0.02 mg/kg.

Residues of 2,6-dichlorobenzamide in Brussels sprouts arising from use of fluopicolide were < 0.01 (8) mg/kg.

In the USA data set for broccoli used by the 2009 Meeting for MRL estimation and dietary risk assessment for Flowerhead brassicas were < 0.01 (6) mg/kg.

The Meeting noted that 2,6-dichlorobenzamide residues of < 0.01 (6), 0.02, and 0.04 mg/kg were found in head cabbage grown as a rotational crop.

The Meeting agreed to combine the head cabbage dataset for 2,6-dichlorobenzamide residues resulting from in-crop use of fluopicolide with the dataset for residues of 2,6-dichlorobenzamide in head cabbage resulting from use of fluopicolide in a preceding crop:

< 0.01 (12), 0.02 (2), and 0.04 mg/kg

Recognizing that residues of 2,6-dichlorobenzamide could occur in brassica vegetables other than cabbage grown in rotation with a crop treated with fluopicolide, the Meeting decided to estimate a group maximum residue level of 0.05 mg/kg, together with an STMR of 0.01 mg/kg and an HR of 0.04 mg/kg, based on the combined head cabbage dataset.

Fruiting vegetables, Cucurbits

In the dataset for melons used by the 2009 Meeting for estimation of a group MRL for fruiting vegetables, Cucurbits, residues of 2,6-dichlorobenzamide were not detected: < 0.01 (9) mg/kg. It is noted that residues of 2,6-dichlorobenzamide were not detected in other cucurbit crops. The Meeting estimated a group maximum residue level of 0.01* mg/kg for fruiting vegetables, Cucurbits, together with an STMR and an HR of 0.01 mg/kg.

Fruiting vegetables, other than Cucurbits

In the data set in peppers, sweet and peppers, Chilli used by the 2009 Meeting to estimate a group maximum residue level for fruiting vegetables, other than Cucurbits, residues of 2,6-dichlorobenzamide were: < 0.01 (10) mg/kg. It is noted that residues of 2,6-dichlorobenzamide were not detected in other non-cucurbit fruiting vegetable crops. The Meeting estimated a group maximum residue level of 0.01* mg/kg for 2,6-dichlorobenzamide in fruiting vegetables, other than Cucurbits, together with an STMR and an HR of 0.01 mg/kg.

As residues of 2,6-dichlorobenzamide were not detected in chilli peppers treated with fluopicolide in the trials reported by the 2009 JMPR, the Meeting estimated an MRL of 0.01* mg/kg for 2,6-dichlorobenzamide in peppers, Chili, dried, together with an STMR and an HR of 0.01 mg/kg.

Leafy vegetables

The spinach data set was used by the 2009 Meeting to estimate a group MRL for leafy vegetables. Residues of 2,6-dichlorobenzamide in spinach arising from use of fluopicolide were 0.02, 0.03, 0.06, 0.07 (2), 0.09, and 0.19 mg/kg. The Meeting estimated a maximum residue level of 0.3 mg/kg for 2,6-dichlorobenzamide in leafy vegetables, together with an STMR of 0.07 mg/kg and an HR of 0.19 mg/kg.

Celery

In the dataset considered by the 2009 Meeting, residues of 2,6-dichlorobenzamide resulting from use of fluopicolide in celery were < 0.01 (4), 0.01, 0.03 and 0.04 mg/kg. The Meeting estimated a maximum residue level of 0.07 mg/kg for 2,6-dichlorobenzamide in celery, together with an STMR of 0.01 mg/kg and an HR of 0.04 mg/kg.

Rotational crops

Residues arising in rotational brassica crops are covered in the appropriate section above.

Low levels of 2,6-dichlorobenzamide residues were found in rotational pulse and cereal forages and fodders after application of fluopicolide to a preceding crop.

Residues of 2,6-dichlorobenzamide in rotational faba beans were < 0.01 (8) mg/kg.

The Meeting estimated a maximum residue level of 0.01* mg/kg for faba bean (dry), together with an STMR and an HR of 0.01 mg/kg. The Meeting agreed to extrapolate these values to establish a group maximum residue level for pulses.

Residues of 2,6-dichlorobenzamide in faba bean forage were < 0.01 (3), 0.01, 0.03, 0.06 (2), and 0.10 mg/kg (as received).

The Meeting estimated a median residue of 0.02 mg/kg, and a highest residue of 0.10 mg/kg for bean forage on an as received basis. The Meeting agreed to extrapolate these values to legume animal feeds.

Residues of 2,6-dichlorobenzamide in rotational wheat were < 0.01 (9) mg/kg.

The Meeting estimated a maximum residue level of 0.01* mg/kg for wheat, together with STMR and HR values of 0.01 mg/kg. The Meeting agreed to extrapolate these values to estimate a group maximum residue level and STMR/HR values for cereal grains.

Residues of 2,6-dichlorobenzamide in rotational wheat forage were < 0.01 (6), 0.01 (2), and 0.02 mg/kg (as received basis).

The Meeting estimated a median residue of 0.01 mg/kg and a highest residue of 0.02 mg/kg for wheat forage on an as received basis. The Meeting agreed to extrapolate these figures to cereal forage.

Residues of 2,6-dichlorobenzamide in rotational wheat hay (stalks and/or ears) were < 0.01 (6), 0.01, 0.03, and 0.06 mg/kg (as received).

As it was not clear that the samples had been dried, these values were converted to a dry weight basis assuming the 25% dry matter content for wheat forage: < 0.04 (6), 0.04, 0.12, and 0.24 mg/kg.

Residues of 2,6-dichlorobenzamide in rotational wheat straw were < 0.01 (7), 0.01, and 0.03 mg/kg on an as received basis.

Based on the hay data, the Meeting estimated a maximum residue level of 0.4 mg/kg for wheat straw and fodder, dry. The Meeting agreed to extrapolate this to straw and fodder (dry) of cereal grains.

The Meeting estimated a median residue of 0.04 mg/kg and a highest residue of 0.24 mg/kg for cereal hays based on the wheat hay data set and a median residue of 0.01 mg/kg and a highest residue of 0.03 mg/kg for cereal straws based on the wheat straw data set.

Fate of residues during processing

Plums

A processing study was provided for plums but as there was insufficient data to estimate a maximum residue level in plums, it will not be considered further.

Grapes

A processing study in grapes was provided. Grapes from a plot treated with dichlobenil at a target rate of 8.74 kg ai/ha were processed into juice and raisins, and processing factors of 1.4 and 2.8 respectively were determined for 2,6-dichlorobenzamide.

Based on the processing factor of 2.8 and the grape MRL of 0.05 mg/kg, the Meeting estimated a maximum residue level of 0.15 mg/kg for dried grapes. Based on the STMR and HR of 0.01 and 0.04 mg/kg respectively for grapes, the Meeting estimated an STMR-P of 0.028 mg/kg, and an HR-P of 0.11 mg/kg for dried grapes.

Based on the processing factor of 1.4, and the grape MRL of 0.05 mg/kg, the Meeting estimated a maximum residue level of 0.07 mg/kg for grape juice. Based on the STMR and HR values for grapes, the Meeting estimated an STMR-P of 0.014 mg/kg and an HR-P of 0.056 mg/kg.

Grape pomace is used as a feed for cattle. However, the processing study did not include results for pomace. Given that pomace consists of the dry matter remaining after manufacture of juice or wine, the Meeting estimated an STMR-P value of 0.028 mg/kg for grape pomace, dry, based on the processing data for raisins.

Tomatoes

As residues of 2,6-dichlorobenzamide were not detected in tomatoes, the Meeting confirmed the median residues and STMRs of 0.01 mg/kg estimated by the 2009 Meeting for tomato pomace, juice, paste and ketchup.

Residues in animal commodities

Feeding studies

A feeding study for 2,6-dichlorobenzamide in lactating dairy cattle was provided to the Meeting.

Lactating Holstein dairy cow were dosed daily by capsule with 2,6-dichlorobenzamide at the equivalent of 0.6, 1.6 and 5.3 ppm in the dry weight diet for 28 consecutive days. Milk was collected throughout, and the cattle were slaughtered within 6 hours of the final dose for tissue sampling. 2,6-Dichlorobenzamide residues were found at 0.011 mg/kg in milk from the 1.6 ppm group, and at 0.031 mg/kg in milk from the 5.3 ppm group. Residues of 2,6-dichlorobenzamide were found in most tissues at all feeding levels, except for fat. The relationships between dose and residues of 2,6-dichlorobenzamide in muscle, kidney and liver were linear.

Farm animal dietary burden

Dietary burden calculations incorporating all commodities considered by the current and 2003 Meetings for beef cattle, dairy cattle, broilers and laying poultry are presented in Annex 6. The

calculations are made according to the livestock diets of the USA/Canada, the European Union, Australia and Japan as laid out in the OECD table.

	US/CAN		EU		AU		Japan	
	Max.	Mean	Max.	Mean	Max.	Mean	Max.	Mean
Beef cattle	0.064	0.010	0.31	0.063	0.38	0.077	0.019	0.002
Dairy cattle	0.25	0.054	0.091	0.031	0.39	0.075	0.031	0.013
Poultry – broiler	0.009	0	0.01	0	0.012	0	0.022	0.029
Poultry – layer	0.009	0	0.096	0.017	0.012	0	0.006	0

Animal commodity maximum residue levels

The highest dietary burden for cattle is for Australia (maximum of 0.39 ppm for both beef and dairy cattle and mean values of 0.077 mg/kg for beef cattle and 0.075 mg/kg for dairy cattle).

Scaling the residues observed in milk at a feeding level of 1.6 ppm for the expected Maximum Feeding Level of 0.39 ppm shows that residues are not expected to be found above the LOQ in milk. The Meeting estimated a maximum residue level of 0.01* mg/kg for milk, together with an STMR of 0.01 mg/kg.

The calculated maximum residues for muscle, liver and kidney are tabulated below.

Tissue	Regression equation (forced through origin)	Calculated residue for 0.39 ppm residues of 2,6-dichlorobenzamide in feed (mg/kg)
Liver	$y = 0.0804x$	0.031
Kidney	$y = 0.01x$	0.004
Muscle	$y = 0.0128x$	0.005
Fat	Scaled from residue at LOQ (0.01 mg/kg) at 1.6 ppm feeding level	0.002

The calculated mean residues for muscle, liver and kidney are tabulated below.

Tissue	Regression equation (forced through origin)	Calculated residue for 0.075 ppm residues of 2,6-dichlorobenzamide in feed (mg/kg)
Liver	$y = 0.0584x$	0.004
Kidney	$y = 0.0078x$	0.0006
Muscle	$y = 0.0114x$	0.0009
Fat	Scaled from residue at LOQ (0.01 mg/kg) at 1.6 ppm feeding level	0.0004

Based on the calculated residues, the Meeting estimated a maximum residue level of 0.04 mg/kg for edible offal, mammalian, together with an STMR of 0.01 mg/kg and an HR of 0.031 mg/kg. The Meeting estimated maximum residue levels of 0.01* mg/kg for meat (mammalian, except marine mammals) and mammalian fats, together with STMR and HR values of 0.01 mg/kg.

Poultry

The highest dietary burden for poultry is for European laying hens (mean of 0.017 ppm and maximum of 0.096 ppm).

A poultry feeding study was not provided to the Meeting. In a metabolism study for 2,6-dichlorobenzamide in laying hens, after dosing at 10 ppm daily for five days, residues of 2,6-dichlorobenzamide were 8.5 mg/kg in liver, 4.8 mg/kg in kidney, 1.8 mg/kg in skin, 1.9 mg/kg in muscle, 1.3 mg/kg in fat, and 2.0 mg/kg in eggs.

Scaling the residues in hen matrices for the expected maximum and mean feeding levels in poultry, the following median and highest residues were calculated.

Tissue	Maximum residue	Median residue
Liver	0.081	0.014
Kidney	0.046	0.008
Muscle	0.018	0.003
Fat	0.012	0.002
Eggs	0.019	0.003

Based on the calculations for eggs, the Meeting estimated a maximum residue level of 0.03 mg/kg for eggs, together with an STMR of 0.01 mg/kg and an HR of 0.019 mg/kg.

Based on the calculations for liver, the Meeting estimated a maximum residue level of 0.1 mg/kg for poultry edible offal, together with an STMR of 0.014 mg/kg and an HR of 0.081 mg/kg.

Based on the calculations for muscle, the Meeting estimated an MRL of 0.03 mg/kg for poultry meat, together with an STMR of 0.01 mg/kg and an HR of 0.018 mg/kg.

Based on the calculations for fat, the Meeting estimated an MRL of 0.02 mg/kg for poultry fats, together with an STMR of 0.01 mg/kg and an HR value of 0.012 mg/kg.

RECOMMENDATIONS

Definition of the residue (for compliance with the MRL and for estimation of dietary intake for plant and animal commodities): *2,6-Dichlorobenzamide*.

The residue is not fat soluble.

The Meeting estimated the maximum residue levels, STMR and HR values below:

CCN	Commodity name	Recommended maximum residue level, mg/kg		STMR (P), mg/kg	HR (P), mg/kg
		New	Previous		
VB 0040	Brassica vegetables	0.05 (FL)	-	0.01	0.04
FB 2005	Caneberries	0.2 (D)	-	0.034	0.13
VS 0624	Celery	0.07 (FL)	-	0.01	0.04
GC 0080	Cereal grains	0.01* (FL)	-	0.01	-
DF 0269	Dried grapes (= Currants, Raisins and Sultanas)	0.15 (FL)	-	0.028	0.11
MO 0105	Edible offal (mammalian)	0.04 (FL)	-	0.01	0.031
PE 0112	Eggs	0.03 (FL)	-	0.01	0.019
VC 0045	Fruiting vegetables, Cucurbits	0.01* (FL)	-	0.01	0.01
VO 0050	Fruiting vegetables, other than Cucurbits (except sweetcorn and mushrooms)	0.01* (FL)	-	0.01	0.01
	Tomato juice	-	-	0.01	-
	Tomato purée	-	-	0.01	-
	Tomato paste	-	-	0.01	-
JF 0269	Grape juice	0.07 (FL)	-	0.014	0.056
FB 0269	Grapes	0.05 (FL)	-	0.01	0.04
VL 0053	Leafy vegetables	0.3 (FL)	-	0.07	0.19
MF 0100	Mammalian fats (except milk fats)	0.01* (FL)	-	0.01	0.01
MM 0095	Meat (from mammals other than marine mammals)	0.01* (FL)	-	0.01	0.01
ML 0106	Milks	0.01* (FL)	-	0.01	-
VA 0385	Onion, Bulb	0.01* (FL)	-	0.01	0.01
VA 0387	Onion, Welsh	0.02 (FL)	-	0.01	0.01
HS 0444	Peppers, Chili, dried	0.01* (FL)	-	0.01	0.01
PO 0111	Poultry, edible offal of	0.1 (FL)	-	0.014	0.081
PF 0111	Poultry fats	0.02 (FL)	-	0.01	0.012
PM 0110	Poultry meats	0.03 (FL)	-	0.01	0.018
VD 0070	Pulses (FL)	0.01* (FL)	-	0.01	-
AS 0081	Straw and fodder (dry) of cereal grains (FL)	0.4 (FL)	-	-	-

D = indicates values estimated to accommodate residues of 2,6-dichlorobenzamide arising from dichlobenil use.

FL = indicates values estimated to accommodate residues of 2,6-dichlorobenzamide arising from fluopicolide use.

CCN	Commodity name	Median residue, mg/kg	Highest residue, mg/kg
	Cereal forage (as received) (FL)	0.01	0.02
	Cereal hay (FL)	0.04	0.24
	Cereal straw (FL)	0.011	0.034
	Grape pomace (FL)	0.028	-
	Legume animal feeds (as received) (FL)	0.02	0.10
	Tomato pomace (FL)	0.01	-

FL = indicates values estimated to accommodate residues of 2,6-dichlorobenzamide arising from fluopicolide use.

DIETARY RISK ASSESSMENT

Long-term intake

The ADI for dichlobenil is 0–0.01 mg/kg bw. Finite residues of dichlobenil parent compound are not expected to be found in edible commodities, and parent compound has not been recommended for inclusion in the residue definition for dietary risk assessment. The Meeting concluded that the long-term intake of residues of dichlobenil when used in ways that have been considered by the JMPR is unlikely to present a public health concern.

The ADI for 2,6-dichlorobenzamide is 0–0.05 mg/kg bw. The International Estimated Dietary Intakes (IEDIs) for 2,6-dichlorobenzamide arising from both dichlobenil and fluopicolide were calculated for the 17 GEMS/food cluster diets using STMRs/STMR-Ps estimated by the current Meeting (see Annex 3 of the 2014 JMPR Report). The calculated IEDIs were 0–1% of the maximum ADI (0.05 mg/kg bw). The Meeting concluded that the long-term intakes of residues of 2,6-dichlorobenzamide, resulting from the uses of dichlobenil considered by the current Meeting and from the uses of fluopicolide considered by the 2009 Meeting are unlikely to present a public health concern.

Short-term intake

The ARfD for dichlobenil is 0.5 mg/kg bw (for women of childbearing age only). Finite residues of dichlobenil parent compound are not expected to be found in edible commodities, and parent compound has not been recommended for inclusion in the residue definition for dietary risk assessment. The Meeting concluded that the short-term intake of residues of dichlobenil when used in ways that have been considered by the JMPR is unlikely to present a public health concern.

The ARfD for 2,6-dichlorobenzamide is 0.3 mg/kg bw (for women of childbearing age only). The International Estimated Short-Term Intakes (IESTIs) for 2,6-dichlorobenzamide arising from dichlobenil and fluopicolide were calculated for food commodities and their processed commodities using HRs/HR-Ps or STMRs/STMR-Ps estimated by the current Meeting and by the 2009 Meeting as part of the evaluation of fluopicolide (see Annex 4 of the 2014 JMPR Report). The calculated IESTIs were 0–2% of the ARfD for all commodities. The Meeting concluded that the short-term intake of residues of 2,6-dichlorobenzamide, when dichlobenil and fluopicolide are used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

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DIMETHOMORPH (225)

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EXPLANATION

Dimethomorph, a cinnamic acid derivative, is a member of the morpholine group of fungicides and consists of a mixture of the E and Z isomers in approximately equal proportions. Its mode of action is through the disruption of fungal cell wall formation. When applied as a foliar spray dimethomorph penetrates the leaf surface and is translocated within the leaf to provide protectant action against plant pathogens belonging to class Oomycetes order Peronosporales. The fungicide is selective for downy mildews and members of the genus *Phytophthora*. Dimethomorph was evaluated for the first time by the JMPR in 2007. The 2007 meeting established an acceptable daily intake (ADI) of 0–0.2 mg/kg bw and an acute reference dose (ARfD) of 0.6 mg/kg bw. The residue (for compliance with the MRL and for estimation of dietary intake) for plant and animal commodities was defined as dimethomorph (sum of isomers). Maximum residues levels for 20 commodities were proposed by JMPR 2007.

Dimethomorph was listed by the Forty-fifth Session of the CCPR (2013) for evaluation of additional MRLs. The 2014 Meeting received GAP information and residue trial data from uses as foliar spray, soil drench or drip irrigation in oranges, strawberries, grapes, papaya, bulb onions, leek, spring onions, head cabbage, broccoli, pepper, lettuce leaf, spinach, lettuce head, taro, green peas, vining peas, lima beans, artichoke, and celery. An analytical method for animal matrices was provided as well as validation data for an analytical method in oranges. Processing studies in oranges, strawberry, onion, lettuce head and peas were also provided.

ANALYTICAL METHODS

The Meeting received a summary of validation data for the method Agriquem PE-804 which was developed for determination of dimethomorph residues in citrus fruits (doc. code 2010/1089351). The method (extraction method and extraction efficiency) was already reviewed by the JMPR in 2007. The method validation data for citrus was carried out concurrently with the analysis of the residue trials and not carried out as a separate project. The recoveries obtained as part of method validation for the parent dimethomorph (sum of isomers) in citrus fruit are presented in Table 1.

Table 1 Recovery data for the method Agriquem PE-804 for dimethomorph in citrus

Matrix	reported LOQ mg/kg	spike level	n	% recovery		RSD	Trial references
				mean	range		
Orange pulp	0.01	0.025	8	101.0	96–108	5.9	2006/1015077
		0.05	8	110.8	92–110	6.5	2005/1034175 2006/1015076
		0.1	8	81.6	73–88	6.0	2006/1015076
		0.2	4	97.3	94–104	4.9	2005/1034175
Orange whole fruit	0.01	0.05	8	88.5	78–106	9.9	2006/1015076 2006/1015076
		0.075	4	91.0	84–106.67	15.8	2005/1034175
		0.1	4	95.5	88–106.0	9.4	2006/1015076
		0.15	8	88.4	81.33–109.33	10.9	2006/1015076
		0.3	4	81.3	81.0–81.67	0.4	2005/1034175
Orange peel	0.01	0.075	6	87.1	72–104	14.4	2006/1015076
		0.1	4	91.0	87.0–95.0	4.5	2005/1034175
		0.15	6	75.3	71.33–78.67	3.9	2006/1015076
		0.2	4	100.4	92.0–106.5	6.1	2006/1015076
		0.25	4	87.0	77.2–79.2	12.2	2006/1015077
		0.4	4	86.7	73.0–93.25	10.7	2005/1034175

The recovery in all the matrices and fortification levels was in the acceptable range 70–110%. The relative standard deviation for all matrices was within the adequate level below 20%.

A new analytical method for animal matrices was submitted to the 2014 Meeting. In this method dimethomorph is extracted with methanol/water/hydrochloric acid. The extract is partitioned against cyclohexane at alkaline conditions and then evaporated to dryness and dissolved in a methanol/water mixture. The final determination of dimethomorph is performed by HPLC-MS/MS at two transitions. Transition m/z 388 $\rightarrow$ 301 is proposed as target transition for quantification and transition m/z 388 $\rightarrow$ 165 for confirmatory purposes. The limit of quantification is 0.01 mg/kg for dimethomorph in milk, egg, muscle and liver.

Table 2 Recovery result of dimethomorph residues using method LO138/01 in different animal tissues

Sample Matrix	Dimethomorph	Fortification level	Average recovery%	RSD%	No of analysis
Egg	Dimethomorph m/z 388-301	0.01	100.4	1.9	5
		0.1	100.9	2.3	5
	Dimethomorph m/z 388-165	0.01	97.8	1.9	5
		0.1	100.9	1.6	5
Fat	Dimethomorph m/z 388-301	0.01	95.1	0.9	5
		0.1	105.2	2.5	5
	Dimethomorph m/z 388-165	0.01	96.7	2.6	5
		0.1	104.8	1.8	5
Kidney	Dimethomorph m/z 388-301	0.01	99.5	3.9	5
		0.1	98.1	2.6	5
	Dimethomorph m/z 388-165	0.01	97.6	4.0	5
		0.1	99.1	3.8	5
Liver	Dimethomorph m/z 388-301	0.01	97.2	4.8	5
		0.1	98.8	2.9	5
	Dimethomorph m/z 388-165	0.01	98.0	2.4	5
		0.1	98.7	3.1	5
Milk	Dimethomorph m/z 388-301	0.01	103.8	2.9	5
		0.1	101.4	4.9	5
	Dimethomorph m/z 388-165	0.01	105.4	3.3	5
		0.1	103.4	4.5	5
Muscle	Dimethomorph m/z 388-301	0.01	101.6	3.4	5
		0.1	96.8	5.2	5
	Dimethomorph m/z 388-165	0.01	103.4	3.0	5
		0.1	98.5	5.4	5

The method was independently validated with recoveries in milk, egg, muscle and liver in the range of 70–110% and a RSD of $\leq 20\%$.

USE PATTERN

Dimethomorph is a morpholine fungicide with protective action against plant pathogenic Phytophthora species and a number of downy mildew diseases on fruit, vegetables and potato.

The fungicide is registered in around 100 countries worldwide. Information on registered uses together with labels for dimethomorph was provided to the Meeting by one manufacturer. The representative uses relating to the crops under consideration for additional MRLs and revising some of the existing CODEX MRLs are summarized in the following tables.

Table 3 Selected representative uses of dimethomorph on berries and other small fruit as well as fruit and assorted and sub-tropical fruits crops and bulb vegetables (from labels provided)

Crop	Country	Form*	Application per treatment			Interval (days)	PHI (days)	Notes
			Max no	Conc kg ai/hL	Rate kg ai/ha			
Grapes	US (not registered for use in California)	SC 43.5%	4	0.03–0.05 (concentrated) 0.006–0.02 diluted 0.12 per ground application 0.47 per aerial application	0.219 ^a	7 ⁱ	14	Spray
Grapes	US	SC 20.2%	2	0.12 per ground application 0.48 per aerial application	0.16–0.206 ^b	7–10 ^j	14	Spray
Strawberries (outdoor and protected)	Belgium	WP 50%	3	0.02–0.05	0.05 g ai/plant	1		Root drench
Strawberries (outdoor and protected)	Belgium	WP 50%	1	0.025–0.05		^m	35	Drip irrigation
Strawberries (protected)	Ireland	WP 50%	1	0.003–0.05	0.05 g ai/plant at least	^m	35	Drip irrigation or root drench
Strawberries (outdoor)	Ireland	WP 50%	1	0.15–0.25	1.5	^m	35	Spray
Strawberries (protected)	The Netherlands	WP 50%	1	0.05	0.05 g ai/plant	^m	35	Drip irrigation
Strawberries (outdoor)	The Netherlands	WP 50%	1	0.375–0.75	1.5	^p	35	Spray
Strawberries	Taiwan	WG 18.7%	2	0.006	0.072–0.096	7 ⁿ	5	Spray
Strawberries	Taiwan	WP 50%	2	0.004–0.01	0.15–0.2	7 ⁿ	6	Spray
Strawberries (outdoor)	UK	WP 50%	1	0.15–0.25	1.5	^m	35	Spray
Strawberries (protected)	UK	WP 50%	1	0.025–0.05	0.05 g ai/plant	^m	35	Drip irrigation or root drench
Bulb vegetables Garlic, Garlic great headed, Leek, Onion dry bulb, Onion green, Onion Welsh Shallot	Canada	SC 22.5	3 ^g	0.12 per ground application 0.49 per aerial application	0.225 ^c	5–7 ^k	0	Spray
Bulb vegetables Garlic, Garlic great headed, Leek, Onion dry bulb,	USA	SC 43.5%	3	0.12 per ground application 0.47 per aerial application	0.19 ^d	5 ^k	0	Spray

Crop	Country	Form*	Application per treatment			Interval (days)	PHI (days)	Notes
			Max no	Conc kg ai/hL	Rate kg ai/ha			
Onion green, Onion Welsh Shallot								
Bulb vegetables Garlic, Garlic great headed, Leek, Onion dry bulb, Onion green, Onion Welsh Shallot	USA	SC 20.2%	3 ^g	0.12 per ground application 0.49 per aerial application	0.206 ^e	5–7 ^k	0	Spray
Leek	Austria	WG 90 g/kg	3	0.03–0.045	0.18	7–14	21	Spray
Leek	France	DG 9%	2	0.023–0.036	0.18	7–10 ^q	14	Spray
Leek	Germany	WG 90 g/kg	3	0.03–0.045	0.18	7–14	21	Spray
Leek	Mexico	SC 8%	3	0.029–0.83	0.20–0.25	7–14	7	Spray
Leek	Norway	WG 90 g/kg	4	0.045–0.06	0.18	7–14	14	Spray
Leek	UK	DG 7.5%	3	0.03–0.075	0.15	7–14 ^k	21	Spray
Spring onions (Green onions)	Belgium	WG 7.5%	4	0.038–0.09	0.188	7–10	14	Spray
Spring onions (Green onions)	Hungary	WG 9%	4	0.03–0.09	0.180	7 ^o	14	Spray
Spring onions	Luxemburg	WG 7.5%	4	0.038–0.09	0.188	7–10	14	Spray

^a Maximum rate per season is 0.95 kg ai/ha

^b Maximum rate per season is 0.826 kg ai/ha

^c Maximum rate per season is 1.12 kg ai/ha

^d Maximum rate per season is 0.953 kg ai/ha

^e Maximum rate of 0.619 kg ai/ha

^f Maximum rate of 0.44 kg ai/ha

^g Do not make more than two sequential applications before alternating to a labelled fungicide with different mode of action fungicide

^h Apply at transplanting

ⁱ Apply after transplanting

^j From transplanting until growth resumption in spring

^k Apply prior to disease development

^l Apply at transplanting, one month later and at growth resumption in spring

^m Apply direct after planting

ⁿ Apply at beginning of infection

^o Last treatment at skin development

^p Apply direct after planting in propagation fields, waiting beds and production fields

^q Carry out two applications during the winter protection period at a rate of a minimum of 14 days, of a maximum of 21 days

Table 4 Representative uses of dimethomorph on brassica vegetables (from labels provided)

Crop	Country	Form*	Application per treatment			Interval (days)	PHI (days)	Notes
			Max no	Conc kg ai/hL	Rate kg ai/ha			
Head cabbage	Belgium	WP 50 g/L	1–2		1.5	7–10 ^f	na	Spray
Head cabbage	USA	SC 20.2%	3 ^a	0.12 per ground application 0.49 per aerial application	0.206 ^b	7 ^g	0	Spray
Head cabbage	USA	SC 43.5%	3	0.12 per ground application 0.47 per aerial application	0.19 ^c	7 ^g	0	Spray
Broccoli	Belgium	WP 50 g/L	2		1.5	7–10 ^h	na	Spray
Broccoli	USA	SC 20.2%	3 ^a	0.12 per ground application 0.49 per aerial application	0.206 ^b	7 ^g	0	Spray
Broccoli	USA	SC 43.5%	3	0.12 per ground application 0.47 per aerial application	0.19 ^c	7 ^g	0	Spray
Brassica leafy vegetables, (Broccoli and others).	Canada	SC 22.5%	3 ^e	0.12 per ground application 0.49 per aerial application	0.225	5–7 ^g	0	Spray

^a Two is maximum number of sequential applications before altering to a labelled fungicide with different mode of action

^b Maximum rate per season is 0.619 kg ai/ha

^c Maximum rate per season is 0.953 kg ai/ha

^d Maximum rate per season is 1.169 kg ai/ha

^e Do not make more than 2 sequential applications before alternating to a labelled fungicide with different mode of action fungicide

na = Not applicable

^f Treatment before planting

^g Apply prior to disease development

^h Treatment before planting

Table 5 Selected representative uses of dimethomorph on fruiting vegetables other than cucurbits

Crop	Country	Form*	Application per treatment			Interval (days)	PHI (days)	Notes
			Max no	Conc kg ai/hL	Rate kg ai/ha			
Pepper, all varieties	Canada	SC 225 g/L	3 ^a	use min. 200 L/ha per ground and 50 L/ha per aerial application	0.225	5–7	4	Spray
Fruiting vegetable group tomato, eggplant, ground cherry (Physalis spp.) Pepper (all varieties) Pepino	Canada	WP 50%	5	use min 200 L/ha per ground and 20 L/ha for aerial applications	0.225	5–10 ^h	0	Spray

Crop	Country	Form*	Application per treatment			Interval (days)	PHI (days)	Notes
			Max no	Conc kg ai/hL	Rate kg ai/ha			
Tomatillo								
Pepper	Chile	SC 500 g/L	3	0.036	0.18	10–14	7	Spray
Fruiting vegetables group Eggplant Ground cheery Pepino (all varieties) Tomatillo Tomato	USA	SC 20.2%	2 ^g	0.12 per ground application 0.49 per aerial application	0.206 ^b	5–7 ⁱ	4	Spray
Fruiting vegetables (except tomatoes) Eggplant Ground cherry Pepper (all varieties) Tomatillo	USA Not registered for use in California	SC 43.5%	2 ^{a–5}	0.12 per ground application 0.47 per aerial application	0.19 ^c	5 ^h	0	Spray
Pepper	Vietnam	WP 90 g/kg		0.0036–0.0045/16 L water		7–14	7	Spray
Tomato Non-stalked	USA	SC 43.5%	5 ^f		0.19 ^c	5 ^h	4	Spray
Tomato stalked	USA	SC 43.5%	5 ^f		0.19 ^c	5 ^h	4	Spray

^a Two is maximum number of sequential applications before altering to a labelled fungicide with different mode of action.

^b Maximum rate per season is 0.619 kg ai/ha

^c Maximum rate per season is 0.953 kg ai/ha

^d Maximum rate per season is 1.169 kg ai/ha

^e Maximum rate per season is 1.14 kg ai/ha

^f One is maximum number of sequential applications before altering to a labelled fungicide with different mode of action

^g Do not make more than two sequential applications before alternating to a labelled fungicide with different mode of action fungicide

^h Begin applications when plants are 4–6 inches high and prior to onset of disease infection.

ⁱ Apply prior to disease development

Table 6 Selected representative uses of dimethomorph on Leafy vegetables, Legume vegetable, Root and tuber vegetable (from labels provided)

Crop	Country	Form	Application per treatment			Interval (days)	PHI (days)	Notes
			Max no	Conc kg ai/hL	Rate kg ai/ha			
Lettuce only head varieties	Austria	WP 500 g/kg	4		0.36	7–10	14	Spray
Leafy vegetables	Belgium	WP 500 g/kg	3		0.36	7	14	Spray
Leafy vegetables	Canada	SC 225	3		0.225	5–7	0	Spray
Lettuce	Cyprus	EC 72 g/L	3		0.144–0.18	7–8	7	Spray
Lettuce, open field exclusively	France	90 g/kg WG	3		0.144		28	Spray
Lettuce	Czech Republic	90 g/kg WG	3		0.18	7–12	21	Spray

Crop	Country	Form	Application per treatment			Interval (days)	PHI (days)	Notes
			Max no	Conc kg ai/hL	Rate kg ai/ha			
Leafy vegetables	Germany	WG 90 g/kg	3		0.18	7–12	21	Spray
Leafy vegetables	Greece	SC 22.5%	2		0.18	7–10	7	Spray
Leafy vegetables	Italy	SC 22.5%	2	0.018	0.18	7–10	7	Spray
Lettuce	Italy	EC 72 g/L	3	0.014	0.144	7–10	3	Spray
Protected cultivation on lettuce and lambs lettuce	Netherlands	SC 300 g/L	3		0.24 (0.8 L/ha)	7–12	7	Spray
Protected lettuce	Netherlands	WG 50%			0.15	7–12	14	Spray
Lettuce	Spain	WG 7.5%		0.3–0.35	max 3 kg/ha		7	Spray
Lettuce	Taiwan	WG 50%	3		0.15–0.2	7	6	Spray
Leafy vegetable (except Brassica vegetables)	USA	SC 20.2%	3 ^d	0.12 per ground application 0.49 per aerial application	0.206 ^b	7	0	Spray
Leafy vegetable (except Brassica)	USA	SC 43.5%	3	0.12 per ground application 0.47 per aerial application	0.19 ^c	7	0	Spray
Taro	USA Not for use in California	SC 43.5%	5	0.12 per ground application 0.47 per aerial application	0.19 ^c	7	7 days (leaves) 30 days (corms)	Spray
Peas without pods	Belgium	WG 7.5%	2		0.15	BBCH 15–60	21	Spray
Canned Peas	France	DG 9%	2		0.18	7–10 ^e	21	
Green Peas	Germany	WG 90 g/kg	2		0.18	8–14	21	Spray
Peas	Hungary	WG 90 g/kg	1		0.18			Spray
Peas without pods	Luxemburg	WG 7.5%	2		0.15	BBCH 15–60	21	Spray
Peas	Portugal	DG 9%		0.2	0.18	7–10		Spray
Lima Bean, succulent seed.	USA In CO, MT, NM, WY and states eastward	SC 43.5%	5		0.19 ^c	7 ^f	0	Spray Application to lima bean must be made to Lima beans intended to harvest as succulent seed only
Ginseng	USA Not registered for use in CA.	SC 43.5%	5		0.19 ^c	7	14	Spray Aerial application is not permitted

Crop	Country	Form	Application per treatment			Interval (days)	PHI (days)	Notes
			Max no	Conc kg ai/hL	Rate kg ai/ha			
Artichoke	France	EC 72 g/L	3	0.018–0.09	0.18	14 ^g	3	Spray
Artichoke	Italy	EC 72g/l	3	0.14–0.09	0.14–0.18	7–10	3	Spray
Celery	Germany	90 g/kg WG	2	0.018	0.18	10–14		Spray
Celery	Trinidad and Tobago	WP 69	2–3	0.024–0.045	0.135–0.18	3–4 weeks	7	Spray
Leafy vegetable (except Brassica) includes celery	USA	SC 43.5%	3	0.12 per ground application 0.47 per aerial application	0.19 ^c	7 ^f	0	Spray
Leafy vegetable (except Brassica vegetables) includes celery	USA	SC 20.2%	3 ^d	0.12 per ground application 0.49 per aerial application	0.206 ^b	7 ^f	0	Spray

^a Maximum rate per season is 1.169 kg ai/ha

^b Maximum rate per season is 0.9535 kg ai/ha

^c Maximum rate per season is 1.14 kg ai/ha

^d Do not make more than two sequential applications before alternating to a labelled fungicide with different mode of action fungicide

^e Preventive, intervene at the beginning of risk period

^f Preventive application before infection occurs

^g Carry out two applications with COACH PLUS in the risk period which generally begins at the beginning of the strong leaf growth period

Residues resulting from supervised trials on crops

The Meeting received information on supervised field trials involving dimethomorph for the following crops and commodities:

Group	Crop commodity	Portion of commodity to which MRL apply	Countries	Table No
FC, Citrus fruits	Orange	Whole fruit, peel and pulp	Spain	7
	FC 0004, oranges sweet, sour			
FB, Berries and other Small Fruits	Grapes	Whole fruit	USA	8
	FB 0269			
FB, Berries and other Small Fruits	Strawberry,	Whole fruit	Belgium, Denmark, south and north France, Germany, the Netherlands, Spain and United Kingdom	9–13
	FB 0275			
FI, Assorted Tropical and Subtropical Fruit-inedible peel	Papaya	Whole fruit (peel and pulp)	Brazil	14
	FI 0350			

Group	Crop commodity	Portion of commodity to which MRL apply	Countries	Table No
VA, Bulb Vegetables	Onion, bulb	Bulbs	USA	15
VA, Bulb Vegetables	VA 0385 Leek	Leek stems	Belgium, Germany, Greece, France (north and south), Italy, the Netherlands, Spain and UK	16
VA, Bulb Vegetables	VA 0384 Spring onions	Whole plants	USA	17–18
VB, Brassica Vegetables, Head Cabbage, Flowerhead Brassicas	VA 0389 Head cabbage	Cabbage heads	USA, Canada	19
	VB 0041	(including wrappers leaves)		
VB, Brassica Vegetables, Head Cabbage, Flowerhead Brassicas	Broccoli	Flower heads and stem	USA, Canada	20
	VB 0040			
VO, Fruiting vegetables, other than Cucurbits	Pepper	Fruits	USA	21
	VO 0051 representing VO 0050 (fruiting vegetables other than cucurbits)			
VO, Fruiting vegetables, other than Cucurbits	Tomato	Mature fruits	USA	22*
VL, Leafy vegetables including Brassica Leafy vegetables	Lettuce Head	Fresh heads with wrappers leaves	USA	23–24
	VL 0482			
VL, Leafy vegetables including Brassica Leafy vegetables	Lettuce leaf	Leaves	USA	25
	VL 0483			
VL, Leafy vegetables including Brassica Leafy vegetables	Spinach	Leaves	USA	26
	VL 0502			
VL, Leafy vegetables including Brassica Leafy vegetables	Taro leaves	Leaf	USA	27
	VL 0505			
VP, Legume Vegetables	Green Peas	Whole commodity	France (north and south), Germany, Spain and the Netherlands, Denmark,	28–29
	Peas shelled (succulent			

Group	Crop commodity	Portion of commodity to which MRL apply	Countries	Table No
	seed)			
	VP 0064			
VP, Legume Vegetables	Lima Beans (young pods and/or immature beans)	Whole commodity	USA	30
	VP 0534			
VR, Root and tuber vegetable	Ginseng	Whole commodity after removing tops	USA	31
	VR 0604			
VS, Stalk and stem vegetables	Globe artichoke	Whole commodity	Germany, Italy, France (north and south), the Netherlands and Spain	32
	VS0620			
VS, Stalk and stem vegetables	Celery	Whole commodity	USA and Canada	33
	VS0624			
VR, Root and tuber vegetable	Taro, corms	Whole commodity after removing tops	USA	34
	VR 0505			

* Trials were evaluated by JMPR 2007. See Evaluation report for dimethomorph, Table 48

Conditions of the supervised residue trials were generally well reported in detailed field reports. In most trials treated plots were not replicated but where results were reported from replicate plots, these are presented as individual values. Field reports provided data on the sprayers used and their calibration, plot size, residue sample size and sampling date. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Residue data are recorded unadjusted for % recovery. When residues were not detected they are shown as below the LOQ (e.g. < 0.01 mg/kg). Laboratory reports included methods validation with procedural recoveries from spiking at residue levels similar to those occurring in samples from the supervised trials. Data on duration of residue sample under storage were also provided. Residues and application rates have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Where trials have involved two or more applications, the mean target application rate has been recorded unless the individual rates differ by more than 10%.

Residues values from trials conducted according to a maximum registered GAP with supporting trials have been used for the estimation of maximum residue levels. The results included in the evaluation of the MRL, STMR and HR is underlined.

Oranges

Eight outdoor trials performed during growing season 2004–2005 in Spain were presented to the 2007 and 2014 Meeting. Two broadcast spray applications of dimethomorph at 0.41 kg ai/ha were made at 15 day intervals 28 ± 1 and 14 ± 1 days before harvest.

Mature fruit were sampled and both the whole fruit and the pulp were analysed separately by LC-MS/MS, using Method PE 804 approved at this Meeting (see chapter analytical method). The limit of quantification was 0.01 mg/kg for and the mean recovery was 73–104.5.2% at fortification levels of 0.05–0.3 mg/kg for pulp, whole fruit and peel. The results of the residues from dimethomorph are summarized in Table 7.

Table 7 Residues in oranges from foliar application of dimethomorph from field trials in Spain

ORANGE Country, year (variety)	Form	Application				PHI (days)	Dimethomorph Residues mg/kg			Reference & Comments
		kg ai/ha ^a	kg ai/hL	no	Growth stage at last treatment BBCH		Pulp	Peel	Whole fruit	
Spain 2004 (Valencia Late)	DC 150 g/L	0.41	0.014	2	85–89	0			0.152	2005/1034175 04/S/05
						7	< 0.01	0.219	0.066	
						14	< 0.01	0.167	0.042	
						21	< 0.01	0.105	0.037	
						28	–	–	0.019	
Spain 2004 (Valencia Late)	DC 150 g/L	0.41	0.014	2	85–89	0			0.060	2005/1034175 04/S/06
						7	< 0.01	0.130	0.059	
						14	< 0.01	0.075	0.035	
						21	–	0.027	0.016	
						28	–	–	< 0.01	
Spain 2005 (Clemenules)	DC 150 g/L	0.41	0.014	2	81	7	< 0.01	0.138	0.091	2006/1015076 05/S/41
						14	< 0.01	0.144	0.047	
Spain 2005 (Clemenules)	DC 150 g/L	0.41	0.014	2	81	7	< 0.01	0.265	0.237	2006/1015076 05/S/42
						14	< 0.01	0.199	0.144	
Spain 2005 (Salustiana)	DC 150 g/L	0.41	0.014	2	83	0		–	0.368	2006/1015077 (05/S/01)
						7	< 0.01	0.868	0.170	
						14	< 0.01	0.548	0.227	
						21	< 0.01	0.520	0.111	
						28	–	–	0.084	
Spain 2005 (Salustiana)	DC 150 g/L	0.41	0.014	2	83	0			0.607	2006/1015077 (05/S/02)
						7	< 0.01	1.243	0.388	
						14	< 0.01	1.216	0.604	
						21	< 0.01	0.644	0.330	
						28			0.178	
Spain 2005 (Lane Late)	DC 150 g/L	0.41	0.014	2	83	0			0.310	2006/1015077 (05/S/03)
						7	< 0.01	1.349	0.187	
						14	< 0.01	0.931	0.173	
						21	< 0.01	0.959	0.204	
						28			0.082	
Spain 2004 (Valencia Late)	DC 150 g/L	0.41	0.014	2	83	0			0.147	2006/1015077 (05/S/04)
						7	< 0.01	0.617	0.175	
						14	< 0.01	0.440	0.069	
						21	< 0.01	0.451	0.075	
						28			0.040	

^a Spray volumes of 3000 L/ha

Grapes

During growing season 2008 eight outdoor trials on grapes were conducted in the USA. Dimethomorph was applied four times as a foliar spray using a 6–8 days interval. The applications of the concentrate (0.04–0.06 kg ai/hL) and the dilute (0.009–0.02 kg ai/hL) were made to side by side plots. The timing of the applications was from BBCH 7 (developing of fruits) until BBCH 8 (ripening of fruits).

The samplings of fresh grape fruits were analysed using HPLC-MS/MS Method 575/0. The limit of quantification was 0.01 mg/kg and the mean recovery was 71–115% at fortification levels of 0.01–5.0 mg/kg. The results are summarized in Table 8.

Table 8 Residues in grapes from foliar application of dimethomorph in field trials in the USA

GRAPES Country, year (variety)	Form	Application				PHI (days)	Dimethomorph Residues mg/kg Fruits		Reference & Comments
		kg ai/ha	kg ai/hL	no	Growth stage at last treatment		Conc.	Dilute	
Critical GAP USA		0.219		4		14			
USA (NY) 2008 (Cayuga White)	SC 500 g	0.225	0.05 conc. ^a 0.03 dilute ^b	4	BBCH 81	14	0.3	0.41	2009/7002489 R080044
					–	28	0.23	0.19	
USA (PA) 2008 (Concord)	SC 500 g	0.225	0.05 conc. ^a 0.009 dilute ^b	4	BBCH 85	14	0.11	0.46	2009/7002489
					–	28	0.10	0.11	
USA (WI) 2008 (King of the North)	SC 500 g	0.225	0.06 conc. ^a 0.03 dilute ^b	4	BBCH 85	14	1.77	1.28	2009/7002489 R080046
					–	28	1.53	1.22	
USA (MI) 2008 (Concord)	SC 500 g	0.225	0.05 conc. ^a 0.02 dilute ^b	4	Berries touching	14	0.39	0.49	2009/7002489 R080047
					–	28	0.48	0.34	
CA (ON) 2008 (Concord)	SC 500 g	0.225	0.05 conc. ^a 0.03 dilute ^b	4	BBCH 83	14	0.05	0.11	2009/7002489 R080048
					–	28	0.05	0.11	
USA (CA) 2008 (Ruby red)	SC 500 g	0.225	0.05 conc. ^a 0.03 dilute ^b	4	Berry colour adv.	14	0.26	0.15	2009/7002489 R080049
					–	28	0.16	0.08	
USA (CA) Fresno 2008 (Thompson Seedless)	SC 500 g	0.225	0.04 conc. ^a 0.01 dilute ^b	4	Fruits 1.3 cm	14	0.76	1.86	2009/7002489 R080050
					–	28	0.53	0.90	
USA (CA) Fresno 2008 (Thompson Seedless) USA (CA) Fresno 2008 (Fiesta)	SC 500 g	0.225	0.04 conc. ^a 0.01 dilute ^b	4	Fruits 5 cm	0	2.44	1.68	2009/7002489 R080051
					–	7	1.43	1.88	
					–	14	1.83	1.51	
					–	28	1.16	0.89	
					–	35	0.76	1.57	
USA (CA) Madera 2008 (Thompson Seedless)	SC 500 g	0.225	0.04 conc. ^a 0.02 dilute ^b	4	Near mature	14	0.40	0.75	2009/7002489 R080052
					–	28	0.29	0.66	
USA (CA) Tulare 2008 (Red Globe)	SC 500 g	0.225	0.04 conc. ^a 0.02 dilute ^b	4	BBCH 88	14	0.51	0.71	2009/7002489 R080053
					–	28	0.33	0.55	
USA (WA) 2008 (White Riesling)	SC 500 g	0.225	0.05 conc. ^a 0.02 dilute ^b	4	BBCH 82	14	0.56	0.92	2009/7002489 R080054
					–	28	0.59	0.61	
USA (OR)	SC	0.225	0.06	4	BBCH 85	14	1.33	0.33	2009/7002489

GRAPES Country, year (variety)	Form	Application				PHI (days)	Dimethomorph Residues mg/kg Fruits		Reference & Comments
		kg ai/ha	kg ai/hL	no	Growth stage at last treatment		Conc.	Dilute	
2008 (Pinot Noir)	500 g		conc. ^a 0.02 dilute ^b		–	28	0.55	0.46	R080055
USA (CA) Kern 2008 (Crimson)	SC 500 g	0.225	0.04 conc. ^a 0.02 dilute ^b	4	BBCH 88 –	14 28	0.14 0.18	0.65 0.56	2009/7002489 R080547

^a Spray volumes of 458–713 L/ha

^b Spray volumes of 936–3663 L/ha

Strawberries

Twenty one additional (protected and outdoor) trials where dimethomorph was applied as root drench or drip irrigation and four additional (outdoor) trials where dimethomorph was applied as a foliar spray, were presented.

Root drench /drip application

In eight protected trials performed 2004 and 2007 on strawberries in three cultivars in Europe (Belgium, the Netherlands, Germany, Denmark, and northern France) dimethomorph was applied once as a root drench (green house) or (plastic tunnel) at planting/transplanting. The trials from 2004 included in addition to the treatment 0.125 g ai/plant, the treatment 0.0625 g ai/plant. The PHI was 34–46 days. The results are summarized in Table 9.

In two greenhouse trials on strawberries in Spain and south of France during growing season 2007 dimethomorph was added twice with a 14 days interval to the drip irrigation system in an amount of 0.75 kg ai/ha. The residue decline was measured 0, 1, 3 ± 1 and 7 ± 1 days after treatment. The results are summarized in Table 10.

In three outdoor trials on strawberries in Europe 2004 (Belgium, Germany and the Netherlands) dimethomorph was applied once as a root drench at planting/transplanting. The trials from 2004 included in addition to the treatment 0.125 g ai/plant, the treatment 0.0625 g ai/plant. The PHI was 43 ± day. The results are summarized in Table 11.

In eight outdoor strawberry trials in Spain dimethomorph) was added twice to the drip irrigation system in an amount of 0.75 kg ai/ha 15 ± 1 day before harvest. Duplicate samples of strawberry fruits were sampled 0, 3, 7, 21 and 28 days after last application. PHI was one day. The results are summarized in Table 12.

Foliar spray application

In four independent outdoor strawberry trials (four cultivars) from northern Europe (Belgium, Germany, the Netherlands and United Kingdom) dimethomorph was applied once as a foliar spray at 1.5 kg ai/ha, the PHI was 35 days. The results are summarized in Table 13.

Analytical methodology

Mature fruit were sampled. In the trials reported from 2004 the strawberry fruits were analysed using GC-NDP and an extended version of the DFG S19 method reviewed by JMPR 2007. The mean recovery rate for the revised method was 90.5–111.5% at fortification levels of 0.01–1 mg/kg. The limit of quantification was 0.01 mg/kg.

In outdoor drip irrigations trials from Spain the analytical was LC-MS method PE 804 with a limit of quantification of 0.01 mg/kg, and the mean recovery rates were 89–99.2% at fortification levels of 0.015–0.3 mg/kg. The analytical method 575/0 using HPLC-MS/MS were used in the trials reported from 2008 and onwards. The limit of quantification for this method was 0.01 mg/kg and the mean recovery rates were 92–106% at fortification levels of 0.01–1 mg/kg.

Table 9 Residues in strawberries from applications of dimethomorph as root drench in protected trials in northern Europe (Belgium, the Netherlands, Germany, Northern France and Denmark)

STRAWBERRY Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues mg/kg Fruits	Reference & Comments
		g ai/plant ^a	no	Growth stage at treatment (BBCH)			
Critical GAP Ireland		0.05	1		35		
Belgium 2004 (Elsanta)	WP 500 g	0.125	1	19	34	0.511	2005/1004964/ 2010/1051738 AGR/21/04
Belgium 2004 (Elsanta)	WP 500 g	0.0625	1	19	34	0.304	
The Netherlands 2004 (Elsanta)	WP 500 g	0.125	1	19	34	0.289	2005/1004964/ 2010/1051738 AGR/22/04
The Netherlands 2004 (Elsanta)	WP 500 g	0.0625	1	19	34	0.257	
Germany 2004 (Elsanta)	WP 500 g	0.125	1	10	43 50	0.047 0.032	2005/1004964/ 2010/1051738 ACK/12/04
Germany 2004 (Elsanta)	WP 500 g	0.0625	1	10	43 50	0.030 0.028	
Denmark 2004 (Everst)	WP 500 g	0.125	1	12	42 80	0.228 0.055	2005/1004964/ 2010/1051738 ALB/14/04
Denmark 2004 (Everst)	WP 500 g	0.0625	1	12	42 80	0.181 0.030	
Northern France 2007 (Matis)	WP 500 g	0.125	1	11	36 46	0.18 0.21	2008/1051524 A7047 OB1
Germany 2007 (Elsanta)	WP 500 g	0.125	1	at planting	35 44	0.05 0.03	2008/1051524 A7047 GE1
The Netherlands 2007 (Elsanta)	WP 500 g	0.125	1	at planting	34 43	0.04 0.03	2008/1051524 A7047 NL1
Belgium 2007 (Elsanta)	WP 500 g	0.125	1	at planting	34 45	0.04 0.02	2008/1051524 A7047 BE1

^a 250 mL/plant

Table 10 Residues in strawberries from applications of dimethomorph as drip irrigation in protected in southern Europe, (Spain and southern France).

STRAWBERRY Country, year (variety)	Form	Application			PHI (day s)	Dimethomorph Residues mg/kg Fruits	Reference & Comments
		kg ai/ha ^a	no	Growth stage at last treatment (BBCH)			
Spain 2007 (Camarosa)	DC 150 g	0.75	2	87–89	0 1 4 7	0.06 0.08 0.08 0.09	2008/1005528 L070724
South France	DC	0.75	2	85–89	0	0.08	

STRAWBERRY Country, year	Form	Application			PHI (day)	Dimethomorph	Reference & Comments
2007 (Clery)	150 g				1 4 7	< 0.01 < 0.01 < 0.01	L070725

^a With 2500 L water/ha

Table 11 Residues in strawberries from applications of dimethomorph as a root drench in outdoor trials in northern Europe (Belgium, the Netherlands and Germany).

STRAWBERRY Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues mg/kg Fruits	Reference & Comments
		g ai/plant ^a	no	Growth stage at treatment (BBCH)			
Critical GAP Ireland		0.05	1		35		
Belgium 2004 (Elsanta)	WP 500 g	0.125	1	15	43 57	0.04 0.03	2005/1004964 AGR/19/04
Belgium 2004 (Elsanta)	WP 500 g	0.0625	1	15	43 57	0.02 0.02	
The Netherlands 2004 (Elsanta)	WP 500 g	0.125	1	15	43 61	0.01 < 0.01	2005/1004964 AGR/20/04
The Netherlands 2004 (Elsanta)	WP 500 g	0.0625	1	15	43 61	< 0.01 < 0.01	
Germany 2004 (Elsanta)	WP 500 g	0.125	1	10	44 56	0.02 0.02	2005/1004964 ACK/11/04
Germany 2004 (Elsanta)	WP 500 g	0.0625	1	10	44 56	0.01 0.02	

^a 250 mL/plant

Table 12 Residues in strawberries from applications of dimethomorph, as drip irrigation in outdoor trials in southern Europe (Spain)

STRAWBERRY Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues mg/kg Fruits		Reference & Comments
		kg ai/ha ^a	no	Growth stage (BBCH)		ind, value	average	
Spain 2004 (Camerosa)	DC 150	0.75	2	87–89	1	0.21	0.022	2006/1015075 04/S/01
					3	0.022	0.033	
					7	0.033	0.062	
					21	0.063	0.051	
					28	0.060	0.036	
						0.052		
Spain 2004 (Camerosa)	DC 150	0.75	2	87–89	1	0.049		2006/1015075 04/S/02
					3	0.035		
					7	0.037		
					21	< 0.01	< 0.01	
					28	< 0.01	< 0.01	
						0.022	0.023	
						0.023	0.016	
						0.016		
						0.015		
						< 0.01	< 0.01	

STRAWBERRY Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues mg/kg Fruits		Reference & Comments
		kg ai/ha ^a	no	Growth stage (BBCH)		ind, value	average	
Spain 2004 (Camerosa)	DC 150	0.75	2	87–89	1 3 7 21 28	< 0.01 0.01 < 0.01 0.019 0.019 0.063 0.063 0.034 0.034	< 0.01 0.01 0.019 0.063 0.034	2006/1015075 04/S/03
Spain 2004 (Camerosa)	DC 150	0.75	2	87–89	1 3 7 21 28	0.129 0.125 0.057 0.058 0.180 0.184 0.043 0.043 0.031 0.031	0.127 0.058 0.182 0.043 0.031	2006/1015075 04/S/04
Spain 2005 (Camerosa)	DC 150	0.75	2	89	1 3 7 22 28	0.032 0.033 0.124 0.122 0.099 0.096 0.028 0.028 0.052 0.052	0.033 0.123 0.098 0.028 0.052	2006/1015079 05/S/05
Spain 2005 (Camerosa)	DC 150	0.75	2	89	1 3 7 22 28	0.026 0.025 0.064 0.066 0.048 0.048 0.026 0.026 0.023 0.023	0.026 0.065 0.048 0.026 0.023	2006/1015079 05/S/06
Spain 2005 (Camerosa)	DC 150	0.75	2	89	1 2 6 21 28	0.014 0.015 0.030 0.030 0.025 0.025 0.029 0.028 0.023 0.023	0.015 0.030 0.025 0.029 0.023	2006/1015079 05/S/07
Spain 2005 (Camerosa)	DC 150	0.75	2	89	1 2 6 21	0.055 0.055 0.111 0.111 0.117 0.116 0.069 0.069	0.055 0.111 0.117 0.069	2006/1015079 05/S/08

STRAWBERRY Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues mg/kg Fruits		Reference & Comments
		kg ai/ha ^a	no	Growth stage (BBCH)		ind, value	average	
					28	0.042 0.042	0.042	

^a With 4444 L water/ha

Table 13 Residues in strawberries from foliar applications of dimethomorph in outdoor trials in northern Europe (United Kingdom, the Netherlands, Germany and Belgium)

STRAWBERRY Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues mg/kg Fruits	Reference & Comments
		kg ai/ha ^a	no	Growth stage at treatment (BBCH)			
United Kingdom 2009 (Elsanta)	WP 500 g	1.478	1	65–71	0 28 35 42	131 ^b < 0.01 0.018 0.013	2009/1112576 LO90258
The Netherlands 2009 (Mara des Bois)	WP 500 g	1.59	1	61	0 29 36 43	115 ^b < 0.01 < 0.01 < 0.01	2009/1112576 LO90259
Germany 2009 (Dorselect)	WP 500 g	1.65	1	61	0 28 35 42	103 ^b 0.014 0.028 0.013	2009/1112576 LO90260 ^c
Belgium 2009 (Charlotte)	WP 500 g	1.598	1	65–81	0 28 35 42	105 ^b 0.061 0.012 0.012	2009/1112576 LO90261

^a With 197–220 L water/ha

^b Leaves

^c Residues taken from specimen labelled as untreated. The corresponding treated specimen had residue levels < 0.01 mg/kg. It is assumed that there has been a mistake at the labelling either in the field or in the analytical phase.

Papaya

Four field trials were conducted 2011 in Brazil. Dimethomorph was applied four times as a foliar spray from the beginning of ripening until full maturity of the fruits with a 6–8 days interval.

Samples of mature whole fruit (peel and pulp) was analysed with LC/MS/MS/ using Method 535/1, The limit of quantification was 0.01 mg/kg and the mean recovery was 95–92% at fortification levels of 0.01–1.0 mg/kg. The results are summarized in Table 14 below.

Table 14 Residues in papaya from foliar application of dimethomorph in field trials in Brazil

PAPAYA Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues (mg/kg)	Reference & Comments
		kg ai/ha ^a	no	Growth stage at last treatment			
Brazil/SP 2011 (Formosa)	WP 500 g	0.5 0.5 0.5 0.5	4	BBCH 81–83	0 3 7 14	0.39 0.38 0.19 0.07	2012/3001445 G100547
Brazil/SP 2011	WP	0.5	4	BBCH	0	0.40	2012/3001445

PAPAYA Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues (mg/kg)	Reference & Comments
		kg ai/ha ^a	no	Growth stage at last treatment			
(Formosa)	500 g	0.5 0.5 0.5		81–78 ^b	3 7 14	0.18 0.26 0.03	G100548
Brazil/ES 2011 (Goldhem)	WP 500 g	0.5	4	BBCH 81–78 ^b	7	0.44	2012/3001445 G100549
Brazil/ES 2011 (THB)	WP 500 g	0.5	4	BBCH 83–78 ^b	7	0.67	2012/3001445 G100550

^a with 1000 L water/ha^b During conduction of this trial ripe fruit were discarded, therefore a decrease in BBCH is observed.

Bulb onions

Ten residue decline trials were conducted in the USA and Canada during growing season 2008. Three broadcast spray applications of dimethomorph were done at a 4–8 days interval. The growth stage for the applications varied from BBCH 19 (vegetative) until BBCH 81 (mature bulbs). Duplicate samples of onion bulb were analysed using HPLC-MS/MS Method 575/0. The limit of quantification was 0.01 mg/kg and the mean recovery was $85.9 \pm 11.8\%$ at fortification levels of 0.01–1.0 mg/kg. The results are summarized in Table 15.

Eight residue trials were performed in the USA during growing season 1999. Seven foliar directed applications were done with a 6–8 days interval. The growth stage of the applications varied from vegetative to mature bulbs. Duplicated samples were analysed using HPLV/UV Method FAMS 002/004 approved grapes at the 2007 Meeting. The limit of quantification was 0.01 mg/kg and the recovery was 70–120% at fortification levels of 0.01–0.1 mg/kg. The results are summarized in Table 15.

Table 15 Residues in onion bulbs from foliar application of dimethomorph in field trials in the USA

Head Cabbage Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues in bulbs		Reference & Comments
		kg ai/ha	no	Growth stage at last treatment		ind. value	mean	
Critical GAP USA, Canada		0.21–0.23	3		0			
USA (NY) 2008 (Super Star Onion)	SC 500 g	0.227 ^a	3	2.5 bulbs	0 1 3 7 10	0.10 0.10 0.08 0.08 0.09 0.09 0.09 0.12 0.16 0.13	0.10 0.08 0.09 0.11 0.13	2009/7004732 R080022
CAN (MN) 2008 (Candy)	SC 500 g	0.226 ^a	3	vegetative	0 1 3 7 10	0.09 0.07 0.06 0.04 0.03 0.03 0.02 0.02 0.01 0.01	0.08 0.05 0.03 0.02 0.01	2009/7004732 R080023
CAN (KS)	SC	0.226 ^a	3	BBCH 48	0	0.16	0.16	2009/7004732

Head Cabbage Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues in bulbs		Reference & Comments
2008 (Candy)	500 g				1 3 7 10	0.15 0.07 0.06 0.08 0.04 0.04 0.05 0.05 0.06	0.07 0.06 0.05 0.06	R080024
CAN (MB) 2008 (Genisis Hybrid)	SC 500 g	0.220 ^a	3	BBCH 47	0 1 3 7 10	0.37 0.40 0.31 0.28 0.21 0.21 0.06 0.05 0.13 0.12	0.38 0.3 0.21 0.06 0.13	2009/7004732 R080025
USA (CA) 2008 (White)	SC 500 g	0.221 ^a	3	Mature	0 1 3 7 10	0.04 0.04 0.06 0.03 0.10 0.11 0.21 0.15 0.02 0.03	0.04 0.05 0.02 0.18 0.03	2009/7004732 R080027
USA (TX) 2008 (Yellow Spanish)	SC 500 g	0.23 ^a	3	Tops falling	0 1 3 7 10	0.16 0.20 0.03 0.11 0.04 0.08 0.02 0.03 0.01 0.02	0.18 0.07 0.06 0.03 0.02	2009/7004732 R080028
USA (CA) 2008 (Southport)	SC 500 g	0.226 ^a	3	2.5 th bulbs	0 1 3 7 10	0.25 0.26 0.04 0.22 0.29 0.26 0.17 0.14 0.14 0.19	0.26 0.13 0.28 0.155 0.165	2009/7004732 R080029
USA (ID) 2008 (Vaquero)	SC 500 g	0.223 ^a	3	growth complete	0 1 3 7 10	0.05 0.06 0.04 0.05 0.03 0.03 0.02 0.03 < 0.01 0.01	0.06 0.045 0.03 0.025 0.01	2009/7004732 R080030
USA (OR) 2008 (White Sweet Spanish)	SC 500 g	0.230 ^a	3	BBCH 49	0 1	0.05 0.05 0.04 0.03	0.05 0.04	2009/7004732 R080684

Head Cabbage Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues in bulbs		Reference & Comments
					3	0.05 0.19	0.12	
					7	0.07 0.05	0.06	
					10	0.07 0.04	0.055	
USA (TX) 2008 (Yellow Granex)	SC 500 g	0.23 ^a	3	BBCH 47	0	0.24 0.21	0.23	2009/7004732 R080690
					1	0.21 0.20	0.205	
					3	0.19 0.19	0.19	
					7	0.13 0.11	0.12	
					10	0.09 0.09	0.09	
USA (CA) 1999 (Early red burger)	WP 500 g	0.225 ^b	7	Mature bulbs	0	0.10 0.22	0.16	2002/7013745 CA 06
USA (CA) 1999 (Reina Blanca)	WP 500 g	0.225 ^b	7	Bulbs formed	0	0.08 0.10	0.09	2002/7013745 CA 07
USA (CO) 1999 (Teton)	WP 500 g	0.225 ^b	7	Mature bulbs	0	0.10 0.12	0.11	2002/7013745 CO 01
USA (ID) 1999 (Vega)	WP 500 g	0.225 ^b	7	Mature bulbs	0	0.03 0.06	0.04	2002/7013745 ID 01
USA (NY) 1999 (Hustler)	WP 500 g	0.225	7	Mature bulbs	0	0.07 0.13	0.10	2002/7013745 NY 02
USA (NY) 1999 (Burgos)	WP 500 g	0.225 ^b	7	Vegetative ^c	0	1.411– 0.220	0.82	2002/7013745 OH 06
USA (OR) 1999 (Santos FI)	WP 500 g	0.225 ^b	7	Mature bulbs	0	0.27 0.28	0.27	2002/7013745 OR 10
USA (TX) 1999 (1015)	WP 500 g	0.225 ^b	7	Mature bulbs	0	0.07 0.08	0.08	2002/7013745 TX02
					3	0.04 0.05	0.05	
					7	0.05 0.06	0.06	

^a With 274–312 L water/ha^b With 284–730 L water/ha^c Crop height 12–15"

Leek

Nineteen residue decline trials were conducted in Europe during growing seasons 1999 and 2006. Dimethomorph was applied three times as a foliar spray. The applications were made 34–14 ± 1 days before harvest with a spray interval of 10–11 days.

Samples of leek stems were analysed using LC/MS/MS Method 575/0. The limit of quantification was 0.01–0.02 mg/kg and the recovery was 74–103%. The results are summarized in Table 16.

Table 16 Residues of dimethomorph in leek from field trials conducted in Europe

LEEK Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues (mg/kg) stem	Reference & Comments
		kg ai/ha ^a	no	Growth stage at last treatment		ind. value	
Critical GAP France		0.18	2		14		
Germany, 1999 (Lancelot)	WG 9 g	0.18	3	12 leaves	0 7 14 21 28	0.67 0.24 0.08 0.08 0.03	2001/1026153 FR22/06/40
Germany, 2001 (Hiberna)	WG 9 g	0.18	3	2.5–3.0 cm	0 7 14 21 28	0.62 0.44 0.07 0.06 0.03	2002/1019891 01/047
Germany, 2001 (Hiberna)	WG 9 g	0.18	3	mature	14 21	0.05 0.04	2002/1019892 01/048
Germany, 2001 (Florina)	WG 9 g	0.18	3	2 weeks before harvest	0 7 14 21 28	1.6 0.05 < 0.02 < 0.02 < 0.02	2002/1019893 01/090
Germany, 2001 (Amundo)	WG 9 g	0.18	3	43	14 21	< 0.02 < 0.02	2002/1019893 01/091
Germany, 2001 (Werdea)	WG 9 g	0.18	3	2 weeks before harvest.	14 21	0.03 0.02	2002/1019893 01/092
Germany, 2001 (Werdea)	WG 9 g	0.18	3	2 weeks before harvest	14 21	0.04 0.05	2002/1019893 01/093
Northern France, 2005 (Elektra)	WG 9 g	0.18	3	45	0 7 14 21	2.23 0.20 0.10 0.06	2007/1008491 A/NF/F/05/222
Southern France 2005 (Amundo)	WG 9 g	0.18	3	47	0 7 14 21	0.56 1.02 0.69 0.18	2007/1008491 A/SF/F/05/223
Germany, 2005 (Appolo)	WG 9 g	0.18	3	45–49	0 7 15 22	1.42 0.16 0.08 0.07	2007/1008491 A/NF/F/05/224
Belgium, 2005 (Sheltar)	WG 9 g	0.18	3	47–49	0 7 14 21	0.29 0.22 0.04 0.03	2007/1008491 A/NF/F/05/225
The Netherlands, 2005 (Kenton)	WG 9 g	0.18	3	44	0 7 14 21	0.34 0.21 0.11 0.03	2007/1008491 A/NF/F/05/226
Italy, 2005 (Kenton F1)	WG 9 g	0.18	3	46–47	0 7 14 21	0.39 0.54 0.21 0.30	2007/1008491 A/IT/F/05/227
N. France 2006 (Axima)	WG 9	0.18	3	43	0 7 14 21	0.81 0.15 0.10 0.05	2007/1008500 A/NF/F/06/81

LEEK Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues (mg/kg) stem		Reference & Comments
		kg ai/ha ^a	no	Growth stage at last treatment		ind. value		
The Netherlands, 2006 (Kenton)	WG 9 g	0.18	3	47–48	0 7 14 21	0.33 0.08 0.04 0.02		2007/1008500 A/NF/F/06/82
Denmark, 2006 (Pandora)	WG 9 g	0.18	3	49	0 7 14 21	0.46 0.05 < 0.01 0.01		2007/1008500 A/DK/F/06/83
UK, 2006 (Pancho)	WG 9 g	0.18	3	44	0 6 13 21	0.84 0.17 0.13 0.02		2007/1008500 A/UK/F/06/84
Spain, 2006 (Atal)	WG 9 g	0.18	3	46–47	0 6 13 20	0.27 0.39 0.06 0.02		2007/1008500 A/SP/F/06/85
Greece, 2006 (Demylemy)	WG 9 g	0.18	3	43–46	0 7 14 21	0.88 0.11 0.08 0.05		2007/1008500 A/GR/F/06/86

^a Spray volumes of 372–600 L/ha

Spring onions

During growing season 2011 three outdoor trials were conducted in the USA. Three broadcast spray applications of dimethomorph were made at a 5–6 days interval. The growth stage for the applications varied within BBCH 41–49 (development of harvestable vegetative plant parts).

Three outdoor residue decline trials were conducted in USA 2008. Three broad cast spray applications of dimethomorph were made at a 4–8 days interval. The growth stage for the applications varied from 12 days before first sampling until growth complete.

Duplicate samples from treated plots were analysed with LC/MS/MS using Method 575/0. The limit of quantification was 0.01 mg/kg and the mean recovery was 76–114% and 74–93% for the trials from 2011 and 2008 respectively at fortification levels of 0.01–10.0 mg/kg. The results are summarized in Table 17.

Table 17 Residues of dimethomorph in spring onions from field trials conducted in USA

SPRING ONION Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues in whole plants		Reference & Comments
		kg ai/ha	no	Growth stage at last treatment		ind. value	mean	
Critical GAP USA, Canada		0.21–0.23	3		0			
USA (OK) 2011 (Walla, Walla)	SC 225 g	0.25 ^a	3	BBCH 43	0 0	1.92 2.78	2.35	2013-7001787 R110160
					1 1	1.97 2.21	2.09	
USA (TX) 2011 (Local sort not specified)	SC 225 g	0.25 ^a	3	BBCH 41	0 0	1.78 3.12	2.45	2013-7001787 R110161
					1 1	2.07 1.99	2.03	

SPRING ONION Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues in whole plants		Reference & Comments
		kg ai/ha	no	Growth stage at last treatment		ind. value	mean	
USA (CA) 2011 (Evergreen White)	SC 225 g	0.25 ^a	3	BBCH 49	0	1.26	1.27	2013-7001787 R110162
					0	1.28		
					1	0.88	0.91	
					1	0.93		
USA (CA) 2008 (APT 410)	SC 500 g	0.225 ^b	3	Growth complete	0	1.34	1.56	2009/7004732 R080033
					0	1.78		
					1	1.36	1.42	
					1	1.48		
					3	0.78	0.72	
					3	0.66		
					7	0.28	0.22	
					7	0.15		
USA (CA) 2008 (Southport White)	SC 500 g	0.225 ^b	3	Mature	0	1.79	1.79	2009/7004732 R080034
					0	1.79		
					1	1.73	1.76	
					1	1.78		
					3	1.46	1.64	
					3	1.81		
					7	0.79	0.92	
					7	1.04		
USA (TX) 2008 (Yellow Granex)	SC 500 g	0.225 ^b	3	BBCH 15	0	6.60	5.36	2009/7004732 R080691
					0	4.11		
					1	3.19	3.24	
					1	3.28		
					3	2.49	2.64	
					3	2.78		
					7	0.99	0.92	
					7	0.85		
					10	0.75	0.72	
					10	0.69		

^a With 187–337 L water/ha^b With 278–291 L water/ha

During growing season 1999 four trials were performed on spring onions in the USA. Seven to eight foliar directed spray applications of dimethomorph formulation were made at 5–8 days interval. The growth stage for applications varied within growth stage development of harvestable vegetative plant parts (BBCH 41–49) from vegetative until vegetative (mature).

Duplicate samples (whole vegetable) from treated plots were sampled on the day of seventh application. The samples were analysed with LC/MS/MD using Method 575/0. The limit of quantification was 0.05 mg/kg and the mean recovery was 92–114% at fortification levels of 0.05–5.0 mg/kg. The results are summarized in Table 18.

Table 18 Residues in spring onions from foliar application of dimethomorph in field trials in the USA

SPRING ONION Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues in whole plants		Reference & Comments
		kg ai/ha	no	Growth stage at last treatment		ind. value	mean	
Critical GAP USA		0.21	3		0			
USA (NJ) 1999 (Bunchine)	WP 50	0.225	7	Vegetative mature	0 0	0.641 0.653	0.647	2002/7013746 NJ 05
USA (FL) 1999 (White Spear)	WP 50	0.225	7	Vegetative mature	0 0	0.721 0.776	0.749	2002/7013746 FL 18
USA (WI) 1999 (Evergreen)	WP 50	0.225	7	vegetative	0 0	0.887 0.945	0.915	2002/7013746 WI 24
USA (OR) 1999 (Feast)	WP 50	0.225	7	Mature	0 0	1.21 1.22	1.22	2002/7013746 OR09
			8		0 0	0.862 0.899	0.881	

^a With 265–474 L water/ha

Head cabbage

Ten field trials were conducted in the USA (nine) and Canada (one) during growing season 2008. Three foliar broadcast applications of dimethomorph were made with a 5–9 days interval. The growth stage for the applications varied from vegetative stage to mature heads.

Duplicate samples of cabbage heads (including wrapper leaves) were analysed with LC-MS/MS using Method 575/0. The limit of quantification was 0.01 mg/kg and the mean recovery was 108% at fortification levels of 0.01–10.0 mg/kg. The results are summarized in Table 19.

Table 19 Residues in head cabbage (with wrapper) from foliar application of dimethomorph in field trials from the USA

Head Cabbage Country, year (variety)	Form	Application				PHI (days)	Dimethomorph Residues Head cabbage with wrapper leaves (mg/kg)		Reference & Comments
		kg ai/ha ^a	kg ai/hL	no	Growth stage at last treatment		ind. value	mean	
Critical GAP USA		0.21		3		0			
USA (NY) 2008 (Rocket)	SC 500 g	0.225	0.106	3	mature heads	0	0.74 0.97	0.86	2009/7006205 RCN R080068
						1	0.86 0.85	0.86	
						3	0.21 0.28	0.25	
						7	0.11 0.08	0.095	
						10	0.07 0.06	0.07	
USA (GA)	SC	0.225	0.107	3	4–8"	0	1.15	1.08	2009/7006205

Head Cabbage Country, year (variety)	Form	Application				PHI (days)	Dimethomorph Residues Head cabbage with wrapper leaves (mg/kg)		Reference & Comments
2008 (Thunder)	500 g				heads	1 3 7 10	1.01 0.97 0.95 0.45 0.51 0.12 0.09 0.09 0.08	0.96 0.48 0.11 0.09	RCN R080069
USA (FL) 2008 (White)	SC 500 g	0.225	0.105	3	mature heads	0 1 3 7 10	1.24 1.49 1.28 0.30 0.27 0.18 0.23 0.14 0.19	1.37 1.28 0.29 0.21 0.17	2009/7006205 RCN R080070
USA (MN) 2008 (Market Pride)	SC 500 g	0.225	0.107	3	vegetativ e	0 1 3 7 10	0.14 0.13 0.14 0.13 0.15 0.19 0.03 0.05 0.03 0.04	0.14 0.14 0.17 0.04 0.04	2009/7006205 RCN R080071
Canada (QC) 2008 (Stonehead)	SC 500 g	0.225	0.105	3	BBCH 49	0 1 3 7 10	1.54 1.17 1.51 1.51 0.25 0.24 0.22 0.22 0.08 0.13	1.36 1.51 0.25 0.22 0.11	2009/7006205 RCN R080072
USA (WI) 2008 (Artost)	SC 500 g	0.225	0.106	3	BBCH 48	0 1 3 7 10	0.68 1.22 0.90 1.25 0.07 0.08 0.80 0.21 0.13 0.10	0.95 1.08 0.08 0.51 0.12	2009/7006205 RCN R080073
USA (TX) 2008 (Pennant)	SC 500 g	0.225	0.107	3	BBCH 49	0 1 3 7 10	0.91 0.66 1.28 1.15 0.82 1.14 0.20 0.17 0.97 0.27	0.79 1.22 0.51 0.19 0.62	2009/7006205 RCN R080074
USA (KS) 2008 (Stonehead)	SC 500 g	0.225	0.106	3	mature heads	0	3.90 4.61	4.26	2009/7006205 RCN R080075

Head Cabbage Country, year (variety)	Form	Application				PHI (days)	Dimethomorph Residues Head cabbage with wrapper leaves (mg/kg)		Reference & Comments
						1	4.09 4.21	4.15	
						3	3.23 3.65	3.44	
						7	1.27 1.21	1.24	
						10	0.78 0.62	0.07	
USA (CA) 2008 (Copenhagen)	SC 500 g	0.225	0.110	3	mature heads	0	0.45 0.42	0.44	2009/7006205 RCN R080076
						1	0.48 0.43	0.46	
						3	0.23 0.16	0.19	
						7	0.19 0.16	0.18	
						10	0.09 0.11	0.10	
USA (OR) 2008 (Primo)	SC 500 g	0.225	0.106	3	BBCH 49	0	0.52 0.38	0.45	2009/7006205 RCN R080077
						1	0.23 0.29	0.26	
						3	0.26 0.22	0.24	
						7	0.04 0.05	0.05	
						10	0.03 0.03	0.03	

^a With 267–295 L water/ha

Broccoli

Ten field trials were conducted in the USA (nine) and Canada (one) during growing season 2008. Three foliar broadcast applications of dimethomorph were applied with a spray interval of 5–9 days. The growth stage for the applications varied from vegetative stage to mature heads.

Duplicate samples of flower heads and stem were and analysed with LC-MS/MS using Method 575/0. The limit of quantification was 0.01 mg/kg and the mean recovery was 99% at fortification levels of 0.01–5.0 mg/kg. The results are summarized in Table 20.

Table 20 Residues in broccoli from foliar application of dimethomorph in field trials in the USA

BROCCOLI Country, year (variety)	Form	Application				PHI (days)	Dimethomorph Residues in flower head (inflorescence) (mg/kg)		Reference & Comments
		kg ai/ha	kg ai/hL	no	Growth stage at last treatment		ind. value	mean	
Critical GAP USA		0.21		3		0			
CAN (QC) 2008 (Packman)	SC 500 g	0.230	0.08	3	BBCH 605	0	1.23 1.64	1.435	2009/7006205 RCN R080058
						1	1.45 1.53	1.49	
						3	0.44 0.34	0.39	
						7	0.16	0.155	

BROCCOLI Country, year (variety)	Form	Application				PHI (days)	Dimethomorph Residues in flower head (inflorescence) (mg/kg)		Reference & Comments
						10	0.15 0.03 0.02	0.025	
USA (WI) 2008 ^a (Packman)	SC 500 g	0.226	0.08	3	6–10" heads	0 1 3 7 10	1.61 1.62 0.82 1.02 0.53 0.53 0.16 0.14 0.06 0.04	1.62 0.92 0.53 0.15 0.05	2009/7006205 RCN R080059
USA (WI) 2008 (Packman)	SC 500 g	0.240	0.086	3	BBCH 48	0 1 3 7 10	0.93 0.87 0.78 0.81 0.27 0.29 0.20 0.19 0.10 0.11	0.9 0.795 0.28 0.195 0.105	2009/7006205 RCN R080060
USA (CA) 2008 (Green Majic)	SC 500 g	0.221	0.079	3	Mature	0 1 3 7 10	1.82 1.67 1.64 1.24 0.72 0.84 0.38 0.46 0.16 0.22	1.745 1.44 0.78 0.42 0.19	2009/7006205 RCN R080062
USA (CA) 2008 (Marathon)	SC 500 g	0.225	0.08	3	Mature heads	0 1 3 7 10	2.14 1.96 2.62 2.04 1.40 1.46 0.85 0.98 0.79 0.72	2.05 2.33 1.43 0.915 0.755	2009/7006205 R080063
USA (CA) 2008 (Green Magic)	SC 500 g	0.223	0.079	3	Mature heads	0 1 3 7 10	0.71 0.64 0.51 0.36 0.38 0.41 0.15 0.14 0.08 0.12	0.675 0.435 0.395 0.145 0.10	2009/7006205 RCN R080064
USA (AZ) 2008 (Crown Set)	SC 500 g	0.229	0.08	3	Early maturity	0 1 3 7	0.30 0.19 0.23 0.13 0.12 0.12 0.03 0.09	0.245 0.18 0.12 0.06	2009/7006205 RCN R080065

BROCCOLI Country, year (variety)	Form	Application				PHI (days)	Dimethomorph Residues in flower head (inflorescence) (mg/kg)		Reference & Comments
						10	0.03 0.08	0.55	
USA (CA) 2008 (Greenbelt)	SC 500 g	0.227	0.081	3	BBCH 49	0	1.87 1.89	1.88	2009/7006205 RCN R080066
						1	1.64 1.41	1.525	
						3	1.41 0.99	1.2	
						7	0.85 0.77	0.81	
						10	0.66 0.50	0.58	
USA (OR) 2008 (Emerald Pride)	SC 500 g	0.226	0.079	3	BBCH 47	0	0.98 0.91	0.945	2009/7006205 RCN R080067
						1	0.74 0.71	0.725	
						3	0.07 0.11	0.09	
						7	0.04 0.05	0.045	
						10	< 0.01 0.03	0.02	
USA (TX) 2008 (Marathon)	SC 500 g	0.223	0.084	3	BBCH 49	0	0.84 0.64	0.74	2009/7006205 RCN R080685
						1	0.59 0.53	0.56	
						3	0.47 0.72	0.595	
						7	0.31 0.26	0.285	
						10	0.12 0.15	0.135	

^a With 267–295 L water/ha

Pepper (bell and non-bell)

Twelve outdoor trials on bell peppers and non-bell peppers were conducted in the USA during growing seasons 1998 and 1999. Seven foliar broadcast applications of dimethomorph were made with a 4–15 days interval. The growth stage for the applications was from vegetative (5–16 cm) to mature heads. Three of the trials were decline trials.

Duplicate samples were analysed using GC-ECD method FAMS 002-04. The average procedural recovery range for dimethomorph was (95–119%) at the fortification level of 0.025–5 mg/kg and the limit of quantification was 0.025 mg/kg. The results are summarized in Table 21.

Table 21 Residues in pepper (bell and non-bell) from outdoor foliar application of dimethomorph in trials in USA

PEPPER Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues (mg/kg) fruit		Reference & Comments
		kg ai/ha ^d	Growth stage at last treatment	no		ind. value	mean	
Critical GAP Canada		0.225		5	0			
USA (NJ) 1999 (bell, Bell Captain)	WP 500 g	0.225	Mature fruit	7	0	0.125 0.126	0.126	2002/7013750 NJ01
USA (NJ) 1998	WP	0.225	Mature	7	6 ^a	0.165	0.179	2002/7013750

PEPPER Country, year	Form	Application			PHI (days)	Dimethomorph Residues (mg/kg) fruit		Reference & Comments
(bell King Arthur)	500 g		fruit			0.192		NJ10
USA (GA) 1998 (bell, Camelot)	WP 500 g	0.225	Mature fruit	7	0 0	0.429 0.526 0.753 0.921	0.478 ^b 0.837 ^c	2002/7013750 GA 08
USA (GA) 1998 (non-bell, Mesilla)	WP 500 g	0.225	Mature fruit	7	0	0.884 1.205	1.045	2002/7013750 GA 09
USA (FL) 1998 (bell, Camelot)	WP 500 g	0.225	Mature fruit	7	0 7	0.084 0.088 0.035 0.061	0.057 0.048	2002/7013750 FL 26
USA (FL) 1998 (non-bell, Jalapeno)	WP 500 g	0.225	Mature fruit	7	0	0.078 0.081	0.08	2002/7013750 FL 27
USA (OH) 1998 (bell, King Arthur)	WP 500 g	0.225	Fruiting	7	0	< 0.025 0.044	0.035	2002/7013750 OH 03
USA (TX) 1998 (bell, Capistrano)	WP 500 g	0.225	Mature fruit	7	0 7	0.107 0.110 0.027 0.030	0.109 0.029	2002/7013750 TX 09
USA (TX) 1998 (bell, TAM Veracruz)	WP 500 g	0.225	Mature fruit	7	0	0.660 0.730	0.695	2002/7013750 TX 10
USA (CA) 1998 (bell, Valiant)	WP 500 g	0.225	Fruiting	7	0	0.130 0.134	0.132	2002/7013750 CA 23
USA (CA) 1998 (bell, Indra)	WP 500 g	0.225	Mature fruit	7	0 6	0.137 0.144 0.163 0.166	0.141 0.165	2002/7013750 CA 27
USA (CA) 1998 (non-bell, Rogers Jalapone M)	WP 500 g	0.225	Mature fruit	7	0	0.211 0.296	0.254	2002/7013750 CA 28

^a At the NJ10 trial samples were collected at 6 days after last application, the 0 day was lost due to freezer failure. This trial was repeated the following year (NJ01)

^b Harvested after six applications, because the peppers were ripening faster than expected and the fruit was not expected to last for the 7th application

^c Harvested after seven applications. At the 7th application there was sufficient fruit for a good harvest and a sample good be taken

^d Spray volumes of 275–625 L/ha

Tomato

Twelve outdoor trials on tomato were evaluated by JMPR 2007. Results from those trials are presented in Table 22 below.

Table 22 Residues in tomato from outdoor foliar application of dimethomorph in trials in the USA

TOMATO Country, year (variety)	Form	Application			Dimethomorph Residues (mg/kg)	Reference & Comments
		kg ai/ha	no	PHI (days)	fruit	
Critical GAP Canada		0.225	5	0		
USA (CA) 1995 (Flaver saver Calgene)	WP 500 g	0.22	6	0 3 7 14 21	0.07 < 0.05 0.06 < 0.05 0.05	DK-723-028
USA (CA) 1995 (Heniz 8892)	WP 500 g	0.22	6	0 3 7	0.2 0.41 0.22	DK-723-031

TOMATO Country, year (variety)	Form	Application			Dimethomorph Residues (mg/kg)	Reference & Comments
		kg ai/ha	no	PHI (days)	fruit	
USA (CA) 1995 (Peach Mech)	WP 500 g	0.22	6	0 3 7	0.38 0.26 0.16	DK-723-030
USA (CA) 1995 (Shady Lady)	WP 500 g	0.22	6	0 3 7 14 21	0.11 0.08 0.06 0.05 < 0.05	DK-723-029
USA (TE) 1995 (Better Boy)	WP 500 g	0.22	6	0 3 7 14 21	0.14 0.21 0.17 < 0.05 < 0.05	DK-723-027
USA (CA) 1998 (Rio Grande)	WP 90 g	0.22	6	0 3 7	0.06 0.05 < 0.05	DK-723-034
USA (CA) 1998 (Celebrity)	WP 90 g	0.22	7	0 3 7	0.35 0.51 0.35	DK-723-043
USA (CA) 1998 (Roma Hybrid 882)	WP 90 g	0.22	7	0 3 7	0.13 0.14 0.1	DK-123-249
USA (CA) 1998 (Sun 6200)	WP 90 g	0.22	7	0 3 7	0.3 0.14 0.09	DK-723-048
USA (FL) 1996 (Agriset)	WP 90 g	0.22	7	0 3 7	< 0.05 < 0.05 < 0.05	DK-723-032
USA (PE) 1996 (La Roma)	WP 90 g	0.22	7	0 3 7	< 0.05 < 0.05 < 0.05	DK-723-036
USA (SC) 1996 (Celebrity)	WP 90 g	0.22	7	0 3 7	0.06 < 0.05 < 0.05	DK-723-035

Lettuce, head

Eight outdoor residue decline trials from the USA (seven) and Canada (one) in lettuce head were conducted during growing season 2008. Dimethomorph was applied three times as a foliar broadcast spray and a spray interval of 4–8 days. The timing of the applications was from head development until mature heads. Duplicate samples (fresh heads with wrappers leaves) were analysed with HPLC-MS/MS using Method 575/0. The limit of quantification was 0.01 mg/kg and the mean recovery 75% at fortification levels from 0.01–20.0 mg/kg. The results are summarized in Table 23.

In six similar trials from the USA made in during the growing season of 1998, dimethomorph was applied seven to eight times as foliar spray with a spray interval of 5 to 11 days. Mature heads were analysed using GC-ECD and Method FAMS 002-04. The limit of quantification was 0.025 mg/kg and the mean recovery 101 ± 15 fortification level from 0.025–10 mg/kg. The results are summarized in Table 24.

Table 23 Residues in outdoor lettuce head from foliar application of dimethomorph in trials from the USA

LETTUCE, HEAD Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues (mg/kg)		Reference & Comments
		kg ^b ai/ha	no	Growth stage at last treatment		Heads, Fresh with wrapper leaves		
						individual value	average value	
Critical GAP USA	SC	0.19	3		0			
USA (NY) 2008 (Ithaca MTP)	SC 250 g/L	0.225	3	BBCH 49 Mature Heads	0	1.42	1.355	2009/7003324 RCN R080224
					0	1.29		
					1	1.22	1.195	
					1	1.17		
					3	0.21 ^a	0.17	
					3	0.13 ^a		
					7	0.05	0.03	
					7	0.1		
					10	0.04	0.045	
					10	0.05		
USA (FL) 2008 (Great Lakes)	SC 250 g/L	0.225	3	BBCH 49 Mature Heads	0	2.49	2.815	2009/7003324 RCN R08025
					0	3.14		
					1	2.91	2.87	
					1	2.83		
					3	2.23	1.925	
					3	1.62		
					7	0.89	0.975	
					7	1.06		
					10	0.69	0.082	
					10	0.48		
USA (WI) 2008 (Fall Green MTO)	SC 250 g/L	0.225	3	Small medium heads	0	1.77	1.420	2009/7003324 RCN R080226
					0	1.07		
					1	1.09	1.110	
					1	1.13		
					3	0.17	0.12	
					3	0.07		
					7	0.08	0.08	
					7	0.08		
					10	0.05	0.05	
					10	0.05		
CA (QC) 2008 (Grand Rapid)	SC 250 g/L	0.225	3	BBCH 47 (70% of expected head size)	0	2.09	2.055	2009/7003324 RCN R080227
					0	2.02		
					1	1.47	1.885	
					1	2.30		
					3	0.51	0.505	
					3	0.50		
					7	0.39	0.34	
					7	0.29		
					10	0.20	0.175	
					10	0.15		
USA (CA) 2008 (Sidewinter)	SC 250 g/L	0.225	3	Head development	0	4.10	4.095	2009/7003324 RCN R080228
					0	4.09		
					1	3.77	3.495	
					1	3.22		
					3	2.91	2.85	
					3	2.79		
					7	3.52	3.65	
					7	3.78		
					10	2.83	2.425	
					10	2.02		
USA (CA) 2008 (Great Lakes 659)	SC 250 g/L	0.225	3	BBCH 49 Mature Heads	0	2.43	2.295	2009/7003324 RCN R08029
					0	2.16		
					1	1.17	0.93	
					1	0.69		
					3	0.52	0.52	

LETTUCE, HEAD Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues (mg/kg)		Reference & Comments
		kg ^b ai/ha	no	Growth stage at last treatment		Heads, Fresh with wrapper leaves		
						individual value	average value	
					3 7 7 10 10	0.52 0.11 ^a 0.23 ^a 0.14 0.17	0.17 0.155	
USA (CA) 2008 (Tellmark)	SC 250 g/L	0.225	3	Heading	0 0 1 1 3 3 7 7 10 10	1.61 1.83 1.62 1.23 0.78 0.97 0.20 0.34 0.45 ^a 0.31 ^a	1.72 1.425 0.875 0.27 0.38	2009/7003324 RCN R08030
USA (CA) 2008 (Telluride)	SC 250 g/L	0.225	3	BBCH 49 Mature Heads	0 0 1 1 3 3 7 7 10 10	1.23 1.19 1.07 1.08 0.84 0.84 0.3 0.24 0.21 0.26	1.21 1.075 0.84 0.27 0.235	2009/7003324 RCN R08031

^a Mean of multiple analysis of the same field sample.^b Spray volumes of 279–295 L/ha

Table 24 Residues in outdoor lettuce head from foliar application of dimethomorph in trials from the USA

LETTUCE, HEAD Country, year (variety)	Form	Application			PHI (days)	Heads Fresh with wrapper leaf		Heads Fresh without wrapper leaf	2001/50033 06
		kg ^a ai/ha	no	Growth stage at last treatment		individ. value	mean		
Critical GAP USA	SC	0.19	3		0				
USA (CA) 1998 (Winterhaven M.I.)	WP 50	0.199	7	Mature Heads	0	4.372		0.441	2001/50033 06 CA 113
					2	1.091	1.125		
					2	1.159			
					7	0.672	0.853		
					7	1.033			
					14	0.453	0.487		
					14	0.521			
USA (FL) 1998 (Crispino)	WP 50	0.198	7	Beginning to bolt	0	3.634		0.198	2001/50033 06 FL 67
					3	0.446	0.499		
					3	0.551			
					7	0.198	0.223		
					7	0.247			
					14	0.030	0.024		
					14	0.018			
USA (TN) 1998 (cultivar not stated)	WP 50	0.203	7	Mature Heads	0	6.447		0.638	2001/50033 06 TN 08
USA (CA) 1998 (251)	WP 50	0.200	7	Mature Heads	0	1.076		0.080	2001/50033 06

LETTUCE, HEAD Country, year (variety)	Form	Application			PHI (days)	Heads Fresh with wrapper leaf		Heads Fresh without wrapper leaf	2001/50033 06
		kg ^a ai/ha	no	Growth stage at last treatment		individ. value	mean		
Critical GAP USA	SC	0.19	3		0				
									CA 116
USA (CA) 1998 (Salinas, M.I.)	WP 50	0.200	8	Mature Heads	0	1.454		0.247	2001/50033 06 CA 117
USA (CA) 1998 (Great Lake 659)	WP 50	0.200	7	Mature Heads	0	1.675		0.046	2001/50033 06 CA 118

^a Spray volumes of 285–380 L/ha

Lettuce, leaf

Nine field trials were conducted in USA in during growing season 2008. Dimethomorph was applied three times as a foliar broadcast spray and intervals of 4–8 days. The growth stage for the applications varied from vegetative stage until BBCH 49. Duplicate samples of mature leaves were sampled and analysed with HPLC-MS/MS using Method 575/0. The limit of quantification was 0.01 mg/kg and the mean recovery 75% at fortification levels from 0.01–20.0 mg/kg. The results are summarized in Table 25.

Nine field trials were conducted in the USA during growing season 1998. Dimethomorph was applied seven times as foliar spray at 0.2 kg ai/ha and spray interval of 3–6 days. Duplicate samples of leaves were analysed with GC-ECD using Method FAMS 002-04. The limit of quantification was 0.1 mg/kg and the mean recovery 98 ± 8 fortification level from 0.1–10 mg/kg. The results are summarized in Table 25.

Table 25 Residues in outdoor lettuce from foliar application of dimethomorph in trials from the USA

LETTUCE Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues (mg/kg)		Reference & Comments
		kg ai/ha	Growth stage at last treatment	no		Leaves		
						individual value	average value	
Critical GAP USA	SC	0.19		3	0			
USA (GA) 2008 (Italien Isher)	SC 250 g/L ^a	0.225	7–10 ^c	3	0 1 3 7 10	6.05 5.60 1.31 1.25 0.46 0.39 0.18 0.14 0.05 0.07	5.83 1.28 0.43 0.16 0.60	2009/7003324 RCN R080215
USA (FL) 2008 (Bibb)	SC 250 g/L ^a	0.225	vegetative	3	0 1 3 7 10	4.78 5.60 4.48 4.29 2.52 2.41 0.93 0.55 0.34 0.23	5.19 4.39 1.26 0.74 0.29	2009/7003324 RCN R080216
USA (WI) 2008 (Black Seeded)	SC 250 g/L ^a	0.225	vegetative	3	0	10.42 ^c 9.11 ^c	9.77	2009/7003324 RCN R080217

LETTUCE Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues (mg/kg)		Reference & Comments
		kg ai/ha	Growth stage at last treatment	no		Leaves		
						individual value	average value	
Simpson)					1 3 7 10	7.38 7.81 1.22 1.17 0.43 0.51 0.20 0.27	7.6 1.2 0.47 0.24	
USA (QC) 2008 (Great Leak)	SC 250 g/L ^a	0.225	BBCH 49 (typical leaf mass reached)	3	0 1 3 7 10	3.69 3.67 0.47 0.41 0.26 0.26 0.16 0.16 0.11 0.11	3.68 0.44 0.26 0.16 0.11	2009/7003324 RCN R080218
USA (CA) 2008 (Tohema)	SC 250 g/L ^a	0.225	mature	3	0 1 3 7 10	10.53 9.22 8.68 10.7 8.92 9.81 9.24 10.09 5.94 6.85	9.88 9.69 9.37 9.67 6.39	2009/7003324 RCN R080219
USA (CA) 2008 (Salad Bowl)	SC 250 g/L ^a	0.225	24 leaves	3	0 1 3 7 10	2.15 2.04 3.48 2.44 2.20 3.11 1.09 0.91 0.7 ^c 0.36 ^c	2.09 2.96 3.16 1.00 0.53	2009/7003324 RCN R08020
USA (CA) 2008 (Butter Crunch)	SC 250 g/L ^a	0.225	7–9 leaves	3	0 1 3 7 10	3.44 3.29 2.85 2.83 2.43 2.67 1.67 1.35 0.48 0.50	3.37 2.84 2.55 1.01 0.49	2009/7003324 RCN R08021
USA (CA) 2008 (Sunbelt)	SC 250 g/L ^a	0.225	8–11 leaves	3	0 1 3 7 10	4.68 4.53 3.56 3.60 0.47 0.43 0.09 0.08 0.06 0.05	4.61 3.58 0.45 0.09 0.55	2009/7003324 RCN R08022
USA (OR) 2008 (Red Sails)	SC 250 g/L ^a	0.225	BBCH 49 (typical	3	0	5.10 5.66	5.38	2009/7003324 RCN R08023

LETTUCE Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues (mg/kg)		Reference & Comments
		kg ai/ha	Growth stage at last treatment	no		Leaves		
						individual value	average value	
			leaf mass reached)		1 3 7 10	3.52 3.66 0.95 1.23 0.58 0.57 0.37 0.56	3.59 1.09 2.57 0.37	
USA (CA) 1998 (Green Vision)	WP 50 ^b	0.198	Mature Leaves	7	0	2.7 3.4	3.1	2001/5003305 CA 25
USA (CA) 1998 (Paris Island, Cos M.1.)	WP 50 ^b	0.2	Mature Leaves	7	0	6.6 8.0	7.3	2001/5003305 CA 26
USA (CA) 1998 (Waldman’s Green M1)	WP 50 ^b	0.199	Mature Leaves	7	0	5.6 6.3	5.95	2001/5003305 CA 114
USA (CA) 1998 (Waldman’s Green M1)	WP 50 ^b	0.201	Mature Leaves	7	0	4.9 5.3	5.1	2001/5003305 CA 115
USA (FL) 1998 (Romaine)	WP 50 ^b	0.201	Beginning to bolt	7	0	6.8 7.2	7.0	2001/5003305 FL 25
USA (NY) 1998 (New Red Fire MTO)	WP 50 ^b	0.2	Mature Leaves	7	0	3.0 4.1	3.55	2001/5003305 NY 08
USA (OR) 1998 (Paris Island Cos)	WP 50 ^b	0.199	Mature Leaves	7	0	3.3 3.5	3.4	2001/5003305 OR 09
USA (TX) 1998 (Black Seed Simpson)	WP 50 ^b	0.199	Vegetativ e	7	0	8.6 9.1	8.85	2001/5003305 TX 08
USA (TX) 1998 (Paris Island Cos	WP 50	0.199	Mature Leaves	7	0 3 7 12	2.6 3.1 2.4 2.5 1.1 1.2 0.16 0.31	2.85 2.45 1.15 0.24	2001/5003305 TX 26

^a Spray volumes of 270–301 L/ha

^b Spray volumes of 285–740 L/ha

^c Mean of multiple analysis of the same field sample.

Spinach

Eight field trials were conducted in the USA in during growing season 2008. Dimethomorph was applied three times as a foliar broadcast spray and spray intervals of 4–8 days. The timing of the applications was from 6–8 leaves until BBCH 49. Duplicate samples of mature leaves were analysed with LC-MS/MS using Method 575/0. The limit of quantification was 0.01 mg/kg and the mean recovery 75% at fortification levels from 0.01–20.0 mg/kg. The results are summarized in Table 26.

Table 26 Residues in outdoor spinach from foliar application of dimethomorph in trials from the USA

SPINACH Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues (mg/kg)	Reference & Comments
		kg	no	Growth stage		Leaves	

		ai/ha		at last treatment		individual value	average value	
Critical GAP USA	SC	0.19	3		0			
USA (NY) 2008 (Melody)	SC 250 g/L ^b	0.225	3	plants close to maturity	0 0 1 1 3 3 7 7 10 10	6.01 5.8 1.24 1.19 0.65 0.68 0.38 0.39 0.16 0.19	5.91 1.22 0.67 0.39 0.18	2009/7003324 RCN R080232
USA (GA) 2008 (Space FI)	SC 250 g/L ^b	0.225	3	9–16 leaves	0 0 1 1 3 3 7 7 10 10	8.59 7.83 6.13 7.22 2.25 ^a 1.91 ^a 0.02 0.02 < 0.01 < 0.01	8.21 6.68 2.44 0.02 < 0.01	2009/7003324 RCN R08033
USA (WI) 2008 (Unipack 151)	SC 250 g/L ^b	0.225	3	15–20 cm	0 0 1 1 3 3 7 7 10 10	11.04 11.47 8.43 8.10 3.11 3.17 0.52 0.59 0.20 0.21	11.26 8.27 3.14 0.56 0.21	2009/7003324 RCN R080234
USA (QC) 2008 (Tyee)	SC 250 g/L ^b	0.225	3	BBCH 51	0 0 1 1 3 3 7 7 10 10	5.19 4.20 2.17 2.13 0.67 0.76 0.21 0.23 0.08 0.09	4.69 2.15 0.72 0.22 0.09	2009/7003324 RCN R080235
USA (TX) 2008 (Siena)	SC 250 g/L ^b	0.225	3	BBCH 49	0 0 1 1 3 3 7 7 10 10	9.32 7.37 7.21 6.01 6.78 6.34 3.98 3.89 2.02 2.24	8.35 6.61 6.56 3.94 2.13	2009/7003324 RCN R080236
USA (ID) 2008 (Unipack 151)	SC 250 g/L ^b	0.225	3	BBCH 48	0 0 1 1 3 3 7 7 10 10	8.47 8.48 7.39 7.19 6.28 6.36 1.25 1.33 0.23 0.20	8.48 7.29 6.32 1.29 0.22	2009/7003324 RCN R08037
USA (CA) 2008	SC	0.225	3	30.5 cm	0	9.67	10.18	2009/7003324

SPINACH Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues (mg/kg)		Reference & Comments
		kg ai/ha	no	Growth stage at last treatment		Leaves		
						individual value	average value	
(Crocodile)	250 g/L ^b				0	10.69		RCN R08038
					1	9.72		
					1	7.55		
					3	8.44		
					3	9.71		
					7	8.30		
					7	9.17		
					10	3.43		
					10	3.13		
					USA (OR) 2008 (Avenger)	SC 250 g/L ^b		
0	4.63							
1	0.74							
1	0.80							
3	0.69							
3	0.56							
7	0.10							
7	0.10							
10	0.07							
10	0.06							

^a Mean of multiple analysis of the same field sample.

^b Spray volumes of 274–275 L/ha.

Taro, leaf

Three field trials on taro were conducted in Hawai during growing season 2000. Dimethomorph was applied seven times as a foliar spray (spray interval of 7 to 8 days) or drip irrigation.

Duplicate samples of leaves were analysed using GC-ECD and Method FAMS 002-04. The limit of quantification was 0.01 mg/kg and the recovery 76–119% at fortification level 0.01–10 mg/kg for leaves. The results are summarized in Table 27.

Table 27 Residues in outdoor Taro from foliar application of dimethomorph in trials from the USA

TARO Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues (mg/kg)	Reference & Comments
		kg ai/ha ^a	no	Growth stage at last treatment			
Critical GAP USA, except California		0.19	5		7		
USA (HI) 2000 (Chinese)	WP 500 g	0.225 ^b	7	vegetative, 1.83 m	7	3.65 5.40 mean 4.525	2003/7010215 IR-4 PR 07335 H109
USA (HI) 2000 (Chinese - 'Bun Long')	WP 500 g	0.225	7	vegetative 1.53 m	7	1.78 1.49 mean 1.63	2003/7010215 IR-4 PR 07335 H110
USA (HI) 2000 (Chinese - 'Bun Long')	WP 500 g	0.225	7	vegetative 1.83 m	0 28	0.633 2.24 mean 1.437	2003/7010215 IR-4 PR 07335 H11

^a Spray volumes of 1400–1900 L/ha

^b Drip irrigation

*Legume vegetables**Pea, Shelled, (succulent seed)*

Two field trials were conducted in France and Germany during the growing season of 2005. Dimethomorph was applied as foliar broadcast application two times 29 and 21± 1 day before harvest. Samples of peas were analysed by LC-MS/MS using Method 575/0. The limit of quantification was 0.01 mg/kg and recovery 79–89% for peas with pods and 83–96% for rest of plants without roots at fortification level 0.01–10 mg/kg. The results are summarized in Table 28.

During the growing season of 2006 ten field trials were conducted on green peas in Spain, France (north and south), Germany, the Netherlands and Denmark. Dimethomorph was applied as foliar broadcast application two times 29 and 21± 1 day before harvest. Samples were analysed using LC-MS/MS and Method 575/0. The limit of quantification was 0.01 mg/kg and recovery 83–95% for peas with pods and 80–97% for rest of plants without roots at fortification level 0.01–10 mg/kg. The results are summarized in Table 28.

Table 28 Residues in Green Peas from foliar application of dimethomorph in field trials in Europe

GREEN PEAS Country, year (variety)	Form	Application			PHI (days)	Commodity	Residues (mg/kg)	Reference & Comments
		kg ai/ha ^a	no	Growth stage at last treatment BBCH				
Critical GAP France		0.18	2		21			
France, 2005/6 (Barely)	WG 90 g/kg	0.180	2	71	0	peas with pods	0.615	2006/1032765 05 CL FR P37
					0	rest of plant w/o roots	7.032	
					13	fresh peas w/o pods	< 0.01	
					13	rest of plant w/o roots	5.167	
					20	fresh peas w/o pods	< 0.01	
					20	rest of plant w/o pods	3.583	
					28	fresh peas w/o roots	0.023	
					28	rest of plant w/o pods	4.973	
Germany 2005/6 (Trompet)	WG 90 g/kg	0.180	2	67	0	peas with pods	1.175	2006/1032765 AC/05/100
					0	rest of plant w/o pods	4.186	
					14	fresh peas w/o pods	< 0.01	
					14	rest of plant w/o roots	0.511	
					20	fresh peas w/o pods	< 0.01	
					20	rest of plant w/o roots	1.060	

GREEN PEAS Country, year (variety)	Form	Application			PHI (days)	Commodity	Residues (mg/kg)	Reference & Comments
		kg ai/ha ^a	no	Growth stage at last treatment BBCH				
Spain, 2006 (Jumbo)	WG 90 g/kg	0.18	2	73	27	fresh peas w/o pods	< 0.01	2007/1005061 06ES/094R
					27	rest of plant w/o roots	1.884	
					0	peas with pods	0.568	
					0	rest of plant w/o roots w/o pods	4,95	
					13	fresh peas w/o pods	< 0.01	
					13	rest of plant w/o roots	2,96	
					20	fresh peas w/o pods	< 0.01	
					20	rest of plant w/o roots	2.03	
Spain, 2006 (Argona)	WG 90 g/kg	0.18	2	73	28	fresh peas w/o pods	< 0.01	2007/1005061 06ES/095R
					28	rest of plant w/o roots	5.45	
					0	peas with pods	0.324	
					0	rest of plant w/o roots w/o pods	3.35	
					14	fresh peas w/o pods	< 0.01	
					14	rest of plant w/o roots	0.575	
					21	fresh peas w/o pods	< 0.01	
					21	rest of plant w/o roots	0.620	
France, 2006 (Frediro)	WG 90 g/kg	0.18	2	75	28	fresh peas w/o pods	< 0.01	2007/1005061 06FR/096R
					28	rest of plant w/o roots	1.57	
					0	peas with pods	0.447	
					0	rest of plant w/o roots w/o pods	3.65	
					14	fresh peas w/o pods	< 0.01	
					14	rest of plant w/o roots	0.368	
					21	fresh peas w/o pods	< 0.01	
					21	rest of plant w/o roots	0.311	
					28	fresh peas w/o pods	< 0.01	
					28	rest of plant	0.588	

GREEN PEAS Country, year (variety)	Form	Application			PHI (days)	Commodity	Residues (mg/kg)	Reference & Comments
		kg ai/ha ^a	no	Growth stage at last treatment BBCH				
France, 2006 (Frediro)	WG 90 g/kg	0.18	2	75		w/o roots		2007/1005061 06FR/097R
					0	peas with pods	0.358	
					0	rest of plant w/o roots w/o pods	2.86	
					14	fresh peas w/o pods	< 0.01	
					14	rest of plant w/o roots	0.836	
					21	fresh peas with pods	< 0.01	
					21	rest of plant w/o roots	1.59	
					28	fresh peas w/o pods	< 0.01	
France, 2006 (Cepia)	WG 90 g/kg	0.18	2	71		rest of plant w/o roots	0.874	2007/1005061 06FR/098R
					0	peas with pods	0.505	
					0	rest of plant w/o pods w/o roots	6.130	
					15	fresh peas w/o pods	< 0.01	
					15	rest of plant w/o roots	0.0929	
					21	fresh peas w/o pods	< 0.01	
					21	rest of plant w/o roots	1.521	
					27	fresh peas w/o pods	< 0.01	
Germany, 2006 (Wunder von Kelvedon)	WG 90 g/kg	0.18	2	73		rest of plant w/o roots	2.400	2007/1005061 06FR/099R
					0	peas with pods	0.337	
					0	rest of plant w/o pods w/o roots	4.28	
					15	fresh peas w/o pods	< 0.01	
					15	rest of plant w/o roots	4.79	
					20	fresh peas w/o pods	0.063	
					20	rest of plant w/o roots	4.18	
					27	fresh peas w/o pods	< 0.041	
The Netherlands, 2006	WG 90 g/kg	0.18	2	73		rest of plant w/o roots	4.60	2007/1005061 06FR/100R
					0	peas with pods	0.351	

GREEN PEAS Country, year (variety)	Form	Application			PHI (days)	Commodity	Residues (mg/kg)	Reference & Comments
		kg ai/ha ^a	no	Growth stage at last treatment BBCH				
(Wunder von Kelvedon)					0	rest of plant w/o pods w/o roots	3.39	
					13	fresh peas with pods	0.015	
					13	rest of plant w/o roots	8.09	
					20	fresh peas w/o pods	0.044	
					20	rest of plant w/o roots	9.53	
					27	fresh peas w/o pods	0.025	
					27	rest of plant w/o roots	9.58	
Denmark, 2006 (Progress no 9 Lot ZP1065)	WG 90 g/kg	0.18	2	73	0	peas with pods	0.662	2007/1005061 06FR/101R
					0	rest of plant w/o pods w/o roots	3.52	
					13	fresh peas with pods	< 0.01	
					13	rest of plant w/o roots	0.242	
					22	fresh peas w/o pods	< 0.01	
					22	rest of plant w/o roots	0.353	
					28	fresh peas w/o pods	< 0.01	
					28	rest of plant w/o roots	0.420	

^a Spray volumes of 285–300 L/ha.

Vining peas

During the growing season of 2008 four field trials were conducted on vining peas in Northern France, Germany and the Netherlands. Dimethomorph was applied as a foliar broad cast application two times 29 ± 1 and 21 ± 1 days before harvest. Samples (peas with pods and rest of plant w/o roots) were collected directly after the second application. Samples of pods and seed were taken at and 20 ± 1 day after the last treatment. Pea seeds were analysed using LC-MS/MS and the Method 535/1. The limit of quantification was 0.01 mg/kg and the recovery 72–110% for the different samples (n2) at fortification levels 0.01–10 mg/kg. The results are summarized in Table 29.

Table 29 Residues in Vining peas from foliar application of dimethomorph in field trials from Europe

VINING PEAS Country, year (variety)	Form	Application			PHI (days)	Commodity	Residues (mg/kg)	Reference & Comments
		kg ai/ha ^a	no	Growth stage at last treatment BBCH				
Critical GAP France		0.18	2		21			
France, 2008 (Cariboue)	WG 90 g/kg	0.180 0.187	2	62 71	0	peas with pods	0.768	2009/1069170 A8096 BM1
					0	rest of plant without roots	3.416	
					20	seeds (manual)	< 0.01	
					20	Pods	0.021	
					20	seeds (mechanical)	< 0.01	
France, 2008 (Louvette)	WG 90 g/kg	0.207 0.208	2	74 75	0	peas with pods	0.983	2009/1069170 A8096 OB1
					0	rest of plant without roots	9.327	
					20	seeds (manual)	0.016	
					20	Pods	0.237	
					20	seeds (mechanical)	0.071	
Germany, 2008 (Esprit)	WG 90 g/kg	0.196 0.205	2	73 75	0	peas with pods	0.372	2009/1069170 A8096 DE1
					0	rest of plant without roots	2.323	
					21	seeds (manual)	< 0.01	
					21	Pods	< 0.01	
					21	seeds (mechanical)	< 0.01	
The Netherlands, 2008 (Arlette)	WG 90 g/kg	0.202 0.202	2	73 75	0	peas with pods	0.506	2009/1069170 A8096 NL1
					0	rest of plant without roots	3.234	
					22	seeds (manual)	< 0.01	
					22	Pods	0.011	
					22	seeds (mechanical)	< 0.01	

^a Spray volumes of 200 L/ha

Lima Beans

During the 2002 and 2003 growing season seven field trials on Lima beans were conducted in the USA. Dimethomorph was applied as a foliar broad cast spray seven times with an interval of 5–11 days. Duplicate samples were analysed using GC-MSD and the Method FAMS 002-04 approved for grapes the 2007 Meeting. The limit of quantification was 0.01 mg/kg and the recovery 71–123% for the different samples at fortification levels 0.01–1 mg/kg. The results are summarized in Table 27.

During the growing season 2010 three field trials on Lima Beans was conducted in the USA. Dimethomorph was applied as foliar broad cast spray five to six times with an interval of 6–8 days. Duplicate samples were collected (pods) at the same day as the last application and frozen within 30 minutes. The lima beans were analysed with GC-MSD using Method FAMS 002-04. The limit of quantification was 0.01 mg/kg and the recovery 85–102% for the different samples at fortification levels 0.01–1 mg/kg. The results are summarized in Table 30.

Table 30 Residues in Lima beans from foliar application of dimethomorph in field trials from the USA

LIMA BEANS Country, year (variety)	Form	Application			PHI (days)	Commodity succulent seed without pod	Residues (mg/kg)		Reference & Comments
		kg ai/ha ^a	no	Growth stage at last treatment			ind. value	mean	
Critical GAP USA		0.19	5		0				
USA, 2002/3 (GA) (Cangreen)	WP 50%	0.225	7	mature	0	bean mechanical	0.48	0.465	2007/7016820 GA10
					0		0.45		
USA, 2002/3 (GA) (Henderson Bush)	WP 50%	0.225	8	fruiting	0	bean mechanical	0.03	0.03	2007/7016820 GA11
					0		0.03		
USA, 2002/3 (GA) (Jackson Wonder)	WP 50%	0.225	7	mature	0	bean mechanical	0.09	0.1	2007/7016820 GA12
					0		0.11		
					3	bean mechanical	0.04	0.045	
					3	bean mechanical	0.05		
					5	bean mechanical	0.01	0.01	
					5	bean mechanical	0.01		
					11	bean mechanical	0.02	0.02	
					11	bean mechanical	0.02		
USA, 2002/3 (MD) (Fordhook 242)	WP 50%	0.225	7	mature	0	bean mechanical	0.21	0.21	2007/7016820 MD 14
					0		0.21		
USA, 2003 (MD) (Burpee Improved Bush)	WP 50%	0.225	7	mature	0	bean mechanical	0.03	0.03	2007/7016820 MD 03
					0		0.03		
USA, 2003 (NJ) (Bridgeton)	WP 50%	0.225	7	fruiting	0	bean mechanical	0.05	0.05	2007/7016820 NJ07
					0		0.05		
USA, 2002/3 (MD) (Improved Kingston)	WP 50%	0.225	7	fruiting	0	bean manual	0.03	0.03	2007/7016820 WI 33
					0		0.03		
USA, 2010 (CA) (Luna Baby Lima Beans)	WP ^b 50%	0.225	5	Pods Setting	0	bean manual	0.0245	0.0257	2013/1335899 CA 140
							0.0268		
	SC ^b 500 g/L		5	Pods Setting	0	bean manual	0.0199	0.0218	
							0.0236		
USA, 2010 (MD) (Eastland)	WP ^b 50%	0.225 ^b	6	Pods filling out, Some Mature	0	bean manual	0.0329	0.0403	2013/1335899 MD19
							0.0476		
	SC ^b 500 g/L		6	Pods filling out, Some Mature	0	bean manual	0.0585	0.0593	
							0.0601		
USA, 2010 (SC) (Fordhook # 242)	WP ^b 50%	0.225 ^b	5	Fruiting	0	bean manual	0.0954	0.0778	2013/1335899 SC18
							0.0602		
	SC ^b 500 g/L		5	Fruiting	0	bean manual	0.0587	0.0623	
							0.0659		

^a Spray volume of 284–488 L/ha^b Adjuvant used

*Root and vegetable crop**Ginseng*

Four outdoor trials were conducted in the USA during growing season 2004. Dimethomorph was applied seven times from blooming until berries dropping. In three trials dimethomorph was applied as a broadcast foliar spray and in one trial via drip application.

Duplicate samples were analysed using Method FAMS 073-03 to measure dimethomorph residues in ginseng roots. The limit of quantification of this method was 0.08 mg/kg and the mean recovery was 87.3–96.9% at fortification levels of 0.05–5 mg/kg. The results are summarized in Table 31.

Table 31 Residues in outdoor Ginseng from foliar application of dimethomorph in trials from the USA

Ginseng Country, year (variety)	Form	Application			PHI (days)	Commodity	Residues (mg/kg)		Reference & Comments
		kg ai/ha	no	Growth stage at last treatment			ind. value	mean	
Critical GAP USA		0.19	5		14				
USA, 2004 (MI) (American Ginseng)	WP ^a 50%	0.270 ^a	7	Fruiting	14	root	0.6	0.61	2007/1068765 MI 123
							0.62		
USA, 2004 (WI) (American Ginseng)	WP 50%	0.225 ^a	7	Red berries	13	root	0.43	0.42	2007/1068765 WI 25
							0.41		
USA, 2004 (WI) (American Ginseng)	WP 50%	0.225 ^a	7	Berries dropping	14	root	0.28	0.28	2007/1068765 WI 29
							0.27		
USA, 2004 (WI) (American Ginseng)	WP 50%	0.225 ^a	7	Berries dropping	15	root	0.28	0.28	2007/1068765 WI 30
							0.29		

^a Drip irrigation

*Stalk and stem vegetable**Globe artichoke*

Ten outdoor trials on globe artichoke from Germany, northern and southern France, the Netherlands, Spain and Italy conducted during the growing season 2006 and 2007. Dimethomorph was applied three times by foliar broadcast spray in intervals of 7 ± 1 day. The timing of the applications was from 17 until $3 \pm$ days before harvest.

Samples (artichoke heads) were analysed by HPLC-MS/MS using method 575/0. The limit of quantification of this method was 0.01 mg/kg and the recovery was 70–110% at fortification levels of 0.01 and 1.0 mg/kg. The results are summarized in Table 32.

Table 32 Residues in Artichoke from foliar application of dimethomorph in outdoor trials from Europe

ARTICHOKE (VS 0620) Country, year (variety)	Form	Application			PHI (days)	Commodity	Residues (mg/kg)	Reference & Comments
		kg ai/ha ^a	no	Growth stage at last treatment				
Critical GAP France, Italy		0.18	3		3			
N France, 2006 (Camus)	EC 72 g/L + py	0.18	3	3 days before harvest	0	heads	0.13	2008/1068911 A6025 BM1
					3		0.26	
					8		0.06	
Netherlands, 2006 (Concerto)	EC 72 g/L + py	0.18	3	3 days before harvest	0	heads	0.58	2008/1068911 A6025 NL1
					3		0.75	
					7		0.69	
Germany, 2006 (Imperial Star)	EC 72 g/L + py	0.18	3	3 days before harvest	0	heads	0.42	2008/1068911 A6025 HA1
					3		0.55	
					8		0.2	
N France, 2007 (Camus)	EC 72 g/L + py	0.18	3	4 days before harvest	0	heads	0.22	2009/1013323 A7037 BM1
					4		0.11	
					7		0.08	
Germany, 2007 (Green Globe)	EC 72 g/L + py	0.18	3	3 days before harvest	0	heads	0.36	2009/1013323 A7037 HA1
					3		0.24	
					7		0.09	
Italy, 2006 (Spinosiso, DI Albenga)	EC 72 g/L + py	0.18	3	4 days before harvest	0	heads	0.10	2008/1068911 A6025 IT1
					4		0.06	
					7		0.04	
S France, 2006 (Makau)	EC 72 g/L + py	0.18	3	3 days before harvest	0	heads	0.32	2008/1068911 A6025 TL 1
					3		0.08	
					8		0.09	
Italy, 2007 (Brindisino)	EC 72 g/L + py	0.18	3	3 days before harvest	0	heads	1.27	2009/1013323 A7037 IT1
					3		1.14	
					7		0.79	
S France, 2006 (Makau)	EC 72 g/L + py	0.18	3	3 days before harvest	0	heads	0.42	2009/1013323 A7037 TL1
					3		0.32	
					7		0.15	
Spain, 2007 (Prat)	EC 72 g/L + py	0.18	3	3 days before harvest	0	heads	0.31	2009/1013323 A7037 ES2
					4		0.14	
					7		0.10	

^a Spray volumes of 400 L/ha*Celery*

Nine outdoor residue decline trials on celery from the USA and Canada performed during growing season 2008. Dimethomorph was applied three times as a foliar broadcast spray with and spray intervals of 4–6 days. The timing of the applications was from vegetative stage (35–40 cm) until stalk elongation.

Duplicate samples were analysed by HPLC-MS/MS using Method 575/0. The limit of quantification was 0.01 mg/kg and the mean recovery $78 \pm 8\%$ at fortification levels from 0.01–10.0 mg/kg. The results are summarized in Table 33.

Table 33 Residues in celery from foliar application of dimethomorph in outdoor trials from the USA and Canada

CELERY Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues (mg/kg)		Reference & Comments
		kg ^a ai/ha	No	Growth stage at last treatment		Leaf stalk		
						individual value	average value	
Critical GAP USA		0.21	3		0			
USA (FL), 2008 (AB52)	SC 225 g/L	0.225	3	vegetative 14– 16”	0	1.25 1.28	1.265	2009/7003324 RCN R08240
					1	0.85 1.11	0.98	
					3	0.25 0.27	0.26	
					7	0.24 0.23	0.235	
					10	0.15 0.15	0.15	
USA (FL), 2008 (Utah)	SC 225 g/L	0.225	3	BBCH 49	0	5.8 5.27	5.535	2009/7003324 RCN R08241
					1	5.35 5.19	5.27	
					3	1.02 0.99	1.01	
					7	1.07 1.01	1.04	
					10	0.92 0.89	0.905	
USA (WI), 2008 (Tango)	SC 225 g/L	0.225	3	BBCH 46	0	3.12 2.8	2.96	2009/7003324 RCN R08242
					1	3.34 3.19	3.265	
					3	2.09 1.83	1.96	
					7	0.51 0.58	0.545	
					10	0.34 0.37	0.355	
Canada (QC), 2008 (Victoria)	SC 225 g/L	0.225	3	BBCH 49	0	1.47 1.62	1.545	2009/7003324 RCN R08243
					1	1.03 1.03	1.03	
					3	0.85 0.87	0.86	
					7	0.06 0.06	0.06	
					10	0.06 0.05*	0.055	
Canada (QC), 2008 (XP266)	SC 225 g/L	0.225	3	BBCH 49	0	2.1 1.6	1.85	2009/7003324 RCN R08244
					1	1.26 1.17	1.215	
					3	1.15 1.1	1.125	
					7	0.09 0.08	0.085	
					10	0.07 0.07	0.07	
USA (CA), 2008 (Mission)	SC 225 g/L	0.225	3	BBCH 49	0	7.6 8.82	8.21	2009/7003324 RCN R08245
					1	7.89 7.16	7.525	

CELERY Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues (mg/kg)		Reference & Comments		
		kg ^a ai/ha	No	Growth stage at last treatment		Leaf stalk				
						individual value	average value			
							3		5.12 5.05	5.085
							7		4.88 5.37	5.125
					10	3.32 2.3	2.81			
USA (CA), 2008 (Mission)	SC 225 g/L	0.225	3	BBCH 49	0	3.99 4.05	4.02	2009/7003324 RCN R08246		
					1	1.56 1.15	1.355			
					3	1.04 1.17	1.105			
					7	0.61 0.42	0.515			
					10	0.51 0.45	0.48			
USA (CA), 2008 (G15)	SC 225 g/L	0.230	3	BBCH 49	0	1.97 1.84	1.905	2009/7003324 RCN R08247		
					1	1.8 1.76	1.78			
					3	1.05 1.22	1.135			
					7	0.16 0.13	0.145			
					10	0.05 0.06	0.055			
USA (CA), 2008 (Sinora)	SC 2025 g/L	0.225	3	Stalk elongation	0	2.39 2.48	2.435	2009/7003324 RCN R08248		
					1	1.76 2.03	1.895			
					3	1.45 1.56	1.505			
					7	0.25 0.41	0.33			
					10	0.34 0.52	0.43			

^a Spray volumes of 277–301 L/ha

Taro corm (root)

Three field trials on taro were conducted in Hawai during growing season 2000. Meeting. Dimethomorph was applied seven times as a foliar spray or drip irrigation. Dimethomorph was applied with a spray interval of 7 to 8 days to the vegetative stage (growth height 1.2 m to 1.7 m).

The cormes were analysed using GC-ECD and Method FAMS 002-04. The limit of quantification was 0.01 mg/kg and the recovery 76–119% at fortification level 0.01–10 mg/kg for leaves. The results are summarized in Table 34.

Table 34 Residues in outdoor Taro corm from foliar application of dimethomorph in trials from USA

TARO Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues (mg/kg)	Reference & Comments
		kg ai/ha ^a	no	Growth stage at last treatment		Corms	
Critical GAP USA, except California		0.19	5		30		
USA (HI) 2000	WP	0.225 ^b	7	vegetative,	28	0.0494	2003/7010215

TARO Country, year (variety)	Form	Application			PHI (days)	Dimethomorph Residues (mg/kg)	Reference & Comments
		kg ai/ha ^a	no	Growth stage at last treatment		Corms	
(Chinese)	500 g			1.83 m		0.0464 mean 0.0479	IR-4 PR 07335 H109
USA (HI) 2000 (Chinese - 'Bun Long')	WP 500 g	0.225	7	vegetative 1.53 m	28	0.343 0.195 mean 0.269	2003/7010215 IR-4 PR 07335 H110
USA (HI) 2000 (Chinese - 'Bun Long')	WP 500 g	0.225	7	vegetative 1.83 m	28	0.0523 0.0477 mean 0.05	2003/7010215 IR-4 PR 07335 H11

^a Spray volumes of 1400–1900 L/ha

^b Drip irrigation

Fate of residues in storage and processing

Oranges

During the 2011 growing season four independent field trials were conducted in oranges (sweet) in Spain to determine the potential for concentration of residues in juice, marmalade, pulp, pomace, pulp and oil after processing to these commodities. Treated plots received two foliar applications of dimethomorph (DC formulation) at a rate of 1.22 kg ai/ha 28 and 14 days before harvest. Samples of whole fruits were taken on the day of the last application (BBCH 83–85) and 14 ± 1 day later (BBCH 89).

The processing of oranges was conducted with samples taken at the last sampling using simulated commercial processing procedures according to the flowchart below. During processing, nine different fractions of orange products or intermediates were collected for analysis specifically; juice, wet pomace, dried pomace, marmalade, peel after oil extraction, pulp, peel, dried pulp and oil.

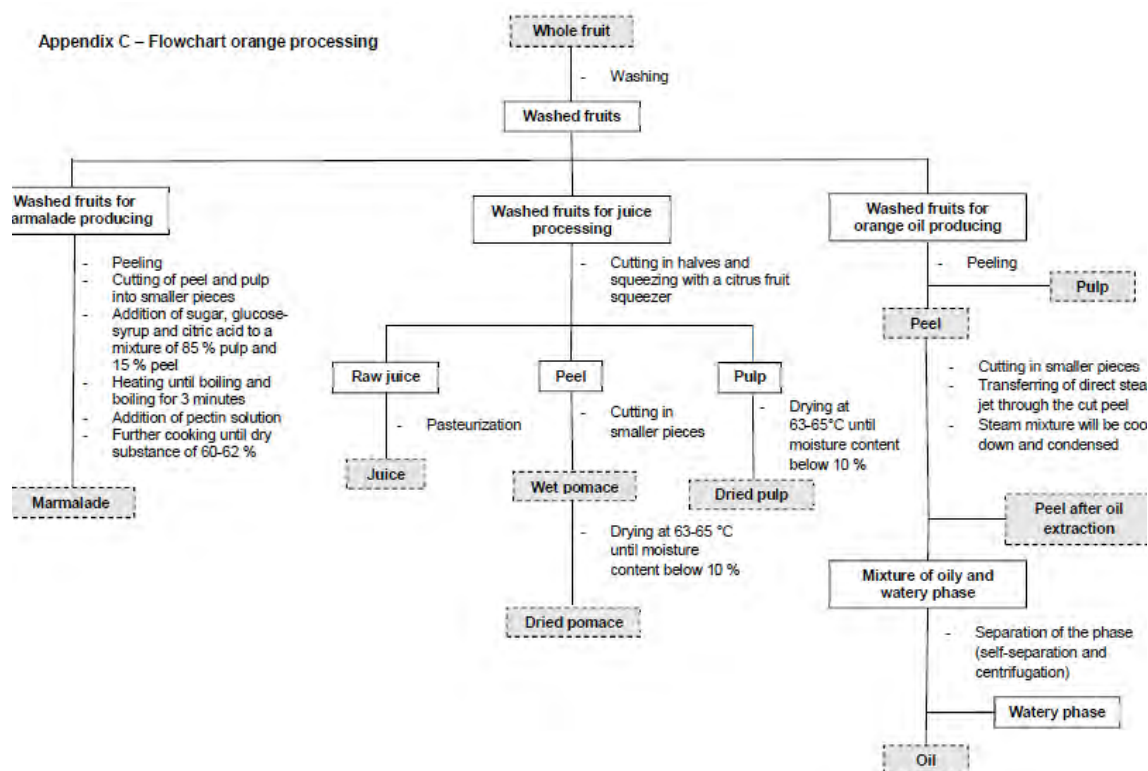


Figure 1 Flow chart of processing procedure for oranges

Table 35 Residues of dimethomorph in processed commodities of oranges

Portion analysed	Dimethomorph residues mg/kg					transfer factor (Tf) ^a				
	Days after last treatment	1	2	3	4	1	2	3	4	mean Tf best estimate
Trial ^b										
Whole fruit	0	0.34	0.31	0.32	0.73	—	—	—	—	—
Whole fruit	14–15	0.12	0.13	0.058	0.15	—	—	—	—	—
Peel	14–15	0.30	0.35	0.23	0.41	—	—	—	—	—
Pulp	14–15	0.022	0.013	< 0.010	0.013	—	—	—	—	—
Whole fruit, RAC ^c		0.079	0.12	0.12	0.25	1	1	1	1	
Juice		< 0.01 ^d	< 0.01 ^d	< 0.01 ^d	< 0.01 ^d	< 0.13	< 0.08	< 0.08	< 0.04	< 0.08
Wet pomace		0.14	0.12	0.16	0.35	1.77	1.00	1.33	1.40	1.38
Dried pomace		0.39	0.34	1.2	1.2	4.94	2.83	10.00	4.80	5.64
Marmalade		< 0.01 ^d	0.012	< 0.01	< 0.01	< 0.13	0.10	< 0.08	< 0.04	0.09
Peel after oil extraction		0.14	0.19	0.21	0.78	1.77	1.58	1.75	3.12	2.06
Pulp		< 0.010 ^d	< 0.01 ^d	< 0.01 ^d	< 0.01 ^d	< 0.13	< 0.08	< 0.08	< 0.04	< 0.08
Peel		0.11	0.18	0.18	0.53	1.39	1.50	1.50	2.12	1.62
Dried Pulp		0.026	0.014	0.026	0.048	0.33	0.12	0.22	0.19	0.22
Oil		< 0.01 ^d	< 0.01 ^d	< 0.01 ^d	< 0.01 ^d	< 0.13	< 0.08	< 0.08	< 0.04	< 0.08

^a Transfer factor = residue in processed fraction (PF)/ residue in RAC^b Trial 1: L110357, Trial 2: L110358, Trial 3: L110359, Trial 4: L110360. These trials are not reported in details here.^c At processing start^d For calculation purposes < 0.01 is set 0.01

Strawberry (doc. code 2012/1002401)

During the 2007 growing season four independent field trials in strawberries were conducted in Germany to study the potential of concentration of residues in jam and canned fruit after processing to these commodities. Dimethomorph (WP formulation) was applied once at (0.375 g ai/plant) which are 2–3 times maximum label rate for foliar and soil drench applications respectively. Samples of strawberries were collected 35 days after last application according to critical GAP.

The samples were processed into washed fruit, jam and canned fruit. During processing using simulated commercial processing procedures according to flow diagram in flowchart below different fractions of strawberry intermediates were collected for analysis; specifically washed strawberries, wash water, jam before cooking, jam after cooking, canned strawberries and vegetable stock.

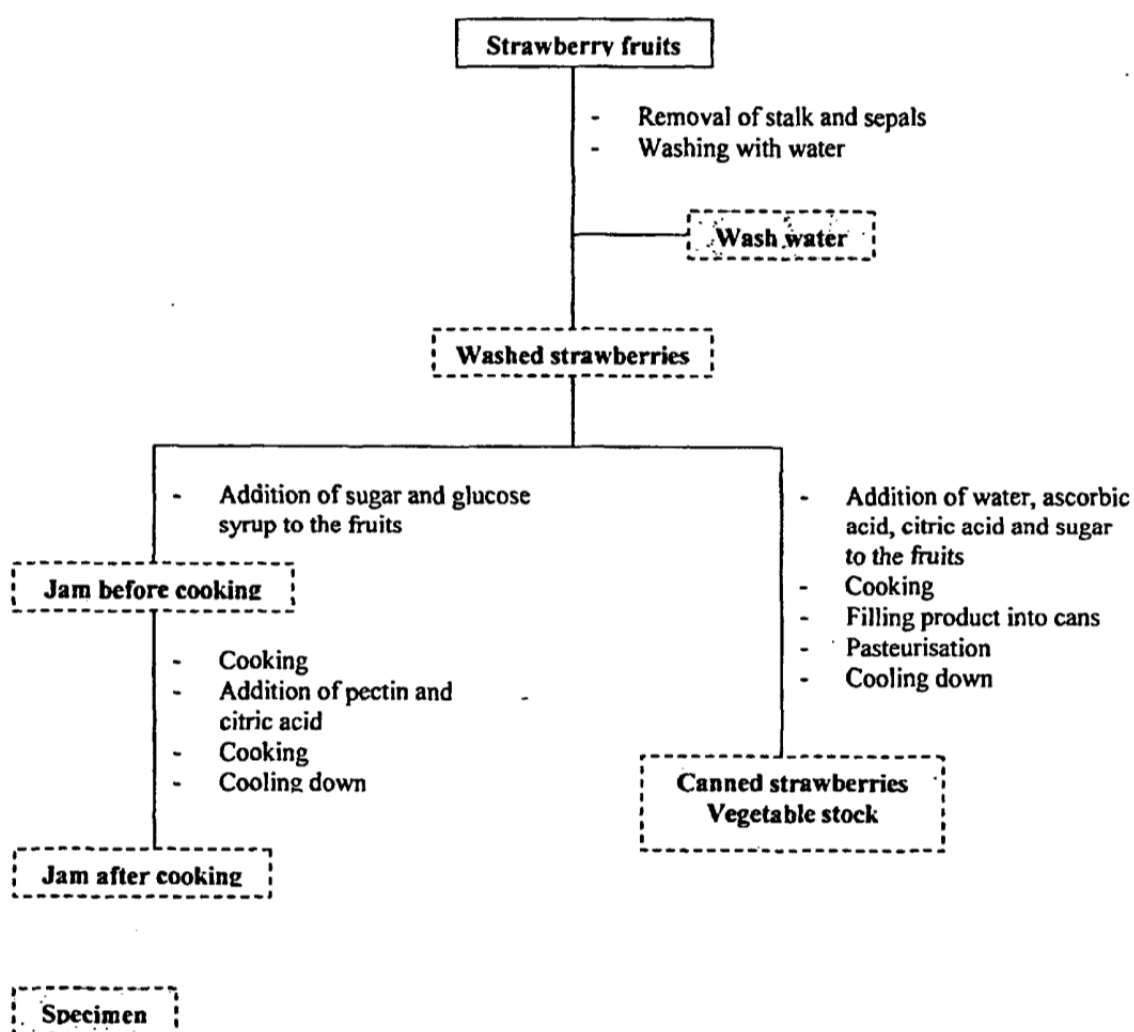


Figure 2 Flow chart of processing procedure for strawberry

Table 36 Summary of dimethomorph residues in process fractions and transfer factors, based on residue levels determined in unwashed strawberry fruit

Portion analysed	DALA	Dimethomorph residues mg/kg				transfer factor (Tf) ^a					
Trial ^b		1	2	3	4	1	2	3	4	median Tf best estimate	mean Tf excl. trials 2 and 4
Strawberry, fruit, RAC ^d	35	0.14	0.092 ^c	0.25	0.091 ^c	1	1	1	1	1	1
washed strawberries		0.11	0.17	0.27	0.2	0.79	1.85	1.08	2.20	1.47	0.94
wash water		0.13	0.07	0.19	0.065	0.93	0.76	0.76	0.71	0.76	0.85
jam before cooking		0.037	0.051	0.19	0.054	0.26	0.55	0.76	0.59	0.57	0.51
jam after cooking		0.034	0.050	0.11	0.039	0.24	0.54	0.44	0.43	0.44	0.34
canned strawberries		0.080	0.14	0.25	0.11	0.57	1.52	1	1.21	1.11	0.79
vegetable stock		0.055	0.080	0.17	0.057	0.38	0.87	0.68	0.63	0.66	0.53

^a Transfer factor = residue in processed fraction (PF)/ residue in RAC^b Trial 1: FR 24/07/50, Trial 2: FR 24/07/70, Trial 3: 24/07/30, Trial 4: 24/07/75. These trials are not reported in details here^c Unusually low values (maybe due to analytical problems)^d At processing start

Table 37 Summary of dimethomorph residues in process fractions and transfer factors based on residue levels determined in washed strawberries

Portion analysed	DALA	Dimethomorph residues mg/kg				transfer factor (Tf) ^a					
Trial ^b		1	2	3	4	1	2	3	4	median Tf	
washed strawberries	35	0.11	0.17	0.27	0.2	1	1	1	1	1	
wash water		0.13	0.07	0.19	0.065	1.18	0.41	0.7	0.32	0.56	
jam before cooking		0.037	0.051	0.19	0.054	0.34	0.30	0.7	0.27	0.32	
jam after cooking		0.034	0.050	0.11	0.039	0.31	0.29	0.4	0.195	0.3	
canned strawberries		0.080	0.14	0.25	0.11	0.73	0.82	0.93	0.55	0.78	
vegetable stock		0.055	0.080	0.17	0.057	0.50	0.47	0.63	0.28	0.49	

^a Transfer factor = residue in processed fraction (PF)/ residue in RAC^b Trial 1: FR 24/07/50, Trial 2: FR 24/07/70, Trial 3: 24/07/30, Trial 4: 24/07/75. These trials are not reported in details here*Onion (doc. code 2010/1093126)*

During the 2009 growing season four independent trials in bulb onions (*Allium cepa*) were conducted in Germany to study the potential of concentration residues in dried onion, peeled onions and in peel after processing to these commodities. Dimethomorph was applied two times 28 and 14 days before harvest at 0.54 kg ai/ha which is two times the label rate but one time less than maximum number of applications. Samples of onions bulbs were collected on the day of last application and 7±1 days later. The processing of onions was conducted with samples taken at the last sampling which is 7 days later than in cGAP.

Onion bulbs were washed (3 min) with cold tap water immediately before processing. The green leaves were cut off followed by peeling using a knife. The peeled onions were cut into slices and dried in an oven in cycles from 90 °C 20 minutes, 60 °C 20 minutes and 50 °C until the dry weight of 4–6% was reached. After processing four different fractions on onion products or intermediates were collected for analysis, specifically onion bulbs, dried onions, peeled onions and peel.

Residues of dimethomorph were determined by LC-MS/MS using method No. 575/0. The limit of quantitation was 0.01 mg/kg and the mean recovery rate from 84–99% at fortification levels of 0.01–10 mg/kg in the different matrices. The results are summarized in Table 38..

Table 38 Summary of dimethomorph residues and transfer factors in processed commodities from onion bulb

Portion analysed	DALA	Dimethomorph residues mg/kg				transfer factor (Tf) ^a					
Trial ^b		1 ^b	2 ^b	3 ^b	4 ^b	1	2	3	4	Median Tf	mean Tf best estimate
Onion bulb	0	0.45	0.29	0.14	0.38	—	—	—	—		
Onion bulb	7 ± 1	0.22	0.98	0.30	0.098						
Onion bulb (RAC) ^c	7 ± 1	0.66	0.083	0.25	0.029	1	1	1	1	1	1
Dried onions		0.02	< 0.01 _d	< 0.01 _d	< 0.01 _d	0.03	0.12	0.04	0.34	0.08	0.13
Peeled onions		< 0.01 _d	< 0.01 _d	< 0.01 _d	< 0.01 _d	0.02	0.12	0.04	0.34	0.08	0.13
Peel		2.8	0.88	1.5	0.35	4.24	10.6	6.0	12.1	8.0	8.2

^a Transfer factor = residue in processed fraction (PF)/ residue in RAC^b Trial 1: L090306, Trial 2: L090307, Trial 3: L090308, Trial 4: L090309. These trials are not reported in details here^c At processing start^d For calculation purposes < 0.01 is set 0.01*Lettuce (doc. code 2008/1004853)*

During the growing season 2007, four independent field trials were conducted in Northern France, Germany, Spain and Italy to determine the residue levels of dimethomorph in lettuce head (open and closed head cultivars) and its processed parts. The trials were treated three times with a dose rate of 0.18 kg ai/ha which generally is according to label rates for outdoor lettuce head and leaf, but below (70%) label rates for protected cultivation of lettuce in the Netherlands.

Samples of lettuce heads were collected 0, 7, 14 and 21 days after the last application for analysis of the raw agricultural commodity. The samples for processing were taken 7 days after last treatment which is 7 days later than in cGAP. The processing procedure included dividing lettuce heads into inner and outer leaves and washing of the leaves.

Residues of dimethomorph were determined by LC-MS/MS using method No. 575/0. The limit of quantitation was 0.01 mg/kg and the mean recovery rate from 71–106% at fortification levels of 0.01–20 mg/kg in the different matrices. The result are summarized in Table 39.

Table 39 Summary of dimethomorph residues in processed commodities of lettuce head and transfer factors

Portion analysed	DALA	Dimethomorph residues mg/kg				transfer factor (Tf) ^a					
Trial ^b		1 ^b	2 ^b	3 ^b	4 ^b	1	2	3	4	median Tf best estimate	Mean Tf
Head, RAC	7	0.28	0.15	1.46	0.59	1	1	1	1	1	1
Inner Leaves		0.071	0.012	0.14	0.14	0.25	0.08	0.10	0.24	0.17	0.17
Inner leaves washed		< 0.01 ^c	< 0.01 ^c	0.21	0.063	0.04	0.07	0.14	0.11	0.09	0.09
Outer leaves		0.27	0.28	3.11	1.03	0.96	1.9	2.13	1.75	1.82	1.69
Outer leaves washed		0.083	0.11	1.45	0.39	0.30	0.73	0.99	0.66	0.7	0.67
wash water (inner leaves)		0.016	0.014	0.028	0.025	0.06	0.09	0.02	0.04	0.05	0.05
wash water (outer leaves)		0.21	0.049	0.23	0.23	0.75	0.33	0.16	0.39	0.36	0.41

^a Transfer factor = residue in processed fraction (PF)/ residue in RAC^b Trial 1: L070028 (open/leafy head), Trial 2: L070029 (closed head), Trial 3: L070034 (open/leafy head), Trial 4: L070035 (open/leafy head). These trials are not reported in details here^c For calculation purposes < 0.01 is set 0.01

Pea (doc. code 2007/1044681)

During the 2006 growing season four independent trials were conducted on fresh peas (*Pisum sativum*) in Germany to study the potential of residues its processed products cooked and canned peas. Dimethomorph (WG formulation) was applied as a foliar spray two times 29 and 21 days $\pm$ 1 day prior to harvest, at 0.51–0.53 kg ai/ha which is more than two times the label rate. Samples (plants without pods and whole pods) were collected on the day of last application and 21 $\pm$ 1 days after last application.

The processing of peas was performed with green seeds taken at the last sampling which is according to cGAP. The green seeds were first washed in tap water for three minutes and then boiled in a saucepan covered with water added with salt 2 g/100 mL until the peas were cooked through. For preserving procedure the peas was blanched by staying 2 minutes $<$ 85 °C. After blanching the peas were put in tins covered with 2% NaCL-solution. The tins with the peas were sterilized at 120 °C in an autoclave for 20 minutes. During processing different fractions from pea products or intermediates were collected for analysis, specifically washed peas, cooked peas, blanched seed and canned peas.

Table 40 Summary of dimethomorph residues in processed commodities of peas and transfer factors

Portion analysed	DALA	Dimethomorph residues mg/kg				transfer factor (Tf) ^a				
Trial ^b		1 ^b	2 ^b	3 ^b	4 ^b	1	2	3	4	median Tf
Plants without pods	0	29.067	29.638	20.719	27.746	–	–	–	–	–
Whole pods	0	1.304	2.147	1.521	1.648	–	–	–	–	–
Green seed (RAC) ^c	20–22	0.168	0.042	0.050	0.122	1	1	1	1	1
Washed peas		0.035	0.017	0.046	0.063	0.208	0.405	0.092	0.516	0.307
Wash water		0.111	0.021	0.017	0.048	0.661	0.500	0.340	0.393	0.447
Cooked peas		0.014	$<$ 0.01 _d	0.013	0.021	0.083	0.238	0.260	0.172	0.205
Cooking liquid		0.016	$<$ 0.0 _{d1}	0.012	0.020	0.095	0.238	0.240	0.164	0.201
Blanched seed		$<$ 0.01	0.011	$<$ 0.01 _d	0.021	0.060	0.262	0.200	0.172	0.186
Blanching water		0.021	$<$ 0.01 _d	$<$ 0.01 _d	0.026	0.125	0.238	0.200	0.213	0.207
Canned peas		$<$ 0.01	$<$ 0.01 _d	$<$ 0.01 _d	$<$ 0.01 _d	0.06	0.238	0.200	0.082	0.141/mean 0.145
Vegetable stock		$<$ 0.01	$<$ 0.01 _d	$<$ 0.01 _d	$<$ 0.01 _d	0.06	0.238	0.200	0.082	0.141

^a Transfer factor = residue in processed fraction (PF)/ residue in RAC

^b Trial 1: FR 22/06/40, Trial 2: FR 22/06/70, Trial 3: FR 22/06/50, Trial 4 FR 22/06/60. These trials are not reported in details here

^c At processing start

^d For calculation purposes $<$ 0.01 is set 0.01

Table 41 Calculated processing factors from studies provided for dimethomorph residues in oranges, strawberry, onions, lettuce head and pea.

Processed com	pf	n ^a	STMR-P	HR-P
Orange, juice	0.08	4	0.015	0.072
Oranges, peeled	0.08	4	0.015	0.072
Oranges, marmalade	0.09	4	0.017	0.081
Oranges, oil	0.08	4	0.015	0.072
Orange, peel	1.62		0.308	1.458
Orange peel after oil extraction	2.06	4	0.391	1.854
Oranges, dried pulp	0.22	4	0.042	0.198

Processed com	pf	n ^a	STMR-P	HR-P
Orange pomace, wet	1.38	4	0.262	1.242
Orange pomace, dry	5.64	4	1.072	5.076
Grapes, wine	0.29 ^b	4	0.189	0.87
Raisin	1.8 ^b	4	1.17	5.4
Grape, pomace wet	2.75 ^b	4	1.787	8.25
Strawberry, washed	1.47	4	0.191	1.323
Strawberry, jam before cooking	0.57	4	0.074	0.513
Strawberry, jam after cooking	0.44	4	0.057	0.396
Strawberry, canned	1.11	4	0.632	0.999
Onions, dried	0.13	4	0.009	0.666
Onions, peeled	0.13	4	0.009	0.666
Peel of onions	8.2	4	0.574	4.92
Lettuce head (outdoor), inner leaves	0.17	4	0.165	1.53
Lettuce head (outdoor), inner leaves washed	0.09	4	0.087	0.81
Lettuce head (outdoor), outer leaves washed	0.7	4	0.679	6.3
Peas (<i>Pisum sativum</i>), washed	0.31	4	0.031	0.047

^aNumber

Residues in animal commodities

New residue data on residues of dimethomorph in animal feed have been provided to the 2014 Meeting. A new livestock dietary burden calculation has therefore been performed (Annex 6 to the 2014 Report).

The new estimation did not result in significant change of the dietary burdens of farm animals, please see Appraisal.

APPRAISAL

Dimethomorph is a fungicide with protective action against plant pathogenic *Phytophthora* species and a number of downy mildew diseases of fruit, vegetables and potatoes. It consists of a mixture of an E and Z isomers in approximately equal proportions. Its mode of action is through disruption of fungal cell wall formation.

Dimethomorph was evaluated for the first time by the JMPR in 2007 and the Meeting established an acceptable daily intake (ADI) of 0–0.2 mg/kg bw and an acute reference dose (ARfD) of 0.6 mg/kg bw. The residue (for compliance with the MRL and for the estimation of dietary intake) for plant and animal commodities was defined as dimethomorph (sum of isomers). Maximum residues levels for 20 commodities were proposed by the JMPR in 2007.

The current Meeting received information on supervised residue trial for dimethomorph in oranges, strawberries, grapes, papaya, bulb onions, leek, spring onions, head cabbage, broccoli, pepper, lettuce leaf, spinach, lettuce head, taro, green peas, vining peas, lima beans, artichoke, and celery. An analytical method for determination of dimethomorph in animal matrices were provided as well as validation data for an analytical method in oranges and processing studies in oranges, strawberry, onion, lettuce head, and peas.

Methods of analysis

The 2007 Meeting evaluated methods of analysis for dimethomorph in different plant and animal matrices with a LOQ of 0.01 mg/kg (LC-MS/MS or GC-NDP), LOQ of 0.02 mg/kg (GC-NDP or GC-MS) based on the multi-residue method DFG-S19.

The current Meeting received information on a new analytical method LO138/01 for dimethomorph in animal matrices. In this method dimethomorph is extracted with methanol/water/hydrochloric acid. The final determination of dimethomorph is performed by HPLC-MS/MS at two transitions. Transition m/z 388 → 301 is the target transition for quantification and transition m/z 388 → 165 for confirmatory purposes. The method is suitable for measuring residues of 0.01 mg/kg for dimethomorph in milk, egg, muscle and liver.

Stability of pesticide residues in stored analytical samples

In 2007 the Meeting concluded that dimethomorph is stable (less than 10% loss of residues) under frozen conditions in stored samples in most crops and animal commodities if stored under frozen conditions at 18-24 months and 16 months, respectively.

Results of supervised residue trials on crops

Oranges

Data from supervised trials on oranges from Spain were presented to the Meeting. However, no registered GAP from Spain was available for oranges. As a result no estimation of a maximum residue level was made.

Grapes

Data from supervised trials on grapes from the USA were presented to the Meeting. The registered critical GAP in the USA is four foliar applications of 0.219 kg ai/ha and PHI of 14 days.

In twelve independent residue trials from USA matching the cGAP the residues of dimethomorph in grapes were (n=12): 0.11, 0.26, 0.41, 0.46, 0.49, 0.55, 0.65, 0.71, 0.75, 0.92, 1.77, 1.86 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for dimethomorph in grapes of 3 mg/kg, 0.60 mg/kg and 1.9 mg/kg, respectively. The Meeting replaces its previous recommendation of 2 mg/kg for a maximum residue level for grapes.

Strawberry

Data from supervised trials on strawberries were presented to the Meeting.

The critical GAP is from Belgium (protected and outdoor) with three successive root drench applications of 0.05 g ai/plant and a PHI of 35 days. No trials supporting the GAP were provided.

In Ireland the GAP (protected and outdoor) is one root drench application of 0.05 g ai/plant with a PHI of 35 days. A dataset on protected strawberries was submitted consisting of four trials with replicate plots treated either at 0.0625 g ai/plant or 0.125 g ai/plant and with four additional trials solely treated at 0.125 g ai/plant.

In four GAP compliant plots conducted at 0.0625 g ai/plant residues in strawberry fruits were (n=4): 0.03, 0.18, 0.26, 0.3 mg/kg.

In four additional trials solely conducted at 0.125 g ai/plant residues in strawberry fruits were (n=4): 0.04 (2), 0.05 and 0.21 mg/kg.

Since the four trials provided according to GAP are insufficient for an evaluation of residues in strawberries, the Meeting decided to extend the dataset by applying the proportionality approach. In accordance to the general principles outlined in the 2012 JMPR report, all residue values within and above 25% deviation from GAP were scaled to match the application rate of 0.05 g ai/plant. From

replicated plots conducted at different application rates, the higher scaled residue was selected for the assessment. Scaled residues in strawberry fruits were: 0.024, 0.14, 0.21, 0.24 mg/kg (factor 0.8, based on 0.0625 g ai/plant → 0.05 g ai/plant and 0.016, 0.016, 0.02, 0.084 mg/kg (factor 0.4, based on 0.125 g ai/plant → 0.05 g ai/plant).

The combined scaled dataset is (n=8) 0.016, 0.016, 0.02, 0.024, 0.084, 0.14, 0.21 and 0.24 mg/kg.

Three similar outdoor trials with a PHI of 43 days from Belgium, the Netherlands and Germany were also available. The residues in these trials were for 0.125 g ai/plant (n=3) 0.01, 0.02 and 0.04 mg/kg.

For drip irrigation eight trials from Spain using 2×0.75 kg ai/ha with a PHI of one day were presented to the Meeting. No registered GAP from Spain was available for strawberries.

In the United Kingdom the registered GAP for outdoor is one foliar spray at 1.5 kg ai/ha applied just after planting/transplanting with a PHI of 35 days. Eight trials presented to JMPR 2007 from the Netherlands and four trials from northern Europe presented to the current Meeting match this GAP and could be combined. The residues found in these combined trials are (n=12) < 0.01 mg/kg (9), 0.01, 0.02 and 0.03 mg/kg.

The highest residues came from the protected root drench treatment. Based on the combined scaled dataset from protected root drench trials for strawberries in Ireland, the Meeting estimated a maximum residue level, an STMR value and an HR value for dimethomorph for strawberries of 0.5 mg/kg, 0.05 mg/kg and 0.24 mg/kg, respectively. The Meeting replaces its previous recommendation of 0.05 mg/kg for maximum residue level for strawberry.

Papaya

Data from supervised trials on papaya from Brazil were presented to the Meeting. No registered GAP from Brazil was available for papaya. As a result no maximum residue level estimation was made.

Bulb vegetables

Bulb onion

The Meeting received results from supervised trials with dimethomorph on bulb onions. The critical GAP is for Bulb Vegetables (Garlic, Garlic great headed, Leek, Onion dry bulb, Onion green, Onion Welsh Shallot) in the USA and Canada with three foliar applications of 0.21 kg ai/ha and PHI of 0 days.

Ten independent residue trials from the USA (nine) and Canada (one) were presented on bulb onions matching the cGAP. Residues of dimethomorph in bulb onions were (n=10) 0.06, 0.08, 0.10, 0.12, 0.16, 0.18 (2), 0.23, 0.28 and 0.38 mg/kg. The highest residue of 0.40 mg/kg was measured in individual onion samples.

The Meeting estimated a maximum residue level, an STMR value and an HR value for dimethomorph in bulb onions of 0.6 mg/kg, 0.17 mg/kg and 0.40 mg/kg, respectively. The Meeting also agreed to extrapolate these estimations to shallot and garlic.

Leek

Data from supervised trials on leek were presented to the Meeting. The critical GAP is in France with two foliar applications of 0.18 kg ai/ha and a PHI of 14 days.

Eighteen independent trials from Belgium, Germany, Greece, Italy, France, the Netherlands, Spain, and the UK matching this GAP were presented. The dimethomorph residue from trials on leek was in south EU (n=4) 0.06, 0.08, 0.3 and 0.69 mg/kg and north EU (n=14) 0.01 (2), < 0.02 (2), 0.03, 0.04 (2), 0.07, 0.08 (2), 0.10 (2), 0.11, 0.13.

The combined data set is (n= 18) 0.01 < 0.02 (2), 0.03 0.04 (2), 0.05, 0.06, 0.07, 0.08 (3), 0.10 (2), 0.11, 0.13, 0.30 and 0.69 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for dimethomorph in leek of 0.8 mg/kg, 0.08 mg/kg and 0.69 mg/kg, respectively.

Spring onion

Data from supervised trials on spring onion were presented to the Meeting. The critical GAP is for Bulb Vegetables (Garlic, Garlic great headed, Leek, Onion dry bulb, Onion green, Onion Welsh Shallot) in USA and Canada with three foliar applications of 0.21 kg ai/ha and a PHI of 0 days.

Six independent residue trials from the USA matched the cGAP. Residue of dimethomorph in whole plant were (n=6) 1.27, 1.56, 1.79, 2.35, 2.45 and 5.36 mg/kg. The highest residue of 6.6 mg/kg was measured in individual spring onion samples.

The Meeting estimated a maximum residue level, an STMR value and an HR value for dimethomorph in spring onion of 9 mg/kg, 2.1 mg/kg and 6.6 mg/kg, respectively. The Meeting also agreed to extrapolate this estimation to Onion, Welsh.

Brassica vegetables

Head cabbage

The Meeting received results from supervised trials with dimethomorph on head cabbage. The critical GAP is in USA with three foliar applications of 0.21 kg ai/ha and a PHI of 0 days.

Ten independent residue trials from USA matched the cGAP. Residue from dimethomorph in cabbage heads were (n=10) 0.17, 0.45, 0.46, 0.86, 1.08 (2), 1.22, 1.37 1.51 and 4.26 mg/kg. The highest residue of 4.6 mg/kg was measured in individual head cabbage samples.

The Meeting estimated a maximum residue level, an STMR value and an HR value for dimethomorph in head cabbage of 6 mg/kg, 1.1 mg/kg and 4.6 mg/kg, respectively. The Meeting replaces its previous recommendation of 2 mg/kg for maximum residue level for head cabbage.

Broccoli

Data from supervised trials on broccoli were presented to the Meeting. The critical GAP is from the USA which consists of three foliar applications of 0.21 kg ai/ha and a PHI of 0 days.

Ten independent residue trials performed in USA match the US GAP. Residues for dimethomorph in broccoli were (n=10): 0.25, 0.68, 0.74, 0.90, 0.95, 1.49, 1.62, 1.75, 1.88 and 2.33 mg/kg. The highest residue of 2.6 mg/kg was measured in an individual broccoli sample.

The Meeting estimated a maximum residue level, an STMR value and an HR value for dimethomorph in broccoli of 4 mg/kg, 1.3 mg/kg and 2.6 mg/kg, respectively. The Meeting replaces its previous recommendation of 1 mg/kg for a maximum residue level for broccoli.

Fruiting vegetables, other than cucurbits

Peppers

Data from supervised trials on pepper were presented to the Meeting. The GAP in Canada for Fruiting vegetables (tomato, eggplant, ground cherry, peppers (all varieties), pepino and tomatillo) is five foliar applications of 0.225 kg ai/ha and a PHI of 0 days.

In eleven outdoor independent residue trials in USA matching the Canadian GAP the residues of dimethomorph in peppers were (n= 11): 0.04, 0.06, 0.08, 0.11, 0.13(2), 0.14, 0.25, 0.7, 0.84 and 1.05 mg/kg.

Tomato

Data from supervised trials on tomato were presented to the 2007 Meeting. The critical GAP is from Canada for Fruiting vegetable with five foliar applications of 0.225 kg ai/ha and PHI of 0 day.

In twelve outdoor independent residue trials on tomato from the USA matching the cGAP the residue of dimethomorph in tomato fruit were (n= 12): 0.05(2), 0.06(2), 0.07, 0.11, 0.14, 0.21, 0.3, and 0.38, 0.41 and 0.51 mg/kg.

The Meeting noted that the GAP in Canada was for fruiting vegetables, other than cucurbits, the medians of the data sets for peppers and tomatoes differed by less than 5-fold and that the residue populations were statistically similar. The Meeting therefore decided to consider recommending a crop group maximum residue level. The combined data set matching the Canadian GAP is (n= 23) 0.04, 0.05(2) 0.06(3), 0.07, 0.08, 0.11(2), 0.13(2), 0.14(2), 0.21, 0.25, 0.30, 0.38, 0.41, 0.51 0.70, 0.84 and 1.05 mg/kg. The highest residue of 1.2 mg/kg was measured in an individual pepper sample.

The Meeting estimated a group maximum residue level, an STMR value and an HR value for dimethomorph in fruiting vegetables, other than cucurbits except mushrooms and sweet corn of 1.5 mg/kg, 0.13 mg/kg and 1.2 mg/kg, respectively.

The Meeting withdraws its previous recommendation of 1 mg/kg for fruiting vegetable, other than cucurbits except mushrooms and sweet corn.

*Leafy vegetables**Lettuce, Head*

Data from supervised field trials on head lettuce were presented to the Meeting. The critical GAP is from USA for Leafy vegetables, except brassica vegetables, consisting of three foliar applications at 0.19 kg/ha and a PHI of 0 days.

In fourteen independent trials from the USA matching the cGAP the residues from dimethomorph in head lettuce were (n= 14): 1.08, 1.21, 1.36, 1.42, 1.46, 1.68, 1.72, 2.06, 2.3, 2.82, 3.63, 4.1, 4.37 and 6.45 mg/kg.

The Meeting noted that the US trials reported by the 2007 JMPR resulted in higher residues. The Meeting therefore, confirmed the previous recommendations made by the 2007 JMPR.

Lettuce, Leaf

Data from supervised trials on leaf lettuce were presented to the Meeting. The critical GAP is from USA for Leafy vegetables, except brassica vegetables with three foliar applications 0.19 kg/ha and a PHI of 0 days.

In nine independent trials from the USA matching the cGAP the residues from dimethomorph in lettuce leaf were (n= 9) 2.09, 3.37, 3.68, 4.61, 5.19, 5.38, 5.83, 9.77 and 9.88 mg/kg. The highest residue of 10.5 mg/kg was measured in an individual lettuce sample.

The Meeting estimated a maximum residue level, an STMR value and a HR value for dimethomorph in leaf lettuce of 20 mg/kg, 5.2 mg/kg and 10.5 mg/kg.

Short-term intake assessment showed that residues in leaf lettuce exceeded the acute reference dose of 0.6 mg/kg bw by 110% for children.

Spinach

Data from supervised trials on spinach were presented to the Meeting. The critical GAP is from USA for Leafy vegetables, except brassica vegetables with three foliar applications 0.19 kg/ha and a PHI of 0 days.

In eight independent trials from the USA matching the cGAP the residues from dimethomorph in spinach leaves were (n= 8) 4.69, 5.30, 5.91, 8.21, 8.35, 8.48, 10.18 and 11.26 mg/kg. The highest residue of 11.5 mg/kg was measured in individual spinach samples.

The Meeting estimated a maximum residue level, an STMR value and an HR value for dimethomorph in spinach of 30 mg/kg, 8.3 mg/kg and 11.5 mg/kg, respectively.

Taro leaves

Data from supervised trials on taro leaves from USA were presented to the Meeting. The critical GAP is five foliar applications of 0.19 kg ai/ha and PHI 7 days for use in USA except California.

Dimethomorph was applied seven times to taro at the rate of 0.225 kg ai/ha with an interval of 7–8 days. Presented residue trials from head lettuce, leaf lettuce and spinach show that residues of dimethomorph decline significantly three days after application. The Meeting, therefore, concluded that the first two applications would not contribute significantly to the residues in leaves at harvest.

In three independent trials from USA matching the cGAP residues in taro leaves were (n=3): 1.44, 1.64 and 4.53 mg/kg. The highest residue of 5.4 mg/kg was measured in an individual taro sample.

The Meeting estimated a maximum residue level, an STMR value and an HR value for dimethomorph in taro leaves of 10 mg/kg, 1.64 mg/kg and 5.4 mg/kg, respectively.

Legume vegetable

Peas, shelled (succulent seed)

Data on supervised trials on peas were presented to the Meeting. The critical GAP is from France with two foliar applications at 0.18 kg ai/ha and a PHI of 21 days.

In twelve independent trials from north (eight) and south (four) Europe matching the cGAP residues of dimethomorph in fresh peas without pods were (n= 12) < 0.01 (8), 0.016, 0.044 and 0.063 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for dimethomorph in peas without pods of 0.15 mg/kg, of 0.01 mg/kg and 0.063 mg/kg, respectively.

Lima bean

Data from supervised trials on Lima bean were presented to the Meeting. The critical GAP is from the USA with five applications at 0.19 kg ai/ha and a PHI of 0 days.

Ten independent trials matching the cGAP were conducted on Lima bean in USA. Residues of dimethomorph in beans (succulent seed without pods) (n=10) were 0.01, 0.03(4), 0.05, 0.06, 0.08, 0.1, 0.21 and 0.47 mg/kg. The highest residue of 0.48 mg/kg was measured in an individual Lima bean sample.

The Meeting estimated a maximum residue level, an STMR value and an HR value for dimethomorph in Lima bean of 0.7 mg/kg, 0.055 mg/kg and 0.48 mg/kg, respectively.

Root and tuber vegetables

Ginseng

Data from supervised trials on ginseng were presented to the Meeting. The critical GAP from the USA, except California, is five applications of 0.19 kg ai/ha and a PHI of 14 days. Four trials were presented where dimethomorph was applied seven times at 0.225 kg ai/ha. Residue decline trials were not provided for any root and tuber vegetable to support that the first two applications did not contribute to the residues at harvest. Consequently, no maximum residue level estimation was made.

Taro root

Data on supervised trials on taro corms from Hawaii were presented to the Meeting. The critical GAP is five applications of 0.19 kg ai/ha and a PHI of 30 days in the USA, except California. Three trials were presented where dimethomorph was applied seven times at 0.225 kg ai/ha. Residue decline trials were not presented for any root and tuber vegetable to support that the first two applications not contribute to the residues at harvest. Consequently, no maximum residue level estimation was made.

*Stalk and stem vegetables**Globe artichoke*

Data from supervised trials on globe artichoke were presented to the Meeting. The critical GAP from France is three foliar applications of 0.18 kg ai/ha and PHI of 3 days.

In ten independent trials from Europe matching the French GAP residues of dimethomorph in artichoke heads were in North Europe (n=5) 0.11, 0.24, 0.26, 0.55 and 0.75 mg/kg and in South EU (n=5) 0.06, 0.09, 0.14, 0.32 and 1.14 mg/kg.

The combined data set was (n=10): 0.06, 0.09, 0.11, 0.14, 0.24, 0.26, 0.32, 0.55, 0.75 and 1.14 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for dimethomorph in globe artichoke of 2 mg/kg, 0.25 mg/kg and 1.14 mg/kg, respectively.

Celery

Data from supervised trials on celery were presented to the Meeting. The critical GAP is from the USA with three foliar applications of 0.21 g ai/ha and a PHI of 0 days.

In nine independent trials from Canada (two) and from USA (seven) matching the cGAP residues of dimethomorph in leaf stalks were (n=9): 1.27, 1.55, 1.85, 1.91, 2.44, 3.27, 4.02, 5.54 and 8.21 mg/kg.

The highest residue of 8.8 mg/kg was measured from an individual celery sample.

The Meeting estimated a maximum residue level, an STMR value and an HR value for dimethomorph in celery of 15 mg/kg, 2.44 mg/kg and 8.8 mg/kg, respectively.

*Animal feeds**Pea forage*

Data on supervised trials on peas were presented to the Meeting. The critical GAP is from France with two foliar applications at 0.18 kg ai/ha and a PHI of 21 days. However, the residues (10) 28 days after the second treatment were found to be higher and were used for animal burden calculation: 0.42, 0.59, 1.57, 1.59, 1.88, 2.40, 4.60, 4.97, 5.45 and 9.58 mg/kg.

The Meeting estimated in pea forage a median residue of 2.14 mg/kg (fresh weight) and a highest residue of 9.58 mg/kg (fresh weight).

Fate of residue during processing

The Meeting received information on processing of oranges, strawberries, onions, lettuce head and peas.

Processing factors calculated for the processed commodities for the above raw agricultural commodities, including previously estimated, are shown in the table below. STMP-Ps was calculated for processed commodities of strawberry, onion and peas for which maximum residue levels were estimated.

Processed commodity	Processing factor	PF (Best estimate)	STMR-P	HR-P
Strawberry, jam	0.24, 0.43, 0.44, 0.54	0.435	0.02	
Strawberry, canned	0.57, 1, 1.21, 1.52	1.11	0.0555	
Onions, raw without skin	0.02, 0.04, 0.12, 0.34	0.08	0.014	0.032
Dried onion	0.03, 0.12, 0.04, 0.34	0.13	0.022	0.053
Peas (cooked)	0.08, 0.17, 0.24, 0.26	0.21	0.002	
Peas (canned)	0.06, 0.08, 0.20, 0.24	0.14	0.0014	

*estimated by 2007JMPR Meeting

** PF =processing factor

^b Data from a 5-week special neurotoxicity study, using a limited number of dogs.

The Meeting confirmed its previous maximum residue level estimation of 5 mg/kg for dried grapes.

Residues in animal commodities

Farm animal dietary burden

Dietary burden calculation for beef cattle, dairy cattle, broilers and laying poultry based on feed items evaluated by JMPR in 2007 and 2014 are provided in table below. The calculations were made according to the livestock diets from US-Canada, EU, Australia and Japan according to OECD feeding table. Noting that fresh forage commodities are not significant in international trade, the Meeting only included the burden contributions from the pea forages in the European dietary burden calculation, as dimethomorph is not authorised for use on peas in US-Canada, Australia or Japan.

Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are presented in Annex 6 and are summarised below.

Estimated maximum and mean dietary burden of farm animals Summary (ppm of dry matter diet)

	US-Canada		EU		Australia		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	0.4	0.3	14.2 ^a	3.6 ^c	2.6	2.6	0.007	0.007
Dairy cow	0.1	0.1	14.1 ^b	3.5 ^d	2.6	2.6	0.01	0.01
Poultry-broiler	0.002	0.002	0.06	0.05	0.04	0.04	0.002	0.002
Poultry layer	0.002	0.002	5.4 ^{e g}	1.27 ^{f h}	0.04	0.04	0.007	0.007

^a Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian tissues

^b Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk

^c Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

^d Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

^e Highest maximum poultry dietary burden suitable for MRL estimates for poultry tissues.

^f Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues.

^g Highest maximum poultry dietary burden suitable for MRL estimates for poultry eggs.

^h Highest mean poultry dietary burden suitable for STMR estimates for poultry eggs

For beef and dairy cattle, the calculated maximum dietary burdens suitable for estimating maximum residue levels in mammalian tissues and milk are 14.2 and 14.1 ppm dry weight of feed respectively.

The calculated mean dietary burden, suitable for estimating STMRs in mammalian tissues and in milk is 3.6 and 3.5 ppm, dry weight of feed, respectively.

In the cattle feeding study evaluated by JMPR in 2007 where lactating cows were dosed at 37.5 ppm (approximately 40% higher than estimated maximum burden) no residues of parent dimethomorph were detected in edible tissue or milk. Therefore the Meeting concluded that no residues are to be expected at the maximum calculated dietary burden for ruminants.

The calculated maximum dietary burden suitable for estimating maximum residue levels in poultry tissues and eggs is 5.4 ppm dry weight of feed and the calculated mean dietary burden, suitable for estimating STMRs in poultry tissues and in eggs is 1 ppm dry weight of feed.

In the metabolism study where laying hens were fed the equivalent of 40 ppm in the feed for seven days, dimethomorph residues in fat and skin were < 0.02 mg/kg and were not detected in tissue or eggs. On the basis that the maximum calculated dietary burden is eight times lower than the dose rate in the metabolism study the Meeting concluded that no residues of dimethomorph are to be expected at the maximum calculated dietary burden for poultry.

The Meeting confirmed the previous recommendations for animal commodities.

RECOMMENDATIONS

On the basis of the data from supervised residue trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for the IEDI and IESTI assessment.

Definition of the residue (for compliance with the MRL and for estimation of dietary intake) for plant and animal commodities: dimethomorph (sum of isomers).

The residue is not fat soluble.

MRL recommendations and dietary intake

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
VS 0620	Artichoke, Globe	2		0.25	1.14
VB 0400	Broccoli	4	1	1.3	2.6
VB 0041	Cabbages, Head	6	2	1.1	4.6
VS 0624	Celery	15		2.44	8.8
DF 0269	Dried grapes (= currants, Raisins and Sultanas)	5	5	1.17	3.4
VO 0050	Fruiting vegetables, other than Cucurbits	1.5	1	0.13	1.2
VA 0381	Garlic	0.6		0.17	0.4
FB 0269	Grapes	3	2	0.60	1.9
VA 0384	Leek	0.8		0.08	0.69
VL 0482	Lettuce, Head	10	10	3.6 ^a	7.2 ^a
VL 0483	Lettuce, Leaf	20		5.19	10.5
VP 0534	Lima bean (young pods and/or immature beans)	0.7		0.055	0.48
VA 0385	Onion, Bulb	0.6		0.17	0.4
VA 0387	Onion, Welsh	9		2.1	6.6
VP 0064	Peas, shelled (succulent seeds)	0.15		0.01	0.63
VA 0388	Shallots	0.6		0.17	0.4
VL 0502	Spinach	30		8.3	11.5
VA 0389	Spring onion	9		2.1	6.6
FB 0275	Strawberry	0.5	0.05	0.02	0.24
VL 0505	Taro leaves	10		1.64	5.4
	Dried onion			0.022	0.053
	Grapes, wine			0.18	

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
	Onions, raw without skin			0.014	0.03
	Peas (canned)			0.0014	
	Peas (cooked)			0.002	
	Strawberry jam			0.02	
	Strawberry, canned			0.056	
JF 0448	Tomato juice			0.065	
VW 0448	Tomato paste			0.31	2.88

^a recommendations from the 2007 Meeting

Additional values used to calculate the livestock animal dietary burden

CCN	Commodity name	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
AL 0528	Pea vines (green)	2.14	9.58
	Grape pomace wet	1.95 ^a	
	Potato process, waste	0.12 ^a	
	Potato culls	0.02 ^a	0.05 ^a
	Rape meal	0.04 ^a	

^a recommendations from the 2007 Meeting

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDI) of dimethomorph, based on the STMRs estimated for 30 commodities, for the 17 cluster diets were in the range of 0–2% of maximum ADI (0.2 mg/kg bw), see Annex 3. The Meeting concluded that the long-term intake of residues of dimethomorph resulting from its uses that have been considered by JMPR is unlikely to present a health concern.

Short-term intake

The WHO Panel of the 2007 JMPR established an Acute Reference Dose (ARfD) of 0.6 mg/kg bw for dimethomorph.

The International Estimated Short Intake (IESTI) for dimethomorph was calculated for new food commodities and their processed fractions for which maximum residue levels were estimated and for which consumption data were available, see Annex 4.

For lettuce leaf, the IESTI represented 110% of the ARfD of 0.6 mg/kg bw. On the basis of the information provided to the JMPR it was not possible to conclude that the estimate of the short-term intake of dimethomorph, from the consumption of lettuce leaf, was less than the ARfD. The Meeting noted that an alternative GAP for lettuce leaf was not available.

For the other commodities the IESTI for dimethomorph calculated on the basis of recommendations made by the JMPR represented 0-90% of the ARfD (0.6 mg/kg bw) for children and 0-30% for the general population.

The Meeting concluded that except for lettuce leaf, the short-term intake of residues of dimethomorph, when used in ways that have been considered by the JMPR is unlikely to present a public concern.

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2001/5003306	Corley, J	2001 d	Dimethomorph: Magnitude of the residue on lettuce (head) IR-4 (Interregional Research Project No.4.) North Brunswick NJ. USA. Unpublished.
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Code	Author	Year	Title, Institute, Report reference
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2013/1335899	Corley, J	2013 a	Dimethomorph: Magnitude of the residue on bean (lima) IR-4 Project Headquarters Princeton NJ. USA. Unpublished.
2007/1068765	Corley, J	2007 d	Dimethomorph: Magnitude of the residue on ginseng. Center for Minor Crop Pest Management, North Brunswick NJ. USA. Unpublished.
2008/1068911	Perny, A	2008 c	Study on the residue behavior of Dimethomorph and Pyraclostrobin in artichokes after treatment with BAS 536 01 F under field conditions in Northern and Southern France, Germany, Italy and the Netherlands, 2006. Anadiag SA, Haguenau France. Unpublished.
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2009/1051339	Strobl, M	2009 a	Validation of BASF method L0138/01-Method of the determination of Dimethomorph (BAS 550F) in animal matrices. BASF SE, Limburgerhof, Germany Fed Rep. Unpublished
2009/1078429	Wolf, S	2009 a	Independent laboratory validation (ILV) of an analytical method for the determination of Dimethomorph (BAS 550 F) in animal tissues (milk, egg, muscle and liver). Harlan Laboratories Ltd., Itingen, Switzerland. Unpublished

DITHIOCARBAMATES (105) - MANCOZEB (050)

The first draft was prepared by Professor Mi-Gyung Lee, Andong National University, Republic of Korea

EXPLANATION

Mancozeb was evaluated in 1993 in the CCPR periodic review programme. Since then, in 2012, the Meeting evaluated residue data on mancozeb and maneb, however, made no recommendation for MRLs. Currently, a number of MRLs for dithiocarbamates are established based on the residue data derived from use of mancozeb, maneb, metiram, thiram, ziram and propineb.

The 1993 JMPR evaluated several dithiocarbamate fungicides for toxicology and residues when the Meeting established an ADI of 0–0.03 mg/kg bw (group or in any combination) for ethylene-bis-dithiocarbamates (EBDCs: mancozeb, maneb, metiram and zineb). In addition, for their metabolite ethylenethiourea (ETU), the Meeting established an ADI of 0–0.004 mg/kg bw. At present, there are no ARfDs established by the JMPR for ethylene-bis-dithiocarbamates or ETU.

The mancozeb residue is defined as total dithiocarbamates, determined as CS₂, evolved during acid digestion and expressed as mg CS₂/kg, for compliance with MRLs in plant and animal commodities. Dithiocarbamate residues are not fat soluble.

Dithiocarbamates-Mancozeb was listed by the Forty-fifth Session of the CCPR (2013) for the evaluation of additional MRL in 2014 JMPR. Further, it was requested by the Session that 2014 JMPR evaluate monitoring data on spices produced by India for additional MRLs. The Meeting received supervised residue trial data from Thailand (chili pepper) and the Republic of Korea (ginseng and the processed products). From India, monitoring data on spices (cardamom, coriander, cumin, fennel and black pepper) were submitted.

RESIDUE ANALYSIS*Analytical methods*

The analytical method used for determination of dithiocarbamates in chili pepper was based on the Dutch manual of analytical methods (Ministry of Welfare, Health and Cultural Affairs, 1988), which was reviewed in 1995 by JMPR: dithiocarbamates are converted to CS₂ by treatment with hydrochloric acid in the presence of stannous chloride. The CS₂ in the head-space is determined by GC with either ECD or FPD. For analysis of dithiocarbamates in chili pepper, ECD was used. The limit of quantification was 0.05 mg/kg as CS₂. Recovery tests were performed at five levels of 0.05–5.0 mg CS₂/kg (n=3 at each level). The average recoveries were in the range of 71–101%. In procedural recovery tests conducted at a fortified level of 0.1 mg CS₂/kg, the average recoveries (n=3) were 81–100% with an acceptable RSDs. ETU residue was not analysed.

Information on an analytical method ABY0064 with a validation study was submitted, for the analysis of mancozeb in ginseng and the processed products. This method is based on the direct determination, by HPLC-MS/MS, of a derivatized EBDC (EBDC-dimethyl produced by S-methylation). In the mass analysis, individual EBDCs are distinguished using compound-specific transition ions.

The validation study was performed with orange, olive and their products. An aliquot of extracting solvent and iodomethane were added to sample (2 g). The extracting solvent was a mixture of disodium EDTA (ethylenediaminetetraacetic acid), NaOH, L-cysteine, water and methanol. The sample extract was cleaned up using C₁₈ SPE cartridge with an eluting solvent of water:methanol (40:60, v:v). Residues were quantified with by HPLC-MS/MS (MRM *m/z* 241 > 134). The responses of the HPLC/MS/MS systems were linear over the range 0.1 to 5 ng/mL (*r*², > 0.99). At the fortification levels of 0.01 and 0.1 mg/kg of mancozeb, individual recoveries were in the range of 70% to 108% (RSD, < 15%). The limit of quantification was 0.01 mg/kg in all matrices (Table 1).

In the validation study, additionally, a method for ETU analysis was tested for its performance. The method involved extraction with dichloromethane, clean-up by liquid-liquid partition with hexane and quantification by HPLC-MS/MS (MRM m/z 103 > 44 for ETU; MRM m/z 107 > 48 for internal standard, ETU D4). The linearity was over the range 2.5 to 100 ng/mL. At the fortification levels of 0.01 and 0.1 mg/kg of ETU, individual recoveries were in the range of 79% and 109% (CV, < 12%). The limit of quantification was 0.01 mg/kg in the all matrices (Table 2).

The control sample extracts from all matrices (oranges, olives and their by-products) were fortified with mancozeb (as the methylated derivative) or ETU, the residues in the final extracts were stable for seven days stored at approximately -20 °C. The storage period was sufficient to cover the maximum storage period of the extracts from this validation study.

Table 1 Recoveries of mancozeb in orange, olive and the processed products using the method ABY0064

Matrix	Fortification, mg/kg	No.	Range of recoveries, %	Mean recovery, %	CV, %
Orange pulp	0.01	5	73-98	82	12.5
	0.1	5	71-76	73	2.6
Orange peel	0.01	5	96-101	97	2.3
	0.1	5	96-104	101	3.6
Orange juice	0.01	5	85-95	90	4.4
	0.1	5	75-78	77	2.0
Orange oil	0.01	5	97-103	94	14.7
	0.1	5	98-108	101	4.4
Orange wet pomace	0.01	5	80-100	90	7.9
	0.1	5	80-93	88	5.9
Orange dry pomace	0.01	5	70-84	74	7.8
	0.1	5	71-81	76	6.8
Olive oil	0.01	5	72-85	78	7.0
	0.1	5	70-86	78	9.6

Mancozeb was not detected (< 0.01 mg/kg) in control samples.

Table 2 Recoveries of ETU from oranges, olives and the processed by-products

Matrix	Fortification, mg/kg	No.	Range of recoveries, %	Mean recovery, %	CV, %
Orange whole fruit	0.01	5	79-98	88	9.5
	0.1	5	94-106	100	4.4
Orange pulp	0.01	5	94-107	103	5.0
	0.1	5	97-107	102	3.7
Orange peel	0.01	5	88-104	97	7.0
	0.1	5	89-95	92	2.4
Orange juice	0.01	5	80-103	89	12.4
	0.1	5	96-109	104	5.0
Orange oil	0.01	5	88-93	89	5.4
	0.1	5	80-92	87	5.4
Orange wet pomace	0.01	5	91-100	96	3.5
	0.1	5	99-104	102	2.3
Orange dry pomace	0.01	5	90-109	98	7.7
	0.1	5	100-106	103	2.5
Olive whole fruit	0.01	5	79-109	98	12.3
	0.1	5	95-108	103	5.4

Matrix	Fortification, mg/kg	No.	Range of recoveries, %	Mean recovery, %	CV, %
Olive oil	0.01	5	85-94	91	4.7
	0.1	5	89-98	94	3.7

ETU was not detected (< 0.01 mg/kg) in control samples.

Analyses of mancozeb and ETU in ginseng and the processed products were conducted by the analytical methods mentioned above, except minor modifications in certain trials, e.g., use of HPL SPE instead of C18 SPE or without involvement of internal standard in ETU analysis. LOQ of mancozeb in fresh ginseng by HPLC-MS/MS was 0.01 mg/kg and 0.02 mg/kg for the other matrices (dried ginseng, red ginseng, water extract of dried ginseng and water extract of red ginseng). For ETU, the LOQs were 0.01mg/kg or 0.02 mg/kg in fresh ginseng and 0.02 mg/kg in dried and red ginseng. In the ginseng matrices (fresh and processed), individual procedural recoveries (n=3 or 5) for mancozeb and ETU were between 70% and 120% (RSD, <20%) at the various fortification levels (0.01–1.0 mg/kg in mancozeb; 0.02–1.0 mg/kg in ETU).

In the analysis of dithiocarbamates in spices employed in monitoring, spectrometry was used. The samples (10–20 g) were hydrolysed with hydrochloric acid and stannous chloride solution at elevated temperature, and the evolved carbon disulphide (CS₂) passed through two gas washing tubes in series containing lead acetate and sodium hydroxide solutions and an absorption tube containing an ethanolic solution of cupric acetate and diethanol amine. Formed two cupric complexes of N,N-bis (2-hydroxyethyl) dithiocarbamic acid were measured by spectrometry at 453 nm (Kepple G.E. J AOAC 54, 528-532, 1971). LOQs of dithiocarbamates in spices (cardamom, coriander, cumin, fennel and pepper) were 0.1 mg CS₂/kg. At fortification levels of 0.1–1.0 mg CS₂/kg, recoveries were 90–110% in cumin, coriander and fennel and 88-107% in cardamom and black pepper.

Stability of residues in stored analytical samples

In ginseng trials, fortified control samples were concurrently treated with the field trial samples during storage and analysis. The ginseng samples (fresh and processed) fortified with mancozeb and ETU were stored together with the field trial samples at -20 °C or below until analysis. Recoveries of mancozeb in fresh ginseng were 81–92% (0.1 mg/kg, n=5) at 56 days or 86–94% (0.1–0.5 mg/kg, n=6) at 103 days. In the processed ginseng, recoveries of mancozeb were 81–117% (0.2 mg/kg, n=20 for all processed types) at 11–56 days or 78–94% (0.2–1.0 mg/kg, n=6 for dried ginseng) at 100 days. For ETU, the recoveries in fresh ginseng were 84–92% (0.2 mg/kg, n=5) at 71 days or 85–96% (0.1–0.5 mg/kg, n=6) at 103 days; in the processed, 83–96% (0.2 mg/kg, n=10 for dried and red ginseng) at 55–62 days or 90–96% (0.2–1.0 mg/kg, n=6 for dried ginseng) at 100 days. The days refer to mean storage days of fortified samples and covered the actual storage days of the samples, except for two cases in fresh ginseng, analysis of mancozeb (max. actual storage 76 days over test 56 days,) and analysis of ETU (max. actual storage 92 days over test 71 days). Based on the results, it is considered that the residues were stable during the study period.

As the analysis of mancozeb residues in chili peppers was undertaken on the day of harvest, storage stability tests were not conducted.

USE PATTERN

Mancozeb is a protective fungicide effective against a wide range of foliar fungal diseases. The registered uses on ginseng, chili pepper, cumin and black pepper are summarized in Table 3.

Table 3 Registered uses of mancozeb submitted for evaluation

Crop	Country	Formulation	Application					PHI, days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hl	No.	Interval days	
Chili pepper	Thailand	80% WP	Foliar spraying	2.0 ^a	0.4	3	5	7
Ginseng	Rep. of Korea	75% WP	Foliar spraying	2.4 ^b	0.12	5	10	45

Location Year (Variety)	Application					DAT, days	Residue ^a , mg/kg as CS ₂	Report No.
	kg ai/ha	n	Interval days	L/ha	kg ai/hL			
						9	1.2	
						12	1.0	
						15	0.46	
Ratchaburi 2005 (n.r.)	2.0	3	7	637	0.32	0	2.3	MA-CH-03
						1	1.6	
						3	1.3	
						5	1.0	
						7	<u>0.80</u>	
						9	0.69	
						12	0.21	
						15	0.083	
Kanchanaburi 2005 (n.r.)	2.0	3	7	635	0.32	0	3.2	MA-CH-04
						1	2.3	
						3	1.4	
						5	1.1	
						7	<u>0.75</u>	
						9	0.49	
						12	0.21	
						15	0.13	
Ratchaburi 2007 (n.r.)	2.1	3	7	643	0.32	0	1.1	CY-CH-05
						1	0.88	
						3	0.68	
						5	0.31	
						7	<u>0.31</u>	
						9	0.20	
						12	0.28	
						15	0.22	
Rachaburi 2007 (n.r.)	2.0	3	7-8	614	0.32	0	0.61	
						1	1.5	
						3	0.79	
						5	0.70	
						7	<u>0.48</u>	
						9	0.20	
						12	0.23	
						15	0.17	

Chili pepper: Capsicum annuum Linn. var. *acuminatum* Fingerh

WP 80% formulation was used.

Portion analysed: whole fruit without stem

^a The residue value is an average of analytical results for three harvested samples.

Root and tuber vegetables

Ginseng

Four residue trials were conducted in the Republic of Korea in 2013. Mancozeb WP 75% formulation was applied five times (10 day intervals) to four-year old ginseng as a foliar application. Three trials of which were decline trials. The concentration in spray solution was 0.12 kg ai/hL and a spray volume of 2,000 L/ha was used. The calculated dose rate of each application was 12-13 kg ai/ha. Ginseng was collected at 45 days (0, 25, 35, 45, 55 days in the decline trials) after the last application. Foliage and stem were removed before shipment to an analytical laboratory. After removing the soil particles on ginseng roots by washing with tap water, it was blended and stored at -20 °C or -60 °C until analysis. The mass of sample was more than 2 kg.

The residues from application of mancozeb formulation were analysed for both mancozeb and ETU. In control samples, no residue was not detected (< LQO). Ginseng processing studies were also conducted. These are described in the following section.

Table 5 Mancozeb and ETU residues in ginseng from supervised trials performed in the Republic of Korea

Location Year (Variety) ^a	Application ^b				DAT, days	Residue ^c , mg/kg		Study No. or ID
	kg ai/ha	n	Interval days	kg ai/hL		Mancozeb	ETU	
GAP		5	10	0.12	45			
Yeongju 2013	2.4-2.6	5	10	0.12	45	0.05	0.01	MFDS13162FS009
Geumsan 2013	2.4-2.5	5	10	0.12	0 ^e	0.19	0.04	MFDS13162FS009
					25	0.09	0.04	
					35	0.07	0.08	
					45	0.07	0.04	
					55	0.11	0.04	
Sunheung ^d 2013	2.5	5	10	0.12	0 ^e	0.03	< 0.01	CRI-2013-C-1
					25	0.02	< 0.01	
					35	0.05	< 0.01	
					45 ^f	0.02	< 0.01	
					45	0.02	< 0.01	
					55	0.02	< 0.01	
Punggi ^d 2013 CRI-2013-C-1	2.5	5	10	0.12	0 ^e	0.03	< 0.01	CRI-2013-C-1
					25	0.02	< 0.01	
					35	0.03	< 0.01	
					45 ^{e,f}	0.05	< 0.01	
					45	0.03	< 0.01	
					55	0.02	< 0.01	

^a *Panax ginseng* C.A. Mey (Violet-stem variant)

^b WP 75% formulation was used.

^c The residue value is an average of analytical results for three harvested samples.

^d The two trials were not independent. These were conducted in an adjacent location of 8 km distance and by the same application method and on the same days.

^e In the trial conducted in Sunheung, analysis of control sample was performed only at 0 day; while for trial conducted in Punggi only at 0 and 45 days.

^f At 45 days, duplicate samples were collected.

Spices

Spices (cardamom, coriander, cumin, fennel and black pepper) were collected from the retail outlets during 2009–2014 in India. About 20–500 g of sample of each spice was collected and out of which 10–20 g of homogenized sample was taken for the analysis.

Table 6 Dithiocarbamate residues in spices monitored in India during 2009-2014

Spice		No. of samples	Residues (mg CS ₂ /kg)		
Group	Name		Median	Max	Detections
Seed	Cumin	384	3.6	17	0.11, 0.12, 0.14, 0.15, 0.26, 0.27, 0.29 (2), 0.31, 0.36, 0.41, 0.46, 0.53, 0.54, 0.55, 0.60 (2), 0.61, 0.63, 0.64, 0.65 (2), 0.69, 0.71, 0.73, 0.74 0.75, 0.76 (2), 0.78 (2), 0.79, 0.81(2), 0.82(2), 0.83 (2), 0.85, 0.88, 0.90 (2), 0.92, 0.93, 0.94, 0.97 (2), 0.98, 1.00, 1.02, 1.04 (2), 1.05, 1.07 (2), 1.08, 1.11, 1.14, 1.19, 1.20, 1.23, 1.25, 1.27, 1.28, 1.30, 1.33, 1.35, 1.37, 1.41, 1.43 1.45, 1.48, 1.50 (2), 1.51, 1.53, 1.57, 1.60, 1.69, 1.70 (2), 1.71, 1.79, 1.80, 1.83, 1.87, 1.92, 1.96, 1.97 (2), 1.98, 2.01 (2), 2.02, 2.05, 2.06, 2.09, 2.10, 2.13 (2), 2.15, 2.16,

Spice		No. of samples	Residues (mg CS ₂ /kg)		
Group	Name		Median	Max	Detections
					2.20 (2), 2.23, 2.35, 2.36, 2.39, 2.40, 2.42 (3), 2.44 (2), 2.47, 2.55, 2.62, 2.66 (2), 2.67 (4), 2.70, 2.72, 2.73, 2.74, 2.76 (3), 2.79, 2.80, 2.88 (4), 2.89, 2.90 (3), 2.93 (2), 2.94, 2.95, 2.99 (2), 3.00, 3.01, 3.02, 3.04 (3), 3.07, 3.09, 3.10 (2), 3.14, 3.18, 3.20 (2), 3.23, 3.29, 3.30 (2), 3.32 (3), 3.33 (2), 3.36 (4), 3.40 (4), 3.42, 3.44, 3.47, 3.49 (2), 3.50 (3), 3.52, 3.55, 3.59, 3.60 (2), 3.61, 3.62, 3.67, 3.68, 3.73, 3.80 (3), 3.82, 3.84, 3.89, 3.92, 3.98, 4.01, 4.03, 4.10 (2), 4.13 (2), 4.16, 4.20, 4.34, 4.36, 4.38, 4.48, 4.63, 4.64, 4.67, 4.68, 4.75, 4.80 (3), 4.85, 4.90, 4.91, 5.00, 5.09, 5.10, 5.11, 5.15, 5.20 (2), 5.22, 5.30, 5.38, 5.40 (2), 5.56, 5.60 (3), 5.62, 5.70 (2), 5.72, 5.78, 5.80, 6.01 (2), 6.05, 6.07, 6.10 (2), 6.11, 6.14, 6.16, 6.20 (6), 6.27, 6.30 (4), 6.32, 6.38 (2), 6.40 (2), 6.41, 6.43, 6.44, 6.50 (7), 6.60 (2), 6.65 (4), 6.70, 6.80 (3), 6.82, 6.84, 6.88, 6.90 (3), 6.91, 6.94, 6.96, 7.04, 7.10 (3), 7.20 (10), 7.30 (2), 7.31 (2), 7.40 (4), 7.50 (2), 7.59 (2), 7.60 (5), 7.70, 7.77, 7.84, 7.90 (2), 8.01, 8.03, 8.10, 8.13, 8.20 (3), 8.30 (2), 8.40 (3), 8.41, 8.57, 8.60 (2), 8.70 (2), 8.90 (3), 9.04, 9.10, 9.28, 9.35, 9.39, 9.40 (2), 9.43 (2), 9.88 (2), 9.98, 10.20, 10.69 (2), 11.38, 11.44, 12.53, 13.17, 13.32, 14.54, 15.47, 16.88, 17.20
Fruit or berry	Cardamom	1,037	< 0.1	< 0.1	0
Seeds	Coriander	248	< 0.1	0.4	0.4
Fruit or berry	Black pepper	272	< 0.1	< 0.1	0
Seeds	Fennel	286	2.0	2.8	1.06, 2.01, 2.83

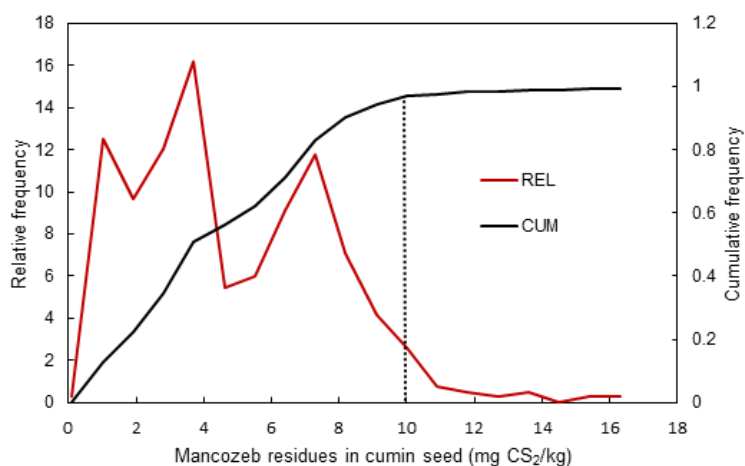


Figure 1 Distribution of mancozeb residues in cumin seed (n=383)

Dotted line indicates that the residue value (10 mg CS₂/kg) covers at least 99th percentile of residues.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

The Meeting received information on the fate of mancozeb residues during the processing of ginseng.

Ginseng

Four-year old ginseng cultivated under shaded conditions was treated five times with a 75% WP formulation at a spray concentration of 0.12 kg ai/hL (12–13 kg ai/ha per application) with 10 day treatment intervals. The fresh ginseng was collected 45 days or decline days (0, 25, 35, 45, and 55 days) after the last application. The harvested ginseng was treated with tap water to remove the soil particles and then dried.

To produce dried ginseng, the washed fresh ginseng was air-dried at 60 °C. Red ginseng was prepared by steaming fresh ginseng for three hours at 98 °C and then by air-drying at 65 °C and further drying under the sunlight. The dried or red ginseng were cut and extracted three times by refluxing in water at 85 °C. The extract was concentrated to 72° Brix. Dried ginseng, red ginseng and the water extracts were packed in inert plastic bottles and stored at -20 °C until analysis.

Water contents in fresh ginseng, dried ginseng, red ginseng, water extract of dried ginseng and water extract of red ginseng were 71%, 8%, 9%, 47% and 52%, respectively.

Table 7 Mancozeb and ETU residues in processing ginseng products

Location Year (Variety)	Application, WP 75%			PHI, days	Sample analysed	Residues, mg/kg		Pf		Study No. or ID
	kg ai/ha	n	kg ai/hL			Mancozeb	ETU	Mancozeb	ETU	
GAP		5	0.12	45						
Yeongju 2013	2.4-2.6	5	0.12	45	Fresh ginseng	0.05	0.01			
					Dried ginseng	0.14	0.05	2.8	1.75	MFDS1316 2FS009
					Red ginseng	0.08	0.12	1.6	4.2	
					Water extract of dried ginseng	< 0.02	n.a.	< 0.4		
					Water extract of red ginseng	< 0.02	n.a.	< 0.4		
Sunheung 2013	2.5	5	0.12	0	Fresh ginseng	0.03	< 0.01			
					Dried ginseng	0.04	< 0.02	1.3		CRI-2013- C-1
				25	Fresh ginseng	0.02	< 0.01			
					Dried ginseng	0.04	< 0.02	2.0		
				35	Fresh ginseng	0.05	< 0.01			
					Dried ginseng	0.06	< 0.02	1.2		
				45	Fresh ginseng	0.02	< 0.01			
					Dried ginseng	0.04, 0.04	< 0.02	2.0		
				55	Fresh ginseng	0.02	< 0.01			
					Dried ginseng	0.04	< 0.02	2.0		
Punggi 2013	2.5	5	0.12	0	Fresh ginseng	0.03	< 0.01			CRI-2013- C-1
					Dried ginseng	0.04	< 0.02	1.3		
				25	Fresh ginseng	0.02	< 0.01			
					Dried ginseng	< 0.02	< 0.02	<1.0		
				35	Fresh ginseng	0.03	< 0.01			
					Dried ginseng	0.04	< 0.02	1.3		
				45	Fresh ginseng	0.04	< 0.01			
					Dried ginseng	0.04, 0.07	< 0.02	1.4		
				55	Fresh ginseng	0.02	< 0.01			
					Dried ginseng	0.04	< 0.02	2.0		

Processing studies were performed once in the Yeongju location and three times in Sunheung and Punggi location each. Pre-processing samples were not analysed. Residue values for fresh ginseng were taken from field trial data.

n.a.: not analysed

Ginseng variety: Panax ginseng C.A. Mey (Violet-stem variant)

Processing factors were calculated:

For mancozeb, the concentration of mancozeb in dried ginseng was divided by the concentration of mancozeb in fresh ginseng.

For ETU, the concentration of ETU in dried ginseng was divided by the sum of ETU and mancozeb (expressed as ETU equivalents by multiplying 0.378 of conversion factor) in fresh ginseng in order to account for mancozeb as a potential source of ETU during processing.

Table 8 Summary of processing factors for mancozeb in ginseng (RAC)

Processed commodity	Calculated processing factors		Pf, best estimate	
	Mancozeb	ETU	Mancozeb	ETU
Dried ginseng	< 1.0, 1.2, 1.3, 1.3, 1.3, 1.4, 2.0, 2.0, 2.0, 2.0, 2.8	1.75		
Red ginseng	1.6	4.2		
Ginseng, dried including red ginseng	< 1.0, 1.2, 1.3, 1.3, 1.3, <u>1.4</u> , <u>1.6</u> , 2.0, 2.0, 2.0, 2.0, 2.8	1.75, 4.2	1.5	4.2
Water extract of dried ginseng	< 0.4			
Water extract of red ginseng	< 0.4			
Ginseng, extracts	< 0.4, < 0.4		< 0.4	

APPRAISAL

Mancozeb was evaluated in 1993 in the CCPR periodic review programme. Since then, in 2012, the Meeting evaluated residue data on mancozeb and maneb, however, made no recommendation for MRLs. Currently, a number of MRLs for dithiocarbamates are established based on the residue data derived from use of mancozeb, maneb, metiram, thiram, ziram and propineb.

Dithiocarbamates-Mancozeb was listed by the Forty-fifth Session of the CCPR (2013) for the evaluation of additional MRL in 2014 JMPR. The Meeting received supervised residue trial data from Thailand (chili pepper) and the Republic of Korea (ginseng and the processed products). In addition, India submitted monitoring data on spices (cardamom, coriander, cumin, fennel and black pepper).

The mancozeb residue is defined as total dithiocarbamates, determined as CS₂, evolved during acid digestion and expressed as mg CS₂/kg, for compliance with MRLs. Dithiocarbamate residues are not fat soluble.

In 1993, the JMPR established a group (or in any combination) ADI of 0–0.03 mg/kg bw for ethylene-bis-dithiocarbamates (EBDCs: mancozeb, maneb, metiram and zineb) and an ADI of 0–0.004 mg/kg bw for their metabolite ethylenethiourea (ETU). The parent EBDC and ETU are defined as the residues for evaluating dietary intake. The Meeting is assessing combined residues of mancozeb and ETU using the ratio of the ADIs (7.5) to express residues in terms of mancozeb-toxicity-equivalents (MTE).

Methods of analysis

The analytical method used for the determination of dithiocarbamates in chili pepper was considered by the JMPR in 1995. According to the method, dithiocarbamate residues in chili pepper were converted to CS₂ by treatment with hydrochloric acid in the presence of stannous chloride. The CS₂ in the head-space was determined by GC-ECD and the limit of quantification was 0.05 mg CS₂/kg. The validity of the analytical results was supported by a set of recovery test and procedural recoveries.

Mancozeb and ETU in ginseng and its processed products were directly measured using analytical method ABY0064.

The method ABY0064 is based on the direct determination, by HPLC-MS/MS, of a derivatized EBDC (EBDC-dimethyl produced by S-methylation). In the method, disodium EDTA and iodomethane are used for decomposing and methylating EBDC compounds, and then the extracts are cleaned up with C₁₈ SPE cartridge. In the mass analysis, individual EBDCs are distinguished using compound-specific transition ions.

The method ABY0064 was fully validated with analytical matrices of orange, olive and their processed products. The recoveries of mancozeb were within an acceptable range (70–120%) at fortification levels of 0.01 mg/kg and 0.1 mg/kg. The LOQs were 0.01 mg/kg. In ginseng residue trials, procedural recoveries were acceptable and the LOQs were 0.01 mg/kg or 0.02 mg/kg.

A method developed for determination of ETU was fully validated for the same matrices used in a validation study of ABY0064. The recoveries and RSDs were within an acceptable range at fortification levels of 0.01 mg/kg and 0.1 mg/kg and the LOQs were 0.01 mg/kg. In ginseng residue trials, procedural recoveries were acceptable and the LOQs were 0.01 mg/kg.

Spectrometry was used for the analysis of dithiocarbamates in spices. Dithiocarbamate residues in spices were converted to CS₂ and two cupric complexes of N,N-bis (2-hydroxyethyl) dithiocarbamic acid, which were measured at 453 nm. LOQs of dithiocarbamates in spices (cardamom, coriander, cumin, fennel and pepper) were 0.1 mg/kg. At fortification levels of 0.1–1.0 mg CS₂/kg, recoveries were 88–110% in cumin, coriander, fennel, cardamom and black pepper.

Stability of residues in stored analytical samples

Storage stability tests for ginseng samples were performed simultaneously with freezer storage of field trial samples or processed samples. Mancozeb and ETU residues were stable during the study period, 103 days until analysis of mancozeb and ETU for fresh ginseng and 11–100 days (mancozeb analysis) or 55–100 days (ETU analysis) for the processed products.

In residue trials of chili pepper, analysis of mancozeb, as CS₂, was conducted on the day the samples were harvested.

Results of supervised trials on crops

The MRLs for mancozeb are expressed as CS₂. For trials using the headspace method, residues are reported in terms of CS₂. For trials using the HPLC-MS/MS method, residues are reported as mancozeb and ETU. For those trials, maximum residue estimates are made by converting mancozeb to CS₂-equivalents.

To estimate dietary intakes, residues are expressed in terms of mancozeb toxicity-equivalents (MTE). The conversion factor for ETU to MTE is the ratio of the ADI for mancozeb to that of ETU, which is 7.5. Thus, when residues were measured as mancozeb and ETU, the total MTE was estimated by multiplying the ETU residue by 7.5 and adding the result to the measured mancozeb residue. The resulting ETU-equivalent was then converted to MTE using the 7.5 factor. The molecular weights of these compounds are CS₂=76.1 g/mol, ETU=102.2 g/mol, and mancozeb=541.0 g/mol, assuming that the 2 moles of ETU are formed from one mole of mancozeb, leading to the following conversion factors:

$$\text{CS}_2 \text{ mancozeb equivalent: } 541.045 / (4 \times 76.139) = 1.777 \times \text{CS}_2 \text{ mg/kg}$$

$$\text{Mancozeb MTE equivalent is: mancozeb mg/kg (measured as mancozeb)}$$

$$\text{MTE for ETU} = 7.5 \times \text{ETU mg/kg}$$

$$\text{MTE for combined residues of mancozeb measured as CS}_2 \text{ mg/kg and ETU mg/kg:}$$

$$\text{MTE}_{(\text{MCZ}+\text{ETU})} = 1.777 \times \text{CS}_2 \text{ mg/kg} + 7.5 \times \text{ETU mg/kg}$$

*Fruiting vegetables, other than Cucurbits**Peppers, Chili*

Mancozeb is registered in Thailand for use on chili peppers at a GAP of 3×0.4 kg ai/hL, with 5 day intervals and a PHI of 7 days. A total of six trials were conducted in Thailand in 2005–2008, matching the GAP.

The residues, as CS₂ were (n=6): 0.31, 0.48, 0.75, 0.80, 1.2 and 1.7 mg CS₂/kg.

The Meeting estimated a maximum residue level of 3 mg CS₂/kg, an STMR of 1.4 mg MTE/kg and an HR of 3.0 mg MTE/kg for chili pepper. Using the default factor of 7 for dried chili pepper, the Meeting estimated a maximum residue level of 20 mg CS₂/kg, a STMR-P of 9.8 mg MET/kg and an HR-P of 21 mg MET/kg for dried chili pepper.

*Root and tuber vegetables**Ginseng*

Mancozeb is registered in the Republic of Korea for use on ginseng at a GAP of 5×0.12 kg ai/hL, with 10 day intervals and a PHI of 45 days. Four trials matching the GAP conducted in the Republic of Korea in 2013 were submitted. Two trials were conducted on the same dates of application in sites closely located and with the same application method; therefore, the trials are not considered to be independent. Only three trials could be considered for estimation of a maximum residue level.

The measured concentrations of mancozeb in ginseng were (n=3): 0.05, 0.05 and 0.11 mg/kg, which is equivalent to 0.028, 0.028 and 0.062 mg/kg as CS₂.

The measured concentrations of ETU in ginseng were (n=3): < 0.01, 0.01 and 0.04 mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg CS₂/kg.

For dietary intake, the STMR is 0.125 mg MTE/kg and the HR is 0.41 mg MTE/kg.

Results of monitoring studies on spices

Monitoring of spices was conducted for dithiocarbamates in India during 2009–2014. Residues in spices came from the use of mancozeb and other dithiocarbamates. Spice samples were analysed based on the determination of CS₂.

The numbers of samples analysed were: 383 for cumin seed, 1,037 for cardamom, 248 for coriander seed, 272 for black pepper and 286 for fennel seed.

In all of the cardamom (1,037 samples) and black pepper (272 samples), CS₂ residues were < 0.1 mg/kg.

In coriander seed, CS₂ residues were < 0.1 (247), 0.4 mg CS₂/kg.

In fennel seed, CS₂ residues were < 0.1 (283), 1.1, 2.0 and 2.8 mg CS₂/kg.

In cumin seed (383 samples), CS₂ residues ranged from 0.11 mg/kg to 17 mg CS₂/kg, with a median of 3.6 mg CS₂/kg.

For compliance, the Meeting estimated maximum residue levels of 0.1 mg CS₂/kg for black pepper, cardamom, coriander seed, and fennel seed, and 10 mg CS₂/kg for cumin seed. The Meeting noted that the maximum residue estimate covers at least 98% of the observed residues.

For dietary intake, the Meeting estimated STMRs of < 0.18 mg MTE/kg for black pepper, cardamom, coriander seed, and fennel seed, and 6.4 mg MTE/kg for cumin seed.

Fate of residues during processing

The Meeting received information on the fate of mancozeb residues during the processing of ginseng. Conversion of residues to their CS₂, mancozeb, and/or ETU equivalents was done, as needed, as described above for supervised residue trials.

RAC and processed	Pf, best estimate		STMRRAC		Median-P		HRRAC		Highest residue-P	
	mancozeb	ETU*	mancozeb (mg/ kg)	ETU (mg/kg)	mancozeb (mg /kg)	ETU equiv. (mg/kg)	mancozeb (mg/ kg)	ETU (mg/kg)	mancozeb (mg /kg)	ETU equiv. (mg/kg)
Ginseng			0.05	0.01			0.11	0.04		
Ginseng, dried including red ginseng	1.5	4.2			0.075	0.1197			0.17	0.3389

* Fraction yield of ETU = $[ETU]_{proc}/([ETU]_{rac} + 0.378 \times [mancozeb]_{rac})$

In dried ginseng, each processing factor for mancozeb, ETU and CS₂ residue was calculated as follows:

For mancozeb, the concentration of mancozeb in dried ginseng was divided by the concentration of mancozeb in fresh ginseng.

For ETU, the concentration of ETU in dried ginseng was divided by the sum of ETU and mancozeb (expressed as ETU equivalents) in fresh ginseng in order to account for mancozeb as a potential source of ETU during processing. The stoichiometric conversion factor for mancozeb to ETU is 0.377 (102.2 g ETU/mol ÷ 271.2 g mancozeb/mol).

For compliance, the Meeting estimated a maximum residue level of 0.3 mg CS₂/kg, based on a maximum residue level of 0.15 mg CS₂/kg for ginseng and the processing factor of dried ginseng, 1.5 (1.5×0.15=0.225 mg/kg).

For dietary intake, the Meeting estimated an STMR-P of 0.075+7.5×0.1197=0.97 mg MTE/kg and an HR-P of 0.17+7.5×0.33894=2.71 mg MTE/kg.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

The mancozeb residue is defined as total dithiocarbamates, determined as CS₂, evolved during acid digestion and expressed as mg CS₂/kg, for compliance with MRLs in plant and animal commodities.

For estimation of dietary intake in plant and animal commodities, the residue definition is mancozeb and ETU.

Dithiocarbamate residues are not fat soluble.

DIETARY RISK ASSESSMENT

Long-term intake

A group ADI (or in any combination) for ethylene-bis-dithiocarbamates (EBDCs: mancozeb, maneb, metiram and zineb) is 0–0.03 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for mancozeb were estimated based on the 17 GEMS/Food Consumption Cluster Diets using only the STMR or STMR-P values estimated by the current JMPR. The results are shown in Annex 3 of the 2014 JMPR Report. The IEDIs ranged 0-6% of the maximum ADI. The Meeting concluded that the

long-term intake of residues of mancozeb from uses added by the current JMPR is unlikely to present a public health concern.

Short-term intake

The ARfD for mancozeb and its metabolite ETU is not available currently. The Meeting noted that the dithiocarbamates were last evaluated in 1993 before the ARfD established by the JMPR.

REFERENCES

Author	Study No./ID or Report No.	Year	Title, Institute
Kyung, K. S. and Kim, T. H.	MFDS13d162 FS009	2013	Residues of Mancozeb and Its Metabolite ETU in Ginsengs and their Processing Products, Korea Chungbuk National University and Analysis Technology and Tomorrow, LTD Non - GLP. Unpublished
Lee, Y C.	CRI-2013-C-1	2013	Residues of Mancozeb and ETU in Ginseng from Korea, Korea Croen Research Non - GLP. Unpublished
Stephen Brewin	ABY0064	2010	Mancozeb and ETU: Validation of Methodology for the Determination of Residues in Orange and Olive Processed Commodities Huntingdon Life Sciences GLP. Unpublished
PANIDA CHAIYANBON	MA-CH-01-06	2014	Residue data: Mancozeb for peppers, chili Ministry of Agriculture and Cooperatives, Thailand

EMAMECTIN BENZOATE (247)

First draft prepared by Ms T. van der Velde-Koerts, Centre for Nutrition, Prevention and Health Services (VPZ), National Institute for Public Health and the Environment (RIVM), The Netherlands

EXPLANATION

Emamectin benzoate is a foliar insecticide derivative of abamectin, a naturally occurring soil actinomycete. Emamectin benzoate was first evaluated by the JMPR in 2011 for toxicology and residues. The 2011 Meeting established an ADI of 0–0.0005 mg/kg bw and an acute RfD of 0.03 mg/kg bw, expressed as emamectin benzoate. The 2011 Meeting defined the residue as emamectin B1a benzoate for plant and animal commodities for compliance with the MRL and for estimation of dietary intake. Since the molecular weight difference between emamectin B1a benzoate and emamectin benzoate (consisting of 90% emamectin B1a benzoate and 10% emamectin B1b benzoate) is marginal, residues are not corrected for molecular weight. The 2011 Meeting considered the residue not fat soluble.

Emamectin benzoate was scheduled at the 45th session of the CCPR (2013) for the evaluation of additional maximum residue levels by the 2014 JMPR. The manufacturer submitted additional supervised residue trials on rape forage, almonds, pecans and rape seeds, which were evaluated by the present Meeting.

RESIDUE ANALYSIS

The Meeting received information on analytical methods for the determination of emamectin benzoate and its avermectin-like metabolites as used in the supervised residue trials. The analytical residue methods have been evaluated according to the guidance provided by OECD (Series on Pesticides number 39) as indicated on page 25 of the FAO manual 2009.

Validation results are required for every commodity submitted for MRL-setting: at least one full validation for a commodity within the five defined crop groups (high acid content, high water content, high oil content, high protein content, high starch content) and a reduced validation for every other commodity within a certain crop group. Where validation results do not meet the criteria given below, this is indicated.

When the analytical method is validated according to a full validation scheme, it means that

- at least 5 recovery experiments per level were conducted on at least 2 levels (LOQ and 10× LOQ) and average recovery per level was shown to be between 70–120% and the relative standard deviation (RSD_r or CV) per level was shown to be < 20%,
- at least two control samples were analysed and were shown to be below 0.3×LOQ and
- the calibration was conducted with at least 5 single points or at least 3 duplicate points and was shown to be linear (either standards in solvent or matrix matched standards).

When the analytical method is validated according to a reduced validation scheme, it means that

- a full validation is available for a crop in the same crop group (high acid content, high water content, high oil content, high protein content, high starch content);
- at least 3 recovery experiments per level were conducted on at least 2 levels (LOQ and 10× LOQ) and the average recovery per level was shown to be between 70–120% and the relative standard deviation (RSD_r or CV) per level was < 20%;
- at least two control samples were analysed and shown to be below 0.3× LOQ
- the calibration was conducted with at least 5 single points or at least 3 duplicate points and was shown to be linear (only relevant for matrix matched standards; standards in solvent are already covered by full validation).

Analytical methods used in study reports in plant commodities

HPLC-MS/MS method RAM 465/01

HPLC-MS/MS Method RAM 465/01 is used for the determination of emamectin benzoate and its avermectin-like residues in almond and pecan nutmeat and almond hulls. Description and validation of RAM 465/01 and its modifications has previously been submitted for the JMPR 2011 evaluation. A modification of the method was issued on 20 March 2007 for almonds and pecans as described in the JMPR 2011 evaluation report [Ediger, 2007, MK244/0714]. The 30 May 2008 modification used in the 2008 supervised residue trials on almonds and pecans [Oakes, 2009, MK244/50014] is the same as the 20 March 2007 modification.

Additional validation results are available from the 2008 supervised residue trials [Oakes, 2009, MK244/50014]. Since all analyses were performed in the same laboratory, the present reviewer combined the recovery results from identical commodities to get within-laboratory reproducibility and recovery results. Reduced validation results are available at 0.001 and 0.005 mg/kg (n=3–4, each level) in almond nutmeat and pecan nutmeat and at 0.001 mg/kg (n=3, each level) in almond hulls for MAB1a (NOA 426007), MAB1b (NOA 422390), 8,9-ZMa (NOA 438376), AB1a (NOA 438309), MFB1a (NOA 415692) and FAB1a (NOA 415693). Limited validation results are available at 0.05–0.1 mg/kg in almond hulls (n=2, each level). Average recoveries for these validations ranged between 70–120% with an RSD < 20% for reduced validations. The only exception is 0.001 mg/kg FAB1a in almond nutmeat where the RSD = 25%. Residues of the analytes were below LOQ (i.e. < 0.001 mg/kg) when measured in untreated control samples of almond nutmeat, pecan nutmeat or almond hulls. Linearity of the response was assessed using 5 single standards in solvent. The response was shown to be linear (1/× weighted) across the range 0.005–0.1 µg/L or 0.05–20 ng/L for ionisable analytes and 0.02–2.0 µg/L for neutral analytes. The correlation coefficient (r) for the calibration regression lines was greater than 0.999 in all cases.

The 20 March 2007 modification of HPLC-MS/MS method RAM 465/01 is valid for quantification of emamectin benzoate (B1a and B1b) and its avermectin-like residues at levels between 0.001–0.005 mg/kg in almond nutmeat and pecan nutmeat and at 0.001–0.1 mg/kg in almond hulls. The only exception is 0.001 mg/kg FAB1a (NOA 415693) in almond nutmeat where the RSD = 25% (n=3). Since this compound is not in the residue definition for enforcement or dietary risk assessment, this has no impact on the MRL derivation.

HPLC-MS/MS method TP/03

Method TP/03 is used for the determination of emamectin benzoate and its isomer 8,9-ZMa in rape seeds and rape forage.

Method TP/03 involves extraction of residues of emamectin B1a (NOA 426007), emamectin B1b (NOA 422390) and 8,9-ZMa (NOA 438376) from rape seeds and rape forage [Balshaw and Fernandez, not dated]. Homogenised sample (5 or 10 g) is mixed with acetonitrile acidified to 1% with acetic acid. Sodium chloride and magnesium sulphate are added and mixed vigorously followed by centrifugation. Where the sample to solvent ratio is not 1:1 a concentration step is included using a nitrogen evaporator. The acetonitrile layer is cleaned up with a primary secondary amine cartridge and magnesium sulphate. The extract is then analysed using HPLC-MS/MS using external standards for emamectin benzoate and 8,9-ZMa. For emamectin B1a benzoate the MRM transition ions were m/z 886.6 to m/z 158 (quantification) and m/z 126 and 302 for confirmation. For emamectin B1b benzoate the MRM transitions were m/z 872.5 to m/z 158 (quantification) and m/z 82 and 126 for confirmation. For 8,9-ZMa the MRM transitions were m/z 890.6 to m/z 305 (quantification) and m/z 193 and 567 for confirmation.

Validation results for rape seeds and rape forage are reported in the supervised residue trials [Lean, 2014a/b, MK244/10298, MK244/10300] as well as a separate validation report [Balshaw and Fernandez, not dated]. Full validation results are available for rape seeds at 0.005 and 0.05 mg/kg

(n=6, each level) for emamectin benzoate (B1a and B1b). Reduced validation results are available at 0.05 mg/kg (n=3) for 8,9-Zma in rape seeds and at 0.05 mg/kg (n=3) for emamectin benzoate (B1a and B1b) and 8,9-Zma in rape forage. Limited validation results are available at 0.005 mg/kg (n=2) for 8,9-Zma in rape seeds and rape forage and for emamectin benzoate (B1a and B1b) in rape forage. Limited validation results are available at 0.1 mg/kg (n=1) for emamectin benzoate (B1a and B1b) and 8,9-Zma in rape forage. Average recoveries for these validations ranged between 70%-120% with an RSD < 20% for full or reduced validations. Residues of the analytes were below 0.3LOQ (i.e. < 0.002 mg/kg) when measured in untreated control samples of rape seeds and rape forage. The linearity of response was verified for 5–7 single standards in solvent. The linear range was 0.005 to 0.20 mg/kg for 1/× weighted regression and forced origin with a coefficient of determination (R^2) > 0.99 for each analyte.

HPLC-MS/MS method TP/03 is considered valid for quantification of emamectin benzoate (B1a and B1b) at levels between 0.005–0.05 mg/kg in rape seeds and rape forage. The quantification of 8,9-Zma at levels of 0.005–0.05 mg/kg in rape seeds and rape forage is considered insufficiently validated. Since 8,9-Zma is not in the residue definition for enforcement or dietary risk assessment, this has no impact on the MRL derivation.

Stability of pesticide residues in stored analytical samples

Emamectin benzoate is stable in commodities with high oil content such as nutmeat and canola grain for a minimum of 9 months and for a minimum of 2 years in high water content crops and dry crops under deep-frozen conditions. The reports of all relevant storage stability studies have previously been submitted [JMPR 2011 evaluation].

USE PATTERN

Emamectin benzoate is registered for use in several countries for control of insects on several commodities as is indicated in the JMPR 2011 evaluation. Additional label information on tree nuts and rape seed was submitted to the present Meeting and is presented in table 1.

Table 1 lists only the uses for which an original label was available and the dose rate could be verified by the Meeting. The label information on tree nuts is the same as for the JMPR 2011 evaluation and this information is repeated here for convenience. The labels on rape seed and tree nuts indicate that thorough spray coverage is essential for optimum performance. The use of greater water volumes generally results in better coverage, especially in hot and dry weather conditions or when the plant canopy is dense. The use of an adjuvant typically improves coverage and penetration and results in optimum insect control, especially in crops with hard-to-wet leaf surfaces.

Table 1 Registered pre-harvest uses of emamectin benzoate, relevant for the present evaluation

Crop	Country	Formul g ai/kg or g ai/L	Method	Rate per applic (g ai/ha)	Spray concentr (g ai/hL)	Number	Spray interval (days)	PHI, days
Tree nuts ^a including pistachios	USA	SG 50 ^c	concentrate airblast foliar spray ground equipment	11.2-16.8	3.0-4.5	1-3, max 50.4 g ai/ha per season	7	14
	USA	SG 50 ^c	dilute airblast foliar spray ground equipment	11.2-16.8	0.30-0.45	1-3, max 50.4 g ai/ha per season	7	14
Rape seed (canola) for	Australia	EC 17 ^c	foliar spray ground	2.6-5.1	2.6-5.1	1-2	^d	14

Crop	Country	Formul g ai/kg or g ai/L	Method	Rate per applic (g ai/ha)	Spray concentr (g ai/hL)	Number	Spray interval (days)	PHI, days
seed production ^b			equipment					
	Australia	EC 17 ^c	foliar aircraft spray	2.6-5.1	13-26	1-2	^d	14

SG = water soluble granule, EC = emulsifiable concentrate

^a The tree nut class in the USA includes: almond, beechnut, Brazil nut, butternut, cashew, chestnut, chinquapin, filbert (hazelnut), hickory nut, macadamia nut (bush nut), pecan, walnut (black and English/Persian).

^b The Australian label on rape seed contains a restriction for use as feed: "do not use on canola grown as forage crop and also the intended use table indicates: canola for grain production"

^c The use of a penetrating type spray adjuvant (tree nuts) or a non-ionic surfactant (rape seed, tree nuts) is recommended. Do not use sticker/binder type adjuvant for tree nuts.

^d Sample crops twice a week after application to determine if a second application is required.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received supervised residue trials on rape forage, almonds, pecans and rape seed.

Crop group	Commodity	Table no.
Tree nuts	Almonds	Table 2
	Pecans	Table 3
	Rape seed	Table 4
Oilseeds	Almond hulls	Table 5
	Rape forage	Table 6

Application rates are reported as emamectin benzoate (g ai/ha). Unquantifiable residues are shown as below the reported LOQ (e.g., < 0.001 mg/kg). Residues, application rates and spray concentrations have been rounded to two significant figures. Residue data are recorded unadjusted for percentage recoveries or for residue values in control samples unless otherwise stated. Where multiple samples were taken from a single plot individual values are reported, but the mean value is selected for MRL derivation, if according to cGAP. Where multiple analyses were conducted on a single sample, the average value is reported. Where results from separate plots with distinguishing characteristics such as different formulations, crop varieties or treatment schedules were reported, results are listed for each plot. However in such cases only one residue value may be selected per location. Residues from the trials conducted according to critical GAP have been used for the estimation of maximum residue levels, STMR and HR values. Those results are underlined.

Tree nuts

The Meeting received supervised residue trials on almonds and pecans.

Supervised residue field trials on almonds and pecans were carried out in the USA in 2006 and 2007 [Ediger 2007, MK244/0714, Oakes, 2009, MK244/50014]. The 2006 trials were summarized and evaluated by the 2011 JMPR. The 2007 trials are summarized here. Almond and pecan trees were treated with three foliar cover spray applications using a tractor mounted sprayer or airblast sprayer. The spray volume was either 93–95 L/ha (very concentrate spray as used for aircraft equipment), 450–780 L/ha (concentrate spray as used for airblast sprayers) or 1300–2300 L/ha (dilute spray as used for ground equipment). The spray mix included a horticultural oil, non-ionic surfactant or organo-silicone adjuvant. There were no unusual weather conditions. Whole nuts were harvested at maturity. Samples were prepared by separating shells from nutmeat to generate at least 1.4 kg of nutmeat (=RAC). Samples were stored at -20 °C for a maximum of 9 months. Samples were analysed for MAB1a (NOA 426007), MAB1b (NOA 422390), 8,9-Zma (NOA 438376), AB1a (NOA 438309), MFB1a (NOA 415692) and FAB1a (NOA 415693) by HPLC-MS/MS Method RAM 465/01

(modification 30 May 2008). Results were not corrected for control levels (< 0.001 mg/kg for each analyte) nor for average concurrent method recoveries (70%-120% for each analyte). Results are shown in table 2 and table 3. Results represent the mean of two replicate samples taken from each plot.

Residues of avermectin-like residues were not found in any of the almond or pecan nutmeat samples (< 0.001 mg/kg). Several trials have been conducted on the same location. From such trials only the highest (mean) value is selected for MRL derivation if compliant with cGAP.

Table 2 Residue results from supervised field trials on almonds (nutmeat) using an SG formulation (50 g ai/kg) with adjuvant

ALMONDS Location, year, (Variety)	Number (interval) soil type	g ai/ha	g ai/hL	Method, last application date, growth stage (BBCH)	DAT (days)	MAB1a (mg/kg)	MAB1b (mg/kg)	Sum1a (mg/kg)	Report, Trial no
Madera, CA, USA, 2007 (Butte)	3 (7, 7), sandy loam	17 17 17	18 18 18	very concentrate foliar spray 27 Aug, BBCH 85	14	< 0.001 < 0.001 mean <u>< 0.001</u> ^a	< 0.001 < 0.001 mean < 0.001 ^a	< 0.001 < 0.001 mean < 0.001 ^a	Report: T002811-07 Trial: W30CA078291
Madera, CA, USA, 2007, (Mission)	3 (7, 7) loamy sand	17 17 17	2.2 2.2 2.2	concentrate foliar spray 29 Aug, BBCH 85	14	< 0.001 < 0.001 mean < 0.001 ^a	< 0.001 < 0.001 mean < 0.001 ^a	< 0.001 < 0.001 mean < 0.001 ^a	Report: T002811-07 Trial: W29CA078294
Madera, CA, USA, 2007, (Mission)	3 (7, 7) loamy sand	17 17 17	0.90 0.90 0.90	dilute foliar spray 29 Aug, BBCH 85	14	< 0.001 < 0.001 mean < 0.001 ^a	< 0.001 < 0.001 mean < 0.001 ^a	< 0.001 < 0.001 mean < 0.001 ^a	Report: T002811-07 Trial: W29CA078294
Fresno, CA, USA, 2007 (Non-Pareil)	3 (7, 7) sandy loam	16 17 17	1.0 1.0 1.0	dilute foliar spray 27 Aug, BBCH 85	14	< 0.001 < 0.001 mean <u>< 0.001</u> ^a	< 0.001 < 0.001 mean < 0.001 ^a	< 0.001 < 0.001 mean < 0.001 ^a	Report: T002811-07 Trial: W30CA078292
Kerman, CA, USA, 2007, (Carmel)	3 (7, 7) loamy sand	17 17 17	2.4 2.4 2.4	concentrate foliar spray 27 Aug, BBCH 85	14	< 0.001 < 0.001 mean <u>< 0.001</u> ^a	< 0.001 < 0.001 mean < 0.001 ^a	< 0.001 < 0.001 mean < 0.001 ^a	Report: T002811-07 Trial: W30CA078293

^a Results are from two replicate field samples, the mean may be selected for MRL derivation if compliant with cGAP.

Sum1a expressed as MAB1a = sum of MAB1a plus its avermectin-like metabolites, corrected for molecular weight (MAB1a + 1.000 × 8,9-Zma + 1.016 × AB1a + 0.9693 × MFB1a + 0.9844 FAB1a). Metabolites < LOQ were assumed not to be present

Table 3 Residue results from supervised field trials on pecans (nutmeat) using an SG formulation (50 g ai/kg) with adjuvant

PECANS Location, year, (Variety)	Number (interval) soil type	g ai/ha	g ai/hL	Method, last application date, growth stage (BBCH)	DAT (days)	MAB1a (mg/kg)	MAB1b (mg/kg)	Sum1a (mg/kg)	Report, Trial no
Hawkinsville, GA, USA, 2007, (Desirable)	3 (7, 6) sandy clay loam	17 17 17	18 18 18	concentrate foliar spray 25 Oct, BBCH 83	14	< 0.001 < 0.001 mean <u>< 0.001</u> ^a	< 0.001 < 0.001 mean < 0.001	< 0.001 < 0.001 mean < 0.001	Report: T002811-07 Trial: E12GA078295

PECANS Location, year, (Variety)	Number (interval) soil type	g ai/ha	g ai/hL	Method, last application date, growth stage (BBCH)	DAT (days)	MAB1a (mg/kg)	MAB1b (mg/kg)	Sum1a (mg/kg)	Report, Trial no
							^a	^a	
Vienna, GA, USA, 2007, (Desirable)	3 (7, 6) sandy loam	17 17 17	1.3 1.3 1.3	dilute foliar spray 25 Oct, BBCH 83	14	< 0.001 < 0.001 mean <u>< 0.001</u> ^a	< 0.001 < 0.001 mean < 0.001 ^a	< 0.001 < 0.001 mean ^a < 0.001	Report: T002811-07 Trial: E12GA078296
Alexandria, LA, USA, 2007, (Sumner)	3 (7, 7) loam	17 17 17	3.5 3.6 3.6	concentrate foliar spray 16 Oct, BBCH 88-89	14	< 0.001 < 0.001 mean <u>< 0.001</u> ^a	< 0.001 < 0.001 mean < 0.001 ^a	< 0.001 < 0.001 mean ^a < 0.001	Report: T002811-07 Trial: E17LA078297
Yoakum, TX, USA, 2007, (Cheyenne)	3 (8, 7) sandy loam	17 17 17	1.1 1.1 1.1	dilute foliar spray 1 Oct, BBCH 94-98	14	< 0.001 < 0.001 mean <u>< 0.001</u> ^a	< 0.001 < 0.001 mean ^a < 0.001	< 0.001 < 0.001 mean ^a < 0.001	Report: T002811-07 Trial: W05TX078298

^a Results are from two replicate field samples, the mean may be selected for MRL derivation if compliant with cGAP. Sum1a expressed as MAB1a = sum of MAB1a plus its avermectin-like metabolites, corrected for molecular weight (MAB1a + 1.000 × 8,9-Zma + 1.016 × AB1a + 0.9693 × MFB1a + 0.9844 FAB1a). Metabolites < LOQ were assumed not to be present

Rape seed

The Meeting received supervised residue trials on rape seed (canola).

Supervised residue field trials on rape seed (canola) were carried out in 2009, 2010 and 2011 in Australia [Lean, 2014a/b, MK244/10298, MK244/10300 and additional information in Syngenta, 2014]. Rape seed on 12–160 m² plots was treated with two foliar applications using a gas powered hand-held boom sprayer. The spray volume was 74–88 L/ha (concentrate spray) or 154–250 L/ha (intermediate spray) or 403–575 L/ha (dilute spray). The spray mix included a non-ionic adjuvant. There were no unusual weather conditions. The mature crop was desiccated by wind-rowing, herbicide desiccation (Reglone® with 200 g/L diquat) or unaided natural desiccation. Herbicide desiccation involves the use of a herbicide to dry off the plant, which leaves it attached in the ground, preventing the crop from blowing away. Whereas wind-rowing involves the cutting of the crop and laying it in the rows to dry, providing a greater chance for loss of crop due to wind. Seeds (at least 2 kg, from at least 12 plants) were collected from each plot 0–15 days after cutting or chemical desiccation. Crops were headed directly in the field in case of unaided natural desiccation. Samples were stored at -2 °C or lower for a maximum of 265 days (2009–2010 samples) and 99 days (2011 samples).

Samples were analysed for MAB1a (NOA 426007), MAB1b (NOA 422390) and 8,9-Zma (NOA 438376) by HPLC-MS/MS Method TP/03. The isomer 8,9-Zma was only analysed in the 2009 and 2010 residue trials. The other avermectin-like metabolites were not quantified at all. Results were not corrected for control levels (< 0.005 mg/kg for each analyte) nor for average concurrent method recoveries (70–120% for each analyte). Results are shown in Table 4.

HPLC-MS/MS method TP/03 is considered valid for quantification of emamectin benzoate (B1a and B1b) in rape seeds at levels between 0.005–0.05 mg/kg. The quantification of 8,9-Zma at levels of 0.005–0.05 mg/kg in rape seeds is considered insufficiently validated. Since 8,9-Zma is not in the residue definition for enforcement or dietary risk assessment, this has no impact on the MRL derivation.

No residues of MAB1a, MAB1b or 8,9-Zma were not found in any of rape seed samples (< 0.005 mg/kg). The other avermectin-like metabolites were not quantified at all. Several trials have

been conducted on the same location. From such trials only the highest value is selected for MRL derivation if compliant with cGAP.

Table 4 Results from supervised field trials on rape (seeds) using an EC formulation (17 g ai/L) with adjuvant

RAPE SEEDS, Location, year, (Variety)	Number (interval) soil type	g ai/h a	g ai/hL	Method, last application date, GS (BBCH), harvest method	DAT (DAC*)	MAB1a (mg/kg)	MAB1b (mg/kg)	8,9-Zma (mg/kg)	Report, Trial
Inverleigh, VIC, Australia, 2009, (cv Jardee)	2 (14) brown clay loam	6.1 5.8	7.0 7.0	Concentrate foliar spray, 1 Nov 2009 GS 70 natural desiccation	51 (0*)	< 0.005	< 0.005	< 0.005	Report: 09-SAR-018GLP_SR Trial: Site 1
Inverleigh, VIC, Australia, 2009, (cv Jardee)	2 (14) brown clay loam	12 11	14 14	Concentrate foliar spray, 1 Nov 2009 GS 70 natural desiccation	51 (0*)	< 0.005	< 0.005	< 0.005	Report: 09-SAR-018GLP_SR Trial: Site 1
Glenthompson, VIC, Australia, 2009, (cv TT)	2 (14) brown clay loam	6.2 5.3	7.0 7.0	Concentrate foliar spray, 1 Nov 2009 GS 67 natural desiccation	47 (0*)	< 0.005	< 0.005	< 0.005	Report: 09-SAR-018GLP_SR Trial: Site 2
Northam, WA, Australia, 2009, (cv 46Y78)	2 (14) river loam	5.8 6.1	7.0 7.0	Concentrate foliar spray 16 Oct 2009 GS 69 natural desiccation	42 (0*)	< 0.005	< 0.005	< 0.005	Report: 09-SAR-018GLP_SR Trial: Site 3
Northam, WA, Australia, 2009, (cv 46Y78)	2 (14) river loam	11 12	14 14	Concentrate foliar spray 16 Oct 2009 GS 69 natural desiccation	42 (0*)	< 0.005	< 0.005	< 0.005	Report: 09-SAR-018GLP_SR Trial: Site 3
Williams, WA, Australia, 2009, (cv Manola 76TT)	2 (14), sandy loam	6.1 5.7	7.0 7.0	Concentrate foliar spray 15 Oct 2009 GS 68 natural desiccation	50 (0*)	< 0.005	< 0.005	< 0.005	Report: 09-SAR-018GLP_SR Trial: Site 4
Inverleigh, VIC, Australia, 2010, (cv 46Y78)	2 (7), brown clay loam	5.4 5.2	1.1 1.1	Dilute foliar spray, 1 Nov 2010 GS 76 herbicide desiccation	28 (0*) ^b	< 0.005	< 0.005	< 0.005	Report: 09-SAR-018GLP_SR Trial: Site 5, treatment 2
Inverleigh, VIC, Australia, 2010,	2 (8), brown clay loam	5.2 6.3	1.1 1.1	Dilute foliar spray, 16 Nov 2010 GS 78	13 (0*) ^b 21 (8*) 28 (15*)	< 0.005 ^a < 0.005 < 0.005	< 0.005 ^a < 0.005 < 0.005	< 0.005 a < 0.005 < 0.005	Report: 09-SAR-018GLP_SR Trial: Site 5,

RAPE SEEDS, Location, year, (Variety)	Number (inter val) soil type	g ai/h a	g ai/ hL	Method, last application date, GS (BBCH), harvest method	DAT (DAC*)	MAB1a (mg/kg)	MAB1b (mg/kg)	8,9- Zma (mg/kg)	Report, Trial
(cv 46Y78)				herbicide desiccation					treatment 3
Inverleigh, VIC, Australia, 2010, (cv 46Y78)	2 (6), brown clay loam	5.9 5.5	1.1 1.1	Dilute foliar spray, 22 Nov 2010 GS 79 herbicide desiccation	7 (0*) ^b 15 (8*) 22 (15*)	< 0.005 ^a < 0.005 < 0.005	< 0.005 ^a < 0.005 < 0.005	< 0.005 ^a < 0.005 < 0.005	Report: 09- SAR- 018GLP_SR Trial: Site 5, treatment 4
Inverleigh, VIC, Australia, 2010, (cv 46Y78)	2 (8), brown clay loam	12 12	2.2 2.2	Dilute foliar spray, 16 Nov 2010 GS 78 herbicide desiccation	13 (0*) ^b 21 (8*) 28 (15*)	< 0.005 ^a < 0.005 < 0.005	< 0.005 ^a < 0.005 < 0.005	< 0.005 ^a < 0.005 < 0.005	Report: 09- SAR- 018GLP_SR Trial: Site 5, treatment 5
Inverleigh, VIC, Australia, 2010, (cv 46Y78)	2 (6), brown clay loam	12 5.6	2.2 1.1	Dilute foliar spray 22 Nov 2010 GS 79 herbicide desiccation	7 (0*) ^b 15 (8*) 22 (15*)	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	Report: 09- SAR- 018GLP_SR Trial: Site 5, treatment 6
Lake Bolac, VIC, Australia, 2010, (cv 46Y78)	2 (8) soil not stated	5.4 5.9	1.1 1.1	Dilute foliar spray, 16 Nov 2010 GS 78 wind-rowed	13 (0*) ^b 21 (8*) 28 (15*)	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	Report: 09- SAR- 018GLP_SR Trial: Site 6
Lake Bolac, VIC, Australia, 2010, (cv 46Y78)	2 (8) soil not stated	11 13	2.2 2.2	Dilute foliar spray, 16 Nov 2010 GS 78 wind-rowed	13 (0*) ^b 21 (8*) 28 (15*)	< 0.005 ^a < 0.005 < 0.005	< 0.005 ^a < 0.005 < 0.005	< 0.005 ^a < 0.005 < 0.005	Report: 09- SAR- 018GLP_SR Trial: Site 6
Longford, TAS, Australia, 2010, (cv 517CL)	2 (7) black brown sandy loam	6.1 4.5	1.1 1.1	Dilute foliar spray, 27 Oct 2010 GS 67 wind-rowed	29 (0*) ^b	< 0.005 ^a	< 0.005 ^a	< 0.005 ^a	Report: 09- SAR- 018GLP_SR Trial: Site 7, treatment 2
Longford, TAS, Australia, 2010, (cv 517CL)	2 (7) black brown sandy loam	5.3 4.8	1.1 1.1	Dilute foliar spray 10 Nov 2010 GS 75 wind-rowed	15 (0*) ^b 21 (6*) 27 (12*)	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	Report: 09- SAR- 018GLP_SR Trial: Site 7, treatment 3
Longford, TAS, Australia, 2010, (cv 517CL)	2 (8) black brown sandy loam	5.7 5.3	1.1 1.1	Dilute foliar spray, 18 Nov 2010 GS 81 wind-rowed	7 (0*) ^b 13 (6*) 19 (12*)	< 0.005 ^a < 0.005 < 0.005	< 0.005 ^a < 0.005 < 0.005	< 0.005 ^a < 0.005 < 0.005	Report: 09- SAR- 018GLP_SR Trial: Site 7, treatment 4
Longford, TAS, Australia, 2010, (cv 517CL)	2 (7) black brown sandy loam	13 11	2.2 2.2	Dilute foliar spray, 10 Nov 2010 GS 75 wind-rowed	15 (0*) ^b 21 (6*) 27 (12*)	< 0.005 ^a < 0.005 < 0.005	< 0.005 ^a < 0.005 < 0.005	< 0.005 ^a < 0.005 < 0.005	Report: 09- SAR- 018GLP_SR Trial: Site 7, treatment 5
Longford,	2 (8)	12	2.2	Dilute foliar	7 (0*) ^b	< 0.005	< 0.005	< 0.005	Report: 09-

RAPE SEEDS, Location, year, (Variety)	Number (inter val) soil type	g ai/h a	g ai/ hL	Method, last application date, GS (BBCH), harvest method	DAT (DAC*)	MAB1a (mg/kg)	MAB1b (mg/kg)	8,9- Zma (mg/kg)	Report, Trial
TAS, Australia, 2010, (cv 517CL)	black brown sandy loam	12	2.2	spray, 18 Nov 2010 GS 81 wind-rowed	13 (6*) 19 (12*)	< 0.005 < 0.005	< 0.005 < 0.005	< 0.005 < 0.005	SAR- 018GLP_SR Trial: Site 7, treatment 6
Beverly, WA, Australia, 2010, (cv Hyola 502RR)	2 (7), red loam	5.3 5.5	7.0 7.0	Concentrate foliar spray, 19 Oct 2010 GS 86 herbicide desiccation	13 (0*) ^b 20 (7*) 27 (14*)	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	Report: 09- SAR- 018GLP_SR Trial: Site 8
Beverly, WA, Australia, 2010, (cv Hyola 502RR)	2 (7), red loam	11 11	14 14	Concentrate foliar spray, 19 Oct 2010 GS 86 herbicide desiccation	13 (0*) ^b 20 (7*) 27 (14*)	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	Report: 09- SAR- 018GLP_SR Trial: Site 8
Rosedale, VIC, Australia, 2011, (cv. Crusher TT)	2 (9), gray clay loam	5.4 5.8	2.8 2.8	intermediate foliar spray, 11 Nov 2011, GS not stated wind-rowed	7 (0*) ^b	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 1, treatment 2
Rosedale, VIC, Australia, 2011, (cv. Crusher TT)	2 (9), gray clay loam	11 11	5.6 5.6	intermediate foliar spray, 11 Nov 2011, GS not stated. wind-rowed	7 (0*) ^b	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 1, treatment 8
Rosedale, VIC, Australia, 2011, (cv. Crusher TT)	2 (5), gray clay loam	5.8 6.3	2.8 2.8	intermediate foliar spray, 23 Nov 2011, GS not stated herbicide desiccation	8 (8*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 1, treatment 3
Rosedale, VIC, Australia, 2011, (cv. Crusher TT)	2 (5), gray clay loam	11 11	5.6 5.6	intermediate foliar spray, 23 Nov 2011, GS not stated, herbicide desiccation	8 (8*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 1, treatment 9
Rosedale, VIC, Australia, 2011, (cv. Crusher TT)	2 (8), gray clay loam	6.5 5.4	2.8 2.8	intermediate foliar spray, 1 Dec 2011, GS not stated, natural desiccation	7 (0*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 1, treatment 4
Rosedale, VIC, Australia, 2011, (cv. Crusher TT)	2 (8), gray clay loam	11 12	5.6 5.6	intermediate foliar spray, 1 Dec 2011, GS not stated, natural	7 (0*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 1, treatment 10

RAPE SEEDS, Location, year, (Variety)	Number (inter val) soil type	g ai/h a	g ai/ hL	Method, last application date, GS (BBCH), harvest method	DAT (DAC*)	MAB1a (mg/kg)	MAB1b (mg/kg)	8,9- Zma (mg/kg)	Report, Trial
TT)				desiccation					
Rosedale, VIC, Australia, 2011, (cv. Crusher TT)	2 (6), gray clay loam	6.3 4.7	2.8 2.8	intermediate foliar spray, 2 Nov 2011, GS not stated wind-rowed	16 (0*) ^b	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 1, treatment 5
Rosedale, VIC, Australia, 2011, (cv. Crusher TT)	2 (6), gray clay loam	12 12	5.6 5.8	intermediate foliar spray, 2 Nov 2011, GS not stated wind-rowed	16 (0*) ^b	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 1, treatment 11
Rosedale, VIC, Australia, 2011, (cv. Crusher TT)	2 (7), gray clay loam	5.6 5.6	2.8 2.8	intermediate foliar spray, 18 Nov 2011, GS not stated herbicide desiccation	13 (8*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 1, treatment 6
Rosedale, VIC, Australia, 2011, (cv. Crusher TT)	2 (7), gray clay loam	12 11	5.6 5.6	intermediate foliar spray, 18 Nov 2011, GS not stated herbicide desiccation	13 (8*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 1, treatment 12
Rosedale, VIC, Australia, 2011, (cv. Crusher TT)	2 (5), gray clay loam	5.8 6.3	2.8 2.8	intermediate foliar spray, 23 Nov 2011, GS not stated natural desiccation	15 (0*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 1, treatment 7
Rosedale, VIC, Australia, 2011, (cv. Crusher TT)	2 (5), gray clay loam	11 11	5.6 5.6	intermediate foliar spray, 23 Nov 2011, GS not stated, natural desiccation	15 (0*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 1, treatment 13
Francis, SA, Australia, 2011 (cv. Crusher TT)	2 (8) sandy clay	5.7 5.4	2.8 2.8	intermediate foliar spray, 24 Nov 2011, GS not stated, herbicide desiccation	9 (9*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 2, treatment 3
Francis, SA, Australia, 2011 (cv. Crusher TT)	2 (8) sandy clay	12 11	5.6 5.6	intermediate foliar spray, 24 Nov 2011, GS not stated, herbicide desiccation	9 (9*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 2, treatment 9

RAPE SEEDS, Location, year, (Variety)	Number (inter val) soil type	g ai/h a	g ai/ hL	Method, last application date, GS (BBCH), harvest method	DAT (DAC*)	MAB1a (mg/kg)	MAB1b (mg/kg)	8,9- Zma (mg/kg)	Report, Trial
Francis, SA, Australia, 2011 (cv. Crusher TT)	2 (8) sandy clay	5.6 5.4	2.8 2.8	intermediate foliar spray, 2 Dec 2011, GS not stated, natural dessication	5 (0*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 2, treatment 4
Francis, SA, Australia, 2011 (cv. Crusher TT)	2 (8) sandy clay	13 11	5.6 5.6	intermediate foliar spray, 2 Dec 2011, GS not stated, natural dessication	5 (0*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 2, treatment 10
Francis, SA, Australia, 2011 (cv. Crusher TT)	2 (7) sandy clay	5.5 5.8	2.8 2.8	intermediate foliar spray, 16 Nov 2011, GS not stated, herbicide desiccation	17 (9*)	≤ 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 2, treatment 6
Francis, SA, Australia, 2011 (cv. Crusher TT)	2 (7) sandy clay	11 11	5.6 5.6	intermediate foliar spray, 16 Nov 2011, GS not stated, herbicide desiccation	17 (9*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 2, treatment 12
Francis, SA, Australia, 2011 (cv. Crusher TT)	2 (8) sandy clay	5.6 7.0	2.8 2.8	intermediate foliar spray, 24 Nov 2011, GS not stated, natural desiccation	13 (0*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 2, treatment 7
Francis, SA, Australia, 2011 (cv. Crusher TT)	2 (8) sandy clay	11 12	5.6 5.6	intermediate foliar spray, 24 Nov 2011, GS not stated, natural desiccation	13 (0*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 2, treatment 13
Marrar, NSW, Australia, 2011, (cv. Stingray)	2 (9) loam	5.8 6.1	3.7 3.7	intermediate foliar spray 3 Nov 2011, GS not stated, wind-rowed	6 (0*) ^b	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 3 treatment 2
Marrar, NSW, Australia, 2011, (cv. Stingray)	2 (9) loam	12 12	7.5 7.5	intermediate foliar spray 3 Nov 2011, GS not stated, wind-rowed	6 (0*) ^b	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 3 treatment 8
Marrar, NSW, Australia, 2011,	2 (9) loam	6.1 6.0	3.7 3.7	intermediate foliar spray 17 Nov 2011, GS not stated,	6 (6*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 3

RAPE SEEDS, Location, year, (Variety)	Number (inter val) soil type	g ai/h a	g ai/ hL	Method, last application date, GS (BBCH), harvest method	DAT (DAC*)	MAB1a (mg/kg)	MAB1b (mg/kg)	8,9- Zma (mg/kg)	Report, Trial
(cv. Stingray)				herbicide desiccation					treatment 3
Marrar, NSW, Australia, 2011, (cv. Stingray)	2 (9) loam	12 12	7.5 7.5	intermediate foliar spray 17 Nov 2011, GS not stated, herbicide desiccation	6 (6*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 3 treatment 9
Marrar, NSW, Australia, 2011, (cv. Stingray)	2 (6) loam	6.0 5.8	3.7 3.7	intermediate foliar spray 23 Nov 2011, GS not stated, natural desiccation	8 (0*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 3 treatment 4
Marrar, NSW, Australia, 2011, (cv. Stingray)	2 (6) loam	12 12	7.5 7.5	intermediate foliar spray 23 Nov 2011, GS not stated, natural desiccation	8 (0*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 3 treatment 10
Marrar, NSW, Australia, 2011, (cv. Stingray)	2 (5) loam	6.1 5.8	3.7 3.7	intermediate foliar spray, 25 Oct 2011, GS not stated, wind-rowed	15 (0*) ^b	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 3 treatment 5
Marrar, NSW, Australia, 2011, (cv. Stingray)	2 (6) loam	14 12	7.5 7.5	intermediate foliar spray, 25 Oct 2011, GS not stated, wind-rowed	15 (0*) ^b	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 3 treatment 11
Marrar, NSW, Australia, 2011, (cv. Stingray)	2 (5) loam	6.1 6.1	3.7 3.7	intermediate foliar spray, 8 Nov 2011, GS not stated, herbicide desiccation	15 (6*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 3 treatment 6
Marrar, NSW, Australia, 2011, (cv. Stingray)	2 (5) loam	12 12	7.5 7.5	intermediate foliar spray, 8 Nov 2011, GS not stated, herbicide desiccation	15 (6*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 3 treatment 12
Marrar, NSW, Australia, 2011, (cv. Stingray)	2 (9) loam	6.1 6.0	3.7 3.7	intermediate foliar spray, 17 Nov 2011, GS not stated, natural desiccation	<u>14</u> (0*)	<u>< 0.005</u>	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 3 treatment 7

RAPE SEEDS, Location, year, (Variety)	Number (inter val) soil type	g ai/h a	g ai/ hL	Method, last application date, GS (BBCH), harvest method	DAT (DAC*)	MAB1a (mg/kg)	MAB1b (mg/kg)	8,9- Zma (mg/kg)	Report, Trial
Marrar, NSW, Australia, 2011, (cv. Stingray)	2 (9) loam	12 12	7.5 7.5	intermediate foliar spray, 17 Nov 2011, GS not stated, natural desiccation	14 (0*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 3 treatment 13
Inverleigh, VIC, Australia, 2011, (cv. Crusher TT)	2 (8) clay	5.4 5.9	2.8 2.8	intermediate foliar spray, 24 Nov 2011, GS not stated, herbicide desiccation	9 (8*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 4 treatment 3
Inverleigh, VIC, Australia, 2011, (cv. Crusher TT)	2 (8) clay	12 12	5.6 5.6	intermediate foliar spray, 24 Nov 2011, GS not stated, herbicide desiccation	9 (8*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 4 treatment 9
Inverleigh, VIC, Australia, 2011, (cv. Crusher TT)	2 (8) clay	5.8 4.9	2.8 2.8	intermediate foliar spray, 2 Dec 2011, GS not stated, natural desiccation	6 (0*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 4 treatment 4
Inverleigh, VIC, Australia, 2011, (cv. Crusher TT)	2 (8) clay	11 11	5.6 5.6	intermediate foliar spray, 2 Dec 2011, GS not stated, natural desiccation	6 (0*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 4 treatment 10
Inverleigh, VIC, Australia, 2011, (cv. Crusher TT)	2 (6) clay	6.1 5.5	2.8 2.8	intermediate foliar spray, 16 Nov 2011, GS not stated, herbicide desiccation	17 (8*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 4 treatment 6
Inverleigh, VIC, Australia, 2011, (cv. Crusher TT)	2 (6) clay	13 11	5.6 5.6	intermediate foliar spray, 16 Nov 2011, GS not stated, herbicide desiccation	17 (8*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 4 treatment 12
Inverleigh, VIC, Australia, 2011, (cv. Crusher TT)	2 (8) clay	5.4 5.8	2.8 2.8	intermediate foliar spray, 24 Nov 2011, GS not stated, natural desiccation	14 (0*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP Trial: site 4 treatment 7
Inverleigh, VIC, Australia,	2 (8) clay	11 12	5.6 5.6	intermediate foliar spray, 24 Nov 2011,	14 (0*)	< 0.005	< 0.005	NA	Report: 11- SAR- 002GLP

RAPE SEEDS, Location, year, (Variety)	Number (inter val) soil type	g ai/h a	g ai/ hL	Method, last application date, GS (BBCH), harvest method	DAT (DAC*)	MAB1a (mg/kg)	MAB1b (mg/kg)	8,9- Zma (mg/kg)	Report, Trial
2011, (cv. Crusher TT)				GS not stated, natural desiccation					Trial: site 4 treatment 13

*DAT = days after treatment. This timepoint includes the time the crop lies or stands drying in the field (in case of wind-rowing or herbicide desiccation) and refers to the time at which the seeds were collected from the plant.

DAC = days after cutting or desiccation, where the crop was kept in the field, before actual samples were taken.

^a Results are for seeds + pods. Since the residues are < LOQ, results can be used for MRL derivation for seeds if according to cGAP

^b Windrowed crop was harvested at 0 days after cutting or herbicide desiccated crop was harvested at the day of desiccation, meaning the crop was not dried off at harvest. Such samples are not considered representative for MRL derivation and the corresponding residues are not selected.

Miscellaneous fodder and forage crops

The Meeting received supervised residue trials on almonds and rape.

Almond hulls

Supervised residue field trials on almonds were carried out in 2006 and 2007 in the USA [Ediger, 2007, MK244/0714, Oakes, 2009, MK50014]. The 2006 trials were summarized and evaluated by the 2011 JMPR. The 2007 trials are summarized here. Almond trees were treated with three foliar cover spray applications using a tractor mounted sprayer or airblast sprayer. The spray volume was either 93-94 L/ha (very concentrate spray as used for aircraft equipment), 700- 780 L/ha (concentrate spray as used for airblast sprayers) or 1600-1900 L/ha (dilute spray as used for ground equipment). The spray mix included a horticultural oil, non-ionic surfactant or organo-silicone adjuvant. There were no unusual weather conditions. Whole nuts were harvested at maturity. Samples were prepared by separating shells from nutmeat to generate at least 1.4 kg of nutmeat (=RAC). Samples were stored at -20 °C for a maximum of 9 months. Samples were analysed for MAB1a (NOA 426007), MAB1b (NOA 422390), 8,9-Zma (NOA 438376), AB1a (NOA 438309), MFB1a (NOA 415692) and FAB1a (NOA 415693) by HPLC-MS/MS Method RAM 465/01 (modification 30 May 2008). Results were not corrected for control levels (< 0.001 mg/kg for each analyte) nor for average concurrent method recoveries (70%-120% for each analyte). Results are shown in table 5. Results represent the mean of two replicate samples taken from each plot.

Several trials have been conducted on the same location. From such trials only the highest (mean) value is selected for MRL derivation if compliant with cGAP.

For the overall conclusion on avermectin-like metabolites also the data from the 2011 JMPR are taken into account. Residues of avermectin-like metabolites were found at levels (< 0.001–0.010 mg/kg for individual analytes) in several almond hull samples at DALT = 7–35, except in trial WC-IR-06-7116/CA (JMPR 2011). In this single trial MFB1a residues were particularly high: 0.018–0.036 mg/kg at DALT 7–21. Where metabolites were > LOQ, the sum of the four avermectin-like metabolites ranged from 0.002–0.010 mg/kg, expressed as MAB1a, except in trial WC-IR-06-7116/CA (JMPR 2011), where the sum ranged from 0.019–0.038 mg/kg at DALT 7–21. Where MAB1a was at least 0.01 mg/kg, the ratio of the sum of the avermectin-like metabolites to MAB1a ranged from 0.04–0.30 (median 0.09), except in trial WC-IR-06-7116/CA, where the ratio ranged from 0.28–0.78 at DALT=7–35.

Table 5 Residue results from supervised field trials on almonds (hulls) using an SG formulation (50 g ai/kg) with adjuvant

ALMOND HULLS Location, year, (Variety)	Number (interval) soil type	g ai/ha	g ai/hL	Method, last application date, growth stage (BBCH)	DAT (days)	MAB1a (mg/kg)	MAB1b (mg/kg)	Sum1a (mg/kg)	Report, Trial no
Madera, CA, USA, 2007 (Butte)	3 (7, 7), sandy loam	17 17 17	18 18 18	very concentrated foliar spray 27 Aug, BBCH 85	14	0.070 0.048 mean 0.059 ^a	0.004 0.002 mean 0.003 ^a	0.073 0.050 mean 0.061 ^a	Report: T002811-07 Trial: W30CA078291
Madera, CA, USA, 2007, (Mission)	3 (7, 7) loamy sand	17 17 17	2.2 2.2 2.2	concentrated foliar spray 29 Aug, BBCH 85	14	0.088 0.088 mean <u>0.088</u> ^a	0.005 0.006 mean 0.006 ^a	0.095 0.098 mean 0.097 ^a	Report: T002811-07 Trial: W29CA078294
Madera, CA, USA, 2007, (Mission)	3 (7, 7) loamy sand	17 17 17	0.90 0.90 0.90	dilute foliar spray 29 Aug, BBCH 85	14	0.062 0.052 mean 0.057 ^a	0.003 0.003 mean 0.003 ^a	0.065 0.055 mean 0.060 ^a	Report: T002811-07 Trial: W29CA078294
Fresno, CA, USA, 2007 (Non- Pareil)	3 (7, 7) sandy loam	16, 17 17	1.0 1.0 1.0	dilute foliar spray 27 Aug, BBCH 85	14	0.021 0.020 mean <u>0.020</u> ^a	0.001 0.001 mean 0.001 ^a	0.025 0.024 mean 0.024 ^a	Report: T002811-07 Trial: W30CA078292
Kerman, CA, USA, 2007, (Carmel)	3 (7, 7) loamy sand	17 17 17	2.4 2.4 2.4	concentrated foliar spray 27 Aug, BBCH 85	14	0.013 0.023 mean <u>0.018</u> ^a	< 0.001 0.002 mean 0.002 ^a	0.017 0.028 mean 0.022 ^a	Report: T002811-07 Trial: W30CA078293

^a Results are from two replicate field samples, the mean may be selected for MRL derivation if compliant with cGAP.

Sum1a expressed as MAB1a = sum of MAB1a plus its avermectin-like metabolites, corrected for molecular weight (MAB1a + 1.000 × 8,9-Zma + 1.016 × AB1a + 0.9693 × MFB1a + 0.9844 FAB1a). Metabolites < LOQ were assumed not to be present

Rape forage

Supervised residue field trials on rape forage were carried out in 2009 and 2010 in Australia [Lean, 2014a, MK244/10298 and additional information in Syngenta, 2014]. Rape forage on 12–160 m² plots were treated with two foliar applications using a gas powered hand-held boom sprayer. The spray volume was 74–88 L/ha (concentrate spray) or 403–575 L/ha (dilute spray). The spray mix included a non-ionic adjuvant. There were no unusual weather conditions. Immature foliage samples (at least 1 kg, from at least 12 plants) were collected 36–6 days before normal desiccation of the crop. Samples were stored at -2 °C or lower for a maximum of 274 days.

Samples were analysed for MAB1a (NOA 426007), MAB1b (NOA 422390) and 8,9-Zma (NOA 438376) by HPLC-MS/MS Method TP/03. Results were not corrected for control levels (< 0.005 mg/kg for each analyte) nor for average concurrent method recoveries (70%-120% for each analyte). Results are shown in table 6.

HPLC-MS/MS method TP/03 is considered valid for quantification of emamectin benzoate (B1a and B1b) at levels between 0.005–0.05 mg/kg in rape forage. The quantification of 8,9-Zma at levels of 0.005–0.05 mg/kg in rape seeds and rape forage is considered insufficiently validated. Since 8,9-Zma is not in the residue definition for enforcement or dietary risk assessment, this has no impact on the MRL derivation.

Residues of MAB1a in rape forage ranged between < 0.005–0.014 mg/kg; residues of MAB1b ranged between < 0.005–0.006 mg/kg. Residues of 8,9-Zma were not found in any of the samples. The other avermectin-like metabolites were not analysed.

Table 6 Results from supervised field trials on rape forage using an EC formulation (17 g ai/L) with adjuvant

RAPE FORAGE, Location, year, (Variety)	Number (interval) soil type	g ai/ha	g ai/hL	Method, last application date, growth stage, harvest method	DAT (DBC*)	MAB1a (mg/kg)	MAB1b (mg/kg)	8,9-Zma (mg/kg)	Report, Trial
Inverleigh, VIC, Australia, 2009, (cv Jardee)	2 (14) brown clay loam	6.1 5.8	7.0 7.0	Concentrate foliar spray, 1 Nov 2009 BBCH 70 natural desiccation	16 (35*) 29 (22*)	< 0.005 < 0.005	< 0.005 < 0.005	< 0.005 < 0.005	Report: 09-SAR-018GLP_SR Trial: Site 1
Inverleigh, VIC, Australia, 2009, (cv Jardee)	2 (14) brown clay loam	12 11	14 14	Concentrate foliar spray, 1 Nov 2009, BBCH 70, natural desiccation	16 (35*) 29 (22*)	< 0.005 < 0.005	< 0.005 < 0.005	< 0.005 < 0.005	Report: 09-SAR-018GLP_SR Trial: Site 1
Glenhompson, VIC, Australia, 2009, (cv TT)	2 (14) brown clay loam	6.2 5.3	7.0 7.0	Concentrate foliar spray, 1 Nov 2009 BBCH 67, natural desiccation	16 (31*) 29 (18*)	< 0.005 < 0.005	< 0.005 < 0.005	< 0.005 < 0.005	Report: 09-SAR-018GLP_SR Trial: Site 2
Northam, WA, Australia, 2009, (cv 46Y78)	2 (14) river loam	5.8 6.1	7.0 7.0	Concentrate foliar spray 16 Oct 2009 BBCH 69, natural desiccation	14 (28*) 28 (14*)	< 0.005 < 0.005	< 0.005 < 0.005	< 0.005 < 0.005	Report: 09-SAR-018GLP_SR Trial: Site 3
Northam, WA, Australia, 2009, (cv 46Y78)	2 (14) river loam	11 12	14 14	Concentrate foliar spray 16 Oct 2009 BBCH 69, natural desiccation	14 (28*) 28 (14*)	0.005 < 0.005	< 0.005 < 0.005	< 0.005 < 0.005	Report: 09-SAR-018GLP_SR Trial: Site 3
Williams, WA, Australia, 2009, (cv Manola 76TT)	2 (14), sandy loam	6.1 5.7	7.0 7.0	Concentrate foliar spray 15 Oct 2009 BBCH 68, natural desiccation	14 (36*) 28 (22*) 42 (8*)	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	Report: 09-SAR-018GLP_SR Trial: Site 4
Inverleigh, VIC, Australia, 2010, (cv 46Y78)	2 (7), brown clay loam	5.4 5.2	1.1 1.1	Dilute foliar spray, 1 Nov 2010 BBCH 76, herbicide desiccation	15 (13*) 21 (7*)	< 0.005 < 0.005	< 0.005 < 0.005	< 0.005 < 0.005	Report: 09-SAR-018GLP_SR Trial: Site 5, treatment 2
Inverleigh, VIC, Australia, 2010,	2 (8), brown clay loam	5.2 6.3	1.1 1.1	Dilute foliar spray, 16 Nov 2010 BBCH 78,	0 (13*) 6 (7*)	< 0.005 < 0.005	< 0.005 < 0.005	< 0.005 < 0.005	Report: 09-SAR-018GLP_SR Trial: Site 5,

RAPE FORAGE, Location, year, (Variety)	Number (interval) soil type	g ai/ha	g ai/ hL	Method, last application date, growth stage, harvest method	DAT (DBC*)	MAB1a (mg/kg)	MAB1b (mg/kg)	8,9- Zma (mg/kg)	Report, Trial
(cv 46Y78)				herbicide desiccation					treatment 3
Inverleigh, VIC, Australia, 2010, (cv 46Y78)	2 (6), brown clay loam	5.9 5.5	1.1 1.1	Dilute foliar spray, 22 Nov 2010 BBCH 79, herbicide desiccation	not sampled	-	-	-	Report: 09- SAR- 018GLP_SR Trial: Site 5, treatment 4
Inverleigh, VIC, Australia, 2010, (cv 46Y78)	2 (8), brown clay loam	12 12	2.2 2.2	Dilute foliar spray, 16 Nov 2010 BBCH 78, herbicide desiccation	0 (13*) 6 (7*)	0.006 < 0.005	< 0.005 < 0.005	< 0.005 < 0.005	Report: 09- SAR- 018GLP_SR Trial: Site 5, treatment 5
Inverleigh, VIC, Australia, 2010, (cv 46Y78)	2 (6), brown clay loam	12 5.6	2.2 1.1	Dilute foliar spray 22 Nov 2010 BBCH 79, herbicide desiccation	not sampled				Report: 09- SAR- 018GLP_SR Trial: Site 5, treatment 6
Lake Bolac, VIC, Australia, 2010, (cv 46Y78)	2 (8) soil not stated	5.4 5.9	1.1 1.1	Dilute foliar spray, 16 Nov 2010 BBCH 78, wind-rowed	0 (13*) 6 (7*)	0.013 < 0.005	0.005 < 0.005	< 0.005 < 0.005	Report: 09- SAR- 018GLP_SR Trial: Site 6
Lake Bolac, VIC, Australia, 2010, (cv 46Y78)	2 (8) soil not stated	11 13	2.2 2.2	Dilute foliar spray, 16 Nov 2010 BBCH 78, wind-rowed	0 (13*) 6 (7*)	< 0.005 0.011	< 0.005 < 0.005	< 0.005 < 0.005	Report: 09- SAR- 018GLP_SR Trial: Site 6
Longford, TAS, Australia, 2010, (cv 517CL)	2 (7) black brown sandy loam	6.1 4.5	1.1 1.1	Dilute foliar spray, 27 Oct 2010 BBCH 67, wind-rowed	14 (15*) 21 (8*)	< 0.005 < 0.005	< 0.005 < 0.005	< 0.005 < 0.005	Report: 09- SAR- 018GLP_SR Trial: Site 7, treatment 2
Longford, TAS, Australia, 2010, (cv 517CL)	2 (7) black brown sandy loam	5.3 4.8	1.1 1.1	Dilute foliar spray 10 Nov 2010 BBCH 75, wind-rowed	0 (15*) 7 (8*)	< 0.005 < 0.005	< 0.005 < 0.005	< 0.005 < 0.005	Report: 09- SAR- 018GLP_SR Trial: Site 7, treatment 3
Longford, TAS, Australia, 2010, (cv 517CL)	2 (8) black brown sandy loam	5.7 5.3	1.1 1.1	Dilute foliar spray, 18 Nov 2010 GS 81 wind-rowed	not sampled				Report: 09- SAR- 018GLP_SR Trial: Site 7, treatment 4
Longford, TAS, Australia, 2010, (cv 517CL)	2 (7) black brown sandy loam	13 11	2.2 2.2	Dilute foliar spray, 10 Nov 2010 BBCH 75, wind-rowed	0 (15*) 7 (8*)	0.014 < 0.005	0.006 < 0.005	< 0.005 < 0.005	Report: 09- SAR- 018GLP_SR Trial: Site 7, treatment 5
Longford, TAS, Australia,	2 (8) black brown sandy	12 12	2.2 2.2	Dilute foliar spray, 18 Nov 2010	not sampled				Report: 09- SAR- 018GLP_SR

RAPE FORAGE, Location, year, (Variety)	Number (interval) soil type	g ai/ha	g ai/hL	Method, last application date, growth stage, harvest method	DAT (DBC*)	MAB1a (mg/kg)	MAB1b (mg/kg)	8,9-Zma (mg/kg)	Report, Trial
2010, (cv 517CL)	loam			BBCH 81, wind-rowed					Trial: Site 7, treatment 6
Beverly, WA, Australia, 2010, (cv Hyola 502RR)	2 (7), red loam	5.3 5.5	7.0 7.0	Concentrate foliar spray, 19 Oct 2010 BBCH 86, herbicide desiccation	0 (13*) 7 (6*)	< 0.005 < 0.005	< 0.005 < 0.005	< 0.005 < 0.005	Report: 09-SAR-018GLP_SR Trial: Site 8
Beverly, WA, Australia, 2010, (cv Hyola 502RR)	2 (7), red loam	11 11	14 14	Concentrate foliar spray, 19 Oct 2010 BBCH 86, herbicide desiccation	0 (13*) 7 (6*)	< 0.005 0.008	< 0.005 < 0.005	< 0.005 < 0.005	Report: 09-SAR-018GLP_SR Trial: Site 8

* = days before cutting or days before desiccation, i.e. the period before normal harvest of the crop

APPRAISAL

Emamectin benzoate is a insecticide derivative of abamectin. Emamectin benzoate was first evaluated by the JMPR in 2011 for toxicology and residues. The 2011 Meeting established an ADI of 0–0.0005 mg/kg bw, expressed as emamectin benzoate. The ARfD was re-evaluated by the 2014 JMPR which reduced the ARfD to 0.02 mg/kg bw expressed as emamectin benzoate. The 2011 Meeting defined the residue as emamectin B1a benzoate for plant and animal commodities for compliance with the MRL and for estimation of dietary intake. Since the molecular weight difference between emamectin B1a benzoate and emamectin benzoate (consisting of 90% emamectin B1a benzoate and 10% emamectin B1b benzoate) is marginal, residues are not corrected for molecular weight. The 2011 Meeting considered the residue not fat soluble.

Emamectin benzoate was scheduled by the Forty-fifth Session of the CCPR (2013) for the evaluation of additional maximum residue levels by the 2014 JMPR. The manufacturer submitted additional supervised residue trials on almonds, pecans and rape, which were evaluated by the present Meeting.

Methods of Analysis

The Meeting received description and validation data for an analytical method for emamectin B1a and B1b benzoate and its 8,9-ZMa isomer in rape commodities for use in supervised residue trials. The analytical method is based on extraction with acidified acetonitrile and analysis by HPLC-MS/MS. The Meeting considered the method valid in the range 0.005–0.05 mg/kg emamectin B1a in rape seeds.

The analytical method for the determination of residues in almonds, pecans and almond hulls was considered valid by the 2011 Meeting.

Stability of pesticide residues in stored analytical samples

Storage stability studies were provided to the 2011 Meeting demonstrating the stability of emamectin B1a benzoate for at least 27 months at -20 °C or lower in stored plant commodities with high water content, 18 months in plant commodities with high starch content and at least 9 months in plant commodities with high oil content.

All crop commodities from supervised residue trials were analysed within the verified storage stability period. Oilseeds and rape forage were stored at -2 °C. Since parent is shown to be stable for a long period of time, trials where temperatures during storage were raised to -2 °C, were not rejected.

Results of supervised residue trials on crops

The Meeting received supervised trials data for emamectin benzoate on rape forage, tree nuts, rape seed and almond hulls. In addition, the 2011 JMPR trials on lettuces were re-evaluated by the present Meeting because of an ARfD exceedance for leaf lettuce.

Lettuce

The International Estimated Short Term Intake (IESTI) for emamectin benzoate was recalculated as the ARfD was changed from 0.03 to 0.02 mg/kg bw and a revised IESTI calculation model was available at the 2014 Meeting. The IESTI for the diets submitted to the JMPR represented 0–190% of the ARfD (0.02 mg/kg bw, expressed as emamectin benzoate) for children. The ARfD is exceeded for leaf lettuce (total, i.e., raw and processed commodities) in the diet for children.

At the 2011 JMPR, maximum residue levels for head lettuce, leaf lettuce and Cos lettuce were recommended based on head lettuce data, of 1 mg/kg. The present Meeting re-evaluated the separate datasets for head lettuce, Cos lettuce and leaf lettuce that were available to the 2011 JMPR. The leaf lettuce dataset was considered insufficient to recommend a maximum residue level (n=1–3, depending on the GAP used). The Cos lettuce dataset could however be used to propose a maximum residue level for leaf lettuce and Cos lettuce. The Meeting decided to retain the previous recommendation for head lettuce (current Codex MRL of 1 mg/kg) based on head lettuce data and to propose a new maximum residue level for Cos lettuce and leaf lettuce, based on the Cos lettuce data.

The 2011 Meeting agreed to combine the dataset for indoor and field grown Cos lettuce matching the Italian GAP (3 foliar spray applications, interval 7 days, 14.2 g ai/ha with a 3 day PHI) to represent residues in field and indoor grown Cos lettuce. This resulted in the following dataset: 0.030, 0.033, 0.042, 0.052, 0.10, 0.11, 0.30 and 0.33 mg/kg (n=8).

The present Meeting agreed that the dataset for Cos lettuce matching Italian GAP could be used to support a maximum residue level recommendation for Cos lettuce and leaf lettuce. The Meeting decided to withdraw its previous recommendations for Cos lettuce and leaf lettuce of 1 mg/kg and estimated a new maximum residue level 0.7 mg/kg for Cos lettuce and leaf lettuce. The Meeting estimated an STMR of 0.076 mg/kg and a HR of 0.33 mg/kg.

Tree nuts

The 2011 JMPR Meeting was unable to estimate a maximum residue level for almonds or pecans as the dataset was considered insufficient. Additional trials were submitted to the present Meeting and these were combined with the trials evaluated by the 2011 JMPR.

Field trials involving almonds were performed in the USA.

Critical GAP for tree nuts in the USA is for three foliar spray applications at 16.8 g ai/ha (maximum of 50.4 g ai/ha per season, interval 7 days) and a PHI of 14 days, with adjuvant. In almond trials from the USA (3 × 17 g ai/ha; interval 7 days and a 14 day PHI, applied with adjuvant) matching US GAP emamectin B1a benzoate residues in almonds (nutmeat) were: < 0.001 (4) mg/kg (including 1 value from the 2011 JMPR).

Field trials involving pecans were performed in the USA.

Critical GAP for tree nuts in the USA is for three foliar spray applications at 16.8 g ai/ha (maximum of 50.4 g ai/ha per season, interval 7 days) and a 14 day PHI, with adjuvant. In pecan trials from the USA (3 × 17 g ai/ha; interval 7 days and PHI 14 days, with adjuvant) matching US GAP emamectin B1a benzoate residues in pecans (nutmeat) were < 0.001 (5) mg/kg (including 1 value from the 2011 JMPR).

The Meeting agreed that the dataset for almonds and pecans matching USA GAP could be used to support a maximum residue level recommendation for tree nuts, and estimated a maximum residue level of 0.001* mg/kg in/on tree nuts and estimated an STMR of 0.001 mg/kg and an HR of 0.001 mg/kg.

Rape seed

Field trials involving rape were performed in Australia. Rape seeds were harvested using three different techniques: natural desiccation, herbicide desiccation, and wind-rowing. In natural desiccation seeds were collected after the plants had dried off naturally. Herbicide desiccation involves the use of a herbicide to dry off the green plant followed by seed collection up to 15 days later. Wind-rowing involves the cutting of the green crop and laying it in the rows to dry, followed by seed collection up to 15 days later. Since no residues were found in rape seeds in any of these trials, the impact of the harvest technique could not be assessed. Therefore, trials matching cGAP were selected irrespective of the harvest technique. Trials where the seeds were collected at the day of cutting (wind-rowing technique) or at the day of desiccation (herbicide desiccation technique) were excluded, since in this case the seeds were harvested from the green plant and such samples are not representative for maximum residue level derivation.

Critical GAP for rape in Australia is for two foliar spray applications at 5.1 g ai/ha with an unspecified interval and PHI 14 days. In rape trials from Australia (2×5.3 –6.1 g ai/ha; interval 6–9 days and PHI 13–17 days, with adjuvant) matching this GAP emamectin B1a benzoate residues in rape seeds were < 0.005 (6) mg/kg. Trials at higher dose rate and shorter PHI (2×11 –13 g ai/ha, interval 5–8 days and PHI 5–8 days) confirmed the non-residue situation: < 0.005 (4) mg/kg.

The Meeting agreed that the dataset for rape matching Australian GAP could be used to support a maximum residue level recommendation for rape seeds, and estimated a maximum residue level of 0.005* mg/kg in/on rape seed and estimated an STMR of 0 mg/kg. An HR is not considered necessary, since bulking/blending of the seeds is likely for a pre-harvest application.

Almond hulls

The 2011 JMPR Meeting was unable to estimate a maximum residue level for almond hulls as the dataset was considered insufficient. Additional trials were submitted to the present Meeting and these were combined with the trials evaluated in the 2011 JMPR report.

Field trials involving almonds were performed in the USA. Three spray concentrations were tested in one trial: very concentrated (for aircraft equipment), concentrated (for airblast equipment) and dilute (for ground equipment). In this one trial, the highest residue was found for the concentrated spray concentration as used for airblast equipment: 0.088 mg/kg versus 0.057–0.059 mg/kg. Since one trial is not sufficient to conclude on the effect of spray concentration and a second trial, where a concentrate spray concentration was used, produced much lower residues (0.018 mg/kg), the Meeting decided to take the highest residue from each location irrespective of the spray concentration.

Critical GAP for tree nuts in the USA is for three foliar spray applications at 16.8 g ai/ha (maximum of 50.4 g ai/ha per season, interval 7 days) and PHI of 14 days, with adjuvant. In almond trials from the USA (3×17 g ai/ha; interval 7 days and PHI 14 days, with adjuvant) matching this GAP emamectin B1a benzoate residues in almond hulls were 0.018, 0.020, 0.043, 0.088 mg/kg (n=4), as received (including 1 value from the 2011 JMPR). Since the dry weight for almond hulls is 90%, no dry weight correction is needed.

The Meeting agreed that the dataset for almond hulls matching USA GAP could be used to support a maximum residue level recommendation for almond hulls, and estimated a maximum residue level of 0.2 mg/kg in/on almond hulls and estimated a median residue of 0.0315 mg/kg. A highest residue is not considered necessary, since bulking/blending of the hulls is likely for use as feed commodity.

Rape forage

Field trials involving rape forage were performed in Australia. The only GAP available on rape is from Australia and this GAP contains a restriction not to use emamectin benzoate on rape grown as forage crop (i.e., rape forage). The Meeting decided not to use the trials.

Residues in animal commodities

The Meeting estimated the dietary burden of emamectin benzoate residues on the basis of the livestock diets listed in the FAO manual appendix IX (OECD feedstuff table). Calculation from highest residue, STMR (some bulk commodities) and STMR-P values provides the levels in feed suitable for estimating maximum residue levels, while calculation from STMR and STMR-P values from feed is suitable for estimating STMR values for animal commodities.

The 2014 JMPR Meeting recalculated the livestock dietary burden based on the uses presented by the 2011 JMPR and including the residue values for almond hulls from the 2014 JMPR Meeting. The maximum dietary burden for cattle for maximum residue level setting is not changed, while the mean dietary burden for cattle changed only marginally from 0.018 ppm in the 2011 JMPR Meeting to 0.021 ppm in the 2014 JMPR Meeting. Poultry is not exposed to emamectin benzoate through feed treated with emamectin benzoate. The Meeting therefore confirmed its previous recommendations for animal commodities.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits.

Definition of the residue for compliance with the MRL and for dietary risk assessment for plant and animal commodities: *emamectin B1a benzoate*

The Meeting considers the residue not fat soluble.

Summary of recommendations

CCN	Commodity name	MRL proposed mg/kg	MRL Previous mg/kg	STMR mg/kg	HR mg/kg	Comment
VL 0483	Leaf lettuce	0.7	1	0.076	0.33	-
VL 0510	Cos lettuce	0.7	1	0.076	0.33	-
TN 0085	tree nuts	0.001*		0.001	0.001	-
SO 0495-	rape seed	0.005*		0	-	-

CCN	Commodity name	MRL mg/kg	Median residue mg/kg	Highest residue mg/kg	Comment
-	almond hulls	0.2	0.0315	-	feed stuff

DIETARY RISK ASSESSMENT**Long-term intake**

The International Estimated Daily Intakes (IEDI) for emamectin benzoate was calculated from recommendations for STMRs for raw and processed commodities in combination with consumption data for corresponding food commodities. The results are shown in Annex 3.

The IEDI of in the 17 GEMS/Food cluster diets, based on the estimated STMRs in the 2011 and 2014 JMPR represented 1–9% of the maximum ADI of 0.0005 mg/kg bw, expressed as

emamectin benzoate. No conversion factor is needed to convert emamectin B1a benzoate residues to emamectin benzoate residues.

The Meeting concluded that the long-term intake of residues of emamectin benzoate from uses considered by the 2011 and 2014 Meeting is unlikely to present a public health concern.

Short-term intake

The International Estimated Short Term Intake (IESTI) for emamectin benzoate was recalculated due to the ARfD being changed from 0.03 to 0.02 mg/kg bw and the availability of a revised IESTI calculation model at the present Meeting. The IESTI was calculated from recommendations for STMRs and HRs for raw and processed commodities evaluated in the 2011 and 2014 JMPR Meeting in combination with consumption data for corresponding food commodities. The results are shown in Annex 4 to the 2014 Report.

The IESTI for the general population represented 0–30% of the ARfD (0.02 mg/kg bw, expressed as emamectin benzoate) and the IESTI for children represented 0–30% of the ARfD. No conversion factor is needed to convert emamectin B1a benzoate residues to emamectin benzoate residues.

The Meeting concluded that the short-term intake of residues of emamectin benzoate from uses considered by the 2011 and 2014 Meeting are unlikely to present a public health concern.

REFERENCES

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
-	Balshaw and Fernandez	not indicated	Determination of emamectin residues in canola, foliage and straw using LC-MS/MS Agrifood Technology, city, country, data not indicated. Agrifood Report No. GLP TP 03/10/20100817, Submitted by Syngenta. non-GLP, Unpublished.
MK244/0714	Ediger K	2007	Emamectin benzoate – Magnitude of the Residues in or on Almond and Pecan as Representative Commodities of Nut, Tree, Group 14. Syngenta Crop Protection Inc. Greensboro, NC, USA. Morse Laboratories Inc, Sacramento, CA, USA. 28 June 2007, Syngenta Report No. 007157-05, Morse Report No ML07-1339-SYN Task number T007157-05 Submitted by Syngenta RDN MK244/0714. GLP, Unpublished.
MK244/10298	Lean R	2014a	Determination of emamectin residues in canola after foliar application of Affirm® Insecticide. Crop Protection Research Pty Ltd., Mt Eliza, VIC, Australia. 23 May 2011, with amendment 04, 2014 Report No. 09-SAR-018GLP_SR, Submitted by Syngenta RDN MK244/10298. GLP, Unpublished.
MK244/10300	Lean R	2014b	Determination of residues of emamectin in canola after foliar applications of Affirm® Insecticide (17 g/L) at a rate of 300 mL/ha and 600 mL/ha. Crop Protection Research Pty Ltd., Mt Eliza, VIC, Australia. 15 August 2012, with amendment 04 and 05, 2014. Report No. 11-SAR-002GLP_SR, Submitted by Syngenta RDN MK244/10300. GLP, Unpublished
MK244/50014	Oakes T	2009	Emamectin benzoate – Magnitude of the Residues in or on Almond and Pecan as Representative Commodities of Nut, Tree, Group 14: Final

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
			Report. Syngenta Crop Protection Inc., Greensboro, NC, USA. Morse Laboratories Inc, Sacramento, CA, USA 4 February 2009. Syngenta Report No. T002811-07, Morse Report No ML08-1427-SYN, Task Number T002811-07 Submitted by Syngenta RDN MK244/50014. GLP, Unpublished
-	Syngenta	2014	Syngenta answers to questions, 30 July 2014

FENAMIDONE (264)

First draft was prepared by Dr Ursula Banasiak, Delegate of the Federal Institute for Risk Assessment, Berlin, Germany

EXPLANATION

Fenamidone is a broad-spectrum fungicide belonging to the imidazolinone group. The compound was evaluated the first time by the 2013 JMPR for toxicology where an ADI of 0–0.03 mg/kg bw and an ARfD of 1 mg/kg bw was allocated. The evaluation for residues was scheduled for the 2014 JMPR.

The current Meeting received information on physical and chemical properties, metabolism studies on plants and animals, analytical methods, supervised trials data, processing studies as well as use patterns.

IDENTITY

Fenamidone (RPA 407213) is the S-enantiomer of the stereoisomeric molecule RPA 405803 with the chiral centre in the 5-position. The S-isomer has been shown to be the biologically active enantiomer.

Common name: Fenamidone

Chemical name:

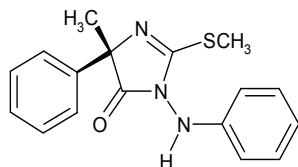
IUPAC: (S)-5-Methyl-2-methylthio-5-phenyl-3-(phenylamino)-3,5-dihydro-4H-imidazol-4-one

CA: (5S)-3,5-dihydro-5-methyl-2-(methylthio)-5-phenyl-3-(phenylamino)-4H-imidazol-4-one

CAS number: 161326-34-7

Molecular formula: C₁₇H₁₇N₃OS

Structural formula:



Molecular mass: 311.4

Minimum purity: Active substance (ai) manufactured 975 g/kg

Formulations

Fenamidone is available in numerous commercial formulations in different types (SC, WG, WP-soluble bag) in many countries.

PHYSICAL AND CHEMICAL PROPERTIES

Fenamidone is a thermally stable solid. The compound is poorly soluble in water and soluble in toluene, dichloromethane and acetone. It is stable at pH 5 and pH 7 and hydrolyses under acidic or alkaline conditions, is not explosive and not self-igniting. For details see Table 1.

Table 1 Physical and chemical properties of fenamidone

Property	Findings	Report, Bayer reference
Active ingredient		
Melting point	137 °C	R003094, M-163995-01-1

Property	Findings		Report, Bayer reference
Decomposition	At about 240 °C		R003102, M-164004-01-1
Relative density	1.29		R003094, M-163995-01-1
Vapour pressure	33 °C	1.48×10^{-6} Pa	R003104, M-164006-01-1
	41 °C	5.95×10^{-6} Pa	
	49 °C	2.28×10^{-5} Pa	
	25 °C (calculated)	3.4×10^{-7} Pa	
	20 °C (calculated)	1.32×10^{-7} Pa	
Henry’s law constant	0.5×10^{-3} Pa m ³ /mol (20 °C)		R003104, M-164006-01-1
Physical state, colour	White woolly powder		R003094, M-163995-01-1
Odour	No characteristic odour		R003094, M-163995-01-1
Solubility in water	7.8 mg/L (20 °C), pH 4–10 has no significant effect on water solubility		R003098, M-164000-01-1 R004781, M-166509-01-1
Solubility in organic solvents (20 °C)	Acetone	ca. 250 g/L	R003098, M-164000-01-1
	Acetonitrile	86.1 g/L	
	Dichloromethane	ca. 330 g/L	
	Ethyl acetate	105.7 g/L	
	n-Heptane	0.3 g/L	
	Toluene	40.1 g/L	
	Methanol	43.1 g/L	
	n-Octanol	9.7 g/L	
Partition coefficient	Log P _{ow} = 2.8 (20 °C)		R003100, M-164002-01-1
Hydrolysis rate		DT ₅₀ (days)	R003342, M-164512-01-1
	pH 4	41.7	
	pH 5	222	
	pH 7	411	
	pH 9	27.6	
	Fenamidone is stable at pH 5 and pH 7 and was hydro-lysed at pH 4 and pH 9. Major hydrolysis products: —RPA 410193 at pH 4 —RPA 413350 & RPA 412708 at pH 9.		
Photochemical degradation	[C-phenyl-UL- ¹⁴ C]-fenamidone DT ₅₀ = 25.7 hours, corresponding to 5 days Florida summer sunlight. Major metabolites: RPA 412708 & RPA 410193 [N-phenyl-UL- ¹⁴ C]-fenamidone DT ₅₀ = 29.5 hours, corresponding to 5.8 days of Florida summer sunlight. Major metabolite: RPA418915 (but < 10%)		R006697, M-173299-01-1 R005035, M-170334-01-1
Quantum yield	Quantum yield (Φ): 3.38×10^{-2} Theoretical environmental half-life ranged from 25843 hours in July to approximately 1.2×10^9 hours in December.		CR&D/CRLD/AN/9716610, M-164514-01-1
Dissociation constant	Not ionisable in water At pH 4: neutral fenamidone (modelling)		R003096, M-163997-02-1 C026172, M-210906-01-1
Flammability	Not classified as highly flammable		R003437, M-164707-01-1
Auto-flammability	No self-ignition temperature observed up to 400 °C		R003437, M-164707-01-1
Explosive properties	No danger of explosion		R003437, M-164707-01-1
Oxidising properties	No oxidizing properties under test conditions		R005016, M-170307-02-1 C026164, M-210891-01-1
Technical material			
Physical state, colour	White to cream fine powder		R003094, M-163995-01-1
Odour	No characteristic odour		R003094, M-163995-01-1

METABOLISM AND ENVIRONMENTAL FATE

Fenamidone active substance as defined by the ISO common name and the CA name is the S-enantiomer of the stereoisomeric molecule RPA405803 with the chiral centre in the 5-position of the dihydro-imidazolone ring. This S-enantiomer has been shown to be the biologically (fungicidally) active enantiomer. The minimum purity of fenamidone in the technical substance is 975 g/kg.

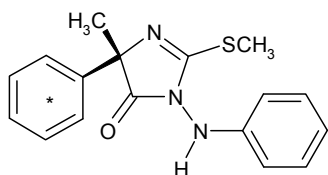
The chiral carbon atom in the 5-position of the dihydro-imidazolone ring is substituted by 4 non-hydrogen substituents, i.e., by an amino, carbonyl, methyl and phenyl group. Prerequisite for isomerisation/racemisation of this type of centre of chirality is the presence of a hydrogen atom as a substituent that can be split off easily. In fenamidone, no hydrogen is linked to the chiral centre.

Therefore, racemisation is chemically not possible and the configuration and the optical purity, respectively, established by synthesis (5S, ee > 99%) does not change afterwards.

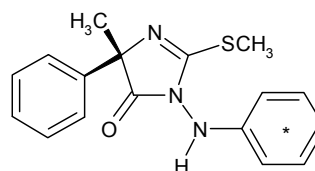
All fenamidone studies on metabolism, residues and toxicology were conducted with this pure S-enantiomer as test substance. Accompanying analysis was usually conducted using achiral methods without the possibility to examine a potential isomerisation/racemisation during the interaction with animal, plant and environmental matrices. In order to investigate this possibility a study (M-173254-01-1) was performed using chiral HPLC analysis of different samples out of “nature-of-residue” studies on soil metabolism, water-sediment, hydrolysis, aqueous and soil photolysis as well as metabolism on lettuce, grape, potato and rat (ADME) and on the livestock animals hen and goat. In no case, there was any evidence of enantiomerization or racemization of the parent substance and its metabolites during metabolic conversions or physico-chemical degradation.

The metabolism and distribution of fenamidone in plants and animals was investigated using the ^{14}C -labelled test materials as shown below.

[C-phenyl-UL- ^{14}C]-fenamidone



[N-phenyl-UL- ^{14}C]-fenamidone



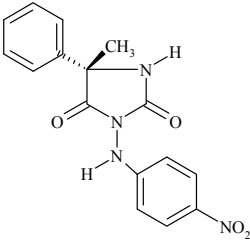
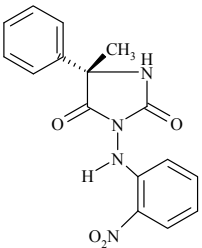
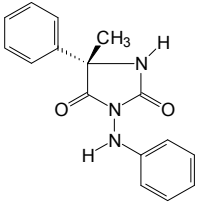
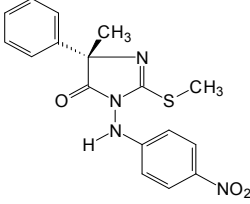
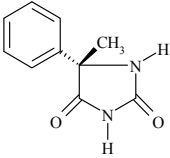
* Denotes position of ^{14}C -label

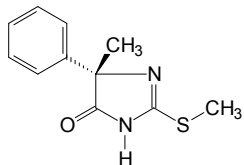
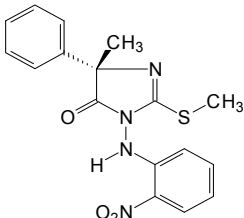
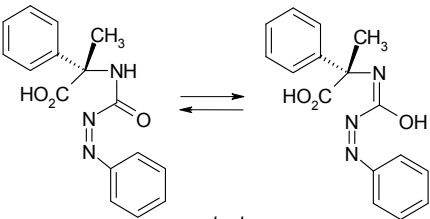
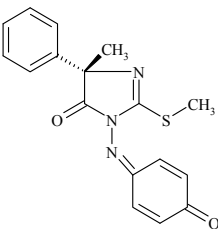
Chemical names, structures and code names of metabolites and degradation products of fenamidone are summarized in Table 2. Compounds are referred to primarily by the code number of the S-enantiomer, with the code of the corresponding racemic compound additionally mentioned.

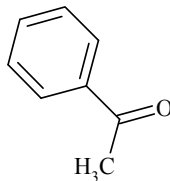
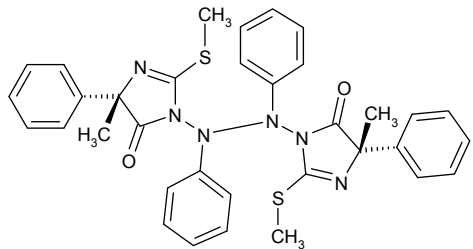
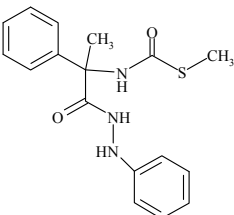
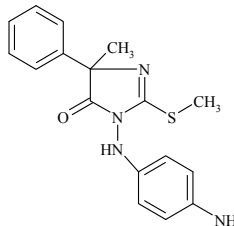
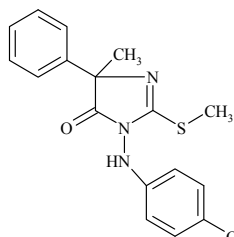
Racemic compounds are generally used as reference standards in metabolism/degradation and residue studies. Nevertheless, the codes for the S-enantiomer were frequently used below for describing the metabolites, because their enantiomeric stability in the S-configuration has been demonstrated.

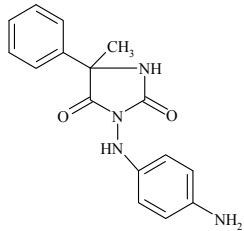
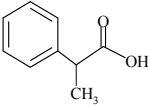
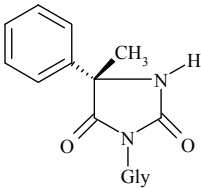
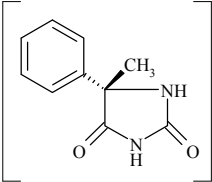
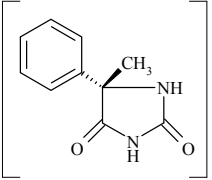
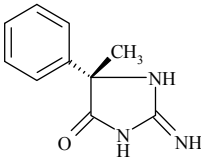
Table 2 Code names, chemical names and structures of fenamidone related substances

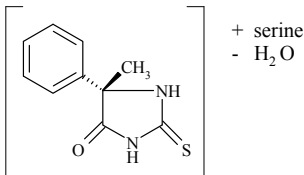
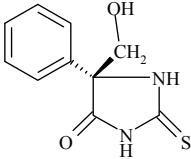
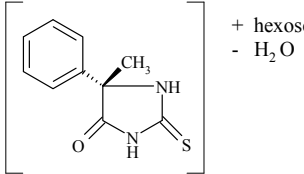
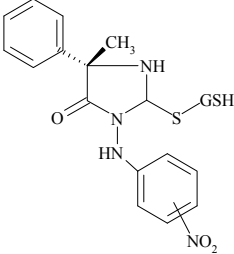
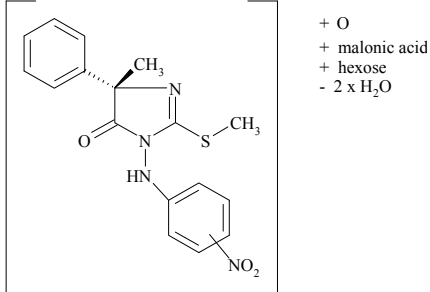
No.	Name, Structure, IUPAC name, CAS name, [CAS number]	Mol. formula, molar mass Other names / codes	Occurrence, Compartment
ai	FENAMIDONE (parent substance) (5S)-5-methyl-2-methylthio-5-phenyl-3-(phenylamino)-3,5-dihydro-4H-imidazol-4-one (IUPAC) 4H-imidazol-4-one, 3,5-dihydro-5-methyl-2-(methylthio)-5-phenyl-3-(phenylamino)-, (5S) (CAS) CAS No.: 161326-34-7 (S-Enantiomer) 151022-37-6 (Racemate)	$\text{C}_{17}\text{H}_{17}\text{N}_3\text{OS}$ 311.4 g mol^{-1} S-Enantiomer: RPA 407213 AE C649693 RPA 410287 BCS-AB63420 <u>Racemate:</u> RPA 405803 AE C643114	Active substance Rat Plants Rotated crops Livestock Soil Water
1	RPA 221607	$\text{C}_{16}\text{H}_{14}\text{N}_4\text{O}_4$ 326.3 g mol^{-1} S-Enantiomer: RPA 221607 AE 0591776 BCS-AX84896 2,4-imidazolidinedione,	Soil, aerobic Carrot Rotational crops: Swiss chard

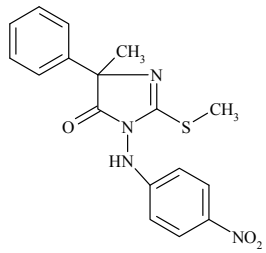
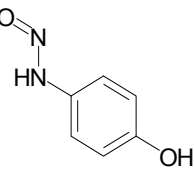
No.	Name, Structure, IUPAC name, CAS name, [CAS number]	Mol. formula, molar mass Other names / codes	Occurrence, Compartment
	 (5S)-5-methyl-3-[(4-nitrophenyl)amino]-5-phenyl- 2,4-imidazolidinedione (IUPAC) CAS No.: N/A (S-Enantiomer) N/A (Racemate)	5-methyl-3[(4nitrophenyl)-amino]-5-phenyl-, (5S)- <u>Racemate:</u> RPA 409446 AE 0591779	
2	RPA 221701  (5S)-5-methyl-3-[(2-nitrophenyl)amino]-5-phenyl- 2,4-imidazolidinedione (IUPAC) CAS No.: N/A (S-Enantiomer) N/A (Racemate)	C₁₆H₁₄N₄O₄ 326.3 g mol⁻¹ S-Enantiomer: RPA 221701 AE 0591777 BCS-AX84897 2,4-imidazolidinedione, 5-methyl-3-[(2-nitrophenyl)-amino]-5-phenyl-, (5S)- <u>Racemate:</u> RPA 410995 AE 0591778	Soil, aerobic Carrot Rotational crops: Turnip, Swiss chard
3	RPA 410193  (S)-5-Methyl-5-phenyl-3-(phenylamino)- 2,4-imidazolidinedione (IUPAC) 2,4-Imidazolidinedione, 5-methyl-5-phenyl-3-(phenylamino)-, (5S)- (CAS) CAS No.: 332855-88-6 (S-Enantiomer) 153969-11-0 (Racemate)	C₁₆H₁₅N₃O₃ 281.3 g mol⁻¹ S-Enantiomer: RPA 410193 AE 0540049 BCS-AX71129 Diketo-fenamidone (DK-Fen) <u>Racemate:</u> RPA 405862 AE C650488	Soil, aerobic Soil, photolysis Hydrolysis, abiotic Photolysis, buffer Vine grapes, Lettuce, Tomato, Potato, Carrot Hen
4	RPA 411639  (5S)-5-methyl-2-(methylthio)-3-(4-nitrophenylamino)-5-phenyl-3,5-dihydro-4H-imidazol-4-one (IUPAC) 4H-imidazol-4-one, 3,5-dihydro-5-methyl-2-(methylthio)-3-[(4-nitrophenyl)amino]-5-phenyl-, (5S) (CAS) CAS No.: 776299-39-9 (S-Enantiomer) 151022-56-9 (Racemate)	C₁₇H₁₆N₄O₃S 356.4 g mol⁻¹ S-Enantiomer: RPA 411639 AE 0540054 BCS-AX71134 <u>Racemate:</u> RPA 406012 AE 0540056	Soil, aerobic Carrot Rotational crops: Turnip, Swiss chard Rat (postulated intermediate)
5	RPA 412636  (S)-5-methyl-5-phenyl-2,4-imidazolidine-dione (IUPAC)	C₁₀H₁₀N₂O₂ 190.2 g mol⁻¹ S-Enantiomer: RPA 412636 AE 0540051 BCS-AX71131 “ fenamidone-hydantoin” Desanilino-diketo-	Soil, aerobic Soil, photolysis Water / sediment Lettuce, Potato, Rotational crops: Wheat, Turnip,

No.	Name, Structure, IUPAC name, CAS name, [CAS number]	Mol. formula, molar mass Other names / codes	Occurrence, Compartment
	2,4-imidazolidinedione, 5-methyl-5-phenyl-, (5S) (CAS) CAS No.: 27539-12-4 (S-Enantiomer) 6843-49-8 (Racemate)	fenamidone (DADK-Fen) <u>Racemate</u> : RPA 717879 AE C415557	Swiss chard Rat, Goat, Hen
6	RPA 412708  (5S)-5-methyl-2-(methylthio)-5-phenyl-3,5-dihydro-4H-imidazol-4-one (IUPAC) 4H-imidazol-4-one, 3,5-dihydro-5-methyl-2-(methylthio)-5-phenyl-, (5S)- (CAS) CAS No.: 332855-82-0 (S-Enantiomer) 151023-66-4 (Racemate)	C ₁₁ H ₁₂ N ₂ OS 220.9 g mol ⁻¹ S-Enantiomer: RPA 412708 AE 0540050 BCS-AX71130 Desanilino-fenamidone (DA-Fen) <u>Racemate</u> : RPA 408056 AE 0540057	Soil, aerobic Soil, photolysis Hydrolysis, abiotic Photolysis, buffer Photolysis, nat. water Water / sediment Lettuce, Potato Rat, Goat, Hen
7	RPA 413255  (5S)-5-methyl-2-(methylthio)-3-(2-nitrophenylamino)-5-phenyl-3,5-dihydro-4H-imidazol-4-one (IUPAC) 4H-imidazol-4-one, 3,5-dihydro-5-methyl-2-(methylthio)-3-[(2-nitrophenylamino)]-5-phenyl-, (5S) (CAS) CAS No.: 776299-40-2 (S-Enantiomer) 151022-56-9 (Racemate)	C ₁₇ H ₁₆ N ₄ O ₃ S 356.4 g mol ⁻¹ S-Enantiomer: RPA 413255 AE 0540053 BCS-AX71133 <u>Racemate</u> : RPA 410914 AE 0540055	Soil, aerobic Soil, photolysis Carrot Rotational crops: Turnip, Swiss chard
8	RPA 413350  tautomers 2-phenyl-N-[(E)-phenyldiazenyl]carbonyl-D-alanine (IUPAC) benzenecetic acid, □-methyl □[[phenylazo]] carbonyl-amino]-, (□S)- (CAS) CAS No.: 775326-19-7 (S-Enantiomer) N/A (Racemate)	C ₁₆ H ₁₅ N ₃ O ₃ 297.3 g mol ⁻¹ S-Enantiomer: RPA 413350 AE 0540052 BCS-AX71132 <u>Racemate</u> : RPA 409344 AE 0841910	Hydrolysis, abiotic
9	RPA 418915  (5S)-5-methyl-2-(methylthio)-3-[(4-oxo-2,5-cyclohexadien-1-ylidene)amino]-5-phenyl-3,5-dihydro-4H-imidazol-4-one (IUPAC) CAS No.: N/A (S-Enantiomer) N/A (Racemate)	C ₁₇ H ₁₅ N ₃ O ₂ S 325.4 g mol ⁻¹ S-Enantiomer: RPA 418915 AE R418915 BCS-AZ94979 4H-imidazol-4-one, 3,5-dihydro-5-methyl-2-(methylthio)-3-[(4-oxo-2,5-cyclohexadien-1-yl)-amino]-5-phenyl-, (5S)	Aqueous photolysis, buffer
10	Acetophenone	C ₈ H ₈ O 120.2 g mol ⁻¹	Photolysis, nat. water

No.	Name, Structure, IUPAC name, CAS name, [CAS number]	Mol. formula, molar mass Other names / codes	Occurrence, Compartment
	 1-phenylethanone (IUPAC) ethanone, 1-phenyl- (CAS) CAS No.: 98-86-2	AE C419259	
11	BCS-CV62706  (5S,5'S)-3,3'-(1,2-diphenylhydrazine-1,2-diyl)bis[5-methyl-2-(methylthio)-5-phenyl-3,5-dihydro-4H-imidazol-4-one] (IUPAC) CAS No.: N/A	C₃₄H₃₂N₆O₂S₂ 620.8 g mol⁻¹ S,S-Enantiomer: RPA: None AE: None BCS-CV62706 “Dimer” “Hedgehog 4”	Soil, aerobic Carrot
12	RPA 407599 (only racemic code available)  [1-phenyl-1-(N'-phenylhydrazinocarbonyl)-ethyl]-thiocarbamic acid methyl ester (IUPAC) benzeneacetic acid, α-methyl-α-[[methylthio]-carbonyl]amino]-, 2-phenylhydrazide (CAS) CAS No.: N/A (S-Enantiomer) N/A (Racemate)	C ₁₇ H ₁₉ N ₃ O ₂ S 329.42 g mol ⁻¹	Tomato
13	RPA 409352 (only racemic code available)  5-methyl-2-(methylthio)-3-[(4-aminophenyl)amino]-5-phenyl-3,5-dihydro-4H-imidazole-4-one (IUPAC) 4H-imidazol-4-one, 3,5-dihydro-5-methyl-2-(methylthio)-3-[(4-aminophenyl)amino]-5-phenyl (CAS) CAS No.: N/A	C ₁₇ H ₁₈ N ₄ OS 326.42 g mol ⁻¹	Rat (major, also conjugate with glucuronic acid) Goat
14	RPA 409361, AE 0540047, (only racemic code available) 	C ₁₇ H ₁₇ N ₃ O ₂ S 327.41 g mol ⁻¹	Rat (major, also conjugate with glucuronic acid) Hen, Goat (OH-position undefined) as aglycon and glucoside conjugate

No.	Name, Structure, IUPAC name, CAS name, [CAS number]	Mol. formula, molar mass Other names / codes	Occurrence, Compartment
	5-methyl-2-(methylthio)-3-[(4-hydroxyphenyl)amino]-5-phenyl-3,5-dihydro-4H-imidazol-4-one (IUPAC) 4H-imidazol-4-one, 3,5-dihydro-5-methyl-2-(methylthio)-3-[(4-hydroxyphenyl)amino]-5-phenyl (CAS) CAS No.: N/A		
15	RPA 409445 , AE 0652676, (only racemic code available)  5-methyl-3-[(4-aminophenyl)amino]-5-phenyl- 2,4-imidazolidinedione (IUPAC) 2,4-imidazolidinedione, 5-methyl-3-[(4-aminophenyl)amino]-5-phenyl- (CAS) CAS No.: N/A	$C_{16}H_{16}N_4O_2$ 296.33 g mol ⁻¹	Rat (major in conjugated form) Hen (minor)
16	2-Phenyl-propionic acid  Configuration in plants not known CAS No.: 492-37-5	$C_9H_{10}O_2$ 150.18 g mol ⁻¹ P2	Rotational crops: Wheat, Turnip, Swiss chard
17	Hydantoin glycoside  (S)-5-methyl-5-phenyl-2,4-imidazolidine-dione-3-N-glycoside	$C_{16}H_{20}N_2O_8$ 368.35 g mol ⁻¹ P7	Rotational crops: Wheat, Turnip, Swiss chard Rat (only aglycon)
18	Hydantoin malonyl glycoside  + hexose + malonic acid - 2 x H ₂ O Appears as S-enantiomer in plants, exact structure not known CAS No.: N/A	P9	Rotational crops: Wheat, Turnip, Swiss chard
19	Hydantoin serine  + serine - H ₂ O Appears as S-enantiomer in plants, exact structure not known CAS No.: N/A	P4	Rotational crops: Wheat, Turnip, Swiss chard
20	imino hydantoin 	$C_{10}H_{11}N_3O$ 189.22 g mol ⁻¹ P5	Rotational crops: Wheat, Turnip, Swiss chard

No.	Name, Structure, IUPAC name, CAS name, [CAS number]	Mol. formula, molar mass Other names / codes	Occurrence, Compartment
	(5S)-2-imino-5-methyl-5-phenylimidazolidin-4-one CAS No.: N/A		
21	Thiohydantoin serine  Appears as S-enantiomer in plants, exact structure not known CAS No.: N/A	P6	Rotational crops: Wheat, Swiss chard
22	Hydroxymethyl thiohydantoin  (5R)-5-(hydroxymethyl)-5-phenyl-2-thioxoimidazolidin-4-one (IUPAC) CAS No.: N/A	C ₁₀ H ₁₀ N ₂ O ₂ S 222.27 g mol ⁻¹ P13	Rotational crops: Wheat, Swiss chard
23	Thiohydantoin glycoside  Appears as S-enantiomer in plants, exact structure not known CAS No.: N/A	P8	Rotational crops: Wheat, Turnip, Swiss chard
24	Nitro-GSH  Appears as S-enantiomer in plants, exact structure not known CAS No.: N/A	A7	Rotational crops: Swiss chard (GSH: glutathionate conjugate)
25	Hydroxy-nitro-malonyl-glycoside  Appears as S-enantiomer in plants, exact structure not known CAS No.: N/A	A8	Rotational crops: Turnip
26	RPA 411639 (only racemic code available)	C ₁₇ H ₁₆ N ₄ O ₃ S 356.41 g mol ⁻¹	Carrot

No.	Name, Structure, IUPAC name, CAS name, [CAS number]	Mol. formula, molar mass Other names / codes	Occurrence, Compartment
	 5-methyl-2-(methylsulfanyl)-3-[(4-nitrophenyl)amino]-5-phenyl-3,5-dihydro-4H-imidazol-4-one (IUPAC) CAS No.: N/A		
27	UMET/10  4-(nitrosoamino)phenol (IUPAC)	$C_6H_6N_2O$ $138.13 \text{ g mol}^{-1}$	Rat

Animal metabolism

The metabolism of fenamidone has been studied in laboratory rats, lactating goats and laying hens.

Table 3 Characterization of radio-labelled test materials of fenamidone

	[C-Phenyl-UL- ^{14}C]-fenamidone, reference	[N-Phenyl-UL- ^{14}C]-fenamidone, reference
Studies on goats	643724, M-183880-01-1	684088, M-183833-01-1
Studies on hens	643735, M-183883-01-1	684090, M-183837-01-1
Radiochemical purity	99.5% (HPLC), 98.7% (TLC)	100% (HPLC), 98.2% (TLC)
Specific activity	1.41 GBq/mM, 38 mCi/mM	1.37 GBq/mM, 37 mCi/mM

Rats

Rat metabolism studies were evaluated by the WHO Core Assessment Group of the 2013 JMPR and are summarized as follows: In experiments conducted in rats using [^{14}C]fenamidone labelled at either the C-phenyl or N-phenyl part of the molecule, the time to reach the maximum plasma concentration of radioactivity was 2 hours after a single oral dose of 3 mg/kg bw and 26 hours after a single oral dose of 300 mg/kg bw. Gastrointestinal absorption was greater than 80%. Radioactivity distributed to most tissues, with no evidence of accumulation. Relatively high concentrations of radioactivity (approximately 400 times higher than that in plasma) were detected in the thyroid following dosing with C-phenyl- but not N- phenyl-labelled fenamidone, suggesting the distribution of a radiolabelled metabolite to the thyroid. Fenamidone undergoes extensive metabolism in the rat by phase I (oxidation, reduction and hydrolysis) and phase II reactions (conjugation). The plasma elimination half-life was at least 60 hours. More than 20 metabolites were detected in rat excreta, with the majority of radioactivity excreted in the faeces (up to approximately 90% of the administered dose) and the remainder in urine. Mass balance data indicated that the majority of radioactivity (> 80%) was eliminated within 48 hours of dosing.

Lactating goats

Burri (1999) investigated the fate of fenamidone following repeated oral administration to the lactating goat in two separate studies (643724, M-183880-01-1 and 684088, M-183833-01-1, see Table 3).

Two dose levels were used for each label position, with one lactating Saanen goat being used per dose and label. The dose levels were 1.0 ppm and 10.4 ppm for [C-phenyl-UL- ^{14}C]-fenamidone and 1.5 ppm and 11.5 ppm for [N-phenyl-UL- ^{14}C]-fenamidone in the diet. Dosing was performed twice daily by capsule during seven consecutive days. Goats were sacrificed at 22 hours after the last

(14th) administration. Milk, urine and faeces were sampled during the course of the study and liver, kidney, muscles and fat were obtained after the end of the experiment. The total radioactive residues (TRR) were determined by LSC in liquid matrices after solubilisation, in solid matrices by combustion. The samples were prepared as follows:

Milk

Studies 643724 and 684088: Radioactivity in the milk was determined by LSC after solubilisation. The milk was defatted by centrifugation. The defatted milk was de-proteinised by adding acetone (1 + 4, v/v). After shaking for 30 min. at room temperature, an incubation for 16 hours at about 4 °C and centrifugation, the supernatant was removed and the pellet was further extracted twice with acetone, twice with acetone/water (1 + 1, v/v) and once with methanol/water (C-phenyl-label: 1 + 1, v/v; N-phenyl-label: 2 + 8, v/v). The remaining non-extracted radioactivity was determined by combustion. The five fractions (whey) were pooled for partitioning. The whey was concentrated to about 800 mL at 35 °C. Thereafter, the whey was partitioned twice with dichloromethane, once with ethyl acetate and twice with acidic ethyl acetate (pH 1–2). The radioactivity in the milk fat was determined by combustion.

Liver and kidney

Study 643724: The tissue was homogenized and extracted once with acetonitrile, twice with acetonitrile/water (1 + 1, v/v), twice with methanol/water (1 + 1, v/v) and once with methanol/water (2 + 8, v/v). Additionally, acidic (2 × methanol/0.2 M HCl, 8 + 2, v/v) and basic extractions (methanol/0.5 M NH₄OH, 8 + 2, v/v) were performed. Radioactivity in each extract was measured after centrifugation. Thereafter, a Soxhlet extraction in methanol/water (8 + 2, v/v) for 16 hours was performed. Liver and kidney were further extracted with Tris buffer (0.1 M), incubated in pronase/Tris buffer and extracted with Tris buffer. The remaining non-extracted radioactivity was determined by combustion. For liver, hydrolysis was performed in 2 M HCl at 70 °C for approximately 16 hours.

Study 684088: The tissue was incubated in pronase/phosphate buffer for 6 h at 40 °C and then methanol was added to facilitate precipitation of the tissue. Thereafter, the tissue was extracted three times with methanol/water (1 + 1, v/v) and once with methanol/water (2 + 8, v/v). Radioactivity in each extract was measured after centrifugation. Thereafter, a Soxhlet extraction in methanol/water (1 + 1, v/v) at 70 °C for 16 hours was performed. The remaining non-extracted radioactivity was determined by combustion.

Muscle

Study 643724: The tissue was extracted (1–3 mL solvent per g tissue) once with acetonitrile, twice with acetonitrile/water (1 + 1, v/v), once with methanol/water (1 + 1, v/v), followed by a Soxhlet extraction in methanol/water (8 + 2, v/v) for 16 hours. Due to the low radioactivity amounts, the muscle extracts were not further worked up.

Study 684088: The tissue was homogenised for radioactivity determination. Due to low radioactivity amounts no extraction of muscle tissue was performed.

Fat, omental and perirenal

Study 643724: Samples were extracted twice with dichloromethane and once with dichloromethane/ethanol (1 + 1, v/v).

Study 684088: Samples were extracted twice with dichloromethane, once with dichloromethane/ethanol (1 + 1, v/v) and once again with dichloromethane. A further extraction with hexane was performed. The remaining tissue was extracted three times with methanol/water (1 + 1, v/v) and once with methanol/water (2 + 8, v/v). Thereafter, the residue was incubated with pronase/phosphate buffer for 6 h at 40 °C, three times extracted with methanol/water (1 + 1, v/v) and once with methanol/water (2 + 8, v/v). Thereafter, a Soxhlet extraction in methanol/water (1 + 1, v/v) at about 70 °C for 16 hours was performed.

Faeces

Studies 643724 and 684088: Samples collected from 24 to 48 hours and from 144 to 168 hours after the first administration were extracted once with acetonitrile, twice with acetonitrile/water (1 + 1, v/v) and three times with methanol/water (1 + 1, v/v), followed by a Soxhlet extraction in methanol/water (8 + 2, v/v). Extractions were performed as described for organs/tissues.

After extraction, the remaining solid was dried, and aliquots were combusted to obtain material balances of radioactivity for each examined sample. The extracts were partitioned further with different solvents and analysed by HPLC, TLC and LC-MS to determine the metabolic profiles. The TRR in mg/kg as parent equivalents in goat milk are shown in Table 4.

Table 4 TRR in goat milk following oral administration of [^{14}C]fenamidone for 7 days.

Time (hours) after		TRR ^a in mg/kg as parent eq		N-phenyl-label	
First admin.	Last admin.	C-phenyl-label		Low dose	High dose
0–8	8	< 0.001	0.008	0.003	0.012
8–24	16	0.001	0.012	0.003	0.014
24–32	8	0.001	0.018	0.004	0.025
32–48	16	0.001	0.015	0.004	0.020
48–56	8	0.001	0.018	0.005	0.015
56–72	16	0.002	0.016	0.006	0.019
72–80	8	0.001	0.018	0.005	0.022
80–96	16	0.002	0.016	0.005	0.015
96–104	8	0.001	0.018	0.004	0.020
104–120	16	0.001	0.017	0.005	0.016
120–128	8	0.001	0.016	0.005	0.013
128–144	16	0.002	0.016	0.004	0.015
144–152	8	0.001	0.020	0.004	0.019
152–168	16	0.001	0.016	0.004	0.014
168–174	22	0.001	0.011	0.002	0.006

^a Values corrected for background

The balance of the applied radioactivity and the excretion pattern of the lactating goats treated with radiolabelled fenamidone are presented in Table 5. At sacrifice, at both dose levels and with both labels, small amounts of radioactivity (totally 0.6 to 1.0% of the administered radioactivity) were detected in edible organs/tissues and blood. The ratio of residues in high dose to low dose animals was approximately 10, reflecting the ratio of the doses (see Table 6).

Table 5 Balance of radioactivity in goats following oral administration of [^{14}C]fenamidone for 7 days

Matrix	% TAR			
	C-phenyl-label		N-phenyl-label	
	Low dose	High dose	Low dose	High dose
Urine (0–174 hours)	26	17	40	36
Faeces (0–174 hours)	75	80	45	52
Milk (0–174 hours)	0.1	0.1	0.2	0.1
Cage wash	0.9	1.3	5.0	1.1
Total excreted	102	99	91	90
Total in edible tissues/organs	1.0	1.0	0.6	0.7
Gastro-intestinal tract (GIT)	2.9	2.3	1.1	0.2
Total	106	102	92	90

Table 6 TRR in goat tissues and blood following oral administration of [^{14}C]fenamidone for 7 days

Matrix	TRR (mg parent eq/kg) ^a			
	C-phenyl-label		N-phenyl-label	
	Low dose	High dose	Low dose	High dose
Liver	0.077	0.94	0.061	0.68
Kidney	0.013	0.12	0.007	0.08
Muscle	0.002	0.02	< 0.001	0.006
Fat	0.003	0.02	0.002	0.02

Matrix	TRR (mg parent eq/kg) ^a			
	C-phenyl-label		N-phenyl-label	
	Low dose	High dose	Low dose	High dose
Blood	0.005	0.09	0.009	0.054

^a Values corrected for background

The distribution of TRR in goat matrices following treatment at the high dose with [¹⁴C]fenamidone is summarised in Tables 7 (milk and fat) and 8 (liver and kidney). The majority of the TRR could be extracted using organic solvents, Soxhlet extraction, pronase incubation (liver and kidney) and hydrolysis (liver). The characterization and identification of metabolites was carried out with matrices of the high dosed goats only. Due to low TRR in muscle, no extraction of the tissue was performed in case of the N-phenyl-label study. In case of the C-phenyl-label study, the muscle was extracted but the extracts were not further worked up due to the low radioactivity amounts. The total extracted radioactivity amounted to 63.6% (0.01 mg eq/kg) of the TRR. The radioactivity remaining in the muscle was 36.4% of the TRR (0.006 mg eq/kg).

Table 7 Characterization and identification of radioactivity in goat milk and fat (high dose)

Component/fraction	Milk				Fat, omental and perirenal			
	C-phenyl-label		N-phenyl-label		C-phenyl-label		N-phenyl-label	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Fenamidone	0.001	0.7	ND	ND	0.013	53	ND	ND
RPA 409445	< 0.001	0.7	ND	ND	ND	ND	ND	ND
RPA 412708	0.001	1.7	ND	ND	ND	ND	ND	ND
RPA 412636	0.002	11.1	ND	ND	ND	ND	ND	ND
OH-fenamidone	0.001	1.0	0.001	1.9	ND	ND	ND	ND
OH-RPA 412708	< 0.001	1.0	ND	ND	ND	ND	ND	ND
Unidentified metabolites (number)	0.011 (16)	68 (16)	0.012 (13)	84 (13)	0.005 (9)	24 (9)	0.004 (4)	25 (4)
Sub-total	0.013	84	0.013	86	0.018	76	0.004	25
Extracted, but not characterized					0.001	6.4	0.016 ^b	75 ^b
Total extracted ¹⁴ C	0.013 ^a	84 ^a	0.013 ^a	86 ^a	0.019	83	0.02	100

^a Total in the whey

^b Assumed to be related to non-fat tissue

Table 8 Characterization and identification of radioactivity in goat liver and kidney (high dose)

Component/fraction	Liver				Kidney			
	C-phenyl-label		N-phenyl-label		C-phenyl-label		N-phenyl-label	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Fenamidone	0.003	0.3	ND	ND	0.001	0.6	ND	ND
RPA 409445	ND	ND	ND	ND	ND	ND	ND	ND
RPA 412708	ND	ND	ND	ND	0.001	0.9	ND	ND
RPA 412636	0.055	5.9	ND	ND	0.018	15	ND	ND
OH-fenamidone	0.011	1.2	0.039	5.6	0.004	4.0	0.004	4.3
OH-RPA 412708	0.004	0.4	ND	ND	0.003	2.9	ND	ND
Unidentified metabolites (number)	0.149 (10)	16 (10)	0.607 (5)	87 (5)	0.061 (9)	49 (9)	0.070 (7)	81 (7)
Sub-total	0.222	24	0.646	92	0.088	72	0.074	86
Extracted, but not characterized	0.062	6.7	0.013	1.9	0.011	8.2	0.003	3.2
After pronase incubation	0.439	47	ND	ND	0.021	18	ND	ND
After hydrolysis	0.148	16	ND	ND	ND	ND	ND	ND
Total extracted ¹⁴ C	0.87	93	0.66	94	0.12	98	0.077	89

Laying hens

Burri (1999) investigated the fate of fenamidone following repeated oral administration to the laying hen in two separate studies (643735, M-183883-01-1 and 684090, M-183837-01-1, see Table 3).

Two dose levels were used for each label position, with one group of five white leghorn hybrid hens being used per dose level and label. Two groups of hens were orally dosed daily by

capsule with [C-phenyl-UL-¹⁴C]-fenamidone at doses of 1.3 ppm and 13.8 ppm in the diet for fourteen consecutive days. Two other groups of hens were similarly dosed with [N-phenyl-UL-¹⁴C]-fenamidone at doses of 1.0 ppm and 9.8 ppm in the diet for fourteen consecutive days. The concentration profiles of radioactivity in eggs as well as the excretion patterns via excreta were followed. The hens were sacrificed at approximately 24 hours after the last (14th) dose and blood, plasma, liver, muscle, fat and skin were collected. The TRR were determined by LSC in liquid matrices after solubilisation, in solid matrices by combustion. The samples were prepared as follows:

Eggs

Study 643735: The egg samples (white and yolk) were de-proteinised by adding acetone (1 + 2 or 1 + 4, v/v). Following centrifugation, the supernatant was removed and the pellet extracted once with acetone. Further extraction steps with acetonitrile/water (1 + 1, v/v) and methanol/water (2 + 8, v/v) as well as a Soxhlet extraction in methanol/water (8 + 2, v/v) were performed. The egg yolk of the high dose group was further extracted with Tris buffer (0.1 M), incubated in pronase/Tris buffer and extracted with Tris buffer. The remaining non-extractable radioactivity was determined by combustion. Due to the low radioactivity amounts, the low dose egg yolk and egg white samples were not further worked up.

Study 684090: Egg white and egg yolk of eggs between 144 and 168 hours as well as 312 and 336 hours after the first administration was pooled per time point and group and radioactivity was re-determined. These pools were used for further work-up. Due to the low radioactivity amounts, the high dose egg white samples, the low dose egg yolk and egg white samples were not analysed. The egg yolk samples were de-proteinised by adding acetone (1 + 4, v/v). After shaking for 30 min at room temperature, the samples were left for 16 hours at about 4 °C. Following centrifugation (10 min at 1600 g), the supernatant was removed and the pellet extracted once with acetone. Further extraction steps with acetone/water (1 + 1, v/v) and methanol/water (1 + 1, v/v) as well as a Soxhlet extraction in methanol/water (1 + 1, v/v) were performed.

Muscle

Study 643735: The tissue was homogenised for radioactivity determination. Thereafter, the samples were pooled and extracted (high dose group only). The extractions were performed with acetonitrile, methanol and methanol/water in different ratios. Muscle tissue was extracted once with acetonitrile, twice with acetonitrile/water (1 + 1, v/v), twice with methanol/water (1 + 1, v/v) and once with methanol/water (2 + 8, v/v). Radioactivity in each extract was measured after centrifugation. Additionally, a Soxhlet extraction in methanol/water (8 + 2, v/v) for 16 hours was performed.

Study 684090: The tissue was homogenised for radioactivity determination. Due to low radioactivity amounts no extraction of muscle tissue was performed.

Liver

Study 643735: The tissue was homogenised for radioactivity determination, pooled per group and extracted. The extractions were performed with acetonitrile, methanol and methanol/water in different ratios. Liver tissue was extracted once with acetonitrile, twice with acetonitrile/water (1 + 1, v/v), twice with methanol/water (1 + 1, v/v) and once with methanol/water (2 + 8, v/v). Radioactivity in each extract was measured after centrifugation. Additionally, a Soxhlet extraction in methanol/water (8 + 2, v/v) for 16 hours was performed. The liver tissue of the high dose group was additionally extracted/incubated with Tris buffer and pronase.

Study 684090: The organs were homogenised for radioactivity determination. Thereafter, the liver tissue was pooled per group and extracted by incubation with pronase for 6 hours at 40 °C. Thereafter, the tissue was further extracted three times with methanol/water (1+1, v/v) and twice with methanol/water (2 + 8, v/v). Radioactivity in each extract was measured after centrifugation. Additionally, a Soxhlet extraction in methanol/water (1 + 1, v/v) at about 80 °C for 16 hours was performed. Finally, the residual sample was hydrolysed in 1 N HCl at 70 °C for approximately 16 hours.

Fat and skin

Study 643735: The tissues were extracted as follows: twice with dichloromethane, once with dichloromethane/acetone (1 + 1, v/v), once with acetone, twice with acetonitrile/water (1 + 1, v/v). Additionally, the skin sample was extracted with methanol/water (2 + 8, v/v) followed by a Soxhlet extraction in methanol/water (8 + 2, v/v).

Study 684090: The tissue was extracted as follows: twice with dichloromethane, twice with dichloromethane/ethanol (1 + 1, v/v), three time with methanol/water (1 + 1, v/v) and once with methanol/water (2 + 8, v/v). Thereafter, the tissue was extracted by incubation with pronase for approximately 16 hours at 40 °C. Thereafter, the tissue was further extracted twice with methanol/water (1 + 1, v/v).

Excreta

Individual excreta samples from the respective time intervals were homogenised in water (1 + 1, w/v) and the radioactivity determined in subsamples by combustion.

Studies 643735 and 684090: The excreta samples collected from 96 to 120 hours and from 312 to 336 hours after the first administration were pooled by time point and by group and were extracted once with acetonitrile, twice with acetonitrile/water (1 + 1, v/v), with methanol/water and water, followed by a Soxhlet extraction in methanol/water (8 + 2, v/v).

After extraction, the remaining solid was dried, and aliquots were combusted to obtain material balances of radioactivity for each examined sample. The extracts were partitioned further with different solvents and analysed by HPLC and as far as appropriate by TLC and LC-MS to determine the metabolic profiles. The TRRs in mg/kg (as parent equivalents) in eggs after administration of radiolabelled fenamidone are shown in Table 9. At the plateau after 9 days, the residues in egg yolk were about 7 times (C-label) higher than in the egg white.

Table 9 TRR in eggs following oral administration of [¹⁴C]fenamidone for 14 days.

Day	TRR ^a in mg/kg as parent eq							
	C-phenyl-label				N-phenyl-label			
	Low dose		High dose		Low dose		High dose	
	White	Yolk	White	Yolk	White	Yolk	White	Yolk
1	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
2	0.001	0.001	0.004	0.009	< 0.001	< 0.001	0.005	0.002
3	0.001	0.003	0.008	0.021	0.001	0.001	0.005	0.023
4	0.001	0.007	0.011	0.043	0.001	0.003	0.005	0.045
5	0.001	0.008	0.012	0.077	0.001	0.005	0.004	0.072
6	0.002	0.010	0.016	0.095	0.001	0.007	0.004	0.090
7	0.002	0.009	0.018	0.105	0.001	0.008	0.004	0.098
8	0.001	0.010	0.015	0.120	0.001	0.008	0.003	0.100
9	< 0.001	0.010	0.016	0.119	0.001	0.009	0.004	0.097
10	0.001	0.010	0.020	0.122	0.001	0.009	0.004	0.097
11	0.002	0.012	0.018	0.129	0.001	0.009	0.004	0.087
12	0.003	0.013	0.018	0.131	0.001	0.009	0.005	0.094
13	0.002	0.013	0.019	0.135	0.001	0.009	0.006	0.098
14	0.001	0.011	0.020	0.144	0.001	0.009	0.005	0.099

^a Mean of 5 birds, values corrected for backgrounds obtained from untreated hens

As shown in Table 10, the overall recoveries of radioactivity were > 91% of the total applied radioactivity (TAR) for the low and high dose groups, respectively. The majority of the radioactivity was in excreta (approximately 90% TAR). All eggs together contained 0.1% of the dose administered, most of which was retained in the yolk. At sacrifice, at both dose levels and in both labels, negligible amounts of radioactivity (0.1% of TAR) were detected in edible organs/tissues and blood. The ratio of residues in high dose to low dose animals ranged from 5 to 16 (see Table 11). The major amounts of radioactivity in the excreta could be extracted. Table 13 shows the results for the low dose (C-phenyl label only) and the high dose samples (both labels).

Table 10 Balance of radioactivity following oral administration of [^{14}C]fenamidone for to laying hens

Matrix	% TAR ^a			
	C-phenyl-label		N-phenyl-label	
	Low dose	High dose	Low dose	High dose
Excreta, 0–336 hours	91	89	91	94
Cage wash	1.3	2.0	2.0	1.5
Total excreted	92	91	93	96
Eggs, 0–336 hours	0.1	0.1	0.1	0.1
Total in edible tissues/organs ^b	< 0.1	< 0.1	< 0.1	< 0.1
Total	92	91	93	96

^a Mean of 5 animals^b Liver, muscle, fat, skin, blood, developing eggsTable 11 TRR in hen tissues and blood following oral administration of [^{14}C]fenamidone for 14 days

Matrix	TRR (mg parent eq/kg) ^a			
	C-phenyl-label		N-phenyl-label	
	Low dose	High dose	Low dose	High dose
Liver	0.013	0.160	0.005	0.051
Muscle	< 0.001	0.010	< 0.001	0.002
Fat	< 0.001	0.014	< 0.001	0.005
Skin	0.002	0.019	0.001	0.016
Blood	0.004	0.049	0.002	0.022
Plasma	0.002	0.021	< 0.001	0.006
Developing eggs (white)	0.001	0.016	0.001	0.005
Developing eggs (yolk)	0.013	0.149	0.009	0.095

^a Mean of 5 animals, values corrected for backgroundTable 12 Extraction of radioactivity from excreta following oral administration of [^{14}C]fenamidone

Time, hours	C-phenyl-label				N-phenyl-label	
	Low dose		High dose		High dose	
	% TRR	% TAR	% TRR	% TAR	% TRR	% TAR
96–120	83	6.1	83	5.5	85	6.1
312–336	80	6.1	85	5.8	80	5.7

In the excreta extracts of the C-phenyl-label groups, at least sixteen radioactive fractions were found, in amounts ranging from < 0.1% to 1.9% of the TAR (low dose) and < 0.1% to 1.8% (high dose). Fenamidone was detected in small amounts (0.1–0.2% TAR low and high dose). RPA 409445, RPA 412708, RPA 412636 and hydroxy-RPA 412708 were found in minor amounts (< 0.1 to 0.6% TAR). Hydroxy-fenamidone was found in amounts of 1.3 to 1.9% of the TAR. Unknown fractions occurred at levels up to 1.6% TAR. The excreta extracts from the N-phenyl-label application (high dose only) showed at least nine radioactive fractions ranging from < 0.1% to 1.6% of the TAR. The parent compound occurred in a minor amount of 0.1% of the TAR. Hydroxy-fenamidone was found in a maximal amount of 1.1%. Unknown metabolite fractions occurred in maximal amounts of 1.6% of the radioactivity administered.

The characterization of the TRR and identification of metabolites (components) in eggs and edible tissues was carried out for the high dose samples only. The findings are summarized in Table 13 for egg white and egg yolk and in Table 14 for liver, fat and skin.

Table 13 Characterization and identification of radioactivity in eggs (high dose)

Component/fraction	Egg white		Egg yolk							
	C-phenyl-label (312–336 h)		C-phenyl-label (96–120 h)		N-phenyl-label (144–168 h)		C-phenyl-label (312–336 h)		N-phenyl-label (312–336 h)	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Fenamidone	ND	ND	ND	ND	ND	ND	0.014	11	ND	ND
RPA 409445	ND	ND	0.004	3.4	ND	ND	0.003	2.7	ND	ND
RPA 412708	0.001	2.9	0.028	25	ND	ND	0.022	17	ND	ND
RPA 412636	0.011	74	0.008	8.3	ND	ND	0.014	11	ND	ND

Component/fraction	Egg white		Egg yolk							
	C-phenyl-label (312–336 h)		C-phenyl-label (96–120 h)		N-phenyl-label (144–168 h)		C-phenyl-label (312–336 h)		N-phenyl-label (312–336 h)	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
OH-fenamidone	ND	ND	0.007	6.8	0.005	5.1	0.008	7.0	0.013	12
OH-RPA 412708	ND	ND	0.004	2.8	ND	ND	0.002	2.0	ND	ND
Metabolites unidentified (n)	0.001 (1)	7.6 (1)	0.04 ^a (9)	37 (9)	0.051 ^b (2)	48 (2)	0.037 ^c (8)	31 (8)	0.045 ^d (2)	44 (2)
Sub-total	0.013	84	0.091	83	0.056	53	0.1	81	0.058	56
Non-characterized	0.002	16	0.006	5.1			0.008	6.6		
Soxhlet extract	–	–	–	–	0.011	11	–	–	0.012	12
Hexane phase	–	–	–	–	0.001	1.3	–	–	0.002	1.7
Aqueous phase	–	–	–	–	0.021	20	–	–	0.015	15
Pronase incubation	ND	ND	0.015	12.7	–	–	0.019	15	–	–
Total extracted ¹⁴ C	0.015	100	0.11	101	0.09	85	0.13	102	0.09	85

– Not carried out

ND not detected

^a range: 0.001–0.01 mg eq/kg^b range: 0.009–0.042 mg eq/kg^c range: 0.001–0.015 mg eq/kg^d range: 0.002–0.043 mg eq/kg

Table 14 Characterization and identification of radioactivity in tissues (high dose)

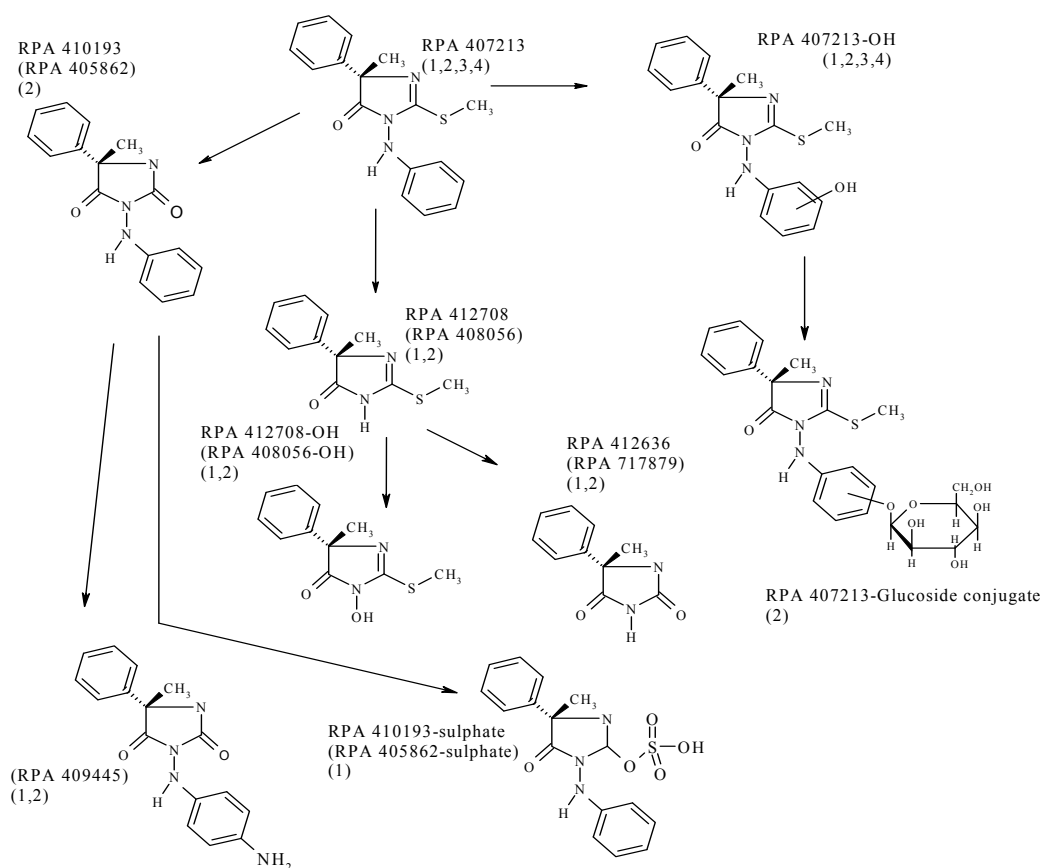
Component/fraction	Liver				Fat		Skin	
	C-phenyl-label		N-phenyl-label		C-phenyl-label		C-phenyl-label	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Fenamidone	0.003	1.7	ND	ND	< 0.001	4.6	0.002	14
RPA 409445	ND	ND	ND	ND	0.003	26	ND	ND
RPA 412708	0.002	1.3	ND	ND	ND	ND	ND	ND
RPA 412636	0.023	15	ND	ND	ND	ND	0.002	16
OH-fenamidone	ND	ND	ND	ND	ND	ND	ND	ND
OH-RPA 412708	< 0.001	0.3	ND	ND	ND	ND	ND	ND
Fenamidone Glu ^a	0.009	5.8	ND	ND	ND	ND	ND	ND
Unidentified metabolites (n)	0.072 ^b (6)	47 (6)	0.035 ^c (2)	68 (2)	0.006 (1)	60 (1)	0.006 (2)	38 (2)
Subtotal	0.11	72	0.035	68	0.009	90	0.01	68
Non-characterized	0.013	9.0			0.001	6.5	0.003	22
Soxhlet extracted	–	–	0.002	4.2	–	–	–	–
Hydrolysis extract	–	–	0.001	1.2	–	–	–	–
Hexane phase	–	–	0.000	0.4	–	–	–	–
Aqueous phase	–	–	ND	ND	–	–	–	–
Organic phase	–	–	0.01	21	–	–	–	–
Pronase incubation	0.027	18	–	–	ND	ND	ND	ND
Total extracted ¹⁴ C	0.15	98	0.048	94	0.01	97	0.013	90

– Not carried out

ND not detected

^a Fenamidone glucoside^b range: < 0.001–0.057 mg eq/kg^c range: 0.002–0.033 mg eq/kg

Fenamidone is extensively metabolised in animals. The metabolic profiles differ quantitatively between the species investigated, but qualitatively there are no major differences. The routes and metabolites in animals are consistent across the studies. Figure 1 presents the proposed metabolic pathway for fenamidone in livestock.



- (1) In goats dosed with [C-phenyl-UL-¹⁴C]-fenamidone
 (2) In hens dosed with [C-phenyl-UL-¹⁴C]-fenamidone
 (3) In goats dosed with [N-phenyl-UL-¹⁴C]-fenamidone
 (4) In hens dosed with [N-phenyl-UL-¹⁴C]-fenamidone

Figure 1 Proposed metabolic pathway of fenamidone in livestock.

Metabolite codes in parentheses are the numbers for the racemic compounds, as used in the original study report. The metabolites have been shown to be present as the S-enantiomers.

Plant metabolism

The metabolism of fenamidone has been studied in grapes, tomatoes, lettuce, carrots and potatoes. The test material used in the studies is characterized in Table 15.

Table 15 Characterization of radio-labelled test material

Crop	[C-phenyl-UL- ¹⁴ C]-fenamidone			[N-phenyl-UL- ¹⁴ C]-fenamidone		
	Report, Bayer reference	Radiochem. Purity ^a , %	Spec. activity, MBq/mg	Report, Bayer reference	Radiochem. Purity ^a , %	Spec. activity, MBq/mg
Grape	655266, M-183815-01-1	≥ 98.7	1.25	—	—	—
Tomato	10616, M-173256-01-1	99.6	2.51	10616, M-173256-01-1	99.7	2.51
Lettuce	10617, M-173236-01-1	98.5	2.53	10617, M-173236-01-1	98.6	2.55
Potato	10615, M-173249-02-1	99.6 ^b 100 ^c	2.50 ^b 3.27 ^c	10615, M-173249-02-1	99.7 ^b 100 ^c	2.53 ^b 2.95 ^c
Carrot	—	—	—	MEF-06/243 M-275069-02-1	99	3.7

^a by HPLC, TLC

^b Outdoor experiment

^c Indoor experiment

Grapes

The metabolism of fenamidone in grapes was investigated by Voelkel in 1999 (655266, M-183815-01-1). C-phenyl-¹⁴C-fenamidone alone was used in this study. Grape vine plants (Pinot Noir variety) were treated with four foliar applications of C-phenyl-¹⁴C-fenamidone during the course of berry growth and maturation to a total application of 1.65 kg ai/ha. The details of grape vine treatment are given in Table 16.

Table 16 Timing and details of grape vine treatments with [¹⁴C]fenamidone

Treatment	Rate, g ai/ha	Timing	Growth stage
1	505	136 days before harvest	Before flowering Flower buds starting to separate
2	485	100 days before harvest, 36 days following first treatment	80% flower hoods fallen, Young fruits starting to swell
3	504	78 days before harvest, 58 days following first treatment	Well-formed bunches start to hang
4	156	24 days before harvest, 112 days following first treatment	Beginning of maturation

Before treatment the soil surface around the base of each vine as well as the adjoining vine plants were protected with plastic sheeting so as to prevent loss of the radioactive material into the surrounding environment and, therefore, potential uptake by the roots of the vine plants. At harvest, 24 days following the last treatment, all remaining grape bunches on the vines were removed. Prior to processing, all bunches were washed using water, followed by aqueous methanol and finally methanol. The grapes were then removed from the stems and then, following chilling to reduce enzyme activity, separated into skin, pips and pulp. All samples were assayed for TRR by LSC, either directly or following processing and sample combustion.

The individual components of the grape bunches were subjected to a succession of solvent partition and extraction procedures culminating in enzyme treatment followed by harsh hydrolytic conditions, the last being used to investigate the small amounts of unextracted radioactive residue. The extracts obtained were subjected to TLC as well as HPLC in order to investigate the extracted components of the residue. Selected extracts were then analysed using LC/MS to obtain structural information with regard to those components resolved by the chromatography used. The distribution of radioactivity in the washes and various grape bunch parts and for the grapes for the pre-harvest and for the mature harvest samples is shown in Table 17.

Table 17 Distribution of radioactive residues in the pre-harvest and mature grapes

Sampling date	Pre-harvest grape bunches (prior of last application)				Mature harvest grape bunches (24 days after fourth treatment)			
Samples	Whole bunches Incl. washes & stems		Grapes Without washes & stems		Whole bunches Incl. washes & stems		Grapes Without washes & stems	
	mg/kg ^a	% TRR	mg/kg ^a	% TRR	mg/kg ^a	% TRR	mg/kg ^a	% TRR
Surface wash	0.79	45	–	–	0.40	34	–	–
Stems	0.27	16	–	–	0.22	19	–	–
Skin	0.28	16	0.3	41	0.25	21	0.27	44
Pulp	0.295	17	0.32	44	0.27	22	0.29	47
Pips	0.11	6.3	0.12	16	0.048	4.1	0.052	8.6
Total	1.7	100	0.74	100	1.2	100	0.61	100

^a mg parent eq/kg

Similar results for the distribution of radioactivity were obtained for the pre-harvested grapes and the mature harvest grapes. The major part of the radioactivity associated with the grape bunches could be extracted and represented 89.1% of the TRR (including the washes) for mature harvest grape bunches. The non-extracted radioactivity was confined mainly to the stem, skin and pips and some effort was made to release it for further analysis. The procedures used included enzyme treatment, followed by acid and alkali hydrolysis. Table 18 shows the metabolites identified and components resolved in mature harvest grapes from vines treated four times to a total of a nominal 1.65 kg fenamidone/ha. The extracted radioactivity from each grape bunch part as well as the washes were

separately analysed by HPLC. Parent compound was the only component detected in the washings demonstrating the stability of fenamidone on the surface of the grape bunches when exposed to field conditions. Results for the stem, skin, pips and pulp extracts showed the presence of two major components, which were indicated to be parent compound and the metabolite RPA 410193. In addition to fenamidone and RPA 410193 up to eleven additional radioactive components were detected in the plant fractions. Individually these radioactive components did not exceed 4.2% of the TRR in the mature grapes. Despite this, attempts were made to obtain additional information regarding the structure of two of the components in the most abundance (M1 at 4.2% TRR and M2 at 3.4% of the TRR). From the evidence obtained, it is suggested that M1 may be hydroxylated RPA 410193, and M2 may be hydroxylated parent compound. In both cases it was reasonable to assume that the hydroxylation has taken place in the amino-phenyl ring and that the basic ring structure of the parent compound remained intact.

Table 18 Metabolites identified and components resolved in mature harvest grapes matrices.

Component	Stems		Skin		Pips		Pulp		Washes		Total	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Extracted												
Parent (RPA 407213)	0.095	8.0	0.061	5.1	0.004	0.3	0.098	8.2	0.404	34.0	0.662	56
RPA 410193	0.018	1.5	0.065	5.4	0.014	1.2	0.107	9.0	ND	ND	0.204	17
M1 (RPA 410193-OH) ^a	0.009	0.8	0.023	1.9	0.003	0.2	0.015	1.2	ND	ND	0.050	4.2
M2 (RPA 407213-OH) ^a	0.012	1.0	0.018	1.5	0.001	0.1	0.009	0.8	ND	ND	0.040	3.4
M3 ^b	0.005	0.5	0.015	1.2	ND	ND	0.018	1.5	ND	ND	0.038	3.2
M4	0.009	0.8	ND	ND	0.001	0.1	ND	ND	ND	ND	0.010	0.9
M5	< 0.001	< 0.1	ND	ND	ND	ND	ND	ND	ND	ND	< 0.001	< 0.1
M6	ND	ND	0.011	1.0	0.001	0.1	0.006	0.5	ND	ND	0.029	2.4
M7	0.003	0.2	0.009	0.7	ND	ND	ND	ND	ND	ND	0.011	0.9
M8	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
M9	0.002	0.2	ND	ND	ND	ND	0.003	0.3	ND	ND	0.005	0.5
M10	0.003	0.2	ND	ND	ND	ND	ND	ND	ND	ND	0.003	0.2
M11	< 0.001	< 0.1	0.003	0.2	ND	ND	0.005	0.4	ND	ND	0.008	0.6
Extracted Subtotal	0.166	14	0.205	17	0.024	2.0	0.261	22	0.404	34	1.060	89
Non-extracted Subtotal	0.056	4.67	0.045	3.79	0.024	2.05	0.005	0.039	< 0.001	0.00	0.13	10.9
Released after protease treatment	0.008	0.68	0.004	0.30	< 0.001	0.04	0.001	< 0.11	NA	NA	0.013	1.1
Released after 'harsh' extractions ^c	0.006	0.54	0.003	0.23	< 0.001	0.03	0.002	0.13	NA	NA	0.011	0.9
Released after hydrolysis ^d	0.01	0.84	0.007	0.62	0.01	0.83	0.002	0.17	NA	NA	0.029	2.5
Non-released Radioactivity	0.031	2.61	0.031	2.64	0.014	1.16	< 0.001	0.03	NA	NA	0.077	6.5
Total	0.22	19	0.25	21	0.048	4.1	0.27	22	0.40	34	1.2	100

^a Tentative identity

^b Possibly comprising two components

^c Solids successively suspended in 0.1 M HCl and 0.1 N NaOH

^d Solids successively refluxed with 6 M HCl and 6 M NaOH

ND not detected (or below the LOQ) NA not applicable

The metabolism of fenamidone in field grown grapes is mainly characterised by the formation of RPA 410193, i.e. loss of the thiomethyl group and formation of an imidazolidinedione. Some evidence was obtained to suggest that hydroxylation of parent compound as well of the major metabolite, RPA 410193 also occurred. The number of other metabolites detected, albeit at very low levels, demonstrated the further metabolism of fenamidone. With regard to the TRR in the grapes in excess of 80% was accounted for in terms of identified and tentatively identified metabolites. Figure 2 shows the proposed metabolic pathway for fenamidone in grapes.

Fenamidone

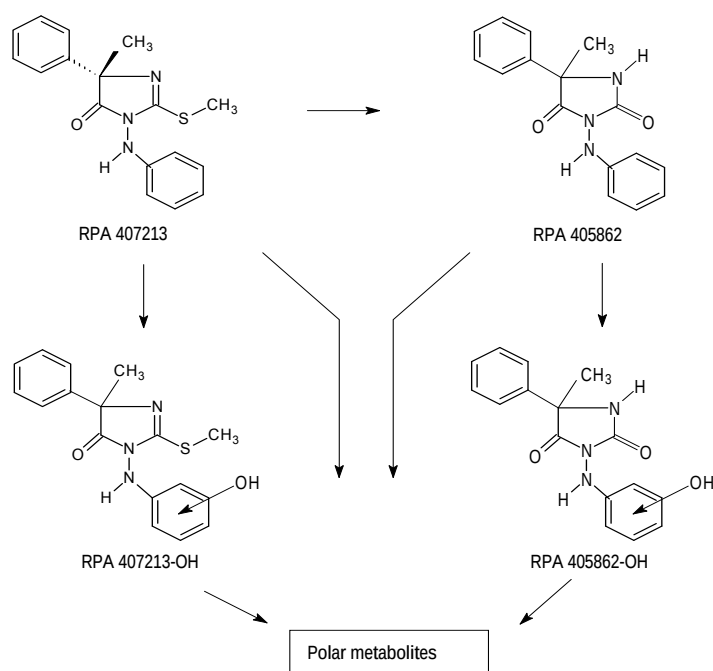


Figure 2 Proposed metabolic pathway for fenamidone (RPA 407213) in grapes

Metabolite codes in this pathway are the numbers for the racemic reference compounds, as used in the original study report. In an additional study, it has been shown that all metabolites and degradation products were formed as the S-enantiomers.

Tomato

The metabolism of fenamidone in tomatoes was investigated by Oliver in 1999 (10616, M-173256-01-1) on the variety “Gardeners Delight” following treatment with [^{14}C]fenamidone at a cumulative rate equivalent to 1.5 kg ai/ha. This was applied in three foliar spray treatments, each of 0.5 kg ai/ha, during the course of fruit growth. Untreated control plants were grown among the treated plants and also at some distance from the treated plants. Both labels, C-phenyl and N-phenyl, were used separately to study the metabolism of the molecule. For each treatment fenamidone was formulated as a 10% EC. The crop was grown to maturity under glass and was sampled at two interim time points and at maturity. There was an interval of 7 days between the last application and harvest. Following harvest, the fruits were picked, weighed fresh, rinsed with acetonitrile and then frozen prior to further analysis. The frozen fruits were then homogenised and extracted with acetonitrile. The rinses and extracts were concentrated prior to analysis by HPLC in order to establish the quantitative and qualitative nature of the residue. Selected representative extracts were cleaned-up using C_{18} solid phase extraction cartridges and analysed using LC-MS/MS to obtain confirmation of the identities indicated by HPLC.

The distribution of radioactivity and the calculated TRR for the treated plants are shown in Table 19. For both labels, extraction efficiencies of 87.1% and 91.8% were achieved. The components of the TRR at final harvest are summarised in Tables 20.

Table 19 Characterisation of radioactivity in tomato fruits 7 days after 3rd treatment

^{14}C -Label	Acetonitrile rinses		Acetonitrile extracts		Unextracted		TRR
	mg/kg ^a	% TRR	mg/kg ^a	% TRR	mg/kg ^a	% TRR	
C-Phenyl	0.06	31	0.105	57	0.024	13	0.19
N-Phenyl	0.085	41	0.105	51	0.017	8.2	0.21

^a mg parent eq/kg

Table 20 Identification of residues in [^{14}C]fenamidone treated tomatoes

Component	C-phenyl label		N-phenyl label	
	mg parent eq/kg	% TRR	mg parent eq/kg	% TRR
Polar unknowns ^a	0.017 (6)	9.7	0.009 (4)	4.7
Fenamidone	0.12	66	0.16	76
RPA 410193	0.017	9.4	0.019	9.3
RPA 407599	0.004	2.3	0.004	2.1
Non-polar unknowns	< 0.001	0.2	< 0.001	0.1
Unextracted	0.024	13	0.017	8.2
Total	0.185	100	0.21	100

^a Numbers in brackets indicate the number of components present.

The metabolic profile obtained with each label was similar. Parent fenamidone was the major component present, with significant amounts of RPA 410193 along with a number of minor components. A metabolic pathway for fenamidone metabolism in tomato is proposed in Figure 3.

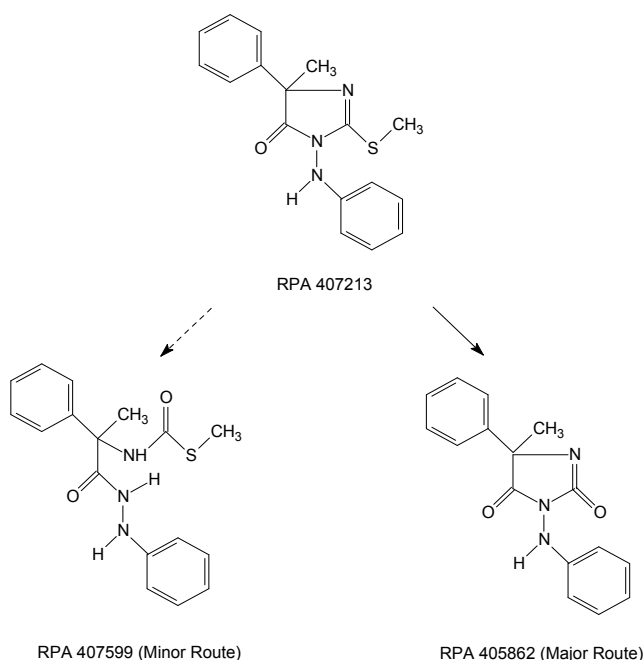


Figure 3 Proposed metabolic pathway for fenamidone (RPA 407213) in tomato

Metabolite codes in this pathway are the numbers for the racemic reference compounds, as used in the original study report. In an additional study, it has been shown that all metabolites and degradation products were formed as the S-enantiomers.

Lettuce

The metabolism of fenamidone on iceberg lettuce was investigated by Oliver and Clarke in 1999 (10617, M-173236-01-1) using [^{14}C]fenamidone labelled on two positions. Fenamidone was applied to lettuces in a 10% EC formulation. Four foliar spray applications were made at the nominal field rate of 0.4 kg ai/ha, the total treatment rates achieved over four applications were 1.4 kg ai/ha for the C-phenyl and the N-phenyl treated lettuces. The plants were grown to maturity outdoors, under ambient environmental conditions. After final harvest, 7 days after the fourth application, the outer wrapper leaves were separated from the lettuce heads. The wrapper leaves were rinsed with acetonitrile. Both plant parts were frozen, then homogenised and extracted by maceration with acetonitrile, methanol/water and acetonitrile/water. Aliquots of the dry extracted residue were combusted to determine the amount of unextracted radioactivity.

Table 21 shows the TRR in the different plant parts at harvest, seven days after 4th application. The acetonitrile rinses and combined plant extracts were subjected to quantitative and qualitative analysis by reverse-phase HPLC and normal phase TLC. Metabolite identification was confirmed by LC-MS. The principal components of the TRR are given in Table 22.

Table 21 Distribution of radioactivity in lettuce at final harvest 7 days after 4th treatment

¹⁴ C-Label	Plant part	TRR, mg/kg	Rinse, %	Extracted, %	Unextracted, %
	Wrapper leaves	12	9.2	86	4.5
C-phenyl	Heads	0.29	NA	94	6.5
	Whole lettuce	9.3	9.1	86	4.5
	Wrapper leaves	12	13	81	6.0
N-phenyl	Heads	0.21	NA	94	6.5
	Whole lettuce	9.0	12.7	81	6.0

NA: not applicable

Table 22 Identification of residues at final harvest in lettuces

Component	C-phenyl label			N-phenyl label		
	Heads, mg/kg (%TRR)	Wrapper leaves mg/kg (%TRR)	Whole lettuce, mg/kg (%TRR)	Heads mg/kg (%TRR)	Wrapper leaves, mg/kg (%TRR)	Whole lettuce mg/kg (%TRR)
Polar multi-component	0.043 (15)	0.36 (2.9)	0.28 (3.0)	0.04 (17)	0.24 (2.1)	0.19 (2.1)
Fenamidone	0.21 (70)	11 (92)	8.6 (92)	0.16 (73)	11 (91)	8.2 (91)
RPA410193	0.008 (0.26)	0.08 (0.65)	0.06 (0.66)	0.007 (3.2)	0.07 (0.57)	0.053 (0.59)
RPA412636	0.008 (2.7)	ND	0.002 (0.02)	ND	ND	ND
Glucoside conjugates ^a	0.008 (2.9)	ND	0.002 (0.02)	ND	ND	ND
Unextracted	0.02 (6.5)	0.56 (4.5)	0.42 (4.5)	0.014 (6.5)	0.69 (6.0)	0.54 (6.0)

^a RPA 412708 and fenamidone glucoside conjugate eluted from HPLC column with same retention time

The metabolic profile obtained with each label was similar. The major component in each crop part was parent fenamidone. The remaining extracted radioactivity was comprised of RPA 410193 and multiple unidentified polar components. In addition, trace levels of RPA 412636 and a glucoside conjugate of fenamidone were present in extracts from the C-phenyl-label treatment. A metabolic pathway for fenamidone metabolism in lettuce is proposed in Figure 4.

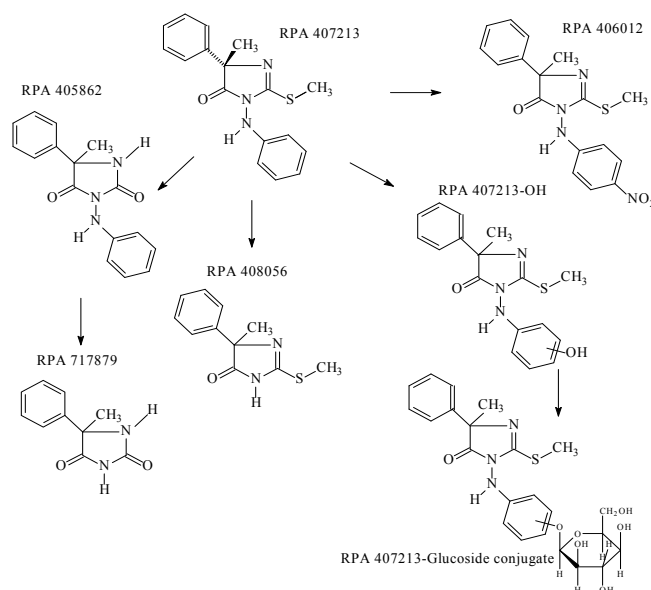


Figure 4 Proposed metabolic pathway for fenamidone (RPA 407213) in lettuce

Metabolite codes in this pathway are the numbers for the racemic reference compounds, as used in the original study report. In an additional study, it has been shown that all metabolites and degradation products were formed as the S-enantiomers.

Potato

An outdoor study was conducted by Clark in 1999 on potatoes (10615, M-173249-02-1) using N-phenyl and C-phenyl-[^{14}C]fenamidone in order to investigate plant metabolism. Three foliar spray applications were made each at the nominal field rate of 0.5 kg ai/ha. The total treatment rates achieved over three applications were 1.30 kg ai/ha and 1.36 kg ai/ha for the C-phenyl and N-phenyl label respectively. The potatoes were grown to maturity outdoors, under ambient environmental conditions. After final harvest, 14 days after the third application, the haulm was separated from the tubers. The tubers were rinsed with water and peeled. All plant parts were frozen, then homogenised and extracted by maceration with acetonitrile, acetonitrile/water and methanol. Aliquots of the dry extracted residue were combusted to determine the amount of unextracted radioactivity. The TRR are given in Table 23. Following concentration, the combined plant extracts were subjected to quantitative and qualitative analysis by HPLC and TLC, employing certified reference standards as retention time and relative front markers. The compound identification was confirmed by LC-MS/MS for parent, RPA 412636 and RPA 412708. The principal components of the TRR are given in Table 24. All concentration data are given in mg parent eq/kg.

Table 23 Potato TRR at final harvest, 14 days after 3rd application.

^{14}C -Label	Plant part	TRR, mg/kg	% Extracted	% Unextracted
C-phenyl	Tubers (peeled)	0.08	76	24
	Peel	0.12	66	34
	Whole tubers	0.09	73	27
	Haulm	6.6	77	23
	Tubers (peeled)	0.06	46	55
N-phenyl	Peel	0.032	48	53
	Whole tubers	0.038	46	54
	Haulm	5.9	78	22

Table 24 Distribution of metabolites in mature potatoes treated by ^{14}C -labelled fenamidone

Component/fraction	C-phenyl label							
	Peeled tuber		Peel		Whole tuber		Haulm	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Fenamidone	< 0.001	0.2	0.008	6.7	0.002	2.3	3.4	51
RPA412708 ^a	0.005	6.4	0.007	5.8	0.006	6.4	0.13	2.0
RPA410193	ND	–	0.001	0.8	< 0.001	0.3	0.062	0.9
RPA412636 ^a	0.004	5.5	0.009	7.4	0.005	6.3	ND	–
Unknown 1	ND	–	0.009	7.4	0.002	2.5	ND	–
Unretained metabolites (polar unknowns)	0.037	46	0.027	23	0.034	40	1.5	22
Retained metabolites (non-polar unknowns + RPA 409446)	ND	–	0.001	2.1	< 0.001	0.7	ND	–
Procedural losses	0.014	17	0.014	12	0.014	16	< 0.001	–
Unextracted radioactivity	0.019	24	0.040	34	0.023	27	1.5	23
Total	0.08	100	0.12	100	0.09	101	6.6	100
	N-phenyl label							
	Peeled tuber		Peel		Whole tuber		Haulm	
Fenamidone	ND	–	0.004	12	0.002	5.8	4.1	69
RPA 410193	ND	–	< 0.001	0.3	< 0.001	0.2	0.06	1.0
Unknown 1	ND	–	0.001	4.4	0.001	2.1	ND	–
Unretained metabolites (polar unknowns)	0.021	34	0.007	21	0.012	31	0.46	7.7
Retained metabolites (non-polar unknowns + RPA 409446)	ND	–	0.002	6.4	0.001	3.1	ND	–
Procedural losses	0.007	12	0.001	3.1	0.003	8.5	< 0.001	< 0.1
Unextracted radioactivity	0.033	55	0.017	53	0.020	54	1.3	22
Total	0.06	101	0.032	100	0.04	105	5.9	100

^a C-phenyl specific metabolites

An additional indoor experiment was carried out in order to determine the nature of the major unidentified polar component of the potato tuber TRR which was unretained on the initial reversed-phase HPLC. Two post emergence applications, one of C-phenyl-[^{14}C : ^{13}C]fenamidone (4:1) at a treatment rate of 1.6 kg ai/ha, the other of N-phenyl-[^{14}C : ^{13}C]fenamidone (4:1) at a treatment rate of 1.4 kg ai/ha were made to separate groups of potato plants grown in pots under greenhouse conditions. The treatment solutions were applied to the haulm (cut stem, mature and immature leaves) and at the soil surface surrounding the base of the stem to try to maximise the incurred TRR, and reproduce the field situation as far as practicable. These plants were grown to maturity and then harvested. The resultant potato tubers were rinsed with water and homogenised. Representative subsamples were extracted by maceration with acetone/water. The TRR for greenhouse grown potato tubers are summarised in Table 25.

As previously, the TRR for greenhouse grown tubers was also low. HPLC analysis of extracts from greenhouse grown potato tubers was carried out. These were shown to have similar profiles to those previously obtained from field grown potato tubers, for both labels. The C-phenyl-[^{14}C , ^{13}C]fenamidone treated tuber extract was subjected to solid phase extraction (SPE) to separate the polar residue from the parent compound. The appropriate fractions were concentrated and analysed using a second HPLC method to retain and resolve individual polar components. This method resolved the previously unretained material, corresponding to that which accounted for 39.5% TRR (0.034 mg/kg) in extracts derived from C-phenyl-fenamidone treated field grown potato tubers, into nine discrete entities (Table 26) confirming its multicomponent nature.

Table 25 TRR in whole potato tubers (grown indoors)

Labels	TRR, mg parent eq/kg	TRR extracted, %	TRR unextracted, %
C-Phenyl-[^{14}C , ^{13}C]	0.023	33	67
N-Phenyl-[^{14}C , ^{13}C]	0.006	25	75

Table 26 Composition of polar material in C-phenyl treated potato tubers

Tubers	Concentration, mg parent eq/kg									
	P1	P2	P3	P4	P5	P6	P7	P8	P9	Total
Greenhouse ^a	< 0.001	0.001	0.001	< 0.001	0.001	< 0.001	< 0.001	< 0.001	< 0.001	0.005
Field grown	0.001	0.008	0.008	0.002	0.006	0.001	0.001	0.002	0.004	0.034

^a Treated with C-phenyl-[^{14}C : ^{13}C]fenamidone (4 : 1)

P1–P9: Individual polar fractions

Additional analysis indicated that P3 and P4 are likely to be RPA 412636 conjugates, which hydrolyse to release free RPA 412636. Thus, a further 0.01 mg/kg (11.5% TRR) of the C-phenyl-fenamidone treated field grown potato tubers extractable radioactive residue was characterised as being conjugated RPA 412636. The total amount of RPA 412636 found in treated potato tuber extracts as free or conjugated material was 0.015 mg/kg (17.8% TRR).

The N-phenyl-[^{14}C , ^{13}C]fenamidone treated tuber extract concentrate was also analysed on the second HPLC system. The resulting chromatogram showed the presence of four peaks of polar origin. Therefore, the polar material, which accounted for 30.8% TRR (0.012 mg/kg) in extracts derived from N-phenyl-fenamidone treated field grown potato tubers, is postulated to be composed of at least four discrete entities, as presented in Table 27.

Table 27 Composition of polar material in N-phenyl treated potato tubers

Sample	Concentration, mg parent eq/kg				
	PA	PB	PC	PD	Total
Greenhouse grown potato tubers ^a	< 0.001	< 0.001	< 0.001	< 0.001	0.001
Field grown potato tubers	0.004	0.003	0.002	0.003	0.012

^a Treated with N-phenyl-[^{14}C : ^{13}C]fenamidone (4 : 1)

PA–PD: Individual polar fractions

The presence of fewer polar entities in sample extracts derived from N-phenyl-fenamidone compared to C-phenyl-fenamidone treated potato tubers is due to the position of the ^{14}C -radiolabel. Cleavage of the N-N bond during potato metabolism of N-phenyl-fenamidone results in loss of non-radiolabelled RPA 412636 and RPA 412708. This process is likely to be accompanied by formation of radiolabelled aniline analogues. These are known to bind to plant matrices and are likely to contribute to the 53.9% (0.02 mg/kg) unextracted radioactive residue for N-phenyl-fenamidone treated tubers, compared to the 26.8% (0.023 mg/kg) unextracted radioactive residue for C-phenyl-fenamidone treated tubers.

In summary, the TRR found in potato tubers treated with fenamidone at 3×0.5 kg ai/ha and grown to maturity were very low (0.09 mg/kg and 0.04 mg/kg for the C-phenyl- and N-phenyl-label, respectively). Parent fenamidone accounted for 2.3 to 6 % (0.002 mg/kg) of TRR. The metabolites RPA 412708 and RPA 412636, each accounting for 6% of TRR and very low in absolute concentration (0.005–0.006 mg/kg) were identified. Because these metabolites were also identified as soil metabolites, it is assumed that they were formed in the soil and taken up directly from the soil. Furthermore, about 40% (C-phenyl-label) or 31% (N-phenyl-label) of TRR could be characterized as polar material. Figure 5 shows the proposed metabolic pathway for fenamidone in potatoes.

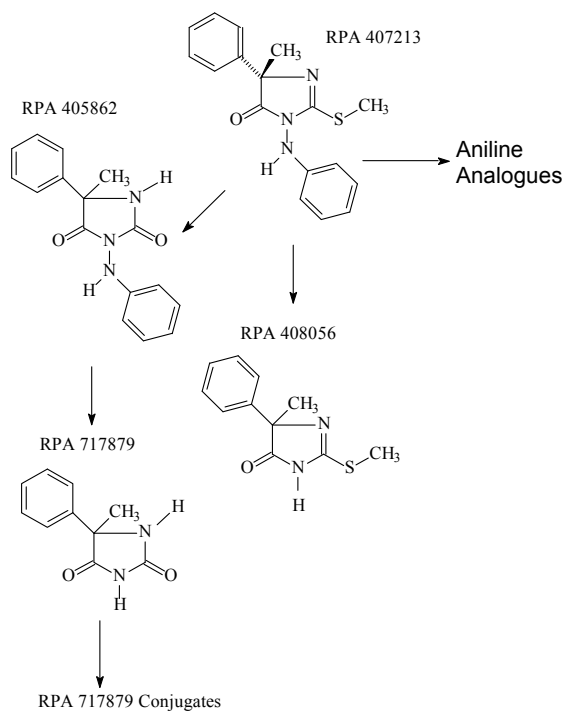


Figure 5 Proposed metabolic pathway for fenamidone (RPA 407213) in potatoes

Metabolite codes in this pathway are the numbers for the racemic reference compounds, as used in the original study report. In an additional study, it has been shown that all metabolites and degradation products were formed as the S-enantiomers.

Carrots

The metabolism of fenamidone was investigated in carrots grown outdoors under ambient conditions (Sur, 2006; MEF-06/243, M-275069-02-1). The test material was [N-phenyl-UL- ^{14}C]-fenamidone. The test material was applied three times as an SC 500 formulation at a rate of 0.3 kg ai/ha, giving a seasonal application rate of 0.9 kg ai/ha. The first application was made pre-emergence (one week after sowing), followed by two foliar applications to the emerged crop, 8 and 14 weeks after sowing.

Carrot leaves and roots were harvested at maturity, 14 days after the last application. The TRR found in the fractions are summarised in Table 28.

Table 28 TRR in carrots following application of [N-phenyl-UL-¹⁴C]-fenamidone

Plant part	TRR, mg fenamidone eq/kg	% Extracted	% Unextracted
Leaves	30.5	99.7	0.33
Roots	0.04	93	7.3

The majority of the radioactivity (between 81.3% and 98.7% of the TRR) was effectively extracted at ambient temperature with acetonitrile/water using a high-speed blender. After evaporation of the combined extracts of leaves, the radioactivity was partitioned between a dichloromethane and an aqueous phase. Only 1.2% of the TRR remained in the aqueous phase, whereas 97.5% of TRR was found in the organic phase. For roots, the combined acetonitrile/water extracts were simply concentrated without partitioning.

The leaf solids still contained 0.41 mg/kg (1.3% of TRR). A two-step microwave-assisted extraction, with neutral and acidic solvent then yielded the final post-extraction solids (PES), which contained only 0.104 mg/kg (0.33% of TRR). The radioactive residue in the root solids was already very low (0.008 mg/kg, 18.7% of TRR). After the same microwave extraction procedure, only 0.003 mg/kg (7.25% of TRR) remained in the final post-extraction solids (PES).

The extracts were analysed by HPLC or TLC for quantification of the parent compound and metabolites. These were identified by co-chromatography with radiolabelled or non-radiolabelled reference compounds and/or by mass spectroscopy. The distribution of radioactivity in identified or characterised components is summarised in Table 29.

Table 29 Distribution of radioactivity in carrots after treatment with [N-phenyl-UL-¹⁴C]-fenamidone

Fraction/component	Leaves		Roots	
	mg/kg	% TRR	mg/kg	% TRR
Fenamidone	27	89	0.012	29
RPA 411639	1.4	4.5	NA	NA
RPA 410193	0.48	1.6	0.003	7.2
RPA 413255	0.13	0.44	0.008	19
RPA 221701	0.056	0.19	0.002	5.4
Fenamidone dimer	0.050	0.17	ND	ND
RPA 221607	0.039	0.13	0.002	5.2
Carbohydrates	0.14	0.45	NA	NA
Total identified	29	96	0.027	65
Total characterised	1.03	3.3	0.010	27
Total extracted	30	100	0.037	93
Post extraction solids (PES)	0.104	0.33	0.003	7.3
Total	31	100	0.04	100

NA not applicable or not analysed

ND not detected

The residue in both leaves and roots consisted predominantly of unchanged fenamidone. The metabolic pattern showed that the three rings of fenamidone remain intact with no intermediate metabolites resulting from cleavage on the N-phenyl ring, although there was clear evidence for degradation of the N-phenyl ring and re-incorporation of the ¹⁴CO₂ in the plant leaves in the form of radiolabelled glucose. Aniline, the three isomer aminophenols (2-, 3-, 4-aminophenol) and 2- and 4-nitroaniline or conjugates thereof were specifically included as targets for analysis, but were not detected in either leaves or roots. A minor metabolic reaction was the nitration of the N-phenyl ring in the 2- and 4-positions to produce RPA 413255 and RPA 411639, respectively. A further route of degradation was hydrolysis of the N=C bond of the imidazolinone ring leading to the metabolites RPA 410193, RPA 221701, and RPA 221607 by replacement of the thiomethyl substituent by a carbonyl group. Figure 6 shows the proposed metabolic pathway for fenamidone in carrots.

Overall, the five plant metabolism studies submitted provide a clear understanding of the fate of the fenamidone in plants, both the C-phenyl and N-phenyl portions. The metabolic profiles differ

quantitatively between the plant species, but qualitatively there are no major differences; the routes and products of metabolism in plants are consistent across the studies. The overall metabolic pathway is consistent between the different crop categories fruits and fruiting vegetables, leafy vegetables, root and tuber vegetables. Parent fenamidone forms the largest part of the residue and the only significant metabolite is RPA 410193, diketo-fenamidone, formed by oxidative hydrolysis of the thiomethyl side chain.

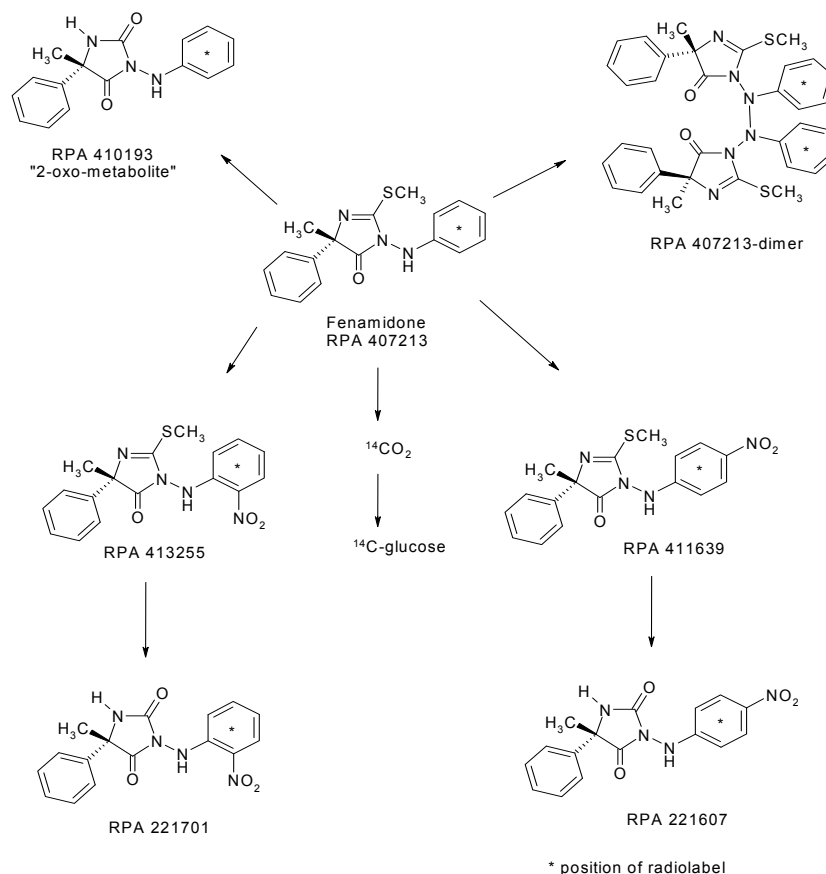


Figure 6 Proposed metabolic pathway for fenamidone in carrots

Metabolite codes in this pathway are the numbers for the racemic reference compounds, as used in the original study report. In an additional study, it has been shown that all metabolites and degradation products were formed as the S-enantiomers.

Environmental fate in soil and water-sediment systems

The FAO Manual (FAO, 2009) explained the data requirements for studies of environmental fate. The focus should be on those aspects that are most relevant to MRL setting. For fenamidone, supervised residue trials data were received for foliar spray on permanent crops and on annual crops. A further application is on cotton seed as in furrow treatment. Therefore, according to the FAO manual, studies on the aerobic degradation in soil, photolysis, hydrolysis, rotational crops (confined, field) and field dissipation were evaluated. For information on hydrolysis and photolysis see also chapter “Physical and chemical properties”, Table 1. The fate and behaviour of fenamidone in soils was investigated using fenamidone radio-labelled in two different positions, [C-phenyl-UL-¹⁴C]- and [N-phenyl-UL-¹⁴C]-fenamidone.

Aerobic degradation in soil

The route of aerobic degradation of fenamidone has been investigated in three soils: a clay loam (Loam by USDA classification; Simmonds and Burr, 1999; Report 201609, M-173243-01-1), a sandy loam and a loam (Burr and Bullus, 2000; Report 202200, M-240551-01-1). Fenamidone radio-labelled in two different positions, [C-phenyl-UL-¹⁴C]-fenamidone and [N-phenyl-UL-¹⁴C]-fenamidone, was applied separately to the clay loam and sandy loam soils, only [C-phenyl-UL-¹⁴C]-fenamidone was applied to the loam. The clay loam was treated at a rate equivalent to 1.6 kg ai/ha, the sandy loam and loam at rates equivalent to 1.2 kg ai/ha. The samples were also connected to a series of traps to retain any volatiles. Throughout the 365-day duration of the studies, the soil samples were maintained in the dark at 20 ± 2 °C and at known moisture contents.

Good overall radiochemical balances were obtained in each case; 94% to 97% of TAR at day 0, 92% to 99% of TAR at mean, over all incubation intervals. Fenamidone was rapidly metabolised in the three soils to two major metabolites, RPA 412708 and RPA 412636, both observed only from the C-phenyl treatment. RPA 411639 and RPA 413255 were observed as minor metabolites in the clay loam and loam, and at higher levels in the sandy loam, where two other major metabolites were found, RPA 221607 and RPA 221701. There were no unique metabolites observed from the N-phenyl label. Levels of volatile radioactivity remained quite low, < 10% of the TAR after 365 days. Unextracted radioactivity increased with time, reaching 31% and 46% for the C-phenyl-label and N-phenyl-label respectively in the clay loam, 14% and 26% respectively in the sandy loam and 21% for the N-phenyl-label in the loam. Various unknown metabolites were formed, which could be separated into numerous individual components, none of which reached a significant level (< 8% of TAR) and consequently the unknown components were not considered to contain any major metabolites. The composition of radioactivity is summarized in Tables 30 to 34.

Table 30 Composition of radioactivity in a clay loam soil by HPLC (C-phenyl-fenamidone)

Time, days	% TAR								
	% Extracted	Fenamidone	Unknown ^a (group 1)	RPA 412636	RPA 412708	RPA 411639	RPA 413255	Unknown ^a (group 2)	Others (< 5% each)
0	97	97	0.00	0.00	0.00	0.00	0.00	0.00	0.00
1	92	88	0.09	0.26	3.4	0.00	0.00	0.00	0.25
3	88	75	0.46	1.5	7.8	0.00	0.48	1.9	0.94
7	77	50	1.1	2.6	13	1.3	2.0	6.1	1.5
14	66	24	2.1	7.1	15	4.9	7.9	1.5	4.1
28	63	12	2.3	11	14	4.8	7.9	5.8	4.5
64	52	4.3	3.6	12	8.7	4.7	5.8	8.8	4.0
90	55	3.3	3.1	14	6.1	4.1	4.8	8.6	10
120	53	2.6	3.3	19	5.4	3.9	5.3	6.3	7.8
182	56	2.3	5.7	19	3.5	2.2	3.7	11	8.5
273	48	2.1	4.8	18	1.9	1.3	3.2	8.2	8.4
365	50	1.6	6.2	23	1.2	0.44	1.7	6.7	8.4

^a Two groups of unknown metabolites: polar (group 1) and non-polar (group 2)

Table 31 Composition of radioactivity in a clay loam soil by HPLC (N-phenyl-fenamidone)

Time, days	% TAR						
	% Extracted	Fenamidone	Unknown ^a (group 1)	RPA 411639	RPA 413255	Unknown ^a (group 2)	Others (< 5% each)
0	98	98	0.00	0.00	0.00	0.00	0.00
1	88	84	0.54	0.87	0.00	1.9	0.62
3	82	77	0.95	2.1	0.58	1.1	1.2
7	69	59	1.4	2.3	1.9	2.3	1.5
14	55	35	3.1	4.4	5.8	4.4	2.0
28	43	17	3.8	5.4	7.9	7.3	1.8
64	39	5.0	2.8	6.8	9.6	10	4.5
90	36	4.3	3.4	5.5	6.9	11	5.7

Time, days	% TAR						
	% Extracted	Fenamidone	Unknown ^a (group 1)	RPA 411639	RPA 413255	Unknown ^a (group 2)	Others ($< 5\%$ each)
120	33	2.9	4.4	3.9	5.7	9.9	5.9
182	34	2.7	5.9	3.0	4.4	11	6.5
273	29	2.6	4.5	2.7	4.2	9.0	6.0
365	32	2.1	2.9	1.9	3.9	12	9.2

^a Two groups of unknown metabolites: polar (group 1) and non-polar (group 2)

Table 32 Composition of radioactivity in a sandy loam soil by HPLC (C-phenyl-fenamidone)

Time, days	% TAR										
	% Extracted	Fenamidone	RPA 412636	RPA 412708	RPA 410193	RPA 409446	RPA 410995	RPA 411639	RPA 413255	Late group	Others ^a
0	98	98	ND	ND	ND	ND	ND	ND	ND	ND	ND
1	92	78	ND	9.2	ND	ND	ND	ND	ND	5.3	ND
3	93	52	6.0	6.1	2.4	ND	ND	3.2	3.3	16	3.3
7	92	35	10	6.8	1.5	ND	ND	5.3	4.6	24	4.6
14	92	25	10	13	1.1	0.31	0.56	7.8	8.4	20	4.8
28	91	15	20	8.6	0.94	0.70	0.53	7.6	9.4	22	6.1
56	87	12	33	5.9	2.6	5.9	3.2	7.1	11	2.3	4.4
121	84	7.6	28	1.3	2.1	9.2	7.0	4.2	6.4	10	7.5
181	81	4.3	33	0.57	1.5	9.8	9.5	1.7	4.1	13	3.6
272	80	3.7	30	2.0	1.6	7.6	6.7	1.9	3.4	16	7.7
365	76	3.4	35	1.0	1.5	8.4	8.7	1.3	2.4	9.4	4.8

^a Up to 11 components, each $< 5\%$ of applied radioactivity

Table 33 Composition of radioactivity in a sandy loam soil by HPLC (N-phenyl-fenamidone)

Time, days	% TAR								
	% Extracted	Fenamidon e	RPA 410193	RPA 409446	RPA 410995	RPA 411639	RPA 413255	Late group	Others ^a
0	98	98	ND	ND	ND	ND	ND	ND	ND
1	93	80	ND	ND	ND	ND	ND	13	ND
3	86	50	2.3	0.17	0.04	3.1	4.0	22	3.9
7	85	40	2.5	ND	ND	5.3	6.4	27	4.3
14	80	33	1.3	ND	ND	8.0	7.1	29	1.9
28	77	19	0.11	0.35	0.09	8.7	9.7	35	4.3
56	71	14	2.3	6.8	6.2	12	12	13	4.7
121	71	11	3.1	9.1	7.4	6.2	8.9	15	9.4
181	66	6.7	2.1	13	12	4.1	7.1	19	3.2
272	63	5.8	1.8	10	8.3	3.2	5.3	21	7.4
365	62	4.0	2.2	9.4	9.3	1.4	3.7	28	3.8

^a Up to 12 components, each $< 5\%$ of applied radioactivity

Table 34 Composition of radioactivity in a loam soil by HPLC (C-phenyl-fenamidone)

Time, days	% TAR									
	% Extracted	Fenamidone	RPA 412636	RPA 412708	RPA 410193	RPA 409446	RPA 411639	RPA 413255	Late group	Others ^a
0	94	94	ND	ND	ND	ND	ND	ND	ND	ND
1	90	90	ND	ND	ND	ND	ND	ND	ND	ND
3	89	73	0.38	4.2	0.40	ND	ND	0.32	10	0.16
7	85	52	2.3	7.0	0.26	ND	0.34	2.1	21	0.10
14	84	29	2.0	17	ND	ND	2.5	4.5	26	2.3
28	80	23	6.7	14	0.13	ND	0.27	2.2	30	4.1
56	71	11	10	17	0.19	ND	5.7	5.9	15	4.9

Time, days	% TAR									
	% Extracted	Fenamidone	RPA 412636	RPA 412708	RPA 410193	RPA 409446	RPA 411639	RPA 413255	Late group	Others ^a
121	49	7.7	11	11	0.14	0.11	1.9	3.2	19	13
181	63	2.8	13	13	0.07	ND	1.1	2.5	19	11
272	66	1.7	22	12	ND	ND	0.75	3.0	9.1	17
365	58	2.7	21	10	ND	ND	0.21	1.9	8.6	14

^a Up to 7 components, each < 8% of TAR

Under aerobic conditions, fenamidone was rapidly metabolised by three significant degradation routes. In the first route of degradation two major metabolites, RPA 412708 and RPA 412636 are formed. RPA 412708 is formed through the loss of the aniline ring. This is followed by further degradation with hydrolysis and the loss of the S-methyl group to form RPA 412636. In addition RPA 407213 was transformed by nitro addition in the 2 or 4 position to form RPA 411639 and RPA 413255. Mineralisation of both C-phenyl and N-phenyl rings resulted in the evolution of carbon dioxide. No unique metabolites were observed from N-phenyl labelled fenamidone. The proposed principal degradation pathways are presented in Figure 7.

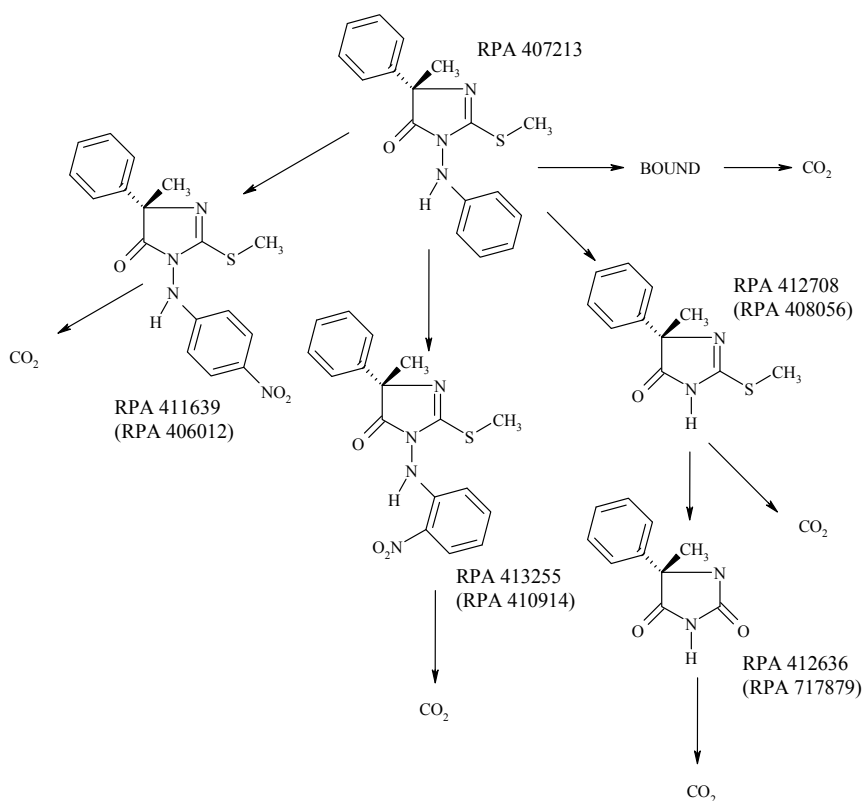


Figure 7 Proposed degradation pathways for fenamidone in aerobic soil

Metabolite codes in brackets are the numbers for the racemic compounds, as used in the original study report. The metabolites have been shown to be present as the S-enantiomers.

Soil photolysis

The photolytic degradation of fenamidone has been studied on a sandy loam soil. Two studies have been conducted, one with C-phenyl fenamidone (Burr and McDonald, 1999; Report 201428, M-173233-01-1) and the other with N-phenyl fenamidone (Burr, 1999; Report 202094, M-173271-01-1).

Portions of the soil, at a moisture content equivalent to 75% of the 1/3 bar moisture holding capacity, were surface treated with radio-labelled fenamidone to achieve an application rate

approximately equal to a cumulative field application rate of 1.5 kg ai/ha. The samples were placed inside glass incubation units fitted with quartz glass lids and aerobically incubated at $20 \pm 1^\circ\text{C}$. Irradiated samples were exposed to an artificial xenon light source (290 to 800 nm) for 13.3 or 12.7 hours each day (for the C-phenyl-label and N-phenyl-label, respectively), the equivalent of 1 day of natural summer sunlight. Non-irradiated control samples were incubated in continuous darkness. Trap solutions (ethylene glycol and potassium hydroxide) were connected to the incubation units to retain any volatile degradates.

At intervals up to 30 days after treatment, duplicate samples were removed for analysis. Good radiochemical balances were achieved for both the irradiated and non-irradiated soil (94.9 to 96.0% of TAR). In the C-phenyl-labelled experiment, over the 30-day study duration, carbon dioxide accounted for 2.3% and 8.1% of TAR in the irradiated and non-irradiated soils, respectively. Unextracted radioactivity accounted for 10% and 15% of TAR, respectively. In the N-phenyl-labelled experiment, the corresponding values were, respectively, 3.6%, 4.2%, 9.6% and 13%. Fenamidone was rapidly degraded in both irradiated and non-irradiated soils, by the end of the study reaching mean values of approximately 26% and 10.6% of TAR respectively in the C-phenyl labelled study and 31% and 17% of TAR in the N-phenyl labelled study. The degradates present in the irradiated and non-irradiated soils showed no significant differences. The composition of radioactivity is summarized in Tables 35 and 36.

Table 35 Composition of radioactivity by HPLC (C-phenyl-fenamidone)

Time, days	% TAR							
	Fenamidone	RPA 412636	RPA 412708	RPA 409445	RPA 410193	RPA 411639	Minor ^a unknowns	Total late eluting
Irradiated								
0	100	0.00	0.00	0.00	0.00	0.00	0.00	0.00
2	73	0.97	4.6	0.00	0.59	0.53	0.85	6.4
5	65	0.98	10	0.69	1.04	0.72	1.6	8.5
9	49	4.7	16	2.5	1.8	0.92	3.2	11
15	37	9.4	12	2.4	1.8	0.69	2.6	15
21	33	9.6	15	1.9	1.5	0.48	3.4	15
30	26	12	17	2.7	1.9	0.53	5.0	18
Non-irradiated								
0	100	0.00	0.00	0.00	0.00	0.00	0.00	0.00
2	63	3.2	2.9	0.67	0.70	0.72	0.54	12
5	46	2.4	11	1.3	0.81	0.68	1.4	19
9	30	4.6	20	2.4	0.61	0.73	2.9	25
15	23	7.3	12	2.1	1.1	0.68	3.6	27
21	14	10	9.0	0.68	2.4	0.16	5.0	26
30	11	9.6	12	1.3	1.7	0.46	7.9	29

^a Up to 6 peaks each < 4% of TAR

Table 36 Composition of radioactivity by HPLC (N-phenyl-fenamidone)

Time, days	% TAR					
	Fenamidone	RPA 410193	RPA 411639	RPA 413255	Total unknowns ^a	Total late eluting
Irradiated						
0	99	0.0	0.0	0.0	0.0	0.0
2	79	2.0	1.9	3.6	0.8	9.1
5	67	2.5	2.6	4.7	1.7	11
9	57	3.9	3.1	4.1	3.7	13
15	46	5.1	2.2	6.1	7.5	16
21	43	7.6	3.7	4.7	7.8	12
30	31	7.4	2.5	5.0	7.3	20
Non-irradiated						
0	99	0.0	0.0	0.0	0.0	0.0

Time, days	% TAR					
	Fenamidone	RPA 410193	RPA 411639	RPA 413255	Total unknowns ^a	Total late eluting
2	74	2.2	1.3	2.6	1.1	13
5	58	3.0	1.7	3.6	1.5	19
9	55	2.3	1.0	3.2	3.5	23
15	24	0.29	3.0	5.5	7.9	32
21	16	0.43	0.47	5.6	7.6	35
30	17	1.0	1.3	6.7	7.9	37

^a With RRT < 1.0, up to 9 peaks (C-phenyl label) and 5 peaks (N-phenyl label) each < 5% of TAR.

Fenamidone was readily degraded under both irradiated and non-irradiated soil conditions and it is concluded that photolytic processes do not contribute significantly to the degradation of fenamidone applied to the soil surface. Two major metabolites, RPA 412708 and RPA 412636, were observed, together with a large number of minor metabolites, each less than 5% of TAR, with similar quantities in both irradiated and non-irradiated conditions. Mineralisation of the C-phenyl ring resulted in the formation of carbon dioxide. The proposed photolytic degradation pathway for fenamidone in soil is presented in Figure 8.

With regard of the route of degradation in soil it can be concluded, that the degradation of fenamidone primarily proceeds by the action of aerobic soil microorganisms. Irradiation with sunlight does not influence the degradation pathway.

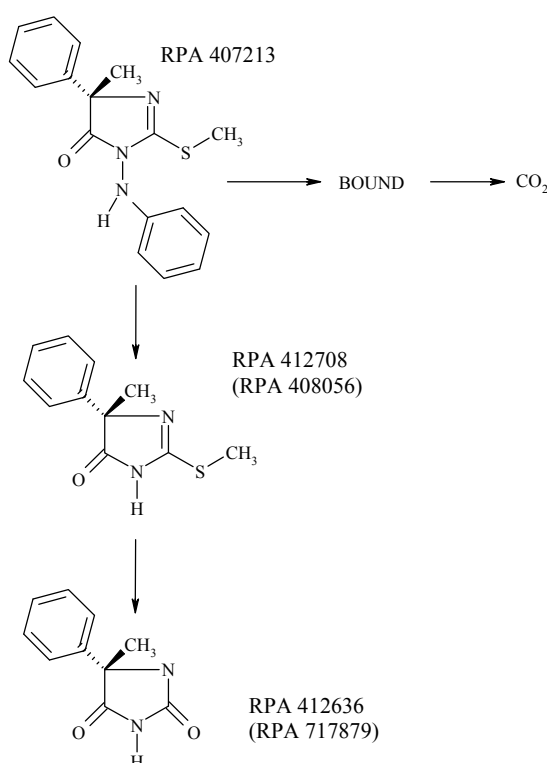


Figure 8 Proposed degradation pathway for fenamidone in soil under dark and irradiated conditions

Metabolite codes in parentheses are the numbers for the racemic compounds, as used in the original study report. The metabolites have been shown to be present as the S-enantiomers.

Rate of aerobic degradation in soil

A laboratory study has been conducted (Simmonds and Burr, 1999; Report 201610, M-173246-01-1) in which the rate of degradation of [C-phenyl-UL-¹⁴C]-fenamidone under aerobic conditions has been

investigated in three soils incubated for 6 months at 20 °C and one soil for 12 months at 10 °C in the dark. A high pH calcareous silty clay loam, a sandy loam and a clay loam were all incubated at 20 ± 2 °C, and a different clay loam was incubated at 10 ± 2 °C, to cover a range of pH, organic matter and clay content. Throughout the study, the moisture content of the soils was maintained at approximately 45% of the maximum water holding capacity. The soils were treated at a nominal application rate of 1.6 kg ai/ha. Volatile traps were attached to the samples flask to retain any volatile metabolites. At intervals during the study samples were removed for analysis. The overall mean recoveries were good, ranging from 93% to 96% of TAR. Fenamidone was rapidly metabolised. The abundance and distribution of metabolites was consistent with the aerobic soil degradation study report above. The DT₅₀ and DT₉₀ values obtained for fenamidone and for the metabolites RPA 412708, RPA 411639 and RPA 413255 are presented in Table 37 for the soils incubated at 20 °C and 10 °C (in the dark). All values calculated using a kinetic modelling program (KIM).

Table 37 DT₅₀ and DT₉₀ values for soils incubated at 20 °C and 10 °C

Compound/soil	T, °C	DT ₅₀ , days	DT ₉₀ , days	Fit
Fenamidone				
Silty clay loam	20	4.5	17.2	-0.9981
Sandy loam	20	3.1	19.8	-0.9999
Clay loam	20	0.9	5.1	-0.9986
RPA 412708				
Silty clay loam	20	214	Not calculated	-0.9857
Sandy loam	20	28.4	177	-0.9947
Clay loam	20	18	60	-0.9810
RPA 413255				
Silty clay loam	20	139	Not calculated	-0.9956
RPA 411639				
Silty clay loam	20	52.2	313	-0.9747
Fenamidone		22.7	97	-0.9994
Clay loam	10			
RPA 412708		195	475	-0.9898
Clay loam	10			

The rate of degradation of fenamidone and metabolites in a clay loam, a sandy loam and a loam soil at 20 °C were also determined in the two aerobic soil degradation studies (Simmonds and Burr, 1999; Report 201609, M-173243-01-1; Burr and Bullus, 2000; Report 202200, M-240551-01-1). All values calculated using a kinetic modelling program (KIM) are shown in Table 38.

Table 38 DT₅₀ and DT₉₀ values for soil incubated at 20 °C (from aerobic degradation studies)

Compound/soil	Label	DT ₅₀ , days	DT ₉₀ , days	Fit
Fenamidone				
Clay loam	C-phenyl	7.1	29.3	0.99950
	N-phenyl	9.6	39.1	0.99950
Sandy loam	C-phenyl	3.5	68.6	0.99862
	N-phenyl	3.7	115.6	0.99649
Loam	C-phenyl	7.8	81.5	0.99550
RPA 412708				
Clay loam	C-phenyl	55	300	0.99870
	N-phenyl	—	—	—
RPA 413255				
Clay loam	C-phenyl	135	394	0.97980
	N-phenyl	120	Not calculated	0.98630
RPA 411639				
Clay loam	C-phenyl	124	643	0.99550
	N-phenyl	129	Not calculated	0.99350

In summary, under aerobic conditions, fenamidone was rapidly degraded at 20 °C in all tested soils leading to the formation of two major metabolites RPA 408065 and RPA 412636. The DT_{50s} ranged from 0.9 to 9.6 days. The rate of degradation was slower at 10 °C (DT₅₀ 22.7 days).

Field dissipation

The behaviour of fenamidone was investigated at four field sites in Europe, in Italy, France, Germany and the UK (Wicks, 1999; Report 202140, Bayer reference M-183886-01-1). Fenamidone formulated as a suspension concentrate was sprayed onto a smooth seed bed at a nominal rate of 1.6 kg ai/ha, before growing grass cover. Irrigation water was applied to the treated plots as required so that precipitation plus irrigation was usually in excess of historical average precipitation. Soil samples from different depths to a maximum of 0.6 m below the surface were then collected at regular intervals up to 12 months and analysed for fenamidone, RPA 411639, RPA 413255, RPA 412708 and RPA 412636 using LC-MS/MS. The LOQ of each compound in soil was 0.005 mg/kg. Residues of the five analytes at each of the four test sites are presented in Table 39. The calculated DT₅₀ and DT₉₀ values for the four sites are presented in Table 40.

Table 39 Residues of fenamidone and metabolites in field soils at four different locations

Time, days	Residue, g/ha				
	Fenamidone	RPA 411639	RPA 413255	RPA 412708	RPA 412636
Bologna, Italy, clay loam					
0	889	< LOQ	< LOQ	ND	ND
3	884	36	40	29	< LOQ
7	1135	94	89	76	39
14	552	110	110	69	33
30	96	53	57	57	46
60	< LOQ	37	45	55	61
122	< LOQ	13	19	< LOQ	28
179	ND	10	23	< LOQ	17
260	ND	< LOQ	10	ND	< LOQ
Chazay, France, clay loam					
0	840	< LOQ	< LOQ	ND	ND
3	1105	42	36	43	35
7	368	38	34	27	47
14	88	42	28	63	101
30	< LOQ	12	17	11	52
56	< LOQ	12	26	12	77
119	< LOQ	< LOQ	< LOQ	< LOQ	20
182	ND	< LOQ	9	ND	< LOQ
261	ND	ND	11	ND	< LOQ
Goch, Germany, silt loam					
0	1226	< LOQ	< LOQ	ND	ND
3	742	< LOQ	< LOQ	45	< LOQ
7	642	< LOQ	< LOQ	122	< LOQ
14	186	< LOQ	13	214	34
31	60	< LOQ	< LOQ	103	68
60	< LOQ	< LOQ	< LOQ	24	74
126	< LOQ	< LOQ	< LOQ	< LOQ	57
190	ND	ND	ND	ND	43
288	ND	ND	ND	ND	32
Manningtree, UK, sandy loam					
0	1720	< LOQ	< LOQ	n/d	n/d
3	1071	28	29	20	16
7	1010	145	57	28	54
15	778	122	69	28	72
32	14	62	65	30	41
77	< LOQ	37	85	21	109
126	< LOQ	17	56	12	89
182	ND	< LOQ	15	< LOQ	40
275	ND	ND	21	ND	47

< LOQ = mean residue value was below the limit of quantification (9 g/ha)

Table 40 Residue decline results for fenamidone in field soils

Location	r ²	DT ₅₀ , days	Confidence interval	DT ₉₀ , days	Confidence interval
Bologna, Italy	0.82	14.3	11.6–17.0	47.4	38.5–56.3
Chazay, France	0.94	3.7	3.3–4.2	12.4	10.9–13.9
Goch, Germany	0.97	7.7	7.1–8.3	25.5	23.5–27.4
Manningtree, UK	0.85	8.5	6.9–10.1	28.2	22.9–33.5
Mean		8.5		28.4	

The data from the field study were analysed under a separate study (Reinken, 2002; Report OE02/062, M-210997-02-01). Environmental conditions like fluctuating soil moisture and soil temperature have a significant impact on the degradation of plant protection products under outdoor conditions. In order to achieve a comparability of half-lives or rate constants observed at various field locations the measured field data were analysed employing a normalisation procedure. In the original study reports the estimation of first-order dissipation half-lives was based only on standard kinetic modelling which does not take into account the specific soil-climate conditions at the different field sites.

Standardised transformation rates of fenamidone and the four principal metabolites (RPA 412708, RPA 412636, RPA 411639 & RPA 413255) were investigated in this study by a detailed kinetic analysis based on the reported field residue data using a special computer program. A mathematical model was employed able to describe the observed time-dependent field concentrations based on a first-order kinetic scheme, a reference soil temperature of 20 °C and a reference soil moisture of 100% FC (field capacity). The evaluation method accounted for effects of daily changing temperature and moisture according to established model assumptions.

The agreement of measured and calculated concentrations was good, with coefficients of determination (r²) between 0.86 and 0.96. Normalised half-life values and DT₅₀ values and field capacity for fenamidone and metabolites are given for each field site in Table 41 (all average values are calculated as geometric means). The degradation of fenamidone at reference conditions (20 °C and 100% field capacity) was consistent between the individual sites, giving half-lives between 4 and 7 days. Normalised half-life values for the four metabolites were also very similar across the sites. The representative average half-lives were 5 days for fenamidone, 12 days for RPA 412708, 47 days for RPA 412636, 21 days for RPA 411639 and 43 days for RPA 413255. The calculated half-lives (t_{1/2}) are identical to first-order DT₅₀ values.

Table 41 Standard first order dissipation rates (SFO) and DT₅₀ values at 20 °C

Location	Fenamidone		RPA 412708		RPA 412636		RPA 411639		RPA 413255	
	SFO, days ⁻¹	DT ₅₀ , days	SFO, days ⁻¹	DT ₅₀ , days	SFO, days ⁻¹	DT ₅₀ , days	SFO, days ⁻¹	DT ₅₀ , Days	SFO, days ⁻¹	DT ₅₀ , Days
Bologna	0.09492	7.3	0.04165	19.6	0.02237	31.0	0.02720	25.5	0.01922	36.1
Chazay	0.18140	3.8	0.07256	9.6	0.02605	26.6	0.02832	24.5	0.01360	510
Goch	0.17386	4.0	0.06383	10.9	0.00823	84.2	0.0400	17.3	0.02000	34.7
Manningtree	0.13354	5.2	0.06958	10.0	0.01002	69.2	0.04237	16.4	0.01274	54.4
Geom. mean	0.14140	4.9	0.06053	11.5	0.01481	46.8	0.03380	20.5	0.01607	43.1

In summary, field dissipation studies were undertaken at four sites in Europe using clay loam, silt loam and sandy loam soils. In all cases dissipation of fenamidone was rapid with a mean half live of 5 days. Half-lives for the metabolites were 12 days for RPA 412708, 47 days for RPA 412636, 21 days for RPA 411639 and 43 days for RPA 413255.

Hydrolytic degradation

The hydrolytic degradation of fenamidone was examined by Corgier and Turier (1996; Report 442527, M-164512-01-1). [C-phenyl-UL-¹⁴C]-fenamidone was used as test material in aqueous buffered solutions at pH values of 4, 5, 7 and 9 under sterile conditions at 25 °C and at an initial concentration of 3.9 mg/L. Duplicate samples were taken for analysis at intervals up to 31 days. In addition, two samples at each pH were attached to suitable trapping solutions and removed after 35 days. Recovery of radioactivity ranged from 96.2 to 102.3% with a mean value of 100.3 ± 0.9%.

No volatile compounds were found in the traps at any pH. Sterility of the samples was maintained throughout the incubation. During the course of the test, the concentration of fenamidone decreased and other compounds were detected. Table 42 presents the amounts of fenamidone and its main hydrolysis products occurring during the study.

Table 42 Hydrolytic degradation of fenamidone at different pH values

pH	Sampling time, days	Residue, % TAR				
		Fenamidone	RPA 410193	RPA 413350	RPA 412708	Unknown
4	0	100	0.0			
	5	92	8.4			
	10	85	15			
	14	80	20			
	19	70	26			
	25	67	33			
	31	60	39			
5	0	100	0.0			
	5	98	1.7			
	10	97	2.6			
	14	96	3.4			
	19	94	4.4			
	25	92	5.7			
	31	91	6.9			
7	0	100		0.0		
	5	100		0.0		
	10	98		1.1		
	14	98		1.3		
	19	97		2.1		
	25	96		2.5		
	31	95		2.9		
9	0	100	0.0	0.0	0.0	0.0
	5	95	0.0	4.6	0.0	0.0
	10	82	1.2	12	3.3	0.0
	14	73	1.7	17	4.6	0.7
	19	66	2.2	24	5.9	1.3
	25	56	3.1	28	7.9	3.4
	31	47	3.8	32	10	4.6

Hydrolytic degradation of fenamidone was shown to follow pseudo-first-order reaction kinetics resulting in three major hydrolysis products that exceeded 10% of TAR; RPA 410193 at pH 4, RPA 413350 and RPA 412708 at pH 9. Fenamidone is considered stable at pH 5 and 7 (less than 10% degradation after one month), but half-lives could be estimated at these pH values. The hydrolytic degradation characteristics are presented in Table 43.

Table 43 Rates of hydrolytic degradation of fenamidone at different pH values

pH	r ²	K, day ⁻¹	DT ₅₀ , days
4	0.993	-0.0166	42
5	0.981	-0.0031	222
7	0.942	-0.0017	411
9	0.991	-0.0251	28

In summary, fenamidone can be considered to be stable at pH 5 and 7. The compound is hydrolysed at pH 4 and 9 with DT₅₀ values of 42 and 28 days respectively. A proposed degradation pathway is given in Figure 9.

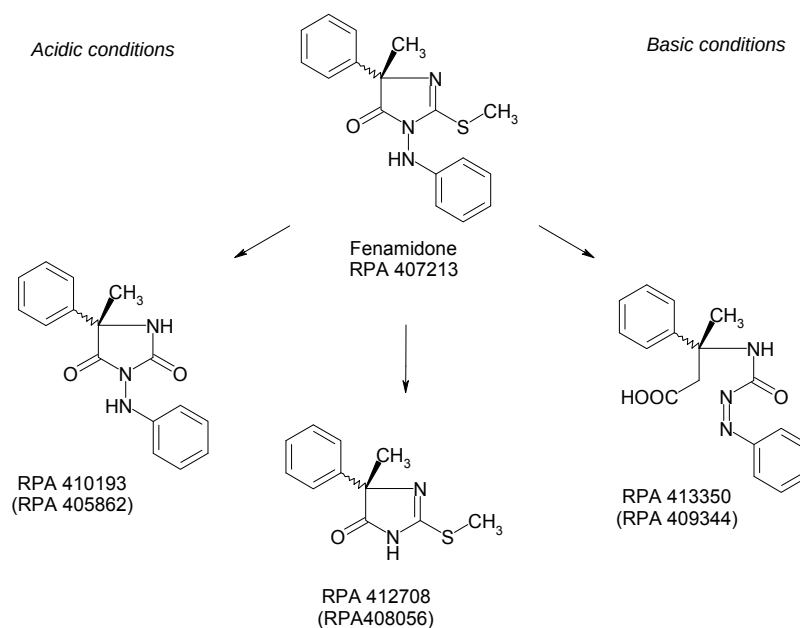


Figure 9 Proposed hydrolytic degradation pathway for fenamidone (RPA 407213)

Metabolite codes in parentheses are the numbers for the racemic compounds, as used in the original study report. The metabolites have been shown to be present as the S-enantiomers.

Residues in rotational crops

Confined radio-labelled succeeding crop studies

Three confined rotational crop studies have been conducted, one in 2001 and two in 2013. The first study was conducted by McMillan-Staff *et al.* in 2001 (report C010569 with amendments C021273 and C021274, M-199243-03-1). The quantity and nature of the residues in following crops were investigated after application of [C-phenyl-UL-¹⁴C]-fenamidone to bare sandy loam soil at an application rate equivalent to 2.02 kg ai/ha (exception: 1.6 kg ai/ha for the 30 and 120 day lettuce). Representative rotational crops, a leafy crop (lettuce), a root crop (turnip) and a grain crop (barley), were sown after ageing periods of 30, 120 (lettuce, turnip), 150 (barley) and 365 days. The rotational crops were grown outdoors in circular vessels 80 cm in diameter and 60 cm deep. The treatment was applied to the surface of each treated vessel and crops planted after the specified intervals. Plant samples were harvested at maturity; interim immature samples were also taken and analysed. At harvest, the lettuce and barley were cut at ground level and only the parts above the soil analysed, the turnips were harvested intact and then divided into leaf and root. The plant parts were homogenised and analysed. Extracts containing sufficient radioactivity were characterised by HPLC, TLC and LC-MS as appropriate.

The highest residues in all the plant commodities were found at the 30-day planting interval. Residues after 120/150 and 365 days were significantly lower. The proportions of the radioactivity extracted, identified and characterised in each of the plant commodities are summarised in Table 44 and the nature of the extractable radioactivity is presented in Table 45.

Table 44 Distribution of TRR in plants at harvest

Interval, days	Plant part	TRR, mg/kg	% TRR					
			Washings	Extracted	Un-extracted	Total identified	Total characterised	Total identified and characterised
30	Lettuce	0.72	2.6	80	20	55	20	75
	Turnip top	1.02	—	93	6.7	62	20	82
	Turnip root	0.25	19	62	19	46	1.1	47
	Barley chaff	7.19	—	91	9.0	79	5.1	84

Interval, days	Plant part	TRR, mg/kg	% TRR					
			Washings	Extracted	Un-extracted	Total identified	Total characterised	Total identified and characterised
120	Barley grain	1.34	—	88	12	75	1.4	76
	Barley straw	7.05	—	82	18	63	—	63
	Lettuce	0.14	—	69	31	67	2.3	69
	Turnip top	0.43	—	76	24	63	12	76
	Turnip root	0.15	0.99	80	19	80	—	80
150	Barley chaff	0.35	—	84	15	65	—	65
	Barley grain	0.07	—	66	34	66	—	66
	Barley straw	0.60	—	73	27	54	—	54
365	Lettuce	0.12	5.4	76	19	74	—	74
	Turnip top	0.18	—	86	14	70	16	86
	Turnip root	0.14	27	30	43	28	1.3	30
	Barley chaff	0.39	—	81	19	57	3.4	60
	Barley grain	0.09	—	73	27	74	—	74
	Barley straw	0.77	—	80	20	68	—	68

Table 45 Characterisation of radioactivity in plants at harvest

Interval days	Plant part	Radioactivity, % TRR (mg/kg in parentheses)									
		Unretained ^a	Slightly Retained ^b	Unknown ⁵	Unknown ⁶	RPA 412708 Conjugate	RPA 412636	RPA 412708	RPA 410193	Polar unknowns < 10%	Non-polar unknowns < 10%
30	Lettuce	4.5 (0.033)	2.9 (0.021)	10 (0.075)	0.54 (0.004)	22 (0.16)	18 (0.13)	5.7 (0.041)	2.1 (0.015)	0.29 (0.002)	8.7 (0.063)
	Turnip top	24 (0.25)		7.4 (0.075)	11 (0.12)	34 (0.34)	3.5 (0.036)				1.1 (0.011)
	Turnip root	3.6 (0.009)	26 (0.065)	0.41 (0.001)		7.8 (0.02)	8.4 (0.021)				0.7 (0.002)
	Barley chaff	48 (3.46)		0.66 (0.05)		28 (1.98)	3.5 (0.25)			3.1 (0.24)	1.1 (0.082)
	Barley grain	55 (0.74)	0.73 (0.01)			17 (0.23)	2.0 (0.027)				1.4 (0.018)
	Barley straw	34 (2.4)					29 (2.04)				
120	Lettuce	0.00 (0.0)	3.9 (0.005)	2.3 (0.003)		41 (0.056)	20 (0.028)	1.3 (0.002)			
	Turnip top	10 (0.045)		9.8 (0.043)	2.4 (0.01)	47 (0.21)	5.7 (0.025)				
	Turnip root	41 (0.06)	13 (0.02)			19 (0.03)	7.5 (0.011)				
150	Barley chaff	3.4 (0.012)	15 (0.054)			32 (0.11)	11 (0.039)	2.8 (0.01)			
	Barley grain	30 (0.021)	18 (0.013)			14 (0.01)	3.1 (0.002)				
	Barley straw	3.5 (0.021)	6.8 (0.04)			38 (0.23)	6.3 (0.038)				
365	Lettuce	9.9 (0.012)				44 (0.052)	20 (0.024)				
	Turnip top		13 (0.023)	16 (0.03)		46 (0.09)	11 (0.02)				
	Turnip root		4.1 (0.006)	1.3 (0.002)		19 (0.026)	5.3 (0.007)				
	Barley chaff		7.0 (0.027)	3.4 (0.013)		37 (0.14)	13 (0.051)				
	Barley grain		34 (0.031)			33 (0.03)	5.9 (0.005)				
	Barley straw					56 (0.43)	12 (0.094)				

^a Unretained compounds^b Slightly retained compounds

The profile of identified and characterised components was similar in each of the crops and at each planting interval. RPA 412636 and a conjugate of RPA 412708 were the major components in all crop parts, unconjugated RPA 412708 was also detected in some plant parts. The RPA 412708 conjugate was putatively identified as the glucoside conjugate in many samples. Additional work proposed the presence of amino acids in two regions (“unretained compounds” and “slightly retained compounds” in Table 45), these were classified as tentatively identified in the plants and demonstrate that fenamidone residues were reincorporated into natural occurring compounds in the plant. Two distinct unknowns (U5 and U6) were characterised and a number of polar and non-polar components were also detected. No parent fenamidone was detected in any plant part.

Two further confined rotational crop studies have been conducted with radiolabelled fenamidone (C-phenyl label: Bongartz and Miebach, 2013, report M1301876-6, M-447085-01-1, M-447085-02-1; N-phenyl label: Bongartz and Miebach, 2013, report M1301877-7, M-451106-01-1). The quantity and nature of the residues in following crops were investigated after single applications of C-phenyl-UL- $^{14}\text{C}/^{13}\text{C}_6$]-fenamidone (referred as C-phenyl label) and [aniline-UL- $^{14}\text{C}/^{13}\text{C}_6$]-fenamidone (referred as N-phenyl label) to bare sandy loam soils at application rates of 0.97 kg ai/ha for the C-phenyl label and 0.96 kg ai/ha for the N-phenyl label. Representative rotational crops, a leafy crop (Swiss chard), a root crop (turnip) and a cereal crop (wheat), were sown after ageing periods of 30, 191 and 324 days.

The rotational crops were grown outdoors in 1 m² planting containers. The treatment was applied to the surface of each treated vessel and crops planted after the specified intervals. The application to the soil and the ageing of the soil (0 to 30 days after soil treatment) were conducted in the outdoor “vegetation area”, the plant cultivation took place under artificial temperature and light conditions in a greenhouse. At harvest, the Swiss chard and wheat were cut at ground level and only the parts above the soil analysed, the turnips were harvested intact and then divided into leaf and root. The plant parts were homogenised and analysed as follows:

Conventional extraction and sample preparation of all plant parts (RAC):

Aliquots of the homogenised samples were extracted 3 × with acetonitrile/water (8/2, v/v) and 1 × with pure acetonitrile. The extraction steps were conducted using a homogeniser. The residues were air-dried and weighed yielding the solids. The TRR of each RAC was calculated from the specific radioactivity of the test compound, the amount of the sample used for extraction and the sum of radioactivity, measured in the extracts and the remaining solids. The combined and concentrated conventional extracts were subjected to HPLC analysis. Significant losses of radioactivity during the concentration process or radioactivity in the distillates were not observed. Recoveries of the concentration processes ranged from 95.6% to 103%.

Exhaustive extraction of solids:

Depending on the amount of residues in the solids of the conventional extraction, an exhaustive extraction was performed with 1 × acetonitrile/water (1/1, v/v) and 1 × with acetonitrile/water (1/1, v/v) plus formic acid. The exhaustive extracts were concentrated and analysed by HPLC. Significant losses of radioactivity during the concentration process or radioactivity in the distillates were not observed. Recoveries of the concentration processes ranged from 95.5% to 104%.

Enzyme digestion of solids 2 of wheat grain (1st rotation) after exhaustive extraction with microwave assistance:

Further characterisation of the solids 2 of wheat grain was performed by an enzyme digestion with diastase followed by a partition of the digestion solution against ethyl acetate. After partition, the aqueous phase was concentrated and further analysed by HPLC.

Hydrolysis of fenamidone-hydantoin-conjugates to fenamidone-hydantoin (P10):

The hydrolysis of fenamidone-hydantoin-conjugates to fenamidone-hydantoin was investigated under extreme conditions with hydrochloric acid and sodium hydroxide at 100 °C for at least 1 h and resulted into multiple degradation products or non-reproducible results. Therefore the hydrolysis was

performed according to the residue method 00863. Representative conventional extracts of the first and second rotation were hydrolysed under mild acidic conditions with formic acid for approx. 2 hours at 70 °C.

Extracts containing sufficient radioactivity were characterised by HPLC, TLC and LC-MS as appropriate. The TRR found in each crop part are presented in Table 46. The highest residues in all the plant commodities were found at the 30-day planting interval for both labels, with the exception of wheat straw treated with the phenyl label, where higher residues were found at the second rotation (191 days). In general, residues after 191 and 324 days were significantly lower. The proportions of the radioactivity extracted, identified and characterised in each of the plant commodities are summarised in Tables 47 (C-phenyl label) and 48 (N-phenyl label). The nature of the extracted radioactivity is shown in Tables 49 and 50. In general, the TRR in the plants were significantly higher in the C-phenyl label study compared with the N-phenyl label study, primarily because of the higher uptake of C-phenyl label-specific metabolites from the soil following soil cleavage of the parent compound. For both labels, the majority of the radioactivity was extracted in the initial extraction, with the exception of wheat grain. Extraction of the remaining residues was performed with exhaustive extraction techniques, using elevated temperature conditions (microwave) and where required enzyme digestion. Parent compound and non-label-specific metabolites were isolated from crop extracts and their identities confirmed by spectroscopic methods.

The extracted residues were systematically analysed for the presence of potentially released aniline. In the extract of Swiss chard (TRR: 0.13 mg eq/kg) rotated after application of aniline-labelled fenamidone to soil, no aniline could be detected.

Table 46 TRR in plant commodities of rotational crops

Plant part	TRR, mg eq/kg					
	30 day PBI ^a		191 day PBI		324 day PBI	
	C-phenyl	N-phenyl	C-phenyl	N-phenyl	C-phenyl	N-phenyl
Wheat forage	0.50	0.04	0.20	0.02	0.10	0.02
Wheat hay	2.24	0.32	0.92	0.08	0.41	0.08
Wheat straw	1.00	0.27	1.67	0.09	0.43	0.08
Wheat grain	0.09	0.03	0.07	0.01	0.03	0.01
Turnip leaves	1.00	0.04	0.18	0.02	0.39	0.02
Turnip roots	0.07	0.01	0.02	0.01	0.03	0.01
Swiss chard (intermediate)	0.67	0.06	0.14	0.02	0.15	0.01
Swiss chard (at maturity)	0.95	0.13	0.27	0.03	0.12	0.01

^a PBI Plant back interval

Table 47 TRR in plants at harvest following application of [C-phenyl]-UL-¹⁴C/¹³C₆]-fenamidone

Interval, days	Plant part	TRR, mg eq/kg	% TRR					
			Initial extract	Exhaustive extracts + digests	Un-extracted	Total identified	Total characterised	Total identified and characterised
30	Wheat forage	0.50	90	–	9.8	84	6.5	90
	Wheat hay	2.24	67	26	7.4	83	9.3	93
	Wheat straw	1.00	66	24	9.5	88	2.7	91
	Wheat grain	0.09	27	72	1.2	83	16	99
	Turnip leaves	1.00	93	4.2	2.5	94	3.2	98
	Turnip roots	0.07	89	–	11	84	4.5	89
	Swiss chard (intermediate)	0.67	93	–	6.8	89	4.5	93
	Swiss chard (maturity)	0.95	91	8.7	3.0	92	4.9	97
191	Wheat forage	0.20	93	–	7.4	90	2.2	93
	Wheat hay	0.92	73	22	5.3	92	3.2	95
	Wheat straw	1.67	77	18	4.4	92	3.4	96
	Wheat grain	0.07	21	40	7.0	90	2.6	93
	Turnip leaves	0.18	95	–	5.5	92	2.3	95
	Turnip roots	0.02	85	–	15	79	6.2	85
	Swiss chard (intermediate)	0.14	94	–	6.4	92	1.6	94

Interval, days	Plant part	TRR, mg eq/kg	% TRR					
			Initial extract	Exhaustive extracts + digests	Un-extracted	Total identified	Total characterised	Total identified and characterised
	Swiss chard (maturity)	0.27	92	–	8.0	89	2.7	92
324	Wheat forage	0.095	90	–	9.6	90	–	90
	Wheat hay	0.41	74	18	8.1	90	2.2	92
	Wheat straw	0.43	71	20	8.8	90	1.1	91
	Wheat grain	0.03	28	37	35	27	39	65
	Turnip leaves	0.39	96	–	4.4	93	2.9	96
	Turnip roots	0.03	82	–	18	78	4.5	82
	Swiss chard (intermediate)	0.15	92	–	8.2	90	2.3	92
	Swiss chard (maturity)	0.12	94	–	6.0	89	5.0	94

Table 48 TRR in plants at harvest following application of [N-phenyl-UL-¹⁴C/¹³C₆]-fenamidone

Interval, days	Plant part	TRR, mg eq/kg	% TRR					
			Initial extract	Exhaustive extracts + digests	Un-extracted	Total identified	Total characterised	Total identified and characterised
30	Wheat forage	0.04	63	28	9.4	27	64	91
	Wheat hay	0.32	47	40	13	40	48	87
	Wheat straw	0.27	50	35	15	47	38	85
	Wheat grain	0.03	–	65	2.5	–	98	98
	Turnip leaves	0.04	78	–	22	32	46	78
	Turnip roots	0.01	77	–	23	17	60	77
	Swiss chard (intermediate)	0.06	67	23	10	34	56	90
	Swiss chard (maturity)	0.13	62	25	13	36	51	87
191	Wheat forage	0.02	66	–	34	5.5	61	66
	Wheat hay	0.08	48	36	16	25	59	84
	Wheat straw	0.09	54	34	11	23	66	89
	Wheat grain	0.01	9.5	–	91	–	9.5	9.5
	Turnip leaves	0.02	78	–	22	17	61	78
	Turnip roots	0.01	62	–	38	–	62	62
	Swiss chard (intermediate)	0.02	62	–	38	31	31	62
	Swiss chard (maturity)	0.03	56	29	15	26	59	85
324	Wheat forage	0.02	67	–	33	27	39	67
	Wheat hay	0.08	47	34	20	17	63	80
	Wheat straw	0.08	44	33	23	16	61	77
	Wheat grain	0.01	–	31	69	–	31	31
	Turnip leaves	0.02	73	–	27	17	56	73
	Turnip roots	0.01	–	–	–	–	–	–
	Swiss chard (intermediate)	0.01	55	–	45	–	55	55
	Swiss chard (maturity)	0.01	67	–	33	–	67	67

Table 49 Characterization of radioactivity in plants at harvest following application of [C-phenyl-UL-¹⁴C/¹³C₆]-fenamidone

Compound/fracti on	Wheat forage	Wheat hay	Wheat straw	Wheat grain	Turnip leaves	Turnip roots	Swiss chard (intermediat e)	Swiss chard (mature)
Fenamidone-	C-phenyl label, interval 30 days,% TRR (mg/kg in parentheses)							
2-phenyl-propionic acid (P2)	5.7 (0.028)	1.2 (0.027)	2.5 (0.025)	58 (0.051)	7.1 (0.071)	29 (0.022)	3.7 (0.025)	3.7 (0.035)
-hydantoin-serine	2.3	0.4	0.6	ND	30	4.0	1.1	1.7

Fenamidone

Compound/fracti on	Wheat forage	Wheat hay	Wheat straw	Wheat grain	Turnip leaves	Turnip roots	Swiss chard (intermediat e)	Swiss chard (mature)
(P4)	(0.012)	(0.009)	(0.006)		(0.29)	(0.003)	(0.007)	(0.017)
-imino-hydantoin (P5)	8.3 (0.041)	15 (0.34)	15 (0.147)	3.7 (0.003)	2.2 (0.022)	9.6 (0.007)	3.8 (0.026)	5.9 (0.056)
-thiohydantoin- serine (P6)	1.1 (0.005)	2.8 (0.062)	4.2 (0.042)	1.0 (0.001)	4.7 (0.047)	ND	2.9 (0.019)	2.6 (0.025)
-hydantoin- glucoside (P7)	45 (0.22)	46 (1.02)	51 (0.51)	19 (0.017)	31 (0.31)	11 (0.008)	35 (0.23)	36 (0.34)
-thiohydantoin- glycoside (P8)	2.5 (0.013)	3.1 (0.069)	1.6 (0.016)	ND	5.8 (0.058)	ND	2.1 (0.014)	3.2 (0.03)
-hydantoin-malo- nyl-glucoside (P9)	1.5 (0.008)	2.5 (0.057)	1.6 (0.016)	ND	3.5 (0.03)	ND	16 (0.11)	22 (0.2)
-hydantoin (P10), (RPA 412636)	15 (0.072)	8.7 (0.19)	11 (0.11)	1.5 (0.001)	6.6 (0.066)	29 (0.021)	21 (0.14)	14 (0.13)
-hydroxymethyl- thiohydantoin (P13)	1.5 (0.007)	1.8 (0.04)	0.5 (0.005)	ND	2.9 (0.029)	0.5 (< 0.001)	0.9 (0.006)	1.3 (0.012)
-nitro-GSH (P14)	1.0 (0.005)	1.5 (0.032)	ND	ND	–	–	1.9 (0.013)	2.1 (0.02)
-hydroxy-nitro- malonyl- glycoside (P16)	ND	0.4 (0.009)	ND	ND	0.6 (0.006)	ND	–	–
-desthiomethyl-2- nitro (P17)	0.2 (0.001)	ND	0.3 (0.003)	ND	–	–	0.2 (0.001)	0.2 (0.002)
Parent and -4-nitro (P19)	–	–	–	–	–	–	–	0.2 (0.002)
-2-nitro (P20)	0.2 (0.001)	0.2 (0.005)	0.2 (0.002)	ND	0.1 (0.001)	0.6 (< 0.001)	–	0.1 (0.001)
Sum total unknowns $< 10\%$	6.4 (0.032)	9.2 (0.21)	2.6 (0.21)	16.2 (0.014) ^a	3.1 (0.031)	4.4 (0.005)	4.5 (0.03)	4.9 (0.047)
Fenamidone-	C-phenyl label, interval 191 days,% TRR (mg/kg in parentheses)							
2-phenyl- propionic acid (P2)	1.9 (0.004)	3.0 (0.028)	1.3 (0.022)	73 (0.05)	9.6 (0.017)	28 (0.005)	ND	3.2 (0.009)
-hydantoin-serine (P4)	0.8 (0.002)	0.6 (0.005)	1.2 (0.02)	ND	39 (0.071)	6.9 (0.001)	–	–
-imino-hydantoin (P5)	11 (0.022)	17 (0.15)	19 (0.32)	ND	2.7 (0.005)	4.5 (0.001)	2.8 (0.004)	1.9 (0.005)
-thiohydantoin- serine (P6)	–	–	–	–	–	–	0.8 (0.001)	4.2 (0.011)
-hydantoin- glucoside (P7)	57 (0.11)	61 (0.56)	56 (0.93)	13 (0.009)	26 (0.047)	4.3 (0.001)	41 (0.06)	38 (0.104)
-thiohydantoin- glycoside (P8)	1.0 (0.002)	1.2 (0.011)	1.5 (0.025)	1.1 (0.001)	2.3 (0.004)	0.8 (< 0.001)	0.6 (0.001)	0.6 (0.002)
-hydantoin-malo- nyl-glucoside (P9)	2.1 (0.004)	1.6 (0.015)	2.2 (0.037)	0.4 (< 0.001)	2.8 (0.005)	1.0 (< 0.001)	40 (0.06)	38 (0.103)
-hydantoin (P10) (RPA 412636)	16 (0.03)	5.9 (0.06)	8.7 (0.15)	2.4 (0.002)	7.4 (0.013)	32 (0.005)	5.8 (0.008)	2.2 (0.006)
-hydroxymethyl- thiohydantoin (P13)	0.7 (0.001)	1.0 (0.009)	0.7 (0.011)	ND	2.5 (0.005)	1.3 (< 0.001)	0.8 (0.001)	0.8 (0.002)
-nitro-GSH (P14)	ND	ND	0.9 (0.015)	ND	–	–	0.5 (0.001)	0.3 (0.001)
-hydroxy-nitro- malonyl- glycoside (P16)	ND	ND	0.4 (0.006)	ND	0.2 (< 0.001)	ND	–	–
-desthiomethyl-2- nitro (P17)	ND	ND	0.3 (0.005)	ND	–	–	–	–
Parent and -4-nitro (P19)	–	–	–	–	–	–	–	–
-2-nitro (P20)	ND	0.7	0.1	ND	ND	0.8	–	–

Compound/fracti on	Wheat forage	Wheat hay	Wheat straw	Wheat grain	Turnip leaves	Turnip roots	Swiss chard (intermediat e)	Swiss chard (mature)
		(0.007)	(0.002)			(< 0.001)		
Sum total unknowns <10%	0.21 (0.004)	3.2 (0.029)	3.4 (0.056)	2.6 (0.002)	0.23 (0.004)	6.1 (0.002)	1.6 (0.003)	2.7 (0.008)
Fenamidone-	C-phenyl label, interval 324 days,% TRR (mg/kg in parentheses)							
2-phenyl- propionic acid (P2)	4.8 (0.005)	2.9 (0.012)	3.7 (0.016)	15 (0.004)	9.1 (0.035)	36 (0.011)	2.0 (0.003)	1.6 (0.002)
-hydantoin-serine (P4)	–	–	–	–	34 (0.13)	7.5 (0.002)	2.0 (0.003)	1.0 (0.001)
-imino-hydantoin (P5)	7.6 (0.007)	15 (0.06)	11 (0.05)	–	3.2 (0.012)	6.9 (0.002)	2.9 (0.004)	1.2 (0.002)
-thiohydantoin- serine (P6)	–	2.5 (0.01)	2.9 (0.013)	–	–	–	1.1 (0.002)	3.0 (0.004)
-hydantoin- glucoside (P7)	63 (0.06)	60 (0.24)	62 (0.27)	9.4 (0.003)	34 (0.13)	3.9 (0.001)	59 (0.09)	49 (0.06)
-thiohydantoin- glycoside (P8)	1.4 (0.001)	0.8 (0.003)	0.9 (0.004)	1.3 (< 0.001)	3.2 (0.012)	–	0.6 (0.001)	2.5 (0.003)
-hydantoin-malo- nyl-glucoside (P9)	1.1 (0.001)	1.3 (0.005)	0.8 (0.004)	–	4.3 (0.017)	–	17 (0.026)	25 (0.031)
-hydantoin (P10) (RPA 412636)	13 (0.012)	6.6 (0.027)	7.5 (0.032)	–	3.2 (0.013)	23 (0.007)	3.9 (0.006)	4.4 (0.005)
–hydroxymethyl- thiohydantoin (P13)	–	0.7 (0.003)	0.5 (0.002)	–	2.0 (0.008)	–	1.3 (0.002)	1.4 (0.002)
-nitro-GSH (P14)	–	–	–	–	–	–	–	–
-hydroxy-nitro- malonyl- glycoside (P16)	–	–	–	–	0.6 (0.002)	–	–	–
-desthiomethyl-2- nitro (P17)	–	–	0.3 (0.001)	–	–	–	–	–
Parent and -4-nitro (P19)	–	–	–	–	–	–	–	–
-2-nitro (P20)	–	–	0.1 (0.001)	–	–	–	–	–
Sum total unknowns <10%	–	2.2 (0.009)	1.1 (0.005)	1.6 (< 0.001)	3.0 (0.012)	4.5 (0.001)	2.2 (0.004)	5.0 (0.007)

^a Incorporated CO₂ (characterized by enzymatic digestion)

Parent compound was detected together with fenamidone-4-nitro in one region of the conventional extract of Swiss chard of the first rotation at maturity, only. The region amounted to 0.002 mg/kg (0.2% of TRR). The major metabolites were fenamidone-hydantoin (RPA 412636) and its conjugates fenamidone-hydantoin-glucoside, fenamidone-hydantoin-serine and fenamidone-hydantoin-malonyl-glycoside. These metabolites represented in sum approx. 50 to 70% of the TRR in the plants of all rotations, except wheat grain and turnip roots. 2-phenyl-propionic acid was detected in higher amounts in these plants and ranged from approx. 15 to 73% of the TRR. Another important metabolite in wheat was fenamidone-imino-hydantoin and amounted up to approx. 19% of the TRR in wheat straw.

Table 50 Characterization of radioactivity in plants at harvest, [N-phenyl-UL-¹⁴C/¹³C₆]-fenamidone

Compound/fracti on	Wheat forage	Wheat hay	Wheat straw	Wheat grain	Turnip leaves	Turnip roots	Swiss chard (intermediate)	Swiss chard (mature)
Fenamidone-	N-phenyl label, interval 30 days,% TRR (mg/kg in parentheses)							
-nitro-GSH (A7)	3.2 (0.001)	1.4 (0.004)	–	–	3.3 (0.001)	–	18 (0.01)	20 (0.03)
-hydroxy- malonyl-	3.5 (0.001)	1.9 (0.006)	2.1 (0.006)	–	14 (0.005)	–	1.1 (0.001)	2.4 (0.003)

Fenamidone

Compound/fracti on	Wheat forage	Wheat hay	Wheat straw	Wheat grain	Turnip leaves	Turnip roots	Swiss chard (intermediate)	Swiss chard (mature)
glycoside (A8)								
-desthiomethyl (A9)	1.3 (< 0.001)	—	—	—	0.8 (< 0.001)	—	3.0 (0.002)	2.5 (0.003)
-desthiomethyl-4- nitro (A10)	2.9 (0.001)	3.7 (0.01)	3.5 (0.009)	—	1.9 (0.001)	—	2.7 (0.002)	2.1 (0.003)
-desthiomethyl-2- nitro (A11)	5.1 (0.002)	9.7 (0.03)	15 (0.04)	—	1.7 (0.001)	6.7 (0.001)	5.2 (0.003)	5.2 (0.006)
Parent and -4-nitro (A13)	2.6 (0.001)	6.8 (0.02)	5.2 (0.01)	—	3.5 (0.001)	3.7 (< 0.001)	1.9 (0.001)	2.5 (0.003)
-2-nitro (A14)	8.2 (0.003)	16 (0.05)	22 (0.06)	—	6.7 (0.003)	6.9 (0.001)	2.1 (0.001)	1.2 (0.001)
Unknown (A1)	14 (0.005)	3.0 (0.009)	12 (0.03)	—	7.1 (0.003)	43 (0.005)	5.2 (0.003)	4.2 (0.005)
Unknown (A2)	—	0.5 (0.002)	—	56 (0.02)	1.2 (< 0.001)	—	0.5 (< 0.001)	0.9 (0.001)
Unknown (A3)	8.2 (0.003)	12 (0.04)	12 (0.03)	—	—	—	7.8 (0.005)	11 (0.01)
Unknown (A4)	9.2 (0.003)	12 (0.04)	11 (0.03)	9.7 (0.003)	1.6 (0.001)	—	7.3 (0.004)	3.8 (0.005)
Unknown (A6)	9.1 (0.003)	11 (0.035)	2.5 (0.007)	—	2.4 (0.001)	—	5.8 (0.004)	6.4 (0.008)
Unknown (A12)	1.0 (< 0.001)	—	—	—	2.4 (0.001)	16.8 (0.002)	0.7 (< 0.001)	1.5 (0.002)
Unknown (A-Gr- 1)	7.1 (0.003)—	—	—	—	6.3 (0.002)	—	3.0 (0.002)	1.9 (0.002)
Sum total unknowns $< 10\%$	16 (0.005)	9.7 (0.03)	—	32 (0.01) ^a	25 (0.01)	—	26 (0.02)	22 (0.03)
Fenamidone-								
N-phenyl label, interval 191 days,% TRR (mg/kg in parentheses)								
-nitro-GSH (A7)	—	—	—	—	3.3 (0.001)	—	31 (0.006)	7.9 (0.003)
-hydroxyl- malonyl- glycoside (A8)	—	2.9 (0.002)	3.1 (0.002)	—	14 (0.003)	—	—	—
-desthiomethyl (A9)	—	—	—	—	—	—	—	—
-desthiomethyl-4- nitro (A10)	—	—	1.6 (0.001)	—	—	—	—	—
-desthiomethyl-2- nitro (A11)	—	9.6 (0.007)	12 (0.01)	—	—	—	—	10.5 (0.004)
Parent and -4-nitro (A13)	—	1.8 (0.001)	—	—	—	—	—	3.5 (0.001)
-2-nitro (A14)	5.5 (0.001)	10 (0.008)	5.6 (0.005)	—	—	—	—	4.4 (0.002)
Unknown (A1)	35 (0.006)	11 (0.008)	10 (0.009)	—	20 (0.004)	—	25 (0.005)	20 (0.007)
Unknown (A2)	—	—	9.3 (0.008)	—	6.6 (0.001)	—	—	—
Unknown (A3)	—	—	—	—	—	—	—	—
Unknown (A4)	4.2 (0.001)	5.2 (0.004)	6.6 (0.006)	—	—	—	—	2.4 (0.001)
Unknown (A6)	—	—	—	—	—	—	—	—
Unknown (A12)	—	—	—	—	—	—	—	—
Unknown (A-Gr- 1)	22 (0.004)	7.4 (0.006)	36 (0.03)	—	14 (0.003)	—	—	—
Sum total unknowns $< 10\%$	—	36 (0.03)	3.7 (0.003)	9.5 (0.001)	21 (0.005)	62 (0.004)	5.6 (0.001)	37 (0.01)
Fenamidone-								
N-phenyl label, interval 324 days,% TRR (mg/kg in parentheses)								
-nitro-GSH (A7)	—	—	—	—	4.0 (0.001)	—	—	—
-hydroxy- malonyl-	—	2.7 (0.002)	2.0 (0.002)	—	13 (0.003)	—	—	—

Compound/fracti on	Wheat forage	Wheat hay	Wheat straw	Wheat grain	Turnip leaves	Turnip roots	Swiss chard (intermediate)	Swiss chard (mature)
glycoside (A8)								
-desthiomethyl (A9)	–	–	–	–	–	–	–	–
-desthiomethyl-4- nitro (A10)	–	2.6 (0.002)	1.0 (0.001)	–	–	–	–	–
-desthiomethyl-2- nitro (A11)	17 (0.003)	8.9 (0.007)	7.7 (0.006)	–	–	–	–	–
Parent and -4-nitro (A13)	–	–	0.8 (0.001)	–	–	–	–	–
-2-nitro (A14)	10 (0.001)	2.9 (0.002)	4.7 (0.004)	–	–	–	–	–
Unknown (A1)	20 (0.003)	6.1 (0.005)	7.0 (0.005)	–	12 (0.002)	–	–	–
Unknown (A2)	–	4.7 (0.004)	7.0 (0.005)	–	8.6 (0.002)	–	–	–
Unknown (A3)	–	–	–	–	–	–	–	–
Unknown (A4)	–	9.4 (0.008)	–	–	3.3 (0.001)	–	–	–
Unknown (A6)	–	–	–	–	–	–	–	–
Unknown (A12)	–	–	–	–	–	–	–	–
Unknown (A-Gr- 1)	12 (0.002)	9.2 (0.007)	13 (0.01)	–	13 (0.003)	–	–	–
Sum total unknowns < 10%	7.7 (0.001)	34 (0.03)	33 (0.03)	31 (0.004)	19 (0.006)	–	55 (0.007)	67 (0.007)

^a Incorporated CO₂ (characterized by enzymatic digestion)

Low amounts of parent compound and fenamidone-4-nitro were detected together in one region of the conventional extracts, mainly in crops of the first rotation. The region amounted from < 0.001 mg/kg (3.7% of TRR) for turnip roots (1st rotation) to 0.02 mg/kg (6.8% of TRR) for wheat hay (1st rotation). Parent compound, fenamidone-desthiomethyl-2-nitro, fenamidone-desthiomethyl-4-nitro and fenamidone-2-nitro were prominent in the conventional extracts of the non-edible parts of wheat and amounted to ≤ 0.06 mg/kg. Fenamidone-nitro-GSH was a prominent metabolite in Swiss chard and amounted to ≤ 0.03 mg/kg.

In contrast to the earlier study, where the major components of the residue in succeeding crops were RPA 410193, RPA 412636, RPA 412708 and RPA 412708 conjugate, neither RPA 410193 nor RPA 412708 but fenamidone-hydantoin (RPA 412636) were found in the two newer studies. No nitro products were seen in the earlier study, whereas this route of metabolism is significant in the two newer studies. The following metabolic reactions were observed:

- Electrophilic nitration in the aniline ring of parent compound forming fenamidone-2-nitro and fenamidone-4-nitro (most likely in the soil)
- Conjugation with hexose and malonic acid after electrophilic nitration in the aniline ring and hydroxylation of the molecule forming fenamidone-hydroxy-nitro-malonyl-glycoside
- Nucleophilic substitution of methyl mercaptane in the imidazolinone ring of fenamidone-2-nitro by addition of water forming fenamidone-desthiomethyl-2-nitro
- Conjugation with glutathione after electrophilic nitration in the aniline ring of parent and nucleophilic substitution of methyl mercaptane forming fenamidone-nitro-GSH
- Cleavage of the aniline moiety of fenamidone-desthiomethyl-2-nitro forming fenamidone-hydantoin (most likely in the soil)
- Conjugation of fenamidone-hydantoin with glucose, hexose plus malonic acid and serine forming fenamidone-hydantoin-glucoside, fenamidone-hydantoin-malonyl-glycoside and fenamidone-hydantoin-serine

- Addition of ammonia by simultaneous elimination of water in the hydantoin ring forming fenamidone-imino-hydantoin
- Degradation of fenamidone-hydantoin to 2-phenyl-propionic acid
- Cleavage of the aniline moiety of parent compound followed by conjugation with hexose or serine forming fenamidone-thiohydantoin-glycoside and fenamidone-thiohydantoin-serine
- Cleavage of the aniline moiety of parent compound followed by hydroxylation of the methyl group of the imidazolinone ring forming fenamidone-hydroxymethyl-thiohydantoin
- Mineralisation of parent compound leading to carbon dioxide. Carbon dioxide was incorporated into natural compounds (e.g. starch).

Proposed degradation pathways for both the phenyl- and alanine-labelled forms of fenamidone in succeeding crops are presented in Figures 10 and 11.

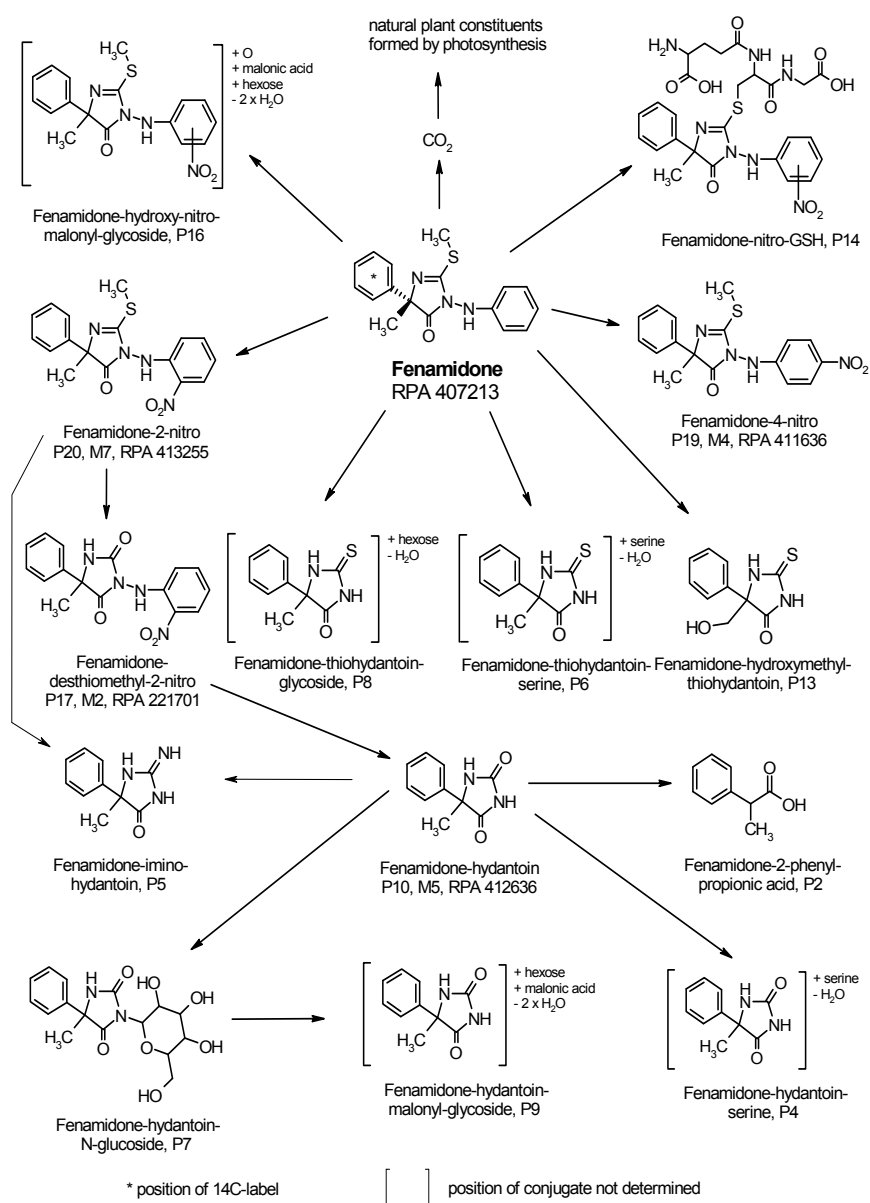


Figure 10 Proposed metabolic pathway of C-phenyl-¹⁴C-fenamidone in succeeding crops

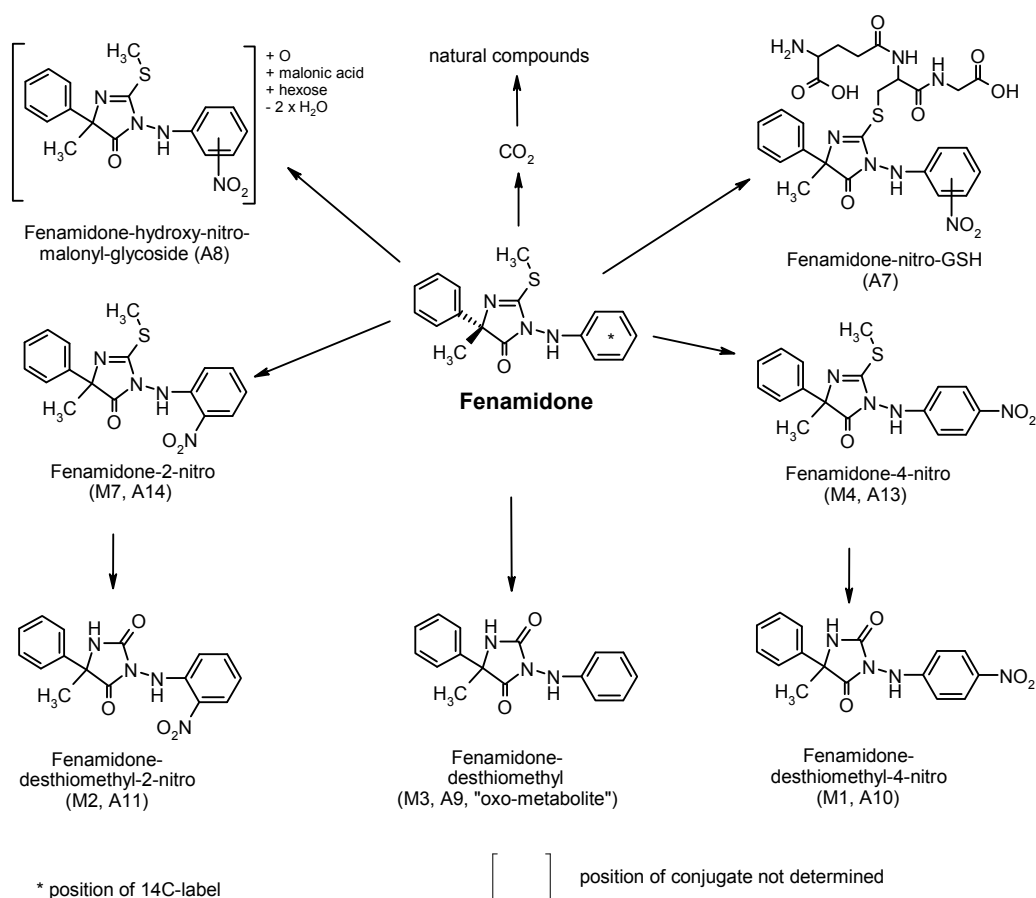


Figure 11 Proposed metabolic pathway of N-phenyl- ^{14}C -fenamidone in succeeding crops

Field succeeding crop studies

Seven field succeeding studies were carried out, one in Europe and six in the USA. Rotational crops were cereals (wheat, maize), fruiting vegetables (sweet corn) root and tuber vegetables (turnip, radish) leafy vegetables (lettuce, spinach), pulses (soya bean, dry) and strawberries.

Study by Cavallé, 2006; Report RA-2750/03, M-274302-01-1

Two field rotational crop trials with fenamidone have been conducted in Northern Europe, one in the UK and one in Germany. Six spray applications were made to target crops of potatoes, using fenamidone as SC formulation at a rate of 0.15 kg ai/ha. Rotational crops of wheat, turnip and lettuce were grown. Three plant back intervals of 30, 180 and 365 days were investigated. Samples of wheat forage, wheat grain, wheat straw, turnip leaf, turnip root and lettuce head were analysed at harvest for residues of fenamidone and its metabolites (RPA 410193, RPA 412636, RPA 412708 and RPA 412708 conjugate). The days after the last treatment of the previous crop ranged in the UK trial from 265 to 617 days for wheat forage, 361 to 719 days for wheat grain and straw, from 300 to 449 days for turnip leaf and root and from 118 to 449 days for lettuce head. In the German trial, the days after the last treatment of the previous crop ranged from 245 to 419 days for wheat forage, 354 to 740 days for wheat grain and straw, from 110 to 445 days for turnip leaf and root and from 83 to 419 days for lettuce head. In all samples, the residues were for all analytes lower than the LOQ of 0.01 mg/kg, with the exception of two wheat straw samples and one lettuce sample with residues of RPA 412708 conjugate at 0.01 mg/kg.

Samples were stored deep-frozen for periods up to 316 days for fenamidone and its metabolites (RPA 410193, RPA 412636 and RPA 412708), except for lettuce samples in the first rotation where the maximum storage interval was 480 days and for turnip in the first rotation where

the maximum storage interval was 463 days. It has been shown that fenamidone and its metabolites are stable for at least 12 months in stored frozen samples of potato (tuber), wheat (grain, hay, straw, forage) and leaf lettuce. The stability of fenamidone and its main plant metabolite RPA 410193 was demonstrated on strawberries over 18 months (M-247921-01-1) which is a representative commodity with high water content. Therefore the storage period of leaf samples (lettuce and turnip) is well covered by appropriate storage data.

Study by Norris, 2001; Report B003034, M-238683-01-1

A crop rotation study with fenamidone has been conducted in the USA. Two field rotational crop trials were conducted in California and Washington. Six broadcast applications were made to bare soil, using fenamidone as SC formulation at a rate of 0.2 kg ai/ha. Rotational crops of wheat, radish and spinach were grown. Two plant-back periods were investigated: 28/30 days and 201/234 days. Radish roots and tops, spinach leaves, and wheat forage (immature), hay, straw and grain, were harvested at maturity and analysed for residues of fenamidone, RPA 412636, RPA 412708, and RPA 410193. No measurable residues were found in any of the rotational crop fractions from the Washington site. However, measurable residues of RPA 412636 were found in many of the fractions from the California site (see Table 51). The maximum storage interval from harvest to extraction was 269 days. Adequate storage stability data are available to support the storage conditions and intervals.

Table 51 Residues of fenamidone and metabolites in rotated crops in California (two treated plots)

Plant material	Plant back interval ^a	DAT ^b	Residues, mg/kg			
			Fenamidone	RPA 410193	RPA 412636	RPA 412708
Spinach leaf	28	76	< 0.02	< 0.02	0.071	< 0.02
			< 0.02	< 0.02	0.12	< 0.02
	201	264	< 0.02	< 0.02	< 0.02	< 0.02
			< 0.02	< 0.02	< 0.02	< 0.02
Radish tops	28	76	< 0.02	< 0.02	0.022	< 0.02
			< 0.02	< 0.02	0.044	< 0.02
	201	264	< 0.02	< 0.02	< 0.02	< 0.02
			< 0.02	< 0.02	< 0.02	< 0.02
Radish roots	28	76	< 0.02	< 0.02	0.039	< 0.02
			< 0.02	< 0.02	< 0.02	< 0.02
	201	264	< 0.02	< 0.02	< 0.02	< 0.02
			< 0.02	< 0.02	< 0.02	< 0.02
Wheat forage	28	76	< 0.02	< 0.02	0.026	< 0.02
			< 0.02	< 0.02	0.028	< 0.02
	201	264	< 0.02	< 0.02	0.093	< 0.02
			< 0.02	< 0.02	0.075	< 0.02
Wheat hay	28	131	< 0.02	< 0.02	0.054	< 0.02
			< 0.02	< 0.02	0.036	< 0.02
	201	280	< 0.02	< 0.02	0.39	< 0.02
			< 0.02	< 0.02	0.45	< 0.02
Wheat straw	28	278	< 0.02	< 0.02	0.10	< 0.02
			< 0.02	< 0.02	0.076	< 0.02
	201	311	< 0.02	< 0.02	0.27	< 0.02
			< 0.02	< 0.02	0.17	< 0.02
Wheat grain	28	278	< 0.02	< 0.02	0.061	< 0.02
			< 0.02	< 0.02	< 0.02	< 0.02
	201	311	< 0.02	< 0.02	< 0.02	< 0.02
			< 0.02	< 0.02	< 0.02	< 0.02

^a Plant back interval: days between last treatment and planting or sowing

^b DAT: days after last treatment of previous crop

Study by Mackie, 2001; Report B003122, M-238779-01-1

A field rotational crop study has been conducted on wheat in the USA. Twenty-two field trials were conducted during the 1999/2000 growing season. A single application of fenamidone was made to bare soil as SC formulation at a rate of 1.2 kg ai/ha. Application was 30 ± 1 day before planting at test sites growing winter wheat varieties or spring wheat varieties that are seeded in the fall. Wheat forage,

hay, grain, and straw samples were collected at normal commercial harvest and analysed for fenamidone, RPA 412708, RPA 410193 and RPA 412636. The results are shown in Table 52. The maximum storage interval from harvest to extraction was 358 days. Adequate storage stability data are available to support the storage conditions and intervals.

Table 52 Residues of fenamidone and metabolites in rotated crops (wheat)

Trial	Plant back interval	Plant part	Residue, mg/kg			
			Fenamidone	RPA 412708	RPA 410193	RPA 412636
18273-01 Lucama, USA	31	Forage	< 0.02	< 0.02	< 0.02	< 0.02
		Hay	< 0.02	< 0.02	< 0.02	0.03
		Straw	< 0.02	< 0.02	< 0.02	< 0.02
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-02 Proctor, USA	30	Forage	< 0.02	< 0.02	< 0.02	< 0.02
		Hay	< 0.02	< 0.02	< 0.02	0.02
		Straw	< 0.02	< 0.02	< 0.02	< 0.02
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-03 Carlyle, USA	29	Forage	< 0.02	< 0.02	< 0.02	< 0.02
		Hay	< 0.02	< 0.02	< 0.02	0.02
		Straw	< 0.02	< 0.02	< 0.02	< 0.02
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-04 Bagley, USA	30	Forage	< 0.02	< 0.02	< 0.02	< 0.02
		Hay	< 0.02	< 0.02	< 0.02	0.04
		Straw	< 0.02	< 0.02	< 0.02	0.03
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-05 York, USA	195	Forage	< 0.02	< 0.02	< 0.02	0.07
		Hay	< 0.02	< 0.02	< 0.02	0.05
		Straw	< 0.02	< 0.02	< 0.02	0.02
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-06 Colony, USA	28	Forage	< 0.02	< 0.02	< 0.02	0.05
		Hay	< 0.02	< 0.02	< 0.02	0.32
		Straw	< 0.02	< 0.02	< 0.02	0.08
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-07 Conklin, USA	30	Forage	< 0.02	< 0.02	< 0.02	0.02
		Hay	< 0.02	< 0.02	< 0.02	< 0.02
		Straw	< 0.02	< 0.02	< 0.02	< 0.02
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-08 Uvalde, USA	87	Forage	< 0.02	< 0.02	< 0.02	< 0.02
		Hay	< 0.02	< 0.02	< 0.02	0.10
		Straw	< 0.02	< 0.02	< 0.02	0.02
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-09 Velva, USA	213	Forage	< 0.02	< 0.02	< 0.02	0.02
		Hay	< 0.02	< 0.02	< 0.02	< 0.02
		Straw	< 0.02	< 0.02	< 0.02	< 0.02
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-10 Velva, USA	213	Forage	< 0.02	< 0.02	< 0.02	< 0.02
		Hay	< 0.02	< 0.02	< 0.02	< 0.02
		Straw	< 0.02	< 0.02	< 0.02	< 0.02
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-11 Velva, USA	217	Forage	< 0.02	< 0.02	< 0.02	0.03
		Hay	< 0.02	< 0.02	< 0.02	0.16
		Straw	< 0.02	< 0.02	< 0.02	0.03
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-12 Britton, USA	224	Forage	< 0.02	< 0.02	< 0.02	< 0.02
		Hay	< 0.02	< 0.02	< 0.02	< 0.02
		Straw	< 0.02	< 0.02	< 0.02	< 0.02
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-13 Britton, USA	224	Forage	< 0.02	< 0.02	< 0.02	< 0.02
		Hay	< 0.02	< 0.02	< 0.02	< 0.02
		Straw	< 0.02	< 0.02	< 0.02	< 0.02
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-14 Colony, USA	30	Forage	< 0.02	< 0.02	< 0.02	0.03
		Hay	< 0.02	< 0.02	< 0.02	< 0.02
		Straw	< 0.02	< 0.02	< 0.02	0.02

Fenamidone

Trial	Plant back interval	Plant part	Residue, mg/kg			
			Fenamidone	RPA 412708	RPA 410193	RPA 412636
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-15 Lamed, USA	29	Forage	< 0.02	< 0.02	< 0.02	0.02
		Hay	< 0.02	< 0.02	< 0.02	0.04
		Straw	< 0.02	< 0.02	< 0.02	0.20
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-16 Lamed, USA	30	Forage	< 0.02	< 0.02	< 0.02	0.03
		Hay	< 0.02	< 0.02	< 0.02	0.26
		Straw	< 0.02	< 0.02	< 0.02	0.03
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-17 Plainview, USA	29	Forage	< 0.02	0.04	< 0.02	< 0.02
		Hay	< 0.02	0.02	< 0.02	0.07
		Straw	< 0.02	< 0.02	< 0.02	0.02
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-18 Las Cruces, USA	30	Forage	< 0.02	< 0.02	< 0.02	< 0.02
		Hay	< 0.02	< 0.02	< 0.02	0.07
		Straw	< 0.02	< 0.02	< 0.02	0.04
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-19 Levelland, USA	29	Forage	< 0.02	< 0.02	< 0.02	0.02
		Hay	< 0.02	< 0.02	< 0.02	0.02
		Straw	< 0.02	< 0.02	< 0.02	0.05
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-20 Ephrata, USA	30	Forage	< 0.02	0.07	< 0.02	0.03
		Hay	< 0.02	< 0.02	< 0.02	0.03
		Straw	< 0.02	< 0.02	< 0.02	0.04
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-21 Porterville, USA	30	Forage	< 0.02	< 0.02	< 0.02	< 0.02
		Hay	< 0.02	< 0.02	< 0.02	0.07
		Straw	< 0.02	< 0.02	< 0.02	0.07
		Grain	< 0.02	< 0.02	< 0.02	< 0.02
18273-22 Watsonville, USA	30	Forage	< 0.02	0.04	< 0.02	< 0.02
		Hay	< 0.02	< 0.02	< 0.02	0.05
		Straw	< 0.02	< 0.02	< 0.02	0.04
		Grain	< 0.02	< 0.02	< 0.02	< 0.02

Study by Beedle and Harbin, 2004; Report 201165, M-242538-01-1

A field rotational crop study has been conducted on strawberries in the USA. Eight field trials were conducted in 2003. Four broadcast foliar applications of fenamidone were made to the primary crop (a leafy vegetable, cucurbit or fruiting vegetable) as SC formulation at a rate of 0.3 kg ai/ha, and with 4–5 days between applications. The primary crop was tilled into the ground at least 2 days after the final application, and strawberries were planted as a rotational crop 28 to 30 days after the last application of the previous crop. Strawberries were collected at normal harvest. The residues of fenamidone and its metabolites RPA 412708, RPA 410193 and RPA 412636 were quantitated in strawberries by LC-MS/MS with an LOQ of 0.02 mg/kg. In all eight trials, no residues of fenamidone, RPA 412708, RPA 410193 and RPA 412636 greater than the LOQ of 0.02 mg/kg were observed in strawberries as rotational crop. The maximum storage interval from harvest to extraction was 294 days. Adequate storage stability data are available to support the storage conditions and intervals.

Further to the studies described above, field rotational crop studies have been conducted in the USA with sweet corn, maize and soya beans. The samples were analysed for fenamidone as well as for the sum of RPA 412636 and RPA 412708 (measured as RPA 412636) by LC-MS/MS. The results (mean of two field samples) are summarized in Table 53.

Study by Hartz and Helfrich, 2008; Report RARPY018, M-305323-01-1

A field rotational crop study has been conducted on sweet corn in the USA. Twelve field trials were conducted during the 2007 growing season. Three applications of fenamidone were made to bare soil as a SC formulation at a target rate of 0.3 kg ai/ha, and with 5–9 days between applications. The soil was tilled after the third application to simulate a failed crop and sweet corn was then planted with a

target planting interval of 25 to 30 days. Sweet corn forage and kernel plus cob with husks removed were collected at normal commercial harvest (BBCH 73–79). Stover (fodder) was collected at BBCH 89. All the samples were analysed for fenamidone as well as for RPA 412636 and RPA 412708 (measured as RPA 412636). The results are summarized in Table 53. The maximum storage interval from harvest to extraction was 344 days. Adequate storage stability data are available to support the storage conditions and intervals.

Study by Hartz and Helfrich, 2008; Report RARPY021, M-305368-01-1

A field rotational crop study has been conducted on maize in the USA. Nine field trials were conducted during the 2007 growing season. Three applications of fenamidone were made to bare soil as SC formulation at a target rate of 0.3 kg ai/ha, and with 5–8 days between applications. The soil was tilled after the third application to simulate a failed crop and maize was then planted with a target planting interval of 25 to 30 days. Maize forage was collected at BBCH 85–87. Grain and stover (fodder) were collected at BBCH 89 (normal commercial harvest). All the samples were analysed for fenamidone as well as for RPA 412636 and RPA 412708 (measured as RPA 412636). The results are summarized in Table 53. The maximum storage interval from harvest to extraction was 336 days. Adequate storage stability data are available to support the storage conditions and intervals.

Study by Hartz, 2008; Report RARPY017, M-305308-01-1

A field rotational crop study has been conducted on soya beans in the USA. Eleven field trials were conducted during the 2007 growing season. Three applications of fenamidone were made to bare soil as SC formulation at a target rate of 0.3 kg ai/ha, and with 5–10 days between applications. The soil was tilled after the third application to simulate a failed crop. Soya bean was then sown with an interval of 25 to 30 days. Soya forage and hay were collected at BBCH 65–74. Soya bean seed (grain) was collected at BBCH 89 (normal commercial harvest). All the samples were analysed for fenamidone as well as for RPA 412636 and RPA 412708 (measured as RPA 412636). The results are summarized in Table 53. The maximum storage interval from harvest to extraction was 337 days. Adequate storage stability data are available to support the storage conditions and intervals.

Table 53 Residues of fenamidone, RPA 412636 and RPA 412708 in rotated crops

Rotated crop	Trial, location	Plant back interval, days	Commodity	Fenamidone, mg/kg	412636 + 412708, mg/kg ^a
Sweet corn	RP021-07RA, Germansville, USA	30	Forage	< 0.02	0.03
			Stover	< 0.02	0.09
			Kernels plus cob without husk	< 0.02	< 0.02
Sweet corn	RP022-07RA, North Rose, USA	25	Forage	< 0.02	0.04
			Stover	< 0.02	0.07
			Kernels plus cob without husk	< 0.02	< 0.02
Sweet corn	RP023-07RA, Athens, USA	28	Forage	< 0.02	0.10
			Stover	< 0.02	0.29
			Kernels plus cob without husk	< 0.02	< 0.02
Sweet corn	RP024-07RA, High Springs, USA	30	Forage	< 0.02	< 0.02
			Stover	< 0.02	0.03
			Kernels plus cob without husk	< 0.02	< 0.02
Sweet corn	RP025-07RA, New Holland, USA	26	Forage	< 0.02	0.02
			Stover	< 0.02	0.04
			Kernels plus cob without husk	< 0.02	< 0.02
Sweet corn	RP026-07RA, Richwood, USA	27	Forage	< 0.02	0.03
			Stover	< 0.02	< 0.02
			Kernels plus cob without husk	< 0.02	< 0.02
Sweet corn	RP027-07RA, Richland, USA	30	Forage	< 0.02	< 0.02
			Stover	< 0.02	< 0.02
			Kernels plus cob without husk	< 0.02	< 0.02
Sweet corn	RP028-07RA, Gardner, USA	28	Forage	< 0.02	< 0.02
			Stover	< 0.02	0.04
			Kernels plus cob without husk	< 0.02	< 0.02
Sweet corn	RP029-07RA, Geneva, USA	25	Forage	< 0.02	< 0.02
			Stover	< 0.02	< 0.02
			Kernels plus cob without husk	< 0.02	< 0.02

Fenamidone

Rotated crop	Trial, location	Plant back interval, days	Commodity	Fenamidone, mg/kg	412636 + 412708, mg/kg ^a
Sweet corn	RP030-07RA, Hickman, USA	28	Forage	< 0.02	0.07
			Stover	< 0.02	0.11
			Kernels plus cob without husk	< 0.02	< 0.02
Sweet corn	RP031-07RA, Rupert, USA	29	Forage	< 0.02	0.02
			Stover	< 0.02	0.08
			Kernels plus cob without husk	< 0.02	< 0.02
Sweet corn	RP032-07RA, Cornelius, USA	28	Forage	< 0.02	< 0.02
			Stover	< 0.02	0.02
			Kernels plus cob without husk	< 0.02	< 0.02
Maize (Field corn)	RP035-07RA, North Rose, USA	25	Forage	< 0.02	0.02
			Stover	< 0.02	0.02
			Grain	< 0.02	< 0.02
Maize (Field corn)	RP036-07RA, Athens, USA	28	Forage	< 0.02	0.21
			Stover	< 0.02	0.05
			Grain	< 0.02	< 0.02
Maize (Field corn)	RP037-07RA, Richwood, USA	27	Forage	< 0.02	0.02
			Stover	< 0.02	0.05
			Grain	< 0.02	< 0.02
Maize (Field corn)	RP040-07RA, Richland, USA	26	Forage	< 0.02	< 0.02
			Stover	< 0.02	< 0.02
			Grain	< 0.02	< 0.02
Maize (Field corn)	RP041-07RA, Rockwood, USA	27	Forage	< 0.02	0.02
			Stover	< 0.02	< 0.02
			Grain	< 0.02	< 0.02
Maize (Field corn)	RP042-07RA, Clarence, USA	25	Forage	< 0.02	0.04
			Stover	< 0.02	0.02
			Grain	< 0.02	< 0.02
Maize (Field corn)	RP043-07RA, Campbell, USA	28	Forage	< 0.02	< 0.02
			Stover	< 0.02	< 0.02
			Grain	< 0.02	< 0.02
Maize (Field corn)	RP048-07RA, Springfield, USA	24	Forage	< 0.02	< 0.02
			Stover	< 0.02	< 0.02
			Grain	< 0.02	< 0.02
Maize (Field corn)	RP054-07RA, East Bernard, USA	28	Forage	< 0.02	0.04
			Stover	< 0.02	0.13
			Grain	< 0.02	< 0.02
Soya bean	RP001-07RA, Athens, USA	28	Forage	< 0.02	0.05
			Hay	< 0.02	0.07
			Seed	< 0.02	< 0.02
Soya bean	RP002-07RA, Suffolk, USA	30	Forage	< 0.02	< 0.02
			Seed	< 0.02	< 0.02
Soya bean	RP003-07RA, Newport, USA	25	Forage	< 0.02	< 0.02
			Hay	< 0.02	0.02
			Seed	< 0.02	< 0.02
Soya bean	RP004-07RA, Proctor, USA	30	Forage	< 0.02	< 0.02
			Hay	< 0.02	< 0.02
			Seed	< 0.02	< 0.02
Soya bean	RP005-07RA, Cheneyville, USA	26	Forage	< 0.02	< 0.02
			Hay	< 0.02	0.03
			Seed	< 0.02	< 0.02
Soya bean	RP007-07RA, Carlyle, USA	29	Forage	< 0.02	0.05
			Hay	< 0.02	0.05
			Seed	< 0.02	< 0.02
Soya bean	RP008-07RA, New Holland, USA	25	Forage	< 0.02	0.04
			Hay	< 0.02	0.06
			Seed	< 0.02	< 0.02
Soya bean	RP010-07RA, Richland, USA	25	Seed	< 0.02	< 0.02
Soya bean	RP012-07RA, York, USA	26	Forage	< 0.02	0.03
			Hay	< 0.02	0.05
			Seed	< 0.02	< 0.02
Soya bean	RP015-07RA,	26	Forage	< 0.02	< 0.02

Rotated crop	Trial, location	Plant back interval, days	Commodity	Fenamidone, mg/kg	412636 + 412708, mg/kg ^a
	Sheridan, USA		Hay	< 0.02	0.02
			Seed	< 0.02	< 0.02
Soya bean	RP020-07RA, Breslau, USA	27	Forage	< 0.02	0.08
			Hay	< 0.02	0.14
			Seed	< 0.02	< 0.02

^a Sum of RPA 412636 and RPA 412708, measured as RPA 412636

In summary, after application of 0.9–1.2 kg ai/ha to the bare soil, in the following crops no residues of fenamidone were found at or above the LOQ of 0.02 mg/kg in human food but metabolites as RPA412708 and RPA412636 occurred in commodities that may be used as animal feeds. The sum of RPA412708 and RPA412636 reached up to 0.14 in soya bean hay, 0.29 mg/kg in sweet corn stover and 0.21 mg/kg in field corn forage; RPA412636 occurred up to 0.07 mg/kg in wheat forage, 0.27 mg/kg in wheat straw and 0.45 mg/kg in wheat hay.

RESIDUE ANALYSIS

Analytical methods

The Meeting received descriptions and validation data for analytical methods for residues of fenamidone and its relevant metabolites RPA 410193, RPA 412708 and RPA 17879 in plant and animal commodities. Residue analytical methods rely on GC with NP-detection, GC-MS or LC-MS/MS. Typical LOQs achieved for plant and animal commodities fall in the range of 0.01–0.02 mg/kg. Methods have been subjected to independent laboratory validation (ILV). The methods described briefly below have been used for the analysis of the samples generated during the supervised field trials, processing fraction studies and storage stability investigations (report number in brackets).

Plant commodities

Method number AR 150-97, M-165219-01-1

Validation: Report No. 98-139 (R004058), M-165838-01-1

Radiovalidation: Report No. 99-82 (R004785), M-166513-01-1

ILV: Report No. 99-23 (R005021), M-170314-01-1

Analytes: Fenamidone, RPA 410193

LOQ: 0.02 mg/kg (GC-NPD, GC-MS), 0.01 mg/kg (LC-MS/MS)

Description: Fenamidone and RPA 410193 residues are extracted from homogenized sample by acetone / water. The raw extract is purified using a polystyrene-divinylbenzene cartridge SPE followed by an aminopropyl (NH₂) SPE cartridge. Quantification was by GC-NPD, GC-MS or LC-MS/MS.

Residue trials: Grapes (97–579, 97–580, 97–581, 97–582, 97–636, 97–687, 97–742, 98–561, 98–562, 98–563, 98–631, 98–690, 98–721, R.19/00, R.20/00, R.33/01, R.34/01, R.48/01), Bulb onions (R.75/02, R.82/02, R.83/02), Melons (00–535, 00–536, 02 R 655, 02 R 656), Cucumbers (02 R 651, 02 R 653), Watermelons (UNESP RA-944/05, UNESP RA-945/05, UNESP RA-946/05), Lettuce (98–514, 99–506), Potatoes (97–555, 97–556, 97–557, 97–558, 97–691, 97–745, 98–525, 98–526, 98–527, 98–635, 98–669, 98–688, 98–700, 99–648, 99–664, R.23/01, R.32/01, R.50/01).

Method number 00868, M-229394-01-1

Validation: Report No. P 1980 G, M-370932-01-1

Analytes: Fenamidone, RPA 410193

LOQ: 0.01 mg/kg (LC-MS/MS)

Description: Fenamidone and RPA 410193 residues are extracted by homogenisation with acetone / water. After centrifugation an aliquot was concentrated to the aqueous remainder and dissolved in acidified acetonitrile/water. Quantification was by LC-MS/MS.

Residue trials: Grapes (09-2179, 09-2180), Strawberries (09-2169), Leek (RA-2682/03), Bulb onion (RA-2683/03), Witloof chicory (RA-2566/04, 08-2205), Head cabbage (RA-2900/03, RA-2901/03), Cauliflower (RA-2902/03, RA-2903/03), Melons (RA-2536/05), Cucumbers (RA 2648/03, RA 2540/04), Tomatoes (09-2235, 09-3235, RA-2541/05), Potatoes (09-2170), Lettuce (RA-2542/04, RA-2518/05, RA-2519/05, RA-2553/06, 09-2158, 09-2159, 09-2216)

Method number RPAC 45683, M-238850-01-1, method appended, p. 99

Validation: Report No. 019-056 (B003198), M-238850-01-1
 Analytes: Fenamidone, RPA 410193, RPA 412636, RPA 412708
 LOQ: 0.02 mg/kg (LC-MS/MS)
 Description Method analogue to AR 150-97

Method number unknown, M-238851-01-1, method appended, p. 48

Validation: Report No. B003199, M-238851-01-1
 Radiovalidation: Report No. 019-057 (B003200), M-238852-01-1
 Analytes: Fenamidone, RPA 410193, RPA 412636, RPA 412708
 LOQ: 0.02 mg/kg (LC-MS/MS)
 Description Method analogue to AR 150-97
 Residue trials: Ginseng (09800), Lima beans (09530), Snap beans (08895)

Method number RPAC 45911, M-238680-01-1 (method appended, p. 199)

Analytes: Fenamidone, RPA 412636, RPA 410193 (412708), RPA 410193
 LOQ: 0.02 mg/kg (LC-MS/MS)
 Description: Fenamidone, RPA 412636, RPA 412708 and RPA 410193 are extracted from homogenized sample matrix by accelerated solvent extraction (ASE) with acetone / water at enhanced temperature conditions. After dilution with water and filtration, quantification was by LC-MS/MS.
 Residue trials: Onions (98W14461), Green onions (98W14461), Cabbage and broccoli (201225), Melons (99W14488), Cucumbers (99W14486), Summer squash (99W14487), Peppers (201227), Tomatoes (99W17533), Lettuce (00W13043, 00W14459), Mustard greens (201226), Spinach (201214), Carrots (08524), Potatoes (99W17534, 01AC05), Celery (201214), Sunflower seed (07999) and storage stability study EC-99-448

Method number RPAC 45952, M-240317-01-1

Analytes: Fenamidone, RPA 410193 (RPA 412708), RPA 412636
 LOQ: 0.02 mg/kg (LC-MS/MS)
 Description: Fenamidone and metabolites are extracted from homogenized sample matrix by accelerated solvent extraction (ASE) with acetone / water at enhanced temperature conditions. After dilution with acetone extracts were cleaned up on a laboratory packed column containing carbon, silica and alumina. Final extracts are quantified by LC-MS/MS.
 Residue trials: Cotton seed and by-products (201163)

Method number 01163, M-354028-01-1

Validation: Bayer Ref: M-449868-01-1

Analytes: Fenamidone, RPA 410193

LOQ: 0.01 mg/kg (LC-MS/MS)

Description: Modified QuEChERS method for crop matrices. Water is added to the sample material depending on its moisture content. Residues of fenamidone and RPA 410193 are extracted from sample material by vigorous shaking with acetonitrile. For liquid-liquid partition and phase separation, salt (magnesium sulphate, sodium chloride, sodium citrate and sodium hydrogen citrate) is added to the homogenate and the sample is shaken again. After centrifugation an aliquot of the upper organic phase is diluted with acetonitrile/water. This final extract is quantified by LC-MS/MS.

Residue trials: Grapes (10–2010, 10–2011), Strawberries (10–2014, 11–2004, 12–2039), Tomatoes (10–2013), Lettuce (10–2012), Potatoes (10–2065), Witloof chicory (10–2040)

Method number 00862, Bayer Reference: M-255146-01-1

Analytes: Fenamidone, RPA 410193, RPA 412636 and RPA 412708

LOQ: 0.01 mg/kg (LC-MS/MS)

Description: Fenamidone, RPA 410193, RPA 412636 and RPA 412708 are extracted from crop materials using acetone/water in a high-speed blender. After centrifuging, the supernatant is removed and the solids re-extracted with acetone/water. An aliquot of the combined supernatants is concentrated to the aqueous phase and adjusted to volume with acidified water, then filtered for analysis by LC/MS/MS.

Study: Rotational crops; turnip, lettuce, wheat (RA-2750/03)

Method number 00863, Bayer Reference: M-274241-01-1

Analytes: RPA 412708 conjugate

LOQ: 0.01 mg/kg (LC-MS/MS)

Description: RPA 412708 conjugate is extracted from samples with acetone: water (5:1 v/v) twice, the samples blended in a blender and centrifuged. The supernatant is removed, further acetone added and concentrated to an aqueous remainder using a sample concentrator. Formic acid is added and the sample heated in an oven for 2 hours at 70 °C to hydrolyse. Subsamples of the hydrolysed extracts are mixed with acetonitrile or water, filtered (0.2 µm filter) and analysed by LC-MS/MS.

Study: Rotational crops; turnip, lettuce, wheat (RA-2750/03)

Animal commodities

Method number AR 200-99, M-166398-01-1

Validation: Report No. 99-95, M-183960-01-1

Analytes: Fenamidone, RPA 412708, RPA 412636

LOQ: 0.01 mg/kg (GC-NPD)

Description: Residues of fenamidone, RPA412708 and RPA412636 are extracted from milk samples by homogenisation with acetonitrile. The extracts are purified using an octadecyl cartridge. Final determination of residues is by GC on a semi-capillary column, using a thermionic detector and quantification against external standards.

Residue trials: Milk (00W25043)

Method number AR 178-98, M-166460-01-1

ILV: Report No. 99-52, M-183964-01-1

Analytes: Fenamidone, RPA 412708, RPA 412636

LOQ: 0.05 mg/kg (GC-NPD)

Description: Residues of fenamidone, RPA412708 and RPA412636 are extracted from animal products by homogenisation with acetonitrile. The extracts are purified using a polystyrene divinylbenzene cartridge followed by an aminopropyl cartridge. For kidney, whole egg and muscle, final determination of residues is by gas chromatography on a semi-capillary column, using a thermionic detector and quantification against external standards. For liver, final determination of residues is by GC-MS and quantification against external standards.

Residue trials: Animal products (00W25043)

Multi-residue methods

Plant material: Method number DFG S19, M-368426-01-1

Validation: Report No. R&D/CRLD/AN/9915480, M-166310-01-1

Analytes: Fenamidone, RPA410193

LOQ: 0.01 mg/kg (GC-MS)

Description: Residues of fenamidone and RPA410193 are extracted from the plant samples by homogenisation with water / acetone, then partition into ethyl acetate / cyclohexane. The organic extracts are cleaned up by fractionation using gel-permeation chromatography, followed by purification using a silica column. Final determination of residues is by GC-MS, monitoring three ions per analyte for determination and confirmation.

Animal products: Method number 01323, M-410297-01-1

ILV: Report No. 2011/0107/01, M-440254-01-1

Analyte: Fenamidone, RPA 412708 and RPA 412636

LOQ: 0.01 mg/kg (LC-MS/MS)

Description: QuEChERS multi residue method for animal matrices: For milk, residues of fenamidone, RPA 412708 and RPA 412636 are extracted by vigorous shaking with acetonitrile. Salts (magnesium sulphate, sodium chloride, sodium citrate and sodium hydrogen citrate) are added and the sample is shaken again. A portion of the acetonitrile layer is transferred to a dispersive SPE clean-up tube, shaken and centrifuged before aliquots of the supernatant are acidified for determination of fenamidone, RPA 412708 and RPA 412636 by LC-MS/MS). RPA 412636 is determined separately from fenamidone and RPA 412708.

For eggs, muscle, liver and kidney, residues of fenamidone, RPA 412708 and RPA 412636 are extracted by vigorous shaking with acetonitrile (and homogenisation for muscle, liver and kidney). Salts (magnesium sulphate, sodium chloride, sodium citrate and sodium hydrogen citrate) are added and the sample is shaken again. Fat is frozen out of the extract before a portion of the acetonitrile layer is transferred to a dispersive SPE clean-up tube, shaken and centrifuged before aliquots of the supernatant are acidified for determination of fenamidone, RPA 412708 and RPA 412636 by LC-MS/MS as for milk.

For fat, samples are melted (60 °C) and partitioned with water and acetonitrile. After re-melting, salts (magnesium sulphate, sodium chloride, sodium citrate and sodium hydrogen citrate) are added and the sample is shaken again. Fat is frozen out of the extract before a portion of the acetonitrile layer is transferred to a dispersive SPE clean-up tube, shaken and centrifuged before aliquots of the supernatant are acidified for determination of fenamidone, RPA 412708 and RPA 412636 by LC-MS/MS as for milk.

Validation data for the methods described above are available from specific method validation studies or from residue studies where specific method validation recovery experiments were performed separate from routine sample analysis. These method recovery data, for plant and animal commodities are summarised in Table 54.

Table 54 Method recovery data of fenamidone and metabolites in plants and animal products

Commodity	Analytes	Fortification, mg/kg	n	Recovery, %			Method	Reference
				Range	Mean	RSD		
Plant commodities								
Grapes	Fenamidone	0.02	1	117	117	—	AR 150-97	97-580
	RPA 410193	0.02	1	89	89	—		M-165259-01-1
Grapes	Fenamidone	0.2	1	93	93	—	AR 150-97	97-581
	RPA 410193	0.2	1	116	116	—		M-165494-01-1
Grapes	Fenamidone	0.02	5	70–77	73	3.5	AR 150-97	97-582
	RPA 410193	0.02	5	73–114	96	19		M-165735-01-2
Grapes	Fenamidone	0.1, 0.4	2	70, 80	75	—	AR 150-97	97-636
	RPA 410193	0.1, 0.4	2	94, 100	97	—		M-165270-01-1
Grapes	Fenamidone	0.02	2	90, 99	95	—	AR 150-97	97-687
	RPA 410193	0.02	2	98, 103	101	—		M-165275-01-1
Grapes	Fenamidone	0.02	2	99, 97	98	—	AR 150-97	97-742
	RPA 410193	0.02	2	102, 101	102	—		M-165477-01-1
Grapes	Fenamidone	0.02–0.1	3	82–102	91	11	AR 150-97	98-563
	RPA 410193	0.02–0.1	3	92–101	96	4.9		M-166547-01-1
Grapes	Fenamidone	0.02–0.5	9	84–96	92	4.7	AR 150-97 (R003696)	R.19/00, R.20/00, R.33/01, R.34/01 M-285309-01-1, M-285311-01-1, M-285002-01-1, M-285003-01-1
Grapes	RPA 410193	0.02–0.10	6	91–101	95	3.5	AR 150-97 (R003696)	R.33/01, R.34/01 M-285002-01-1, M-285003-01-1
Grapes	Fenamidone	0.02–0.10	6	96–100	97	1.6	AR 150-97 (R003696)	R.48.01
	RPA 410193	0.02–0.10	6	91–101	95	3.5		M-285004-01-1
Onion (bulb)	Fenamidone	0.02–0.3	6	87–109	97	9.2	RPAC 45911	EC-99-448
	RPA 410193	0.02–0.3	6	102–110	106	3.2		M-238849-01-1
Onion (bulb)	Fenamidone	0.02–0.3	6	87–109	97	8.7	RPAC 45911	98W14461 M-238680-01-1
	RPA 410193	0.02–0.3	6	99–104	101	2.3		
Onion (green)	Fenamidone	0.02–0.3	6	83–110	96	10	RPAC 45911	98W14461 M-238680-01-1
	RPA 410193	0.02–0.3	6	106–119	110	5.3		
Onion (bulb)	Fenamidone	0.02–0.08	6	80–94	89	6.5	AR 150-97 (R003696)	R.75/02, R.82/02, R.83/02 M-285013-01-1, M-284950-01-1, M-284958-01-1
	RPA 410193	0.02–0.08	6	78–87	81	4.3		
Cabbage	Fenamidone	0.01–1.5	12	93–108	102	5.6	00868	RA-2900/03, RA- 2901/03 M-248911-01-1, M-248027-01-1
	RPA 410193	0.01–1.5	12	92–109	100	4.9		
Head cabbage with wrapper leaves	Fenamidone	0.02–1.0	11	84–152	100	18	RPAC 45911	201225 M-243561-01-3
	RPA 410193	0.02–1.0	11	81–111	102	8.9		
Head cabbage w/o wrapper leaves	Fenamidone	0.02–1.0	8	87–121	105	14	RPAC 45911	201225 M-243561-01-3
	RPA 410193	0.02–1.0	8	107–118	112	3.7		
Cauliflower	Fenamidone	0.01–1.0	7	88–104	97	5.8	00868	RA-2902/03, RA- 2903/03 M-247895-01-1, M-247987-01-1
	RPA 410193	0.01–1.0	7	85–104	98	6.8		
Broccoli	Fenamidone	0.02–5.0	10	62–112	87	20	RPAC 45911	201225 M-243561-01-3
	RPA 410193	0.02–5.0	9	74–116	97	14		
Melon (fruit)	Fenamidone	0.02–0.2	9	74–132	101	17	AR 150-97	00-535 M-205672-01-1
	RPA 410193	0.02–0.2	9	88–132	111	12		
Melon (fruit)	Fenamidone	0.02	10	71–116	87	15	AR 150-97	02 R 655 M-219005-01-1
	RPA 410193	0.02	10	93–114	103	6.5		
Melon (peel)	Fenamidone	0.02	7	71–102	86	14		
	RPA 410193	0.02	7	84–107	95	10		
Melon (pulp)	Fenamidone	0.02	6	85–110	98	10		
	RPA 410193	0.02	6	93–112	102	7.5		

Fenamidone

Commodity	Analytes	Fortification, mg/kg	n	Recovery, %			Method	Reference
				Range	Mean	RSD		
Melon (fruit)	Fenamidone	0.01–0.1	6	97–101	99	1.7	00868	RA-2536/05
	RPA 410193	0.01–0.1	6	98–103	101	1.9		M-279019-01-1
Melon (fruit)	Fenamidone	0.02–0.30	6	84–95	89	4.3	RPAC	99W14488
	RPA 410193	0.02–0.30	6	111–118	114	2.3	45911	M-240409-01-1
Cucumber (fruit)	Fenamidone	0.02–0.30	6	97–114	104	6.3	RPAC	EC-99-448
	RPA 410193	0.02–0.30	6	107–120	112	4.7	45911	M-238849-01-1
Cucumber (fruit)	Fenamidone	0.01–0.1	10	89–105	101	6.3	00868	RA 2648/03
	RPA 410193	0.01–0.1	10	91–102	95	3.6		M-235795-01-1
Cucumber (fruit)	Fenamidone	0.02–0.30	6	113–120	117	2.0	RPAC	99W14486
	RPA 410193	0.02–0.30	6	125–134	129	2.7	45911	M-240408-01-1
Squash (fruit)	Fenamidone	0.02–1.0	6	96–113	107	5.6	RPAC	99W14487
	RPA 410193	0.02–1.0	6	99–115	107	5.3	45911	M-240407-01-1
Watermelon (whole fruit)	Fenamidone	0.02–0.80	15	87–98	91	3.3	AR 150-97 (R003696)	UNESP RA-944/05,
	RPA 410193	0.02–0.80	15	85–93	89	3.1		UNESP RA-945/05, UNESP RA-946/05 M-276178-01-1, M-276180-01-1, M-276183-01-1
Pepper	Fenamidone	0.02–3.0	13	73–107	88	10	RPAC	201227
	RPA 410193	0.02–0.50	11	97–117	109	6.6	45911	M-260625-01-1
Tomato (fruit)	Fenamidone	0.02–0.30	6	80–107	96	9.8	RPAC	EC-99-448
	RPA 410193	0.02–0.30	6	93–106	100	5.4	45911	M-238849-01-1
Tomato (paste)	Fenamidone	0.02–0.30	6	75–96	84	10	RPAC	EC-99-448
	RPA 410193	0.02–0.30	6	108–128	115	7.1	45911	M-238849-01-1
Tomato (puree)	Fenamidone	0.02–0.30	6	68–94	78	14	RPAC	EC-99-448
	RPA 410193	0.02–0.30	6	117–128	121	3.7	45911	M-238849-01-1
Tomato (fruit)	Fenamidone	0.01–0.1	3	97–104	101	2.9	00868	RA-2541/05
	RPA 410193	0.01–0.1	3	104–113	107	3.6		M-279022-01-1
Lettuce	Fenamidone	0.02–0.10	9	99–102	101	0.9	AR 150-97	98-139
	RPA 410193	0.02–0.10	9	91–99	95	4.2		M-165838-01-1
Lettuce	Fenamidone	0.02–0.30	6	87–112	98	11.4	RPAC	EC-99-448
	RPA 410193	0.02–0.30	6	96–103	99	2.6	45911	M-238849-01-1
Lettuce	Fenamidone	0.01–5.0	11	92–121	105	6.9	00868	RA-2542/04
	RPA 410193	0.01–5.0	11	74–111	100	10		M-265329-01-1
Lettuce	Fenamidone	0.02–1.0	6	102–109	105	2.8	RPAC	00W13043
	RPA 410193	0.02–1.0	6	98–108	102	3.4	45911	M-238774-01-1
Mustard greens	Fenamidone	0.02–40	11	73–117	93	14	RPAC	201226
	RPA 410193	0.02–0.50	9	99–115	107	6.2	45911	M-260567-01-1
Spinach	Fenamidone	0.02–30	11	74–119	99	16	RPAC	201214
	RPA 410193	0.02–0.30	12	69–114	95	14	45911	M-242541-01-1
Lima beans (succ.) w/o pods	Fenamidone	0.02–2.0	12	77–110	90	12.5		09530
	RPA 410193	0.02–2.0	12	84–119	100	9		IR4 submission
Snap beans, pods w/seed	Fenamidone	0.02–2.0	9	101–116	111	4.6		08895
	RPA 410193	0.02–2.0	9	83–107	94	8.4		IR4 submission
Snap beans, plants w/pods	Fenamidone	0.2–40	12	101–117	108	5.8		08895
	RPA 410193	0.2–2.0	9	80–105	91	10.8		IR4 submission
Carrot	Fenamidone	0.02–2.0	12	74–107	89	11	RPAC	08524
	RPA 410193	0.02–2.0	12	80–114	94	9.3	45911	M-278032-01-1
Potato (chips, crisps)	Fenamidone	0.02–0.3	12	84–111	95	11	RPAC	EC-99-448
	RPA 410193	0.02–0.3	12	86–120	101	9.6	45911	M-238849-01-1
Potato (flakes)	Fenamidone	0.02–0.3	6	87–95	92	3.7	RPAC	EC-99-448
	RPA 410193	0.02–0.3	6	103–119	108	6.2	45911	M-238849-01-1
Potato (tubers)	Fenamidone	0.02–0.3	6	87–109	96	10	RPAC	EC-99-448
	RPA 410193	0.02–0.3	6	102–111	107	3.0	45911	M-238849-01-1
Potato (wet peel)	Fenamidone	0.02–0.3	6	92–113	100	9.9	RPAC	EC-99-448
	RPA 410193	0.02–0.3	6	101–117	107	6.3	45911	M-238849-01-1
Potato (foliage)	Fenamidone	0.02	2	84, 86	85	–	AR 150-97	97-555
	RPA 410193	0.02	2	82, 86	84	–		M-165234-01-1

Commodity	Analytes	Fortification, mg/kg	n	Recovery, %			Method	Reference
				Range	Mean	RSD		
Potato (tubers)	Fenamidone	0.02–0.2	3	99–113	105	6.7		
	RPA 410193	0.02–0.2	3	85–100	93	8.2		
Potato (tubers)	Fenamidone	0.02	2	83, 85	84	–	AR 150-97	97-556 M-165225-01-1
	RPA 410193	0.02	2	84, 102	93	–		
Potato (tubers)	Fenamidone	0.02	4	71–96	85	12	AR 150-97	97-691 M-165758-01-1
	RPA 410193	0.02	4	76–113	96	18		
Potato (tubers)	Fenamidone	0.02–0.04	4	75–94	82	10	AR 150-97	97-745 M-165485-01-1
	RPA 410193	0.02–0.04	4	92–114	106	9.6		
Potato (peel)	Fenamidone	0.02–0.04	4	72–86	80	8.9		
	RPA 410193	0.02–0.04	4	88–108	99	9.9		
Potato (peeled tubers)	Fenamidone	0.02–0.04	5	71–84	79	6.8		
	RPA 410193	0.02–0.04	5	81–120	103	14		
Potato (foliage)	Fenamidone	0.20–4.0	5	68–102	82	21	AR 150-97	98-527 M-166545-01-1
	RPA 410193	0.20–0.4	5	82–115	104	13		
Potato (tubers)	Fenamidone	0.005–0.025	20	72–111	92	12		
	RPA 410193	0.005–0.025	20	67–108	83	15		
Potato (tubers)	Fenamidone	0.005	8	68–109	89	19	AR 150-97	98-669 M-166638-01-1
	RPA 410193	0.005	8	43–101	69	27		
Potato (peel)	Fenamidone	0.005	3	70–107	90	21		
	RPA 410193	0.005	3	56–72	66	13		
Potato (peeled tubers)	Fenamidone	0.005	3	66–79	71	9.5		
	RPA 410193	0.005	3	46–61	52	15		
Potato (tubers)	Fenamidone	0.02–0.20	8	74–103	94	10	RPAC 45911	01AC05 M-394609-01-1
	RPA 410193	0.02–0.20	8	86–108	94	7.5		
Potato (tubers)	Fenamidone	0.02–0.20	10	85–92	88	3.0	AR 150-97 (R003696)	UNESP RA- 1066/06, UNESP RA-1067/06, UNESP RA- 1068/06, UNESP RA-1069/06 M-279947-02-1, M-279942-02-1, M-279946-02-1, M-279941-02-1
	RPA 410193	0.02–0.20	10	85–93	89	2.9		
Ginseng (roots)	Fenamidone	0.02–2.0	9	97–116	104	5.9		09800 M-393676-01-1
	RPA 410193	0.02–2.0	9	89–117	101	9.2		
Witloof (leaves)	Fenamidone	0.01–0.1	7	105–109	107	1.4		
	RPA 410193	0.01–0.1	7	89–105	98	7.0		
Witloof (roots)	Fenamidone	0.01–1.0	8	100–109	104	3.5	00868	RA-2566/04 M-256652-01-1
	RPA 410193	0.01–1.0	8	87–108	98	7.8		
Witloof (leaves)	Fenamidone	0.01–1.0	10	95–107	99	4.3		
	RPA 410193	0.01–1.0	10	81–92	87	4.6		
Witloof (roots)	Fenamidone	0.01–1.0	10	96–103	100	2.4	00868	08-2205 M-363750-03-1
	RPA 410193	0.01–1.0	10	83–94	89	5.1		
Witloof (leaves)	Fenamidone	0.01–0.50	7	75–98	88	10.3		
	RPA 410193	0.01–0.50	7	74–96	85	8.6		
Witloof (roots)	Fenamidone	0.01–0.50	7	88–108	97	7.0	01163	10-2040 M-420668-01-1
	RPA 410193	0.01–0.50	7	80–106	94	9.1		
Celery (whole plant w/o roots)	Fenamidone	0.02–20	14	63–120	97	16		
	RPA 410193	0.02–0.20	15	94–119	109	6.5		
Celery (stalks trimmed)	Fenamidone	0.02–10	14	77–120	94	13	RPAC 45911	201214 M-242541-01-1
	RPA 410193	0.02–0.20	11	91–120	106	9.4		
Wheat (forage)	Fenamidone	0.02–0.3	6	75–105	87	14	RPAC 45911	EC-99-448 M-238849-01-1
	RPA 410193	0.02–0.3	6	108–117	112	3.0		
Wheat (grain)	Fenamidone	0.02–0.3	6	86–107	95	9.7	RPAC 45911	EC-99-448 M-238849-01-1
	RPA 410193	0.02–0.3	6	87–104	97	6.3		
Wheat (hay)	Fenamidone	0.02–0.3	6	68–99	83	15	RPAC 45911	EC-99-448 M-238849-01-1
	RPA 410193	0.02–0.3	6	98–110	105	4.6		
Wheat (straw)	Fenamidone	0.02–0.3	6	77–89	85	5.4	RPAC 45911	EC-99-448 M-238849-01-1
	RPA 410193	0.02–0.3	6	80–95	88	5.9		
Cotton	Fenamidone	0.02–0.3	6	72–88	81	8.0	RPAC	EC-99-448

Fenamidone

Commodity	Analytes	Fortification, mg/kg	n	Recovery, %			Method	Reference
				Range	Mean	RSD		
(gin by-products)	RPA 410193	0.02–0.3	6	121–130	125	2.5	45911	M-238849-01-1
Cotton (hulls)	Fenamidone	0.02–0.3	6	82–123	100	15	RPAC	EC-99-448
	RPA 410193	0.02–0.3	6	108–125	118	4.9	45911	M-238849-01-1
Cotton (meal)	Fenamidone	0.02–0.3	6	81–94	89	5.3	RPAC	EC-99-448
	RPA 410193	0.02–0.3	6	68–119	104	19	45911	M-238849-01-1
Cotton (oil)	Fenamidone	0.02–0.3	6	93–102	97	3.4	RPAC	EC-99-448
	RPA 410193	0.02–0.3	6	116–124	120	2.7	45911	M-238849-01-1
Cotton (seed)	Fenamidone	0.02–0.3	6	78–88	84	4.3	RPAC	EC-99-448
	RPA 410193	0.02–0.3	6	101–118	108	5.1	45911	M-238849-01-1
Sunflower (seed)	Fenamidone	0.02–0.20	9	72–82	78	4.3	RPAC	07999
	RPA 410193	0.02–0.20	9	81–94	87	5.6	45911	M-251287-02-1
Animal commodities								
Milk	Fenamidone	0.05–0.5	10	72–100	89	9.8	AR 200-99	99-68 M-166398-01-1
	RPA 412708	0.05–0.5	9	57–104	89	17		
	RPA 412636	0.05–0.5	10	64–108	95	15		
Whole egg	Fenamidone	0.05–0.5	10	67–88	80	7.3	AR 178-98	98-150 M-166460-01-1
	RPA 412708	0.05–0.5	10	76–96	90	6.9		
	RPA 412636	0.05–0.5	10	87–115	97	7.9		
Meat (beef)	Fenamidone	0.05–0.5	10	66–86	80	8.7	AR 178-98	98-150 M-166460-01-1
	RPA 412708	0.05–0.5	10	67–93	82	11		
	RPA 412636	0.05–0.5	10	84–107	94	7.7		
Kidney	Fenamidone	0.05–0.5	10	68–87	76	7.1	AR 178-98	98-150 M-166460-01-1
	RPA 412708	0.05–0.5	10	67–90	82	9.6		
	RPA 412636	0.05–0.5	10	86–106	96	6.5		
Liver	Fenamidone	0.05–0.5	10	66–79	73	5.7	AR 178-98	98-150 M-166460-01-1
	RPA 412708	0.05–0.5	10	78–110	98	12		
	RPA 412636	0.05–0.5	10	96–117	108	7.0		

Stability of residues in stored analytical samples

Information was received on the freezer storage stability of fenamidone residues and its metabolites in plant commodities. The storage stability data for fenamidone and RPA 410193 in grapes, potatoes, lettuce, strawberries and turnips are described as follows. The results are shown in Table 55.

Grapes, potatoes, lettuce

Reference: Kieken and Queyrel, 1999 (report R&D/CRLD/AN/mr/9915970, M-166527-01-1), Kieken and Queyrel, 2000 (report R&D/CRLD/AN/mba/0015230, M-183934-01-1)

Method: AR 150-97, GC-NPD, LOQ 0.02 mg/kg for both fenamidone and RPA 410193

Description: Untreated control samples were fortified with fenamidone and its metabolite RPA 410193 at a concentration of 0.2 mg/kg and then frozen below –20 °C. Samples were analysed immediately after fortification (0 day) and after storage intervals up to 1 year (12 or 13 months). At each interval, two stored samples were analysed, with one or more procedural recovery samples (control samples spiked just before analysis at 0.2 mg/kg).

Strawberries

Reference: Uceda, 2005 (report 03-21, M-247921-01-1)

Method: AR 306-03, GC-MS, LOQ 0.01 mg/kg

Description: Samples of strawberries were fortified with fenamidone and RPA 405682 at a level of 0.1 mg/kg. The samples were stored in glass bottles at about –18 °C or below and were analysed at nominal storage intervals of 0, 4, 14 and 18 months.

Turnips

Reference: Cavaillé, 2007 (report MR-07/232, M-286141-01-1)

Method: 00868, LC-MS/MS, LOQ 0.01 mg/kg

Description: Turnip root samples were fortified with fenamidone or RPA 404862 at a level of 0.2 mg/kg. The samples were stored in plastic containers at about –18 °C or below and were analysed at nominal storage intervals of 0, 2, 6 and 12 months. Procedural recovery experiments were conducted at all storage intervals by spiking control samples of turnip with a mixture of fenamidone and RPA 410193 at a level of 0.2 mg/kg. Additional recoveries for both analytes at 0.01 mg/kg were also performed at the 0 and 12 month intervals.

Table 55 Storage stability data for fenamidone and RPA 410193 in berries and rooting vegetables

	Fenamidone					RPA 410193				
Time, month	Procedural recovery, %		Residue, mg/kg		% Remaining	Procedural recovery, %		Residue, mg/kg		% Remaining
	Individual	Mean	Individual	Mean		Individual	Mean	Individual	Mean	
Grapes (Report R&D/CRLD/AN/mr/9915970, Bayer reference M-166527-01-1)										
0	95, 82	89	0.18, 0.20	0.19	100	93, 96	95	0.22, 0.20	0.21	100
1	107	107	0.22, 0.22	0.22	115	97	97	0.19, 0.19	0.19	89
4	102	102	0.18, 0.20	0.19	98	115	115	0.21, 0.21	0.21	98
8	108	108	0.18, 0.18	0.18	92	84	84	0.17, 0.21	0.19	88
12	99, 104	102	0.22, 0.22	0.22	114	97, 106	102	0.22, 0.19	0.20	96
Potatoes (Report R&D/CRLD/AN/mr/9915970, Bayer reference M-166527-01-1)										
0	89, 75	82	0.16, 0.14	0.15	100	75, 70	73	0.18, 0.17	0.18	100
1	79	79	0.19, 0.18	0.18	121	89	89	0.19, 0.2	0.19	109
3	84	84	0.15, 0.15	0.15	103	72	72	0.15, 0.15	0.15	84
8	93, 80	87	0.17, 0.16	0.17	111	92, 77	85	0.17, 0.12	0.14	81
12	70, 97	84	0.17, 0.18	0.18	120	68, 94	81	0.16, 0.18	0.17	98
Lettuce (Reports R&D/CRLD/AN/mr/9915970, Bayer reference M-166527-01-1 and R&D/CRLD/AN/mba/0015230, Bayer reference M-183934-01-1)										
0	69, 93, 89, 103	89	0.18, 0.19	0.18	100	96, 91	94	0.22, 0.21	0.22	100
2	86, 75	81	0.20, 0.20	0.20	111	95	95	0.15, 0.15	0.15	69
5	86, 95	91	0.21, 0.22	0.21	117	103, 94	99	0.18, 0.21	0.19	88
9	82, 95	89	0.20, 0.19	0.19	106	83, 89	86	0.21, 0.19	0.20	94
13	99, 88	94	0.16, 0.16	0.16	89	101, 82	92	0.21, 0.21	0.21	96
Strawberries (Report 03-21, Bayer reference M-247921-01-1).										
0	85, 89	87	0.09, 0.09	0.09	100	97, 96	97	0.1, 0.1	0.1	100
4	70, 105	88	0.08, 0.06, 0.11	0.083	93	97	97	0.07, 0.08	0.075	75
14	99	99	0.09, 0.09	0.09	100	82	82	0.10, 0.07	0.085	85
18	105, 106	106	0.07, 0.08	0.08	89	90, 84	87	0.07, 0.09	0.08	80
Turnips (Cavaillé, 2007, Report MR-07/232, Bayer reference M-286141-01-1)										
0	100, 99, 90	96	0.19, 0.21	0.2	100	88, 87, 86	87	0.16, 0.16, 0.15	0.16	100
2	96, 98	97	0.19, 0.19	0.19	95	96, 98	97	0.16, 0.17	0.16	104
6	100, 97	99	0.2, 0.19	0.2	99	99, 96	98	0.19, 0.18	0.18	117
12	99, 98, 100	99	0.19, 0.19	0.19	96	97, 94, 97	96	0.18, 0.18, 0.17	0.18	111

The stability of fenamidone and its metabolites RPA 410193, RPA 412636 and RPA 412708 were examined in nineteen crop commodities. Raw and processed commodities were included: bulb onion, cotton gin trash, cotton hulls, cotton meal, cotton oil, cotton seed, cucumber, leaf lettuce, potato chips (crisps), potato flakes, potato tubers, potato wet peel, tomato fruit, tomato paste, tomato puree, wheat forage, wheat grain, wheat hay, and wheat straw. The study is described as follows below. The results are shown in Table 56 for fenamidone and RPA 412636, and in Table 57 for RPA 412708 and RPA 410193.

Various commodities

Reference: Hudson and Carringer, 2001 (report EC-99-448, M-238849-01-1)

Method: RPAC 45911, LC-MS/MS, LOQ 0.02 mg/kg for each analyte

Description: Untreated control samples were fortified with fenamidone, RPA 412636, RPA 412708 and RPA 410193 at a concentration of 0.3 mg/kg and then frozen below -20°C . Each analyte was fortified into separate control samples. All matrices were analysed for 12 months stability with the exception of cotton. Cotton matrices were analysed from 1 to 6 months. At each interval, two stored samples were analysed, with a procedural recovery sample (control sample spiked just before analysis at 0.2 mg/kg).

Table 56 Storage stability data for fenamidone and RPA 412636 in plant commodities

Interval, month	Fenamidone				RPA 412636			
	Procedural recovery, %	Residues, mg/kg		% Remaining	Procedural recovery, %	Residues, mg/kg		% Remaining
		Individual	Mean			Individual	Mean	
Onion, bulb								
0	88	0.268, 0.276	0.27	100	94	0.274, 0.286	0.28	100
1	91	0.263, 0.278	0.27	99	77	0.212, 0.209	0.21	75
3	101	0.304, 0.290	0.3	109	115	0.330, 0.345	0.34	121
6	100	0.272, 0.281	0.28	102	99	0.285, 0.274	0.28	100
9	66	0.190, 0.207	0.2	73	106	0.304, 0.303	0.30	108
12	82	0.236, 0.247	0.24	89	108	0.316, 0.305	0.31	111
Cotton seed								
0	83	0.244, 0.235	0.24	100	107	0.311, 0.327	0.32	100
1	86	0.234, 0.256	0.25	102	91	0.254, 0.235	0.25	77
6	41	0.145, 0.121	0.13	56	128	0.321, 0.363 ^a	0.34 ^a	107 ^a
Cotton gin trash								
0	75	0.225, 0.210	0.22	100	121	0.327, 0.313	0.32	100
6	111	0.306, 0.300	0.30	139	118	0.336, 0.310	0.32	101
Cotton hulls								
0	79	0.244, 0.242	0.24	100	95	0.287, 0.280	0.28	100
1	76	0.209, 0.209	0.21	86	104	0.259, 0.216	0.24	84
3	100	0.264, 0.232	0.25	102	91	0.296, 0.312	0.30	107
6	96	0.250, 0.248	0.25	102	115	0.307, 0.303	0.31	108
Cotton meal								
0	98	0.289, 0.275	0.28	100	104	0.309, 0.313	0.31	100
1	85	0.240, 0.254	0.25	88	81	0.231, 0.248	0.24	77
3	89	0.228, 0.241	0.24	83	101	0.300, 0.279	0.29	93
6	62	0.217, 0.212	0.22	76	90	0.230, 0.262	0.25	79
Cotton oil								
0	99	0.295, 0.299	0.3	100	118	0.338, 0.331	0.33	100
1	91	0.244, 0.276	0.26	88	109	0.297, 0.298	0.3	89
Cucumber								
0	113	0.329, 0.330	0.33	100	120	0.358, 0.355	0.36	100
1	102	0.181, 0.211	0.2	59	101	0.299, 0.308	0.30	85
3	111	0.289, 0.282	0.29	87	106	0.328, 0.335	0.33	93
6	110	0.246, 0.249	0.25	75	83	0.243, 0.236	0.24	67
14	98	0.260, 0.240	0.25	76	102	0.351, 0.319	0.33	94
Lettuce								
0	79	0.234, 0.247	0.24	100	84	0.255, 0.247	0.25	100
1	82	0.242, 0.220	0.23	96	96	0.311, 0.341	0.33	130
3	79	0.214, 0.222	0.22	91	103	0.303, 0.287	0.3	118
6	98	0.277, 0.305	0.29	121	98	0.283, 0.288	0.29	114
9	99	0.264, 0.201	0.23	97	137	0.369, 0.363	0.37	146
12	82	0.217, 0.210	0.21	89	102	0.307, 0.295	0.30	120
Potato tubers								
0	87	0.240, n/a	0.24	100	83	0.249, 0.264	0.26	100
1	75	0.198, 0.204	0.20	84	99	0.300, 0.322	0.31	121
3	106	0.258, 0.269	0.26	110	97	0.271, 0.264	0.27	104
6	112	0.331, 0.315	0.32	135	106	0.303, 0.292	0.3	116
9	83	0.205, 0.207	0.21	86	113	0.321, 0.323	0.32	126
12	92	0.249, 0.244	0.25	103	110	0.319, 0.319	0.32	124
Potato wet peel								
0	88	0.260, 0.261	0.26	100	87	0.244, 0.247	0.25	100
1	73	0.189, 0.194	0.19	74	90	0.350, 0.275	0.31	127

Interval, month	Fenamidone				RPA 412636			
	Procedural recovery, %	Residues, mg/kg		% Remaining	Procedural recovery, %	Residues, mg/kg		% Remaining
		Individual	Mean			Individual	Mean	
3	74	0.215, 0.207	0.21	81	105	0.304, 0.295	0.30	122
6	96	0.268, 0.277	0.27	105	91	0.278, 0.296	0.29	117
9	113	0.272, 0.281	0.28	106	127	0.363, 0.351	0.36	145
12	81	0.226, 0.209	0.22	83	105	0.302, 0.291	0.3	121
Potato chips (crisps)								
0	70	0.198, 0.185	0.19	100	75	0.239, 0.269	0.25	100
1	81	0.206, 0.213	0.21	109	93	0.256, 0.263	0.26	102
3	101	0.256, 0.242	0.25	130	96	0.257, 0.259	0.26	102
6	83	0.189, 0.197	0.19	101	81	0.215, 0.215	0.22	85
12	93	0.222, 0.226	0.22	117	101	0.242, 0.220	0.23	91
Potato flakes								
0	105	0.323, 0.330	0.33	100	93	0.302, 0.277	0.29	100
1	88	0.308, 0.313	0.31	95	75	0.271, 0.227	0.25	86
3	90	0.259, n/a	0.26	79	101	0.298, 0.301	0.30	103
6	92	0.270, 0.257	0.26	81	86	0.264, 0.257	0.26	90
9	78	0.294, 0.334	0.31	96	158	0.478, 0.500	0.49	169
12	77	0.248, 0.240	0.24	75	95	0.298, 0.274	0.29	99
Tomato fruit								
0	81	0.265, 0.247	0.26	100	95	0.278, 0.303	0.29	100
1	104	0.323, 0.280	0.30	118	70	0.204, 0.211	0.21	71
3	70	0.214, 0.231	0.22	87	97	0.281, 0.286	0.28	98
6	94	0.295, 0.290	0.29	114	73	0.243, 0.245	0.24	84
9	86	0.249, 0.255	0.25	98	122	0.379, 0.377	0.38	130
12	79	0.266, 0.257	0.26	102	109	0.325, 0.322	0.32	111
Tomato paste								
0	75	0.320, 0.283	0.30	100	94	0.274, 0.285	0.28	100
1	105	0.302, 0.301	0.30	100	98	0.221, 0.243	0.23	83
3	92	0.202, 0.238	0.22	73	91	0.247, 0.238	0.24	87
6	73	0.162, 0.189	0.18	58	83	0.172, 0.241	0.21	74
12	91	0.246, 0.226	0.24	78	80	0.254, 0.254	0.25	91
Tomato puree								
0	78	0.239, 0.239	0.24	100	126	0.369, 0.376	0.37	100
1	107	0.309, 0.327	0.32	133	101	0.271, 0.294	0.28	76
3	105	0.318, 0.329	0.32	135	93	0.276, 0.274	0.27	74
12	97	0.287, 0.260	0.27	114	98	0.259, 0.273	0.27	74
Wheat forage								
0	78	0.256, 0.234	0.25	100	119	0.357, 0.356	0.36	100
1	101	0.282, 0.291	0.29	117	101	0.289, 0.300	0.3	83
3	105	0.315, 0.316	0.32	129	119	0.336, 0.338	0.34	95
6	79	0.224, 0.219	0.22	90	107	0.316, 0.302	0.31	87
12	81	0.240, 0.202	0.22	90	80	0.234, 0.258	0.25	69
Wheat grain								
0	76	0.247, 0.222	0.24	100	95	0.282, 0.301	0.29	100
1	105	0.299, 0.294	0.3	126	115	0.332, 0.312	0.32	110
3	98	0.281, 0.292	0.29	122	110	0.330, 0.300	0.315	108
6	92	0.225, 0.224	0.23	96	103	0.300, 0.291	0.3	101
12	91	0.279, 0.268	0.27	117	99	0.273, 0.316	0.3	101
Wheat hay								
0	87	0.243, 0.265	0.25	100	118	0.359, 0.356	0.36	100
1	83	0.255, 0.260	0.26	101	97	0.295, 0.288	0.29	82
3	108	0.298, 0.295	0.3	117	Residues	in control	-	-
6	81	0.237, 0.215	0.23	89	92	0.293, 0.285	0.29	80
12	87	0.220, 0.211	0.22	85	97	0.204, 0.269	0.24	66
Wheat straw								
0	91	0.247, 0.266	0.26	100	92	0.255, 0.256	0.26	100
1	93	0.237, 0.272	0.26	99	103	0.270, 0.264	0.27	105
3	103	0.308, 0.333	0.32	125	103	0.266, 0.266	0.27	105
6	94	0.228, 0.245	0.24	92	101	0.259, 0.278	0.27	105
12	89	0.257, 0.252	0.25	99	73	0.217, 0.230	0.22	87

^a Obtained by calculation using the recovery values

Table 57 Storage stability data for RPA 412708 and RPA 410193 in plant commodities

Interval, month	RPA 412708				RPA 410193			
	Procedural recovery, %	Residues, mg/kg		% Remaining	Procedural recovery, %	Residues, mg/kg		% Remaining
		Individual	Mean			Individual	Mean	
Onion, bulb								
0	113	0.339, 0.335	0.34	100	104	0.313, 0.315	0.31	100
1	113	0.331, 0.326	0.33	97	100	0.276, 0.276	0.28	88
3	96	0.288, 0.296	0.29	87	102	0.295, 0.294	0.295	94
6	85	0.281, 0.267	0.27	81	88	0.244, 0.235	0.24	76
9	116	0.302, 0.278	0.29	86	89	0.263, 0.264	0.26	84
12	100	0.262, 0.267	0.265	78	88	0.257, 0.260	0.26	82
Cotton seed								
0	80	0.232, 0.246	0.24	100	118	0.331, 0.333	0.33	100
1	92	0.274, 0.269	0.27	114	119	0.353, 0.348	0.35	106
6	100	0.282, 0.294	0.29	121	81	0.233, 0.234	0.23	70
Cotton gin trash								
0	83	0.254, 0.245	0.25	100	120	0.357, 0.359	0.36	100
6	95	0.282, 0.273	0.28	111	73	0.223, 0.219	0.22	62
Cotton hulls								
0	96	0.284, 0.261	0.26	100	91	0.269, 0.291	0.28	100
1	100	0.246, 0.270	0.26	99	106	0.298, 0.299	0.3	107
3	106	0.311, 0.306	0.31	118	110	0.313, 0.306	0.31	111
6	92	0.259, 0.250	0.26	98	98	0.289, 0.276	0.28	101
Cotton meal								
0	100	0.260, 0.253	0.26	100	115	0.338, 0.355	0.35	100
1	97	0.292, 0.277	0.29	111	98	0.279, 0.282	0.28	81
3	100	0.270, 0.272	0.27	106	91	0.312, 0.310	0.31	90
6	89	0.257, 0.228	0.24	95	88	0.277, 0.276	0.28	80
Cotton oil								
0	115	0.330, 0.319	0.325	100	121	0.369, 0.351	0.36	100
1	110	0.305, 0.301	0.30	93	110	0.325, 0.307	0.32	88
Cucumber								
0	114	0.330, 0.319	0.325	100	105	0.318, 0.324	0.32	100
1	108	0.308, 0.277	0.29	90	120	0.275, 0.287	0.28	88
3	122	0.349, 0.356	0.35	109	116	0.326, 0.308	0.32	99
6	81	0.211, 0.204	0.21	64	106	0.241, 0.258	0.25	78
14	122	0.353, 0.340	0.35	107	107	0.255, 0.271	0.26	82
Lettuce								
0	102	0.304, 0.312	0.31	100	110	0.311, 0.296	0.30	93
1	88	0.256, 0.251	0.25	82	105	0.295, 0.289	0.29	90
3	98	0.286, 0.273	0.28	91	100	0.331, 0.317	0.32	100
6	101	0.279, 0.278	0.28	90	107	0.334, 0.330	0.33	102
9	104	0.306, 0.280	0.29	95	117	0.279, 0.257	0.27	82
12	99	0.291, 0.280	0.29	93	89	0.311, 0.296	0.30	93
Potato tubers								
0	99	0.298, 0.306	0.30	100	102	0.310, 0.307	0.31	100
1	78	0.230, 0.228	0.23	77	86	0.221, 0.227	0.22	73
3	101	0.269, 0.265	0.27	90	100	0.294, 0.283	0.29	94
6	107	0.305, 0.312	0.31	103	84	0.246, 0.236	0.24	78
9	118	0.342, 0.346	0.34	113	98	0.240, 0.288	0.26	86
12	119	0.358, 0.305	0.33	110	101	0.307, 0.306	0.31	99
Potato wet peel								
0	104	0.310, 0.310	0.31	100	104	0.309, 0.308	0.31	100
1	78	0.207, 0.213	0.21	68	82	0.235, 0.228	0.23	75
3	100	0.297, 0.297	0.3	96	102	0.305, 0.305	0.305	99
6	94	0.267, 0.264	0.27	86	110	0.315, 0.311	0.31	101
9	112	0.325, 0.329	0.33	105	121	0.330, 0.325	0.33	106
12	115	0.316, 0.309	0.31	101	97	0.283, 0.286	0.285	92
Potato chips (crisps)								
0	77	0.255, 0.257	0.26	100	96	0.293, 0.288	0.29	100
1	97	0.269, 0.257	0.26	100	97	0.270, 0.275	0.27	94
3	106	0.273, 0.271	0.27	106	106	0.265, 0.277	0.27	94
6	90	0.238	0.24	93	85	0.211, 0.197	0.20	70

Interval, month	RPA 412708				RPA 410193			
	Procedural recovery, %	Residues, mg/kg		% Remaining	Procedural recovery, %	Residues, mg/kg		% Remaining
		Individual	Mean			Individual	Mean	
12	91	0.266, 0.234	0.25	98	90	0.264, 0.238	0.25	86
Potato flakes								
0	111	0.326, 0.316	0.32	100	107	0.313, 0.318	0.32	100
1	100	0.285, 0.289	0.29	89	85	0.240, 0.251	0.25	78
3	88	0.274, 0.278	0.28	86	91	0.266, 0.266	0.27	84
6	97	0.290, 0.299	0.295	92	95	0.210, 0.271	0.24	76
9	186	0.559, 0.454	0.51	158	117	0.315, 0.323	0.32	101
12	105	0.335, 0.334	0.335	104	87	0.281, 0.270	0.28	87
Tomato fruit								
0	106	0.307, 0.304	0.31	100	110	0.339, 0.345	0.34	100
1	129	0.365, 0.381	0.37	122	101	0.314, 0.657 ^a	0.31	92
3	99	0.276, 0.279	0.28	91	102	0.302, 0.303	0.30	88
6	66	0.184, 0.207	0.2	64	95	0.298, 0.297	0.3	87
9	98	0.290, 0.300	0.295	97	108	0.340, 0.356	0.35	102
12	96	0.283, 0.279	0.28	92	88	0.261, 0.290	0.28	81
Tomato paste								
0	86	0.275, 0.276	0.28	100	106	0.336, 0.309	0.32	100
1	91	0.244, 0.216	0.23	83	108	0.261, 0.252	0.26	80
3	86	0.257, 0.243	0.25	91	93	0.260, 0.253	0.26	80
6	76	0.211, 0.165	0.19	68	64	0.178, 0.155	0.17	52
12	88	0.214, 0.238	0.23	82	91	0.276, 0.279	0.28	86
Tomato puree								
0	96	0.288, 0.293	0.29	100	124	0.373, 0.385	0.38	100
1	102	0.291, 0.233	0.26	90	108	0.317, 0.318	0.32	84
3	97	0.278, 0.289	0.28	98	106	0.316, 0.317	0.32	84
12	89	0.255, 0.217	0.24	81	99	0.255, 0.244	0.25	66
Wheat forage								
0	107	0.319, 0.311	0.315	100	129	0.393, 0.410	0.40	100
1	109	0.292, 0.288	0.29	92	63	0.176, 0.178	0.18	44
3	106	0.298, 0.297	0.3	94	121	0.351, 0.356	0.35	88
6	103	0.256, 0.257	0.26	81	77	0.306, 0.297	0.30	75
12	95	0.235, 0.236	0.24	75	91	0.260, 0.269	0.265	66
Wheat grain								
0	92	0.279, 0.265	0.27	100	95	0.258, 0.299	0.28	100
1	108	0.298, 0.279	0.29	106	117	0.303, 0.331	0.32	114
3	102	0.276, 0.284	0.28	103	106	0.285, 0.287	0.29	103
6	115	0.273, 0.289	0.28	103	95	0.246, 0.220	0.23	84
12	94	0.259, 0.271	0.265	97	95	0.270, 0.284	0.28	100
Wheat hay								
0	104	0.376, 0.392	0.38	100	122	0.376, 0.392	0.38	100
1	98	0.265, 0.271	0.27	70	125	0.343, 0.337	0.34	89
3	91	0.283, 0.285	0.28	74	105	0.321, 0.302	0.31	81
6	90	0.259, 0.247	0.25	66	88	0.276, 0.259	0.27	70
12	90	0.231, 0.219	0.225	59	96	0.243, 0.245	0.24	64
Wheat straw								
0	95	0.268, 0.287	0.28	100	106	0.314, 0.301	0.31	100
1	113	0.288, 0.306	0.3	107	136	0.360, 0.382	0.37	121
3	102	0.289, 0.305	0.3	107	112	0.315, 0.305	0.31	101
6	94	0.272, 0.229	0.25	90	92	0.268, 0.256	0.26	85
12	84	0.219, 0.208	0.21	77	89	0.216, 0.240	0.23	74

^a Unexplained high value not included in calculation of mean (possible fortification error)

The stability of incurred fenamidone residues was examined in tomatoes over a 21-month period after harvest under freezer conditions. Residues of RPA 410193 were below 0.01 mg/kg in all the samples, so it was not possible to assess its stability. The study is described below (see Table 58).

Reference: Schulte, 2011 (report MR-11/028, M-404654-02-1)

Method: 00868, LC-MS/MS, LOQ 0.01 mg/kg for each analyte

Description: Incurred fenamidone residues in tomato fruits ranged from 0.05 to 0.38 mg/kg at the first analysis made 17 months after sampling. The samples were analysed again at nominal storage intervals of 14–21 months after sampling (2–4 months after initial analysis). Procedural recovery experiments were conducted at all storage intervals by spiking control samples of tomato at levels of 0.01 to 1.0 mg/kg. Residues in the control samples were all < 0.01 mg/kg.

Table 58 Storage stability data of incurred fenamidone residues in tomato

Interval month	Procedural recovery, %		Fenamidone residues, mg/kg (Percentage remaining)					Mean %
	Individual	Mean						
Trial 09-2235-02								
Initial residues	74, 81, 88, 89, 87, 89, 84, 85, 105, 112	89.4	0.13	0.38	0.16	0.15	0.08	100
2	82, 98, 93, 95	92	0.12 (92)	0.42 (111)	0.17 (106)	0.16 (107)	0.08 (100)	103
4	96, 97	97	0.13 (100)	0.41 (108)	0.13 (81)	0.12 (80)	0.08 (100)	94
Trial 09-2235-03								
Initial residues	74, 81, 88, 89, 87, 89, 84, 85, 105, 112	89.4	0.05	0.07	0.08	0.10	0.05	100
2	82, 98, 93, 95	92	0.05 (100)	0.07 (100)	0.08 (100)	0.09 (90)	0.04 (80)	94
4	96, 97	97	0.06 (120)	0.08 (114)	0.09 (113)	0.11 (110)	0.05 (100)	111

In summary, residues of fenamidone, RPA412636, RPA412708 and RPA410193 stored under freezer conditions at –20 °C are stable for at least 12 months in a wide range of raw and processed crop matrices, including examples of high-water and high-starch crops and for at least 6 months in cotton products (high oil content). Residues of fenamidone and RPA410193 are stable in high-acid commodities (strawberry) for at least 18 months.

USE PATTERN

The fungicide fenamidone is registered in many countries for control of diseases on a large variety of crops. Fenamidone is applied either as solo product or in combinations with other active substances as propamocarb, mancozeb, iprovalicarb and fosetyl-Al. The information available to the Meeting on registered uses on berries, vegetables and on oil seed is summarized in Table 59 and refers for application rates etc. to fenamidone only. The manufacturer submitted labels for all fenamidone uses.

Table 59 Registered uses of fenamidone

Crop (remarks)	Country	Form.	Application				PHI, Days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hL	No	
Berries and other small fruit							
Grapes	Czech Republic	WG 71.1	Foliar	0.13		3	28
Grapes	Brazil	SC 500	Foliar	0.15		3	7
Grapes	Greece	WG 71.1	Foliar	0.11		2	28
Grapes	Italy	WG 71.1	Foliar	0.13		3	40
Grapes	Romania	WG 71.1	Foliar	0.11		2	28
Grapes	USA	SC 500	Foliar	0.10		3	30
Strawberries (outdoor, just before planting)	Netherlands	WG 66	Dip of plants		0.009	1	35
Strawberries (protected)	Netherlands	WG 66	Drench		0.0045	1	35
Strawberries (protected)	Belgium	WG 66	Drench	0.45		2	35
Strawberries (pre-transplantation dip or post-transplantation drench or spray)	UK	WG 66	Dip or drench + foliar	0.18 + 0.27		1 + 1	35
Bulb vegetables							
Onion, bulb; garlic, shallot, leek	Switzerland	SC 450	Foliar	0.15		3	14
Onion, bulb	Brazil	SC 500	Foliar	0.15		3	7
Onion, bulb	Poland	WG 71.1	Foliar	0.11		2–3	14
Bulb vegetables	Canada	SC 500	Foliar	0.20		4	7
Bulb vegetables	USA	SC 500	Foliar	0.20		4	7

Crop (remarks)	Country	Form.	Application				PHI, Days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hL	No	
Brassica vegetables							
Brassica vegetables	USA	SC 500	Foliar	0.30		3	2
Brassica vegetables	Canada	SC 500	Foliar	0.30		3	2
Cabbages, head; flowerhead brassica	Switzerland	SC 450	Foliar	0.15		3	14
Fruiting vegetables, cucurbits							
Cucumbers, melons, water melons (field and protected)	Greece	WG 71.1	Foliar	0.11		3	3
Cucumbers (field)	Poland	WG 71.1	Foliar	0.11		1–2	3
Cucumbers (protected)	Romania	WG 71.1	Foliar	0.11		3	3
Cucumber, pumpkin, melon, courgette, patisson, rondini	Switzerland	SC 450	Foliar	0.15		3	3
Cucurbits	Canada	SC 500	Foliar	0.20		4	14
Cucurbits	USA	SC 500	Foliar	0.20		4	14
Melons	Brazil	SC 500	Foliar	0.15		3	7
Melons (field and protected)	Italy	WG 71.1	Foliar	0.13		3	15
Watermelons	Brazil	SC 500	Foliar	0.15		3	7
Fruiting vegetables, other than cucurbits							
Aubergine, tomato	Switzerland	SC 450	Foliar	0.15		3	3
Tomato	Brazil	SC 500	Foliar	0.15		3	7
Tomato	Canada	SC 500	Foliar	0.10		6	14
Tomato (field)	Poland	WG 71.1	Foliar	0.11		1–2	7
Tomato (protected)	Poland	WG 71.1	Foliar		0.013	3–5	7
Tomato (field and protected)	Romania	WG 44.4	Foliar	0.11	0.03	1–3	3
Fruiting vegetables, except cucurbits	USA	SC 500	Foliar	0.30		3	14
Leafy vegetables (including brassica leafy vegetables)							
Endive (field)	Netherlands	WG 66	Foliar	0.15		3	14
Endive (protected)	Netherlands	WG 66	Foliar	0.15		3	21/42 ^a
Endive, radicchio rosso, (Treatment from 7 days after planting to BBCH 42)	Belgium	WG 66	Foliar	0.15		2	
Lambs lettuce (Treatment from 7 days after planting to BBCH 42)	Belgium	WG 66	Foliar	0.15		2	
Lettuce	Brazil	SC 500	Foliar	0.15		3	7
Lettuce	Greece	WG 71.1	Foliar	0.11		3	14
Lettuce	Italy	WG 71.1	Foliar	0.11		3	15
Lettuce (protected)	Netherlands	WG 66	Foliar	0.15		3	21/42 ^a
Lettuce, rucola (field)	Switzerland	SC 450	Foliar	0.15		3	14
Leafy vegetables	USA	SC 500	Foliar	0.30		3	2
Turnip greens	Canada	SC 500	Foliar	0.30		2	2
Legume vegetables							
Succulent beans, except cowpea	Canada	SC 500	Foliar	0.30		3	3
Succulent beans, except cowpea	USA	SC 500	Foliar	0.30		3	3
Root and tuber vegetables							
Carrot	Canada	SC 500	Foliar	0.30		2	14
Carrot	USA	SC 500	Foliar	0.30		3	14
Potato	UK	SC 450	Foliar	0.15		6	7
Potato	Canada	SC 500	Foliar	0.10		6	14
Potato	USA	SC 500	Foliar	0.30		3	14
Potato	Brazil	SC 500	Foliar	0.15		3	7
Potato	Switzerland	SC 450	Foliar	0.15		3	14
Root and tuber vegetables ^b , except carrot, potato, sugar beet	USA	SC 500	Foliar	0.30		3	14
Ginseng	USA	SC 500	Foliar	0.30		3	14
Stalk and stem vegetables							
Witloof chicory (hydroponic)	Belgium	WG 66	Dip + Irrigation		0.006 + 0.0006	2	18
Witloof chicory (hydroponic)	Netherlands	WG 66	Irrigation		0.0006	1	
Celery	USA	SC 500	Foliar	0.30		3	2

Fenamidone

Crop (remarks)	Country	Form.	Application				PHI, Days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hL	No	
Oilseed							
Cotton	USA	SC 500	In-furrow	0.30		1	
Sunflower	USA	SC 500	Seed treatment	0.19 (kg/100 kg seed)		1	

^a PHI: 21 days between 21 March and 21 September, 42 days between 22 September and 20 March

^b Root and tuber vegetables: beet (garden), celeriac, chervil (turnip-rooted), chicory, horseradish, oriental radish, parsley (turnip rooted), parsnip, salsify, turnip

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on supervised field trials for fenamidone uses that produced residues on the following commodities:

Commodity	Group	Table No
Grapes	FB Berries and other small fruits	60
Strawberries	VA Bulb vegetables	61
Leek		62
Onion, bulb		63
Spring onion		64
Cabbage	VB Brassica vegetables	65
Flowerhead brassica		66
Cucumber	VC Fruiting vegetables, Cucurbits	67
Summer squash		68
Melons		69
Watermelon		70
Peppers	VO Fruiting vegetables, other than Cucurbits	71
Tomato		72
Lettuce	VL Leafy vegetables	73
Mustard greens		74
Spinach		75
Lima beans	VP Legume vegetables	76
Snap beans		77
Carrots	VR Root and tuber vegetables	78
Potato		79
Ginseng		80
Witloof chicory (sprouts)	VS Stalk and stem vegetables	81
Celery		82
Cotton seed	SO Oilseed	83
Sunflower seed		84
Snap bean forage	AL Legume animal feeds	85
Cotton fodder, dry	AM Miscellaneous fodder and forage crops	86

In the residue supervised trials tables, where two samples were taken from a single plot, the average value is reported (individual sample results in parentheses). Where results from separate plots with distinguishing characteristics such as different formulations, varieties or treatment schedules were reported, results are listed for each plot. In these cases, the higher residue has been used for calculation purposes. Dates of duration of residue sample storage before analysis were provided. If the duration of samples storage was longer than the covered frozen stability, it is indicated in the text.

Residue values from the trials conducted according to the maximum GAP have been used for the estimation of maximum residue levels. Those results included in the calculations by the OECD MRL-calculator are underlined.

Residues are reported separately for fenamidone ("Fen") and diketo-fenamidone (RPA 410193). The code name RPA 410193 is used throughout this section, irrespective of what nomenclature (synonyms: AE C650488, RPA 405862, DK-Fen) was used in the residue studies.

For HR and STMR estimation, sums of fenamidone (MW 311.4) and RPA 410193 (MW 281.3), expressed as fenamidone (conversion factor 1.11), are needed. When residues are undetectable in a commodity, the sum of the LOQs of both components is not appropriate for all plant commodities because the days after the last treatment (DALT) differ from 2 days (e. g. lettuce) to 35 days (strawberry). To select the best option, the relative ratio of parent and metabolite was estimated:

Plant commodity	DAT, Days	Samples with measured residues for both analytes (n)	Residue parent ÷ RPA 410193	
			Range	Average
Strawberries	35	3	1–2	1.7
Grapes	28	33	2–8	4.4
Lettuce	21	5	5–16	9
Lettuce	14	3	19–42	27
Peppers	14	1	36	36
Cauliflower	14	1	8	8
Spring onion	7	1	19	19
Potato foliage	7	3	27.5–37	33
Melons, peel	3	5	2.5–23	11
Tomatoes	3	2	18	18
Lettuce	2	9	148–530	306
Mustard greens	2	8	135–710	403
Spinach	2	5	361–1030	659
Celery	2	4	111–220	145

The residues of RPA 410193 are found in the same order of magnitude as the parent in berries harvested 4 to 5 weeks after treatment. In plant commodities harvested at shorter periods (2–21 days), the level of the metabolite is much lower than the parent in most cases. The method for calculation of the total residues of the sum of fenamidone and RPA 410193 is illustrated as follows:

- Plant commodities except for grapes and strawberries

Fenamidone, mg/kg	RPA 410193, mg/kg	Total, mg/kg
< 0.02	< 0.02	< 0.02
0.05	< 0.02	0.05
0.42	0.08	0.51 ^a
^a 0.42 + (0.08 × 1.11) = 0.5088		

- Grapes and strawberries

Fenamidone, mg/kg	RPA 410193, mg/kg	Total, mg/kg
< 0.02	< 0.02	< 0.04
0.05	< 0.02	0.07
0.42	0.08	0.51

Most of the submitted residue supervised trials have been overdosed in terms of the number of applications. The Meeting received additional information and comparative trials on grapes (40 trials) and tomatoes (16 trials). Grapes were treated either 3 × 0.13 kg ai/ha or 7 × 0.12 kg ai/ha and samples were taken at a 28-day PHI. Tomatoes were treated either 3 × 0.15 kg ai/ha or 5 × 0.15 kg ai/ha and harvested at a PHI of 3 days. The results indicate no significant difference between the data sets (Simonin, 2013; M-451095-01-1).

Berries and other small fruits

Grapes

Thirty-five trials on grapes were conducted in Europe (France, Italy, Spain, Portugal, Greece, Germany and Belgium) between 1997 and 2010 by foliar spray. Trials conducted during 1997 and 1998 received seven applications (interval 14 days), whereas trials conducted during 2009 and 2010 received three applications.

Three supervised residue trials were conducted in Brazil in the growing season 1998/99. The maximum period of sample storage at –20 °C was for the Brazilian trials 795 days prior to analysis

Fenamidone

(storage stability covered for one year only). Storage stability data on strawberries show that the residues are stable for at least 18 months in an acid content commodity. The results are summarized in Table 60.

Table 60 Residues in grapes from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT Days ^a	Commodity	Residues, mg/kg			Reference
	Form	Kg ai/ha	Kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
GAP, Czech Rep.	WG	0.13			3	28					
97579RS1 Verzenay (51), France, 1997 (Pinot Noir)	WG	0.12	0.04–0.079	156–312	7	0 21 27 41	Grapes	0.66 0.27 0.19 0.08	0.088 0.083 0.067 0.04	0.76 0.36 0.26 0.12	97-579 M-165834-01-1
97579BX1 Prignac et Marcamps (33), France, 1997 (Merlot)	WG	0.12	0.067	185	7	0 21 28 41	Grapes	0.39 0.22 0.13 0.29	0.046 0.054 0.029 0.048	0.44 0.28 0.16 0.34	
97579BX2 Virrac (33), France, 1997 (Merlot)	WG	0.12	0.066	189	7	0 21 28 42	Grapes	0.32 0.18 0.22 0.15	0.038 0.047 0.056 0.05	0.36 0.23 0.28 0.21	
97580RS1, Serzy et Prin (51), France, 1997 (Chardonnay)	WG	0.12	0.036–0.037	327–340	7	31	Grapes	0.08	0.02	0.10	
97581BX1 Vaudelnay (49), France, 1997 (Cabernet)	WG	0.12–0.13	0.041	285–309	7	28 39	Grapes	0.15 0.24	0.038 0.031	0.19 0.27	97-581 M-165494-01-1
97582PTS1, Beaumont en Véron (37), France, 1997 (Cabernet)	WG	0.1–0.17	0.019–0.031	385–861	7	0 21 28 42	Grapes	0.72 0.31 0.23 0.14	0.075 0.059 0.052 0.045	0.80 0.38 0.29 0.19	97-582 M-165735-01-2
97636BO1 Bologna, Italy, 1997 (Chardonnay)	WG	0.12	0.011	1120	7	0 20 30 40	Grapes	0.17 0.13 0.11 0.06	0.045 0.048 0.033 0.023	0.22 0.18 0.15 0.09	97-636 M-165270-01-1
97742GR1 Lalioi, Greece, 1997 (Soultaína)	WG	0.12	0.012	931–1027	7	32 42	Grapes	0.042 0.021	0.036 0.021	0.08 0.04	97-742 M-165477-01-1
97687C1 St Pere Molanta, Spain, 1997 (Mocabeo)	WG	0.13	0.025–0.038	333–500	7	30 40	Grapes	0.10 0.12	0.034 0.042	0.14 0.17	97-687 M-165275-01-1
98561RN1, Vallet (44), France, 1998 (Melon de Bourgogne)	WG	0.13	0.054	246	7	28	Grapes	0.08	0.03	0.11	98-561 M-166547-01-1
	WG	0.13	0.053	246	7	28	Grapes	0.14	0.042	0.19	
98562RN1 Vallet (44), France, 1998 (Cabernet)	WG	0.13	0.058	230	7	24	Grapes	0.15	0.04	0.19	98-562 M-170328-01-1
98562AV1 Séguret (84), France, 1998 (Grenache)	WG	0.13	0.04–0.045	293–330	7	25	Grapes	0.12	0.039	0.16	
98562TL1, Montady (34), France, 1998 (Cinsault)	WG	0.13	0.042	319	7	28	Grapes	0.26	0.071	0.34	
98563BX1 Virrac (33), France, 1998 (Merlot)	WG	0.13	0.07–0.18	114–190	7	0 24 32 45	Grapes	0.23 0.23 0.12 0.18	0.04 0.04 0.022 0.028	0.27 0.27 0.14 0.21	98-563 M-166535-02-1
98563DJ1, Brochon (21), France, 1998 (Pinot Noir)	WG	0.13	0.044–0.067	200–300	7	0 23 31 46	Grapes	0.47 0.27 0.17 0.13	0.076 0.062 0.037 0.036	0.55 0.34 0.21 0.17	

Trial, Location, Country, Year (Variety)	Application					DAT Days ^a	Commo dity	Residues, mg/kg			Reference
	Form	Kg ai/ha	Kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
98563RS1, Serzy et Prin (51), France, 1998 (Pinot Meunier)	WG	0.12– 0.14	0.032	387–435	7	0 21 28 42	Grapes	0.37 0.09 0.12 0.09	0.056 0.028 0.031 0.034	0.43 0.12 0.15 0.13	
98563TL1 Pouzolles (34), France, 1998 (Carignan Noir)	WG	0.13	0.034– 0.041	323–388	7	0 21 28 42	Grapes	0.88 0.18 0.13 0.18	0.085 0.048 0.05 0.089	0.97 0.23 0.19 0.28	
98631C1 St Pere Molanta, Spain, 1998 (Macabeo)	WG	0.13	0.017– 0.033	400–800	7	0 20 29 41	Grapes	0.56 0.23 0.21 0.12	0.12 0.071 0.068 0.072	0.69 0.31 0.29 0.20	98-631 M-166092-01-1
98690GR1 Korinthos, Greece, 1998 (Soultanina)	WG	0.13– 0.14	0.013	995– 1029	7	29 40	Grapes	0.06 0.04	0.02 < 0.02	0.08 0.06	98-690 M-166463-01-1
	WG	0.13	0.013	978– 1028	7	29 40	Grapes	0.05 0.06	< 0.02 0.02	0.07 0.08	
98721BO1 Bologna, Italy, 1998 (Malvasia Toscana)	WG	0.13	0.012	1120	7	0 20 30 41	Grapes	0.93 0.46 0.26 0.19	0.15 0.13 0.10 0.076	1.1 0.60 0.37 0.27	98-721 M-170375-01-1
						20 30 41		0.82 0.39 0.24 0.13	0.13 0.12 0.12 0.073	0.96 0.52 0.37 0.21	
						20 30 41		0.82 0.39 0.24 0.13	0.13 0.12 0.12 0.073	0.96 0.52 0.37 0.21	
						20 30 41		0.82 0.39 0.24 0.13	0.13 0.12 0.12 0.073	0.96 0.52 0.37 0.21	
09-2179-01 Saint Nicolas de Bourgueil (37), France, 2009 (Cabernet Franc)	WG	0.13	0.067	200	3	–0 0 21 28 35	Bunches	0.24 0.27 0.21 0.17 0.16	0.04 0.04 0.03 0.02 0.02	0.28 0.31 0.24 0.19 0.18	09-2179 M-404520-01-1
09-2179-02 Sausenheim, Germany, 2009 (Dornfelder)	WG	0.13	0.017	800	3	–0 0 21 28 35	Bunches	0.23 0.44 0.25 0.24 0.26	0.09 0.12 0.08 0.07 0.08	0.33 0.57 0.34 0.32 0.35	
09-2179-03 Emines, Belgium, 2009 (Solaris)	WG	0.13	0.013	1000	3	–0 0 21 28 35	Bunches	0.17 0.24 0.10 0.13 0.10	0.06 0.07 0.03 0.03 0.03	0.24 0.32 0.13 0.16 0.13	
09-2179-04 Steinfeld, Germany, 2009 (Müller-Thurgau)	WG	0.13	0.017	800	3	–0 0 21 28 35	Bunches	0.29 0.63 0.37 0.33 0.26	0.10 0.13 0.11 0.08 0.07	0.40 0.77 0.49 0.42 0.34	
09-2180-01 Graveson (13), France, 2009 (Carignan)	WG	0.13	0.067	200	3	–0 0 21 28 35	Bunches	0.18 0.20 0.15 0.07 0.09	0.03 0.04 0.03 0.02 0.03	0.21 0.24 0.18 0.09 0.12	
09-2180-02 Rocha Macieira, Portugal, 2009 (Syrah)	WG	0.13	0.013	1000	3	–0 0 21 28 35	Bunches	0.08 0.41 0.29 0.22 0.11	0.01 0.06 0.03 0.03 0.02	0.09 0.48 0.32 0.25 0.13	
09-2180-03 La Fortesa–Piera, Spain, 2009 (Macabeo)	WG	0.13	0.022	600	3	–0 0 21 28 35	Bunches	0.28 0.31 0.31 0.21 0.19	0.05 0.06 0.04 0.04 0.03	0.34 0.38 0.35 0.25 0.22	09-2180 M-404444-01-1
09-2180-04 Bologna, Italy, 2009 (Chardonnay)	WG	0.12– 0.13	0.013	928– 1000	3	–0 0 21 28 35	Bunches	0.17 0.18 0.12 0.09 0.06	0.09 0.11 0.06 0.04 0.03	0.27 0.30 0.19 0.13 0.09	

Fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT Days ^a	Commo dity	Residues, mg/kg			Reference
	Form	Kg ai/ha	Kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
10-2010-01, Athée sur Cher (37), France, 2010 (Chardonnay)	WG	0.13	0.067	200	3	0 28	Bunches	0.22 <u>0.13</u>	0.01 0.02	0.23 <u>0.15</u>	10-2010 M-419813-01-1
10-2010-02 St Nicolas de Bourgueil (37), France, 2010 (Cabernet Franc)	WG	0.13	0.067	200	3	0 28	Bunches	0.38 <u>0.10</u>	0.01 < 0.01	0.39 <u>0.11</u>	
10-2010-03 Roschbach, Germany, 2010 (Müller-Thurgau)	WG	0.13	0.017	800	3	0 28	Bunches	0.29 <u>0.30</u>	0.03 0.04	0.32 <u>0.34</u>	
10-2010-04 Uhldingen-Mühl- dorf, Germany, 2010 (Spätburgunder)	WG	0.13	0.013	1000	3	0 28	Bunches	0.40 <u>0.22</u>	0.03 0.03	0.43 <u>0.25</u>	
10-2011-01, Lentilly (69), France, 2010 (Aligate)	WG	0.13	0.067	200	3	0 28	Bunches	0.51 <u>0.04</u>	0.04 0.02	0.55 <u>0.06</u>	10-2011 M-435899-01-1
10-2011-02, Campo Arcis, Spain, 2010 (Bobal)	WG	0.13	0.013	1000	3	0 28	Bunches	0.33 <u>0.08</u>	0.02 0.01	0.35 <u>0.09</u>	
10-2011-03 Bologna, Italy, 2010 (Moscato)	WG	0.13	0.013	1000	3	0 28	Bunches	0.29 <u>0.06</u>	0.07 0.03	0.37 <u>0.09</u>	
10-2011-04 Ribafria, Portugal, 2010 (Syrah)	WG	0.13	0.027	500	3	0 28	Bunches	0.47 <u>0.28</u>	0.04 0.05	0.51 <u>0.34</u>	
GAP, Brazil	SC	0.15		1000	3	7					
BRA98F25- BE298F25 Indaiatuba—SP, Brazil, 1998/99 (Maria)	WG	0.15	0.015	1000	3	7	Bunches	< 0.02			R.19/00 M-285309-01-1
	SC	0.15	0.015	1000	3	7	Bunches	0.02	< 0.02	< 0.04	R.33/01 M-285002-01-1
	WG	0.3	0.03	1000	3	7	Bunches	0.07			R.19/00 M-285309-01-1
	SC	0.3	0.03	1000	3	7	Bunches	0.07	0.04	0.11	R.33/01 M-285002-01-1
BRA98F25- BE198F25 Jundiaí—SP, Brazil, 1998/99 (Niagara)	WG	0.15	0.015	1000	3	1 3 5 7 10	Bunches	0.04 0.04 0.04 0.02 0.03			R.20/00 M-285311-01-1
	SC	0.15	0.015	1000	3	1 3 5 7 10	Bunches	0.04 0.04 0.04 0.02 0.03	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.06 0.06 0.06 0.04 0.05	R.34/01 M-285003-01-1
	WG	0.3	0.03	1000	3	7	Bunches	0.07			R.20/00 M-285311-01-1
	SC	0.3	0.03	1000	3	7	Bunches	0.07	< 0.02	0.09	R.34/01 M-285003-01-1
FD00BRAZ15 Indaiatuba—SP, Brazil, 1998/99 (Niagara)	SC	0.15	0.015	1000	3	7	Bunches	0.03	< 0.02	0.05	R.48/01
	SC	0.3	0.03	1000	3	7	Bunches	0.08	0.03	0.11	M-285004-01-1

Strawberries

Fifteen trials on strawberries were conducted in Europe (Germany, Belgium, Netherlands and France) between 2004 and 2012. The trials were conducted in indoor strawberry crops, housed under plastic or glass. Two applications by drip irrigation to individual strawberry plants or one drip application

followed by one foliar application were made. In the trials matching Belgium GAP, both drip applications in irrigation water, made 14–26 days apart, were at a rate of 0.45 kg ai/ha. In the trials according to the UK GAP, the two applications were made at an interval of 21–22 days, the first application at 0.18 kg ai/ha (drip) and the second at 0.27 kg ai/ha (foliar spray). The results are summarized in Table 61.

Table 61 Residues in strawberries from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	Kg ai/ha	Kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
GAP, Belgium (protected)	WG	0.45		Both drip	2	35					
11-2004-02 Dorn-Esch, Germany, 2011 (Elsanta Tray)	WG	0.45	0.015	3000	2	28 35 42	Fruit	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 <u>< 0.02</u> < 0.02	11-2004 M-445339-01-1
11-2004-03 Villers-Perwin, Belgium, 2011 (Darselect)	WG	0.45	0.0075	6000	2	28 35 42	Fruit	0.02 0.02 0.02	0.01 0.01 0.01	0.03 <u>0.03</u> 0.03	
11-2004-04 Zwaagdijk-Oost, Netherlands, 2011 (Elsanta)	WG	0.45	0.015	3000	2	28 35 42	Fruit	0.01 0.01 0.01	< 0.01 < 0.01 < 0.01	0.02 <u>0.02</u> 0.02	
10-2014-01 Cremery (80), France, 2010 (Darselect)	WG	0.45	0.0009	5000	2	35 42	Fruit	0.02 0.02	0.02 0.02	<u>0.04</u> 0.04	10-2014 M-419363-01-1
10-2014-02 Dorn-Esch, Germany, 2010 (Elsanta Tray)	WG	0.45	0.015	3000	2	28 35 42	Fruit	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 <u>< 0.02</u> < 0.02	
10-2014-03 Bossiere, Belgium, 2010 (Darselect)	WG	0.45	0.011	4000	2	28 35 42	Fruit	0.03 <u>0.02</u> 0.02	0.02 0.01 0.01	0.05 <u>0.03</u> 0.03	
10-2014-04 Zwaagdijk-Oost, Netherlands, 2010 (Elsanta)	WG	0.45	0.015	3000	2	28 35 42	Fruit	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.01	< 0.02 < 0.02 <u>0.02</u>	
GAP, UK (protected)	WG	1) 0.18 2) 0.27		Drip + foliar spray	2	35					
09-2169-01 Carpentras (84), France, 2009 (Pajaro)	WG	1) 0.18 2) 0.27	1) 0.005 2) 0.054	1) 4000 2) 500	2	28 35 42	Fruit	0.04 0.01 < 0.01	< 0.01 < 0.01 < 0.01	0.05 0.02 < 0.02	09-2169 M-401525-01-1
09-2169-02 Reute, Germany, 2009 (Charlotte)	WG	1) 0.18 2) 0.27	1) 0.005 2) 0.054	1) 4000 2) 500	2	28 34 41	Fruit	0.04 0.02 < 0.01	< 0.01 < 0.01 < 0.01	0.05 0.03 < 0.02	
09-2169-03 Bossiere, Belgium, 2009 (Elsanta)	WG	1) 0.18 2) 0.27	1) 0.005 2) 0.054	1) 4000 2) 500	2	28 35 42	Fruit	< 0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 0.02 < 0.02	

Fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	Kg ai/ha	Kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
09-2169-04 Zwaagdijk, Netherlands, 2009 (Elsanta)	WG	1) 0.18 2) 0.27	1) 0.005 2) 0.054	1) 4000 2) 500	2	28 35 42	Fruit	0.02 0.02 < 0.01	< 0.01 < 0.01 < 0.01	0.03 0.03 < 0.02	
12-2039-01 Monteux (84), France, 2012 (Clery)	WG	1) 0.16 2) 0.27	1) 0.005 2) 0.054	1) 100 mL/plant 2) 500	2	28 35 42	Fruit	0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01	0.02 0.02 < 0.02	12-2039 M-438111-02-1
12-2039-02 Warmenhuizen, Netherlands, 2012 (Sonata)	WG	1) 0.18 2) 0.27	1) 0.009 2) 0.054	1) 50 mL/plant 2) 500	2	28 35 42	Fruit	0.02 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	0.03 < 0.02 < 0.02	
12-2039-03 Villers-Perwin, Belgium, 2012 (Dorselect)	WG	1) 0.17 2) 0.27	1) 0.006 2) 0.054	1) 70 mL/plant 2) 500	2	28 35 42	Fruit	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	
12-2039-04 Tricht, Netherlands, 2012 (Elsanta)	WG	1) 0.18 2) 0.27	1) 0.009 2) 0.054	1) 50 mL/plant 2) 500	2	28 35 42	Fruit	0.05 0.02 < 0.01	< 0.01 < 0.01 < 0.01	0.06 0.03 < 0.02	

*Bulb vegetables**Leek*

Five field trials on leek were conducted in Europe (Germany, France and Netherlands) during 2003 (RA-2682/03, M-248031-01-1). Fenamidone was applied by foliar spray four times to leek plants at the rate of 0.15 kg ai/ha. The results are summarized in Table 62.

Table 62 Residues in leek from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT Days	Commodity	Residues, mg/kg		
	Form	Kg ai/ha	Kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum
GAP, Switzerland	SC	0.15		300	3	14				
R 2003 0473/0, Bornheim, Germany, 2003 (Amundo)	SC	0.015	0.005	300	4	0 14	Stem	0.10 < 0.01	< 0.01 < 0.01	0.1 < 0.01
R 2003 0553/2, Höfchen, Germany, 2003 (Ventura)	SC	0.15	0.05	300	4	0 14	Stem	0.70 <u>0.02</u>	0.02 < 0.01	0.72 <u>0.02</u>
R 2003 0554/0, Zwaagdijk, Netherlands, 2003 (Heracles)	SC	0.15	0.05	300	4	0 14	Stem	0.35 <u>0.07</u>	0.01 < 0.01	0.36 <u>0.07</u>
R 2003 0555/9, Langenfeld, Germany, 2003 (Ventura)	SC	0.15	0.05	300	4	0 14	Stem	0.80 <u>0.05</u>	0.02 < 0.01	0.82 <u>0.05</u>
R 2003 0556/7 Criquebeuf sur Seine, France, 2003 (Maine)	SC	0.15	0.05	300	4	0 14	Stem	0.90 <u>0.13</u>	0.02 < 0.01	0.92 <u>0.13</u>

Onion, bulb

Supervised field residue trials were conducted in Brazil in 2001, in the USA in 1999 and in Europe (Germany, France, Netherlands and the UK) in 2003. In Europe, fenamidone was applied by foliar

spray four times to onion plants at the rate of 0.15 kg ai/ha, whereas the US trials were conducted with 6×0.2 kg ai/ha (interval 7 days). The results are summarized in Table 63.

Table 63 Residues in bulb onion from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DA T days	Commodity	Residues, mg/kg			Reference
	Form	Kg ai/ha	Kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
GAP, Switzerland	SC	0.15		500	3	14					
R2003 0474/9 Burscheid, Germany, 2003 (Stuttgarter Riesen)	SC	0.15	0.05	300	4	0 14	Bulb	0.01 < 0.01	< 0.01 < 0.01	0.01 < 0.01	RA-2683/03 M-248949-01-1
R2003 0557/5 Langenfeld, Germany, 2003 (Stuttgarter Riesen)	SC	0.15	0.05	300	4	0 14	Bulb	0.02 < 0.01	< 0.01 < 0.01	0.01 < 0.01	
R2003 0558/3 Epone (78), France, 2003 (Barito)	SC	0.15	0.05	300	4	0 14	Bulb	0.05 0.01	< 0.01 < 0.01	0.05 0.01	
R2003 0559/1 Wieringerwerf, Netherlands, 2003 (Hyskin)	SC	0.15	0.05	300	4	0 14	Bulb	0.06 0.02	< 0.01 < 0.01	0.06 0.02	
R2003 0560/5 Elveden, UK, 2003 (Sherpa)	SC	0.15	0.05	300	4	0 14	Bulb	0.05 0.01	< 0.01 < 0.01	0.05 0.01	
GAP, USA	SC	0.20			4	7					
14461-01 North Rose (NY), USA, 1999 (Crusader)	SC	0.20	0.042	466–489	6	7	Bulb	<u>< 0.02</u> (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	98W14461 M-238680-01-1
14461-02 Arkansaw (WI), USA, 1999 (Walla Walla)	SC	0.20	0.043	463–481	6	7	Bulb	<u>< 0.02</u> (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
14461-03 Uvalde (TX), USA, 1999 (Cimmaron)	SC	0.20	0.042	486–489	6	7	Bulb	<u>< 0.02</u> (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
14461-06 San Juan Bautista (CA), USA, 1999 (Festival)	SC	0.20	0.04	479–500	6	7	Bulb	<u>0.10</u> (0.07, 0.13)	< 0.02 (<u>< 0.02</u> , < 0.02)	0.10	
14461-07 Ephrata (WA), USA, 1999 (Apex)	SC	0.20	0.04	459–467	6	7	Bulb	<u>< 0.02</u> (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
14461-08 Corvallis (OR), USA, 1999 (Copra)	SC	0.19–0.20	0.04	467–490	6	7	Bulb	<u>< 0.02</u> (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
14461-13 Manteca (CA), USA, 1999 (Rio Durado)	SC	0.20	0.043	463–480	6	3 5 7 10 14	Bulb	0.02 < 0.02 <u>0.02</u> (<u>< 0.02</u> , 0.02) < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 (<u>< 0.02</u> , < 0.02) < 0.02 < 0.02	0.02 < 0.02 <u>0.02</u> < 0.02 < 0.02	

Fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	Kg ai/ha	Kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
14461-19 Levelland (TX), USA, 1999 (Candy)	SC	0.20–0.21	0.04–0.086	232–488	6	7	Bulb	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
GAP, Brazil	SC	0.15	0.015	1000	3	7					
ER02BRAFO7-3 São José do Rio Pardo—SP, Brazil, 2001 (Baia Dura Hortiçeres)	SC	0.20	0.02	1000	3	7	Bulb	< 0.02	< 0.02	< 0.02	R.75/02 M-285013-01-1
	SC	0.40	0.04	1000	3	7	Bulb	< 0.02	< 0.02	< 0.02	
ER02BRAFO7-1 Paulinia—SP Brazil, 2001 (Baia Periforme)	SC	0.20	0.02	1000	3	7	Bulb	< 0.02	< 0.02	< 0.02	R.82/02 M-284950-01-1
	SC	0.40	0.04	1000	3	7	Bulb	< 0.02	< 0.02	< 0.02	
ER02BRAFO7-2 Piedade—SP Brazil, 2001 (Crioula)	SC	0.20	0.02	1000	3	7	Bulb	< 0.02	< 0.02	< 0.02	R.83/02 M-284958-01-1
	SC	0.40	0.04	1000	3	7	Bulb	0.03	< 0.02	0.03	

Spring onion

Four trials on spring onion were conducted in the USA in 1999 (98W14461, M-238680-01-1). Fenamidone was applied by foliar spray to onion plants at the rate of 6×0.2 kg ai/ha (interval 7 days). For results see Table 64.

Table 64 Residues in spring onion from trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg		
	Form	Kg ai/ha	Kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum
GAP, USA	SC	0.2			4	7				
14461-14 Conklin (MI), USA, 1999 (Ishikura Improved)	SC	0.2	0.04	458–471	6	3	Plant w/o Roots	0.70	< 0.02	0.70
						5		0.39	< 0.02	0.39
						7		<u>0.24</u> (0.22, 0.25)	< 0.02 (< 0.02, < 0.02)	<u>0.24</u>
						10 14		0.15 0.06	< 0.02 < 0.02	0.15 0.06
14461-16 Kerman (CA), USA, 1999 (Southport 404)	SC	0.2	0.04	467–478	6	7	Plant w/o Roots	<u>0.94</u> (0.77, 1.1)	0.05 (0.03, 0.06)	1.1
14461-17 San Juan Bautista (CA), USA, 1999 (Parade)	SC	0.2	0.04	475–489	6	7	Plant w/o Roots	<u>0.94</u> (0.84, 1.03)	< 0.02 (< 0.02, < 0.02)	0.94
14461-20 Levelland (TX), USA, 1999 (Evergreen White Bunching)	SC	0.2	0.04	468–474	6	7	Plant w/o Roots	<u>0.36</u> (0.41, 0.30)	< 0.02 (< 0.02, < 0.02)	0.36

*Brassica vegetables**Cabbage*

Field trials on head cabbage were conducted in Europe (Germany, France and Portugal) and in the USA during 2003. In the European trials, fenamidone was sprayed three times at the rate of 0.15 kg ai/ha. In the USA, four applications were made at intervals of 4–5 days, at the rate of 0.28–0.31 kg ai/ha. The results are summarized in Table 65.

Table 65 Residues in cabbage from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT Days	Commodity	Residues, mg/kg			Reference
	Form	Kg ai/ha	Kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
GAP, Switzerland	SC	0.15	0.05	500	3	14					
R2003 1153/2 VechtaHagstedt, Germany, 2003 (Lenox)	SC	0.15	0.05	300	3	0 7 14 21	Head	0.09 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.09 < 0.01 < 0.01 < 0.01	RA-2900/03 M-248911-01-1
R2003 1157/5 WerlWestönnen, Germany, 2003 (Lion)	SC	0.15	0.05	300	3	0 7 14 21	Head	0.08 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.08 < 0.01 < 0.01 < 0.01	
R2003 1158/3 Langenfeld, Germany, 2003 (Atraktion)	SC	0.15	0.05	300	3	0 7 14 22	Head	0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.01 < 0.01 < 0.01 < 0.01	
R2003 1159/1 Leichlingen, Germany, 2003 (Rodeo)	SC	0.15	0.05	300	3	0 7 14 22	Head	0.03 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.03 < 0.01 < 0.01 < 0.01	
R2003 1160/5 Rilly sur Vienne (37), France, 2003 (Impala)	SC	0.15	0.05	250	3	0 8 14 21	Head	0.04 < 0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.04 < 0.01 0.01 < 0.01	
R2003 1154/0 Reynies (82), France, 2003 (Rigoletto)	SC	0.15	0.05	300	3	0 7 14 21	Head	0.92 0.18 0.06 0.04	0.04 0.01 < 0.01 < 0.01	0.96 0.19 0.06 0.04	RA-2901/03 M-248027-01-1
R2003 1161/3 Vale Bern Feito, Portugal, 2003 (Merlicias)	SC	0.17	0.05	333	3	0 7 14 21	Head	0.95 0.04 0.02 < 0.01	0.04 < 0.01 < 0.01 < 0.01	0.99 0.04 0.02 < 0.01	
GAP, USA	SC	0.3			3	2					
YW19CB01-YW007-03H York Springs (PA), USA, 2003 (Bonnies Hybrid)	SC	0.3	0.14–0.16	184–210	4	2	Head with wrapper leaves	0.35 (0.07, 0.62)	< 0.02 (< 0.02, < 0.02)	0.35	201225 M-243561-01-3
YW19CB01-YW008-03H Tifton (GA), USA, 2003 (Atlantic)	SC	0.3	0.17–0.19	161–176	4	2	Head with wrapper leaves	0.52 (0.38, 0.65)	< 0.02 (< 0.02, < 0.02)	0.52	
						2	Head w/o wrapper leaves	0.19 (0.19, 0.19)	< 0.02 (< 0.02, < 0.02)	0.19	
YW19CB01-YW009-03D Molino (FL), USA, 2003 (Bravo)	SC	0.28–0.31	0.14	198–216	4	0	Head with wrapper leaves	0.13 (0.09, 0.16)	< 0.02 (< 0.02, < 0.02)	0.13	
						2		0.10 (0.12, 0.07)	< 0.02 (< 0.02, < 0.02)	0.10	
						3		0.06 (0.06, 0.05)	< 0.02 (< 0.02, < 0.02)	0.06	
						5 7		0.08 (0.12, 0.05) 0.05 (0.05, 0.05)	< 0.02 (< 0.02, < 0.02) < 0.02 (< 0.02, < 0.02)	0.08 0.07	

Fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT Days	Commodity	Residues, mg/kg			Reference
	Form	Kg ai/ha	Kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
						2	Head w/o wrapper leaves	0.03 (0.03, < 0.02)	< 0.02 (< 0.02, < 0.02)	0.03	
YW19CB01-YW010-03H Seymour (IL), USA, 2003 (Golden Acre)	SC	0.3	0.2	153–154	4	2	Head with wrapper leaves	0.17 (0.08, 0.26)	< 0.02 (< 0.02, < 0.02)	0.17	
						2	Head w/o wrapper leaves	< 0.02	< 0.02 (< 0.02, < 0.02)	< 0.02	
YW19CB01-YW011-03H Uvalde (TX), USA, 2003 (Blue Thunder)	SC	0.29–0.3	0.14–0.15	198–212	4	2	Head with wrapper leaves	0.22 (0.25, 0.19)	< 0.02 (< 0.02, < 0.02)	0.22	
YW19CB01-YW012-03H Fresno (CA), USA, 2003 (Gazelle)	SC	0.3	0.16–0.17	171–182	4	2	Head with wrapper leaves	0.24 (0.19, 0.28)	< 0.02 (< 0.02, < 0.02)	0.24	
						2	Head w/o wrapper leaves	< 0.02	< 0.02 (< 0.02, < 0.02)	< 0.02	

Flowerhead Brassicas

Field trials on cauliflower were conducted in Europe (Germany, France, Italy and the UK) with 3×0.15 kg ai/ha. In the USA, four treatments at a rate of 0.29–0.31 kg ai/ha were made by foliar spray on broccoli at intervals of 4–6 days. The results are summarized in Table 66.

Table 66 Residues in flowerhead brassicas from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
GAP Switzerland	SC	0.15	0.05	300	3	14					
R2003 1155/9 St Lambert des Levees (49), France, 2003 (Thalassa)	SC	0.15	0.06	250	3	0 7	Whole plant	0.95 0.21	0.02 < 0.01	0.97 0.22	RA-2902/03 M-247895-01-1
						14 21	Inflorescence	< 0.01 0.01	< 0.01 < 0.01	< 0.02 0.02	
R2003 1162/1 Langenfeld, Germany, 2003 (Fremont)	SC	0.15	0.05	300	3	0	Whole plant	0.90	0.02	0.92	
						6 14 22	Inflorescence	0.03 < 0.01 0.01	< 0.01 < 0.01 < 0.01	0.04 < 0.02 0.02	
R2003 1164/8 Falkenham, UK, 2003 (Pierrot)	SC	0.15	0.05	300	3	0 7 14 21	Inflorescence	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02	
R2003 1165/6 Faverolles (80), France, 2003 (Aviso)	SC	0.15	0.05	300	3	0 7 14 21	Inflorescence	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02	

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
R2003 1156/7 Zapponeta, Italy, 2003 (Verde di Macerata)	SC	0.15	0.05	300	3	0 7 14 21	Inflorescence	0.45 0.06 0.08 0.02	0.05 < 0.01 0.01 < 0.01	0.51 0.07 0.09 0.03	RA-2903/03 M-247987- 01-1
R2003 1166/4 St Etienne du Gres (13), France, 2003 (Fremont)	SC	0.15	0.05	300	3	0 7	Whole plant	1.4 0.41	0.16 0.05	1.58 0.47	
						13 21	Inflorescence	< 0.01 < 0.01	< 0.01 < 0.01	< 0.02 < 0.02	
GAP, USA	SC	0.3			3	2					
YW19BR01- YW001-03H Raymondville (TX), USA, 2003 (Green Sprout Calabrese)	SC	0.3– 0.31	0.16	189– 191	4	2	Inflorescence	<u>1.6</u> (1.08, 2.05)	< 0.02 (< 0.02, < 0.02)	1.6	201225 M-243561- 01-1
YW19BR01- YW002-03-D Fresno (CA), USA, 2003 (Bell Star)	SC	0.29– 0.31	0.17	175– 179	4	0 2 3 5 7	Inflorescence	0.95 (0.96, 0.94) <u>0.68</u> (0.73, 0.63) 0.68 (0.47, 0.88) 0.57 (0.54, 0.59) 0.42 (0.52, 0.32)	< 0.02 (< 0.02, < 0.02) < 0.02 (< 0.02, < 0.02) < 0.02 (< 0.02, < 0.02) < 0.02 (< 0.02, < 0.02) < 0.02 (< 0.02, < 0.02)	0.95 <u>0.68</u> 0.68 0.57 0.42	
YW19BR01- YW003-03H Fresno (CA), USA, 2003 (Marathon)	SC	0.3	0.14	208– 212	4	2	Inflorescence	<u>1.5</u> (1.25, 1.66)	< 0.02 (< 0.02, < 0.02)	1.5	
YW19BR01- YW004-03H Porterville (CA), USA, 2003 (Marathon)	SC	0.3– 0.31	0.12– 0.13	242– 244	4	2	Inflorescence	<u>0.51</u> (0.47, 0.55)	< 0.02 (< 0.02, < 0.02)	0.51	
YW19BR01- YW005-03H Glenn (CA), USA, 2003 (Green Belt)	SC	0.3– 0.31	0.14– 0.15	206– 210	4	2	Inflorescence	<u>0.31</u> (0.31, 0.31)	< 0.02 (< 0.02, < 0.02)	0.31	
YW19BR01- YW006-03H Hillsboro (OR), USA, 2003 (Packman)	SC	0.29– 0.3	0.19– 0.2	155– 160	4	2	Inflorescence	<u>2.2</u> (1.71, 2.72)	< 0.02 (< 0.02, < 0.02)	2.2	

Fruiting vegetables, Cucurbits

Cucumber

Nine trials on cucumber performed indoor were conducted in Europe during 2002, 2003 and 2004: two trials in France, three in Spain, two in Italy, one in Greece and one in the Netherlands with 3 × 0.15 kg ai/ha by foliar spray. Nine supervised trials on cucumbers were performed outdoors during 1999 in the USA. Six applications in the range of 0.19–0.215 kg ai/ha at intervals of 3–6 days were made. The results are summarized in Table 67.

Table 67 Residues in cucumber from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
GAP, Switzerland	WG	0.15			3	3					
02R651-1, La Mojonera, Spain, 2002 (Trópico)	WG	0.15	0.013–0.015	1000–1150	3	0 3	Fruit	0.13 <u>0.12</u>	< 0.02 < 0.02	0.13 <u>0.12</u>	02 R 651 M-234100-01-1
02R651-2 Giovinazzo, Italy, 2002 (Sarig)	WG	0.15	0.03	500	3	0 3	Fruit	0.12 <u>0.10</u>	< 0.02 < 0.02	0.12 <u>0.10</u>	
02R651-3, Zwaagdijk, Netherlands, 2002 (Enduro)	WG	0.15	0.01	1500	3	0 3	Fruit	0.17 <u>0.13</u>	0.05 < 0.02	0.23 <u>0.13</u>	
02R653-1, La Mojonera, Spain, 2002 (Trópico)	WG	0.15	0.013–0.015	1000–1150	3	0 3	Fruit	0.14 <u>0.09</u>	< 0.02 < 0.02	0.14 <u>0.09</u>	02 R 653 M-234103-01-1
02R653-2, Esovalta, Greece, 2002 (722)	WG	0.15	0.015	1000	3	0 3	Fruit	0.02 <u>0.04</u>	< 0.02 < 0.02	0.02 <u>0.04</u>	
02R653-3, Molfetta, Italy, 2002 (Locale Di Polignano)	WG	0.15	0.015	1000	3	0 3	Fruit	0.07 <u>< 0.02</u>	< 0.02 < 0.02	0.07 <u>< 0.02</u>	
R 2003 0275/4, St Jory (31), France, 2003 (Aramon)	WG	0.15	0.015	1000	3	0 3	Fruit	0.03 <u>< 0.01</u>	< 0.01 < 0.01	0.03 <u>< 0.01</u>	RA-2648/03, M-235795-01-1
R 2004 0070 5 Chazay d'Azergues (69), France, 2004 (Gardon)	SC	0.15	0.015	1000	3	0 3	Fruit	0.12 <u>0.10</u>	< 0.01 < 0.01	0.12 <u>0.10</u>	RA-2540/04 M-262524-01-1
R 2004 0367 4 Monte Algaida Sanlucar, Spain, 2004 (Alanis F1)	SC	0.15	0.015	1000	3	0 3	Fruit	0.09 <u>0.04</u>	< 0.01 < 0.01	0.09 <u>0.04</u>	
GAP, USA	SC	0.2			4	14					
14486-01, Clayton (NC), USA, 1999 (Sweet Slice F-1)	SC	0.2–0.21	0.12–0.14	149–176	6	14	Fruit	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	99W14486 M-240408-01-1
14486-02, Lucama (NC), USA, 1999 (National Pickling)	SC	0.2–0.22	0.11	175–195	6	15	Fruit	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
14486-03, Chula (GA), 1999 (Lightning)	SC	0.2	0.11–0.15	130–179	6	14	Fruit	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
14486-04, Rose Hill (NC), USA, 1999 (Poinsett)	SC	0.19–0.2	0.14	138–145	6	14	Fruit	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
14486-05, Oviedo (FL), USA, 1999 (Poinsett 76)	SC	0.2	0.14	137–142	6	14	Fruit	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
14486-06, York (NE), USA, 1999 (Straight Eight)	SC	0.2	0.11	182–189	6	13	Fruit	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
14486-07, Arkansaw (WI), USA, 1999 (Lucky Strike)	SC	0.2	0.1–0.11	185–188	6	14	Fruit	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
14486-08, Uvalde (TX), USA, 1999 (Supersett)	SC	0.19–0.2	0.14	136–142	6	14	Fruit	0.02 (0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	0.02	

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
14486-09, San Juan Bautista (CA), USA, 1999 (Dasher 11)	SC	0.2	0.12	169–175	6	14	Fruit	0.04 (0.04, 0.03)	< 0.02 (< 0.02, < 0.02)	0.04	

Summer squash

Nine field trials on summer squash were carried out during 1999 in the USA (99W14487, M-240407-01-1). At each trial, six spray applications in the range of 0.19–0.21 kg ai/ha were made at intervals of 4–6 days. The results are summarized in Table 68.

Table 68 Residues in summer squash from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg		
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum
GAP, USA	SC	0.2			4	14				
14487-01, Germansville (PA), USA, 1999 (Multipik)	SC	0.2–0.21	0.11–0.12	179–185	6	14	Fruit	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02
14487-02, Clayton (NC), USA, 1999 (Seneca Prolific F-1)	SC	0.2–0.21	0.12–0.13	163–177	6	7	Fruit	< 0.02	< 0.02	< 0.02
						10		< 0.02	< 0.02	< 0.02
						14		< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02
						17 21		< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)
14487-03, Lucama (NC), USA, 1999 (Early Prolific Straightneck)	SC	0.2	0.11	175–184	6	14	Fruit	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02
14487-04, Chula (GA), USA, 1999 (Dixie Hybrid)	SC	0.2	0.11–0.16	125–176	6	14	Fruit	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02
14487-05, Oviedo (FL), USA, 1999 (Yellow Crookneck)	SC	0.19–0.2	0.14	135–142	6	14	Fruit	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02
14487-06, Arkansaw (WI), USA, 1999 (Monet)	SC	0.2	0.11	185–189	6	14	Fruit	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02
14487-07, Uvalde (TX), USA, 1999 (Multipik F1)	SC	0.2–0.21	0.14	138–144	6	14	Fruit	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02
14487-08, San Juan Bautista (CA), USA, 1999 (Golden Summer Crookneck)	SC	0.2	0.11–0.12	168–179	6	14	Fruit	0.06 (0.04, 0.08)	< 0.02 (< 0.02, < 0.02)	0.06
14487-09, Ephrata (WA), USA, 1999 (Black Beauty Zucchini)	SC	0.2–0.21	0.14	141–150	6	14	Fruit	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02

Melons

Twenty trials on melon were conducted in Europe during 2000, 2002 and 2005, six trials in France, six in Spain, six in Italy, one in Greece and one in Portugal. The spray application rates were 3×0.14 –0.15 kg ai/ha. Twelve trials were conducted outdoors and eight trials conducted indoors. In the indoor trials (02 R 656, M220462-01-1), the samples were stored frozen for about 18 months.

Fenamidone

Eight supervised trials on cantaloupe melon were carried out outdoors during 1999 in the USA. Fenamidone was applied six times by foliar spray at rates of 0.19–0.21 kg ai/ha at intervals of 5 days. The results are summarized in Table 69.

Table 69 Residues in melons from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference, remarks
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
GAP, Switzerland	SC	0.15			3	3					
00535AV1, Isle sur la Sorgues (84), France, 2000 (Presto F1) (Outdoors)	WG	0.14	0.04	333– 345	3	0 7 14 18 18	Fruit Fruit Fruit Peel Pulp Fruit	0.04 0.03 < 0.02 0.03 < 0.02 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.04 0.03 < 0.02 0.03 < 0.02 0.02	00-535 M-205672- 01-1
00535BX1, Virsac (84), France, 2000 (Cantaloupe charentais) (Outdoors)	WG	0.14	0.04	350	3	0 7 15 21 21 21	Fruit Fruit Fruit Peel Pulp Fruit	0.09 0.04 0.03 0.04 < 0.02 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.09 0.04 0.03 0.04 < 0.02 0.02	
00536AV1, Robion (84), France, 2000 (Manta) (Outdoors)	WG	0.14	0.014	1000	3	7 7 7 14 14 14	Peel Pulp Fruit Peel Pulp Fruit	0.03 < 0.02 0.02 0.03 < 0.02 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.03 < 0.02 0.02 0.03 < 0.02 0.02	00-536 M-205151- 01-1
00536TL1, Béziers (34), France, 2000 (Amigo) (Outdoors)	WG	0.14	0.04	357	3	7 7 7 14 14 14	Peel Pulp Fruit Peel Pulp Fruit	0.03 < 0.02 0.03 0.05 < 0.02 0.03	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.03 < 0.02 0.03 0.05 < 0.02 0.03	
02R655-1, Alginet, Spain, 2002 (Cantalup) (Outdoors)	WG	0.15	0.019	800	3	0 3 3 3 3	Fruit Peel Pulp Fruit	< 0.02 < 0.02 < 0.02 < 0.02 <u>< 0.02</u>	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 <u>< 0.02</u> < 0.02	02 R 655 M-219005- 01-1
02R655-2, San Giovanni In Persiceto, Italy, 2002 (Calipso) (Outdoors)	WG	0.15	0.025	600	3	0 3 3 3	Fruit Peel Pulp Fruit	0.03 0.09 < 0.02 <u>0.04</u>	< 0.02 < 0.02 < 0.02 < 0.02	0.03 0.09 < 0.02 <u>0.04</u>	
R2005 0299/0 Alginet, Spain, 2005 (Sirio) (Outdoors)	SC	0.15	0.015	1000	3	–0 0 1 3 3 3 3 7 7 7 14 14 14	Fruit Fruit Fruit Peel Pulp Fruit Fruit Peel Pulp Fruit Peel Pulp Fruit	0.02 0.03 0.03 0.04 < 0.01 <u>0.04</u> 0.08 < 0.01 0.02 0.04 < 0.01 0.02 <u>0.12</u>	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.03 0.03 0.04 < 0.01 0.04 0.08 < 0.01 0.02 0.04 < 0.01 0.02 0.12	RA-2536/05 M-279019- 01-1
R2005 0300/8 Vilanova i la Geltrú, Spain, 2005 (Piel de Sapo) (Outdoors)	SC	0.15	0.015– 0.019	800– 1000	3	0 0 0 3 3 3 7 7 7	Peel Pulp Fruit Peel Pulp Fruit Peel Pulp Fruit	0.27 < 0.01 0.16 0.23 < 0.01 0.10 0.29 < 0.01 <u>0.12</u>	0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01	0.28 < 0.01 0.16 0.24 < 0.01 0.10 0.30 < 0.01 0.12	

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference, remarks
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
R2005 0903/0 Licodia Eubea, Italy, 2005 (Helios) (Outdoors)	SC	0.15	0.015– 0.25	600– 1000	3	–0	Fruit	0.01	< 0.01	0.01	
						0	Fruit	0.02	< 0.01	0.02	
						1	Fruit	0.02	< 0.01	0.02	
						3	Peel	0.09	< 0.01	0.09	
						3	Pulp	< 0.01	< 0.01	<u>< 0.01</u>	
						3	Fruit	0.02	< 0.01	0.02	
						7	Peel	0.05	< 0.01	0.05	
						7	Pulp	< 0.01	< 0.01	< 0.01	
						7	Fruit	<u>0.03</u>	< 0.01	0.03	
						14	Peel	0.04	< 0.01	0.04	
						14	Pulp	< 0.01	< 0.01	< 0.01	
						14	Fruit	0.02	< 0.01	0.02	
R2005 0904/9 Aronas, Greece, 2005 (Velos) (Outdoors)	SC	0.15	0.03	500	3	–0	Fruit	0.04	< 0.01	0.04	
						0	Fruit	0.12	0.01	0.13	
						1	Fruit	0.10	< 0.01	0.10	
						3	Peel	0.57	0.07	0.65	
						3	Pulp	< 0.01	< 0.01	<u>< 0.01</u>	
						3	Fruit	<u>0.08</u>	< 0.01	0.08	
						7	Peel	0.29	0.03	0.32	
						7	Pulp	< 0.01	< 0.01	< 0.01	
						7	Fruit	0.06	< 0.01	0.06	
						14	Peel	0.14	0.02	0.16	
						14	Pulp	< 0.01	< 0.01	< 0.01	
						14	Fruit	0.04	< 0.01	0.04	
R2005 0905/7 Aldeia Gavinha, Portugal, 2005 (Pele de Sapo) (Outdoors)	SC	0.15	0.019	800	3	–0	Fruit	0.02	< 0.01	0.02	
						0	Fruit	0.07	< 0.01	0.07	
						1	Fruit	0.04	< 0.01	0.04	
						3	Peel	0.11	< 0.01	0.11	
						3	Pulp	< 0.01	< 0.01	<u>< 0.01</u>	
						3	Fruit	<u>0.05</u>	< 0.01	0.05	
						7	Peel	0.06	< 0.01	0.06	
						7	Pulp	< 0.01	< 0.01	< 0.01	
						7	Fruit	0.04	< 0.01	0.04	
						14	Peel	0.04	< 0.01	0.04	
						14	Pulp	< 0.01	< 0.01	< 0.01	
						14	Fruit	0.02	< 0.01	0.02	
R2005 0906/5 Manfredonia, Italy, 2005 (Proteo) (Outdoors)	SC	0.15	0.015	1000	3	0	Peel	0.12	< 0.01	0.12	
						0	Pulp	< 0.01	< 0.01	< 0.01	
						0	Fruit	0.05	< 0.01	0.05	
						3	Peel	0.19	0.01	0.20	
						3	Pulp	< 0.01	< 0.01	<u>< 0.01</u>	
						3	Fruit	<u>0.03</u>	< 0.01	0.03	
						7	Peel	0.07	< 0.01	0.08	
						7	Pulp	< 0.01	< 0.01	< 0.01	
						7	Fruit	0.02	< 0.01	0.02	
02R656-1 Puebla de Vicar, Spain, 2002 (Vulcano) (Indoors)	WG	0.15	0.013	1200	3	0	Fruit	< 0.02	< 0.02	< 0.02	02 R 656, M-220462- 01-1
						1	Fruit	< 0.02	< 0.02	< 0.02	
						3	Peel	< 0.02	< 0.02	< 0.02	
						3	Pulp	< 0.02	< 0.02	<u>< 0.02</u>	
02R656-2 Puebla de Vicar, Spain, 2002 (Vulcano) (Indoors)	WG	0.15	0.014	1100	3	0	Fruit	0.05	< 0.02	0.05	
						3	Peel	0.07	< 0.02	0.07	
						3	Pulp	< 0.02	< 0.02	<u>< 0.02</u>	
						3	Fruit	<u>0.07</u>	< 0.02	0.07	
02R656-3 Sanlucar de Barra- meda, Spain, 2002 (Galia-Hy F1) (Indoors)	WG	0.15	0.015	1000	3	0	Fruit	0.11	< 0.02	0.11	
						1	Fruit	0.11	< 0.02	0.11	
						3	Peel	0.15	< 0.02	0.15	
						3	Pulp	< 0.02	< 0.02	<u>< 0.02</u>	
						3	Fruit	<u>0.09</u>	< 0.02	0.09	

Fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference, remarks
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
02R656-4 Virazeil (47), France, 2002 (Toloza) (Indoors)	WG	0.15	0.06	250	3	0 1 3 3 3	Fruit Fruit Peel Pulp Fruit	0.08 0.12 0.10 < 0.02 <u>0.07</u>	< 0.02 < 0.02 0.04 < 0.02 < 0.02	0.08 0.12 0.14 <u>< 0.02</u> 0.07	
02R656-5 Isle sur la Sorgues (84), France, 2002 (Luna) (Indoors)	WG	0.15	0.03	500	3	0 3 3 3	Fruit Peel Pulp Fruit	< 0.02 0.03 < 0.02 <u>< 0.02</u>	< 0.02 < 0.02 < 0.02 < 0.02	< 0.02 0.03 <u>< 0.02</u> < 0.02	
02R656-6 San Giovanni in Persiceto, Italy, 2002 (Harper) (Indoors)	WG	0.15	0.025	600	3	0 1 3 3 3	Fruit Fruit Peel Pulp Fruit	0.03 < 0.02 0.03 < 0.02 <u>< 0.02</u>	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.03 < 0.02 0.03 <u>< 0.02</u> < 0.02	
02R656-7 Giovinazzo, Italy, 2002 (Proteo) (Indoors)	WG	0.15	0.015	1000	3	0 3 3 3	Fruit Peel Pulp Fruit	0.02 0.14 < 0.02 <u>0.03</u>	< 0.02 < 0.02 < 0.02 < 0.02	0.02 0.14 <u>< 0.02</u> 0.03	
02R656-8 Teriizzi, Italy, 2002 (Proteo) (Indoors)	WG	0.15	0.019	800	3	0 3 3 3	Fruit Peel Pulp Fruit	0.04 0.08 < 0.02 <u>0.04</u>	< 0.02 0.03 < 0.02 < 0.02	0.04 0.11 <u>< 0.02</u> 0.04	
GAP, USA	SC	0.2			4	14					
14488-02, Conklin (MI), USA, 1999 (Cantaloupe Superstar)	SC	0.2	0.13– 0.14	148– 152	6	14	Fruit	0.02 (0.02, 0.02)	< 0.02 (< 0.02, < 0.02)	0.02	99W14488 M-240409- 01-1
14488-03, Uvalde (TX), USA, 1999 (Cantaloupe Caravelle)	SC	0.2– 0.21	0.14	138– 147	6	15	Fruit	< 0.02 (< 0.02 2×)	< 0.02 (< 0.02, < 0.02)	< 0.02	
14488-04, Brookshire (TX), USA, 1999 (Cantaloupe–TAM Uvalde)	SC	0.2	0.12	166– 172	6	14	Fruit	0.07 (0.07, 0.07)	< 0.02 (< 0.02, < 0.02)	0.07	
14488-05, San Juan Bautista (CA), USA, 1999 (Cantaloupe - Trooper)	SC	0.2	0.12	168– 171	6	13	Fruit	0.06 (0.06, 0.05)	< 0.02 (< 0.02, < 0.02)	0.06	
14488-06, Hamilton (CA), USA, 1999 (Cantaloupe - Spanish Cavaillon)	SC	0.2	0.11– 0.13	150– 178	6	14	Fruit	0.08 (0.10, 0.08)	< 0.02 (< 0.02, < 0.02)	0.08	
14488-07, Porterville (CA), USA, 1999 (Cantaloupe - Durango)	SC	0.19– 0.2	0.13	149– 156	6	14	Fruit	0.08 (0.08, 0.08)	< 0.02 (< 0.02, < 0.02)	0.08	
14488-08, San Marcos (CA), USA, 1999 (Cantaloupe Hy Shaw)	SC	0.2 0.2 0.2 0.295 0.2	0.12– 0.18	166– 172	6	14	Fruit	0.09 (0.09, 0.09)	< 0.02 (< 0.02, < 0.02)	0.09	
14488-09, Fresno (CA), USA, 1999 (Cantaloupe -Hales Best Jumbo)	SC	0.19– 0.2	0.11	183– 188	6	14	Fruit	0.04 (0.03, 0.04)	< 0.02 (< 0.02, < 0.02)	0.04	

Watermelons

Three supervised trials on watermelon were carried out in Brazil during 2004. The foliar spray application rates were 5×0.15 kg ai/ha or 5×0.3 kg ai/ha (interval 7 days). The results are summarized in Table 70.

Table 70 Residues in watermelons from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
GAP, Brazil	SC	0.15			3	7					
FR04BRA012-C1 Anhembi—SP, Brazil, 2004 (Favorita)	SC	0.15	0.015	1000	5	0	Fruit	0.71	0.09	0.81	UNESP RA-944/05 M-276178-01-1
						2		0.25	0.10	0.36	
						5		0.15	0.06	0.22	
						7		0.04	< 0.02	0.04	
FR04BRA012-P1 Uberlandia—MG, Brazil, 2004 (Perola)	SC	0.3	0.03	1000	5	10	Fruit	< 0.02	< 0.02	< 0.02	UNESP RA-945/05 M-276180-01-1
						7		0.07	0.02	0.09	
						7		0.03	< 0.02	0.03	
						7		0.07	< 0.02	0.07	
FR04BRA012-P2 Ribeirao Preto—SP, Brazil, 2004 (Perola)	SC	0.15	0.015	1000	5	7	Fruit	0.05	< 0.02	0.05	UNESP RA-946/05 M-276183-01-1
		0.3	0.03	1000	5	7	Fruit	0.08	0.02	0.10	

*Fruiting Vegetables other than Cucurbits**Peppers*

Six supervised field trials on sweet pepper and three trials on chilli pepper were carried out during 2003 in the USA (201227, M-260625-01-1). At each trial, four foliar spray applications were made at intervals of 4–6 days, at rates in the range of 0.27–0.35 kg ai/ha. The results are summarized in Table 71.

Table 71 Residues in peppers from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg		
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum
GAP, USA	SC	0.3			3	14				
YW041-03H, Tifton (GA), USA, 2003 (Sweet pepper, Keystone Bell)	SC	0.3	0.17–0.19	161–176	4	14	Fruit	0.03 (< 0.02, 0.03)	< 0.02 (< 0.02, < 0.02)	0.03
YW042-03D, Molino (FL), USA, 2003 (Sweet pepper, Olympus)	SC	0.29–0.31	0.14–0.15	199–223	4	0	Fruit	0.17 (0.13, 0.21)	< 0.02 (< 0.02, < 0.02)	0.17
						5		0.13 (0.11, 0.15)	< 0.02 (< 0.02, < 0.02)	0.13
						10		0.11 (0.11, 0.11)	< 0.02 (< 0.02, < 0.02)	0.11
						14		0.05 (0.05, 0.04)	< 0.02 (< 0.02, < 0.02)	0.05
						21		< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02
YW043-03HA, Stillwell (KS), USA, 2003 (Sweet pepper, California Wonder)	SC	0.27–0.35	0.22–0.24	126–156	4	14	Fruit	0.08 (0.08, 0.07)	< 0.02 (< 0.02, < 0.02)	0.08

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg		
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum
YW044-03H, East Bernard (TX), USA, 2003 (Sweet pepper, Better Bell)	SC	0.29–0.3	0.17–0.18	161–178	4	14	Fruit	<u>0.08</u> (0.06, 0.09)	< 0.02 (< 0.02, < 0.02)	0.08
YW045-03H, Fresno (CA), USA, 2003 (Sweet pepper, Emerald Giant Bell)	SC	0.3	0.18–0.2	149–161	4	14	Fruit	<u>0.19</u> (0.18, 0.20)	< 0.02 (< 0.02, < 0.02)	0.19
YW046-03H, Visalia (CA), USA, 2003 (Sweet pepper, Valiant)	SC	0.3	0.14	210–218	4	14	Fruit	<u>0.07</u> (0.08 0.06)	< 0.02 (< 0.02, < 0.02)	0.07
YW047-03H, Jerome (ID), USA, 2003 (Chilli pepper, Jalapeno/Tam)	SC	0.3–0.31	0.11	260–272	4	14	Fruit	<u>0.07</u> (0.07, 0.06)	< 0.02 (< 0.02, < 0.02)	0.07
YW048-03H, Uvalde (TX), USA, 2003 (Chilli pepper, Jalapeno—M)	SC	0.29–0.3	0.14–0.15	196–209	4	13	Fruit	<u>1.45</u> (1.24, 1.66)	0.04 (0.04, 0.04)	1.5
YW049-03H, Fresno (CA), USA, 2003 (Chilli pepper, Anaheim TMR 23)	SC	0.29–0.3	0.18–0.2	150–161	4	14	Fruit	<u>1.3</u> (1.47, 1.16)	< 0.02 (< 0.02, < 0.02)	1.3

Tomato

Eight supervised trials on indoor tomatoes and four trials on outdoor tomatoes were conducted in Europe (France, Italy, Spain, Germany and Netherlands) during 2005, 2009 and 2010. Fenamidone was applied by foliar spray three times at a rate of 0.15 kg ai/ha and an interval in the range of 6–7 days. The maximum storage period from harvest to analysis was 512 days.

Seventeen supervised field trials on tomato, including two trials on cherry tomatoes, were carried out during 1999/2000 in the USA (outdoors). Each trial consisted of two plots. The first plot was treated four times at rates in the range of 0.2–0.31 kg ai/ha and intervals of 4–6 days. The second plot of each trial was treated six times at lower rates in the range of 0.19–0.21 kg ai/ha. The results are summarized in Table 72.

Table 72 Residues in tomatoes from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
GAP, Romania (Indoors)	WG	0.11	0.03	500	3	3					
09-2235-01 Honselersdijk, Netherlands, 2009 (Mecano) (Indoors)	SC	0.13	0.013	1000	3	–0 0 1 3 7	Fruit	0.03 0.05 0.05 0.04 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.03 0.05 0.05 0.04 0.01	09-2235 M-405712-01-1
09-2235-02 Zapponeta, Italy, 2009 (Foce) (Indoors)	SC	0.13	0.013	1000	3	–0 0 1 3 7	Fruit	0.13 0.38 0.16 0.15 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.13 0.38 0.16 0.15 0.08	
09-2235-03 Leichlingen, Germany, 2009 (Albis) (Indoors)	SC	0.13	0.017	750	3	–0 0 1 3 7	Fruit	0.05 0.07 0.08 0.10 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.05 0.07 0.08 0.10 0.05	
09-2235-04 Sta Susanna, Spain, 2009 (Cherry, Phiolin) (Indoors)	SC	0.13–0.14	0.013	1000–1129	3	–0 0 1 3 7	Fruit	0.21 0.28 0.28 0.22 0.15	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.21 0.28 0.28 0.22 0.15	

Trial, Location, Country, Year (Variety)	Application					DAT days	Commo dity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
10-2013-01 Sta Susanna, Spain, 2010 (Caramba) (Indoors)	SC	0.15– 0.16	0.015	1000– 1077	3	–0 0 1 3 7	Fruit	0.05 0.09 0.06 0.06 0.09	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.05 0.09 0.06 0.06 0.09	10-2013 M-419802- 01-1
10-2013-02 Palidoro-Fiumi- cino, Italy, 2010 (Ikram) (Indoors)	SC	0.15	0.015	1000	3	–0 0 1 3 7	Fruit	0.06 0.10 0.11 0.11 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.06 0.10 0.11 0.11 0.07	
10-2013-03 Leichlingen, Germany, 2010 (Albis) (Indoors)	SC	0.15	0.015	1000	3	–0 0 1 3 7	Fruit	0.04 0.06 0.07 0.06 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.04 0.06 0.07 0.06 0.06	
10-2013-04 Zevenhuizen, Netherlands, 2010 (Cherry, Santa) (Indoors)	SC	0.15	0.015	1000	3	–0 0 1 3 7	Fruit	0.29 0.38 0.38 0.30 0.26	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.29 0.38 0.38 0.30 0.26	
GAP, Switzerland (field)	SC	0.15		500	3	3					
R 2005 0307 5 Lebrija Sevilla, Spain, 2005 (Elegy) (Outdoors)	SC	0.15	0.015	1000	3	–0 0 1 3 7 13	Fruit	0.05 0.12 0.11 0.09 0.09 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.05 0.12 0.11 0.09 0.09 0.06	RA-2541/05 M-279022- 01-1
R2005 0918 9 Molfetta, Italy, 2005 (Candia) (Outdoors)	SC	0.15	0.019	800	3	–0 0 1 3 7 14	Fruit	0.10 0.22 0.21 0.18 0.05 0.06	< 0.01 0.01 0.01 0.01 < 0.01 < 0.01	0.10 0.23 0.22 0.19 0.05 0.06	
R2005 0919 7 Foros de Salvaterra, Portugal, 2005 (H9661) (Outdoors)	SC	0.15	0.019	800	3	–0 0 1 3 7 14	Fruit	0.07 0.10 0.10 0.10 0.08 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.07 0.10 0.10 0.10 0.08 0.02	
R2005 0920 0 Thina, Greece, 2005 (H9780) (Outdoors)	SC	0.15	0.021	700	3	–0 0 1 3 7 14	Fruit	0.14 0.28 0.34 0.17 0.18 0.08	< 0.01 0.01 0.01 < 0.01 0.01 < 0.01	0.14 0.29 0.35 0.17 0.19 0.08	
GAP, USA	SC	0.3			3	14					
17533-01 North Rose (NY), USA, 1999 (Cherry tomato, Red cherry large)	SC	0.3– 0.31	0.11	282– 291	4	14	Fruit	0.25 (0.24, 0.25)	< 0.02 (< 0.02, < 0.02)	0.25	99W17533 M-238684- 01-1
		0.2	0.072	275– 283	6	14	Fruit	0.13 (0.12, 0.13)	< 0.02 (< 0.02, < 0.02)	0.13	
17533-02 Clayton (NC), USA, 1999 (Cherry tomato, Sweetie)	SC	0.21– 0.31	0.08– 0.12	250– 262	4	14	Fruit	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
		0.2– 0.21	0.08– 0.09	246– 260	6	14	Fruit	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	

Fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference	
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum		
17533-04 Lake Jem (FL), USA, 1999 (Tomato, Super sweet 100VF)	SC	0.27–0.3	0.06–0.14	374–457	4	14	Fruit	<u>0.42</u> (0.56, 0.28)	0.03 (0.03, < 0.02)	0.45		
		0.2	0.045–0.09	384–450	6	15	Fruit	0.37 (0.45, 0.29)	0.03 (0.03, 0.02)	0.40		
17533-05 Oviedo (FL), USA, 1999 (Tomato, Better Boy)	SC	0.3–0.31	0.1	285–298	4	14	Fruit	<u>< 0.02</u> (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02		
		0.2–0.21	0.07	274–375	6	14	Fruit	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02		
17533-06 New Holland (OH), USA, 1999 (Tomato, Heinz H9423)	SC	0.29–0.3	0.14–0.15	193–211	4	14	Fruit	<u>0.09</u> (0.09, 0.09)	< 0.02 (< 0.02, < 0.02)	0.09		
		0.2	0.09–0.1	193–224	6	14	Fruit	0.14 (0.16, 0.11)	< 0.02 (< 0.02, < 0.02)	0.14		
17533-07 San Juan Bautista (CA), USA, 1999 (Tomato, Brigade)	SC	0.3	0.12	255–259	4	14	Fruit	<u>0.33</u> (0.35, 0.30)	< 0.02 (< 0.02, < 0.02)	0.33		
		0.2	0.08	255–259	6	14	Fruit	0.21 (0.22, 0.20)	< 0.02 (< 0.02, < 0.02)	0.21		
17533-08 Medera (CA), USA, 1999 (Tomato, Shady Lady)	SC	0.3	0.11	273–283	4	7	Fruit	0.24 (0.26, 0.22)	< 0.02 (< 0.02, < 0.02)	0.24		
						14		<u>0.10</u> (0.13, 0.07)	< 0.02 (< 0.02, < 0.02)			
						21		0.06 (0.07, 0.05)	< 0.02 (< 0.02, < 0.02)			
						29		< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)			
						35		< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)			
		0.2	0.07–0.08	260–283	6	7	Fruit	0.20 (0.27, 0.13)	< 0.02 (< 0.02, < 0.02)	0.20		
						14		0.14 (0.13, 0.14)	< 0.02 (< 0.02, < 0.02)			
						21		0.03 (< 0.02, 0.04)	< 0.02 (< 0.02, < 0.02)			
						29		0.03 (0.03, < 0.02)	< 0.02 (< 0.02, < 0.02)			
						35		< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)			
						7		0.20 (0.27, 0.13)	< 0.02 (< 0.02, < 0.02)			0.20
						14		0.14 (0.13, 0.14)	< 0.02 (< 0.02, < 0.02)			
						21		0.03 (< 0.02, 0.04)	< 0.02 (< 0.02, < 0.02)			
						29		0.03 (0.03, < 0.02)	< 0.02 (< 0.02, < 0.02)			
						35		< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)			

Trial, Location, Country, Year (Variety)	Application					DAT days	Commo dity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
17533-09 Glenn (CA), USA, 1999 (Tomato, Heinz 8892)	SC	0.3– 0.31	0.11– 0.13	247– 275	4	7	Fruit	0.36 (0.35, 0.37)	< 0.02 (< 0.02, < 0.02)	0.36	
						14		<u>0.47</u> (0.45, 0.48)	< 0.02 (< 0.02, < 0.02)	<u>0.47</u>	
						21		0.32 (0.28, 0.36)	< 0.02 (< 0.02, < 0.02)	0.32	
						28		0.24 (0.21, 0.26)	< 0.02 (< 0.02, < 0.02)	0.24	
						35		0.22 (0.25, 0.18)	< 0.02 (< 0.02, < 0.02)	0.22	
		0.2	0.07– 0.08	245– 277	6	7	Fruit	0.40 (0.39, 0.40)	< 0.02 (< 0.02, < 0.02)	0.40	
						14		0.42 (0.39, 0.44)	< 0.02 (< 0.02, < 0.02)	0.42	
						21		0.41 (0.40, 0.41)	< 0.02 (< 0.02, < 0.02)	0.41	
						28		0.25 (0.27, 0.22)	< 0.02 (< 0.02, < 0.02)	0.25	
						35		0.18 (0.22, 0.13)	< 0.02 (< 0.02, < 0.02)	0.18	
17533-10, Delevan (CA), USA, 1999 (Tomato, Sun 6200)	SC	0.3	0.11– 0.12	249– 262	4	14	Fruit	<u>0.40</u> (0.40, 0.39)	< 0.02 (< 0.02, < 0.02)	0.40	
		0.2	0.08– 0.09	233– 262	6	14	Fruit	0.38 (0.39, 0.37)	< 0.02 (< 0.02, < 0.02)	0.38	
17533-11, Fresno (CA), USA, 1999 (Tomato, Casa del Sol)	SC	0.29– 0.3	0.16	182– 191	4	14	Fruit	<u>0.34</u> (0.36, 0.31)	< 0.02 (< 0.02, < 0.02)	0.34	
		0.2– 0.21	0.11	184– 194	6	14	Fruit	0.25 (0.27, 0.23)	< 0.02 (< 0.02, < 0.02)	0.25	
17533-12, Fresno (CA), USA, 1999 (Tomato, Shady Lady)	SC	0.3	0.16	185– 188	4	14	Fruit	<u>0.11</u> (0.09, 0.13)	< 0.02 (< 0.02, < 0.02)	0.11	
		0.19– 0.2	0.11	183– 190	6	14	Fruit	0.14 (0.13, 0.15)	< 0.02 (< 0.02, < 0.02)	0.14	
17533-13 Fresno (CA), 1999 (Tomato, Sweet Chelsea)	SC	0.3	0.16	184– 189	4	14	Fruit	<u>0.61</u> (0.68, 0.53)	< 0.02 (< 0.02, < 0.02)	0.61	
		0.19– 0.21	0.11	183– 193	6	14	Fruit	0.56 (0.48, 0.64)	< 0.02 (< 0.02, < 0.02)	0.56	
17533-14 Manteca (CA), 1999 (Tomato, HP 108)	SC	0.29– 0.3	0.07	417– 426	4	15	Fruit	<u>0.46</u> (0.46, 0.45)	< 0.02 (< 0.02, < 0.02)	0.46	
		0.19– 0.2	0.045	414– 424	6	15	Fruit	0.50 (0.53, 0.46)	< 0.02 (< 0.02, < 0.02)	0.50	

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
17533-15 Lathrop (CA), USA, 1999 (Tomato, HP 108)	SC	0.29–0.3	0.07	416–426	4	15	Fruit	<u>0.38</u> (0.36, 0.40)	< 0.02 (< 0.02, < 0.02)	0.38	
		0.19–0.2	0.05	410–428	6	15	Fruit	0.29 (0.29, 0.28)	< 0.02 (< 0.02, < 0.02)	0.29	
17533-16 San Marcos (CA), USA, 1999 (Tomato, Red Pear)	SC	0.29–0.3	0.08	366–374	4	13	Fruit	<u>0.80</u> (0.82, 0.77)	< 0.02 (< 0.02, < 0.02)	0.80	
		0.19–0.2	0.05–0.07	274–375	6	13	Fruit	0.64 (0.78, 0.49)	< 0.02 (< 0.02, < 0.02)	0.64	
17533-17 Watsonville (CA), USA, 1999 (Tomato, Sunbelt)	SC	0.3–0.31	0.07	426–436	4	14	Fruit	<u>0.07</u> (0.06, 0.07)	< 0.02 (< 0.02, < 0.02)	0.07	
		0.2	0.05–0.06	314–430	6	14	Fruit	0.08 (0.08, 0.08)	< 0.02 (< 0.02, < 0.02)	0.08	
17533-18 Pikeville (NC), USA, 2000 (Tomato, Celebrity)	SC	0.3–0.31	0.09	327–342	4	14	Fruit	<u>0.07</u> (0.07, 0.07)	< 0.02 (< 0.02, < 0.02)	0.07	
		0.2–0.21	0.06	321–339	6	14	Fruit	0.05 (0.05, 0.04)	< 0.02 (< 0.02, < 0.02)	0.05	

Leafy vegetables

Lettuce

Supervised trials on indoor lettuce (19) and eighteen trials on field lettuce (18) were conducted in Europe (France, Germany, Belgium, Spain and Netherlands) between 1998 and 2010. Fenamidone was applied three times by foliar spray at a rate in the range of 0.11–0.16 kg ai/ha and an interval in the range of 7–47 days. In the 2004 trials (RA-2542/04 (M-265329-01-1), the maximum storage period from harvest to analysis was 17.5 month.

Eighteen field trials on lettuce were conducted during the year 2000 in the USA (outdoors), nine on open-leaf and nine on head varieties. Fenamidone was applied four times at 7-day intervals to lettuce plants at the rate of about 0.3 kg ai/ha (range: 0.29–0.34 kg ai/ha). The results are shown in Table 73.

Table 73 Residues in lettuce from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
GAP, Netherlands (indoor)	WG	0.15		500–1000	3	21					
98515OR1 Ouvrouer les champs (45) France 1998 Colette	WG	0.13	0.017	770	3	22	Head	0.17	< 0.02	0.17	98-515 M-165916-01-1
98515TL1 Ricaud (11) France 1998 Sensai	WG	0.13	0.016–0.032	400–800	3	21	Head	0.33	< 0.02	0.33	98-515 M-165916-01-1

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodit y	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
98516OR1 St Denis en Val (45) France 1998 Colette	WG	0.13	0.026	500	3	0 7 13 20	Head	2.1 1.2 0.67 0.47	0.029 0.031 < 0.02 < 0.02	2.1 1.2 0.67 0.47	98-516 M- 165926- 01-1
98751AV1 Noves (13) France 1998 Sensai	WG	0.13	0.014– 0.017	770–920	3	0 6 13 20	Head	1.5 1.1 0.75 0.70	< 0.02 < 0.02 < 0.02 < 0.02	1.5 1.1 0.75 0.70	98-751 M- 166446- 01-1
99504OR2 Ouvrouer les champs (45) France 1999 Colette	WG	0.15	0.026	600	3	21	Head	0.26	< 0.02	0.26	99-504 M- 233244- 01-1
99504TL1 Nohic (82) France 1998 Sensai	WG	0.13	0.027	480	3	18	Head	0.08	< 0.02	0.08	99-504 M- 233244- 01-1
99505AV1 Noves (13) France 1999 Nalys	WG	0.13	0.017	760	3	0 8 15 22	Head	1.1 0.34 0.17 0.13	< 0.02 < 0.02 < 0.02 < 0.02	1.1 0.34 0.17 0.13	99-505 M- 212029- 01-1
99505OR1 St. Denis en Val (45) France 1999 Colette	WG	0.13	0.027	500	3	0 7 15 22	Head	1.4 0.83 0.44 0.35	0.021 < 0.02 < 0.02 < 0.02	1.4 0.83 0.44 0.35	99-505 M- 212029- 01-1
00531RN1 Romille (35) France 2000 Sensai	WG	0.12	0.025	470–500	3	0 8 16 23	Head	1.0 0.82 0.77 0.13	< 0.02 < 0.02 < 0.02 < 0.02	1.0 0.82 0.77 0.13	00-531 M- 201397- 01-1
00533RN1 St Coulomb (35) France 2000 Angie	WG	0.13	0.025	500–540	3	21	Head	0.31	< 0.02	0.31	00-533 M- 201446- 01-1
R 2005 0796/8 Chazay D'Azergues (69), France, 2005 (head variety: Access)	WG	0.15	0.03	500	3	–0 0 3 7 14 21	Head	1.5 6.0 2.2 0.85 0.16 < 0.01	0.06 0.39 0.11 0.04 0.01 < 0.01	1.6 6.4 2.3 0.89 0.17 < 0.01	RA- 2518/05 M- 277879- 01-1
R 2006 0279/0 Leichlingen, Germany, 2006 (head variety: Alexandria)	WG	0.15	0.03	500	3	–0 0 7 14 21	Head	2.3 6.4 4.7 2.3 1.6	0.16 0.54 0.35 0.15 0.10	2.5 7.0 5.1 2.5 1.7	RA- 2553/06 M- 281157- 01-1
R 2006 0280/4 Villers–Perwin, Belgium, 2006 (head variety: Pullman)	WG	0.15	0.03	500	3	–0 0 7 14 21	Head	0.60 8.8 0.55 0.12 0.01	0.04 1.1 0.04 < 0.01 < 0.01	0.64 10 0.59 0.12 0.01	
09-2159-01 Meckenbeuren/Liebe nau, Germany, 2009 (head variety: Analena)	WG	0.15	0.038	400	3	0 20	Head	6.9 0.02	0.56 < 0.01	7.5 0.02	09-2159 M- 404587- 01-1
09-2159-02, Lucenay (69), France, 2009 (leaf variety: Matinale)	WG	0.15	0.025	600	3	0 21	Leaves	6.2 0.16	0.63 0.02	6.9 0.18	

Fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
09-2159-03, Bigues i Riells, Spain, 2009 (leaf variety: Batavia)	WG	0.15–0.16	0.03–0.038	400–530	3	0 20	Leaves	10 0.10	1.7 0.01	12 0.11	
09-2159-04, Palidoro Fiumicino, Italy, 2009 (leaf variety: Antony)	WG	0.15	0.03	500	3	0 21	Leaves	13 2.1	2.0 0.45	15 2.6	
09-2159-05, Wervers-hoof, Netherlands, 2009 (head lettuce)	WG	0.15	0.025	600	3	0 21	Head	5.8 0.12	0.55 0.02	6.4 0.14	
09-2159-06, Villers-Perwin, Belgium, 2009 (head variety: Tanex)	WG	0.15	0.03	500	3	0 21	Head	6.8 < 0.01	1.1 < 0.01	8.0 < 0.01	
GAP, Switzerland (field)	SC	0.15			3	14					
98514RN1 Domloup (35), France, 1998 (leaf variety: Enrika)	WG	0.15	0.022	577–626	3	0 7 14 22	Leaves	1.3 0.05 <u>< 0.02</u> < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	1.3 0.05 <u>< 0.02</u> < 0.02	98-514 M- 165908- 01-1
98514AV1 Meynes (30), France, 1998 (Grise de Serre)	WG	0.13	0.026	500	3	0 7 13 21	Leaves	0.95 0.29 <u>0.07</u> 0.03	< 0.02 < 0.02 < 0.02 < 0.02	0.95 0.29 <u>0.07</u> 0.03	
99506AM1 Mezerolles (80), France, 1999 (leaf variety: Einstein)	WG	0.13	0.029	442	3	0 8 15 21	Leaves	0.90 0.07 <u>< 0.02</u> < 0.02	0.03 < 0.02 < 0.02 < 0.02	0.93 0.07 <u>< 0.02</u> < 0.02	99-506 M- 212036- 01-1
99506RN1 Romille (35), France, 1999 (Nadine)	WG	0.11–0.14	0.021	505–645	3	0 7 14 21	Leaves	2.8 1.3 <u>< 0.02</u> < 0.02	0.04 0.04 < 0.02 < 0.02	3.2 1.7 <u>< 0.02</u> < 0.02	
R 2004 0371/2 Cambridge, UK, 2004 (head variety: Anthem)	SC	0.15	0.038	400	3	0 3 7 14	Head	3.7 4.6 1.3 0.19	0.08 0.22 0.06 0.01	3.8 4.8 1.4 0.20	RA- 2542/04 M- 265329- 01-1
R 2004 0372/0 Fondettes (37), France, 2004 (head variety: Ponchito)	SC	0.15	0.038	400	3	0 3 7 14	Head	4.4 1.4 0.09 0.03	0.11 0.04 < 0.01 < 0.01	4.5 1.4 0.09 0.03	
R 2004 0373/9 Langenfeld, Germany, 2004 (head variety: Ponchito)	SC	0.15	0.038	400	3	0 3 7 14	Head	2.5 0.64 0.33 0.03	0.10 0.02 0.02 < 0.01	2.6 0.66 0.35 0.03	
R 2004 0374/7 Zwaagdijk-Oost, Netherlands, 2004 (head variety: Ponchito, Butterhead)	SC	0.15	0.038	400	3	0 3 7 14	Head	1.9 2.1 1.1 0.41	0.07 0.08 0.04 0.02	2.0 2.2 1.1 0.43	

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodit y	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
R 2005 0797/6 Fondettes (37), France, 2005 (head variety: Estelle)	WG	0.15	0.03	500	3	–0 0 3 7 14 21	Head	0.03 4.2 2.1 0.09 < 0.01 < 0.01	< 0.01 0.38 0.13 < 0.01 < 0.01 < 0.01	0.03 4.6 2.2 0.09 < 0.01 < 0.01	RA- 2519/05 M- 277880- 01-1
R 2005 0798/4 Langenfeld- Reusrath, Germany, 2005 (head variety: Ponchito)	WG	0.15	0.03	500	3	–0 0 3 7 14 21	Head	0.38 4.0 0.67 0.38 0.07 < 0.01	0.03 0.26 0.04 0.03 < 0.01 < 0.01	0.41 4.3 0.71 0.41 0.07 < 0.01	
R 2005 0799/2 Schauernheim, Germany, 2005 (head variety: Naima)	WG	0.15	0.03	500	3	–0 0 3 7 14 21	Head	< 0.01 3.9 1.4 0.24 0.04 < 0.01	< 0.01 0.28 0.08 0.02 < 0.01 < 0.01	< 0.01 4.2 1.5 0.26 0.04 < 0.01	
09-2158-01 Langenfeld- Reusrath, Germany, 2009 (leaf variety: Quintus)	WG	0.15	0.025	600	3	0 14	Leaves	2.1 <u>0.06</u>	0.04 < 0.01	2.1 <u>0.06</u>	09-2158 M- 404370- 01-1
09-2158-02, Cergy (95), France, 2009 (leaf variety: Quenty)	WG	0.15	0.038	400	3	0 14	Leaves	3.8 <u>0.04</u>	0.08 < 0.01	3.9 <u>0.04</u>	
09-2216-01, Manfre- donia, Italy, 2009 (head variety: Iceberg)	SC	0.13	0.016	800	3	0 14	Head	0.25 < 0.01	< 0.01 < 0.01	0.25 < 0.01	09-2216 M- 405062- 01-1
09-2216-03, Scorbe- Clairvaux, France, 2009 (leaf variety: 9539 bio)	SC	0.13	0.025	500	3	0 14	Leaves	11 <u>0.42</u>	0.42 0.01	12 <u>0.43</u>	
09-2216-04, Vilanova del Vallés, Spain, 2009 (head variety: Dorada de Primavera)	SC	0.13– 0.14	0.025– 0.03	429–500	3	0 13	Head	2.6 0.04	0.14 < 0.01	2.8 0.04	
10-2012-01, Gava, Spain, 2010 (leaf variety: Murai)	SC	0.15	0.025	600	3	0 14	Leaves	6.5 <u>0.48</u>	0.01 < 0.01	6.5 <u>0.48</u>	10-2012 M- 418546- 01-1
10-2012-02, Bologna, Italy, 2010 (leaf variety: Perla)	SC	0.15	0.019	800	3	0 14	Leaves	3.3 <u>0.02</u>	0.01 < 0.01	3.3 <u>0.02</u>	
GAP, USA	SC	0.3			3	2					
13043-01, Germansville (PA), USA, 2000 (leaf variety: Slobolt MTO)	SC	0.31– 0.315	0.18	169–175	4	2	Leaves	15.9 (17.5, 14.3)	0.03 (0.03, 0.03)	16	00W13043 M- 238774- 01-1
13043-02, Oviedo (FL), USA, 2000 (leaf variety: Bibb)	SC	0.3	0.18	167–169	4	2	Leaves	11.8 (15.4, 8.15)	0.03 (0.03, 0.02)	12	

Fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
13043-03, Modesto (CA), USA, 2000 (leaf variety: Mardi Gras)	SC	0.3	0.22	138–144	4	0 2 4 6 8	Leaves	8.40 1.00 (1.02, 0.97) 0.44 0.21 0.04	< 0.02 < 0.02 (< 0.02, 0.02) < 0.02 < 0.02 < 0.02	8.4 1.0 0.44 0.21 0.04	
13043-04, Fresno (CA), USA, 2000 (leaf variety: Waldman's Green)	SC	0.3– 0.31	0.21	138–144	4	2	Leaves	3.41 (3.70, 3.12)	< 0.02 (< 0.02, 0.02)	3.4	
13043-05, Watsonville (CA), USA, 2000 (leaf variety: Athena)	SC	0.3– 0.31	0.18	166–171	4	2	Leaves	7.94 (8.26, 7.61)	0.02 (0.02, < 0.02)	8.0	
13043-06, Watsonville (CA), USA, 2000 (leaf variety: Darkland Romaine)	SC	0.3– 0.34	0.21	139–156	4	2	Leaves	10.1 (11.9, 8.23)	0.03 (0.03, 0.02)	10	
13043-07, Sprekels (CA), USA, 2000 (leaf variety: Shining Star)	SC	0.3– 0.31	0.22	140–144	4	2	Leaves	3.40 (2.97, 3.82)	< 0.02 (< 0.02, 0.02)	3.4	
13043-08, Santa Maria (CA), USA, 2000 (leaf variety: Betteravia 145 Romaine)	SC	0.3– 0.31	0.21	141–147	4	2	Leaves	2.58	< 0.02 (< 0.02, 0.02)	2.6	
13043-09, Maricopa (AZ), USA, 2000 (leaf variety: Paris Island)	SC	0.29– 0.3	0.16	179–184	4	2	Leaves	6.49 (5.18, 7.79)	0.04 (0.03, 0.05)	6.5	
14459-01 Germansville (PA), USA, 2000 (head variety: Ithaca)	SC	0.3– 0.31	0.18	172–175	4	2 2	Head with wrapper Head w/o wrapper	0.82 0.03	< 0.02 < 0.02	0.82 0.03	00W14459 M- 238776- 01-1
14459-02 Oviedo (FL), USA, 2000 (head variety: Great Lakes)	SC	0.3– 0.31	0.18	167–170	4	2 2	Head with wrapper Head w/o wrapper	<u>10.7</u> (9.73, 11.7) 2.7 (2.90, 2.40)	0.03 (0.03, 0.03) < 0.02 (< 0.02, 0.02)	11 2.7	
14459-03 Hickman (CA), USA, 2000 (head variety: Cowboy)	SC	0.3	0.21	138–142	4	2 2	Head with wrapper Head w/o wrapper	<u>4.44</u> (4.77, 4.10) 0.26 ^a (0.26, 0.04)	0.025 (< 0.02,0.0 3) < 0.02 (< 0.02, 0.02)	4.5 0.26	
14459-04 Fresno (CA), USA, 2000 (head variety: Empire M.I.)	SC	0.3– 0.31	0.21	140–142	4	2 2	Head with wrapper Head w/o wrapper	<u>2.28</u> (2.45, 2.11) 0.04 (0.04, 0.04)	< 0.02 (< 0.02, 0.02) < 0.02 (< 0.02, 0.02)	2.3 0.04	

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
14459-05 Watsonville (CA), USA, 2000 (head variety: Salinas)	SC	0.3	0.21	138–142	4	2	Head with wrapper	<u>3.30</u> (2.50, 4.10)	0.02 (< 0.02 , 0.02)	3.3	
						2	Head w/o wrapper	0.03 (0.04, < 0.02)	< 0.02 (< 0.02 , < 0.02)	0.03	
14459-06 Watsonville (CA), USA, 2000 (head variety: Prodigy)	SC	0.3–0.31	0.18	168–173	4	2	Head with wrapper	<u>3.9</u> (3.71, 4.12)	< 0.02 (< 0.02 , < 0.02)	3.9	
						2	Head w/o wrapper	0.22 (0.24, 0.19)	< 0.02 (< 0.02 , < 0.02)	0.22	
14459-07 Spreckels (CA), USA, 2000 (head variety: Progeny)	SC	0.3–0.31	0.21–0.22	139–143	4	2	Head with wrapper	<u>3.7^a</u> (0.70, 3.67)	< 0.02 (< 0.02 , < 0.02)	3.7	
						2	Head w/o wrapper	4.0 ^a (< 0.02 , 4.02)	< 0.02 (< 0.02 , < 0.02)	4.0	
14459-08 San Luis Obispo (CA), USA, 2000 (head variety: Bayview)	SC	0.3–0.34	0.22	140–160	4	2	Head with wrapper	<u>3.3</u> (3.91, 2.65)	< 0.02 (< 0.02 , < 0.02)	3.3	
						2	Head w/o wrapper	< 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02	
14459-09 Maricopa (AZ), USA, 2000 (head variety: Green Lightning)	SC	0.3	0.16	181–186	4	2	Head with wrapper	<u>8.0</u> (7.65, 8.36)	0.03 (0.02, 0.03)	8.0	
						2	Head w/o wrapper	1.4 (1.89, 0.86)	< 0.02 (< 0.02 , < 0.02)	1.4	

^a Highest value selected because of the large divergence between duplicate samples.

Mustard greens

Eight supervised trials on mustard greens were carried out during 2003 in the USA (outdoors) (201226, M-260567-01-1). At each trial, four foliar sprays were applied at intervals of 4–5 days, at rates in the range of 0.29–0.32 kg ai/ha. The results are summarized in Table 74.

Table 74 Residues in mustard greens of supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg		
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum
GAP, USA	SC	0.3			3	2				
YW033-03D Tifton (GA), USA, 2003 (Broadleaf Mustard)	SC	0.3	0.17–0.19	161–175	4	0	Green leaf	21 (20.7, 21.5)	0.04 (0.03, 0.05)	21
						2		<u>17</u> (17.3, 16.4)	0.04 (0.04, 0.04)	<u>17</u>
						3		6.1 (6.58, 5.66)	0.03 (0.03, 0.03)	6.2
						5		3.2 (2.92, 3.49)	0.03 (0.03, 0.02)	3.2
						7		2.7 (2.65, 2.82)	0.02 (0.02, 0.02)	2.8
YW034-03H, Suffolk (VA), USA, 2003 (Savannah)	SC	0.3–0.31	0.14–0.15	204–219	4	2	Green leaf	<u>28</u> (28.5, 28.2)	0.04 (0.03, 0.04)	28
YW035-03H Molino (FL), USA, 2003 (Giant Southern Curled)	SC	0.3–0.32	0.14	203–223	4	2	Green leaf	<u>13</u> (9.85, 15.2)	0.02 (0.02, 0.02)	13

Fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg		
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum
YW036-03H, Newport (AR), USA, 2003 (Florida Broadleaf)	SC	0.3	0.16	186–190	4	2	Green leaf	<u>29</u> (31.5, 26.7)	0.04 (0.04, 0.04)	29
YW037-03H, Seymour (IL), USA, 2003 (Green Wave)	SC	0.3–0.31	0.17	176–178	4	2	Green leaf	<u>24</u> (25.1, 23.5)	0.05 (0.04, 0.05)	24
YW038-03H Colony (OK), USA, 2003 (Florida Broadleaf)	SC	0.3–0.31	0.21–0.22	141–143	4	2	Green leaf	<u>28</u> (24.2, 32.4)	0.21 (0.18, 0.23)	29
YW039-03H, Fresno (CA), USA, 2003 (Florida Broadleaf)	SC	0.3	0.16–0.17	172–182	4	2	Green leaf	<u>11</u> (9.33, 13.3)	0.06 (0.05, 0.06)	11
YW040-03H, Porterville (CA), USA, 2003 (Florida Broadleaf)	SC	0.3–0.31	0.13–0.14	214–239	4	2	Green leaf	<u>12</u> (11.8, 11.8)	0.07 (0.07, 0.06)	12

Spinach

Six supervised trials on spinach were carried out during 2003 in the USA (201214, M-242541-01-1). At each trial, four foliar sprays were made at intervals of 3–6 days, at rates in the range of 0.3 kg ai/ha. The results are summarized in Table 75.

Table 75 Residues in spinach from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg		
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum
GAP, USA	SC	0.3			3	2				
YW050-03HA, Ruther Glen (VA), USA, 2003 (Melody)	SC	0.3–0.31	0.17	170–174	4	2	Leaves	<u>7.2</u> (7.44, 7.00)	0.02 (< 0.02 , 0.02)	7.2
YW051-03D Molino (FL), USA, 2003 (Bloomsdale Long Standing)	SC	0.3–0.31	0.14	209–223	4	0	Leaves	15 (15.8, 13.7)	0.03 (0.02, 0.03)	15
						2		<u>7.3</u> (8.62, 6.03)	0.02 (0.02, 0.01)	<u>7.4</u>
						3		5.9 (6.49, 5.33)	0.02 (0.02, 0.02)	5.9
						5		5.1 (5.17, 5.02)	0.02 (0.02, 0.02)	5.1
						7		4.3 (4.64, 3.90)	0.02 (0.02, 0.02)	4.3
YW052-03H, Raymondville (TX), USA, 2003 (612)	SC	0.3–0.31	0.16	188–192	4	2	Leaves	<u>31</u> (28.7, 32.4)	0.04 (0.03, 0.04)	31
YW053-03H, Jerome (ID), USA, 2003 (Unipack 151)	SC	0.3	0.11–0.12	259–270	4	2	Leaves	<u>11</u> (9.84, 12.5)	< 0.02 (< 0.02 , < 0.02)	11
YW054-03H, Fresno (CA), USA, 2003 (SI-70RZ)	SC	0.3–0.31	0.16–0.17	172–183	4	2	Leaves	<u>23</u> (23.9, 22.4)	0.03 (0.03, 0.03)	23
YW055-03H, Porterville (CA), USA, 2003 (Shasta)	SC	0.3–0.31	0.13	235–242	4	2	Leaves	<u>21</u> (21.0, 20.1)	0.02 (0.02, 0.02)	21

*Legume vegetables**Lima beans*

Eight field trials on lima beans were conducted in the USA (09530, M-489331-01-1). The results are shown in Table 76. At each trial four foliar applications of 0.3 kg ai/ha each plus adjuvant (sticker/spreader) were made. At the NC30 trial, a 5th application was made due to the slow maturation of the lima beans. The applications were timed so that commercially mature succulent lima bean seeds

without pods could be sampled 3–4 days after the final application (at all three Maryland trials the plants with pods on them were harvested 2 days after the final application due to a forecasted heavy rain event). The beans were harvested by hand (either by pulling the entire plant or by handpicking pods in the field). The succulent lima bean seeds were then removed from the pods either by hand or by using a pea sheller.

Table 76 Residues in lima beans from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application			DAT, days	Commodity	Residues, mg/kg		
	Form	kg ai/ha	No			Fen	RPA 410193	Sum
GAP, USA	SC	0.3	3	3				
CA63, Davis, CA, USA, 2008 (LUNA baby)	SC	0.3	4	3	Seeds w/o pods	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02
CA64, Parlier, CA, USA, 2008 (Lee)	SC	0.3	4	3	Seeds w/o pods	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02
ID10, Kimberly, ID, USA, 2008 (M15)	SC	0.3	4	4	Seeds w/o pods	0.08 (0.07, 0.08)	< 0.02 (< 0.02, < 0.02)	0.08
MD09, Salisbury, MD, USA, 2008 (Eastland)	SC	0.3	4	2	Seeds w/o pods	0.04 (0.04, 0.03)	< 0.02 (< 0.02, < 0.02)	0.04
MD10, Salisbury, MD, USA, 2008 (Thorogreen)	SC	0.3	4	2	Seeds w/o pods	0.08 (0.08, 0.07)	< 0.02 (< 0.02, < 0.02)	0.08
MD24, Salisbury, MD, USA, 2008 (Burpee)	SC	0.3	4	2	Seeds w/o pods	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02
NC30, Clinton, NC, USA, 2008 (Fordhook 242)	SC	0.3	5	4	Seeds w/o pods	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02
TN10, Crossville, TN, USA, 2008 (Fordhook 242)	SC	0.3	4	3	Seeds w/o pods	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02
WI07, Arlington, WI, USA, 2008 (Imperial Kingston)	SC	0.3	4	3	Seeds w/o pods	0.03 (0.02, 0.04)	< 0.02 (< 0.02, < 0.02)	0.03

Common beans

Eight field trials on common beans (snap beans) were conducted in the USA and Canada (08895, M-489329-01-1). The results are shown in Table 77. At all trials, four foliar applications of the test substance were made 6–8 days apart at a rate of approximately 0.3 kg ai/ha (for a total application of 1.2 kg ai/ha). An adjuvant was included in each application. At trial CA86 a fifth foliar application of 0.3 kg ai/ha was made, due to slow maturation of the pods at that site. The applications were timed so that mature snap bean pods with seed could be collected 3–4 days after the final application. Samples were harvested by hand or using clippers/pruners.

Table 77 Residues in common beans from trials with fenamidone

Trial, Location, Country, Year (Variety)	Application			DAT, days	Commodity	Residues, mg/kg		
	Form	kg ai/ha	No			Fen	RPA 410193	Sum
GAP, USA (proposed)	SC	0.3	3	3				
CA86, Holtville, CA, USA, 2008 (Veronica)	SC	0.3	5	3	Pods with seed	1.3 (1.07, 1.52)	< 0.02 (< 0.02, < 0.02)	1.3
FL07, Citra, FL, USA, 2008 (Dusky)	SC	0.3	4	4	Pods with seed	0.11 (0.087, 0.13)	< 0.02 (< 0.02, < 0.02)	0.11
MI08, Holt, MI, USA, 2008 (Ulysses)	SC	0.3	4	3	Pods with seed	0.23 (0.22, 0.23)	< 0.02 (< 0.02, < 0.02)	0.23

Fenamidone

Trial, Location, Country, Year (Variety)	Application			DAT, days	Commodity	Residues, mg/kg		
	Form	kg ai/ha	No			Fen	RPA 410193	Sum
NC21, Clinton, NC, USA, 2008 (Ambra)	SC	0.3	4	3	Pods with seed	0.46 (0.45, 0.46)	< 0.02 (< 0.02, < 0.02)	0.46
NY15, Freeville, NY, USA, 2008 (Hystyle)	SC	0.3	4	3	Pods with seed	0.16 (0.16, 0.15)	< 0.02 (< 0.02, < 0.02)	0.16
WA16, Prosser, WA, USA, 2008 (OSU 91G)	SC	0.3	4	3	Pods with seed	0.097 (0.11, 0.084)	< 0.02 (< 0.02, < 0.02)	0.10
WI03, Arlington, WI, USA, 2008 (Hystyle)	SC	0.3	4	3	Pods with seed	0.34 (0.31, 0.37)	< 0.02 (< 0.02, < 0.02)	0.34
WI04, Arlington, WI, USA, 2008 (Hercules)	SC	0.3	4	1	Pods with seed	0.2 (0.20, 0.20)	< 0.02 (< 0.02, < 0.02)	0.20
				3		0.19 (0.18, 0.20)	< 0.02 (< 0.02, < 0.02)	0.19
				7		0.07 (0.071, 0.068)	< 0.02 (< 0.02, < 0.02)	0.07
				13		0.057 (0.059, 0.054)	< 0.02 (< 0.02, < 0.02)	0.06

Root and tuber vegetables

Carrots

Thirteen supervised trials on carrots were carried out in the USA and Canada (08524, M-278032-01-1). At each trial, four foliar spray applications were made at rates in the range of 0.26–0.33 kg ai/ha. Applications were performed at intervals of 6–8 days between the first and second and the third and fourth treatments. There was an extended interval (28 to 104 days) between the second and the third applications. The residue results are summarized in Table 78.

Table 78 Residues in carrots from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT, days	Commodity	Residues, mg/kg		
	Form	kg ai/ha	kg ai/hL	Water, L/ha	No			Fen	RPA 410193	Sum
GAP, USA	SC	0.3			3	14				
08524.03-CA03, Salinas, USA (CA), 2003 (Growers Choice)	SC	0.3	0.11–0.12	247–276	4	14	Roots	<u>0.03</u> (0.03, 0.03)	< 0.02 (< 0.02, < 0.02)	0.03
08524.03-CA04, Holtville, USA (CA), 2003 (Tasty Peel)	SC	0.3–0.31	0.13–0.16	185–232	4	14	Roots	<u>0.02</u> (0.02, 0.02)	< 0.02 (< 0.02, < 0.02)	0.02
08524.03-CA05, Holtville, USA (CA), 2003 (Caropak)	SC	0.3–0.31	0.13–0.16	189–231	4	14	Roots	<u>0.03</u> (0.02, 0.04)	< 0.02 (< 0.02, < 0.02)	0.03

Trial, Location, Country, Year (Variety)	Application					DAT, days	Commodity	Residues, mg/kg		
	Form	kg ai/ha	kg ai/hL	Water, L/ha	No			Fen	RPA 410193	Sum
08524.03-CA157, Parlier, USA (CA), 2003 (Danver's Half Long 126)	SC	0.3– 0.31	0.13	232 – 242	4	3	Roots	0.03 (0.03, 0.03)	< 0.02 (< 0.02, < 0.02)	0.03
						7		0.07 (0.07, 0.06)	< 0.02 (< 0.02, < 0.02)	0.07
						13		<u>0.11</u> (0.10, 0.11)	< 0.02 (< 0.02, < 0.02)	<u>0.11</u>
						20		0.09 (0.08, 0.10)	< 0.02 (< 0.02, < 0.02)	0.09
						27		0.08 (0.07, 0.09)	< 0.02 (< 0.02, < 0.02)	0.08
08524.03-FL04, Citra, USA (FL), 2003 (Triple Play 58 SMS)	SC	0.3– 0.31	0.16	188– 194	4	15	Roots	<u>0.09</u> (0.09, 0.09)	< 0.02 (< 0.02, < 0.02)	0.09
08524.03-MI01, Laingsburg, USA (MI), 2003 (Heritage)	SC	0.3	0.16	186– 189	4	3	Roots	0.05 (0.05, 0.04)	< 0.02 (< 0.02, < 0.02)	0.05
						7		0.06 (0.06, 0.05)	< 0.02 (< 0.02, < 0.02)	0.06
						15		<u>0.06</u> (0.06, 0.05)	< 0.02 (< 0.02, < 0.02)	<u>0.06</u>
						22		0.06 (0.06, 0.05)	< 0.02 (< 0.02, < 0.02)	0.06
						29		0.04 (0.04, 0.03)	< 0.02 (< 0.02, < 0.02)	0.04
08524.03-NJ03, Bridgeton, USA (NJ), 2003 (Nelson F1)	SC	0.29	0.11	254– 259	4	13	Roots	<u>0.07</u> (0.06, 0.08)	< 0.02 (< 0.02, < 0.02)	0.07
08524.03-NS04, Somerset Canada (NS), 2003 (Interceptor)	SC	0.3– 0.1	0.15	201– 207	4	15	Roots	<u>0.03</u> (0.03, 0.03)	< 0.02 (< 0.02, < 0.02)	0.03
08524.03-ON16, Harrow, Canada (ON), 2003 (Cellobunch)	SC	0.29– 0.31	0.15	196– 204	4	16	Roots	<u>0.05</u> (0.03, 0.06)	< 0.02 (< 0.02, < 0.02)	0.05
08524.03-QC17, L'Assomption, Canada (QC), 2003 (Appaches)	SC	0.26– 0.33	0.12	220– 280	4	14	Roots	<u>0.05</u> (0.04, 0.05)	< 0.02 (< 0.02, < 0.02)	0.05
08524.03-QC18, St Sabine, Canada (QC), 2003 (Cellobunch)	SC	0.28– 0.33	0.12	237– 276	4	13	Roots	<u>0.04</u> (0.03, 0.04)	< 0.02 (< 0.02, < 0.02)	0.04
08524.03-TX02, Weslaco, USA (TX), 2003 (Six Pence)	SC	0.3– 0.32	0.15	201– 216	4	14	Roots	<u>0.06</u> (0.06, 0.05)	< 0.02 (< 0.02, < 0.02)	0.06
08524.03-WA01, Prosser, USA (WA), 2003 (DS 140395)	SC	0.3– 0.31	0.13– 0.14	224– 232	4	14	Roots	<u>0.03</u> (0.03, 0.03)	< 0.02 (< 0.02, < 0.02)	0.03

Ginseng

Five supervised trials on ginseng were carried out in 2007 in the USA and Canada. At each trial, nine applications at rates in the range of 0.29–0.32 kg ai/ha were performed at intervals of 7–15 days

between each foliar application. Samples were prepared by washing the roots with water and drying at 18–46 °C in a commercial drying facility. Samples of washed and dried roots were stored frozen at below –20 °C for up to 658 days until extraction and analysis. A sufficient stability of fenamidone was demonstrated for up to 650 days using spiked samples. The residue results are shown in Table 79.

Table 79 Residues in ginseng from supervised trials with fenamidone (09800, M-393676-01-1)

Trial, Location, Country, Year	Application					DA T, days	Commodity	Residues, mg/kg		
	Form	kg ai/ha	kg ai/hL	Water, L/ha	No			Fen	RPA 410193	Sum
GAP, USA	SC	0.3			3	14				
09800.07-BC04, Sommerland, Canada (BC), 2007	SC	0.30–0.32	0.034	895–942	9	15	Dried roots	0.17 (0.13, 0.21)	< 0.02 (< 0.02, < 0.02)	0.17
09800.07-MI29, Edgar, USA (WI), 2007	SC	0.30–0.32	0.046	653–687	9	14	Dried roots	0.10 (0.11, 0.08)	< 0.02 (< 0.02, < 0.02)	0.10
		0.30–0.31	0.046	642–678	3	14	Dried roots	0.06 (0.06, 0.05)	< 0.02 (< 0.02, < 0.02)	0.06
09800.07-MI30, Nutterville, USA (WI), 2007	SC	0.29–0.32	0.046	627–682	9	14	Dried roots	0.03 (0.02, 0.03)	< 0.02 (< 0.02, < 0.02)	0.03
09800.07-MI31, Mosinee, USA (WI), 2007	SC	0.29–0.31	0.046	635–670	9	14	Dried roots	0.29 (0.24, 0.34)	0.02 (< 0.02, 0.02)	0.31
09800.07-ON10, Otterville, (ON), USA, 2007	SC	0.30–0.31	0.030	1004–1037	9	12	Dried roots	0.35 (0.17, 0.53)	0.02 (< 0.02, 0.02)	0.37

Potato

Twenty-seven supervised trials on potato were conducted in Europe (Belgium, France, Germany, Greece, Italy, The Netherlands, Spain and the UK) between 1997 and 2010. Five, six or twelve foliar spray applications of fenamidone were performed at rates in the range of 0.1–0.16 kg ai/ha and intervals in the range of 7–14 days.

Eighteen supervised trials on potato were carried out during 1999 in the USA and three trials on potato were carried out in 2001 in Canada. In the trials fenamidone was applied either with six treatments at a rate of 0.19–0.23 kg ai/ha or with four applications at a rate of 0.29–0.315 kg ai/ha by foliar spray. Applications were performed at intervals of 3–7 days. All samples except those from the Florida trial (17534-16) were stored frozen for 176 days or less. The samples from trial 17534-16 were stored for 394 days (storage stability covered up to 12 month).

Seven residue trials were conducted in potato during 1998, 1999, 2000 and 2006 in Brazil by foliar spray. In trials performed in 1998, 1999 and 2000, fenamidone was applied three times to potato plants at rates of 0.15 kg ai/ha and 0.3 kg ai/ha on different plots. In trials performed in 2006, fenamidone was applied six times to potato plants at a rate of 0.15 kg ai/ha. The results are summarized in Table 80.

Table 80 Residues in potatoes from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
GAP, UK	SC	0.15			6	7					
97555AM1 Barly (80), France, 1997 (Nicolas)	WG	0.15	0.05	300	12	–0 0 7 14	Tuber	< 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	97-555 M-165234-01-1

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
97556RS1 Sept Saulx (51), France, 1997 (Kaptah Vandel)	WG	0.15	0.06	250	12	7	Tuber	< 0.02	< 0.02	< 0.02	97-556 M-165225- 01-1
97557LY1 Pusignan (69), France, 1997 (Monalisa)	WG	0.15	0.045	333	5	-0 0 7 14	Tuber	< 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	97-557 M-165261- 01-1
97558BX1 Pugnac (33), France, 1997 (Monalisa)	WG	0.15	0.03- 0.045	333- 466	5	7	Tuber	< 0.02	< 0.02	< 0.02	97-558 M-165265- 01-1
97691VI Alginet, Spain, 1997 (Obelix)	WG	0.15	0.015	976- 1024	5	7	Tuber	< 0.02	< 0.02	< 0.02	97-691 M-165758- 01-1
97745DE1 Winkelsett, Germany, 1997 (Bintje)	WG	0.13- 0.15	0.05	300	12	0 7 14	Tuber	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02	97-745 M-165485- 01-1
97745DE2 Kindenheim, Germany, 1997 (Quarta)	WG	0.15	0.05	300	12	0 7 14	Tuber	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02	
98525RS1 Sept Saulx (51) France, 1998 (Kaptah Vandel)	WG	0.15	0.05	309	12	8	Tuber	0.006	< 0.005	0.006	98-525 M-166541- 01-1
98526BX1 Pugnac (33), France, 1998 (Lizen)	WG	0.15	0.04	375	5	6	Tuber	< 0.005	< 0.005	< 0.005	98-526 M-166539- 01-1
98526LY1 Crémieu (38), France, 1998 (Europa)	WG	0.15	0.04	381	5	7	Tuber	< 0.005	< 0.005	< 0.005	
98527BX1 Virasac (33), France, 1998 (Bintje)	WG	0.15	0.035	435	5	-0 0 7 14	Tuber	< 0.005 < 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005 < 0.005	98-527 M-166545- 01-1
98527LY1 Pusignan (69), France, 1998 (Agata)	WG	0.15	0.04	375	5	-0 0 7 14	Tuber	< 0.005 < 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005 < 0.005	
98635V1 Alginet Spain, 1998 (Spunta)	WG	0.14- 0.15	0.01	1217- 1303	5	-0 0 7 14 21	Tuber	< 0.005 < 0.005 < 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005 < 0.005 < 0.005	98-635 M-166577- 01-1
98669DE1 Winkelsett, Germany, 1998 (Hansa)	WG	0.14- 0.15	0.04	400	12	0 6 13	Tuber	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	98-669 M-166638- 01-1
98669DE2 Kindenheim, Germany, 1998 (Quarta)	WG	0.14- 0.16	0.05	300	12	0 7 14	Tuber	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	
98688D-B1 Angeren, Netherlands, 1998 (Bintje)	WG	0.15- 0.16	0.05	293- 323	12	7	Tuber	< 0.005	< 0.005	< 0.005	98-688 M-166537- 01-1

Fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
98700AGS1 Stoney Houghton, UK, 1998 (Cara)	WG	0.15– 0.16	0.05	291– 318	12	7	Tuber	< 0.005	< 0.005	< 0.005	98-700 M-166903- 01-1
99648RS1 France, 1999 (Kaptah Vandel)	WG	0.15	0.06	250	12	7	Tuber	< 0.01	< 0.01	< 0.01	99-648 M-211672- 01-1
99664GR1 Avlona Attiki, Greece, 1999 (Naxoy)	WG	0.14– 0.15	0.01	1000– 1040	5	7	Tuber	< 0.01	< 0.01	< 0.01	99-664 M-191567- 01-1
09-2170-01 Bouafle (78), France, 2009 (Amandine)	SC	0.13	0.02	600	6	0 7	Tuber	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	09-2170 M-405055- 01-1
09-2170-02 Burscheid, Germany, 2009 (Laura)	SC	0.13	0.04	300	6	0 7	Tuber	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	
09-2170-03 Ondes (31), France, 2009 (Agata)	SC	0.13	0.04	300	6	0 7	Tuber	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	
09-2170-04 San Jose de la Rinconada, Spain, 2009 (Universal)	SC	0.12– 0.13	0.04	283– 300	6	0 7	Tuber	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	
09-2170-05 Piacenza, Italy, 2009(Kennebek)	SC	0.13	0.03	400	6	0 7	Tuber	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	
09-2170-06 S. Francisco Alcochete, Portugal, 2009 (Monalisa)	SC	0.13	0.03	400	6	0 7	Tuber	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	
10-2065-01 Vilanova del Vallés, Spain, 2010 (Red- Pontiac)	SC	0.13– 0.15	0.03– 0.08	200– 400	6	0 7	Tuber	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	10-2065 M-415558- 01-1
10-2065-02 Bologna, Italy, 2010 (Vivaldi)	SC	0.15	0.04	400	6	0 7	Tuber	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	
GAP, USA / Canada	SC	0.3			3	14					
17534-01 Ephrata (WA), USA, 1999	SC	0.2	0.07	283– 293	6	14	Tuber	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	99W17534 M-238825- 01-1

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
(Russet Burbank)						7		< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
						11		< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
		0.3– 0.31	0.1	283– 291	4	14	Tuber	<u>< 0.02</u> (< 0.02, < 0.02)	<u>< 0.02</u> (< 0.02, < 0.02)	<u>< 0.02</u>	
						17		< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
						21		< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
17534-02 Ephrata (WA), USA, 1999 (Russet Burbank)	SC	0.2	0.07	280– 291	6	14	Tuber	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
		0.3	0.1	282– 287	4	14	Tuber	<u>< 0.02</u> (< 0.02, < 0.02)	<u>< 0.02</u> (< 0.02, < 0.02)	< 0.02	
17534-03 Jerome (ID), USA, 1999 (Russet Burbank)	SC	0.2	0.07	279– 297	6	14	Tuber	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
		0.3	0.1– 0.11	277– 291	4	14	Tuber	<u>< 0.02</u> (< 0.02, < 0.02)	<u>< 0.02</u> (< 0.02, < 0.02)	< 0.02	
17534-04 Minidoka (ID), USA, 1999 (Russet Burbank)	SC	0.2– 0.21	0.06– 0.08	252– 363	6	14	Tuber	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
		0.3– 0.315	0.09	342– 352	4	14	Tuber	<u>< 0.02</u> (< 0.02, < 0.02)	<u>< 0.02</u> (< 0.02, < 0.02)	< 0.02	
17534-05 Harrah (WA), USA, 1999 (Norkotah E 3)	SC	0.2	0.09– 0.11	188– 232	6	14	Tuber	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
		0.3	0.13– 0.15	196– 232	4	14	Tuber	<u>< 0.02</u> (< 0.02, < 0.02)	<u>< 0.02</u> (< 0.02, < 0.02)	< 0.02	
17534-06 Hermiston (OR), USA, 1999 (Russet Burbank)	SC	0.2– 0.215	0.08– 0.09	236– 247	6	14	Tuber	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
		0.29– 0.3	0.12– 0.13	236– 243	4	14	Tuber	<u>< 0.02</u> (< 0.02, < 0.02)	<u>< 0.02</u> (< 0.02, < 0.02)	< 0.02	
17534-07 Comstock Park (MI), USA, 1999 (Dark Red Norland)	SC	0.2	0.07	288– 297	6	14	Tuber	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
		0.3	0.1	289– 297	4	14	Tuber	<u>< 0.02</u> (< 0.02, < 0.02)	<u>< 0.02</u> (< 0.02, < 0.02)	< 0.02	
17534-08 Delavan (WI), USA, 1999 (Superior)	SC	0.2	0.06– 0.066	304– 332	6	14	Tuber	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
		0.3– 0.31	0.09– 0.1	304– 332	4	14	Tuber	<u>< 0.02</u> (< 0.02, < 0.02)	<u>< 0.02</u> (< 0.02, < 0.02)	< 0.02	
17534-09 Fairmount (ND), USA, 1999	SC	0.2	0.07– 0.085	236– 283	6	14	Tuber	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	

Fenamidone

Trial, Location, Country, Year (Variety) (Norland)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
		0.3	0.11– 0.13	234– 283	4	14	Tuber	<u>< 0.02</u> (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
17534-10 Campbell (MN), USA, 1999 (Norkotah)	SC	0.2	0.07	283– 285	6	14	Tuber	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
						7		< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
						11		< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
		0.3– 0.31	0.011	282– 284	4	14	Tuber	<u>< 0.02</u> (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	<u>< 0.02</u>	
						17		< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
						21		< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
17534-11 Center (CO), USA, 1999 (Russet Nugget)	SC	0.2	0.07	281	6	14	Tuber	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
		0.3	0.011	281	4	14	Tuber	<u>< 0.02</u> (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
17534-12 Monte Vista (CO), USA, 1999 (Centennial)	SC	0.2	0.07	281	6	14	Tuber	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
		0.3	0.11	281	4	14	Tuber	<u>< 0.02</u> (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
17534-13 Newell (CA), USA, 1999 (Russet)	SC	0.2– 0.21	0.07	268– 294	6	14	Tuber	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
		0.3– 0.31	0.1	280– 289	4	14	Tuber	<u>< 0.02</u> (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
17534-14 Salisbury (MD), USA, 1999 (Red Norland, Dark)	SC	0.2– 0.21	0.06	329– 337	6	14	Tuber	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
		0.3	0.09	326– 330	4	14	Tuber	<u>< 0.02</u> (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
17534-15 Jamesville (NC), USA, 1999 (Atlantic)	SC	0.19– 0.2	0.07	275– 289	6	14	Tuber	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
		0.29– 0.3	0.1	277– 284	4	14	Tuber	<u>< 0.02</u> (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
17534-16 Oviedo (FL), USA, 1999 (Red Pontiac)	SC	0.2	0.06	324– 331	6	14	Tuber	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
		0.29– 0.3	0.09	318– 327	4	14	Tuber	<u>< 0.02</u> (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	
17534-17 North Rose (NY), USA, 1999	SC	0.2– 0.21	0.07	276– 292	6	14	Tuber	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02 (<u>< 0.02</u> , < 0.02)	< 0.02	

Trial, Location, Country, Year (Variety) (Green Mountain)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
		0.3– 0.31	0.11	277– 286	4	14	Tuber	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
17534-18 Exeter (ME), USA, 1999 (FL 1533)	SC	0.2	0.07	289– 293	6	14	Tuber	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
		0.29– 0.3	0.1	290– 293	4	14	Tuber	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
01AC05-01 Yorkton (SK), Canada, 2001 (Norland (Nebraska) Elite 3)	SC	0.2	0.1	200	6	14	Tuber	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	01AC05 M-394609- 01-1
		0.29– 0.3	0.15	200	4	14	Tuber	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
01AC05-02 Copetown (ON), Canada, 2001 (Red Pontiac)	SC	0.21– 0.23	0.085– 0.093	250	6	13	Tuber	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
01AC05-03 St-Paul d'Abbot- sford, (QC), Canada, 2001 (Chieftain)	SC	0.19– 0.21	0.082– 0.089	235	6	14	Tuber	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02	
GAP, Brazil	SC	0.15			3	7					
BRA98F23- BE298F23 Monte Mor (SP), Brazil, 1998 (Atlantic)	SC	0.15	0.015	1000	3	7	Tuber	< 0.02	< 0.02	< 0.02	R.23/01 M-284927- 01-1
		0.3	0.03	1000	3	7	Tuber	< 0.02	< 0.02	< 0.02	
BRA98F23- BE198F23 Paulinia (SP), Brazil, 1999 (Bintje)	SC	0.15	0.015	1000	3	7	Tuber	< 0.02	< 0.02	< 0.02	R.32/01 M-284937- 01-1
		0.3	0.03	1000	3	7	Tuber	< 0.02	< 0.02	< 0.02	
FD00BRAZ14 Monte Mor (SP), Brazil, 2000 (Baronesa)	SC	0.15	0.015	1000	3	7	Tuber	< 0.02	< 0.02	< 0.02	R.50/01 M-284944- 01-1
		0.3	0.03	1000	3	7	Tuber	< 0.02	< 0.02	< 0.02	
FR06BRA008-P1, Paulinia (SP), Brazil, 2006 (Monalisa)	SC	0.15	0.015	1000	6	7	Tuber	< 0.02	< 0.02	< 0.02	UNESP RA- 1066/06 M-279947- 02-1
FR06BRA008-P2, Londrina (PR), Brazil, 2006 (Monalisa)	SC	0.15	0.015	1000	6	7	Tuber	< 0.02	< 0.02	< 0.02	UNESP RA- 1067/06 M-279942- 02-1
FR06BRA008-P3, Ribeirão Preto (SP), Brazil, 2006 (Monalisa)	SC	0.15	0.015	1000	6	7	Tuber	< 0.02	< 0.02	< 0.02	UNESP RA- 1068/06 M-279946- 02-1
FR06BRA008-P4, Chacara das Flores (RS), Brazil, 2006 (Monalisa)	SC	0.15	0.015	1000	6	7	Tuber	< 0.02	< 0.02	< 0.02	UNESP RA- 1069/06 M-279941- 02-1

*Stalk and stem vegetables**Witloof chicory (sprouts)*

Nine indoor residue trials were conducted in Belgium, France, Germany and Netherlands in 2004, 2008 and 2010. In 2004 four residue trials were conducted in Belgium, France and the Netherlands consisting of one application through the irrigation water system at a maximum rate of 0.6 g ai/hL of fenamidone and a water volume of 40 L/m², at the commencement of forcing.

In 2008 and 2010 five residue trials were conducted in France, Germany and the Netherlands. These trials were treated twice, one dip application for two minutes in a solution containing 6.0 g ai/hL, immediately after field sampling of the roots. After storage of the roots in a cold room for 3–8 months, a second application was made at the commencement of forcing in the irrigation water system at a maximum rate of 0.6 g ai/hL. Sprout and root samples were taken on day 21 (or 20) after the second application. The results are summarized in Table 81.

Table 81 Residues in witloof chicory from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg			Reference
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum	
GAP, Belgium	WG		0.006 + 0.0006		2	18					
R 2004 0878/1 Zwaagdijk-Oost, Netherlands, 2004 (Vintor)	WG		0.0006	400000	1	20	Leaf	< 0.01	< 0.01	< 0.01	RA-2566/04 M-256652-01-1
						20	Root	0.11	< 0.01	0.11	
R 2004 0913/3 St Amand, Belgium, 2004 (Atlas)	WG		0.0006	400000	1	21	Leaf	< 0.01	< 0.01	< 0.01	
						21	Root	0.01	< 0.01	0.01	
R 2004 0914/1 St Amand, Belgium, 2004 (Vintor)	WG		0.0006	400000	1	21	Leaf	< 0.01	< 0.01	< 0.01	
						21	Root	0.02	< 0.01	0.02	
R 2004 0916/8 Fresnoy lès Roye (80), France, 2004 (Passion)	WG		0.0006	400000	1	21	Leaf	< 0.01	< 0.01	< 0.01	08-2205 M-363750-03-1
						21	Root	0.81	0.05	0.87	
08-2205-01 Fresnoy lès Roye (80), France, 2008 (Hermes)	WG		0.006 ^a 0.0006 ^b		2	21	Leaf	< 0.01	< 0.01	< 0.01	
						21	Root	0.26	0.01	0.27	
08-2205-02, Werdau-Torgau, Germany, 2008 (Atlas)	WG		0.006 ^a 0.0006 ^c		2	21	Leaf	< 0.01	< 0.01	< 0.01	
						21	Root	0.56	0.03	0.59	
08-2205-03, Zwaagdijk, Netherlands, 2008 (Atlas)	WG		0.006 ^a 0.0006 ^c		2	21	Leaf	< 0.01	< 0.01	< 0.01	
						21	Root	0.11	< 0.01	0.11	
10-2040-01, Zwaagdijk, Netherlands, 2010 (Fakir)	WG		0.006 ^a 0.0006 ^b		2	21	Leaf	< 0.01	< 0.01	< 0.01	10-2040 M-420668-01-1
						21	Root	0.20	< 0.01	0.20	
10-2040-02, Werdau, Germany, 2010 (Desir)	WG		0.006 ^a 0.0006 ^c		2	20	Leaf	< 0.01	< 0.01	< 0.01	
						20	Root	0.40	0.01	0.41	

^a Dip application, roots were dipped for 2 minutes into the application solution and stored in a cold room.

^b Flooding with treated irrigation water with circulation.

^c Flooding with treated irrigation water without circulation.

Celery

Six supervised trials on celery were carried out during 2003 in the USA (201214, M-242541-01-1). At each trial, fenamidone was applied by foliar spray at rates of about 0.3 kg ai/ha four times at intervals of 4–6 days. The results are summarized in Table 82.

Table 82 Residues in celery from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg		
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum
GAP, USA	SC	0.3			3	2				
YW013-03H Belle Glade (FL), USA, 2003 (683K)	SC	0.29–0.31	0.145–0.17	176–210	4	2	Plant w/o roots	<u>2.3</u> (2.03, 2.51)	< 0.02 (< 0.02, < 0.02)	2.3
						2	Trimmed stalks	0.32 (0.35, 0.29)	< 0.02 (< 0.02, < 0.02)	0.32
YW014-03H Springfield (NE), USA, 2003 (Golden selfblanching)	SC	0.3	0.2	148–151	4	2	Plant w/o roots	<u>8.8</u> (8.05, 9.55)	0.04 (0.04, 0.04)	8.8
						2	Trimmed stalks	1.2 (1.09, 1.35)	< 0.02 (< 0.02, < 0.02)	1.2
YW015-03D Porterville (CA), USA, 2003 (Conquistador)	SC	0.3	0.14	212–218	4	0	Plant w/o roots	5.6 (5.33, 5.78)	< 0.02 (< 0.02, < 0.02)	5.6
						2		<u>4.4</u> (4.67, 4.16)	< 0.02 (< 0.02, < 0.02)	4.4
						3		3.2 (2.83, 3.50)	< 0.02 (< 0.02, < 0.02)	3.2
						5		3.9 (2.21, 5.61)	< 0.02 (< 0.02, < 0.02)	3.9
						7		3.5 (3.66, 3.36)	< 0.02 (< 0.02, < 0.02)	3.5
YW016-03H King City (CA), USA, 2003 (Conquistador)	SC	0.3	0.16	186–192	4	2	Plant w/o roots	<u>4.5</u> (4.47, 4.44)	0.04 (0.04, 0.03)	4.5
						2	Trimmed stalks	0.06 (0.07, 0.04)	< 0.02 (< 0.02, < 0.02)	0.06
YW017-03H Fresno (CA), USA, 2003 (Conquistador)	SC	0.3	0.14	207–214	4	2	Plant w/o roots	<u>18</u> (16.8, 19.8)	0.16 (0.13, 0.18)	19
						2	Trimmed stalks	1.1 (0.95, 1.23)	< 0.02 (< 0.02, < 0.02)	1.1
YW018-03H Visalia (CA), USA, 2003 (Matador)	SC	0.3	0.14	211–218	4	2	Plant w/o roots	<u>15</u> (12.9, 16.5)	0.11 (0.09, 0.13)	15
						2	Trimmed stalks	1.61 (1.53, 1.69)	0.04 (0.04, 0.03)	1.7

*Oilseed**Cotton seed*

Twelve trials were conducted on cotton in the USA during 2003(201163, M-242540-01-1). In each trial, cotton plants were treated at-planting with a single in-furrow over-the-seed application at a target rate of 0.3 kg ai/ha. Samples of mature cotton were harvested 127–190 days post treatment from each test location by hand or using either stripper or picker harvest equipment. Samples were then ginned to yield undelinted cotton seed. The results are summarized in Table 83.

Table 83 Residues in cotton seed from supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg		
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum
GAP, USA	SC	0.3			1					
YW021-03H, Tifton (GA), USA, 2003 (Fibermax 989 RRBt)	SC	0.3	0.955	31	1	154	Undelinted seed	<u>< 0.02</u> (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02
YW022-03H, Leland (MS), USA, 2003 (FM960BR)	SC	0.29	0.694	42	1	161	Undelinted seed	<u>< 0.02</u> (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02

Fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT days	Commodity	Residues, mg/kg		
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum
YW023-03H, Newport (AR), USA, 2003 (PM 1218 BG/RR)	SC	0.3	0.774	39	1	173	Undelinted seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
YW024-03H, Proctor (AR), USA, 2003 (DP451 B/RR)	SC	0.3	0.754	40	1	130	Undelinted seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
YW025-03HA East Bernard (TX), USA, 2003 (DP 555 BG/RR)	SC	0.3	0.792	38	1	127	Undelinted seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
YW026-03H Levelland (TX), USA, 2003 (PM 2280 BG/RR)	SC	0.3	0.8	38	1	151	Undelinted seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
YW027-03H Littlefield (TX), USA, 2003 (PM 2280 BG/RR)	SC	0.3	0.795	38	1	178	Undelinted seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
YW028-03H Colony (OK), 2003(PM 2280 BG/RR)	SC	0.3	0.699	43	1	149	Undelinted seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
YW029-03H Plainview (TX), USA, 2003 (Paymaster 2344 B/R)	SC	0.3	0.603	49	1	173	Undelinted seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
YW030-03H Fresno (CA), USA, 2003(Acala Riata RR)	SC	0.31	0.746	41	1	160	Undelinted seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
YW031-03H Visalia (CA), USA, 2003 (DP6100 R/R Acala)	SC	0.3	0.787	38	1	180	Undelinted seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
YW032-03H Blythe (CA), USA, 2003 (449 BRR)	SC	0.3	0.777	39	1	190	Undelinted seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02

Sunflower seed

Nine trials with fenamidone on sunflower were conducted in 2009 in the USA (07999, M-251287-02-1). In each trial, one plot was planted with sunflower seeds treated with fenamidone resulting in a rate of 0.19 kg ai/100 kg seed. Two trials included an additional plot planted with seed treated at an exaggerated rate of 0.95 kg ai/100 kg seed. Samples of mature sunflower seed were harvested from each test location 104–146 days after sowing of the treated seed. The maximum storage period from sampling to analysis was 433 days. A sufficient stability of fenamidone and its metabolites was demonstrated for up to 438 days using spiked samples. The results are shown in Table 84.

Table 84 Residues in sunflower seed of supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application			DAT, Days ^a	Commodity	Residues, mg/kg		
	Form	kg ai/100 kg seed	No			Fen	RPA 410193	Sum
GAP, USA	SC	0.19	1					
07999.02-CO15, Ft Collins, USA (CO), 2002 (Triumph 665)	SC	0.19	1	104	Seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
07999.02-ND15, Fargo, USA (ND), 2002 (Pioneer 63M80)	SC	0.19	1	145	Seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
		0.95	1	145	Seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
07999.02-ND16, Fargo, USA (ND), 2002 (Pioneer 63M91)	SC	0.19	1	144	Seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02

Trial, Location, Country, Year (Variety)	Application			DAT, Days ^a	Commodity	Residues, mg/kg		
	Form	kg ai/100 kg seed	No			Fen	RPA 410193	Sum
07999.02-ND17, Fargo, USA (ND), 2002 (Pioneer 63M52)	SC	0.19	1	143	Seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
07999.02-ND18, Minot, USA (ND), 2002 (Pioneer 63M80)	SC	0.19	1	120	Seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
07999.02-NE04, USA Scottsbluff, (NE), 2002 (IS 6767)	SC	0.19	1	119	Seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
		0.95	1	119	Seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
07999.02-NE05, Scottsbluff, USA (NE), 2002 (IS 4049 AK)	SC	0.19	1	113	Seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
07999.02-SD12, Southshore, USA (SD), 2002 (NK231 (F))	SC	0.19	1	146	Seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
07999.02-SD13, Southshore, USA (SD), 2002 (NK278 (B))	SC	0.19	1	146	Seed	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02

^a From sowing to sampling

Legume animal feeds

Bean forage (green)

Eight field trials on snap beans were conducted in the USA and Canada (08895, M-489329-01-1). The residues in bean forage are shown in Table 85. At all trials, four foliar applications of the test substance were made 6–8 days apart at a rate of approximately 0.3 kg ai/ha (for a total application of 1.2 kg ai/ha). At trial CA86 a fifth foliar application of 0.3 kg ai/ha was made, due to slow maturation of the pods at that site. Samples were harvested by hand or using clippers/pruners and either placed directly into sample bags or shaken lightly to remove loose soil before being placed in to sample bag. At trial CA86 the samples of plants with pods were sub-sampled by cutting four groups of three plants each into top, middle, and bottom sections and retaining a top, middle, and bottom portion from each group. This subsample was placed into the sample bag and the remaining portions were discarded.

Table 85 Fenamidone residues in bean forage

Trial, Location, Country, Year (Variety)	Application			DALT, days	Commodity	Residues, mg/kg		
	Form	Kg ai/ha	No			Fen	RPA 410193	Sum
GAP, USA (proposed)	SC	0.3	3	3				
CA86, Holtville, CA, USA, 2008 (Veronica)	SC	0.3	5	3	Plant with pods	25 (24, 25)	0.11 (0.11, 0.1)	25
FL07, Citra, FL, USA, 2008 (Dusky)	SC	0.3	4	4	Plant with pods	2.3 (2.0, 2.6)	0.017 (0.015, 0.019)	2.3
MI08, Holt, MI, USA, 2008 (Ulysses)	SC	0.3	4	3	Plant with pods	5.6 (5.78, 5.42)	0.045 (0.046, 0.043)	5.6
NC21, Clinton, NC, USA, 2008 (Ambra)	SC	0.3	4	3	Plant with pods	16 (15.8, 15.9)	0.094 (0.093, 0.094)	16
NY15, Freeville, NY, USA, 2008 (Hystyle)	SC	0.3	4	3	Plant with pods	11 (11.8, 10.2)	0.043 (0.043, 0.042)	11
WA16, Prosser, WA, USA, 2008 (OSU 91G)	SC	0.3	4	3	Plant with pods	4.13 (3.53, 4.73)	0.026 (0.018, 0.029)	4.2
WI03, Arlington, WI, USA, 2008 (Hystyle)	SC	0.3	4	3	Plant with pods	7.6 (7.80, 7.35)	0.031 (0.031, 0.03)	7.6
WI04, Arlington, WI, USA, 2008 (Hercules)	SC	0.3	4	1	Plant with pods	10 (9.89, 10.8)	0.028 (0.025, 0.031)	10
				3		10 (10.6, 9.71)	0.039 (0.038, 0.039)	10

Trial, Location, Country, Year (Variety)	Application			DALT, days	Commodity	Residues, mg/kg		
	Form	Kg ai/ha	No			Fen	RPA 410193	Sum
				7		6.8 (6.68, 6.97)	0.039 (0.042, 0.036)	6.9
				13		4.6 (4.28, 5.00)	0.025 (0.024, 0.026)	4.7

Miscellaneous fodder and forage crops

Cotton fodder, dry

Trials were conducted on cotton in the USA during 2003 (201163, M-242540-01-1). Cotton plants were treated at-planting with a single in-furrow over-the-seed application at a target rate of 0.3 kg ai/ha. Cotton gin by-products (gin trash) were collected from the cotton harvested 127–190 days post treatment mechanically using either a picker or a stripper. Because cotton gin by-products are used as animal feed, the fodder was analysed for residues. The results are summarized in Table 86.

Table 86 Residues in cotton fodder of supervised trials with fenamidone

Trial, Location, Country, Year (Variety)	Application					DAT, Days	Commodity	Residues, mg/kg		
	Form	kg ai/ha	kg ai/hL	Water L/ha	No			Fen	RPA 410193	Sum
GAP, USA	SC	0.3			1					
YW021-03H, Tifton (GA), USA, 2003 (Fibermax 989 RRBT)	SC	0.3	0.955	31	1	154	Cotton gin by-products (picker)	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
YW022-03H, Leland (MS), USA, 2003 (FM960BR)	SC	0.29	0.694	42	1	161	Cotton gin by-products (picker)	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
YW023-03H, Newport (AR), USA, 2003 (PM 1218 BG/RR)	SC	0.3	0.774	39	1	173	Cotton gin by-products (picker)	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
YW025-03HA East Bernard (TX), USA, 2003 (DP 555 BG/RR)	SC	0.3	0.792	38	1	127	Cotton gin by-products (stripper)	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
YW026-03H Levelland (TX), USA, 2003 (PM 2280 BG/RR)	SC	0.3	0.8	38	1	151	Cotton gin by-products (stripper)	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02
YW027-03H Littlefield (TX), USA, 2003 (PM 2280 BG/RR)	SC	0.3	0.795	38	1	178	Cotton gin by-products (stripper)	≤ 0.02 (< 0.02 , < 0.02)	< 0.02 (< 0.02 , < 0.02)	< 0.02

FATE OF RESIDUES IN STORAGE AND PROCESSING

Residues are reported separately for fenamidone (“Fen”), diketo-fenamidone (RPA 410193) and desanilino-fenamidone (RPA 412708). The codes RPA 410193 (synonyms: AE C650488, RPA 405862, DK-Fen) and RPA 412708 (synonyms: AE 050057, RPA 408056, DA-Fen) are used throughout this section, irrespective of what nomenclature was used in the reports.

In processing-nature of residues

To estimate the degradation behaviour of [C-phenyl-UL-¹⁴C]-fenamidone during industrial processing or household preparation, the processes of pasteurization (90 °C, 20 min at pH 4), baking, boiling, brewing (100 °C, 60 min at pH 5) and sterilization (120 °C, 20 min at pH 6) were simulated (17581, M-173268-01-1).

The test system consisted of flasks containing approximately 100 mL of buffer maintained at constant temperature and pH for the duration of the simulation. All samples were analysed on the day

of treatment by concurrent radio-detection and LC/MS. The recovery of TAR is summarized in Table 87 and the composition of TRR in Table 88.

Table 87 Recovery of TAR after hydrolysis

Test System	Fraction	% Applied radioactivity	
		0.1 mg/kg Treatment Rate	1.0 mg/kg Treatment Rate
Pasteurisation (90 °C, pH 4)	Sample	98.3	94.0
	Volatiles	0.0	0.0
	Total	98.3	94.0
Baking (100 °C, pH 5)	Sample	104.6	100.3
	Volatiles	0.01	0.00
	Total	104.6	100.3
Sterilisation (Nominal 120 °C, pH 6)	Sample	109	102.4
	Volatiles	0.0	0.0
	Total	109	102.4

Table 88 Degradation products after hydrolysis

Test System	Application Rate (mg/kg)	Replicate	% Applied radioactivity (mg/kg)		
			Fen	RPA 410193	RPA 412708
Pasteurisation (90 °C, pH 4)	0.1	1	98.34 (0.089)	—	—
		2	98.34 (0.089)	—	—
		Mean	98.34 (0.089)	—	—
	1	1	81.96 (0.775)	12.06 (0.114)	—
		2	81.84 (0.774)	12.18 (0.115)	—
		Mean	81.90 (0.77)	12.12 (0.11)	—
Baking (100 °C, pH 5)	0.1	1	104.63 (0.1)	—	—
		2	104.63 (0.1)	—	—
		Mean	104.63 (0.1)	—	—
	1	1	96.32 (0.925)	4.00 (0.038)	—
		2	94.69 (0.909)	5.62 (0.054)	—
		Mean	95.51 (0.92)	4.81 (0.046)	—
Sterilisation (Nominal 120 °C, pH 6)	0.1	1	109.01 (0.098)	—	—
		2	109.01 (0.098)	—	—
		Mean	109.01 (0.098)	—	—
	1	1	96.59 (0.93)	2.73 (0.026)	3.05 (0.029)
		2	95.86 (0.923)	2.28 (0.022)	4.23 (0.041)
		Mean	96.22 (0.93)	2.50 (0.024)	3.64 (0.035)

In processing-effect on the residue level

The Meeting received information on the fate of fenamidone residues during the processing of raw agricultural commodities (RAC) like grapes to juice, must, wine and pomace and tomatoes into juice, paste, ketchup and canned tomatoes. Because the residues of diketo-fenamidone (RPA 410193) are of the same order of magnitude as the parent concentrations in processed products of grapes and tomatoes, the sum of parent and RPA 410193 is calculated as follows:

Fenamidone, mg/kg	RPA 410193, mg/kg	Total, mg/kg
< 0.02	< 0.02	< 0.04
< 0.02	0.076	0.10
0.05	< 0.02	0.07
0.53	0.13	0.67 ^a

$$^a 0.53 + (0.13 \times 1.11) = 0.6743$$

Two processing studies were carried out on potatoes but were only of limited use because the residues in RAC were < LOQ. Further studies conducted on cabbage, broccoli, peppers, mustard greens and spinach investigated the fate of fenamidone and RPA 410193 after cooking.

Processing of grapes

Ten processing trials were conducted in which grapes were processed into red wine; grape juice was also produced in eight of these trials. Sixteen processing trials were conducted in which grapes were processed into white wine; grape juice was also produced in two of these trials. The results and the calculated processing factors (residue in processed commodity ÷ residue in RAC) for MRL setting and dietary intake purposes are presented in Tables 89 and 90. The information submitted on processing procedures is summarized as follows.

Red and white wine

Grape samples (bunches) were processed simulating commercial practices. The grape bunches were crushed and stemmed and the must collected in stainless steel tanks. Pectolytic enzyme was added to the juice of white grapes. Alcoholic fermentation was started by addition of dry active yeast. The fermentation was controlled on a daily basis by measurement of the temperature, density and pH-value. Alcoholic fermentation was considered complete when the must/juice density fell below 1.000. The must (red grapes) was processed to young wine and pomace by use of a water press. Red wine (only) was then subjected to malolactic fermentation. Alcoholic and malolactic fermentation were stopped by addition of potassium metabisulphite and the young wine was subjected to natural clarification, aided by addition of gelatine and potassium metabisulphite and storage for 26–27 days in a cold chamber at about 5–10 °C. Finally the clarified wine was filtered (2.5 and 1.5 µm) and stabilized by addition of potassium metabisulphite. The production of white wine followed similar processes but without malolactic fermentation.

Grape juice

For processing of red grape juice, grape bunches were crushed and stemmed and the crushed samples placed in a saucepan and treated with pectolytic enzyme for two hours at 50–65 °C. Juice was then obtained by pressing the must in a water press. Samples of white grapes were directly processed into juice using a water press. Pectolytic enzyme was added to the juice of white grapes. Samples were stabilized by addition of potassium metabisulphite.

Table 89 Residues of fenamidone in red grapes (RAC and processed fractions)

Country, Year, Trial (Variety)	Commodity/product	Residue, mg/kg			Processing factor		Reference
		Fen	RPA 410193	Sum	Parent	Sum	
France, 1997 97599AV1 (Grenache)	Grapes	0.11	0.026	0.14		–	97-599 M- 165500- 01-1
	Juice	< 0.02	0.048	0.073		0.52	
	Must	0.28	0.051	0.34	2.55	2.4	
	Pomace	0.38	0.12	0.51	3.45	3.6	
	Wine, alcoholic fermentation	0.047	0.054	0.11		0.79	
	Wine, malolactic fermentation	0.023	0.088	0.12		0.86	
	Red wine	0.022	0.093	0.13		0.93	
France, 1997 97599DJ1 (Pinot noir)	Grapes	0.21	0.038	0.25		–	97-599 M- 165500- 01-1
	Juice	< 0.02	0.07	0.098		0.39	
	Must	0.11	0.042	0.16	0.52	0.64	
	Pomace	0.42	0.162	0.60	2.0	2.4	
	Wine, alcoholic fermentation	0.043	0.056	0.11		0.44	
	Wine, malolactic fermentation	< 0.02	0.054	0.080		0.32	
	Red wine	< 0.02	0.096	0.13		0.52	
France, 1998 98562AV1 (Grenache)	Grapes	0.12	0.039	0.16		–	98-562 M- 170328- 01-1
	Juice	< 0.02	0.041	0.065		0.41	
	Must	0.14	0.026	0.17	1.17	1.1	
	Red wine	< 0.02	0.079	0.11		0.65	
France, 1998 98562RN1 (Cabernet)	Grapes	0.15	0.04	0.19		–	
	Juice	< 0.02	0.03	0.053		0.28	
	Must	0.098	0.035	0.14	0.65	0.74	
	Red wine	< 0.02	0.070	0.097		0.51	
France, 1998 98562TL1 (Cinsault)	Grapes	0.27	0.071	0.35		–	
	Juice	0.11	0.15	0.28		0.8	
	Must	0.53	0.13	0.67	1.96	1.9	

Country, Year, Trial (Variety)	Commodity/product	Residue, mg/kg			Processing factor		Reference
		Fen	RPA 410193	Sum	Parent	Sum	
	Red wine	< 0.02	0.076	0.10		0.29	
France, 1999, 99531DJ1-P3 (Pinot noir)	Grapes	0.034	< 0.02	0.054		–	
	Red wine	0.003	0.074	0.085		1.6	
France, 1999, 99531DJ1-P4 (Pinot noir)	Grapes	0.047	< 0.02	0.067		–	
	Red wine	0.004	0.089	0.10		1.5	
France, 1999 99629AV1 (Grenache)	Grapes	0.23	0.049	0.28		–	
	Juice	0.031	0.045	0.081		0.29	
	Must	< 0.01	< 0.01	0.02	< 0.04	0.07	
	Red wine	0.012	0.11	0.13		0.46	
France, 1999 99689RS1 (Pinot noir)	Grapes	0.069	0.021	0.092		–	
	Juice	< 0.01	0.018	0.03		0.33	
	Must	0.063	0.027	0.093	0.91	1.0	
	Red wine	0.005	0.028	0.036		0.39	
France, 1999 99689BX1 (Merlot)	Grapes	0.13	< 0.02	0.15		–	
	Juice	0.012	0.015	0.029		0.19	
	Must	0.17	0.033	0.21	1.31	1.4	
	Red wine	0.015	0.054	0.075		0.5	

Table 90 Residues of fenamidone in white grapes (RAC and processed fractions)

Country, Year, Trial (Variety)	Commodity/product	Residue, mg/kg			Processing factor		Reference
		Fen	RPA 410193	Sum	Parent	Sum	
France, 1997 97599TL1 (Ugni blanc)	Grapes	0.20	0.040	0.24		–	
	Juice	0.021	0.074	0.10		0.42	
	Must	0.055	0.036	0.095	0.275	0.40	
	Pomace	0.084	0.027	0.11	0.42	0.46	
	Wine, alcoholic fermentation	0.026	0.17	0.21		0.88	
	White wine	< 0.02	0.18	0.22		0.92	
Italy, 1998, 98721BO1 (1 st trial) (Malvasia Toscana)	Grapes	0.19	0.076	0.27		–	
	White wine	< 0.02	0.15	0.19		0.70	
Italy, 1998, 98721BO1 (2 nd trial) (Malvasia Toscana)	Grapes	0.13	0.073	0.21		–	
	White wine	< 0.02	0.17	0.21		1.0	
Italy, 1998, 98730BO1 (Moscato d' Asti)	Grapes	0.064	0.04	0.11		–	
	White wine	< 0.02	0.075	0.10		0.91	
Italy, 1998, 98731BO1 (Pinot bianco)	Grapes	0.36	0.082	0.45		–	
	White wine	0.027	0.34	0.40		0.89	
Italy, 1998, 98736BO1 (Moscato d' Asti)	Grapes	0.052	0.052	0.11		–	
	White wine	< 0.02	< 0.02	0.04		0.36	
Italy, 1998, 98738BO1 (Pinot bianco)	Grapes	0.23	0.18	0.43		–	
	White wine	0.024	0.20	0.25		0.58	
France, 1999, 99531RS1-P3 (Chardonnay)	Grapes	0.034	0.022	0.058		–	
	White wine	< 0.0005	0.034	0.038		0.66	
France, 1999, 99531RS1-P4 (Chardonnay)	Grapes	0.054	0.024	0.081		–	
	White wine	0.0007	0.055	0.062		0.77	
France, 1999, 99629RS1 (Chardonnay)	Grapes	0.093	0.039	0.14		–	
	Juice	0.010	0.024	0.037		0.26	
	Must	0.049	0.027	0.079		0.56	
	White wine	0.0027	0.055	0.064		0.46	
Italy, 1999, 99698BO1-P2 (Garganega)	Grapes	0.030	< 0.02	0.05		–	
	White wine	0.0009	0.056	0.063		1.26	
Italy, 1999, 99698BO1-P3 (Garganega)	Grapes	0.098	0.047	0.15		–	
	White wine	0.0017	0.052	0.059		0.39	
Italy, 1999, 99698BO1-P4 (Garganega)	Grapes	0.049	0.031	0.083		–	
	White wine	0.0034	0.096	0.11		1.3	
Italy, 1999, 99698BO2-P2 (Chardonnay)	Grapes	0.35	0.18	0.55		–	
	White wine	0.064	0.28	0.37		0.67	
Italy, 1999, 99698BO2-P3 (Chardonnay)	Grapes	0.25	0.098	0.36		–	
	White wine	0.022	0.25	0.30		0.83	

Country, Year, Trial (Variety)	Commodity/product	Residue, mg/kg			Processing factor		Reference
		Fen	RPA 410193	Sum	Parent	Sum	
Italy, 1999, 99698BO2-P4 (Chardonnay)	Grapes	0.68	0.19	0.89		—	
	White wine	0.092	0.53	0.68		0.76	
Italy, 1999, 99705BO1 (Malvasia Toscana)	Grapes	0.14	0.037	0.18		—	99-705
	White wine	0.0045	0.11	0.13		0.72	M-249144-01-1
Italy, 1999, R 1999 0302/2 (Contese)	Grapes (bunches)	0.04	< 0.02	0.06		—	0302-99 RA-3186-99 M-078134-01-1
	Grapes (berries)	0.06	0.02	0.082		1.4	
	Must	0.03	< 0.02	0.05		0.83	
	White wine	< 0.02	0.04	0.064		1.1	

Processing of tomatoes

Three processing studies on tomatoes were submitted. The residues of fenamidone and RPA 410193 as well as processing factors are shown in Table 91. Because the residues increase after processing in tomato puree, ketchup and paste, processing factors were calculated for MRL (parent) and for dietary intake (sum) purposes. Further information on the residue trials and the processing procedures are described below.

Study 1

A first study was conducted in 2000 in Southern France, (99-519, M-206274-01-1). The study contained a single field trial conducted in Southern France to determine the magnitude of residues of fenamidone and its metabolite diketo-fenamidone (RPA 410193) in/on tomato and processed products. Fenamidone was applied to the tomato plants in five foliar applications at the rate of 0.15 kg ai/ha. The fruit samples harvested 3 days after the last application were processed into juice, puree, paste, canned tomatoes and ketchup.

Study 2

A second study was conducted following application of fenamidone in three protected trials conducted in Europe (the Netherlands, Italy and Germany) during 2009 (09-3235, M-406443-02-1).

Fenamidone was applied to tomato plants at a rate of 0.127 kg ai/ha in three spray applications at 7-day intervals between growth stages BBCH 77 to 85 at the three trial sites. Samples of tomato fruit to be processed were harvested 3 days after the last treatment (BBCH 83–89). The processing of the fruit samples into the processed fractions juice, puree and canned tomatoes was performed using methods simulating industrial/household practices.

Tomatoes were washed in water, to produce “washings” and “whole fruit, washed” and left to drain. Cut samples were then heated in water to 80–100 °C in order to prevent enzymatic reactions. After this blanching process, the pulp was passed through a strainer to separate juice from “strain rest” (wet pomace). Sodium chloride was added to the raw juice and taken for the preparation of “raw puree”. Aliquots of the raw puree were placed into cans and pasteurized to give puree. A portion of the “raw juice” was used for processing the canned tomatoes, which were placed in cans and pasteurized. Samples of finished products and intermediate processing fractions were collected for analysis.

Study 3

A third study was conducted in California, USA (98W13074, M-238681-01-1). Field grown tomatoes were treated by foliar spray at 7-day intervals by 6 × 1.0 kg ai/ha. Samples were collected 14 days after the last application. The tomatoes were washed twice, first with tap water, then with chlorinated water. The washed tomatoes were ground and crushed, heated to 96–99 °C and fed into a finisher for separation into wet pomace and juice. The juice was concentrated by vacuum evaporation to produce puree, a sample of which was vacuum condensed to paste. The paste fraction was heated (88–89 °C), packed into cans, sealed and held at filling temperature for 10–28 minutes prior to cooling. Triplicate subsamples of the processed fractions (raw, unwashed tomatoes, tomato puree and tomato paste) were taken and all samples frozen.

Table 91 Residues of fenamidone in processed tomato commodities

RAC / Processed commodity	Residues, mg/kg			Processing factor		Reference, trial
	Fen	RPA 410193	Sum	Parent	Sum	
Tomato fruit	0.05 (0.06, 0.05, 0.04)	< 0.02 (< 0.02, < 0.02, < 0.02)	0.07	–	–	99-519, M-206274- 01-1 99519BX1 France
Tomato juice	0.04	< 0.02	0.06	0.8	0.9	
Tomato puree	0.09	0.05	0.15	1.8	2.1	
Tomato ketchup	0.08	0.08	0.17	1.6	2.4	
Tomato paste	0.12	0.14	0.28	2.4	4.0	
Canned tomatoes	< 0.02	< 0.02	< 0.04	< 0.4	< 0.6	
Tomato fruit	0.04	< 0.01	0.05	–	–	09-3235, M-406443- 02-1, 09-3235-01 Netherlands
Raw juice	0.04	0.01	0.05	1.0	1.0	
Tomato juice	0.03	< 0.01	0.04	0.8	0.8	
Peel	0.39	< 0.01	0.4	9.8	8.0	
Peeling water	< 0.01	< 0.01	< 0.02	0.3	< 0.4	
Peeled fruit	< 0.01	< 0.01	< 0.02	0.3	< 0.4	
Canned tomatoes	0.01	< 0.01	0.02	0.3	0.4	
Whole fruit, washed	0.04	< 0.01	0.05	1.0	1.0	
Washings	0.01	< 0.01	0.02	0.3	0.4	
Strain rest (pomace, wet)	0.25	0.01	0.26	6.3	5.2	
Raw puree	0.09	0.02	0.11	2.3	2.2	
Tomato puree	0.12	0.03	0.15	3.0	3.0	
Tomato fruit	0.15	< 0.01	0.16	–	–	09-3235, M-406443- 02-1, 09-3235-02 Italy
Raw juice	0.10	0.03	0.13	0.7	0.8	
Tomato juice	0.09	0.04	0.13	0.6	0.8	
Peel	1.6	0.02	1.62	10.7	10	
Peeling water	0.06	< 0.01	0.07	0.4	0.4	
Peeled fruit	0.03	< 0.01	0.04	0.2	0.3	
Canned tomatoes	0.05	0.02	0.07	0.3	0.4	
Whole fruit, washed	0.11	< 0.01	0.12	0.7	0.8	
Washings	0.05	< 0.01	0.06	0.3	0.4	
Strain rest (pomace, wet)	0.57	0.05	0.63	3.8	3.9	
Raw puree	0.27	0.04	0.31	1.8	1.9	
Tomato puree	0.17	0.11	0.29	1.1	1.8	
Tomato fruit	0.10	< 0.01	0.11	–	–	09-3235, M-406443- 02-1, 09-3235-03 Germany
Raw juice	0.04	< 0.01	0.05	0.4	0.5	
Tomato juice	0.04	0.01	0.05	0.4	0.5	
Peel	0.49	< 0.01	0.5	4.9	4.5	
Peeling water	0.01	< 0.01	0.02	0.1	0.2	
Peeled fruit	< 0.01	< 0.01	< 0.02	0.1	0.2	
Canned tomatoes	0.04	< 0.01	0.05	0.4	0.5	
Whole fruit, washed	0.06	< 0.01	0.07	0.6	0.6	
Washings	0.02	< 0.01	0.03	0.2	0.3	
Strain rest (pomace, wet)	0.45	0.08	0.54	4.5	4.9	
Raw puree	0.08	0.03	0.11	0.8	1.0	
Tomato puree	0.06	0.05	0.12	0.6	1.1	
Tomato fruit	0.43 (0.26, 0.57, 0.45)	< 0.02 (< 0.02, < 0.02, < 0.02)	0.45	–	–	98W13074, M-238681- 01-1 USA
Puree	0.75 (0.76, 0.69, 0.79)	0.16 (0.18, 0.14, 0.17)	0.93	1.7	2.1	
Paste	1.15 (1.15, 1.16, 1.14)	0.28 (0.28, 0.27, 0.3)	1.5	2.7	3.3	

Processing of potatoes

The first potato processing study was conducted in Germany (98-699, M-166638-01-1) in which two trials were performed on potato in 1998. Fenamidone was applied to potato plants by twelve foliar applications at the dose rate of 0.14–0.15 kg ai/ha with a spray interval of 7 days and a PHI of 6 days. The harvested potatoes were peeled and the peels collected. The separate samples of peel and peeled

tubers were then homogenised by grinding. No residues above the LOQ of 0.005 mg/kg could be determined in tubers (RAC), peeled tubers and in peel and no processing factors could be calculated.

The second potato processing study was conducted in Washington (USA) in 1998 (98W13063, M-240410-01-1). Potato plants from a single trial received six foliar applications of fenamidone at a rate of ca. 1 kg ai/ha. Tubers were collected 14 days after the last application and processed using procedures that simulated commercial practices. Duplicate treated samples of whole potato tubers, potato flakes, potato chips (crisps) and potato wet peel were collected for analysis. The results are shown in Table 92.

Table 92 Residues of fenamidone in processed potato commodities

RAC / Processed commodity	Residues, mg/kg			Processing factor	
	Fen	RPA 410193	Sum	Parent	Sum
Potato tuber (RAC)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.04	–	–
Potato flakes	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.04	–	–
Potato chips (crisps)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.04	–	–
Potato wet peel	0.046 (0.049, 0.043)	< 0.02 (< 0.02, < 0.02)	0.066	> 2.3	> 1.7

Washing and cooking of vegetables

Trials were submitted of washing and/or cooking of cabbages, broccoli, peppers, mustard greens and spinach conducted in the USA. The results and the calculated processing factors (for dietary intake purposes only) are presented in Table 93. The information submitted on processing procedures is summarized as follows.

Cooking of cabbage

Bulk samples of cabbage, consisting of composites of at least 12 plants, were taken 2 days following the last application. The outer wrapper leaves were removed from the cabbage heads, which were then washed and cooked as in normal household practice. The outer 3 to 4 leaves were removed and discarded (because there was apparent pest infestation) until a tight head (similar to those sold in the grocery store) was obtained. The heads were rinsed under a stream of cool to lukewarm tap water for 30 sec and allowed to dry for 2 min. The cabbage was cut into smaller pieces and the core and stem ends were removed. Leaves were trimmed if they were discoloured. The cabbage was cooked for 2 min uncovered in water, followed by 8 min of further cooking in the boiling water after covering the pot. The cabbage was strained, allowed to cool, and transferred into a plastic bag for frozen storage up to analysis.

Washing and cooking of broccoli

Bulk samples of broccoli, consisting of composites from at least 12 plants, were collected 2 days following the last application.

To generate washed broccoli, the leaves were removed and discarded but stalks were not trimmed. The heads were rinsed under a stream of cool to lukewarm tap water for 30 sec and allowed to dry for 2 min.

To generate cooked broccoli, the broccoli was trimmed to florets (approximately 15 mm), discarding the stalks. The florets were rinsed under a stream of cool to lukewarm tap water for 30 sec and allowed to dry for 2 min. The florets were then cooked for 8 min uncovered. The florets were strained, allowed to cool, and transferred into a plastic bag for frozen storage up to analysis.

Cooking of peppers

Bulk samples of peppers, consisting of at least 36 fruit from at least 12 plants, were collected 2 days following the last application.

To generate washed peppers, the stem, husk, core and seeds were removed and discarded. The fruit were quartered and rinsed under a stream of cool to lukewarm tap water for 30 sec and allowed to dry for 2 min. The pieces were transferred into a plastic bag for frozen storage.

To generate cooked peppers, the stem, husk, core and seeds were removed and discarded. The fruit were quartered and rinsed under a stream of cool to lukewarm tap water for 30 sec and allowed to dry for 2 min, and cut into strips of approximately 5 mm width. The strips of pepper were cooked in boiling water for 6 to 7 min. The strips were strained, allowed to cool, and transferred into a plastic bag for frozen storage up to analysis.

Cooking of mustard greens

Bulk samples of mustard greens (at least 4 kg, from at least 12 plants) were collected at BBCH 19, 2 days following the last application.

To generate washed mustard greens, the leaves were rinsed in cool to lukewarm tap water in a sink for 30 sec, agitating by hand to ensure that all leaf surfaces were rinsed. The water was drained from the sink, and the rinsing procedure repeated twice more. The leaves were allowed to drain for 2 minutes. The leaves were transferred into a plastic bag for frozen storage.

To generate cooked mustard greens, the leaves were rinsed in cool to lukewarm tap water in a sink for 30 sec, agitating by hand to ensure that all leaf surfaces were rinsed. The water was drained from the sink, and the rinsing procedure repeated twice more. The drained leaves were placed into a pan containing sufficient boiling water to entirely cover the sample, and cooked, covered, with occasional stirring. After 9 minutes cooking, the contents of the pan were strained, allowed to cool, and transferred into a plastic bag for frozen storage.

Cooking of spinach

Bulk samples of spinach were collected 2 days following the last application.

To generate washed spinach, the leaves were washed in a sink and dried in a salad spinner. The leaves were transferred into a plastic bag for frozen storage.

To generate cooked spinach, the leaves were washed in a sink and dried in a salad spinner. The samples were then cooked for 5 minutes and strained, before being allowed to cool and transferred into a plastic bag for frozen storage.

Table 93 Residues of fenamidone in vegetables after cooking

RAC/processed commodity	Residues, mg/kg			Processing Factor (Sum)	Reference
	Fenamidone	RPA 410193	Sum		
Cabbage	0.36 (0.31, 0.44, 0.33)	< 0.02 (< 0.02, < 0.02, < 0.02)	0.36	—	201225, M-243561-01-3, YW19CB01-YW010-03H, USA (IL), 2003
Cooked cabbage	< 0.02 (< 0.02, < 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02, < 0.02)	< 0.02	< 0.06	
Broccoli, untrimmed	0.71 (0.74, 0.69, 0.71)	< 0.02 (< 0.02, < 0.02, < 0.02)	0.71	—	
Washed broccoli	0.43 (0.52, 0.36, 0.40)	< 0.02 (< 0.02, < 0.02, < 0.02)	0.43	0.61	201225, M-243561-01-3, YW19BR01-YW002-03D, USA (CA), 2003
Cooked broccoli ^a	0.97 (0.88, 1.06, 0.96)	< 0.02 (< 0.02, < 0.02, < 0.02)	0.97	1.4	
Peppers	0.62 (0.61, 0.64, 0.61)	< 0.02 (< 0.02, < 0.02, < 0.02)	0.62	—	
Washed peppers	0.31 (0.31, 0.28, 0.33)	< 0.02 (< 0.02, < 0.02, < 0.02)	0.31	0.5	201227, M-260625-01-1, YW19PP01-YW045-03H, USA (CA), 2003
Cooked peppers	0.13 (0.13, 0.14, 0.12)	< 0.02 (< 0.02, < 0.02, < 0.02)	0.13	0.21	
Mustard greens	9.0 (9.25, 9.09, 8.66)	0.04 (0.04, 0.04, 0.04)	9.04	—	201226, M-260567-01-1, YW19MG01-YW035-03H, USA (FL), 2003
Washed mustard greens	7.22 (7.88, 6.49, 7.29)	0.03 (0.03, 0.03, 0.03)	7.25	0.8	
Cooked mustard greens	3.58 (3.83, 3.76, 3.15)	< 0.02 (< 0.02, < 0.02, < 0.02)	3.6	0.4	
Spinach	0.11 (0.11, 0.11, 0.11)	< 0.02 (< 0.02, < 0.02, < 0.02)	0.11	—	201214, M-242541-01-1,

RAC/processed commodity	Residues, mg/kg			Processing Factor (Sum)	Reference
	Fenamidone	RPA 410193	Sum		
Washed spinach	0.06 (0.06, 0.06, 0.05)	< 0.02 (< 0.02, < 0.02, < 0.02)	0.06	0.55	YW19SP01-YW054-03H, USA (CA), 2003
Cooked spinach	< 0.02 (< 0.02, 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02, < 0.02)	< 0.02	< 0.18	

^a Trimming the broccoli to florets and washing and cooking the florets in water resulted in an increase of the residue.

The best estimates of the processing factors for parent residues (for MRL setting in case of residue increasing) and for the sum of fenamidone and RPA 410193 (for dietary intake) are summarized in Table 94 and 95.

Table 94 Summary of processing factors for fenamidone residues

RAC	Processed commodity	Calculated processing factors	
		Fenamidone	Best estimate
Grapes, red and white	Must	< 0.04, 0.275, 0.52, 0.65, <u>0.91</u> , 1.17, 1.31, 1.96, 2.55	0.91 (median)
	Pomace, wet	0.42, <u>2.0</u> , 3.45	2.0 (median)
Tomatoes	Puree	0.6, 1.1, <u>1.7</u> , 1.8, 3.0	1.7 (median)
	Ketchup	1.6	1.6
	Paste	2.4, 2.7	2.55 (mean)
	Pomace, wet	3.8, <u>4.5</u> , 6.3	4.5 (median)
Potatoes	Wet peel	>2.3	>2.3

Table 95 Summary of processing factors for sum of fenamidone and diketo-fenamidone

RAC	Processed commodity	Calculated processing factors	
		Sum of fenamidone + RPA 410193	Best estimate
Grapes, red and white	Juice	0.19, 0.26, 0.28, 0.29, <u>0.33</u> , <u>0.39</u> , 0.41, 0.42, 0.52, 0.8	0.36 (median)
	Must	0.07, 0.4, 0.56, 0.64, 0.74, <u>0.83</u> , 1.0, 1.1, 1.4, 1.9, 2.4	0.83 (median)
	Wine, red and white	0.29, 0.36, 0.39, 0.39, 0.46, 0.46, 0.5, 0.51, 0.52, 0.58, 0.65, 0.66, 0.67, <u>0.7</u> , <u>0.72</u> , 0.76, 0.77, 0.83, 0.89, 0.91, 0.92, 0.93, 1.0, 1.1, 1.26, 1.3, 1.5, 1.6	0.71 (median)
	Pomace, wet	0.46, <u>2.4</u> , 3.6	2.4 (median)
Tomatoes	Juice	0.5, <u>0.8</u> , <u>0.8</u> , 0.9	0.8 (median)
	Puree	1.1, 1.8, <u>2.1</u> , 2.1, 3.0	2.1 (median)
	Ketchup	2.4	2.4
	Paste	3.3, 4.0	3.65 (mean)
	Canned tomatoes	0.4, <u>0.4</u> , <u>0.5</u> , < 0.6	0.45 (median)
	Pomace, wet	3.9, <u>4.9</u> , 5.2	4.9 (median)
Potatoes	Wet peel	>1.7	>1.7
Cabbage, broccoli, peppers, spinach, mustard greens	Washed vegetables	0.5, <u>0.55</u> , <u>0.61</u> , 0.8	0.58 (median)
	Cooked vegetables	< 0.06, < 0.18, <u>0.21</u> , 0.4, 1.4	0.21 (median)

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

Dairy Cattle

One cattle feeding study was conducted (00W25043, M-238835-01-1). Nine lactating Holstein dairy cows were divided into three groups. Animals were treated once daily with gelatine capsules by oral bolus containing fenamidone for 35 consecutive days. One cow was maintained as control. The first group received an amount of fenamidone equivalent to 0.8 ppm, the second was fed 2.4 ppm, and the last group received 8 ppm in the diet. All cows were sacrificed after 35 days of dosing.

Milk samples from study days 18 through 35, the period when residue levels would be at their maximum, were analysed in 29 days or less. Milk samples from the last collection period were analysed 8 days after collection. All tissue samples were analysed within 29 days of collection. Therefore, storage stability data are not necessary.

Milk samples were collected twice daily and composites prepared from the p.m. milks of one day and the a.m. milks of the following day. All samples were frozen at -20°C before analysis. Milk samples from study days 18 to 35, the period when residue levels would be at their maximum, were analysed within 29 days of sampling, milk samples from the last collection period were analysed within 8 days of collection. Following oral administration to lactating cows for 35 consecutive days, at all sampling intervals, no residues of fenamidone or the two major metabolites desanilino-diketo-fenamidone (RPA 412636) and desanilino-fenamidone (RPA 412708) were found in any of the whole milk samples (see Table 96). The LOQ for fenamidone and each of its metabolites in milk is 0.01 mg/kg. The limit of detection (LOD) is 1/3 the LOQ or 0.003 mg/kg.

Table 96 Residues in whole milk following 35 days oral administration of fenamidone to dairy cows

Day	Residues, mg/kg (mean of 3 cows)					
	Control			High dose (8 ppm in the diet)		
	Fenamidone	RPA 412636	RPA 412708	Fenamidone	RPA 412636	RPA 412708
-1	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003
1	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003
4	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003
8	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003
11	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003
15	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003
18	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003
25	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003
28	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003
34	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003

A portion of the day-34 milk from all groups was centrifuged to separate the milk fat for analysis. Analysis of milk samples from the 8 ppm dose group resulted in fenamidone residues below the LOQ (0.01 mg/kg) in every case. Therefore, milk fat samples from the lower dose groups were not prepared.

Samples of liver, kidney, muscle and fat were collected immediately after sacrifice and frozen at -20°C until analysis. All tissue samples were analysed for fenamidone and the two major metabolites within 29 days of collection. At all sampling intervals, no residues of fenamidone or the two major metabolites were found in any of the tissue or milk fat samples, with the exception of a residue of desanilino-fenamidone (RPA 412708) detected in one milk fat sample in the highest dose group (0.011 mg/kg). Analysis of tissue and milk fat samples from the 8 ppm dose group resulted in fenamidone residues generally below the LOQ (0.05 mg/kg), therefore, tissue samples from the lower dose groups were not analysed. A summary of the results is presented in Table 97.

Table 97 Mean residues in tissues and milk fat following 35 days oral administration to dairy cows

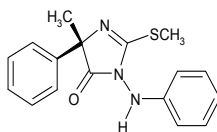
Compound	Residues in mg/kg of 3 cows dosed with 8 ppm in the diet for 35 days				
	Muscle	Liver	Kidney	Fat	Milk fat
Fenamidone	< 0.02 (3)	< 0.02 (3)	< 0.02 (3)	< 0.02 (3)	< 0.003 (3)
RPA 412636	< 0.02 (3)	< 0.02 (3)	< 0.02 (3)	< 0.02 (3)	< 0.003 (3)
RPA 412708	< 0.02 (3)	< 0.02 (3)	< 0.02 (3)	< 0.02 (3)	< 0.01, 0.011, < 0.01

APPRAISAL

Fenamidone is a broad-spectrum fungicide belonging to the imidazolinone group. The compound was evaluated the first time by the 2013 JMPR for toxicology where an ADI of 0–0.03 mg/kg bw and an ARfD of 1 mg/kg bw was allocated. The evaluation for residues was scheduled for the 2014 JMPR.

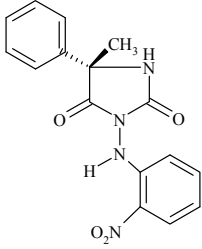
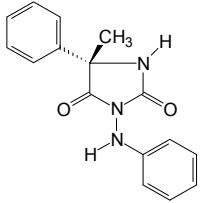
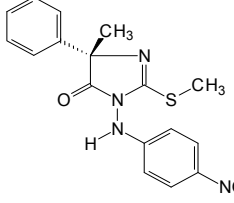
The current Meeting received information on physical and chemical properties, metabolism studies on animals and plants, environmental fate including rotational crops data, analytical methods, use pattern, supervised trials data, processing and feeding studies.

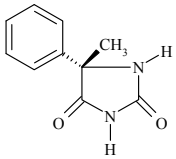
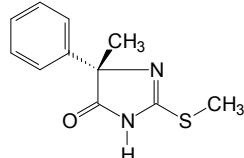
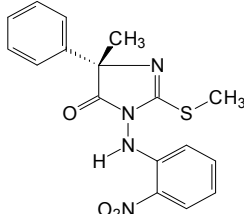
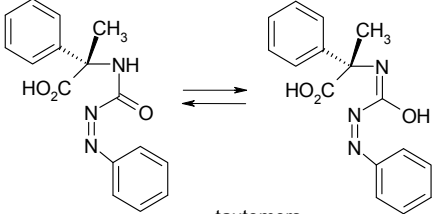
The active substance fenamidone is the S-enantiomer of a stereoisomeric molecule with the chiral centre in the 5-position of the dihydro-imidazolone ring. This S-enantiomer has been shown to be the biologically (fungicidally) active enantiomer.



The chiral carbon atom on the 5-position of the dihydro-imidazolone ring is substituted by 4 non-hydrogen substituents, namely substituted by an amino, carbonyl, methyl and phenyl group. Prerequisite for isomerisation/racemisation of this type of centre of chirality is the presence of a hydrogen atom as a substituent that can be split off easily. In fenamidone, no hydrogen is linked to the chirality centre. Therefore, racemisation is chemically not possible and the configuration and the optical purity, respectively, established by synthesis does not change afterwards. There was also no evidence of enantiomerization or racemization of the parent substance and its metabolites during metabolic conversions in plants and animals or physical-chemical degradation in soil and water.

In this document, the code names of the S-enantiomers were used of the metabolites identified:

Name, Structure, IUPAC name, CAS name, [CAS number]	Mol. formula, molar mass Other names / codes	Occurrence, Compartment
RPA 221701  (S)-5-methyl-3-[(2-nitrophenyl)amino]-5-phenyl- 2,4-imidazolidinedione (IUPAC)	$C_{16}H_{14}N_4O_4$ 326.3 g mol ⁻¹ S-Enantiomer: RPA 221701 AE 0591777 BCS-AX84897 2,4-imidazolidinedione, 5-methyl-3-[(2-nitrophenyl)-amino]-5-phenyl-, (S)- <u>Racemate:</u> RPA 410995 AE 0591778	Soil, aerobic Carrot Rotational crops: Turnip, Swiss chard
RPA 410193  (S)-5-Methyl-5-phenyl-3-(phenylamino)- 2,4-imidazolidinedione (IUPAC)	$C_{16}H_{15}N_3O_3$ 281.3 g mol ⁻¹ S-Enantiomer: RPA 410193 AE 0540049 BCS-AX71129 Diketo-fenamidone (DK-Fen) <u>Racemate:</u> RPA 405862 AE C650488	Soil, aerobic Soil, photolysis Hydrolysis, abiotic Photolysis, buffer Vine grapes, Lettuce, Tomato, Potato, Carrot Hen
RPA 411639  (S)-5-methyl-2-(methylthio)-3-(4-nitrophenylamino)-5-phenyl-3,5-dihydro-4H-imidazol-4-one (IUPAC)	$C_{17}H_{16}N_4O_3S$ 356.4 g mol ⁻¹ S-Enantiomer: RPA 411639 AE 0540054 BCS-AX71134 <u>Racemate:</u> RPA 406012 AE 0540056	Soil, aerobic Carrot Rotational crops: Turnip, Swiss chard Rat (postulated intermediate)
RPA 412636	$C_{10}H_{10}N_2O_2$ 190.2 g mol ⁻¹	Soil, aerobic Soil, photolysis

Name, Structure, IUPAC name, CAS name, [CAS number]	Mol. formula, molar mass Other names / codes	Occurrence, Compartment
 (S)-5-methyl-5-phenyl-2,4-imidazolidine-dione (IUPAC) 2,4-imidazolidinedione, 5-methyl-5-phenyl-, (5S) (CAS)	S-Enantiomer: RPA 412636 AE 0540051 BCS-AX71131 “fenamidone-hydantoin” Desanilino-diketo-fenamidone (DADK-Fen) <u>Racemate</u>: RPA 717879 AE C415557	Water / sediment Lettuce, Potato, Rotational crops: Wheat, Turnip, Swiss chard Rat, Goat, Hen
RPA 412708  (5S)-5-methyl-2-(methylthio)-5-phenyl-3,5-dihydro-4H-imidazol-4-one (IUPAC)	C₁₁H₁₂N₂OS 220.9 g mol⁻¹ S-Enantiomer: RPA 412708 AE 0540050 BCS-AX71130 Desanilino-fenamidone (DA-Fen) <u>Racemate</u>: RPA 408056 AE 0540057	Soil, aerobic Soil, photolysis Hydrolysis, abiotic Photolysis, buffer Photolysis, nat. water Water / sediment Lettuce, Potato Rat, Goat, Hen
RPA 413255  (5S)-5-methyl-2-(methylthio)-3-(2-nitrophenylamino)-5-phenyl-3,5-dihydro-4H-imidazol-4-one (IUPAC)	C₁₇H₁₆N₄O₃S 356.4 g mol⁻¹ S-Enantiomer: RPA 413255 AE 0540053 BCS-AX71133 <u>Racemate</u>: RPA 410914 AE 0540055	Soil, aerobic Soil, photolysis Carrot Rotational crops: Turnip, Swiss chard
RPA 413350  tautomers 2-phenyl-N-[(E)-phenyldiazenyl]carbonyl-D-alanine (IUPAC)	C₁₆H₁₅N₃O₃ 297.3 g mol⁻¹ S-Enantiomer: RPA 413350 AE 0540052 BCS-AX71132 <u>Racemate</u>: RPA 409344 AE 0841910	Hydrolysis, abiotic

Animal metabolism

Metabolism studies on rats reviewed by the 2013 JMPR show that fenamidone undergoes extensive metabolism in the rat by phase I (oxidation, reduction and hydrolysis) and phase II reactions (conjugation). The plasma elimination half-life was at least 60 hours. More than 20 metabolites were detected in rat excreta, with the majority of radioactivity excreted in the faeces (up to approximately 90% of the administered dose) and the remainder in urine. Mass balance data indicated that the majority of radioactivity (> 80%) was eliminated within 48 hours of dosing. The metabolites RPA 412636 and RPA 412708 were present in rat urine and rat bile, respectively.

Metabolism studies have been conducted in lactating goats. Two dose levels were used for each label position: 1 ppm and 10.4 ppm in the diet for [C-phenyl-U-¹⁴C]-fenamidone and 1.5 ppm and 11.5 ppm in the diet for [N-phenyl-U-¹⁴C]-fenamidone. Dosing was performed twice daily by capsule during seven consecutive days. The total administered radioactivity (TAR) was quickly and almost completely eliminated in the excreta. In case of the C-phenyl label, the total excreted residues were 99–102% of TAR (75–80% via faeces, 17–26% via urine, 0.1% via milk). In case of the N-

phenyl label, the total excreted residues were ca. 90% of TAR (45–52% via faeces, 36–40% via urine, 0.1–0.2% via milk). Neither fenamidone nor any of its metabolites accumulated in milk fat or milk proteins. At sacrifice, small amounts of radioactivity (totally 0.6 to 1.0% of TAR) were detected in edible organs/tissues and blood.

Residues in milk and edible tissues of goats were low. RPA 412708 and RPA 412636 were found, as well as hydroxylated fenamidone and hydroxylated RPA 412708. RPA 412636 was the most abundant residue in the goat, reaching after high dose administration 0.055 mg/kg (5.9% TRR) in liver, 0.018 mg/kg (15% TRR) in kidney and 0.002 mg/kg in milk (11% TRR). Fenamidone was identified in fat with 0.013 mg/kg (53% TRR), but was a minor component in milk (0.001 mg/kg, 0.7% TRR), liver (0.003 mg/kg, 0.3% TRR) and kidney (0.001 mg/kg, 0.6% TRR).

Metabolism studies have been conducted in laying hens. Two dose levels were used for each label position: 1.3 ppm and 13.8 ppm in the diet for [C-phenyl-U-¹⁴C]-fenamidone and 1.0 ppm and 9.8 ppm in the diet for [N-phenyl-U-¹⁴C]-fenamidone. Dosing was performed by administration of one capsule per day during fourteen consecutive days. The TAR was quickly and almost completely eliminated in excreta. The recovery of radioactivity was > 91% of the TAR. The majority of the radioactivity was in excreta (approximately 90% TAR). All of the eggs together contained 0.1% of the dose administered, most of which was retained in the yolk. At sacrifice, at both dose levels and in both labels, very minor amounts of radioactivity (0.1% of TAR) were detected in edible organs/tissues and blood.

Residues in eggs and edible tissues of hens were low. RPA 412636 was a major component in the hen, found at 0.011 mg/kg (74% TRR) in egg white, 0.014 mg/kg in egg yolk (11% TRR), 0.023 mg/kg (15% TRR) in liver and 0.002 mg/kg (16% TRR) in skin after high dose administration. RPA 412708 was the highest residue in the hen, reaching 0.028 mg/kg (25% TRR) in egg yolk but only 0.002 mg/kg (1.3% TRR) in liver. Fenamidone was identified, but was a minor component (egg yolk 0.014 mg/kg, 11% TRR; liver 0.003 mg/kg, 1.7% TRR; fat < 0.001 mg/kg, 4.6% TRR and skin 0.002 mg/kg, 14% TRR).

The Meeting concluded that, in all species investigated, the TAR was quickly and almost completely eliminated in excreta. The metabolic profiles differ quantitatively between the species, but qualitatively there are no major differences; the routes and products of metabolism in animals were consistent across the studies. Fenamidone, RPA 412708 (desanilino-fenamidone) and RPA 412636 (desanilino-diketo-fenamidone) were the components identified.

Plant metabolism

The metabolism of fenamidone has been studied in grapes, tomatoes, lettuce, carrots and potatoes.

Grapes

Following four foliar treatments of [C-phenyl-U-¹⁴C]-fenamidone to a total nominal rate of the equivalent of 1.65 kg ai/ha (0.5, 0.49, 0.5 and 0.16 kg ai/ha) to field grown grape vines during the grape berry development period, the terminal residue in the mature harvest grape bunches (1.2 mg eq/kg) was shown to comprise mainly parent compound (56% TRR) and RPA 410193 (17% TRR). The interval between the last application and harvest was 24 days. The major part of the radioactivity associated with the grape bunches could be extracted by methanol/water and represented 89% of the TRR. The non-extracted radioactivity was confined mainly to the stem, skin and pips. Further extraction procedures were made including enzyme treatment followed by acid and alkali hydrolysis (100% TRR).

The metabolism of fenamidone in grapes was characterised by the formation of RPA 410193, i.e. loss of the thiomethyl group and formation of an imidazolidinedione. Some evidence was obtained to suggest that hydroxylation of parent compound (3.4% TRR) as well of RPA 410193 (4.2% TRR) also occurred. A number of other metabolites were detected, albeit polar and at very low levels.

Tomatoes

The metabolism was investigated following three foliar applications of [^{14}C]-fenamidone, each of 0.5 kg ai/ha giving a total nominal application rate of 1.5 kg ai/ha on glasshouse grown tomatoes. Both labels, C-phenyl and N-phenyl, were used separately. At final harvest, 7 days after the last treatment, the TRR in tomato fruits was less than 0.2 mg eq/kg for both labels. About 90% of the TRR was extracted and more than 75% identified.

The major component of the extracted radioactivity in both treatment regimes was unchanged parent accounting for 66–76% TRR. The next largest component was RPA 410193 accounting for 9.3–9.4% TRR. No metabolites were formed at significant levels as a result of cleavage of the two phenyl-rings.

Lettuce

The metabolism was investigated following four foliar applications of [^{14}C]-fenamidone, each of a nominal rate of 0.4 kg ai/ha on outdoor grown iceberg lettuce. Both labels, C-phenyl and N-phenyl, were used separately. At final harvest, 7 days after the last treatment, a total of 93% and 92% of the TRR for the C-phenyl-label and N-phenyl-label treated final harvest lettuces respectively, was identified. The TRR found in the lettuce wrapper leaves (12 mg eq/kg) were significantly higher than that present in the lettuce heads (0.2–0.3 mg eq/kg).

Analysis of the extracted radioactive residues showed the major components of both labels to be parent fenamidone (about 92% TRR in whole lettuce). The remaining extracted radioactivity was comprised of RPA 410193 (0.59–0.66% TRR) and multiple unidentified polar components, which comprised less than 3% TRR in total in whole lettuce. In addition, low levels of RPA 412636 (2.7% TRR) were present in the C-phenyl-label treated lettuce head leaves extracts only.

Potatoes

The metabolism was investigated following three foliar applications of [^{14}C]-fenamidone, each of nominal 0.5 kg ai/ha on outdoor grown potatoes. Both labels, C-phenyl and N-phenyl, were used separately. At final harvest, 14 days after the last treatment, the TRR in the tubers (0.038–0.087 mg eq/kg) was significantly lower than the levels detected in the leafy part of the plant (5.9–6.6 mg eq/kg) with 1.3% of the whole plant TRR being present in the root/tuber. A total of 73% TRR of the intact potato tubers and 77% TRR of the potato haulm could be extracted for C-phenyl-fenamidone treated plants at final harvest. The corresponding values for N-phenyl-fenamidone treated potatoes were 46% and 78% for intact potato tubers and potato haulm respectively.

In potato tubers, parent fenamidone accounted for 2.3 to 6% of TRR (0.002 mg/kg) and two further metabolites were formed: RPA 412708 (desanilino-fenamidone) and RPA 412636 (desanilino-diketo-fenamidone), each accounting for ca. 6% of TRR with low absolute concentrations (0.005–0.006 mg eq/kg). A large portion of the residue was polar in nature and reported to be composed by acid labile conjugates.

Carrots

The metabolism was investigated following three applications of [N-phenyl- ^{14}C]-fenamidone, each of nominal 0.3 kg ai/ha on outdoor grown carrots. The first application was made pre-emergence followed by two foliar applications. At final harvest, 14 days after the last treatment, the TRR in leaves and roots amounted to 30.5 and 0.04 mg eq/kg, respectively. Therefore, basispetal transport of residues from the treated leaves to the roots was very limited. In leaves, 81% of the radioactivity could be extracted with acetonitrile/water using a high-speed blender and the rest by water/dichloromethane partition. In roots, 93% of the radioactivity was extracted with acetonitrile/water.

Fenamidone accounted in leaves for 89% of the TRR and in roots for 29% of the TRR. Six minor metabolites were identified. No metabolite in the roots exceeded a level of 0.01 mg/kg. Two parallel metabolic reactions were found and a combination thereof. The first reaction was an oxidative hydrolysis of the thiomethyl group at the dihydro imidazole ring to a keto substituent resulting in RPA 410193 (diketo-fenamidone). Another reaction was the nitration of the N-phenyl ring. All metabolites

contained the intact basic structure with the two phenyl rings and the imidazole ring. No cleavage product, such as aniline, aminophenols or nitroanilines was detected. In addition, radiolabelled glucose was found in the leaves resulting from complete mineralization of the fenamidone in the soil and photosynthetic uptake of the formed $^{14}\text{CO}_2$ in the plant. Small amounts of a dimer of fenamidone (0.17% of TRR, 0.05 mg/kg) were also detected in leaves.

In summary, the metabolism of fenamidone in plants after foliar application was investigated in three crop categories: fruits and fruiting vegetables, leafy vegetables, root and tuber vegetables. The metabolic pattern was shown to be similar in all these crop groups with the unchanged parent compound being the main compound of the final residue at harvest. The Meeting concluded that after foliar treatment the only significant metabolite in plants was RPA 410193 (diketo-fenamidone), formed by oxidative hydrolysis of the thiomethyl side chain of fenamidone.

Environmental fate

For fenamidone, data were received for foliar spray on permanent crops and on annual crops. A further application is on cotton seed as in furrow treatment. Therefore, according to the FAO manual, studies on the aerobic degradation in soil, photolysis, hydrolysis, rotational crops (confined, field) and field dissipation were evaluated. The fate and behaviour of fenamidone in soils was investigated using fenamidone radio-labelled in two different positions, [C-phenyl- $\text{U-}^{14}\text{C}$]- and [N-phenyl- $\text{U-}^{14}\text{C}$]-fenamidone.

Degradation in soil, photolysis and hydrolysis

Degradation of fenamidone in soil primarily proceeds by the action of aerobic soil microorganisms. In the first major pathway there is loss of the N-phenyl aniline ring to form RPA 412708, followed by loss of the S-methyl moiety and hydrolysis to form RPA 412636. Two further pathways are the concurrent formation of 2- and 4-nitro compounds by addition to parent to form RPA 413255 and RPA 411639. A further route of degradation results in the formation of numerous minor non-polar components. The rate of degradation tested in silty clay loam, sandy loam and in clay loam, is similar in both C-phenyl and N-phenyl rings with DT50s ranging from 0.9 to 9.6 days in all tested soils at 20 °C. The Meeting concluded that fenamidone was rapidly degraded in soil under aerobic conditions leading to the formation of two major metabolites RPA 412708 and RPA 412636.

Field dissipation studies undertaken at four European sites showed that the average half-lives were 5 days for fenamidone, 12 days for RPA 412708, 21 days for RPA 411639, 43 days for RPA 413255 and 47 days for RPA 412636. It can be concluded that the dissipation of fenamidone was rapid in the field.

The photolytic degradation of fenamidone was investigated on a sandy loam soil for 30 days. Artificial sunlight was provided using an artificial xenon light source for 13 hours each day. The Meeting recognized that photolytic processes do not contribute significantly to the degradation of fenamidone applied to the soil surface.

The hydrolytic degradation of fenamidone was examined in aqueous buffered solutions at pH values of 4, 5, 7 and 9 under sterile conditions at 25 °C. The Meeting recognized that fenamidone was stable at pH 5 and pH 7, but was hydrolysed at pH 4 and pH 9.

Rotational crops

One confined radiolabelled succeeding crop study was conducted in 2001 with [C-phenyl- $\text{U-}^{14}\text{C}$]-fenamidone. Following application to the soil at an application rate of 1.6–2.0 kg ai/ha, lettuce, turnip and barley were cultivated after three plant back intervals (30, 120/150 and 365 days). The residues in rotated plants comprised the following major metabolites: RPA 412636 and RPA 412708 and a conjugate thereof. The parent substance could not be detected. Residues of RPA 412636 were in lettuce ca. 20% of TRR at each plant back intervals, in turnip tops 3.5–11% of TRR, in turnip roots 5–8% of TRR and in barley grain 2–6% of TRR. The highest residues of RPA 412636 were found in barley straw with 2.04 mg/kg (29% TRR). RPA 412708 was found in lettuce at a maximum of 5.7% of TRR (0.04 mg/kg).

Two additional confined radiolabelled succeeding crop studies were carried out in 2013. Following application of 0.96 kg ai/ha [N-phenyl-U-¹⁴C]-fenamidone and 0.97 kg ai/ha [C-phenyl-U-¹⁴C]-fenamidone to the soil, wheat, turnip and Swiss chard were sown and cultivated at three plant back intervals (30, 191 and 324 days after soil treatment). Both labelled forms of fenamidone were rapidly degraded in soil with extensive uptake of the metabolites into the plants. The parent compound was only detected in Swiss chard at 30 days plant back interval, however at a low level amounting to a maximum of 0.002 mg/kg (0.2% of TRR). It was not detected in other crops. The pathway is proposed as follows: Electrophilic nitration of the aniline ring formed fenamidone-2-nitro and fenamidone-4-nitro, most probably in the soil, with RPA 413255 (fenamidone-2-nitro) being further converted to RPA 221701 (fenamidone-desthiomethyl-2-nitro) following nucleophilic substitution and the addition of water. Conjugation with hexose and malonic acid and hydroxylation lead to the formation of fenamidone-hydroxy-nitro-malonyl-glycoside. Further conjugation with glutathione and nucleophilic substitution of methyl mercaptan also lead to the formation of fenamidone-nitro-GSH. In addition to these metabolites, the C-phenyl-labelled compound underwent further degradation of the aniline moiety of fenamidone-desthiomethyl-2-nitro to RPA 412636, again, most likely in the soil. Conjugation of RPA 412636 with glucose, hexose plus malonic acid and serine formed fenamidone-hydantoin-glucoside and fenamidone-hydantoin-serine.

The Meeting concluded that parent fenamidone is quickly degraded in soil or in the follow-on crops, not resulting in residues detected at harvest. Residues of RPA 412636, RPA 412708 and conjugates thereof were the major metabolites in commodities used as animal feed and can also occur in the edible parts of rotational crops.

Seven field succeeding crop studies were conducted, one in Europe and six in the USA. Spray applications near GAP were made to bare soil to simulate a treatment of target crops like potatoes, leafy vegetables or fruiting vegetables. Rotational crops were cereals (wheat, maize), fruiting vegetables (sweet corn) root and tuber vegetables (turnip, radish), leafy vegetables (lettuce, spinach), pulses (soya bean, dry) and strawberries.

After application of 0.9–1.2 kg ai/ha per annum to the bare soil, in the follow-on crops no parent fenamidone or its plant metabolite RPA 410193 was found at or above the LOQ of 0.02 mg/kg.

In plant parts used as human food, RPA 412636 occurred up to maximum single values of 0.04 mg/kg in radish roots, 0.04 mg/kg in radish tops and 0.12 mg/kg in spinach. Residues of RPA 412636 in wheat grain were in 22 field trials < 0.02 mg/kg and in only one trial 0.061 mg/kg.

RPA 412708 and RPA 412636 occurred in commodities that may be used as animal feeds. The sum of RPA 412708 and RPA 412636 reached up to 0.14 mg/kg in soya bean hay, 0.29 mg/kg in sweet corn stover and 0.21 mg/kg in field corn forage; RPA 412636 occurred up to 0.07 mg/kg in wheat forage, 0.27 mg/kg in wheat straw and 0.45 mg/kg in wheat hay.

In conclusion, no residues of fenamidone and RPA 410193 above the LOQ of 0.02 mg/kg would be expected in follow-on crops but residues of the metabolites RPA 412708 and RPA 412636 taken up from the soil occurred in commodities that may be used as human food and animal feed.

Methods of analysis

The Meeting received descriptions and validation data for analytical methods for residues of fenamidone and its relevant metabolites RPA 410193, RPA 412708 and RPA 412636 in plant commodities and for fenamidone, RPA 412708 and RPA 412636 in animal commodities. Residue analytical methods rely on GC with NP-detection, GC-MS or LC-MS/MS. Typical LOQs achieved for plant commodities fall in the range of 0.01–0.02 mg/kg for each analyte. The LOQ for milk was 0.01 mg/kg and for animal products (liver, kidney, muscle, eggs) 0.05 mg/kg for each analyte. Methods have been subjected to independent laboratory validation.

Fenamidone and RPA 410193 were analysed in plant material by the multi method S 19 with an LOQ of 0.01 mg/kg by GC-MS. The QuEChERS multi residue method was used for fenamidone and RPA 410193 in plant matrices as well as for fenamidone and RPA 412708 and RPA 412636 in animal matrices with LOQs of 0.01 mg/kg by LC-MS/MS for each analyte.

Stability of residues in stored analytical samples

The Meeting received storage stability studies under freezer conditions at -20 °C for fenamidone, RPA 412636, RPA 412708 and RPA 410193 for the duration of the storage of 12 months in a wide range of raw and processed crop matrices, including examples of high-water and high-starch crops and for duration of 6 months for cotton products. Furthermore, studies were conducted for fenamidone and RPA 410193 in high-water content commodities for duration of 14 months and in strawberries of 18 months.

The Meeting concluded that residues of fenamidone and RPA 410193 are stable in commodities of high-water content for at least 14 months and in strawberries for at least 18 months. Residues of fenamidone, RPA 412636, RPA 412708 and RPA 410193 are also stable for at least 6 months in high-oil content products.

Because milk and tissue samples of the ruminant feeding study were analysed within 29 days of collection, no storage stability data were submitted.

Definition of the residue

Animal metabolism studies were performed in rats, lactating goats and laying hens. The metabolic behaviour of fenamidone in the rat is summarised by the 2013 JMPR and shows pathways and major components similar to those found in ruminants and poultry. RPA 412636 and RPA 412708 are found in the rat metabolism. RPA 410193, which is a plant metabolite, was not identified in the rat ADME studies. The 2013 Meeting considered the named metabolites as toxicological relevant.

The 2014 JMPR agreed that RPA 410193 is covered by the ADI of the parent fenamidone. The toxicological relevance of RPA 412636 and its precursor RPA 412708, both detected as RPA 412636, were confirmed. RPA 412636 was considered as 10 times more toxic than the parent.

Livestock metabolism studies in goat and hen showed no major differences; the routes and products of metabolism in animals are consistent across the studies. The residues in animal products were low. Fenamidone was identified, reaching 0.013 mg/kg in ruminant fat (53% TRR) and 0.014 mg/kg in egg yolk (11% TRR). RPA 412636 was the most abundant residue in the ruminant, reaching 0.055 mg/kg in liver (5.9% TRR) and 11% TRR in milk (0.002 mg/kg). RPA 412636 was also found in the hen, at 0.011 mg/kg (74% TRR) in egg white and 0.014 mg/kg in egg yolk (11% TRR). RPA 412708 was the most abundant residue in the hen, reaching 0.028 mg/kg in egg yolk and 0.002 mg/kg in liver (25 and 1.3% TRR, respectively).

The Meeting concluded that the residue definition for MRL-setting for animal products should be parent fenamidone. For dietary intake, the residue should be defined as the sum of fenamidone, RPA 412636 and RPA 412708, expressed as fenamidone. Due to the 10-fold higher toxicity, a factor of 10 should be applied.

Fenamidone has a log P_{OW} of 2.8. No residues of fenamidone, RPA 412636 and RPA 412708 could be determined in animal products from the cattle feeding study with the exception of one milk fat sample with 0.011 mg/kg of RPA 412708. In the high dose group of the goat metabolism study, the TRR in muscle were low (0.02 mg eq/kg) and could not further identified. Fenamidone was detected in the fat (53% TRR, 0.013 mg eq/kg). In the high dose group of the egg metabolism study, fenamidone was detected in the yolk (11% TRR, 0.014 mg/kg), but not in the egg white (< 0.001 mg/kg). The Meeting decided that the residue is fat-soluble.

Plant metabolism studies for foliar spray application to the plant provide a clear understanding of the fate of the fenamidone molecule, both the C-phenyl and N-phenyl portions. The overall metabolic pathway is consistent between the different crop groups. Fenamidone forms the largest part of the residue and the only significant metabolite is RPA 410193, which reached a maximum of 17% TRR in grape. RPA 412636 and RPA 412708 reached a maximum of 6.3% TRR (0.005 mg/kg) and 6.4% TRR (0.006 mg/kg) in potato tubers. No other metabolite exceeded 5% TRR in any plant commodity.

The Meeting took into account the possibility of residues of the metabolites RPA 412708 and RPA 412636 in follow-on crops used as human food and/or animal feed. The Meeting concluded to

include both metabolites in the residue definition for estimation of the dietary intake of plant commodities. Because of the 10-fold higher toxicity, a factor of 10 should be applied.

The Meeting agreed the following residue definitions:

- Definition of the residue for compliance with the MRL for plant and animal commodities: *Fenamidone*.
- Definition of the residue for estimation of dietary intake for plant commodities: Sum of fenamidone, (S)-5-methyl-5-phenyl-3-(phenylamino)-2,4-imidazolidine-dione (RPA 410193) plus $10 \times$ the sum of both (S)-5-methyl-5-phenyl-2,4-imidazolidine-dione (RPA 412636) and (5S)-5-methyl-2-(methylthio)-5-phenyl-3,5-dihydro-4H-imidazol-4-one (RPA 412708), all calculated as fenamidone.
- Residue concentration $C_{\text{total}} = C_{\text{fenamidone}} + C_{\text{RPA 410193}} + 10 \times (C_{\text{RPA 412636}} + C_{\text{RPA 412708}})$
- Definition of the residue for estimation of dietary intake for animal commodities: *Fenamidone plus $10 \times$ the sum of both (S)-5-methyl-5-phenyl-2,4-imidazolidine-dione (RPA 412636) and (5S)-5-methyl-2-(methylthio)-5-phenyl-3,5-dihydro-4H-imidazol-4-one (RPA 412708), all calculated as fenamidone.*
- Residue concentration $C_{\text{total}} = C_{\text{fenamidone}} + 10 \times (C_{\text{RPA 412636}} + C_{\text{RPA 412708}})$

The residue is fat-soluble.

Results of supervised residue trials on crops

The Meeting received supervised residue trials data for grapes, strawberries, leek, bulb onion, spring onion, cabbage, flowerhead brassica, melons, watermelons, cucumber, summer squash, peppers, tomato, lettuce, mustard greens, spinach, carrots, potato, ginseng, Witloof chicory, celery, cotton seed, sunflower seed, common bean forage and cotton fodder. If two field samples were taken or results of two replicate plots were submitted, the mean value was calculated for estimation of maximum residue levels. For HR estimation, the highest single value of the trials according to GAP was used. From two or more trials carried out side-by-side the higher residue was chosen.

Residues are reported separately for fenamidone and RPA 410193 only because the soil metabolites RPA 412636 and RPA 412708 are not relevant for foliar treated crops. For HR and STMR estimation, the sum of fenamidone (MW 311.4 g/mol) and RPA 410193 (MW 281.3 g/mol), expressed as fenamidone (conversion factor 1.11), is needed. When residues are undetectable in a commodity, the sum of the LOQs of both components is not appropriate for all plant commodities because the days after the last treatment (DALT) differ from 2 days (e. g. lettuce) to 35 days (strawberry). The residues of RPA 410193 are found in the same order of magnitude as the parent in berries harvested 4 to 5 weeks after treatment. In other plant commodities harvested at shorter PHIs (2–21 days), the level of the metabolite is much lower than the parent in most cases. The method for calculation of the total residues of the sum of fenamidone and RPA 410193 is illustrated as follows:

- Plant commodities except grapes and strawberries

Fenamidone, mg/kg	RPA 410193, mg/kg	Total, mg/kg
< 0.02	< 0.02	< 0.02
0.05	< 0.02	0.05
0.42	0.08	0.51 ^a
^a $0.42 + (0.08 \times 1.11) = 0.5088$		

- Grapes and strawberries

Fenamidone, mg/kg	RPA 410193, mg/kg	Total, mg/kg

Fenamidone

< 0.02	< 0.02	< 0.04
0.05	< 0.02	0.07
0.42	0.08	0.51

Trials from the USA or Canada on carrots, potatoes, fruiting—brassica, leafy and legume—as well as stalk and stem vegetables were performed with four instead of three applications and trials on bulb vegetables as well as cucurbits were performed with six instead of four applications. The Meeting accepted the trials as matching the GAP in the USA and Canada since comparative residue trials on grapes and tomatoes as well as decline studies on vegetables indicated that the contribution of two earlier applications to the terminal residue is negligible. The Meeting agreed to use the trials to estimate maximum residue levels.

Rotational crop maximum residue levels, STMRs and HRs

The Meeting noted that no residues of parent fenamidone above the LOQ of 0.02 mg/kg are expected in follow-on crops. It was concluded that the estimation of maximum residue levels is not necessary.

The Meeting recognized that, in commodities used as human food, RPA 412636 were found. The table below shows the highest and the mean residues of two plots found in follow crops as spinach, radish roots and leaves as well as wheat grain after treatment of vegetables with fenamidone at 1.2 kg ai/ha per annum. The Meeting agreed to use the proportionality approach and scaled the residues according to the US GAP of 0.9 kg ai/ha per annum for brassica vegetables, fruiting vegetables, leafy vegetables, root and tuber vegetables and celery. The values measured as RPA 412636 (MW 190.2 g/mol) were expressed as fenamidone (MW 311.4 g/mol) multiplying by 1.64.

Treatment, kg ai/ha	Commodity	RPA 412636, highest residue, mg/kg			RPA 412636, mean residue, mg/kg		
		Measured	Scaled	Calculated as fenamidone eq	Measured	Scaled	Calculated as fenamidone eq
6× 0.2	Spinach	0.12	0.09	0.15	0.096	0.072	0.12
	Radish tops	0.044	0.033	0.054	0.033	0.0275	0.045
	Radish roots	0.039	0.029	0.048	0.03	0.0225	0.037
	Wheat grain	0.061	0.046	0.075			
1× 0.2	Wheat grain				< 0.02		< 0.033 (n = 22)

The Meeting concluded that the contribution of residues of RPA 412636 and RPA 412708 has to be considered for the STMR and HR estimation for annual crops like vegetables and cereals. A factor of 10 is used because of the 10-fold higher toxicity compared to parent.

For brassica vegetables, fruiting vegetables, leafy vegetables, fresh herbs as well as stalk and stem vegetables, the Meeting estimated a rotational crop STMR of 1.2 mg/kg and a rotational crop HR of 1.5 mg/kg, based on the residues analysed in spinach.

For bulb vegetables, root and tuber vegetables as well as their leaves/greens the Meeting estimated a rotational crop STMR of 0.4 mg/kg and an HR of 0.5 mg/kg, respectively.

For cereal grains, residues of RPA 412636 + RPA 412708 were in wheat of 22 field trials as well as of nine trials in maize < 0.033 mg eq/kg and in only one trial 0.075 mg eq/kg. The Meeting agreed to estimate an STMR of 0.33 mg/kg (10 times LOQ) for cereal grains except rice as follow-on crops.

No residues of RPA 412636 or RPA 412708 higher than the LOQ of 0.02 mg/kg occurred in strawberries, sweet corn kernels and soya bean seed grown as follow-on crops. The Meeting concluded that the uptake of substantial concentrations of RPA 412636 or RPA 412708 by strawberry, sweet corn, oil seeds and pulses is negligible. No STMR or HR was recommended.

Grapes

The GAP for fenamidone in Brazil on grapes is foliar spray treatment with 3×0.13 kg ai/ha and a PHI of 7 days. Three Brazilian trials matching the GAP were submitted. The residues in grape bunches were for parent fenamidone 0.02, 0.03, 0.03 mg/kg and for the sum of fenamidone and RPA 410193 0.04, 0.05, 0.05 mg/kg.

Fenamidone is registered for foliar spray treatment on grapes in the Czech Republic with application at 3×0.13 kg ai/ha and a PHI of 28 days. Sixteen European trials according to GAP were conducted in 2009 and 2010 in Belgium, France, Germany, Italy, Portugal and Spain. The residues in grape bunches were for fenamidone ($n = 16$) 0.04, 0.06, 0.08, 0.09, 0.09, 0.10, 0.13, 0.13, 0.17, 0.21, 0.22, 0.22, 0.26, 0.28, 0.30 and 0.33 mg/kg. The total residues (sum of fenamidone and RPA 410193) were 0.06, 0.09, 0.09, 0.11, 0.12, 0.13, 0.15, 0.16, 0.19, 0.25, 0.25, 0.25, 0.34, 0.34, 0.35 and 0.42 mg/kg.

Based on the European residue data, the Meeting estimated a maximum residue level of 0.6 mg/kg, an STMR of 0.175 mg/kg and an HR of 0.42 mg/kg for fenamidone residues in grapes.

Strawberry

The UK GAP for fenamidone in strawberries is one pre-transplantation treatment (0.18 kg ai/ha) followed by one post-transplantation drench or spray (0.27 kg ai/ha) and a PHI of 35 days.

Eight supervised residue trials were carried out in 2009 and 2012 in Europe in greenhouses. The plants were treated by a drip-irrigation with 0.16–0.18 kg ai/ha followed after 21/22 days by a foliar spray of 0.27 kg ai/ha. The residues were for parent fenamidone < 0.01, < 0.01, 0.01, 0.01, 0.01, 0.02, 0.02, 0.02 mg/kg and for the sum of fenamidone and RPA 410193 < 0.02, < 0.02, 0.02, 0.02, 0.02, 0.02, 0.03, 0.03, 0.03 mg/kg.

Fenamidone is registered in Belgium in protected strawberries with two drench treatments of 0.45 kg ai/ha and a PHI of 35 days. Seven trials according to Belgium GAP were conducted in 2010/2011. The residues were for parent fenamidone < 0.01 (3), 0.01, 0.02 (3) mg/kg and for the sum of fenamidone and RPA 410193 < 0.02, < 0.02, 0.02, 0.02, 0.03, 0.03, 0.04 mg/kg.

The Meeting agreed to use the data set according to the Belgium GAP and estimated a maximum residue level of 0.04 mg/kg, an STMR of 0.02 mg/kg and an HR of 0.04 mg/kg for fenamidone residues in strawberries.

Bulb vegetables

Fenamidone is registered for bulb vegetables in the USA and Canada with foliar application at 4×0.2 kg ai/ha and a PHI of 7 days. Field trials on bulb onion (8) and spring onion (4) were carried out in the USA. At each trial, six instead four spray applications of 0.2 kg ai/ha were made. The following residue data were received.

In bulb onion, the fenamidone residues as well as total residues (sum of fenamidone and RPA 410193) were < 0.02 (6), 0.02 and 0.10 mg/kg. The Meeting recognized that one duplicate field sample gave higher residues than the HR based on the mean residues and decided to use this value of 0.13 mg/kg as HR for the short-term dietary intake assessment instead. The Meeting agreed to extrapolate the residue data from bulb onions to garlic and shallots. Furthermore, the contribution in follow-on crops of 0.4 mg/kg has to be added to the STMR and 0.5 mg/kg to the HR.

The Meeting estimated a maximum residue level, an STMR and an HR of 0.15 mg/kg, 0.42 mg/kg and 0.63 mg/kg, respectively, for bulb onion, garlic and shallots.

In spring onion, the fenamidone residues were 0.24, 0.36, 0.94 and 0.94 mg/kg. The corresponding total residues were 0.24, 0.36, 0.94 and 1.1 mg/kg. The Meeting recognized that one duplicate field sample gave higher residues than the HR based on the mean residues and decided to use this value of 1.2 mg/kg as HR for the short-term dietary intake assessment instead. The Meeting agreed to extrapolate the residue data from spring onion to welsh onion. Furthermore, the contribution in follow-on crops of 0.4 mg/kg has to be added to the STMR and 0.5 mg/kg to the HR.

The Meeting estimated a maximum residue level, an STMR and an HR of 3 mg/kg, 1.05 mg/kg and 1.7 mg/kg, respectively, for spring onion and Welsh onion.

The GAP for leek in Switzerland is 3×0.15 kg ai/ha and a PHI of 14 days. Four supervised residue trials (France 1, Germany 2 and the Netherlands 1) with 4×0.15 kg ai/ha were submitted. At a PHI of 14 days, the residues of fenamidone as well as of the sum of fenamidone and RPA 410193 were 0.02, 0.05, 0.07 and 0.13 mg/kg. Furthermore, the contribution in follow-on crops of 0.4 mg/kg has to be added to the STMR and 0.5 mg/kg to the HR.

The Meeting considered four trials on leek as sufficient and estimated a maximum residue level, an STMR and an HR of 0.3 mg/kg, 0.46 mg/kg and 0.63 mg/kg, respectively.

Brassica vegetables

Head cabbage

Fenamidone is registered in Switzerland in head cabbages as foliar spray with 3×0.15 kg ai/ha and a PHI of 14 days. Seven European trials (France 2, Germany 4 and Portugal 1) according to the Swiss GAP were submitted. The residues of fenamidone as well as the sum of fenamidone and RPA 410193 were < 0.01 (4), 0.01, 0.02 and 0.06 mg/kg.

The GAP for Brassica vegetables in Canada and the USA is 3×0.3 kg ai/ha and a PHI of 2 days. Six trials were conducted in 2003 in the USA with 4×0.3 kg ai/ha and a PHI of 2 days. In all trials, heads with wrapper leaves were analysed. The residues were for parent fenamidone 0.10, 0.17, 0.22, 0.24, 0.35 and 0.52 mg/kg in cabbage heads with wrapper leaves. Residue data for heads without wrapper leaves were available from four of the six trials. The fenamidone residues as well as the total residues were < 0.02, < 0.02, 0.03 and 0.19 mg/kg.

The Meeting agreed to use the US residue data on cabbage heads with wrapper leaves for MRL estimation and without wrapper leaves for dietary intake purposes. Furthermore, the contribution in follow-on crops of 1.2 mg/kg has to be added to the STMR and 1.5 mg/kg to the HR.

The Meeting estimated a maximum residue level of 0.9 mg/kg, an STMR of 1.23 mg/kg and an HR of 1.69 mg/kg.

For livestock dietary burden calculation, the Meeting estimated a median residue of 0.22 mg/kg and a highest residues of 0.52 mg/kg based on the fenamidone data for head cabbage with wrapper leaves.

Flowerhead brassica

The GAP for Brassica vegetables in Canada and the USA is 3×0.3 kg ai/ha and a PHI of 2 days. Six trials were conducted in 2003 on broccoli in the USA with 4×0.3 kg ai/ha and a PHI of 2 days. The mean residues of two separate field samples were for parent fenamidone as well as the total residues 0.31, 0.51, 0.68, 1.5, 1.6 and 2.2 mg/kg.

The Meeting recognized that one duplicate field sample gave higher residues than the HR based on the mean residues and decided to use this value of 2.7 mg/kg as HR for the short-term dietary intake assessment instead. Furthermore, the contribution in follow-on crops of 1.2 mg/kg has to be added to the STMR and 1.5 mg/kg to the HR.

The Meeting estimated for fenamidone residues in flowerhead brassica a maximum residue level, an STMR and HR of 4 mg/kg, 2.29 mg/kg and 4.2 mg/kg, respectively.

Fruiting vegetables, cucurbits

The GAP for cucurbits in Canada and the USA is 4×0.2 kg ai/ha and a PHI of 14 days. Field trials on cucumber (9), summer squash (9) and cantaloupe melons (8) were carried out during 1999 in the USA. At each trial, six spray applications in the range of 0.19–0.21 kg ai/ha were made at intervals of 3–6 days. The following residue data were received:

- In cucumber, the residues of fenamidone as well as the total residues were < 0.02 (7), 0.02 and 0.04 mg/kg.
- In summer squash, the residues of fenamidone as well as the total residues were < 0.02 (8) and 0.06 mg/kg.
- In cantaloupe melon, the residues for fenamidone as well as the total residues were in whole fruits < 0.02, 0.02, 0.04, 0.06, 0.07, 0.08, 0.08 and 0.09 mg/kg. No data were submitted for the edible part.

The Brazilian GAP for the use of fenamidone in watermelon is 3×0.15 kg ai/ha and a PHI of 7 days. Three trials on watermelons were conducted in 2004 in Brazil. The residues were in whole fruits for fenamidone as well as the sum of fenamidone and RPA 410193 were 0.03, 0.04 and 0.05 mg/kg. No residue data for pulp were submitted.

Fenamidone is registered in Switzerland for cucumbers, pumpkins, melons, courgettes, patisson and rondini with three foliar spray treatments of 0.15 kg ai/ha and a PHI of 3 days. Trials on cucumber (9 indoor) and on melons (8 indoor, 8 outdoor) carried out between 2002 and 2005 in European countries according to the GAP were submitted. The following residue data were received:

- In indoor grown cucumber, the residues of fenamidone as well as the total residues were < 0.01, < 0.02, 0.04, 0.04, 0.09, 0.10, 0.10, 0.12 and 0.13 mg/kg.
- In outdoor grown melons, the residues of fenamidone in whole fruits were < 0.02, 0.03, 0.03, 0.04, 0.04, 0.05, 0.08 and 0.12 mg/kg
- In indoor grown melons, the residues of fenamidone in whole fruits were < 0.02, < 0.02, < 0.02, 0.03, 0.04, 0.07, 0.07 and 0.09 mg/kg.
- The corresponding residues of the sum of fenamidone and RPA 410193 in melon pulp were < 0.01 (6) and < 0.02 (10).

The Meeting concluded that the maximum residue level should be based on the critical Swiss GAP. It was noted, that the median of the datasets for cucumber and melons (outdoor and indoor grown) differed by less than five times and agreed to consider a group maximum residue level. In deciding on the data set to use for estimating a group maximum residue level, as a Mann-Whitney U-test indicated that the residue populations for cucumber and melons were not different, it was agreed to combine the results to give a data set for fenamidone residues in whole melons and cucumbers of ($n = 25$) < 0.01, < 0.02 (5), 0.03 (3), 0.04 (5), 0.05, 0.07, 0.07, 0.08, 0.09, 0.09, 0.10, 0.10, 0.12, 0.12 and 0.13 mg/kg. Furthermore, the contribution in follow-on crops of 1.2 mg/kg has to be added to the STMR and 1.5 mg/kg to the HR.

The Meeting estimated for fenamidone residues in fruiting vegetables, cucurbits a maximum residue level of 0.2 mg/kg. Based on the cucumber data, an STMR of 1.29 mg/kg and an HR of 1.63 mg/kg were estimated.

Fruiting vegetables other than cucurbits

The GAP for fruiting vegetables except cucurbits in the USA is 3×0.3 kg ai/ha and a PHI of 14 days.

Field US trials on sweet pepper (6), chilli pepper (3) and tomatoes (17) according to the US GAP were provided. At each outdoor trial, four spray treatments of about 0.3 kg ai/ha were applied. The following residue data were received:

- In sweet pepper, the residues were for fenamidone as well as for the sum of fenamidone and RPA 410193 0.03, 0.05, 0.07, 0.08, 0.08 and 0.19 mg/kg.
- In chilli pepper, the fenamidone residues and the total residues were 0.07, 1.3 and 1.5 mg/kg. The Meeting recognized that one duplicate field sample gave higher residues than the HR based on the mean residues and decided to use this value of 1.7 mg/kg as HR for the short-term dietary intake assessment instead.

- In tomatoes, the fenamidone residues were < 0.02, < 0.02, 0.07, 0.07, 0.09, 0.10, 0.11, 0.25, 0.33, 0.34, 0.38, 0.40, 0.42, 0.46, 0.47, 0.61 and 0.80 mg/kg. The total residues were (n = 17) < 0.02, < 0.02, 0.07, 0.07, 0.09, 0.10, 0.11, 0.25, 0.33, 0.34, 0.38, 0.45, 0.46, 0.47, 0.40, 0.61 and 0.80 mg/kg. The Meeting recognized that one duplicate field sample gave higher residues than the HR based on the mean residues and decided to use this value of 0.82 mg/kg as HR for the short-term dietary intake assessment instead.

The Meeting noted that the GAP in the USA was for fruiting vegetables other than cucurbits and considered a group maximum residue level. Furthermore, the Meeting noted that the median of the datasets for sweet pepper and tomatoes differed by less than 5-fold but the median for chilli was 17 times higher than for sweet pepper. The Meeting agreed to consider a group maximum residue level for fruiting vegetables other than cucurbits, except sweet corn, fungi and chilli pepper. In deciding on the data set to use for estimating a group maximum residue level, as a Mann-Whitney U-test indicated that the residue populations for sweet pepper and tomatoes belong to different populations with the highest residues in tomato. Furthermore, the contribution in follow-on crops of 1.2 mg/kg has to be added to the STMR and 1.5 mg/kg to the HR.

Based on the tomato residue data, the Meeting estimated a maximum residue level of 1.5 mg/kg, an STMR of 1.53 mg/kg and an HR of 2.32 mg/kg for fruiting vegetables other than cucurbits, except sweet corn, fungi and chilli pepper.

For chilli pepper, the Meeting estimated a maximum residue level of 4 mg/kg, an STMR of 2.5 mg/kg and an HR of 3.2 mg/kg.

The Meeting also decided to estimate a maximum residue for chilli pepper (dried) of 30 mg/kg following application of a default dehydration factor of 7 to the estimated maximum residue level of 4 mg/kg for chilli pepper ($7 \times 4 = 28$ mg/kg). The STMR for residues of fenamidone in chilli peppers (dry) is estimated to be 18 mg/kg ($7 \times 2.5 = 17.5$ mg/kg).

Leafy vegetables

The use of fenamidone in leafy vegetables in the USA is 3×0.3 kg ai/ha and a PHI of 2 days and the Meeting looked at the possibility to establish a group MRL. However, the Meeting recognized that the ARfD of 1 mg/kg bw was exceeded for the single commodities, if the highest residue of 32.7 mg/kg (mustard greens) is used as HR and concluded that a group MRL cannot be recommended.

Nine outdoor US trials on head lettuce according to the US GAP were provided. At each outdoor trial, four spray treatments of about 0.3 kg ai/ha were applied. The fenamidone residues, as well as the total residues, were: 0.82, 2.3, 3.3, 3.3, 3.7, 3.9, 4.4, 8.0 and 11 mg/kg. The Meeting recognized that one single sample gave higher residues than the HR based on the mean and decided to use this value of 12 mg/kg as HR for the short-term dietary intake assessment instead. Furthermore, the contribution in follow-on crops of 1.2 mg/kg has to be added to estimate the STMR and 1.5 mg/kg to the HR.

The Meeting estimated a maximum residue level of 20 mg/kg, an STMR of 4.9 mg/kg and an HR of 13.5 mg/kg for fenamidone residues in head lettuce.

Nine outdoor US trials on leaf lettuce according to the US GAP were provided. At each outdoor trial, four spray treatments of about 0.3 kg ai/ha were applied. The fenamidone residues were 1.0, 2.6, 3.4, 3.4, 6.5, 7.9, 10, 12 and 16 mg/kg. The corresponding total residues were 1.0, 2.6, 3.4, 3.4, 6.5, 8.0, 10, 12 and 16 mg/kg (residue of one duplicate field sample 17.5 mg/kg).

The Meeting noted that the ARfD of 1 mg/kg bw is exceeded for leaf lettuce by the IESTI for children (110% of ARfD) using 17.5 mg/kg as HR and decided that the US dataset is not appropriate to estimate a maximum residue level for leaf lettuce.

Alternative GAP exist for outdoor use in Switzerland with 3×0.15 kg ai/ha with a PHI of 14 days. Nine trials on leaf lettuce conducted in Europe were submitted. The fenamidone residues in outdoor leaf lettuce at a PHI of 14 days were < 0.02 (3), 0.02, 0.04, 0.06, 0.07, 0.42 and 0.48 mg/kg. The corresponding total residues were < 0.02 (3), 0.02, 0.04, 0.06, 0.07, 0.43 and 0.48 mg/kg.

Furthermore, the contribution in follow-on crops of 1.2 mg/kg has to be added to estimate the STMR and 1.5 mg/kg the HR.

The Meeting estimated a maximum residue level of 0.9 mg/kg, an STMR of 1.24 mg/kg and an HR of 1.98 mg/kg for fenamidone residues in leaf lettuce.

Eight outdoor US trials on mustard greens and six on spinach according to the US GAP were provided. At each outdoor trial, four spray treatments of about 0.3 kg ai/ha were applied.

In mustard greens, the fenamidone residues were 11, 12, 13, 17, 24, 28, 28 and 29 mg/kg. The corresponding total residues were 11, 12, 13, 17, 24, 28, 29 and 29 mg/kg (residue of one duplicate field sample 32.7 mg/kg). In spinach, the fenamidone residues were 7.2, 7.3, 11, 21, 23 and 31 mg/kg. The corresponding total residues were 7.2, 7.4, 11, 21, 23 and 31 mg/kg (residue of one duplicate field sample 32.4 mg/kg).

The similar datasets of mustard greens and spinach were combined for mutual support. The rank order of the combined fenamidone residues in spinach and mustard greens were (n = 14) 7.2, 7.3, 11, 11, 12, 13, 17, 21, 23, 24, 28, 28, 29 and 31 mg/kg. The corresponding total residues were 7.2, 7.4, 11, 11, 12, 13, 17, 21, 23, 24, 28, 29, 29 and 31 mg/kg. The Meeting recognized that one duplicate field sample gave higher residues than the HR based on the mean residues and decided to use this value of 32.7 mg/kg as HR for the short-term dietary intake assessment instead. Furthermore, the contribution in follow-on crops of 1.2 mg/kg has to be added to estimate the STMR and 1.5 mg/kg the HR.

For spinach and mustard greens, the IESTI represented 150% and 170% for children, respectively of the ARfD of 1 mg/kg bw. The Meeting noted that an alternative GAP was not available.

The Meeting estimated a maximum residue level of 60 mg/kg, an STMR of 20 mg/kg and an HR of 34 mg/kg for fenamidone residues in spinach and mustard greens.

Legume vegetables

In the USA and in Canada, fenamidone may be used as foliar spray on succulent beans with 3 × 0.3 kg ai/ha and a PHI of 3 days. Field trials using 4×0.3 kg ai/ha were carried out in the USA for lima beans (8) and seven trials on common beans (7).

In mature succulent lima beans, the fenamidone residues as well as the total residues were in seeds without pods (n = 8) < 0.02 (4), 0.03, 0.04, 0.08 and 0.08 mg/kg. Furthermore, the contribution in follow-on crops of 1.2 mg/kg has to be added to estimate the STMR and 1.5 mg/kg the HR.

The Meeting estimated for fenamidone residues in beans, shelled a maximum residue level, an STMR and an HR of 0.15 mg/kg, 1.2 mg/kg and 1.58 mg/kg, respectively.

In common beans, the fenamidone residues as well as the total residues were in Pods with seeds (n = 7) 0.10, 0.11, 0.16, 0.19, 0.23, 0.34 and 0.46 mg/kg. Furthermore, the contribution in follow-on crops of 1.2 mg/kg has to be added to estimate the STMR and 1.5 mg/kg the HR.

The Meeting estimated for fenamidone residues in beans, except broad bean and soya bean (green pods and immature seeds) a maximum residue level, an STMR and an HR of 0.8 mg/kg, 1.39 mg/kg and 1.96 mg/kg, respectively.

Root and tuber vegetables

Fenamidone is registered in the USA on the one hand for carrots, ginseng and potatoes and on the other hand for other root and tuber vegetables (garden beet, celeriac, horseradish, parsnips, parsley root, radish, salsify, swedes and turnips) except sugar beet with 3×0.3 kg ai/ha and a PHI of 14 days. Residue data were submitted for the use of fenamidone on carrots, potatoes and ginseng, dry.

Field trials (13) on carrots using approximately 4×0.3 kg ai/ha were carried out in the USA and in Canada. The fenamidone as well as the total residues were (n = 13): 0.02, 0.03, 0.03, 0.03, 0.03, 0.04, 0.05, 0.05, 0.06, 0.06, 0.07, 0.09 and 0.11 mg/kg. Furthermore, the contribution in follow-on crops of 0.4 mg/kg has to be added to estimate the STMR and 0.5 mg/kg the HR.

The Meeting estimated a maximum residue level, an STMR and an HR of 0.2 mg/kg, 0.45 mg/kg and 0.61 mg/kg respectively, for carrots.

Field trials (19) on potatoes using approximately 4×0.3 kg ai/ha were carried out in the USA and in Canada. The fenamidone as well as the total residues were < 0.02 (19) mg/kg. Furthermore, the contribution in follow-on crops of 0.4 mg/kg has to be added to estimate the STMR and 0.5 mg/kg the HR.

The Meeting estimated a maximum residue level of 0.02* mg/kg for fenamidone residues in potatoes. Taking into account the uptake of RPA 412363 from the soil by rooting vegetables grown as follow-on crops, the Meeting estimated an STMR of 0.4 mg/kg and an HR of 0.5 mg/kg for fenamidone residues in potatoes.

Six supervised trials on ginseng were carried out in 2007 in the USA and Canada. Only one trial was carried according to GAP with 3×0.3 –0.31 kg ai/ha. At five trials, nine applications with application rates in the range of 0.29–0.32 kg ai/ha were performed at intervals of 7–15 days between each foliar spray. Samples were prepared by washing the roots with water after harvest followed by drying on racks in drying chambers for 14 days at 18–46 °C. The following residue data were received:

- After a treatment with 3×0.3 –0.31 kg ai/ha, the residue of fenamidone as well as the total residue was 0.06 mg/kg.
- After treatment with 9×0.29 –0.32 kg ai/ha, the fenamidone residues were 0.03, 0.10, 0.17, 0.29 and 0.35 mg/kg. The corresponding total residues were 0.03, 0.10, 0.17, 0.31 and 0.37 mg/kg and the highest single value from a duplicate field sample was 0.55 mg/kg.

The Meeting noted that only one of the six trials matched the GAP and concluded that the data submitted are insufficient to estimate a separate maximum residue level for ginseng.

Stalk and stem vegetables

Fenamidone is registered in the USA for celery with 3×0.3 kg ai/ha and a PHI of 2 days. Six supervised trials on celery were carried out during 2003 in the USA. At each trial, fenamidone was applied by foliar spray at rates of about 0.3 kg ai/ha four times at intervals of 4–6 days. In untrimmed plants without roots, the residues of fenamidone as well as the total residues were at a 2-days PHI ($n = 6$) 2.3, 4.4, 4.5, 8.8, 15 and 18 mg/kg. In trimmed stalks, the residues were ($n = 5$) 0.06, 0.32, 1.1, 1.2 and 1.7 mg/kg. Furthermore, the contribution in follow-on crops of 1.2 mg/kg has to be added to estimate the STMR and 1.5 mg/kg the HR.

The Meeting estimated a maximum residue level of 40 mg/kg, an STMR of 2.3 mg/kg and an HR of 3.2 mg/kg for fenamidone residues in celery.

The GAP for Witloof chicory in Belgium is one dip treatment of 0.006 kg ai/hL followed by an irrigation treatment with 0.6 g ai/hL and a PHI of 18 days.

All in all, nine European indoor residue trials were submitted. In 2004 four hydroponic residue trials were conducted in Belgium, France and the Netherlands consisting of one application through the irrigation water system at a maximum rate of 0.6 g ai/hL of fenamidone and a water volume of 40 L/m², at the commencement of forcing. The fenamidone residues as well as total residues in sprouts at PHIs of 20/21 days were < 0.01 mg/kg (4).

In 2008 and 2010 five residue trials were conducted in France, Germany and the Netherlands. These trials were treated twice, one dip application for two minutes in a solution containing 6.0 g ai/hL, immediately after field sampling of the roots. After storage of the roots in a cold room for 3–8 months, a second hydroponic application was made at the commencement of forcing in the irrigation water system at a maximum rate of 0.6 g ai/hL. Sprout samples were taken on day 20/21 after the second application. The fenamidone residues as well as total residues in sprouts at PHIs of 20/21 days were < 0.01 mg/kg (5).

The Meeting estimated a maximum residue level of 0.01* mg/kg, an STMR of 0.01 mg/kg and an HR of 0.01 mg/kg for fenamidone residues in Witloof chicory.

Oilseed

Fenamidone is registered in the USA for cotton as in-furrow treatment with 1×0.3 kg ai/ha.

Twelve trials were conducted on cotton in the USA during 2003. In each trial, cotton plants were treated at-planting with a single in-furrow over-the-seed application at a rate of 0.3 kg ai/ha. Samples of mature cotton seed were harvested 127–190 days post treatment. The residues of fenamidone as well as the total residues were in seed < 0.02 mg/kg (12).

The Meeting estimated a maximum residue level of 0.02^* mg/kg and an STMR of 0.02 mg/kg for fenamidone residues in cotton seed.

The GAP for the use of fenamidone in the USA for sunflower is seed treatment with 0.19 kg/100 kg seed. Nine trials on sunflower were conducted in 2009 in the USA. In each trial, one plot was planted with sunflower seeds treated with fenamidone at a rate of 0.19 kg ai per 100 kg seed. Two trials included an additional plot planted with seed treated at an exaggerated rate of 0.95 kg ai per 100 kg seed. Samples of mature sunflower seed were harvested 104–146 days after sowing of the treated seed. The residues of fenamidone as well as the total residues were in seed < 0.02 mg/kg (9).

The Meeting estimated a maximum residue level of 0.02^* mg/kg and an STMR of zero for fenamidone residues in sunflower seed.

Legume animal feed

In the USA and Canada, fenamidone may be used as foliar spray on succulent beans with 3×0.3 kg ai/ha and a PHI of 3 days. Seven field trials with 4×0.3 kg ai/ha were carried out in the USA on common beans. The fenamidone residues were in plants with pods at a PHI of 3 days 2.3, 4.1, 5.6, 7.6, 10, 11 and 16 mg/kg (fresh weight).

For the calculation of the livestock animal dietary burden, the Meeting estimated a median residue of 7.6 mg/kg and a highest residue of 16 mg/kg bean forage (green).

Cotton fodder, dry

Fenamidone is registered in the USA for cotton as in-furrow treatment with 1×0.3 kg ai/ha. Trials were conducted on cotton in the USA during 2003. In each trial, cotton plants were treated at-planting with a single in-furrow over-the-seed application at a rate of 0.3 kg ai/ha. The harvest was 127–178 days post treatment. The residues of fenamidone as well as the total residues were in cotton gin by-products < 0.02 mg/kg (6).

For the calculation of the livestock animal dietary burden, the Meeting estimated 0.02 mg/kg as median residue and highest residue for cotton fodder, dry.

Fate of residues during processing

Nature of residues

To estimate the degradation behaviour of [C-phenyl-U- ^{14} C]-fenamidone during industrial processing or household preparation, the processes of pasteurization (90 °C, 20 min at pH 4), baking, boiling, brewing (100 °C, 60 min at pH 5) and sterilization (120 °C, 20 min at pH 6) were simulated.

Degradation of fenamidone was limited and appeared to be dependent on the conditions. The largest extent of degradation was ca. 12% TAR under pasteurisation conditions. RPA 410193 was the only degradate under these conditions. The amounts of RPA 410193 decreased with increasing pH, to 2.5% TAR at pH 6. There was additional degradation to RPA 412708 up to 3.6% TAR at higher temperature (120 °C) and pH (6). This degradation path was not seen at the lower temperatures.

The Meeting concluded that, in addition to the parent fenamidone, the only metabolite to consider for processed products is RPA 410193.

Level of residues

The Meeting received information on the fate of fenamidone residues during the processing of raw agricultural commodities (RAC) like grapes to juice, must, wine and pomace and tomatoes into juice, paste, ketchup and canned tomatoes. Because the residues of RPA 410193 are of the same order of magnitude as the parent concentrations in processed products of grapes and tomatoes, the sum of parent and RPA 410193 is calculated as follows:

Fenamidone, mg/kg	RPA 410193, mg/kg	Total, mg/kg
< 0.02	< 0.02	< 0.04
< 0.02	0.076	0.10
0.05	< 0.02	0.07
0.53	0.13	0.67 ^a
^a 0.53 + (0.13 × 1.11) = 0.6743		

Two processing studies were carried out on potatoes but were only of limited use because the residues in RAC were < LOQ. Five further studies conducted on cabbage, broccoli, peppers, mustard greens and spinach investigated the fate of fenamidone and RPA 410193 after washing and cooking. The processing factors were for washed vegetables 0.58 and for cooked vegetables 0.21.

The processing factors for the sum of fenamidone and RPA 410193 obtained in the processing studies and the estimated STMR-P values for dietary intake calculations are summarized below.

Raw agricultural commodity		Processed commodity (food)		
	STMR, mg/kg		Processing factor	STMR-P, mg/kg
Grapes	0.175	Juice	0.36 (median)	0.063
		Must	0.83 (median)	0.145
		Wine	0.71 (median)	0.124
Tomatoes	1.53	Juice	0.8 (median)	1.22
		Puree	2.1 (median)	3.21
		Ketchup	2.4 (single value)	3.67
		Paste	3.65 (mean)	5.58
		Canned fruits	0.45 (median)	0.69

In some tomato processed commodities, the residues increased during processing. For parent fenamidone, the processing factors were 2.55 for paste, 1.7 for puree and 1.6 for ketchup. Based on the recommended MRL of 1.5 mg/kg in fruiting vegetables other than cucurbits, the Meeting estimated maximum residue levels of 4 mg/kg for tomato paste and of 3 mg/kg for tomato puree as well as for ketchup.

The processing factors for parent fenamidone obtained in the processing studies and the median values of fenamidone residues in the RAC (parent only) were used to calculate the residues in the feed items grape pomace, tomato pomace and potato wet peel for animal dietary burden purposes.

Raw agricultural commodity		Processed commodity (feed)		
	Median (mg/kg)		Processing factor	STMR-P (mg/kg)
Grapes	0.15	Pomace, wet	2.0 (median)	0.3
Tomatoes	0.33	Pomace, wet	4.5 (median)	1.49
Potatoes	0.02	Wet peel	> 2.3 (single value)	0.046

The Meeting noted that fenamidone concentrated during processing in grape wet pomace, tomato wet pomace and potato wet peel. Because wet pomace and wet peel are not commodities in the international trade, no maximum residue levels are estimated.

Residues in animal commodities

Farm animal feeding studies

The Meeting received information on the residue levels arising in tissues and milk when three groups of dairy cows were fed with a diet containing 0.8, 2.4 and 8 ppm fenamidone for 35 consecutive days. At the highest dose group, no residues of fenamidone or the two major metabolites RPA 412636 and RPA 412708 were found in any of the tissue or milk fat samples higher than the LOD of 0.003 mg/kg, with the exception that in one milk fat sample, RPA 412708 was detected (0.011 mg/kg).

No poultry feeding study was submitted. In two metabolism studies laying hens were dosed at 13.8 ppm (C-phenyl label) and 9.8 ppm (N-phenyl label) fenamidone in the diet. The maximum residues (sum of fenamidone, RPA 412636 and RPA 412708) were 0.012 mg/kg in egg white, 0.05 mg/kg in egg yolk, 0.028 mg/kg in liver, 0.004 mg/kg in skin and < 0.001 mg/kg in fat.

Estimated dietary burdens of farm animals

Maximum and mean dietary burden calculations for fenamidone are based on the feed items evaluated for cattle and poultry as presented in Annex 6. The calculations were made according to the livestock diets from Australia, the EU, Japan and US-Canada in the OECD feeding table. Furthermore, the Meeting estimated the maximum highest dietary burden for the main metabolites in follow-on crops.

Parent fenamidone in primary commodities

The foliar application of fenamidone to grapes, tomatoes, cabbage, root and tuber vegetables, cotton and sunflower resulted in residues of fenamidone in the following feed items: wet grape pomace, wet tomato pomace, head cabbage, carrot culls, potato culls, potato process waste, turnip roots, swede roots, cassava/tapioca roots, cotton undelinted seed, sunflower seed and cotton fodder, dry. Residue data were also submitted for green bean forage (vines) what is listed as 60–70% of the Australian diet and for beef and dairy cattle and as 20% of the European diet for dairy cattle. Based on the named feed items, the calculated maximum animal dietary burden for dairy or beef cattle was in the USA and Canada 0.13 ppm, in the EU 10 ppm and in Australia 33 ppm. The Meeting noted that the estimated livestock dietary burden (AUS) was up to three times higher than the dose rate in the cow feeding study.

The Meeting recognized that green bean forage (vines) is not used as animal feed in the USA and in Canada but in the EU and in Australia. The Meeting was informed by an official communication of the government of Australia that no fodder crops are imported. Furthermore, the USDA Global Agricultural Trade System database indicates that no animal feed/fodder were exported from the USA to Australia and the EU in 2013. The Meeting concluded that green bean forage is not an exportable commodity and decided to make a refined calculation of the livestock dietary burden without the residues in bean vines (see Annex 6).

In the table below the estimated livestock dietary burden is presented for fenamidone after foliar treatment of plants.

	Livestock dietary burden, fenamidone, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0.15	0.15	1.0 ^a	0.52	1.0 ^a	0.97 ^b	0	0
Dairy cattle	0.13	0.08	0.96	0.49	0.99	0.97	0	0
Poultry–broiler	0	0	0.09	0.04	0	0	0	0
Poultry–layer	0	0	0.27 ^c	0.12 ^d	0	0	0	0

^a Suitable for MRL estimates for mammalian meat, fat, edible offal and milk.

^b Suitable for STMR estimates for mammalian meat, edible offal and milk.

^c Suitable for MRL estimates for eggs, meat, fat and edible offal of poultry.

^d Suitable for STMR estimates for eggs, meat, fat and edible offal of poultry.

Metabolites in follow-on crops

Rotational crop studies showed that in the follow-on crops an uptake from the soil of the metabolites RPA 412708 and RPA 412636 occurred. Their highest concentrations found in follow-on crops as wheat, sweet corn, maize and soya beans after the treatment of bare soil with fenamidone at 1.2 kg ai/ha per annum were scaled according to the critical US GAP of 0.9 kg ai/ha per annum for brassica vegetables, fruiting vegetables, leafy vegetables, root and tuber vegetables and celery. These residues are extrapolated to similar feed items in the OECD feeding table.

The maximum livestock dietary burden for RPA 412708/RPA 412636 in follow-on crops in the USA and Canada was estimated as follows: Beef cattle 0.088 ppm (as RPA 41236), dairy cattle 0.36 ppm (as RPA 41236), poultry broiler 0 ppm and poultry layer 0 ppm. Expressed as fenamidone equivalents, the burden was for beef cattle 0.14 ppm and for dairy cattle 0.59 ppm.

The Meeting noted that RPA 412708 and RPA 412636 are not found in milk or tissues of dairy cows dosed at 8 ppm fenamidone through normal animal metabolism routes. Therefore, the two metabolites are unlikely to be present after direct administration of much lower levels (maximum livestock dietary burden 0.59 ppm).

The Meeting concluded that it is unlikely that residues of RPA 412708 and RPA 412636 in follow on crops of the uses considered by the JMPR result in residues in animal products.

Animal commodities, MRL estimation

The feeding study with fenamidone in dairy cows was performed at actual dose levels of 0.8, 2.4 and 8 ppm in the diet. At the highest dose group, no residues of fenamidone or of the two major metabolites RPA 412636 and RPA 412708 were found in any of the tissue or milk fat samples (< 0.003 mg/kg, LOD; LOQ 0.01 mg/kg), with the exception that in one milk fat sample RPA 412708 was detected (0.011 mg/kg). The overdosing factor is calculated as about 8 ($8 \text{ ppm} \div 1 \text{ ppm}$). Therefore, residues of fenamidone, RPA 412636 and RPA 412708 in cattle tissues and milk arising from a burden of 1 ppm are not expected.

The Meeting estimated maximum residue levels of 0.01^* mg/kg for milks, meat from mammals, other than marine mammals (fat), mammalian fat (except milk fat) and edible offal (mammalian). For milk fat, a maximum residue level of 0.02 mg/kg was estimated. The STMRs for milk and milk fat were 0.01 mg/kg and the STMR/HR values for muscle, fat and edible offal were zero.

No poultry feeding study was submitted. In two metabolism studies, laying hens were dosed at 13.8 ppm (C-phenyl label) and 9.8 ppm (N-phenyl label) fenamidone in the diet. The maximum residues (sum of fenamidone, RPA 412636 and RPA 412708) were 0.012 mg/kg in egg white, 0.05 mg/kg in egg yolk and 0.028 mg/kg in liver.

Allowing for the dose rates in the metabolism studies (overdosing factors about 40–50), it can be seen that at the maximum calculated dietary burden for poultry of 0.27 ppm, no residues of fenamidone or any of its metabolites will be found in poultry commodities at or above the LOQ of 0.01 mg/kg.

The Meeting estimated maximum residue levels of 0.01^* mg/kg poultry meat, poultry fat, poultry edible offal and eggs. The STMR/HR values for poultry meat, poultry fat, poultry edible offal and eggs are zero.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for compliance with the MRL for plant and animal commodities: Fenamidone.

Definition of the residue for estimation of dietary intake for plant commodities: Sum of fenamidone, (S)-5-methyl-5-phenyl-3-(phenylamino)- 2,4-imidazolidine-dione (RPA 410193) plus $10 \times$ the sum of both (S)-5-methyl-5-phenyl-2,4-imidazolidine-dione (RPA 412636) and (5S)-5-methyl-2-(methylthio)-5-phenyl-3,5-dihydro- 4H-imidazol-4-one (RPA 412708), all calculated as fenamidone.

Residue concentration $C_{total} = C_{fenamidone} + C_{RPA\ 410193} + 10 \times (CRPA\ 412636 + CRPA\ 412708)$

Definition of the residue for estimation of dietary intake for animal commodities: Fenamidone plus $10 \times$ the sum of both (S)-5-methyl-5-phenyl-2,4-imidazolidine-dione (RPA 412636) and (5S)-5-methyl-2-(methylthio)-5-phenyl-3,5-dihydro- 4H-imidazol-4-one (RPA 412708), all calculated as fenamidone.

Residue concentration $C_{total} = C_{fenamidone} + 10 \times (CRPA\ 412636 + CRPA\ 412708)$

The residue is fat-soluble.

MRL recommendations and dietary intake

CCN	Commodity Name	MRL, mg/kg Proposed	STMR or STMR-P mg/kg	HR or HR-P mg/kg
VP 0061	Beans, except broad bean and soya bean (green pods and immature seeds)	0.8	1.39	1.96
VP 0062	Beans, shelled	0.15	1.2	1.58
VB 0041	Cabbages, Head	0.9	1.23	1.69
VR 0577	Carrots	0.2	0.45	0.61
VS 0624	Celery	40	2.3	3.2
SO 0691	Cotton seed	0.02*	0.02	
MO 0105	Edible offal (mammalian)	0.01*	0	0
PE 0112	Eggs	0.01*	0	0
VB 0042	Flowerhead brassicas	4	2.29	4.2
VC 0045	Fruiting vegetables, Cucurbits	0.2	1.29	1.63
VO 0050	Fruiting vegetables, other than cucurbits (except chilli pepper, sweet corn and mushrooms)	1.5	1.53	2.32
VA 0381	Garlic	0.15	0.42	0.63
FB 0269	Grapes	0.6	0.175	0.42
JF 0269	Grape juice		0.063	
	Grape must		0.145	
	Grape wine		0.124	
VA 0384	Leek	0.3	0.46	0.63
VL 00482	Lettuce, Head	20	4.9	13.5
VL 00483	Lettuce, Leaf	0.9	1.24	1.98
MF 0100	Mammalian fats (except milk fat)	0.01*	0	0
MM 0095	Meat (from mammals other than marine mammals)	0.01* (fat)	0	0
FM 0183	Milk fats	0.02	0.01	0.01
ML 0106	Milks	0.01*	0.01	0.01
VL 0485	Mustard greens ^a	60	20	34
VA 0385	Onion, Bulb	0.15	0.42	0.63
VA 0287	Onion, Welsh	3	1.05	1.7
VO 0444	Peppers, Chilli	4	2.5	3.2
HS 0444	Peppers, Chilli, dried	30	18	
VR 0589	Potato	0.02*	0.4	0.5
PM 0110	Poultry meat	0.01* (fat)	0	0
PO 0110	Poultry, Edible offal of	0.01*	0	0
PF 0111	Poultry fats	0.01*	0	0
VA 0388	Shallot	0.15	0.42	0.63
VL 0502	Spinach ^a	60	20	34
VA 0389	Spring onion	3	1.05	1.7
FB 0275	Strawberry	0.04	0.02	0.04

Fenamidone

	Commodity	MRL, mg/kg	STMR or STMR-P	HR or HR-P
CCN	Name	Proposed	mg/kg	mg/kg
SO 0702	Sunflower seed	0.02*	0	
	Tomato ketchup	3	3.67	
	Tomato paste	4	5.58	
	Tomato puree	3	3.21	
	Tomato canned fruits		0.69	
JF 0448	Tomato juice		1.22	
VS 0469	Witloof chicory (sprouts)	0.01*	0.01	0.01

* ARfD exceeded

Additional values to calculate the dietary intake

CCN	Commodity name	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
VB 0040	Brassica vegetables, except cabbage head and flowerhead brassicas	1.2	1.5
VA0035	Bulb vegetables, except bulb onion, garlic, shallot, spring onion, welsh onion	0.4	0.5
GC 0080	Cereal grains, except rice	0.33	
	Herbs	1.2	1.5
	Leafy vegetables, except lettuce, spinach, mustard greens	1.2	1.5
	Legume vegetables, except beans, shelled and beans (green pods and immature seeds)	1.2	1.5
	Radish leaves, turnip greens, taro leaves, rape greens	0.4	0.5
VR 0075	Root and tuber vegetables, except carrots and potatoes	0.4	0.5
	Stalk and stem vegetables, except celery, Witloof chicory	1.2	1.5

Additional values used to calculate the livestock animal dietary burden

CCN	Commodity name	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
AL 1030	Bean forage (green)	7.6	16
VB 0041	Cabbages, Head, with wrapper leaves	0.22	0.52
VR 0577	Carrots	0.05	0.11
AM 0691	Cotton fodder, dry	0.02	0.02
	Grape pomace, wet	0.3	
	Potato wet peel	0.046	
	Tomato pomace, wet	1.49	

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Dietary Intakes (IEDIs) of fenamidone were calculated for the 17 GEMS/Food cluster diets using STMRs and STMR-Ps estimated by the current Meeting (Annex 3). The ADI is 0–0.03 mg/kg bw and the calculated IEDIs were 10–60% of the maximum ADI. The Meeting concluded that the long-term intake of residues of fenamidone resulting from the uses considered by the current JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short Term Intake (IESTI) for fenamidone was calculated for food commodities and their processed fractions for which maximum residue levels were estimated and for which consumption data were available. The results are shown in Annex 4.

The Meeting recognized that for leaf lettuce the IESTI calculated according to the maximum GAP exceeded the ARfD of 1 mg/kg bw and used an alternative GAP. For spinach and mustard

greens, the IESTI represented 150% and 170%, respectively of the ARfD of 1 mg/kg bw. The Meeting noted that an alternative GAP was not available. For the other commodities considered by the JMPR, the IESTI represented 0–30% of the ARfD. The Meeting concluded that the short-term intake of residues of fenamidone, when used in ways that have been considered by the JMPR, is unlikely to present a public health concern (except mustard greens and spinach).

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M-285013-01-1	Tornisielo, VL	2002	Determinação de resíduo de Censor (Fenamidone e RPA 405862) em Bulbos de Cebola—São José do Rio Pardo/SP. Universidade de São Paulo, São Paulo, Brazil. Report No. R.75/02. Unpublished
M-285309-01-1	Tornisielo, VL	2000	Determinação de resíduo de SECURE (fenamidone + mancozeb) em frutos de uva.—Indaiatuba/SP. Universidade de São Paulo, São Paulo, Brazil. Report No. R.19/00. Unpublished
M-285311-01-1	Tornisielo, VL	2000	Determinação de resíduo de SECURE (fenamidone + mancozeb) em frutos de uva—Jundiaí/SP. Universidade de São Paulo, São Paulo, Brazil. Report No. R.20/00. Unpublished

Code	Author(s)	Year	Title, Institute, Report reference
Doc-ID No			
M-286141-01-1	Cavaillé, C	2007	Storage Stability of Residues of Fenamidone and its Metabolite AE C650488 in Turnip During Deep Freeze Storage for at least 12 Months. Bayer CropScience, Lyon, France. Report No. 05-15 (MR-07/232). Unpublished
M-305308-01-1	Hartz, AL and Helfrich, KK	2008	REASON 500 SC—Magnitude of the Residue in/on Soybean (Rotational Crop Tolerance). Bayer CropScience, Kansas City, Missouri, USA. Report No. RARPY017. Unpublished
M-305323-01-1	Hartz, AL and Helfrich, KK	2008	REASON 500 SC—Magnitude of the Residue in/on Sweet Corn (Rotational Crop Tolerance). Bayer CropScience, Kansas City, Missouri, USA. Report No. RARPY018. Unpublished
M-305368-01-1	Hartz, AL and Helfrich, KK	2008	REASON 500 SC—Magnitude of the Residue in/on Field Corn (Rotational Crop Tolerance). Bayer CropScience, Kansas City, Missouri, USA. Report No. RARPY021. Unpublished
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M-363750-03-1	Melrose, I	2010	Determination of the residues of Fenamidone and fosetyl-Al in/on Chicory, Witloof after Dip and cold storage and Application in irrigation water, flooding of AE F053618 03 WG66 A1 in the Forcing Room, hall, store, in France (North), Germany and Netherlands. Bayer CropScience, Lyon, France. Report No. 08-2205 (2 nd Amendment). Unpublished
M-368426-01-1	Class, T	2010	Assessment and validation of DFG method S19 (BCS method no. 00086/M091) for the determination of residues of fenamidone and its metabolite AE C650488 in plant materials. PTRL Europe, D-89081 Ulm, Germany. Report No. P/B 1948 G. Unpublished
M-370932-01-1	Senciue, M	2010	Validation of analytical BCS method 00868/M001 for the determination of residues of fenamidone and its metabolite AE C650488 in/on plant materials. PTRL Europe, Ulm, Germany. Report No. P 1980 G. Unpublished
M-393676-01-1	Corley, J	2010	Fenamidone: Magnitude of the Residue on Ginseng. IR-4 Project Headquarters, Rutgers, The State University of New Jersey, USA. IR-4 Project No. 09800. Unpublished
M-394609-01-1	Berriault, J	2002	Magnitude of Fenamidone Residues on Potatoes after Multiple Applications of Reason® Fungicide and a 14 Day PHI. Enviro-Test Laboratories, Edmonton, Alberta, Canada. Report No. 01AC05. Unpublished
M-401525-01-1	Uceda, L and Meilland-Berthier, I	2011	Determination of the residues of fenamidone and fosetyl-Al in/on strawberry after dripping in irrigation water and spraying of AE F053616 03 WG66 A1 in the greenhouse in Belgium, France (South), Germany and Netherlands. Bayer CropScience, Lyon, France. Report No. 09-2169. Unpublished
M-404370-01-1	Uceda, L and Meilland-Berthier, I	2011	Determination of the residues of fenamidone and fosetyl-Al in/on lettuce after spraying of AE F053616 03 WG66 A1 in the field in France (North) and Germany. Bayer CropScience, Lyon, France. Report No. 09-2158. Unpublished
M-404444-01-1	Uceda, L and Meilland-Berthier, I	2011	Determination of the residues of fenamidone and fosetyl-Al in/on grape after spraying and spraying, low-volume of AE F053616 03 WG71 A3 in the field in France (South), Italy, Portugal and Spain. Bayer CropScience, Lyon, France. Report No. 09-2180. Unpublished
M-404520-01-1	Uceda, L and Meilland-Berthier, I	2011	Determination of the residues of fenamidone and fosetyl-Al in/on grape after spraying and spraying, low-volume of AE F053616 03 WG71 A3 in the field in Belgium, France (North) and Germany. Bayer CropScience, Lyon, France. Report No. 09-2179. Unpublished
M-404587-01-1	Uceda, L and Meilland-Berthier, I	2011	Determination of the residues of fenamidone and fosetyl-Al in/on lettuce after spraying of AE F053616 03 WG66 A1 in the greenhouse in Belgium, France (South), Germany, Italy, Netherlands and Spain. Bayer CropScience, Lyon, France. Report No. 09-2159. Unpublished

Code	Author(s)	Year	Title, Institute, Report reference
Doc-ID No			
M-404654-02-1	Schulte, G	2012	Storage Stability of Fenamidone and AE C650488 in/on Tomato Fruit. Amendment No. 1 to report no: MR-11/028. Bayer CropScience AG, Monheim am Rhein, Germany. Report No. MR-11/028 (1 st Amendment M-404654-02-1). Unpublished
M-405055-01-1	Ballesteros, C and Meilland-Berthier, I	2011	Determination of the residues of fenamidone and propamocarb hydrochloride in/on potato after spray application of AE B066752 03 SC40 A1 in the field in France (North), France (South), Germany, Italy, Portugal and Spain. Bayer CropScience, Lyon, France. Report No. 09-2170. Unpublished
M-405062-01-1	Celine, B and Meilland-Berthier, I	2011	Determination of the residues of fenamidone and propamocarb hydrochloride in/on lettuce after spraying of AE B066752 03 SC40 A1 in the field in France (South), Italy and Spain. Bayer CropScience, Lyon, France. Report No. 09-2216. Unpublished
M-405712-01-1	Schulte, G and Bauer, J	2011	Determination of the residues of fenamidone and propamocarb hydrochloride in/on tomato and cherry tomato after spraying of AE B066752 03 SC40 A1 in the greenhouse in Germany, Italy, the Netherlands and Spain. Bayer CropScience, Germany. Report No. 09-2235. Unpublished
M-406443-02-1	Schulte, G and Bauer, J	2012	Determination of the residues of fenamidone and propamocarb in/on tomato and the processed fractions (whole fruit, washed; washings; strain rest; raw juice; juice; peel; peeling water; fruit peeled; preserve; raw puree and puree) after spraying of AE B066752 03 SC40 A1 in the field in the Netherlands, Italy and Germany. Bayer CropScience A, Monheim, Germany. Report No. 09-3235-01 (1 st Amendment to report). Unpublished
M-410297-01-1	Senciuc, M	2011	Validation of analytical BCS method 01323 for the determination of residues of fenamidone and its metabolites AE 0540057 and AE C415557 in foodstuff of animal origin. PTRL Europe GmbH, D-89081 Ulm, Germany. Report No. B 2239 G. Unpublished
M-415558-01-1	Uceda, L	2011	Determination of the residues of fenamidone and propamocarb hydrochloride in/on potato after spray application of Fenamidone & Propamocarb hydrochloride SC 450 in the field in Spain and Italy. Bayer CropScience, Lyon, France. Report No. 10-2065. Unpublished
M-418546-01-1	Uceda, L	2011	Determination of the residues of fenamidone and propamocarb hydrochloride in/on lettuce after spraying of Fenamidone & Propamocarb hydrochloride SC 450 in the field in Spain and Italy. Bayer CropScience, Lyon, France. Report No. 10-2012. Unpublished
M-419363-01-1	Cavaillé, C	2011	Determination of the residues of fenamidone and fosetyl-Al in/on strawberry after Apply in irrigation water, dripping of AE F053616 03 WG66 A1 in the greenhouse in France (North), Germany, Belgium and the Netherlands. Bayer CropScience, Lyon, France. Report No. 10-2014. Unpublished
M-419802-01-1	Noss, G and Ballmann, C	2011	Determination of the residues of fenamidone and propamocarb hydrochloride in/on tomato and tomato, cherry after spraying of Fenamidone & Propamocarb hydrochloride SC 450 in the Greenhouse in Spain, Italy, Germany and the Netherlands. Bayer CropScience, Monheim, Germany. Report No. 10-2013. Unpublished
M-419813-01-1	Cavaillé, C	2011	Determination of the residues of fenamidone and fosetyl-Al in/on grape after spraying and spraying, low-volume of AE F053616 03 WG71 A3 in the field in France (North) and Germany. Bayer CropScience, Lyon, France. Report No. 10-2010. Unpublished
M-420668-01-1	Cavaillé, C and Ratajczak, M	2011	Determination of the residues of Fenamidone and fosetyl-Al in/on Chicory, Witloof after Dipping before cold storage and Application in irrigation water of AE F053616 03 WG66 A1 in the Greenhouse and Room, hall, store, in the Netherlands and Germany. Bayer CropScience, Lyon, France. Report No. 10-2040. Unpublished
M-435899-01-1	Uceda, L	2012	Determination of the residues of fenamidone and fosetyl-Al in/on Grape after Spraying and Spraying, low-volume of AE F053616 03 WG71 A3 in the Field in France (South), Spain, Italy and Portugal. Bayer CropScience, Lyon, France. Report No. 10-2011. Unpublished

Code	Author(s)	Year	Title, Institute, Report reference
Doc-ID No			
M-438111-02-1	Meilland-Berthier, I	2012	Determination of the residues of fenamidone and fosetyl-Al in/on strawberry after application via irrigation water (drip irrigation) and spray application of AE F053616 03 WG66 A1 in the greenhouse in Southern France, the Netherlands and Belgium. Bayer CropScience, Lyon, France. Report No. 12-2039 (1 st Amendment to report). Unpublished
M-440254-01-1	Konrad, S	2012	Independent lab validation of analytical BCS method 01323 for determination of residues of fenamidone and its metabolites AE 0540057 and AE C415557 in animal tissues (whole milk, liver, kidney, fat, muscle, whole egg). Currenta, GmbH, D-51368, Leverkusen, Germany. Report No. 2011/0107/01. Unpublished
M-445339-01-1	Uceda, L	2013	Determination of the residues of fenamidone and fosetyl-Al in/on Strawberry after application of AE F053616 03 WG66 A1 via irrigation water in the Greenhouse in Germany, Belgium and the Netherlands. Bayer CropScience, Lyon, France. Report No. 11-2004. Unpublished
M-447085-01-1	Bongartz, R and Miebach, D	2013	Metabolism of [phenyl-UL- ¹⁴ C/ ¹³ C6] Fenamidone in Confined Rotational Crops. Bayer CropScience AG, Monheim, Germany. Report No. M1301876-6 (Amendment No. 1.). Unpublished
M-449868-01-1	Bourgade, C, Simonin, B and Uceda, L	2013	Validation data of the Multi-Residue Analytical Method 01163 (based on QuEChERS method), for the determination of fenamidone and its metabolite AE C650488 in/on plant materials and Cross Validation results of the three Extraction Methods. Bayer CropScience. Unpublished
M-451095-01-1	Simonin, B	2013	Supplementary information for the dossier of MRLs modifications for fenamidone on certain crops. Unpublished
M-451106-01-1	Bongartz, R and Miebach, D	2013	Metabolism of [aniline-UL- ¹⁴ C/ ¹³ C6] Fenamidone in Confined Rotational Crops. Bayer CropScience AG, Monheim, Germany. Report No. M1301877-7. Unpublished
M-489329-01-1	Corley, J	2012	Fenamidone: Magnitude of the residue on bean (snap). IR-4 Project No. 08895, Headquarters, Rutgers, The State University of New Jersey, Princeton, NJ 08540, USA. Unpublished.
M-489331-01-1	Corley, J	2012	Fenamidone: Magnitude of the residue on bean (lima). IR-4 Project No. 09530, Headquarters, Rutgers, The State University of New Jersey, Princeton, NJ 08540, USA. Unpublished.

FENPROPATHRIN (185)

The first draft was prepared by Professor Árpád Ambrus, temporary adviser, Hungary

EXPLANATION

Fenpropathrin is a type II pyrethroid insecticide and acaricide used for the control of a variety of arthropods including aphids, worms, moths, beetles, mites, spiders, thrips, flies, fleas and other pests in agriculture. It acts by contact and ingestion. It is also a repellent. It interferes with nerve impulse transmission by acting on sodium channels.

Fenpropathrin was first evaluated by JMPR in 1993 when an ADI of 0–0.03 mg/kg bw was established and a number of MRLs recommended. In 2006 MRL for tea was recommended. The compound was re-evaluated for toxicology within the periodic review programme in 2012 when the Meeting reaffirmed the ADI of 0–0.03 mg/kg bw and established an ARfD of 0.03 mg/kg bw.

Fenpropathrin is scheduled for periodic re-evaluation of residues in 2014. Data to support proposed Codex MRLs on a number of commodities and on animal products were submitted for review.

IDENTITY

ISO common name:

Fenpropathrin

IUPAC name:

(*RS*)- α -cyano-3-phenoxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate

Chemical Abstract name:

cyano(3-phenoxyphenyl)methyl 2,2,3,3-tetramethylcyclopropanecarboxylate

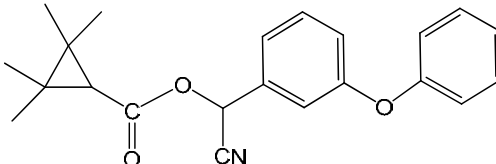
CAS No.:

64257-84-7 (racemate);
39515-41-8 (unstated stereochemistry)

Molecular Formula:

C₂₂H₂₃NO₃

Structural Formula:



Molecular Weight:

349.43

Specifications

Specifications for fenpropathrin have not yet developed by FAO.

Physical and Chemical Properties

Technical material (90%) (Reference: JMPR, 1993)

Chemical/physical property	Results	
Physical state	Liquid	
Colour	Yellow to brown	
Odour	Faint characteristic odour	
Density	1.105	
Vapour pressure	2.15×10^{-6} Pa	
Melting range	45-50 °C	
Flammability	Flash point : 205 °C Ignition point: 325 °C	
Solubility in organic solvents (g/L, 23°C)	Acetone	> 500

Chemical/physical property	Results	
	Acetonitrile	> 500
	Cyclohexanone	> 500
	Ethyl acetate	> 500
	Methanol	216
	Xylene	> 500
Solubility in water	36.3 µg/L at 25.1 °C	
Octanol/water partition coefficient	Log $P_{O/W}$ = 6.0± 0.20	
Hydrolytic half lives	pH 5 and pH: 7 200 days pH 9: 17.1 days (calculated based on hydrolysis of cyclopropyl-1- ¹⁴ C)-fenpropathrin.	
Stability	No significant breakdown after 20 weeks storage at 60 °C	

Hydrolytic degradation

The hydrolytic degradation of fenpropathrin was examined at pH 5, 7, and 9 with [phenoxyphenyl-¹⁴C]-fenpropathrin or [cyclopropyl-1-¹⁴C]-fenpropathrin in sterile aqueous solution containing 1% acetonitrile as co-solvent (Takahashi, *et.al.*, 1983 and 1985, References FM-30-0010 and FM-50-0053; Concha, *et al*, 1992; Reference FM-21-0060).

The predominant hydrolysis reactions were cleavage of the ester linkage and hydration of the cyano group. Major hydrolysis products from cyclopropyl labelled fenpropathrin were TMPA, TMPA-carboxamide and CONH₂-fenpropathrin, while from phenoxyphenyl labelled fenpropathrin, were PBacid and CONH₂-fenpropathrin

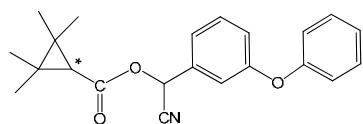
Formulation

The emulsifiable concentrate formulation containing 300 g/L fenpropathrin was used in the residue trials submitted for evaluation.

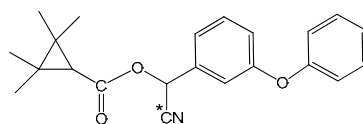
METABOLISM AND ENVIRONMENTAL FATE

The metabolism of fenpropathrin has been investigated in apple, tomato, beans, cotton, cabbage, lactating goat and laying hens. The crops selected represent those for which supervised trials have been provided.

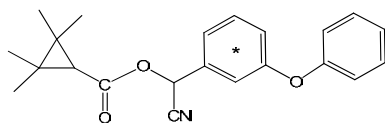
The fate and behaviour of fenpropathrin in plants, animals and soil were investigated using either [cyclopropyl-1-¹⁴C]-fenpropathrin, [benzyl-¹⁴C]-fenpropathrin, [phenoxyphenyl-¹⁴C]-fenpropathrin or [cyano-¹⁴C]-fenpropathrin (all with radiochemical purity >98%).



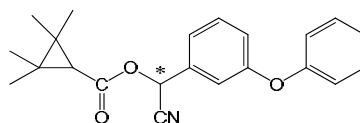
[cyclopropyl-1-¹⁴C]-fenpropathrin



[cyano-¹⁴C]-fenpropathrin



[phenoxyphenyl-¹⁴C]-fenpropathrin



[benzyl-¹⁴C]-fenpropathrin

The chemical structures and code names of metabolites and the major degradation products of fenpropathrin are shown in Table 1.

Table 1 Degradation compounds from the metabolism of fenpropathrin

Compound Name	Structure	Found in:
2'-or 4'-OH-Fenpropathrin [α -cyano-3-(2'- or 4'-hydroxyphenoxy)benzyl 2,2,3,3-tetramethylcyclopropanecarboxylate]		Plant, animal, soil
2'- or 4'-OH-Fenpropathrin-CH ₂ OH [α -cyano-3-(2'- or 4'-hydroxyphenoxy)benzyl-2-hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylate]		Plant, animal
CONH ₂ -Fenpropathrin [α -carbamoyl-3-phenoxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate]		Soil, water plant
COOH-Fenpropathrin [α -carboxy-3-phenoxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate]		Soil, plant
Desphenyl-Fenpropathrin [α -cyano-3-hydroxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate]		Animal, soil, plant
Fenpropathrin-CH ₂ OH [α -cyano-3-phenoxybenzyl 2-hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylate]		Plant, animal
Fenpropathrin-COOH [α -cyano-3-phenoxybenzyl 2-carboxy-2,3,3-trimethylcyclopropanecarboxylate]		Animal
Fenpropathrin-(CH ₂ OH) ₂ [α -cyano-3-phenoxybenzyl 2,2-dihydroxymethyl-3,3-dimethylcyclopropanecarboxylate]		Plant
2'- or 4'-OH-Fenpropathrin-(CH ₂ OH) ₂ [α -cyano-3-(2'- or 4'-hydroxyphenoxy)benzyl 2,2-dihydroxymethyl-3,3-dimethylcyclopropanecarboxylate]		Plant
PB aldehyde (PBald) [3-phenoxybenzaldehyde]		Plant, animal, soil
PB alcohol (PBalc) [3-phenoxybenzyl alcohol]		Plant, soil
PBacid [3-phenoxybenzoic acid]		Plant, animal, soil, water

Compound Name	Structure	Found in:
2'- or 4'-OH-PBalc [3-(2'- or 4'-hydroxyphenoxy)benzyl alcohol]		Plant
2'- or 4'-OH-PBacid [3-(2'- or 4'-hydroxyphenoxy)benzoic acid]		Plant, animal, soil
3-OH-Bacid [3-hydroxy-benzoic acid]		Animal, soil
TPMA [2,2,3,3-tetramethylcyclopropane-carboxylic acid]		Plant, animal, soil, water
TPMA-CH2OH [2-hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylic acid]		Plant, animal
TPMA-lactone [5,6,6-trimethyl-3-oxabicyclohexan-2-one]		Plant
TPMA-CH2OH lactone [5-hydroxymethyl-6,6-dimethyl-3-oxabicyclohexan-2-one]		Plant, animal
TPMA carboxamide [2,2,3,3-tetramethylcyclopropane-carboxamide]		Water
TPMA-COOH [2-carboxy-2,3,3-trimethylcyclopropanecarboxylic acid]		Plant, animal

Animal metabolism

Laboratory animals

Absorption by rats was rapid and excretion almost complete (97%) within 48 hours. About 56% of the administered dose was found in urine and 40% in faeces at 48 hours. The amount of radioactivity excreted via expired air was 0.005%. The low residues found in blood, liver, kidney, fat, muscle and brain 24 hours after dosing depleted rapidly over the following 7 days to barely detectable levels, and less than 1.5% of the administered dose remained in the body 8 days after treatment. The highest residue was found in the fat. About 29–53% of the parent compound was detected in the faeces and no parent compound was detected in the urine. The predominant urinary metabolites derived from the acid moiety were identified as TPMA–glucuronide and TPMA-CH₂OH (*trans*). Other metabolites identified were TPMA-COOH (*trans*) and TPMA-CH₂OH-lactone in free form or as the glucuronide conjugate. The major urinary metabolites derived from the alcohol moiety were PBacid in free form and as the glycine conjugate, 4'-OH-PBacid–sulfate and 2'-OH-PBacid–sulfate. The urinary metabolites from the alcohol moiety were similar to those from other pyrethroids. The major faecal metabolite was identified as CH₂OH *trans*-fenpropathrin, followed by COOH *trans*-fenpropathrin, 4'-

OH-fenpropathrin and 4'-OH,CH₂OH *trans*-fenpropathrin. Depending on the dose administered, 30–50% of the applied radioactivity was excreted in faeces as parent compound. Fenpropathrin and TMPA were the major components of ¹⁴C in tissues. No sex-related differences in tissue distribution were observed.

Lactating goats

The adsorption and deposition of radioactivity was studied in lactating goats (Ku and Doran, 1990, Reference FM-01-0041). Two animals per group were dosed for five consecutive days via capsules with either [phenoxyphenyl-¹⁴C]-fenpropathrin or [cyclopropyl-1-¹⁴C]-fenpropathrin at a rate equivalent to 50 ppm (based on a 2 kg diet, 100 mg per dose of test material). A single control animal was dosed, using a placebo capsule, in an identical manner. Sample collection was initiated on study day 1, with the twice daily collection of milk (morning and afternoon) until sacrifice. Urine and faeces were collected daily from study day 1 to day 4 (with cage wash conducted at study termination). Blood samples were collected at specified times during the last day of dosing. At sacrifice, and within 4 hours of the terminal dose, samples of omental and perirenal fat, kidneys, liver, gall bladder contents, heart, loin muscle and rear leg muscle were taken for analysis. Urine, milk and butterfat (obtained from the milk) were quantified directly by LSC of replicate aliquots. Faeces and non-fatty tissues were homogenized and replicate subsamples taken for combustion and radio-assay. Replicate samples of the fatty tissues were solubilised prior to radio-assay.

Milk, tissues and fat (omental and perirenal) were extracted with either acidified acetonitrile/hexane (milk) or acidified acetonitrile (fat and tissues). Extracts were combined, concentrated and partitioned with either ethyl ether or ethyl acetate with further concentration and subsequent quantification by HPLC. Aqueous fractions from the milk, tissues, urine and faeces extractions were subjected to acid hydrolysis, extracted with either ethyl acetate or ethyl ether and the organo-soluble components analysed. Non extractable residues of milk, tissues, fat and faeces were air dried prior to combustion and radio-assay. Selected non-extractable residues were subjected to acid hydrolysis with analysis of the organo-soluble components.

The mean total recovery of radioactivity following dosing with [cyclopropyl-1-¹⁴C]-fenpropathrin was 65.2% (63.9–66.5%) with 40.9% (40.0–41.8) recovered in the urine and 23.5% (23.0–24.0) in the faeces. Excretion via milk was a minor route with radioactivity accounting for *ca* 0.15% (0.73 (0.497–0.954)) mg fenpropathrin equivalents for the phenoxyphenyl label and *ca* 0.087% (0.43 (0.416–0.449)) mg fenpropathrin equivalents of total dose 500 mg for the cyclopropyl label. Total radioactive residues in the milk reached a steady state by the evening milking on the third day when average residues in the whole milk were 0.11 mg/L for [cyclopropyl-1-¹⁴C]-fenpropathrin treated animals and 0.25 mg/L for the [phenoxyphenyl-¹⁴C]-fenpropathrin treated animals. Less than 3% of the radioactivity in the milk was associated with the butterfat. Most of the retained activity was found in the liver, kidney and fat. Levels in these three organs were in the range of 0.4–0.7 mg/kg fenpropathrin equivalent for both labels. Muscle levels were in the range of 0.02–0.04 mg/kg fenpropathrin equivalent.

Between 20% and 40% of the radiocarbon in the milk from animals receiving the phenoxyphenyl label was associated with the parent compound, with nearly all of the remainder being present as the glycine conjugate of PBacid which reached levels of 0.03–0.15 mg/L (Table 3). There were minor amounts of the hydroxylated derivatives of PBacid (0.003–0.01 mg/kg) and also of fenpropathrin itself (0.02–0.12 mg/kg). With the cyclopropyl label, 57–75% of the activity was associated with the parent material with moderate amounts of TMPA (< 0.002–0.005 mg/kg), its hydroxymethyl (< 0.002–0.003 mg/kg) and carboxy (< 0.002–0.003 mg/kg) derivatives and its hydroxymethylated lactone (< 0.002–0.004 mg/kg) (Table 4). In this case, however, the total recovery was only about 70–80% and the concentration of TMPA and all its derivatives did not exceed 0.01 mg/L. The identity of the compounds associated with the radioactivity in the tissues was somewhat similar except that in the liver and kidney there were only traces left as parent material (Table 5). In the case of the phenoxyphenyl label, most of the activity was in the form of PBacid (kidney 0.22–0.34 mg/kg; liver 0.05–0.07 mg/kg) and its glycine conjugate (kidney 0.21–0.38 mg/kg; liver 0.06–0.11 mg/kg) and in the case of the cyclopropyl label TMPA and its derivatives

predominated. The hydroxymethyl TMPA-lactone (TMPA-CH₂OH lactone) was practically absent from the fat but was prominent in muscle, liver and kidney, accounting for up to 40% of the activity in the kidney, equivalent to about 0.2 mg/kg.

Table 2 Distribution of radioactive residues in milk and tissues following administration of [phenoxyphenyl-¹⁴C]-fenpropathrin or [cyclopropyl-1-¹⁴C]-fenpropathrin to lactating goats for 5 consecutive days

Tissue		Radioactive residue (mg/kg)					
		[phenoxyphenyl- ¹⁴ C]-fenpropathrin			[cyclopropyl-1- ¹⁴ C]-fenpropathrin		
		Goat 12	Goat 16	Mean	Goat 10	Goat 13	Mean
Liver		0.329	0.532	0.431	0.495	0.432	0.464
Kidney		0.514	0.863	0.689	0.521	0.441	0.481
Heart		0.0534	0.113	0.0832	0.130	0.110	0.120
Muscle ^a		0.0177	0.0253	0.0215	0.0428	0.0394	0.0411
Fat ^a		0.451	0.852	0.652	0.748	0.556	0.652
Blood ^b		0.0933	0.127	0.110	0.197	0.245	0.221
Blood ^c		0.0618	0.0332	0.0475	0.184	0.244	0.214
Blood ^d		0.0834	0.093	0.0882	0.178	0.228	0.203
Milk:							
Study day	-1 (am)	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	-1 (pm)	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	1 (am)	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	1 (pm)	0.139	0.282	0.211	0.117	0.0666	0.0918
	2 (am)	0.0682	0.0850	0.0766	0.0609	0.0453	0.0531
	2 (pm)	0.190	0.240	0.215	0.115	0.0877	0.101
	3 (am)	0.0956	0.151	0.123	0.0820	0.0609	0.0715
	3 (pm)	0.186	0.313	0.250	0.141	0.0816	0.111
	4 (am)	0.107	0.0986	0.103	0.0842	0.0624	0.0733
	4 (pm)	0.223	0.278	0.251	0.119	0.0853	0.102
	5 (am)	0.101	0.0958	0.0984	0.103	0.0654	0.0842
	5 (pm)	0.114	0.0989	0.106	0.101	0.0612	0.0811

^a mean mg/kg value for the tissue samples representing a specific sample matrix

^b taken at sacrifice,

^c prior to the final dose,

^d taken at last milking

LOQ – limit of quantification (0.0004 mg/kg)

Table 3 Summary of the nature of the ^{14}C -fenpropathrin residues in milk following dosing with [phenoxyphenyl- ^{14}C]-fenpropathrin

[phenoxyphenyl- ^{14}C]-fenpropathrin														
	4'-OH-PBacid		PBacid-glycine		PBacid		Fenpropathrin-CH ₂ OH		4'-OH-Fenpropathrin		Fenpropathrin		Total ^a	
Milk	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Goat 12														
3 (am)	8.52	0.0083	44.17	0.0431	10.85	0.0106	1.43	< 0.002	0.72	< 0.002	18.98	0.0185	84.67	0.0976
3 (pm)	3.75	0.0073	41.64	0.0815	11.05	0.0216	2.01	0.0039	0.89	< 0.002	24.25	0.0475	83.59	0.1958
4 (pm)	3.83	0.0096	60.66	0.1515	1.27	0.0032	0.73	< 0.002	0.47	< 0.002	27.99	0.0699	94.95	0.2498
5 (am)	5.80	0.0070	56.83	0.0682	2.37	0.0028	0.67	< 0.002	0.34	< 0.002	22.67	0.0272	88.68	0.1200
5 (pm)	4.22	0.0045	37.51	0.0397	5.87	0.0062	2.27	0.0024	2.08	0.0022	28.98	0.0307	80.93	0.1058
Goat 16														
3 (am)	5.53	0.0059	46.98	0.0498	4.68	0.0050	1.28	< 0.002	1.00	< 0.002	28.94	0.0307	88.41	0.1060
3 (pm)	2.93	0.0088	45.06	0.1349	4.96	0.0149	0.86	0.0026	0.54	< 0.002	39.31	0.1177	93.66	0.2994
4 (pm)	3.84	0.0103	48.52	0.1303	2.96	0.0080	0.81	0.0022	0.85	0.0023	36.24	0.0973	93.22	0.2686
5 (am)	8.36	0.0078	32.52	0.0302	5.98	0.0055	1.67	< 0.002	1.09	< 0.002	28.14	0.0261	77.76	0.0928
5 (pm)	3.95	0.0026	41.94	0.0279	3.18	0.0021	6.32	0.0072	2.19	< 0.002	30.31	0.0202	87.89	0.0666

Table 4 Summary of the nature of the ^{14}C -fenpropathrin residues in milk following dosing with [cyclopropyl-1- ^{14}C]-fenpropathrin

[cyclopropyl-1- ^{14}C]-fenpropathrin																
	TMPA-CH ₂ OH		TMPA-COOH		TMPA-CH ₂ OHlactone		TMPA		Fenpropathrin-CH ₂ OH		4'-OH-Fenpropathrin		Fenpropathrin		Total ^a	
	%TR R	mg/kg	%TR R	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Goat 10																
3 (am)	2.88	0.0024	0.45	< 0.002	2.68	0.0022	5.66	0.0047	0.22	< 0.002	0.00	< 0.002	65.95	0.0547	78.10	0.0830
3 (pm)	1.93	0.0030	1.90	0.0029	2.20	0.0034	2.23	0.0034	0.05	< 0.002	0.50	< 0.002	74.83	0.1154	83.64	0.1542
4 (pm)	2.80	0.0034	0.42	< 0.002	3.17	0.0039	3.10	0.0038	0.66	< 0.002	1.98	0.0024	66.09	0.0804	78.22	0.1217
5 (am)	0.92	< 0.002	1.14	< 0.002	1.20	< 0.002	2.14	0.0021	0.42	< 0.002	1.06	< 0.002	72.21	0.0724	79.09	0.1003
5 (pm)	0.32	< 0.002	1.60	< 0.002	1.43	< 0.002	2.34	0.0025	0.63	< 0.002	0.77	< 0.002	74.99	0.0790	82.08	0.1053
Goat 13																
3 (am)	3.27	0.0020	1.01	< 0.002	1.68	< 0.002	2.41	< 0.002	0.27	< 0.002	1.80	< 0.002	62.19	0.0373	72.63	0.0599
3 (pm)	2.71	0.0024	0.90	< 0.002	3.89	0.0034	1.82	< 0.002	0.47	< 0.002	1.00	< 0.002	64.91	0.0570	75.70	0.0878
4 (pm)	3.15	0.0031	1.01	< 0.002	3.30	0.0032	2.38	0.0023	1.11	< 0.002	3.04	0.0030	57.58	0.0565	71.57	0.0982
5 (am)	3.05	< 0.002	1.68	< 0.002	3.62	0.0021	2.07	< 0.002	0.55	< 0.002	1.01	< 0.002	56.53	0.0325	68.51	0.0575
5 (pm)	2.98	< 0.002	0.29	< 0.002	1.82	< 0.002	1.98	< 0.002	0.34	< 0.002	0.33	< 0.002	67.34	0.0398	75.08	0.0591

^a ^{14}C - Fenpropathrin equivalents,^b Fractions containing TMPA-CH₂OH may contain TMPA-COOH,^c Fractions containing Fenpropathrin-CH₂OH may contain Desphenyl-Fenpropathrin or Fenpropathrin-COOH

Table 5 Summary of the nature of the ^{14}C -fenpropathrin residues in tissues following dosing with [phenoxyphenyl- ^{14}C]-fenpropathrin

[phenoxyphenyl- ¹⁴ C]-fenpropathrin														
	4'-OH-PBacid		PBacid-glycine		PBacid		Fenpropathrin-CH ₂ OH ^c		4'-OH-Fenpropathrin		Fenpropathrin		Total ^a	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Goat 12														
Rear leg muscle	2.19	< 0.002	8.73	< 0.002	9.94	< 0.002	5.12	< 0.002	1.16	< 0.002	50.15	0.0075	77.29	0.0150
Loin muscle	2.18	< 0.002	16.62	0.0048	10.03	0.0029	1.33	< 0.002	1.32	< 0.002	51.67	0.0148	83.15	0.0287
Omental fat	0.75	0.0029	4.95	0.0193	1.83	0.0071	0.87	0.0034	0.29	< 0.002	76.65	0.2986	85.34	0.3895
Perirenal fat	0.69	0.0034	5.07	0.0248	1.97	0.0097	0.83	0.0041	0.57	0.0020	78.29	0.3837	87.42	0.4901
Kidney	9.42	0.0533	36.42	0.2059	39.77	0.2248	0.64	0.0036	0.45	0.0025	0.88	0.0050	87.58	0.5653
Heart	1.91	< 0.002	22.97	0.0126	7.38	0.0041	1.76	< 0.002	1.24	< 0.002	40.40	0.0222	75.66	0.0550
Liver	9.77	0.0327	19.31	0.0645	14.80	0.0494	4.84	0.0162	2.54	0.0085	3.02	0.0101	54.28	0.3342
Goat 16														
Rear leg muscle	4.18	< 0.002	30.19	0.0096	10.33	0.0033	1.69	< 0.002	0.88	< 0.002	42.58	0.0135	89.85	0.0317
Loin muscle	1.71	< 0.002	34.00	0.0089	13.42	0.0035	1.11	< 0.002	0.53	< 0.002	34.88	0.0090	85.65	0.0259
Omental fat	0.31	0.0025	3.71	0.0294	1.20	0.0095	0.86	0.0068	0.26	0.0021	81.28	0.6448	87.62	0.7933
Perirenal fat	0.48	0.0043	5.03	0.0454	2.68	0.0242	0.78	0.0070	0.36	0.0032	76.70	0.6918	86.03	0.9020
Kidney	8.76	0.0813	40.70	0.3778	36.80	0.3416	0.38	0.0035	0.60	0.0056	1.60	0.0149	88.84	0.9280
Heart	3.32	0.0038	12.21	0.0141	3.37	0.0039	1.70	0.0020	0.61	< 0.002	61.28	0.0707	82.49	0.1154
Liver	12.15	0.0638	20.56	0.1079	12.48	0.0655	6.51	0.0342	2.38	0.0125	3.34	0.0175	57.42	0.5250

Fenpropathrin

Table 6 Summary of the nature of the ^{14}C -fenpropathrin residues in tissues following dosing with [cyclopropyl-1- ^{14}C]-fenpropathrin

[cyclopropyl-1- ^{14}C]-fenpropathrin																
	TMPA-CH ₂ OH ^b		TMPA-COOH		TMPA-CH ₂ OHlactone		TMPA		Fenpropathrin-CH ₂ OH ^c		4'-OH-Fenpropathrin		Fenpropathrin		Total ^a	
	%TR R	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TR R	mg/kg	%TR R	mg/kg	%TRR	mg/kg	%TR R	mg/kg	%TR R	mg/kg
Goat 10																
Rear leg muscle	15.29	0.0071	3.88	< 0.002	18.84	0.0088	7.32	0.0034	1.97	< 0.002	1.91	< 0.002	12.58	0.0059	61.79	0.0467
Loin muscle	5.61	0.0022	13.04	0.0052	20.48	0.0082	3.74	< 0.002	1.62	< 0.002	0.97	< 0.002	10.30	0.0041	55.76	0.0401
Omental fat	0.75	0.0059	0.26	0.0020	1.80	0.014	1.00	0.008	0.54	0.004	0.48	0.004	88.10	0.688	92.93	0.781
Perirenal fat	0.43	0.0033	0.50	0.0038	2.61	0.02	0.90	0.0068	0.27	0.0021	0.31	0.0024	84.81	0.6447	89.83	0.7602
Kidney	15.94	0.0861	3.71	0.0200	41.33	0.223	6.67	0.0360	1.25	0.0068	0.62	0.0033	1.68	0.0091	71.20	0.5400
Heart	5.98	0.0083	0.93	0.0013	7.69	0.0106	2.42	0.003	0.73	< 0.002	0.36	< 0.002	52.89	0.0732	71.0	0.1384
Liver	15.59	0.0768	7.13	0.0350	14.62	0.0720	19.19	0.094	4.02	0.02	2.51	0.012	2.61	0.013	65.67	0.493
Goat 13																
Rear leg muscle	16.74	0.0076	4.23	< 0.002	18.15	0.0082	5.89	0.003	1.08	< 0.002	0.78	< 0.002	14.9	0.008	61.80	0.046
Loin muscle	6.78	0.0024	14.00	0.0052	20.32	0.0074	7.25	0.003	3.93	< 0.002	0.95	< 0.002	7.30	0.003	60.53	0.038
Omental fat	1.06	0.0062	0.58	0.0034	2.22	0.0130	1.58	0.009	0.86	0.0051	0.56	0.0033	81.4	0.478	88.27	0.588
Perirenal fat	0.60	0.0033	1.18	0.0064	3.80	0.0206	1.62	0.009	0.95	0.0051	0.54	0.0029	68.4	0.371	77.12	0.542
Kidney	13.05	0.0606	4.13	0.0192	40.22	0.1867	7.99	0.037	1.56	0.0072	0.87	0.0040	1.27	0.006	69.09	0.464
Heart	10.32	0.0121	1.95	0.0023	6.74	0.0079	5.24	0.006	0.69	< 0.002	0.77	< 0.002	42.2	0.049	67.89	0.117
Liver	16.05	0.0693	4.39	0.0190	10.47	0.0452	17.30	0.075	2.27	0.0098	2.08	0.0090	2.28	0.01	54.84	0.432

^a ^{14}C - Fenpropathrin equivalents,^bFractions containing TMPA-CH₂OH may containing TMPA-COOH,^cFractions containing Fenpropathrin-CH₂OH may contain Desphenyl-Fenpropathrin or Fenpropathrin-COOH

Laying Hens

Fenpropathrin, labelled in either the cyclopropyl or the benzyl ring was administered in capsules to laying hens daily for 10 days (Ku and Doran, 1990, Reference FM-01-0042). The group of 10 hens were dosed with [phenoxyphenyl- ^{14}C]-fenpropathrin (benzyl-label) and [cyclopropyl-1- ^{14}C]-fenpropathrin (cyclo-label) at a nominal rate of either 0.5 or 5 mg/kg body weight. Eggs were collected every morning and evening and excreta daily. The hens were all sacrificed within four hours of the last dose and kidneys, liver, heart, gizzard (washed without contents), ovaries and contents, muscle and skin were retained for analysis.

The samples were combusted and the ^{14}C activity was determined with liquid scintillation counter. Liquid samples were directly analysed with LSC. The residues in eggs and tissues were extracted with acetonitrile/1% conc. HCl. The organic phase was evaporated and the remaining aqueous phase was diluted and partitioned into ethyl acetate. The concentrated extract was analysed with HPLC. The remaining aqueous fraction was hydrolysed by refluxing it at 80–90 °C with 6N HCl overnight. The radioactivity was determined with LSC. The remaining radioactivity in PES was also determined with LSC. Portions of egg, meat, fat and liver were also analysed with gas chromatography based on acetone/hexane (1/2 v/v) extraction, silica gel clean-up. The polar metabolites were determined after methylation with diazomethane. The compounds were identified with GC/MS applying authentic analytical standards.

The recovery of total radioactivity from excreta, eggs and tissues was between 75 and 84% of the total applied dose. Between 98.9 and 99.6% of the recovered activity was found in the faeces irrespective of the label. Approximately 0.05% of the applied benzyl label was found in the eggs and 0.2% of the cyclopropyl label. The total radioactive residues expressed as fenpropathrin equivalent are summarised in Table 7.

Table 7 Total radioactive residues [mg/kg] expressed as fenpropathrin and parent fenpropathrin found following treatments with benzyl-labelled and cyclopropyl-labelled fenpropathrin at 5 ppm level

Sample	Benzyl -label		Cyclopropyl -label	
	Total, mg/kg	Parent, mg/kg	Total, mg/kg	Parent, mg/kg
Egg	0.05-0.22	0.016-0.07	0.22-0.49	0.014-0.061
Meat, breast	0.07	0.02	0.53	0.0324
Meat, thigh	0.16	0.0376	0.57	0.033
Fat	0.87	0.4315	0.90	0.5781
Liver	1.46	0.0143	2.91	0.0357
Kidney	4.56	0.0963	4.19	0.2127
Heart	0.37	0.0838	1.35	0.0825
Gizzard	5.08	0.6563	4.86	0.9492
Skin	0.48	0.1653	0.81	0.2164
Excreta	48.8-67.5	2.72-4.82	50.7-63.3	3.69-4.01

The percent proportion and concentration of parent compound and the major metabolites, expressed as parent fenpropathrin, in eggs and tissues after treatments at 5 ppm level are shown in Tables 8–13.

Table 8 Distribution of residues in eggs after treatment with [benzyl-¹⁴C]-fenpropathrin at 5 mg/kg bw.

Component	Percent	mg/kg
Fenpropathrin	29.90-32.38	0.0161-0.0698
4'-OH PBacid	4.07-5.88	0.003-0.0129
PBacid-glycine	1.72-5.03	0.0026-0.0093
PBacid	3.23-3.68	0.0017-0.0079
Fenpropathrin-CH ₂ -OH	3.98-6.08	0.0032-0.0117
4'-OH- Fenpropathrin	1.33-1.7	0.0009-0.0029
3-OH-BAacid	4.53-5.68	0.0023-0.0108

Table 9 Distribution of residues in eggs after treatment with [cyclopropyl-1-¹⁴C]-fenpropathrin at 5 mg/kg bw.

Component	Percent	mg/kg
Fenpropathrin	6.05 - 13.34	0.014-0.061
TMPA-CH ₂ OH	10.24-11.73	0.023-0.053
TMPA-COOH	2.23-3.01	0.005-0.013
TMPA-CH ₂ -lactone	5.97-6.96	0.016-0.032
TMPA	5.91-9.02	0.02-0.029
Fenpropathrin-CH ₂ -OH	1.55-2.33	0.004-0.012
4'-OH- Fenpropathrin	1.51-4.29	0.01-0.013

Table 10 Percent distribution of residues in chicken muscle after treatment with [phenoxyphenyl-¹⁴C]-fenpropathrin

Component	Breast muscle		Thigh muscle	
	Percent	mg/kg	Percent	mg/kg
Fenpropathrin	14.75	0.0100	23.12	0.0376
4'-OH PBacid	9.01	0.0061	5.23	0.0085
PBacid-glycine	5.11	0.0034	2.48	0.0040
PBacid	23.90	0.161	19.84	0.0323
Fenpropathrin-CH ₂ -OH	2.33	0.0016	2.65	0.0043
4'-OH- Fenpropathrin	1.73	0.0012	1.95	0.0032
3-OH-BAacid	16.02	0.011	9.34	0.0152

Table 11 Percent distribution of residues in chicken muscle after treatment with [cyclopropyl-1-¹⁴C]-fenpropathrin

Component	Breast muscle		Thigh muscle	
	Percent	mg/kg	Percent	mg/kg
Fenpropathrin	6.09	0.0324	5.82	0.0331
TMPA-CH ₂ OH	14.47	0.0769	18.013	0.1032
TMPA-COOH	7.72	0.0410	4.54	0.0258
TMPA-CH ₂ -lactone	12.93	0.0687	11.66	0.0664
TMPA	15.14	0.0804	16.27	0.0926
Fenpropathrin-CH ₂ -OH	0.60	0.0032	1.06	0.006
4'-OH- Fenpropathrin	0.97	0.0052	1.49	0.0085

Table 12 Percent distribution of residues in chicken liver, kidney skin after treatment with [phenoxyphenyl-¹⁴C]-fenpropathrin

Component	Liver	Kidney	Skin	Fat	Gizzard
Fenpropathrin	0.98	2.11	34.51	49.63	12.91
4'-OH PBacid	16.10	26.02	12.53	0.65	14.38
PBacid-glycine	5.62	6.23	2.4	0.39	8.67
PBacid	14.65	8.73	7.73	1.48	6.89
Fenpropathrin-CH ₂ -OH	1.33	0.63	3.13	2.84	1.70
4'-OH- Fenpropathrin	0.84	0.44	1.01	1.24	0.70
3-OH-BAacid	28.89	35.35	12.70	3.77	12.25

Table 13 Percent distribution of residues in chicken liver, kidney, skin after treatment with [cyclopropyl-1-¹⁴C]-fenpropathrin at 5 ppm level

Component	Liver	Kidney	Skin	Fat	Gizzard
Fenpropathrin	1.23	5.08	26.73	63.91	19.54
TMPA-CH ₂ OH	14.71	7.71	13.00	2.20	5.22
TMPA-COOH	11.37	15.28	3.15	0.85	6.89
TMPA-CH ₂ -lactone	14.81	5.3	6.95	5.64	6.08
TMPA	26.38	47.49	15.37	14.23	17.18
Fenpropathrin-CH ₂ -OH	1.77	1.02	3.28	4.28	22.99
4'-OH- Fenpropathrin	1.13	1.28	1.15	1.57	2.89

Summary of animal metabolism

The major biotransformation reactions of fenpropathrin in animals consisted of oxidation at the methyl groups of the acid moiety and at the 2'- or 4'-positions of the alcohol moiety, cleavage of the ester and ether linkages and conjugation of the resultant carboxylic acids and alcohols. The proposed metabolic pathway for fenpropathrin in animals is shown in Figure 1.

equivalent to 0.45 kg ai/ha (Chen and Abell, Report FM-41-0031,1985 and Reference FM-51-0022, 1986). Samples were collected 14 days after the final application. Apple fruit was washed with methanol and separated into peel and pulp. Samples were pulverized with dry ice then extracted with acetone and methanol/water. Analysis was by autoradiography/TLC/LSC. The radioactivity remaining in un-extractable material was determined by combustion/LSC. Free metabolites were separated from conjugated metabolites by solid phase chromatography on a silica column eluted with hexane/acetone, methanol/acetone and methanol. To release aglycones, conjugated metabolites were subjected either to enzyme hydrolysis using cellulase or β -glucosidase then acidified and extracted with diethyl ether, or to chemical hydrolysis with both acid and base. Characterization of metabolites was by radio-TLC.

Table 14 Distribution of ^{14}C -fenpropathrin in apple tree samples

Sample	Benzyl-label		Cyclopropyl-label	
	mg/kg fenpropathrin equivalent	% TRR	mg/kg fenpropathrin equivalent	% TRR
Leaves	12.2	61.0	15.9	72.1
Branches	4.0	13.9	2.5	4.2
Fruit	2.11	25.1	1.40	24.7

Table 15 Extractability of ^{14}C -fenpropathrin radioactivity (%TRR)

	Leaves		Branches	Fruit	
	Benzyl-label	Cyclopropyl-label	Benzyl-label	Benzyl-label	Cyclopropyl-label
Acetone	81	87	89	94	94
Methanol/water	11	8	6	3	3
Un-extractable	8	5	5	3	3

The cyclopropyl- ^{14}C -treated branches contained only 2.5% of TRR and therefore no further analysis was carried out.

Table 16 Identity of fenpropathrin metabolites in apple fruits and apple plants (%TRR)

	Apple fruits		Apple plants	
	Benzyl-label	Cyclopropyl-label	Benzyl-label	Cyclopropyl-label
Fenpropathrin	91.8	93.6	60.6	65.6
Conjugated metabolites	2.5	2.5		
4'-OH-Fenpropathrin	0.8	0.8	1.9	1.8
Fenpropathrin-CH ₂ OH	0.6	0.6	1.6	1.3
Fenpropathrin-CH ₂ OH conjugates			1.1	0.9
2'- or 4'-OH-Fenpropathrin-CH ₂ OH conjugates			0.1	0.3
PBalc conjugates			4.8	
PBacid conjugates			1.3	
2'- or 4'-OH-PBalc conjugate			1.0	
2'- or 4'-OH-PBacid conjugates			1.2	
TPMA conjugates				4.3
TPMA-CH ₂ OH conjugates				3.4
TPMA-CH ₂ OH lactone conjugates				1.1
Other free metabolites	1.4*	0.2*	6.7**	3.5**
Other conjugated metabolites			12.0***	13.0***
Non-extractable	2.9	2.2	7.7	4.8

* contained at least 2 metabolites

** contained at least 5 metabolites

*** contained at least 10-20 metabolites

Tomatoes

In studies carried out in California, USA in 1983, greenhouse-grown tomato plants (cv. Bonny Best) were treated four times, 7–8 days apart, with [cyclopropyl-1-¹⁴C]-fenpropathrin and [benzyl-¹⁴C]-fenpropathrin (both radiochemical purity >99%) at rates equivalent to 0.224 kg ai/ha (Chen and Abell, 1985 and 1986; References FM-41-0031 and FM-51-0023). Fruit and leaves were extracted at harvest (PHI 19 days) as described for apples.

Table 17 Distribution of ¹⁴C-fenpropathrin in tomato plant samples expressed as fenpropathrin equivalent

Sample	Benzyl-label	Cyclopropyl-label
	mg/kg	mg/kg
Leaves	5.8	4.0
Stem and petioles	0.49	0.53
Fruit	0.10	0.04

Table 18 Extractability of ¹⁴C-fenpropathrin radioactivity (%TRR)

	Leaves		Stems and petioles	
	Benzyl-label	Cyclopropyl-label	Benzyl-label	Cyclopropyl-label
Acetone	78	78	78	80
Methanol/water	13	15	12	10
Unextractable	9	7	10	10

Table 19 Characterization of fenpropathrin metabolites in tomatoes (%TRR)

	Benzyl-label	Cyclopropyl-label
Fenpropathrin	66.1	29.6
Conjugated metabolites	28.2	58.6
Free metabolites	0.2	5.1
Un-extractable	5.5	6.7

Table 20 Identity of fenpropathrin metabolites in tomato plants (%TRR)

	Benzyl-label	Cyclopropyl-label
Fenpropathrin	36.5	39.1
Fenpropathrin-CH ₂ OH conjugates	2.2	2.1
4'-OH-Fenpropathrin-CH ₂ OH conjugates	1.0	0.9
Fenpropathrin-(CH ₂ OH) ₂	3.1	2.7
PBald conjugates	0.4	
4'-OH-PBacid conjugates	7.0	
PBacid conjugates	2.0	
TMPA conjugates		4.6
TMPA-CH ₂ OH conjugates		7.5
Other free metabolites	5.7*	5.1*
Other conjugated metabolites	33.1**	30.6**

	Benzyl-label	Cyclopropyl-label
Non-extractable	9.3	7.4

* contained at least 10 metabolites

** contained at least 15 metabolites

In the fruit, the radioactivity was too low to allow full characterization, but some two thirds were present as unchanged fenpropathrin with a further 28% as conjugated metabolites. In the leaves, only 30% of the total residue was present as parent and just under 60% as conjugated metabolites. In the case of the benzyl label, the most prominent metabolites were conjugates of PBacid and its 4'-hydroxy derivative (4'-OH-PBacid), although these only constituted a minor proportion of the total radioactivity. The main metabolites reported in the case of the cyclopropyl label were conjugates of TMPA and hydroxymethyl-TMPA (TMPA-CH₂OH).

In studies carried out in California, USA in 1994, greenhouse-grown tomato plants (cv. 5715) were sprayed four times with a 2.4% EC formulation containing [cyclopropyl-1-¹⁴C]-fenpropathrin (radiochemical purity 98.7%) and [phenoxyphenyl-¹⁴C]-fenpropathrin (radiochemical purity 98.4%) at rates equivalent to 0.224 kg ai/ha (Toia, *et. al.*, 1995; Reference FM-51-0070). Fruit and leaves were collected at harvest (PHI 3 days); fruit were washed with hexane/acetone. Total radioactivity was determined by combustion/LSC. The washes were evaporated, redissolved in hexane/acetone and analysed by radio-HPLC/TLC. The major radiolabelled zone was removed from the TLC plate, dissolved in acetone, filtered, evaporated and analysed by GC-MS. Tomato fruit were extracted using acetonitrile with the supernatant being analysed using LSC. Further extractions were made using methanol and dichloromethane. Extracts were analysed using radio-HPLC with UV detection and TLC. Radioactivity in dried, un-extracted material was determined by combustion/LSC. Soil samples were analysed by combustion/LSC to determine the total radioactive residue; only small amounts of radioactivity were detected.

Table 21 Radioactive residues in tomatoes following treatment with [¹⁴C]-fenpropathrin

	Phenoxyphenyl-label		Cyclopropyl-label	
	mg/kg	%TRR	mg/kg	%TRR
Rinse	0.34	33.6	0.23	27.3
Rinsed fruit	0.68	66.4	0.62	72.8
Total	1.02	100	0.85	100

The majority of radioactivity was attributed to parent compound but small amounts of *trans*-TMPA-COOH and/or *trans*-TMPA-CH₂OH was tentatively identified.

Table 22 Radioactivity in extracts from tomato plants treated with ¹⁴C-fenpropathrin; mg/kg fenpropathrin equivalents (dichloromethane extracts not included)

	Available	Fenpropathrin		Polar material		Miscellaneous	
		mg/kg	%	mg/kg	%	mg/kg	%
Phenoxyphenyl-label							
Rinses	0.34	0.34	97.9	0.00	0.0	<< 0.01	2.1

Extracts	0.65	0.62	95.3	0.01	2.1	<< 0.01	2.7
Total	0.99	0.96	96.2	0.01	1.3	0.03	2.5
Cyclopropyl-label							
Rinses	0.23	0.23	98.9	0.00	0.0	<< 0.01	1.1
Extracts	0.53	0.52	97.7	0.01	1.8	<< 0.01	0.05
Total	0.76	0.75	98.0	0.01	1.3	0.01	0.7

Beans

In 1984, five pinto bean plants (cv. Idaho 111) grown in a greenhouse in California, was treated three times with [cyclopropyl-1-¹⁴C]-fenpropathrin and [benzyl-¹⁴C]-fenpropathrin (both radiochemical purity >99%) at a rate equivalent to 0.224 kg ai/ha (Chen and Abell, 1985, 1986; References FM-41-0031 and FM-51-0024). Samples were collected 15 days after the final application. Fruit and leaves were extracted at harvest as described for apples.

Table 23 Distribution of ¹⁴C-fenpropathrin in bean plant samples expressed as fenpropathrin equivalent

Sample	Benzyl-label mg/kg	Cyclopropyl-label mg/kg
Leaves	8.8	5.1
Stem and petioles	1.3	0.6
Bean pods	0.1	0.1
Beans	0.03	0.07

Table 24 Extractability of ¹⁴C-fenpropathrin radioactivity (%TRR)

	Leaves		Stems and petioles	
	Benzyl-label	Cyclopropyl-label	Benzyl-label	Cyclopropyl-label
Acetone	63	73	74	81
Methanol/water	32	22	20	13
Unextractable	5	5	6	6

Table 25 Characterization of fenpropathrin metabolites in beans (%TRR)

	Benzyl-label	Cyclopropyl-label
Fenpropathrin	4.1	0.1
Conjugated metabolites	60.7	50.7
Free metabolites	17.0	4.1
Unextractable	18.2	45.1

Table 26 Identity of fenpropathrin metabolites in pinto beans leaves (%TRR)

	benzyl-label	Cyclopropyl-label
Fenpropathrin	46.7	46.4
PBald conjugates	19.5	
PBalc conjugates	4.0	
4'-OH PBacid conjugates	9.8	
2'-OH PBalc conjugates	0.4	
4'-OH PBalc conjugates	0.3	
PBacid conjugates	1.1	
TMPA conjugates		7.0
TMPA-CH ₂ OH conjugates		16.7

	benzyl-label	Cyclopropyl-label
TMPA-CH ₂ OH lactone conjugates		2.2
Other free metabolites	4.6**	3.3**
Other conjugated metabolites	8.5*	8.4**
Unknown conjugate		11.2***
Non-extractable	5.1	4.8

* contained at least 10 metabolites

** contained at least 6 metabolites

*** artefact of the acid hydrolysis product

Cotton

Cotton pants were treated four times with [cyclopropyl-1-¹⁴C]-fenpropathrin and [phenoxyphenyl-¹⁴C]-fenpropathrin (both radiochemical purity > 99%) applying a total of *ca.* 4.7–4.8 mg ¹⁴C-fenpropathrin (Hitchings and Roberts, 1977; Reference FM-81-0005). The plant parts were extracted with acetonitrile/water and the main products in the extracts were found to be fenpropathrin itself with small amounts of TMPA and PBacid together with some polar material which is likely to have consisted primarily of conjugates of either PBacid or TMPA. In the leaves at harvest (the interval between treatment and harvest was 66 days for the benzyl label and 111 days for the cyclopropyl label) the total remaining activity included 70% parent in the case of the cyclopropyl label and 55% parent in the case of the phenoxyphenyl label. Most of the remaining activity was tentatively accounted for by PBacid and TMPA, mainly in conjugated forms. Examination of the plants grown on soils treated with 0.5 kg/ha of fenpropathrin and containing 0.86 mg/kg fenpropathrin equivalent showed only extremely low uptake of radioactivity (0.002 mg/kg in leaves and 0.01 mg/kg in bolls), demonstrating limited tendency for translocation.

In a study carried out outdoors in California in 1992, cotton plants (cv Acala Royale) were sprayed with [phenoxyphenyl-¹⁴C]- or [cyclopropyl-¹⁴C]-fenpropathrin (97.9% or > 99.9% radiochemical purity) on 4 occasions at a rate equivalent to 0.336 kg as/ha (total application rate 1.344 kg as/ha) (Kimmel, 1994, Reference FM-41-0067). Plants were harvested by hand 21 days after the final application. Seeds were delinted by hand and extracted using hexane and extracted radioactivity was analysed by LSC, TLC, HPLC-UV and GC-MS. The un-extracted material was further extracted with methanol/water, followed by diluted hydrochloric acid/methanol. Cotton lint and foliage were extracted using hexane/acetone and analysed by LSC, TLC and HPLC. Unextracted material was further extracted using methanol/water and analysed by LSC. The results are shown in Tables 27–29.

Table 27 Distribution of radioactivity of cotton plant parts expressed as fenpropathrin equivalents

	Phenoxyphenyl label		Cyclopropyl label	
	mg/kg	%TRR	mg/kg	%TRR
Seed rinses	0.71	62.7	1.07	67.5
Rinsed seed	0.42	37.3	0.52	32.6
Total seed	1.14	100.0	1.59	100.0
Lint rinses	48.6	63.9	31.8	76.2

	Phenoxyphenyl label		Cyclopropyl label	
	mg/kg	%TRR	mg/kg	%TRR
Rinsed lint	27.4	36.1	10.0	23.8
Total lint	76.0	100.0	41.8	100.0
Foliage	78.6	100.0	67.7	100.0

Table 28 The extractability of radioactivity from cotton plant parts (mg/kg fenpropathrin equivalents)

Label/sample	Hexane	Hexane/ acetone	Methanol/ water	Methanol/ HCl	Total extracted	Unextracted	Accountability (%TRR)
Phenoxyphenyl							
seeds	0.34	0.04	0.10	0.01	0.49	0.01	118.9
lint	-	17.2	6.2	-	23.4	4.0	100.0
foliage	-	55.8	22.2	-	78.0	-	-
Cyclopropyl							
seeds	0.36	0.05	0.10	0.01	0.51	0.02	101.6
lint	-	6.1	2.7	-	8.8	1.1	100.0
foliage	-	41.9	19.0	-	60.9	-	-

The majority of the radioactivity was associated with parent compound although a small number of low level metabolites were also detected.

Table 29 The Identity of fenpropathrin metabolites in cotton plant parts (mg/kg fenpropathrin equivalents)

	Phenoxyphenyl				Cyclopropyl			
	seed rinse	seed extract	lint	foliage	seed rinse	seed extract	lint	foliage
Fenpropathrin	0.71 (100%)	0.45 (93.8%)	69.2 (96.2%)	54.2 (69.5%)	1.1 (99.5%)	0.43 (85.6%)	40.7 (100%)	41.0 (67.4%)
PBacid		0.01		0.02				
TMPA						0.007		0.08
TMPA-lactone								0.22
Trans-TMPA- CH ₂ OH								1.2
Trans-TMPA- COOH								6.7
4'-OH-PBacid				1.8				
4'-OH- Fenpropathrin								0.06
Desphenyl- Fenpropathrin			0.15					
COOH- Fenpropathrin				0.15				0.26
CONH ₂ - Fenpropathrin				0.13				0.12
Polar material				11.2				9.1
other		12 peaks each < 0.005	Up to 4 peaks each 0.07-1.6	7-12 other peaks	2 peaks 0.002, 0.003	13 peaks each < 0.009- 0.012	3 components	10-17 other peaks

Cabbage

The metabolism of ¹⁴C-fenpropathrin was investigated in cabbages grown and treated in a greenhouse (Mikami, N. *et al.*, 1985; Reference FM-50-0044). ¹⁴C-fenpropathrin labelled at either the cyano

group (referred to as [cyano- ^{14}C]-fenpropathrin), or the $\text{C}_{(1)}$ position of the cyclopropyl ring ([cyclopropyl- ^{14}C]-fenpropathrin), or the phenoxyphenyl ring ([phenoxyphenyl- ^{14}C]-fenpropathrin) were prepared (all radio-chemical purities > 99%). Each of the preparations were dissolved in methanol and evenly applied to the upper surface of two 3rd–4th leaves of cabbage seedlings at a rate of 22 μg per leaf (ca. 25cm², equivalent to 0.09 kg ai/ha). The cabbages were sampled immediately after application and at 3, 7, 14, 21, 28, 35 and 42 days after application.

The cabbage samples were separated into the treated leaves and non-treated shoot portion. The treated leaves were rinsed twice with methanol and the leaves and the untreated shoots were separately homogenized and extracted with a solution of methanol:chloroform:distilled water (4:2:1). Each of the rinse and extract was radioassayed and analysed by TLC. 97.3% of fenpropathrin was recovered from cabbages immediately after application of [cyano- ^{14}C]-fenpropathrin. Direct TLC comparison of metabolites from three labelled preparations was used to distinguish products retaining the ester linkage from hydrolysis products. The extractable components in cabbages harvested 28 and 42 days after application are shown in Table 30.

Table 30 Extractable components in cabbage samples harvested 28 and 42 days after application.

	% of the applied ^{14}C					
	Cyclopropyl- ^{14}C		^{14}CN		phenoxyphenyl- ^{14}C	
	28	42	28	42	28	42
Surface Wash:						
Fenpropathrin	0.6	0.3	1.0	0.6	1.7	0.4
Others	0.3	0.1	0.4	0.3	0.3	0.1
Surface Wash Total	0.9	0.4	1.4	0.9	2.0	0.5
Extracts:						
Fenpropathrin	15.8	11.7	16.9	6.0	12.9	11.3
CONH ₂ -Fenpropathrin	0.7	< 0.1	0.3	< 0.1	0.9	< 0.1
COOH-Fenpropathrin	0.4	0.4	0.6	0.3	0.7	< 0.1
2'-OH-Fenpropathrin	0.1	< 0.1	0.4	< 0.1	0.4	< 0.1
Fenpropathrin-CH ₂ OH	0.4	0.3	0.5	0.5	0.6	0.2
TMPA-lactone	0.1	< 0.1	-	-	-	-
TMPA-CH ₂ OH-lactone	0.8	0.9	-	-	-	-
COOH-Fenpropathrin-conjugate	0.4	0.2	0.6	0.4	0.6	0.7
2'-OH-Fenpropathrin-conjugate	0.2	0.1	0.1	0.1	0.1	0.2
4'-OH-Fenpropathrin-conjugate	1.3	0.7	1.6	1.0	0.6	0.8
Fenpropathrin-CH ₂ OH-conjugate	3.5	4.0	3.4	4.3	4.2	4.0
2'-OH-fenpropathrin-CH ₂ OH-conjugate	4.8	4.5	4.6	4.5	5.9	6.2
4'-OH-fenpropathrin-CH ₂ OH-conjugate						
2'-OH-fenpropathrin-(CH ₂ OH) ₂ -conjugate	20.3	22.0	18.6	20.7	19.4	21.6
4'-OH-fenpropathrin-(CH ₂ OH) ₂ -conjugate						
TMPA-conjugate	0.9	0.8	-	-	-	-
TMPA-CH ₂ OH-conjugate	1.1	1.0	-	-	-	-
TMPA-COOH-conjugate	3.7	4.2	-	-	-	-
TMPA-CH ₂ OH-lactone-conjugate	11.3	11.1	-	-	-	-
PBalc-conjugate	-	-	-	-	0.1	0.1
PBacid-conjugate	-	-	-	-	0.8	1.1
2'-OH-PBacid-conjugate	-	-	-	-	6.9	7.4
4'-OH-PBacid-conjugate	-	-	-	-	4.5	4.6
Others	5.2	5.8	4.2	5.8	9.6	9.1
Extracts Total	71.0	67.7	51.8	43.6	68.2	67.3
Unextractable ^{14}C Total	2.6	5.1	6.7	11.3	4.0	7.5
Treated Leaves Total	74.5	73.2	59.9	55.8	74.2	75.3

	% of the applied ^{14}C					
	Cyclopropyl- ^{14}C		^{14}CN		phenoxyphenyl- ^{14}C	
	28	42	28	42	28	42
Untreated Shoots	0.9	1.2	0.6	0.7	0.4	0.4
Overall Total	75.4	74.4	60.5	56.5	74.6	75.7

The study demonstrated that after foliar application of ^{14}C -fenpropathrin to cabbages the radiocarbon remaining on the treated leaves (as shown by the surface wash) decreased with concomitant increase of ^{14}C in the plant tissues. Most of the recovered radiocarbon was in the treated leaves and less than 1.2% of the applied radiocarbon was found in the untreated shoots indicating that fenpropathrin and its metabolites hardly translocate from the application site to other parts of the plant.

TLC showed that in all cases the predominant radioactive component in the surface washes was the parent, fenpropathrin. Fenpropathrin underwent ester cleavage, hydrolysis of the CN group to the CONH_2 and the COOH groups, hydroxylation at either or both of the gem-dimethyl group with subsequent oxidation to carboxylic acid, and hydroxylation at the 2'- or 4'-position of the phenoxy group. Most of the resultant carboxylic acids and alcohols occurred as glycoside conjugates in plants.

Fate of hydrogen cyanide (HCN) and TMPA in abscised leaves

Mikami, N. *et al.*, also conducted work on the fate of HCN and 2,2,3,3-tetramethylcyclopropanecarboxylic acid (TMPA) in abscised leaves of apple, kidney bean, cabbage, mandarin orange, tomato and vine. 2,2,3,3-tetramethylcyclopropanecarboxylic acid labelled at the $\text{C}_{(1)}$ position of the cyclopropyl ring (^{14}C -TMPA) was prepared. Two abscised leaves from each plant were placed in 100 mL distilled water containing ^{14}C -TMPA at a concentration of 1.0 ppm. After cultivation for five days the leaves were extracted with methanol:chloroform:water (4:2:1).

In a separate experiment, abscised leaves of cabbage and bean plants were placed in a 100 ppm solution of ^{14}C -TMPA in order to obtain large quantities of metabolites for characterization. The extracts were partitioned between ethyl ether and distilled water. After acidification the aqueous layer was partitioned with ethyl acetate. The extractable components in abscised leaves of various plants over a 5 day period are shown in Table 31.

Table 31 Extractable components in abscised leaves of various plants over a 5 day period.

	% of the applied ^{14}C					
	Apple	Bean	Cabbage	Orange	Tomato	Vine
Extracts						
TMPA	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
TMPA-Gu	21.5	14.7	3.2	3.0	2.0	6.6
CH_2OH -TMPA-Gu	5.2	3.2	3.4	0.2	0.2	0.4
TMPA- CH_2OH -Gu	1.3	-	-	-	-	0.1
TMPA-Gu-Gu	5.4	2.8	-	-	12.8	0.7
TMPA-malonyl-Gu	0.7	56.2	70.5	3.1	4.3	0.1
CH_2OH -TMPA-malonyl-Gu	-	-	1.5	0.4	-	-
Others	1.5	3.0	0.2	0.5	1.1	0.2
Extracts Total	35.6	79.9	78.8	7.2	20.4	8.1
Unextractable ^{14}C Total	0.3	0.2	0.2	0.4	0.2	0.3

	% of the applied ^{14}C					
	Apple	Bean	Cabbage	Orange	Tomato	Vine
Abscised Leaves Total	32.9	80.1	79.0	7.6	20.6	8.4
Aqueous Solution						
TMPA	57.1	24.5	13.2	63.8	76.1	83.0
Others	1.2	0.8	0.9	0.7	1.6	0.5
Aqueous Solution Total	58.3	25.3	14.1	64.5	77.7	83.5
Overall Total	94.2	105.4	93.1	72.1	98.3	91.9

TMPA was readily converted in plants to more polar products. The metabolic pathways for TMPA varied dependent upon species of plant. The glucose ester was a main product in apple and vine plants. In orange, cabbages and bean plants, the malonylglucoside was mainly formed.

Further work was carried out using K^{14}CN . Two abscised cabbage leaves were treated for four hours with distilled water containing K^{14}CN and then transferred to K^{14}CN -free distilled water. The treated leaves were extracted at specific intervals after dosing with K^{14}CN , and the extracts were subject to TLC. There was a gradual increase in the amount of volatile ^{14}C trapped in NaOH solution, most of the radiocarbon was considered to be $^{14}\text{CO}_2$. At least six ^{14}C metabolites were present in the extracts of the abscised leaves treated with K^{14}CN . The extractable components in abscised leaves of cabbage over a 2 day period are shown in Table 32.

Table 32 Extractable components in abscised leaves of cabbage over a 2 day period following treatment with K^{14}CN .

Extracts	% of the applied ^{14}C			
	Hours after treatment			
	2	4	8	48
β -Cyanoalanine	0.6	0.9	0.9	0.6
Asparagine	1.8	2.4	2.8	1.1
Aspartic acid	0.7	1.0	1.1	1.3
γ -Glutamyl- β -cyanoalanine	3.8	5.4	5.1	2.4
Others	0.7	1.2	1.1	1.9
Extracts Total	7.6	10.9	11.0	7.3
Unextractable ^{14}C Total	2.9	3.7	4.5	7.7
Treated Leaves Total	10.5	14.6	15.5	15.0
Aqueous Solution	86.9	79.6	83.1	63.2
Overall Total	97.4	94.2	98.6	78.2

This study demonstrates that H^{14}CN liberated on ester hydrolysis of fenpropathrin and its derivatives would be rapidly incorporated into β -cyanoalanine, asparagines, aspartic acid and γ -glutamyl- β -cyanoalanine, with ultimate formation of $^{14}\text{CO}_2$ and unextractable ^{14}C residues.

Summary of plant metabolism

Metabolism of fenpropathrin has been studied in apples, tomatoes (fruiting crops), beans (legume crops), cotton (pulses/oilseed crops) and cabbage (leafy crops). Trials were carried out at approximately recommended rate with fenpropathrin labelled in the phenoxyphenyl, benzyl, cyclopropyl or cyano positions. The majority of radioactivity was found in leaf samples. Low levels of radioactivity were found in fruit/beans. Parent fenpropathrin was the primary component of the

residues in the fruits of the plants, but degradation products constituted the greater part of the residues present in the leaves. Breakdown products/metabolites detected in both fruits and leaves were similar to those detected in rats. It is therefore considered that crop residues are described adequately by defining them as the parent product alone.

The study on cabbage further demonstrated that after foliar application of ^{14}C -fenpropathrin the radiocarbon remaining on the treated leaves (as shown by the surface wash) decreased with concomitant increase of ^{14}C in the plant tissues. Most of the recovered radiocarbon was in the treated leaves and less than 1.2% of the applied radiocarbon was found in the untreated shoots indicating that fenpropathrin and its metabolites hardly translocate from the application site to other parts of the plant.

The general pattern of degradation in all the plant studies include breaking of the ester linkage to produce 3-phenoxybenzoic acid (PBacid) and the corresponding alcohol (PBalc) and aldehyde (PBald). From the acid side of the molecule, the main metabolite is 2,2,3,3-tetramethylcyclopropanecarboxylic acid (TMPA) which can give rise to 2-hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylic acid (TMPA- CH_2OH) and 5-hydroxymethyl-6,6-dimethyl-3-oxabicyclohexan-2-one (TMPA- CH_2OH lactone). PBacid can be hydroxylated at various positions on the phenoxy ring to produce, α -cyano-3-(2'-or 4'-hydroxyphenoxy)benzyl 2,2,3,3-tetramethylcyclopropanecarboxylate (2'-or 4'-OH-fenpropathrin). The proposed metabolic pathway of fenpropathrin in plants is shown in Figure 2.

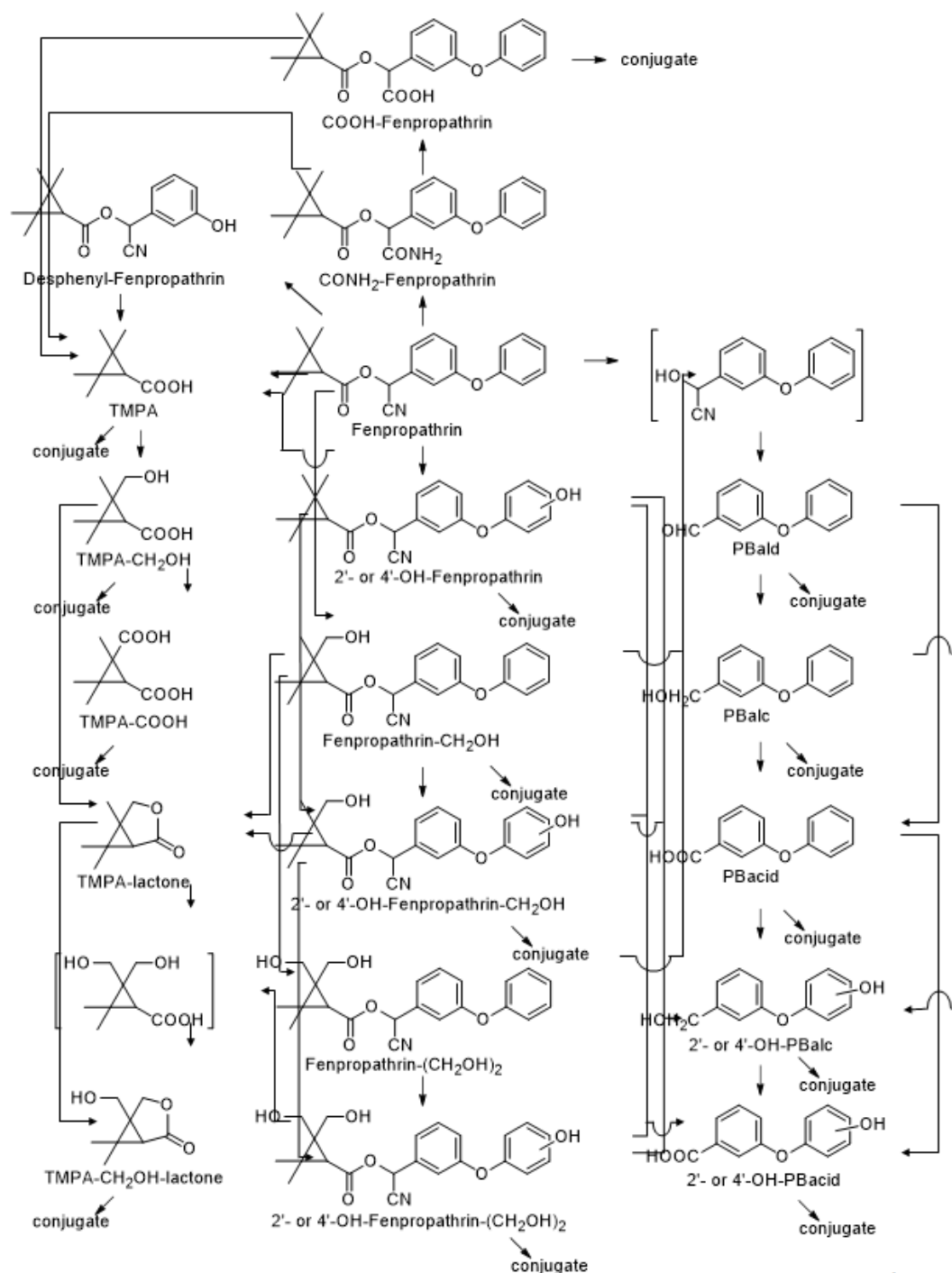


Figure 2 Metabolism and biotransformation pathway for fenpropathrin in plants

Environmental Fate in Soil

The fate and behaviour of fenpropathrin in soils was investigated using fenpropathrin labelled with ^{14}C either in the cyano group, the C-1 position of the cyclopropyl ring, the benzylic carbon or

uniformly in the phenoxyphenyl ring. The reports had been reviewed by the 1993 JMPR, no new study was submitted.

Aerobic soil metabolism

A 365-day aerobic soil metabolism study was conducted in 1989 using [benzyl-¹⁴C]-fenpropathrin (Cranor, 1990, Reference FM-91-0039). The test material was incubated in the dark at 25 °C with silt loam soil confirmed to be biologically active by plating and counting colonies. The nominal concentration of fenpropathrin was 10 µg/g soil; the measured concentration immediately after the addition was 10.2 µg/g. The soils were maintained at 70–75% field capacity throughout the study and sampled at intervals up to the end of the 365-day incubation. The samples were extracted with methanol (3–4 times) and then combusted to determine the un-extracted activity.

After 365 days, 18.4% of the dose remained as parent with accumulated volatiles accounting for 59.9% (99.8% of which was CO₂) and un-extractable residues for 17.8%.

During the whole course of the study the maximum levels of the metabolites were 1.25% PBacid (Day 1), 0.21% CONH₂-fenpropathrin (Day 122), 0.55% desphenyl-fenpropathrin (Day 122), 0.19% 4'-OH-fenpropathrin (Day 122) and 0.39% COOH-fenpropathrin (Day 61), all in terms of per cent of the initial dose. In addition there were maxima of 0.07 (Day 14), 0.28 (Day 365), 0.34 (Day 61) and 0.16% (Day 30) of unidentified products. The ¹⁴C mass balance for the whole period ranged from 98.7% to 107.1% with a mean of 102.3% and a value at the end of the study of 98.7%. The half-life was calculated, using a first order model, to be 152 days. These results were similar to those of previous degradation studies.

The production of labelled carbon dioxide was greatest from the cyano label over 208 days, and least from the phenoxyphenyl label (at day 148), indicating the relative readiness of the different parts of the molecule to mineralize. (Noble, 1976, Reference FR-61-0013).

In the soil extracts from the cyclopropyl label, the parent compound was the main component with small amounts of TMPA and unidentified polar compounds and, in one of the perfusions, the amide of fenpropathrin (CONH₂-fenpropathrin). No amide was found from the cyano label.

In the case of the phenoxyphenyl label, the liberation of carbon dioxide was much slower and the rates were erratic. Neither CONH₂-fenpropathrin nor PBacid was detected in the soils. In the perfusion mixtures, the main components isolated were the parent and unidentified hydrophilic compounds.

In the aerobic soil after 16 weeks, the most important component of the residue was the parent compound. The half-life of the fenpropathrin was about 4 weeks on moist soils (Leiston and moist Brenes) but more than 16 weeks on the drier ones (dry Brenes and Los Palacios). After 16 weeks the most prominent metabolite in drier soil was PBacid in the case of the phenoxyphenyl label and TMPA in the case of the cyclopropyl label (Roberts and Standen, 1976, Reference FR-61-0014). The other

two metabolites found were CONH₂-fenpropathrin and COOH-fenpropathrin, but these were scarcely detectable in the soils with high moisture (Brenes at 16% moisture and Leiston). In the study using Brenes soils, to detect volatile activity, the percentage of the applied radioactivity evolving as CO₂ after 26 weeks was 16.0%, with 71.8% as the intact parent compound and 11.6% as unextracted activity. Other degradation products were only present at negligible levels.

Mikami *et al.*, 1983 (Reference FM-30-0008) investigated two fresh Japanese soils, a sandy clay loam (Azuchi) and a light clay with 15% organic matter (Kodaira), under three different conditions: natural aerobic soil, anaerobic soil, and autoclaved aerobic soil. Under aerobic conditions the half-life of fenpropathrin was 11 and 17 days and after 24 weeks the level of fenpropathrin had declined from 1 mg/kg to 0.025 and 0.040 mg/kg respectively. After 8 weeks approximately 0.93 mg/kg remained under sterilized conditions. Sterilization retarded degradation, demonstrating the importance of biological processes. While there were only minor indications of degradation products in the sterile soils, at least 7 were detected in the methanol extracts of the non-sterile soil. With the cyclopropyl-labelled compound the main components of radioactivity were the parent fenpropathrin, desphenyl-fenpropathrin, 4'-OH-fenpropathrin, and small amounts of CONH₂-fenpropathrin and COOH-fenpropathrin. There were also indications of very small amounts of unidentified products. In the case of the phenoxyphenyl label, very low levels of PBacid were observed during the first 4 weeks. Un-extractable residues at the end of the study reached 44–45% of the added radioactivity in the Kodaira soil (the one containing a high proportion of organic matter), but only 24–32% in the lower-organic Azuchi soil. No TMPA was reported. A major part of the lost radioactivity was recovered as labelled CO₂ in fairly similar amounts from both labels.

Photodegradation in soil

Photodegradation studies carried out in Japan on light clay, sandy loam and sandy clay loam with fenpropathrin labelled with ¹⁴C in the cyano group, the phenoxyphenyl ring or the C-1 position of the cyclopropyl ring and applied to thin-layer soil plates (Takahashi *et al.*, 1983, 1985, Reference FM-30-0011). The fenpropathrin was applied at a rate of 1.1 µg/cm² and the plates exposed to natural sunlight for 14 days. Dark controls were run at the same time. Under irradiation, the half-lives of the CN-labelled fenpropathrin were between 1–5 days on the three soils. The mean fenpropathrin residues left in the soils at the end of the 14-day period amounted to 5.1, 29.4 and 32.9% of the amounts applied. The corresponding figures in the dark controls were 74, 84 and 96%; there was insufficient degradation for half-lives to be estimated. The main degradation product under irradiation with all three labels was CONH₂-fenpropathrin which reached a maximum in the three soils after 5, 7 and 7 days. Substantial amounts also occurred in the dark controls. For the most part, other metabolites were present in the irradiated soils in only very small amounts, except PBacid which reached a maximum of 11.4% of the total applied radioactivity after 7 days. Minor metabolites found were COOH-fenpropathrin, desphenyl-fenpropathrin, and 3-OH-Bacid. Loss of radioactivity from soils was greatly

enhanced by irradiation. The nitrile carbon was the most susceptible followed closely by the C-1 of the cyclopropyl group. The phenoxyphenyl group was a more stable part of the molecule.

Summary: Aerobic soil metabolism

Studies on the metabolism of fenpropathrin in soil demonstrated that fenpropathrin is degraded in the soil by a combination of photochemical and microbiological processes. Metabolism proceeds via cleavage of the ester bonds, hydroxylation, and hydrolysis of the cyano group to CONH_2 and COOH groups. The major products detected after aerobic metabolism were the parent fenpropathrin, CO_2 , and non-extractable residues. Metabolites included desphenyl-fenpropathrin, 4'-OH-fenpropathrin, phenoxybenzoic acid, and CONH_2 -fenpropathrin, which was further degraded to COOH -fenpropathrin. The proposed metabolic pathway of fenpropathrin in soil is shown in Figure 3.

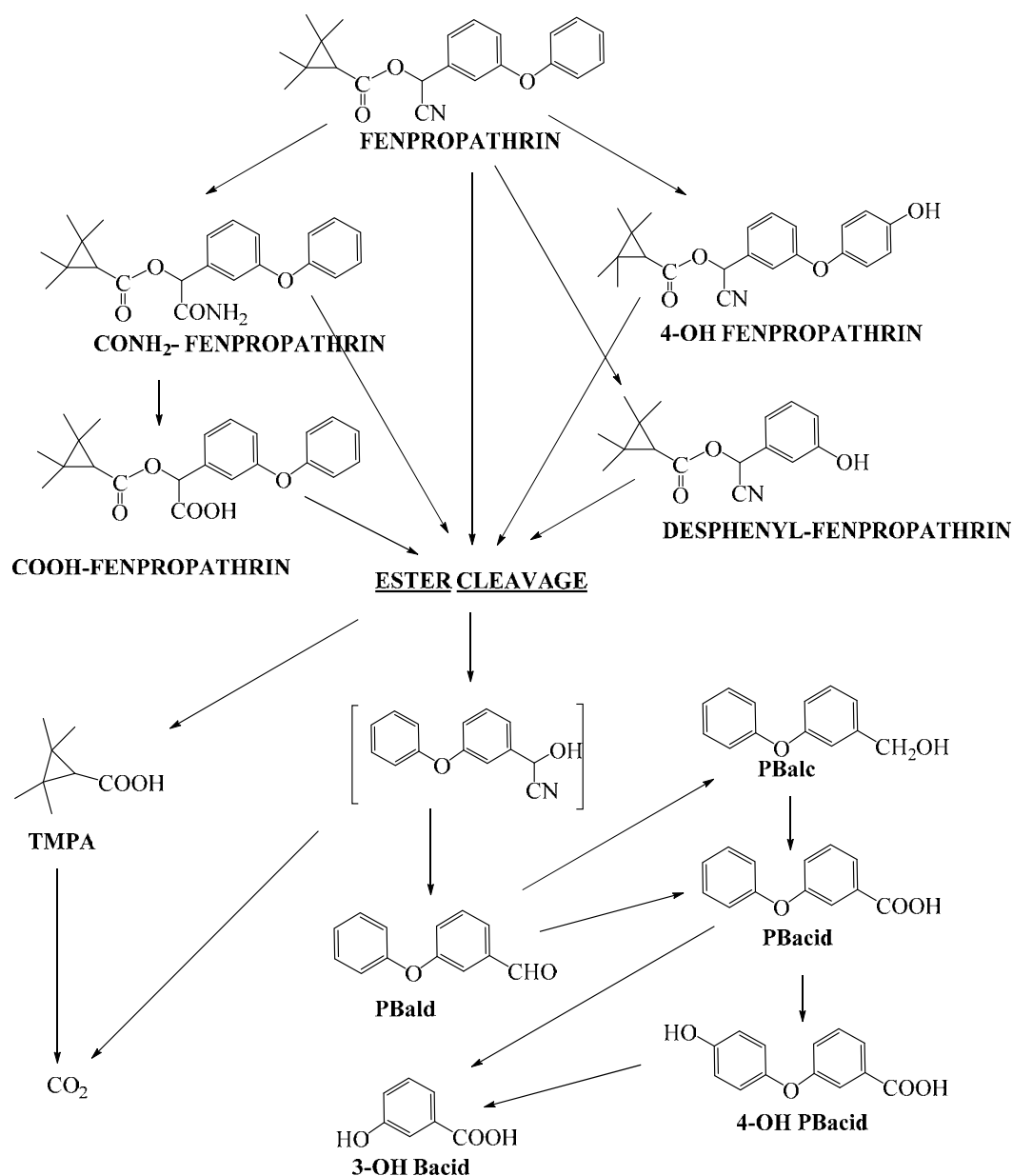


Figure 3 Metabolism and biotransformation pathway for fenpropathrin in soil

Residues in Succeeding or Rotational Crops

The crops included in this evaluation are permanent or semi-permanent crops, for which rotational crop studies are not required. No study was submitted for evaluation.

Residue analytical methods

Analytical methods have been developed for determination of residues of fenpropathrin in plant and animal. In general, the methods involve solvent extraction, clean-up by either silica gel or Florisil column, GLC using electron capture detection. The main variations depending on the substrates are in the extraction and clean-up procedures. Fruits and vegetables may be homogenized with water, shaken with acetone, and extracted with dichloromethane, using NaCl to minimize emulsification. After drying with anhydrous sodium sulfate and clean-up by silica gel column chromatography, the solvent is evaporated at <40 °C and the residue dissolved in acetone before estimation by gas chromatography with electron capture detection. Other extraction procedures involve direct extraction of the homogenized material suspension in water or homogenization with methanol instead of water. The results of recovery studies carried out as part of method validation or concurrent recoveries obtained from supervised trial samples are summarised in Table 33.

Analytical methods for plant matrices

The base method, RM-22-4 consolidates RM-22-2 (for high moisture crops), RM-22-3 (for oily crops) and includes a procedure for processed oil and soapstock (Lai and Fujie, 1983; Reference FA-41-0040). Homogenized samples are extracted with hexane/acetone and cleaned up through silica gel and C18 solid phase extraction (SPE). Additional purification using gel permeation chromatography (GPC) are performed for oily matrices. Soapstock samples are dissolved in water, extracted with dichloromethane, before proceeding with the processed oil method which involves partition between hexane and acetonitrile and clean-up using alumina oxide. Analysis is by GC with electron capture detection (ECD) (column 3% OV 101 - high moisture and oily samples; DB-1 – processed oil and soapstock samples).

Method used for citrus fruit, pome fruit, grapes and their processed fractions

Citrus fruit samples were analysed using method RM-22-4, with modifications. Homogenized samples were extracted with hexane/acetone, partitioned into hexane and cleaned up with silica gel and C18 solid phase extraction (SPE). Residues of fenpropathrin were determined by gas chromatography with electron capture detection. The LOQ for the method was 0.01 mg/kg.

Method used for raspberry

Raspberry samples were analysed using the revised method RM-22-4 (revision May 1993), with modifications. Homogenized samples were extracted with hexane/acetone, partitioned into hexane and cleaned up with silica gel and C18 solid phase extraction (SPE). Residues of fenpropathrin were

determined by gas chromatography with mass spectrometry (MS)-SIM. The LOQ for the method was 0.02 mg/kg.

Method used for tomato and processed fractions, cucumber, melon and squash

Method RM-22-4 was used for the analysis of fenpropathrin in fruiting vegetables, cucurbits and fruiting vegetables other than cucurbits. The LOQ was 0.01 mg/kg. A revised RM-22-4 method (revision May 1993) using a DB-17 column and nitrogen phosphorus detection (NPD) was used for certain raw and processed tomato samples.

Method used for tea

Method RM-22-2 was revised for analysis of fenpropathrin in tea. Tea samples were extracted with an acetonitrile-water mixture and the extracts filtered under suction (Lavakumar, S., *et al.*, 2003; Report No. 11861 and Lavakumar, S., *et al.*, 2004; Report No.14246). The filtrate was concentrated then diluted with 5 percent aqueous sodium chloride solution and subjected to extraction with hexane-ether mixture. The combined extracts were filtered through sodium sulfate and evaporated to near dryness before being dissolved in 5 mL hexane. Clean-up was performed and the eluate evaporated and dissolved in acetone. Fenpropathrin was quantified by gas chromatography using an electron capture detector. The LOQ is 0.05 mg/kg.

Methods used for soya beans

Method RM 22-4 was modified for soya bean determination. Soya bean seeds were extracted with acetonitrile-ethyl acetate mixture, centrifuged and cleaned up through silica gel column with hexane. The extract was eluted with ethyl acetate-hexane mixture, evaporated to dryness and re-suspended in hexane. Fenpropathrin residues were determined by gas chromatography using a micro electron capture detector (Tomaz, 2013; Reference FR-0570). The LOQ for the method is 0.01 mg/kg.

In the other soya bean study, residues were extracted with acetonitrile and salt mixture and fenpropathrin analysed by liquid chromatography with mass spectrophotometric detection (LC/MS/MS) Lopez, 2012; Reference FR-0571). The LOQ for the method was 0.01 mg/kg.

Methods used for cotton matrices

Method RM 22-4 was modified for cotton matrix determination. Fuzzy seed and processing fractions were extracted with hexane-acetone and cleaned up using gel permeation chromatography or partitioned into acetonitrile for oil removal with clean up through silica gel and C-18 Sep Pak columns. For cotton seed oil, samples were dissolved in hexane, portioned into acetonitrile and cleaned up by alumina oxide column chromatography. For soapstock, samples were dissolved in water, partitioned into dichloromethane, partitioned between hexane and acetonitrile and cleaned up by alumina oxide column chromatography. Fenpropathrin residues were determined by gas

chromatography using an electron capture detector (Fujie, 1990; Reference FR-01-0306). The LOQ for the method is 0.01 mg/kg.

In the other soya bean study, residues were extracted with acetonitrile and salts mixture and fenpropathrin analysed by liquid chromatography with mass LC-MS/MS detection *utilising the transition of m/z 350→125*. The LOQ for the method is 0.01 mg/kg (Lopez, 2012; Reference FR-0571).

Method used for coffee beans

Coffee samples were extracted with water and 0.1% acetic acid in acetonitrile and a mixture of salts (anhydrous magnesium sulfate, sodium acetate and sodium chloride are added and the mixture centrifuged. An aliquot of the supernatant was transferred to a tube containing anhydrous magnesium sulfate and centrifuged. The extract was diluted in methanol:water (20:80) and residues of fenpropathrin were analysed by LC-MS/MS. The LOQ was 0.01 mg/kg (Gravena, 2013; Reference FR-0572)

Analytical methods for animal matrices

Method RM-22A-1 was used to analyse for fenpropathrin residues in milk and animal tissue samples in the feeding study (Fujie, 1986; FR-61-0174 and FR-61-0175). In summary, the method involves extraction with hexane/acetone, acetonitrile/hexane partitioning, silica gel column chromatography and measurement by gas chromatography using the electron capture detector. The method was validated with an LOQ of 0.01 mg/kg fenpropathrin. Control samples fortified with fenpropathrin were analysed together with treated samples from the feeding studies and the concurrent recoveries were within the acceptable range of 70–120%.

Multi-residue Methods

The multi-residue method DFG S19 was investigated and validated for the determination of fenpropathrin in acidic plant matrices high water content (tomatoes and oranges) (Class, 2003; Reference FA-0093). Samples were extracted with acetone/water, following neutralization of the pH in the case of oranges. Extracts were partitioned with ethyl acetate/cyclohexane in the presence of sodium chloride and cleaned up using gel permeation chromatography (GPC) and silica gel. Analysis was by GC-MS (ions m/z 181 and 265 for quantitation and 209 and 349 for confirmation). Recoveries (range and mean) and precision were within the acceptable limits. Linearity was demonstrated. There were no interfering peaks at the retention time for fenpropathrin in any of the crop matrices. The method is suitable for monitoring pesticide residues.

The multi-residue method DFG S19 was also validated for the determination of fenpropathrin in foodstuffs of animal origin (milk, bovine meat and fat, eggs) (Class, 2003; Reference FA-0094). Whole milk, egg and bovine meat samples were extracted with acetone/water and the extracts were partitioned with ethyl acetate/cyclohexane in the presence of sodium chloride. Fat samples were

extracted with ethyl acetate/cyclohexane. Extracts were cleaned up using gel permeation chromatography (GPC) and silica gel. Analysis was by GC-MS (ions m/z 181 and 265 for quantitation and 125, 152, 209 and 349 for confirmation). Recoveries (range and mean) and precision were within the acceptable limits. Linearity was demonstrated. There were no interfering peaks at the retention time for fenpropathrin in any of the animal matrices. The validated LOQ is 0.01 mg/kg for animal tissues and eggs. The method can be used for enforcement of MRLs and monitoring of residues.

A summary of recovery data from the methods used for plant and animal commodities supported in this submission are presented in the table 33.

Table 33 Summary of method validation and concurrent recovery data ^a

Commodity	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
Plant commodities							
Almond hulls	0.02-7.5	6	90-115	103	8.9	RM-22-4	FR-0382
Almond nutmeat	0.02-0.1	6	84-119	96	14	RM-22-4	FR-0382
Apples	0.1	9	89-111	98	8	RM-22-4	FR-81-0305
	0.1	6	87-103	97	6		FR-61-0190
	0.1	12	73-119	98	12		FR-0370
	1.0	1	106	-	-		FR-81-0305
Apple juice	0.1	1	106	-	-	RM-22-4	FR-81-0305
	0.1	12	73-96	87	12		FR-0370
Apple wet pomace	0.2	1	109	-	-	RM-22-4	FR-81-0305
	1.0-4.0	4	75-98	89	-		FR-0370
Apple dry pomace	0.2	1	96	-	-	RM-22-4	FR-81-0305
	0.4-8.0	4	71-126	92	26		FR-0370
Bell pepper	0.02-1.0	10	94-107	101	4	RM-22-4	FR-0558
Canned tomatoes	0.01-0.2	3	94-100	97	3	RM-22-4	FR-21-0344
Cherry	0.02-4.0	8	82-110	99	9.6	RM-22-4	FR-0381
Coffee	0.01-0.5	13	79-116	98	15.3	LC-MS/MS	FR-0572
Cotton, fuzzy seed	0.1-0.2	38	70-101	83	-	RM-22-4	Fr-01-0306
Cucumber	0.01-11.4	15	66-115	89	13.8	RM-22-4	FR-0556
Grape	0.1	4	93-102	99	4	RM-22-4	FR-01-0308
	0.1	22	77-123	99	12		FR-010335
	1.0	1	110	-	-		FR-0374
	0.02	1	98	-	-		
	0.4	1	97	-	-		
Grape wet pomace	0.1	3	100-101	100	0.7	RM-22-4	FR-01-0308
	0.2	1	95	-	-		
	0.5	4	97-106	101	4		
	0.5	8	100	-	4		
Grape dry pomace	0.1	3	86-121	101	18	RM-22-4	FR-01-0308
	0.2	2	84-96	90	-		
	0.5	2	100-119	109	-		
	1.0	4	95-108	100	6		
	1.0	11	100	-	12		
Grape juice	0.1	9	87-101	96	5	RM-22-4	FR-01-0308
	0.005	1	98	-	-		FR-01-0335
	0.1	1	98	-	-		
Grapefruit	0.02	2	106-114	110	-	RM-22-4	FR-21-0343
	0.1	9	85-108	95	7		
	0.5	7	90-99	94	3		
		18		96	7		
Lemon	0.02	3	98-120	109	10	RM-22-4	FR-21-0342
	0.1	8	92-117	101	9		
	0.5	5	90-108	99	7		
		16		102	9		
Melon	0.02-1.0	16	82-98	90	4.8	RM-22-4	FR-0375
Non-bell pepper	0.02-10	12	88-115	97	8	RM-22-4	FR-0558

Commodity	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
Olive fruit, no pit	0.02-15	15	70- 121 ^{1/}	91	9.9	RM-22-4	FR-0561
Olive fruit, no pit	0.02-5.0	7	69-103 ^{1/}	88	13.9	RM-22-4	FR-0561
Olive oil	0.02-20	12	76-115 ^{1/}	96	13.0	RM-22-4	FR-0561
Olive oil	0.02-0.10	4	64-81 ^{1/}	73	9.9	RM-22-4	FR-0561
Orange	0.01 – 0.1	10	79-109	98	9	DFG S19	(FA-0093)
Orange	0.01- 0.1	10	79-104	95	7	DFG S19	(FA-0093)
Orange	0.1	34	77-115	96	7	RM-22-4	FR-01-0307
Orange dried peel/fines	0.1	2	90-92	91	-	RM-22-4	FR-01-0307
	1.0	4	77-100	90	11		
Orange juice	0.1	3	97-98	97	0.6	RM-22-4	FR-01-0307
Orange molasses	0.1	3	90-94	92	2	RM-22-4	FR-01-0307
Orange oil	0.4	1	81	81	-	RM-22-4	FR-01-0307
	1.0	2	86-100	93	-		
	2.0	1	86	86	-		
		4		88	9		
Orange peel (wet)	0.1	1	91	91	-	RM-22-4	FR-01-0307
	1.0	4	96-102	100	3		
Orange pulp	0.1	2	82-98	90	-	RM-22-4	FR-01-0307
Peach	0.02-1.0	10	91-113	101	6.4	RM-22-4	FR-0384
Pear	0.1	5	91-102	97	4	RM-22-4	FR-0369
	0.1	7	74-109	95	12		
	0.1	14	94-117	99	6		
Pear pulp	0.1	2	98-101	100	-	RM-22-4	FR-0369
Pear skin	0.5	2	105-108	106	-	RM-22-4	FR-0369
Pear puree	0.1	1	99	-	-	RM-22-4	FR-0369
Pecans	0.02-0.1	6	71-90	82	9.3	RM-22-4	FR-0383
Plum	0.02-0.6	7	97-109	101	4.6	RM-22-4	FR-0385
Plum	0.05-2.0	4	96- 101	99	2.5	RM-22-4	FR-0559
Plum, dried	1.0, 10	2	101, 104	103	-	RM-22-4	FR-0385
Plum, dried	0.1, 2.0	2	94, 99	97	-	RM-22-4	FR-0559
Raisin/raisin waste	0.1	2	82-107	94	-	RM-22-4	FR-01-0308
	0.2	3	81-103	94	12		
	0.5	2	103-104	103	-		
	0.5	7	97	-	11		
Raspberry	0.02-10	10	74-110	88	-	RM-22-4	FR-0560
Soya beans	0.025, 0.25	2	74, 74	74	-	GC-ECD	FR-0570
Soya beans	0.01-0.1	10	77-100	90	8.7	LC-MS/MS	FR-0571
Squash	0.01-11.4	9	85-116	97	11.5	RM-22-4	FR-0557
Strawberry	0.1-0.5	27	81-115	98	8.2	RM-22-4	FR-11-0345
Tea	0.05- 2.0	16	88-93	90	2	RM-22-4	04-2974-2002; 04-4190-2004
Tomato	0.01-0.1	10	67-112	100	13	DFG S19	FA-0093
Tomato	0.01– 0.1	9	96-111	103	5	DFG S19	FA-0093
Tomato dry pomace	0.1	3	75-118)	96	23	RM-22-4	FR-41-0360
Tomato juice	0.1	3	100-101	101	0.7	RM-22-4	FR-41-0360
Tomato paste	0.1	3	100-105	102	3	RM-22-4	FR-41-0360
Tomato wet pomace	0.1	3	100-102	101	1	RM-22-4	FR-41-0360
Tomato, green	0.01-0.2	16	86-130	99	11	RM-22-4	FR-21-0344
Tomato, ripe	0.02-0.2	18	94-102	98	2	RM-22-4	FR-41-0360
Tomato, ripe	0.02- 0.2	10	85-113	95	10	RM-22-4	FR-21-0344
Foodstuffs of animal origin							
Bovine Fat	0.1	1	100	-	-	RM-22A-1	FR—61-0174
Bovine fat	0.01-0.1	10	72-117	94	14	DFG S19	FA-0094
Bovine fat	0.01-0.1	10	80-133	103	14	DFG S19	FA-0094
Bovine Kidney	0.1	3	81-93	88	6.8	RM-22A-1	FR—61-0174
Bovine Liver	0.1	1	114	-	-	RM-22A-1	FR—61-0174
Bovine muscle	0.01- 0.1	10	82-99	89	7	DFG S19	FA-0094
Bovine muscle	0.01-0.1	10	65-106	86	18	DFG S19	FA-0094
Bovine Muscle	0.1	1	-	94	-	RM-22A-1	FR—61-0174
Milk fat	0.4	1	104	-	-	RM-22A-1	FR—61-0174
Poultry fat	0.1	1	94	-	-	RM-22A-1	FR-61-0175
Poultry gizzard	0.1	1	94	-	-	RM-22A-1	FR-61-0175

Commodity	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
Poultry liver	0.1	1	94	-	-	RM-22A-1	FR-61-0175
Poultry muscle	0.1	1	93.4	-	-	RM-22A-1	FR-61-0175
Whole Egg	0.1	6	94-102	96	3.6	RM-22A-1	FR-61-0175
Whole egg	0.01-0.1	10	93-102	99	3	DFG S19	FA-0094
Whole egg	0.01-0.1	10	71-105	99	10	DFG S19	FA-0094
Whole milk	0.1	12	83-119	96	9.4	RM-22A-1	FR—61-0174
Whole milk	0.01-0.1	10	85-93	89	3	DFG S19	FA-0094
Whole milk	0.01-0.1	10	77-105	96	8	DFG S19	FA-0094

^a Data presented are only for registered uses and from studies submitted in this document.

Storage Stability under Frozen Conditions

The stability of fenpropathrin residues in commodities under frozen conditions has been investigated in apples, orange, cotton, pears, grapes, tomato and its processed products as well as in products of animal origin.

In addition to these individual studies, storage stability determinations, together with procedural recovery analyses were also carried out concurrently with the field trial samples for strawberry, olives (fruit and oil), cucumber, squash, melon, peppers, milk, eggs and kidney. All these storage stability studies are summarized below. For samples analysed within 30 days of harvest (soya beans, coffee beans, eggs and poultry tissues), storage stability requirements have been waived. The results are summarised in Table 34.

In general, results of the storage stability tests confirmed that residues of fenpropathrin in stored frozen samples were stable within the period of storage between sampling and analyses.

Apple, orange, cotton, pears and grapes

The stability of field incurred fenpropathrin was investigated in five matrices (Fujie, 1986; Reference FR-61-0190). Samples taken from residues trials conducted with fenpropathrin 2.4 EC were stored at -20 °C and four replicate samples taken at 3, 6, 9 and 12 months during storage. Fortified control samples (0.1 mg/kg) were analysed concurrently with each set of samples to validate analytical method recovery. Homogenized samples were extracted with hexane/acetone and cleaned up through silica gel and C18 solid phase extraction (SPE) (method RM-22-4). Additional purification using gel permeation chromatography (GPC) was performed for oily matrices (method RM-22-4). Analysis was by GC with electron capture detection (ECD).

Orange oil and dried peel

The stability of field incurred fenpropathrin was investigated in orange oil and dried peel samples from a processing study (Fujie, 1990; Reference FR-01-0307). Duplicate samples taken from processing study were stored at -20 °C and samples taken after 11 months storage. Analysis was by methods RM-22-4 and RM-22-2.

Strawberry

The storage stability data for strawberries were obtained by reanalysis of retained samples from a previous field trial (Mitten, 1989; Reference FR-11-0345). Samples harvested on 07.04.89 were received frozen in the laboratory on 18.04.89, macerated on 25.04.89 and were extracted for the initial analysis on 28.04.89. After sub-sampling, the retained samples were stored at -20 °C with periodic subsampling for reanalysis using method RM-22-4. The results demonstrate that fenpropathrin is stable in strawberries up to six months in frozen storage.

Raspberry

The storage stability data for strawberries were obtained by reanalysis of retained samples from a previous field trial (Samoil, 2007; Reference FR-0560). Samples were fortified with 2 mg/kg fenpropathrin then stored frozen for 216 days. Analysis using modified method RM-22-4.

Grape products

The storage stability of fenpropathrin in samples of grape juice, dry and wet pomace, hydrated raisins and raisin waste was determined from incurred residues from the field trial sites (Fujie, 1990; Reference FR-01-0335). Samples were held in frozen storage under similar conditions to the field generated samples.

Olive fruit and olive oil

The storage stability of fenpropathrin in samples of olive fruit without pit and in olive oil was determined by fortifying samples with 2 mg/kg fenpropathrin after receipt from the field trial sites (Samoil, 2007; Reference FR-0561). Samples were held in frozen storage under similar conditions to the field generated samples. After 208 days (olive fruit without pits) and 202 days (olive oil), the storage stability samples were analysed by method RM-22-4.

Cucumber

The stability of fenpropathrin residues in cucumber samples stored frozen under the same conditions as the field trial samples, was determined as part of the field phase of the study (Samoil, 1999; Reference FR-0556). Untreated samples from two field sites were fortified at 11.4 mg/kg fenpropathrin and returned to storage at -20 °C, together with the field trials samples. After 245 and 286 days in storage, the storage stability and field samples were analysed.

Melons

Storage stability of fenpropathrin on melons was determined as part of the field trial study (Green, 1995; Reference FR-0375). Untreated samples fortified at 0.1 mg/kg fenpropathrin were stored at -20°C under the same conditions as the field trial samples. Duplicate analyses were conducted on the day of fortification and again after 187 days of storage.

Squash

Storage stability of fenpropathrin in squash samples was verified by taking untreated control samples, fortifying at 1 and 11 mg/kg with fenpropathrin, and storing in freezers under the same conditions as the field samples. Storage stability samples were analysed after 230 days in storage (Samoil, 1999; Reference FR-0557).

Tomatoes and tomato canning waste

The stability of fenpropathrin was investigated in tomatoes (fortified samples) and tomato canning waste samples (field incurred residues) from a processing study (Lai, 1990; Reference FR-21-0344). Duplicate samples taken from a processing study were stored at -20 °C and samples taken after various time periods up to 6 months storage. Analysis was by method RM-22-4.

Tomato paste, tomato juice, wet and dry tomato pomace

The stability of fenpropathrin was investigated in tomato paste, tomato juice and wet and dry tomato pomace from a processing study (Green, 1994; Reference FR-41-0360). Duplicate control samples taken from a processing study were fortified at 0.1 mg/kg, stored at -20 °C and samples taken after 5 months storage. Analysis was by methods RM-22-4.

Non-bell peppers

Samples from field trials were fortified with fenpropathrin at 10 mg/kg and stored frozen at -25 °C for 323 days (Samoil, 2001; Reference FR-0558). 323

Milk and bovine kidney

The stability of fenpropathrin in milk when stored at -20 °C was determined by fortifying control milk samples bought from a store, with 0.1 mg/kg fenpropathrin and stored at -20 °C along with the milk samples from the residue feeding study (Fujie, 1986; Reference FR-61-0174). The fortified milk samples were analysed at a time interval greater than the storage interval for milk from the feeding study.

To determine the stability of fenpropathrin in tissues, kidney samples from the feeding study were reanalysed following an appropriate interval which reflected the time interval between sacrifice and the initial analyses. The analytical method RM-22A-1 was used to analyse all samples. Reanalysis of kidney samples following an additional 71 days in storage at -20 °C resulted in a mean recovery of 104% of initial analyses results.

Eggs

The stability of fenpropathrin in eggs following storage at -20 °C was determined by fortifying control egg samples bought from the store, with 0.1 mg/kg fenpropathrin and stored at -20 °C along with the egg samples from the residue feeding study (Fujie, 1986; Reference FR-61-0175). The fortified egg

samples were analysed at a time interval greater than the storage interval for eggs from the feeding study.

Table 34 Summary of results of storage stability studies

Commodity	Spike level (mg/kg)	Storage interval (months)	Procedural recoveries (%)	Residues remaining (%)
Apple	2.53 ^a	3	96±8.2	82
		6		78
		9		91
		12		82
Cotton	0.03 ^a	3	96±8.2	133
		6		116
		9		n/a
		12		100
Cucumber	11.4	~9	87±12.6	76-96
Grapes	0.37 ^a	3	96±8.2	n/a
		6		89
		9		87
		12		87
Grape juice		14	94-100	100
Dry pomace		14	98-99	93-98
Hydrated raisins		1-11	92-113	77-106
Raisin waste		1-11	80-112	77-94
Wet pomace		5-12	95-104	89-90
Pear	1.2 ^a	3	96±8.2	99
		6		77
		9		78
		12		89
Melons	0.1	6	89-96	74
Non-bell peppers	10	0	88-104	88.4-104
		11		91-95
Olive fruit	2	7	78-89	78, 88, 89
Olive oil	2	7	83-90	85, 90, 93
Orange	0.25 ^a	3	96±8.2	93
		6		120
		9		n/a
		12		88
Orange oil	0.965	11	81-100	130
Orange dry peel	0.12	11	90-100	91.5
Raspberry	2	7	74-110	86-88
Strawberry ¹	0.83-0.84 ^a	0	98.3±8.2	
		1		91-101
		3		93-98
		6		87-96
Squash	1	7	92.9-94.6	74.2
	11	7	82.4-92.7	91.8
Tomato		6	83-130	100
Tomato paste	0.1	5	100-102	92
Tomato juice	0.1	5	75-118	92
Tomato wet pomace	0.1	5	100-105	102
Tomato dry pomace	0.1	5	100-105	97
Milk	0.1	2.5	96±9.4	100
Kidney	0.155 ^a	2.2	88±6.8	104
Eggs	0.1	5	96±3.6	92

^a: Level of initial residue in retained test portion; n/a = not analysed

USE PATTERN

Fenpropathrin is registered for use on a large number of crops in many countries, but only the registered use patterns for the crops for which supervised trials were provided are summarised in Table 35.

Table 35 Registered uses of fenpropathrin

Crop	Country	Formulation		Application				PHI (days)
		g ai/L or g ai/kg	type	Method	Rate kg ai/ha	Water L/ha	No or/ Season max kg ai/ ha	
Cane fruit	USA	300 g/L	EC	Foliar (by ground or aerial application)	0.22- 0.34	28-94 (aerial); 187 minimum (ground)	0.67 kg ai/ha/season 14-day intervals	3
Citrus	USA	300 g/L	EC	Foliar (by ground or aerial application)	0.22- 0.45	187 (aerial); 468- 4700 (ground)	0.9 kg ai/ha/season; 10-day intervals	1
Coffee	Brazil	300 g/L	EC	Foliar (by ground equipment)	0.06-0.12		2	14
Cotton*	USA	300 g/L	EC	Foliar (by ground or aerial application)	0.22- 0.45	28-94 (aerial); 94- 468 (ground)	0.9 kg ai/ha/season	21
Grapes	USA	300 g/L	EC	Foliar (by ground or aerial application)	0.22- 0.45	230-1870 (air or ground)	0.9 kg ai/ha/season; 7-day intervals	21
Fruiting vegetables, Cucurbits, including cucumber, muskmelon, honeydew melon, pumpkin, squash, watermelon	USA	300 g/L	EC	Foliar (by ground or aerial application)	0.22- 0.34	28-94 (aerial); 187- 935 (ground)	0.9 kg ai/ha/season; 7-day intervals	7
Fruiting vegetables other than cucurbits, including tomato, eggplant, peppers	USA	300 g/L	EC	Foliar (by ground or aerial application)	0.22	47- 94 (aerial); 234- 1122 (ground)	0.9 kg ai/ha/season; 7-days interval	3
Olive	USA	300 g/L	EC	Foliar (by ground equipment)	0.22- 0.34	935	0.9 kg ai/ha/season; 14-day intervals	7
Pome fruit	USA	300 g/L	EC	Foliar	0.22- 0.45	700- 3740	0.9 kg ai/ha/season; 10-days interval	14

Crop	Country	Formulation		Application				PHI (days)
		g ai/L or g ai/kg	type	Method	Rate kg ai/ha	Water L/ha	No or/ Season max kg ai/ ha	
Soya beans	Brazil	300 g/L	EC	Foliar (by ground equipment)	0.045		1	30
Stone fruit	USA	300 g/L	EC	Foliar (by airblast equipment)	0.22- 0.45	935- 3740	0.9 kg ai/ha/ season; 10-days interval	3
Strawberry	USA	300 g/L	EC	Foliar (by ground equipment)	0.22- 0.45	935- 2800	0.9 kg ai/ha/ season ≥30-day intervals	2
Tea	India	300 g/L	EC	Foliar (by knapsack sprayer)	0.05- 0.06 kg ai/ha (0.01- 0.015 kg ai/hL)	400- 500		7
Tree nuts	USA	300 g/L	EC	Foliar (by airblast equipment)	0.22- 0.45	468- 3740	0.9 kg ai/ha/ season; 10-days interval	3

Note: GAPs in the USA specify total seasonal rates in addition to individual application rates, therefore the seasonal maximum dose rate is considered primarily in evaluation of supervised trials.

* Do not feed gin trash or treated forage to livestock or allow animals to graze treated fields

RESIDUES RESULTING FROM SUPERVISED TRIALS

Supervised trials have been conducted to support MRLs for the following crops citrus fruits, peach, plum, strawberries, olives, cucumber, melon, squash, tomato, peppers, soya beans, tree nuts, coffee beans and tea. The results of these supervised trials are summarized in the following tables:

Crop Group	Commodity	Table No.
Citrus fruit	Orange, lemon, grapefruit	36
Pome fruit	Apple, pear	37
Stone fruit	Peach, plum, cherry	38
Berries and other small fruits	Strawberry	39
	Raspberry	40
	Grape	41
Tropical/Subtropical fruits – edible peel	Olives	42
Fruiting vegetables, cucurbits	Cucumber	43
	Melon	44
	Squash	45
Fruiting vegetables other than cucurbits	Tomato	46
	Pepper	47
Pulses	Soya beans	48
Oilseeds	Cottonseed	49

	Cottonseed hulls	53
Tree nuts	Almonds, pecans	50
	Almond hulls	54
Seeds for beverages and sweets	Coffee beans	51
Teas	Tea, green, black	52

In addition to the description and details of the field trials and analytical methods, each report includes a summary of the procedural recoveries, and in many cases, concurrent recoveries in stored frozen samples.

In the trials, where multiple samples were taken from a single plot, the average value is reported. Where results from separate plots with distinguishing characteristics such as different formulations, different crop varieties or different treatment schedules were reported, results are listed for each plot.

Results have not been corrected for concurrent method recoveries. Residues and application rates have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Residue values from the trials conducted according to the maximum GAP have been used for the estimation of maximum residue levels. Those results included in the tables are underlined.

Citrus fruits

A total of 31 supervised trials were conducted in the United States on citrus fruits (18 on oranges, 6 on lemons and 7 on grapefruit). The trials on oranges were carried out from 1984 to 1987 and consisted of one to four applications of an EC formulation containing 300 g/L fenpropathrin, applied at rates ranging from 0.11 to 0.45 kg ai/ha for a total seasonal rate ranging from 0.11 to 1.8 kg ai/ha (Fujie, 1990; Reference FR-01-0307). Duplicate samples of mature oranges, consisting of a minimum of 10 fruits, were collected at various PHIs (0 day to 35 days). In some trials, fenpropathrin was applied pre-planting and sampled at normal harvest, 180 days after application.

The seven supervised trials on grapefruit in 1991 were all according to the US GAP for citrus fruits. The composite samples consisted of 16 fruits.

Upon harvest, all samples were frozen and maintained frozen at -20 °C until analysis. Samples from the field trials were analysed within this demonstrated period of stability (9 months for oranges, one month for lemons, and two months for grapefruit). All samples were analysed using analytical method RM-22-4 with a limit of detection of 0.01 mg/kg.

Table 36 Residues of fenpropathrin in citrus fruits resulting from supervised trials in the USA

CITRUS	Application				DAT	Commodity	Residue	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total kg ai/ha	days		mg/kg	
GAP, USA for citrus fruit	300 EC	0.22-0.45	468-4700	2/0.9	1			
ORANGE								
M335 T-6083 Fresno, CA, 1984 (Navel)	300 g/L	0.113	935	1	3	Whole fruit	0.04, 0.04 (0.04)	Fujie, 1990 (FR-01-0307)
		0.22	935	1	3	Whole fruit	0.07, 0.13 (0.10)	
		0.45	935	1	0	Whole fruit	0.24, 0.40 (0.32)	
					1		0.25, 0.17 (0.21)	
					3		0.23, 0.17 (0.20)	
					7		0.10, 0.20 (0.15)	
					14		0.04, 0.09 (0.07)	
					21		0.06, 0.09 (0.08)	
					28		0.05, 0.12 (0.09)	
					35		0.21, 0.05 (0.13)	
		0.67	935	1	3		0.20, 0.32 (0.26)	
		0.11	935	2/ 0.22	3		0.13, 0.08 (0.11)	
		0.22	935	2/ 0.44	3		0.02, 0.13 (0.08)	
		0.45	935	2/ 0.90	0		0.37, 0.33 (0.35)	
					1		0.28, 0.22 (0.25)	
					3		0.16, 0.27 (0.22)	
					7		0.11, 0.42 (0.27)	
					14		0.27, 0.15 (0.21)	
					21		0.15, 0.19 (0.17)	
					28		0.22, 0.24 (0.23)	
					35		0.08, 0.25 (0.17)	
		0.67	935	2/ 1.34	0		0.51, 0.21 (0.36)	
					1		0.41, 0.32 (0.37)	
					3		0.57, 0.62 (0.60)	
					7		0.40, 0.34	

CITRUS	Application				DAT	Commodity	Residue	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total kg ai/ha	days		mg/kg	
							(0.37)	
					14		0.29, 0.43 (0.36)	
					21		0.21, 0.09 (0.15)	
					28		0.15, 0.17 (0.16)	
					35		0.15, 0.42 (0.29)	
					180		< 0.01, < 0.01 (< 0.01)	
M335 T-6085 Delano, CA, 1984 (Navel)	300 g/L	0.22	5612	2/ 0.44	180	Whole fruit	< 0.01, < 0.01 (< 0.01)	Fujie, 1990 (FR-01-0307)
		0.45	5612	2/ 0.90	180	Whole fruit	< 0.01, < 0.01 (< 0.01)	
M335 T-6085 Porterville, CA, 1984 (Navel)	300 g/L	0.22	5612	2/ 0.44	180	Whole fruit	< 0.01, < 0.01 (< 0.01)	Fujie, 1990 (FR-01-0307)
		0.45	5612	2/ 0.90	180	Whole fruit	< 0.01, < 0.01 (< 0.01)	
M335 T-6087 Madera, CA, USA, 1984 (Navel)	300 g/L	0.22	5612	2/ 0.44	180	Whole fruit	< 0.01, < 0.01 (< 0.01)	Fujie, 1990 (FR-01-0307)
		0.45	5612	2/ 0.90	180	Whole fruit	< 0.01, < 0.01 (< 0.01)	
M335 T-6088 Madera, CA, 1984	300 g/L	0.45	5612	1	0	Whole fruit	0.25, 0.26 (0.26)	Fujie, 1990 (FR-01-0307)
					1		0.20, 0.23 (0.22)	
					3		0.08, 0.09 (0.09)	
					7		0.21, 0.20 (0.21)	
					28		0.26, 0.13 (0.20)	
					35		0.19, 0.13 (0.16)	
	300 g/L	0.45	5612	2/ 0.90	1	Whole fruit	0.35, 0.30 (0.33)	Fujie, 1990 (FR-01-0307)
					3		0.25, 0.26 (0.26)	
					7		0.18, 0.21 (0.20)	
					14		0.32, 0.24 (0.28)	
					21		0.23, 0.18 (0.21)	
M335 T-6157 Lindsay, CA, 1984 (Navel)	300 g/L	0.22	2338	2/ 0.22	180	Whole fruit	< 0.01, < 0.01 (< 0.01)	Fujie, 1990 (FR-01-0307)
		0.45	2338	2/ 0.90	180	Whole fruit	< 0.01, < 0.01 (< 0.01)	
R195 T-6389 Fresno, CA, 1985	300 g/L	0.45	3049	2/ 0.90	1	Whole fruit	0.30, 0.21 (0.26)	Fujie, 1990 (FR-01-0307)
					7		0.17, 0.17	

CITRUS	Application				DAT	Commodity	Residue	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total kg ai/ha	days		mg/kg	
(Valencia)							(0.17)	
					14		0.21, 0.15 (0.18)	
					21		0.05, 0.03 (0.04)	
R195 T-6389 Fresno, CA, USA, 1985 (Navel)	300 g/L	0.45	3002	2/0.90	1	Whole fruit	0.14, 0.10 (0.12)	Fujie, 1990 (FR-01-0307)
		0.45	3002	3/1.4	1	Whole fruit	0.08, 0.17 (0.13)	
		0.45	3002	4/1.8	1	Whole fruit	0.43, 0.51 (0.47)	
R195 T-6391 Tempe, AZ, 1985 (Valencia)	300 g/L	0.11	2104	2/0.22	1	Whole fruit	0.05, 0.03 (0.04)	Fujie, 1990 (FR-01-0307)
		0.22	2104	2/0.44	1	Whole fruit	0.30, 0.17 (0.24)	
		0.45	2104	2/0.90	1	Whole fruit	1.1, 0.81 (0.96)	
		0.90	2104	2/1.8	1	Whole fruit	0.86, 1.3 (1.1)	
R195 T 6392 Donna, TX, 1985 (Valencia)	300 g/L	0.45	6547	2/0.90	1	Whole fruit	0.42, 0.63 (0.53)	Fujie, 1990 (FR-01-0307)
					7		0.45, 0.35 (0.40)	
					14		0.36, 0.43 (0.39)	
					21		0.34, 0.36 (0.35)	
R195 T-6393 Frostproof, FL, 1985 (Valencia)	300 g/L	0.45	5855	2/0.90	1	Whole fruit	0.16, 0.25 (0.21)	Fujie, 1990 (FR-01-0307)
					7		0.30, 0.19 (0.25)	
					14		0.27, 0.17 (0.22)	
					21		0.19, 0.17 (0.18)	
R195 T-6582 Weslaco, TX 1986 (Valencia)	300 g/L	0.45	1871	2/0.90	1	Whole fruit	1.2, 1.2 (1.2)	Fujie, 1990 (FR-01-0307)
					8		0.85, 1.4 (1.1)	
					15		0.57, 0.99 (0.78)	
					22		0.64, 0.81 (0.73)	
R195 T-6583 Frostproof, FL, 1985 (Valencia)	300 g/L	0.45	2245	2/0.90	1	Whole fruit	0.26, 0.56, 0.32; (0.33)	Fujie, 1990 (FR-01-0307)
R195 T-6604 Fresno, CA, 1985 (Valencia)	300 g/L	0.45	3582	2/0.90	7	Whole fruit	0.22, 0.11 (0.17)	Fujie, 1990 (FR-01-0307)
R195 T-6723 Felda, FL, USA, 1986 (Valencia)	300 g/L	0.45	3255	2/0.90	1	Whole fruit	0.44, 0.48 (0.46)	Fujie, 1990 (FR-01-0307)

CITRUS	Application				DAT	Commodity	Residue	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total kg ai/ha	days		mg/kg	
R195 T-6730, Fresno, CA, 1986 (Navel)	300 g/L	0.45	935	2/0.90	1	Whole fruit	0.18, 0.18 (0.18)	Fujie, 1990 (FR-01-0307)
R195 T-6965 Fresno, CA, 1987 (Navel)	300 g/L	0.45	47 (aerial)	2/0.90	1	Whole fruit	0.07, 0.07 (0.07)	Fujie, 1990 (FR-01-0307)
R195 T-6966 Santa Paula, CA, 1987 (Valencia)	300 g/L	0.45	54 (aerial)	2/0.90	1	Whole fruit	0.04, 0.05 (0.05)	Fujie, 1990 (FR-01-0307)
LEMON								
T-7550 Yuma, AZ, 1990 (Unknown)	300 g/L	0.34	281	2/0.78	14	Whole fruit	0.14, 0.11 (0.13)	Lai, 1992 (FR-21-0342)
		0.45	281	2/0.9	14	Whole fruit	0.09, 0.10 (0.10)	
T-7551 Yuma, AZ, 1990 (Unknown)	300 g/L	0.34	271	2/0.78	14	Whole fruit	0.20, 0.22 (0.21)	Lai, 1992 (FR-21-0342)
		0.45	271	2/0.9	14	Whole fruit	0.34, 0.44 (0.39)	
T-7552 Yuma, AZ, 1990 (Rough)	300 g/L	0.34	281	2/0.78	14	Whole fruit	0.16, 0.12 (0.14)	Lai, 1992 (FR-21-0342)
		0.45	281	2/0.9	14	Whole fruit	0.26, 0.22 (0.24)	
V-1004A Porterville, CA, 1991 (Lisbon)	300 g/L	0.45	281	2/0.90	0	Whole fruit	0.21, 0.41 (0.31)	Lai, 1992 (FR-21-0342)
					1		0.62, 0.50 (0.56)	
					7		0.32, 0.38 (0.35)	
					14		0.27, 0.28 (0.28)	
V-1004B Fallbrook, CA, 1991 (Eureka)	300 g/L	0.45	935	2/0.90	0	Whole fruit	0.54, 0.51 (0.53)	Lai, 1992 (FR-21-0342)
					1		0.54, 0.46 (0.50)	
					7		0.50, 0.48 (0.49)	
					14		0.45, 0.57 (0.51)	
V-1004C Yuma, AZ, 1991 (Rough)	300 g/L	0.45	935	2/0.90	0	Whole fruit	0.73, 0.71 (0.72)	Lai, 1992 (FR-21-0342)
					1		0.49, 0.49 (0.49)	
					7		1.5, 0.85 (1.2)	
					14		0.72, 0.80 (0.76)	
GRAPEFRUIT								
V-1003A Mecca, CA, 1991 (Marsh Ruby)	300 g/L	0.45	935	2/0.90	0	Whole fruit	0.04, 0.06 (0.05)	Lai, 1992 (FR-21-0343)
					1		0.13, 0.11 (0.12)	

CITRUS	Application				DAT	Commodity	Residue	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total kg ai/ha	days		mg/kg	
					7		0.05, 0.06 (0.06)	
					14		0.09, 0.11 (0.10)	
V-1003B Bonsall, CA, 1991 (Ruby Red)	300 g/L	0.45	935	2/ 0.90	1	Whole fruit	0.23, 0.16 (0.20)	Lai, 1992 (FR-21-0343)
V-1003C Fillmore, CA, 1991 (Ruby Red)	300 g/L	0.45	935	2/ 0.90	1	Whole fruit	0.18, 0.18 (0.18)	Lai, 1992 (FR-21-0343)
V-1003D Myakka City, FL, 1991 (Thompson; Pink Marsh)	300 g/L	0.45	1758	2/ 0.90	0	Whole fruit	0.42, 0.51 (0.47)	Lai, 1992 (FR-21-0343)
					1		0.57, 0.36 (0.47)	
					7		0.34, 0.32 (0.33)	
					14		0.32, 0.23 (0.28)	
V-1003E Myakka City, FL, 1991 (Duncan)	300 g/L	0.45	1758	2/ 0.90	1	Whole fruit	0.36, 0.38 (0.37)	Lai, 1992 (FR-21-0343)
V-1003G Yuma, AZ, 1991 (Ruby Red)	300 g/L	0.45	935	2/ 0.90	1	Whole fruit	0.40, 0.27 (0.34)	Lai, 1992 (FR-21-0343)
V-1003I Vero Beach, FL, 1991 (Red Ruby)	300 g/L	0.45	1871	2/ 0.90	1	Whole fruit	0.32, 0.36 (0.34)	Lai, 1992 (FR-21-0343)

* Report showed this value for “control samples”. Trial samples residues were reported as < 0.01 mg/kg. The report indicated mislabelling.

Pome fruits

Apple, pear

Forty-seven supervised trials on pome fruit (27 in apples and 20 in pears) were conducted in the USA in 1984–1987, using a higher number of applications compared to the GAP in the USA (up to 0.9 kg ai/ha and a PHI of 14 days) (Fujie, 1990, Fujie and Leary, 1986; Reference: FR-81-0305, FR-01-0370, FR-81-0304, FR-01-0369). The EC formulation of fenpropathrin containing 300 g/L active ingredient, was applied to treated plots at the rates of 0.9–7.2 kg ai/ha from 7 to 42 days prior to harvest.

Samples were analysed within 1 year of harvest using the methods described in section of Methods of residue analysis, which were validated to an LOQ of 0.01 mg/kg. Concurrent recoveries

from control samples fortified with fenpropathrin at levels of 0.1 and 0.2 mg/kg were within the acceptable range of 70-120%, with RSD below 20%. Results of the trials are summarized in Table 37.

Table 37 Residues of fenpropathrin in pome fruits resulting from supervised trials in the USA

Pome fruits	Application				DAT	Commodity	Residue mg/kg	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total (kg ai/ha/ season)	days			
GAP, USA for pome fruit	300 EC (2.4 lb ai/gal)	0.22- 0.45	700-3740	2/ 0.9	14			
APPLE								
R185 T-6719 Fletcher, NC, USA, 1986 (Red Delicious)	300 g/L	0.448	3741	8/3.584	14	Whole fruit	0.14; 0.14 (0.14)	FR-81-0305
R185 T-6720 Phelps, NY, USA, 1986 (Golden Delicious)	300 g/L	0.448	2806	8/3.584	14	Whole fruit	3.0; 4.1 (3.6)	FR-81-0305
R185 T-6721 Fenville, MI, USA, 1986 (Red Delicious)	300 g/L	0.448	1272	8/3.584	14	Whole fruit	1.4; 1.5 (1.5)	FR-81-0305
R185 T-6722 Wenatchee, WA, USA, 1986 (Red Bisbee)	300 g/L	0.448	3741	8/3.584	14	Whole fruit	3.8; 3.6 (3.7)	FR-81-0305
					42	Whole fruit	3.0; 2.5	
					42	Juice	0.37; 0.37	
					42	Wet pomace	5.6; 8.7	
					42	Dry pomace	20; 31	
R185 T-6729 Watsonville, CA, USA, 1986 (Red Delicious)	300 g/L	0.448	1871	8/3.584	14	Whole fruit	2.7; 2.1 (2.4) 2.5 * (* cooked 20 minutes)	FR-81-0305
R185 T-6880 Wenatchee, WA, USA, 1987 (Red Delicious)	300 g/L	0.448	3741	8/3.584	14	Whole fruit	2.4; 2.1 (2.3)	FR-81-0305
R185 T-6969 Wenatchee, WA, USA, 1987 (Aerial) (Red Delicious)	300 g/L	0.448	65	8/3.584	14	Whole fruit	0.39; 0.40 (0.40)	FR-81-0305
R185 T-6971 Waterlief, MI, USA,,1987 (Aerial) (Jonathan)	300 g/L	0.448	47	8/3.808	14	Whole fruit	0.30; 0.14 (0.22)	FR-81-0305
R185 T-6971 Sodus, NY, USA, 1987 (Aerial) (Cortland)	300 g/L	0.448	47	8/3.584	14	Whole fruit	0.78; 0.97 (0.88)	FR-81-0305
R185 T-6055 Hood River	300 g/L	0.336	3741	9/3.024	14	Whole fruit	3.1; 3.4 (3.3)	FR-0370

Pome fruits	Application				DAT	Commodity	Residue mg/kg	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total (kg ai/ha/season)	days			
County, OR, USA, 1984 (Top Red Delicious)		0.448	3741	9/4.032	14	Whole fruit	4.1; 3.6 (3.8)	
					14	Wet pomace	13	
					14	Dry pomace	34	
					14	Juice	0.10	
R185 T-6056 Highland, NY, USA, 1984 (McIntosh)	300 g/L	0.336	3741	6/2.016	14	Whole fruit	1.1; 0.82 (0.96)	FR-0370
		0.448	3741	6/2.688	14	Whole fruit	1.2; 1.2 (1.2)	
R185 T-6058 Winchester, VA, USA, 1984 (Red Delicious)	300 g/L	0.336	3741	8/2.688	14	Whole fruit	1.3; 1.4 (1.4)	FR-0370
		0.448	3741	8/3.584	14	Whole fruit	1.1; 0.96 (1.0)	
R185 T-6059 Clemson, SC, USA, 1984 (Red Chief Red Delicious)	300 g/L	0.336	3741	8/2.688	14	Whole fruit	0.38	FR-0370
		0.448	3741	8/3.584	14	Whole fruit	0.57	
R185 T-6060 Fennville, MI, USA, 1984 (Jonathan)	300 g/L	0.336	1300	8/2.688	14	Whole fruit	1.4; 1.8 (1.6)	FR-0370
		0.448	1300	8/3.584	14	Whole fruit	2.6; 2.0 (2.3)	
R185 T-6061 Hulst Farms, CA, USA, 1984 (Pippen)	300 g/L	0.448	1871	2/0.896	14	Whole fruit	0.36; 1.4 (0.88)	FR-0370
		0.448	1871	4/1.792	14	Whole fruit	2.7; 2.4 (2.6)	
		0.448	1871	6/2.688	14	Whole fruit	2.7; 2.3 (2.5)	
		0.448	1871	8/3.584	14	Whole fruit	1.7; 1.6 (1.7)	
R185 T-6150 Biglerville, PA, USA, 1984 (York Imperial)	300 g/L	0.336	2806	8/2.688	10	Whole fruit	1.2; 3.0 (2.1)	FR-0370
		0.448	2806	8/3.584	10	Whole fruit	3.7; 3.6 (3.7)	
R185 T-6151 Phelps, NY, USA, 1984	300 g/L	0.448	2806	2/0.896	14	Whole fruit	0.95; 1.3 (1.1)	FR-0370
		0.448	2806	4/1.792	14	Whole fruit	2.1; 2.7 (2.4)	
		0.448	2806	5/2.688	14	Whole fruit	2.7; 3.2 (3.0)	
		0.448	2806	8/3.584	14	Whole fruit	2.6; 2.5 (2.6)	
R185 T-6152 Phelps, NY, USA, 1984 (Golden Delicious)	300 g/L	0.448	2806	8/3.584	7	Whole fruit	3.2; 3.2	FR-0370
		0.448	2806	8/3.584	14	Whole fruit	2.7; 2.3 (2.5)	
					14	Wet pomace	13; 11	
					14	Dry pomace	44; 45	
					14	Juice	< 0.01; < 0.01	
		0.448	2806	8/3.584	21	Whole fruit	2.1; 2.5 (2.3)	
		0.448	2806	8/3.584	28	Whole fruit	3.4; 2.9 (3.2)	
R185 T-6154 Wenatchee, WA, USA, 1984 (Golden Delicious)	300 g/L	0.336	3741	8/2.688	14	Whole fruit	1.5; 2.4 (2.0)	FR-0370
		0.448	3741	8/3.584	7	Whole fruit	2.3; 2.0 (2.2)	
		0.448	3741	8/3.584		Pulp	0.77; 0.20	
		0.448	3741	8/3.584		Peel	12; 11	
		0.448	3741	8/3.584	14	Whole fruit	2.2; 2.6 (2.4)	
		0.448	3741	8/3.584	21	Whole fruit	2.1; 2.1 (2.1)	
		0.448	3741	8/3.584	28	Whole fruit	2.2; 2.1 (2.2)	

Pome fruits	Application				DAT	Commodity	Residue mg/kg	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total (kg ai/ha/season)	days			
R185 T-6400) Wenatchee, WA, USA, 1985 (Golden Delicious)	300 g/L	0.112	3741	8/0.896	14	Whole fruit	1.3; 1.7 (1.5)	FR-0370
		0.224	3741	8/1.792	14	Whole fruit	2.4; 3.2 (2.8)	
		0.448	3741	8/3.584	14	Whole fruit	5.1; 7.3 (6.2)	
		0.896	3741	8/7.168	14	Whole fruit	7.6; 8.2 (7.9)	
R185 T-6401, Wenatchee WA, USA, 1985 (Golden Delicious)	300 g/L	0.448	3741	8/3.584	14	Whole fruit	5.2; 4.2 (4.7)	FR-0370
					14	Wet pomace	12; 12	
					14	Dry pomace	47; 38	
					14	Juice	0.11, 0.12	
							0.15, 0.16	
R185 T-6402 Hood River, OR, USA, 1985 (Red Delicious)	300 g/L	0.448	3741	2/0.896	14	Whole fruit	0.51; 0.65 (0.58)	FR-0370
		0.448	3741	4/1.792	14	Whole fruit	1.3; 1.5 (1.4)	
		0.448	3741	6/2.688	14	Whole fruit	1.6; 2.0 (1.8)	
		0.448	3741	8/3.584	14	Whole fruit	1.4; 1.1 (1.3)	
		0.448	3741	8/3.584	14	Whole fruit	1.4; 1.4	
					14	Pulp	0.07; 0.15	
					14	Peel	4.0; 5.5	
R185 T-6403 Haslet, MI, USA, 1985 (Red Delicious)	300 g/L	0.448	1010	2/0.896	14	Whole fruit	0.40; 0.56 (0.48)	FR-0370
		0.448	1010	4/1.792	14	Whole fruit	1.6; 2.1 (1.9)	
		0.448	1010	6/2.688	14	Whole fruit	2.8; 2.3 (2.6)	
		0.448	1010	8/3.584	14	Whole fruit	3.3; 4.0 (3.8)	
R185 T-6404 Fennville, MI, USA, 1985 (Red Delicious)	300 g/L	0.112	1132	8/0.896	14	Whole fruit	0.02 *	FR-0370 * Duplicate sample broken in shipment.
		0.224	1132	8/1.792	14	Whole fruit	2.6 *	
		0.448	1132	8/3.584	14	Whole fruit	3.8; 5.2 (4.5)	
		0.896	1132	8/7.168	14	Whole fruit	7.8; 8.8	
R185 T-6405 Wooster, OH, USA, 1985 (Cortland)	300 g/L	0.448	2806	8/3.584	14	Whole fruit	1.5; 1.5; 1.7; 5.7*; 1.8; 2.1 (1.7)	FR-0370 * Outlier, based on Dixon's Q test
R185 T-6406 Phelps, NY, USA, 1985 (Rhode Island Greening)	300 g/L	0.448	2806	8/3.584	14	Whole fruit	1.6; 1.6 (1.6)	FR-0370
					14	Whole fruit	1.4; 1.1 (1.3)	
					21	We pomace	3.8; 5.1	
					21	Dry pomace	12; 8.4	
					21	Juice	0.01; 0.01	
PEAR								
R186 T-6709 River Bottom, CA (Bartlett)	300 g/L	0.448	935	6/2.688	14	Whole fruit	1.3; 0.96 (1.1) 0.94*	FR-81-0304 * Washed under tap water with gentle hand rubbing
R186 T-6711 Medford, OR, USA, 1986 (Bartlett)	300 g/L	0.448	3741	6/2.688	14	Whole fruit	3.0; 2.8 (2.9)	FR-81-0304
R186 T-6712 Fennville, MI, USA, 1968	300 g/L	0.488	1300	6/2.688	14	Whole fruit	2.0; 1.6 (1.8)	FR-81-0304

Pome fruits	Application				DAT	Commodity	Residue mg/kg	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total (kg ai/ha/ season)	days			
(Bartlett)								
R186 T-6713 Phelps, NY, USA, 1986 (Bartlett)	300 g/L	0.448	2806	6/2.688	14	Whole fruit	2.5; 2.3 (2.4)	FR-81-0304
R186 T-6886 Lagrange, CA, USA, 1987 (Bartlett)	300 g/L	0.448	935	6/2.688	14	Whole fruit	1.9; 1.6 (1.8)	FR-81-0304
R186 T-6972 Live Oak, CA (Aerial), USA, 1987 (Bartlett)	300 g/L	0.448	1878	6/2.688	14	Whole fruit	0.26; 0.32 (0.29)	FR-81-0304
R186 T-6973 Sodus, NY (Aerial), USA, 1987 (Bartlett)	300 g/L	0.448	47	6/2.688	14	Whole fruit	0.76; 0.96 (0.86)	FR-81-0304
R186 T-6062 Live Oak, CA, USA, 1984 (Bartlett)	300 g/L	0.336	3741	6/2.016	14	Whole fruit	0.84; 0.34 (0.59)	FR-0369
		0.448	3741	6/2.688	7	Whole fruit	0.52; 0.84	
					14		0.77; 0.46 (0.62)	
					21		0.59; 0.52	
					28		0.56; 0.42	
R186 T-6063 Live Oak, CA, USA, 1984 (Bartlett)	300 g/L	0.336	3741	6/2.016	14	Whole fruit	0.42; 0.36 (0.39)	FR-0369
		0.448	3741	2/0.892	14	Whole fruit	0.27; 0.27 (0.27)	
		0.448	3741	3/1.344	14	Whole fruit	0.55; 0.34 (0.45)	
		0.448	3741	5/2.24	14	Whole fruit	0.41; 0.42 (0.42)	
		0.448	3741	6/2.688	14	Whole fruit	0.65; 0.50 (0.53)	
R186 T-6064 Medford, OR, USA, 1984 (Bartlett)	300 g/L	0.448	3741	6/2.688	14	Whole fruit	0.96; 1.4 (1.2)	FR-0369
R186 T-6065 Wenatchee, WA, USA, 1984 (Bartlett)	300 g/L	0.336	3741	6/2.196	14	Whole fruit	0.87; 1.0 (0.94)	FR-0369
		0.448	3741	6/2.688	14	Whole fruit	1.5; 1.1 (1.3)	
R186 T-6066 Fenville, MI, USA, 1984 (Bartlett)	300 g/L	0.336	1422	6/2.016	14	Whole fruit	1.3	FR-0369
R186 T-6067 Phelps, NY, USA, 1984 (Bartlett)	300 g/L	0.448	2806	2/0.896	14	Whole fruit	1.2; 1.1 (<u>1.2</u>)	FR-0369
		0.448	2806	4/1.792	14	Whole fruit	1.5; 1.4 (1.5)	
		0.448	2806	5/2.24	14	Whole fruit	1.4; 1.4 (1.4)	
		0.448	2806	6/2.688	14	Whole fruit	1.7; 1.7 (1.7)	
					14	Pear halves in	< 0.01;	

Pome fruits	Application				DAT	Commodity	Residue mg/kg	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total (kg ai/ha/ season)	days			
						syrup	< 0.01	
					14	Puree	< 0.01; < 0.01	
R186 T-6153 Phelps, NY, USA, 1984 (Bartlett)	300 g/L	0.448	2806	6/2.688	7	Whole fruit	2.7; 1.8 (2.3)	FR-0369
		0.448	2806	6/2.688	14	Whole fruit	2.2; 1.6 (1.9)	
		0.448	2806	6/2.688	21	Whole fruit	2.0; 1.9 (2.0)	
		0.448	2806	6/2.688	28	Whole fruit	1.0; 0.89 (0.95)	
R186 T-6394 Wenatchee, WA, USA, 1985 (Bartlett)	300 g/L	0.112	3741	6/0.672	14	Whole fruit	0.82; 0.57 (0.70)	FR-0369
		0.224	3741	6/1.344	14	Whole fruit	1.2; 0.72 (0.96)	
		0.448	3741	6/2.688	14	Whole fruit	1.7; 0.31 (1.0)	
		0.896	3741	6/5.376	14	Whole fruit	3.8; 2.6 (3.2)	
R186 T-6395 Hood River, OR, USA, 1985 (Bartlett)	300 g/L	0.448	3741	2/0.896	14	Whole fruit	0.31; 0.29 (0.30)	FR-0369 * Washed
		0.448	3741	4/1.792	14	Whole fruit	0.47; 0.78 (0.63)	
		0.448	3741	6/2.688	14	Whole fruit	0.62; 0.85 (0.74)	
		0.448	3741	8/3.584	14	Whole fruit	1.0; 0.89 (0.95)	
		0.448	3741	8/3.584	21	Whole fruit	0.91; 0.86	
					21		0.72*	
					21	Peel	5.9	
					21	Pulp	0.01	
R186 T-6396 Ukiah, CA, USA, 1985 (Bartlett)	300 g/L	0.448	3741	6/2.688	1 1 1	Whole fruit Pulp Peel	1.6; 1.4 0.01; < 0.01 12; 13	FR-0369
		0.448	3741	6/2.688	7	Whole fruit	2.0; 2.3 (2.2)	
		0.448	3741	6/2.688	14	Whole fruit	1.5; 1.6 (1.6)	
		0.448	3741	6/2.688	21	Whole fruit	1.4; 1.1 (1.3)	
		0.448	3741	6/2.688	28	Whole fruit	0.78; 0.91 (0.85)	
		0.448	3741	6/2.688	35	Whole fruit	0.79; 1.1 (0.95)	
R186 T-6397 Fennville, MI, USA.1985 (Bartlett)	300 g/L	0.448	1085	2/0.896	14	Whole fruit	1.6; 2.0 (1.8)	FR-0369
		0.448	1085	4/1.792	14	Whole fruit	2.4; 2.8 (2.6)	
		0.448	1085	6/2.688	14	Whole fruit	2.9; 4.5 (3.7)	
		0.448	1085	8/3.584	14	Whole fruit	4.8; 3.9 (4.4)	
R186 T-6398 Phelps, NY, USA, 1985 (Bartlett)	300 g/L	0.112	2806	6/0.672	14	Whole fruit	0.48; 0.54 (0.51)	FR-0369
		0.224	2806	6/1.344	14	Whole fruit	1.2; 1.0 (1.1)	
		0.448	2806	6/2.688	14	Whole fruit	2.2; 2.1 (2.2)	
		0.896	2806	6/5.376	14	Whole fruit	3.3; 3.2 (3.3)	

*Stone fruit**Peach, plum, cherry*

Supervised trials were conducted on stone fruits in the USA during 2003. Ten of the trials were on peaches (Green, 2004; Reference FR-0384), six on cherries (Green, 2004; Reference FR-0381), and seven on plums (Green, 2004; FR-0385). All trials were according to the US GAP on stone fruit, which includes applications of fenpropathrin 300 g/L EC formulation at the rate of 0.45 kg ai/ha for a maximum seasonal application of 0.9 kg ai/ha. Duplicate samples of mature fruits were collected 3 days after the last application. Sample size for peach ranged from 2.6 to 8.5 kg, ≥ 24 fruit. Upon collection, all samples were frozen at -20 °C until extraction and analysis. All of the samples were analysed within the documented storage stability interval analysed using analytical method RM-22-4 (LOQ 0.01 mg/kg). Concurrent recoveries were within the acceptable range of 70 to 120%.

Table 38 Residues of fenpropathrin in stone fruit resulting from supervised trials in the United States

STONE FRUIT	Application				DAT	Commodity	Residue	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha/	days		mg/kg	
GAP, USA	300 g/L EC	0.22-0.45	935- 3740	0.9	3			
PEACHES								
V-25419-A Berks County, PA, USA, 2003 (John Boy)	300 EC	0.45 0.46	1122 1141	2/0.91	3	Whole fruit	0.66, 0.66 (0.66)	FR-0384
V-25419-B Saluda County, SC, USA, 2003 (Sureprince)	300 EC	0.45 0.49	1207 1178	2/0.94	3	Whole fruit	0.76, 0.63 (0.70)	FR-0384
		0.90 0.89	1216 1169	2/1.79	3	Whole fruit	1.3, 0.90 (1.1)	
V-25419-C Aiken, County, SC, USA, 2003 (Contender)	300 EC	0.44 0.44	1066 1057	2/0.88	3	Whole fruit	0.56, 0.73 (0.65)	FR-0384
V-25419-D Houston County, AL, USA, 2003 (Harvester)	300 EC	0.45 0.45	1094 1104	2/0.90	3	Whole fruit	0.91, 1.1 (1.0)	FR-0384
V-25419-E Clarke County, GA, USA, 2003 (Contender)	300 EC	0.44 0.45	1141 1225	2/0.89	3	Whole fruit	1.0, 1.0 (1.0)	FR-0384
V-25419-F Ottawa County, MI, USA, 2003 (Red Haven)	300 EC	0.44 0.44	1132 1132	2/0.88	3	Whole fruit	0.69, 0.72 (0.71)	FR-0384
V-25419-G Freestone County, TX, USA, 2003 (Fairtime)	300 EC	0.46 0.44	945 1085	2/0.90	3	Whole fruit	0.79, 0.67 (0.73)	FR-0384
		0.92 0.89	945 1085	2/1.8	3	Whole fruit	1.1, 0.66 (0.88)	
V-25419-H Sutter County, CA, USA, 2003 (Loadel)	300 EC	0.45 0.45	1066 1066	2/0.90	3	Whole fruit	0.61, 0.54 (0.58)	FR-0384

STONE FRUIT	Application				DAT	Commodity	Residue	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha/	days		mg/kg	
V-25419-I Stanislaus, County CA, USA, 2003 (Summerset)	300 EC	0.45 0.44	1291 1272	2/0.89	1	Whole fruit	0.50, 0.45 (0.48)	FR-0384
					4	Whole fruit	0.49, 0.39 (0.44)	
					7	Whole fruit	0.35, 0.24 (0.30)	
					10	Whole fruit	0.34, 0.34 (0.34)	
V-25419-J Fresno County, CA, USA, 2003 (Fayette)	300 EC	0.43 0.44	1178 1197	2/0.87	3	Whole fruit	1.0, 0.84 (0.92)	FR-0384
CHERRIES								
V-25380-A Wayne County, NY, USA, 2003 (Montmorency/ tart)	300 EC	0.46 0.47	973 982	2/0.93	3	Whole fruit	1.83, 1.97 (1.9)	FR-0381
		0.92 0.94	963 982	2/1.86	3	Whole fruit	5.16, 4.21 (4.7)	
V-25380-B Ottawa County, MI, USA, 2003 (Napoleon/ sweet)	300 EC	0.45 0.45	1104 1094	2/0.90	1	Whole fruit	1.76, 1.62 (1.7)	FR-0381
					3		2.02, 1.6 (1.8)	
					7		1.43, 1.4 (1.4)	
					10		1.38, 1.16 (1.3)	
V-25380-C Delta County, CO, USA, 2003 (Montmorency/ tart)	300 EC	0.45 0.45	954 945	2/0.90	3	Whole fruit	1.44, 1.43 (1.4)	FR-0381
V-25380-D Lehigh County, PA, USA, 2003 (Montmorency/ tart)	300 EC	0.45 0.45	991 1001	2/0.90	3	Whole fruit	3.18, 3.33 (3.3)	FR-0381
V-25380-E Santa Clara, CA, USA, 2003 (Bing/sweet)	300 EC	0.44 0.43	1001 982	2/0.87	3	Whole fruit	1.59, 1.43 (1.5)	FR-0381
		0.88 0.86	1010 982	2/1.74	3	Whole fruit	2.26, 1.90 (2.1)	
V-25380-F Grant County, WA, USA, 2003 (Van/ sweet)	300 EC	0.45 0.45	945 935	2/0.90	1	Whole fruit	2.74, 2.98 (2.9)	FR-0381
					3	Whole fruit	3.53, 3.23 (3.4)	
					7	Whole fruit	3.06, 2.76 (2.9)	
					10	Whole fruit	2.41, 2.3 (2.4)	
PLUMS								
V-25427-A Ottawa County, MI, USA, 2003	300 EC	0.44 0.45	1057 1094	2/0.89	1	Whole fruit	0.57, 0.62 (0.60)	FR-0385
					3	Whole fruit	0.51, 0.58	

STONE FRUIT	Application				DAT	Commodity	Residue	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha/	days		mg/kg	
(Stanley)							(0.55)	
					7	Whole fruit	0.71, 0.63 (0.67)	
					10	Whole fruit	0.44, 0.46 (0.45)	
V-25427-B Tulare County, CA, USA, 2003 (Angelona's)	300 EC	0.45 0.46	1029 1048	2/0.91	3	Whole fruit	0.23, 0.23 (0.23)	FR-0385
V-25427-C Sutter County, CA, USA, 2003 (French Prune)	300 EC	0.45 0.45	1038 1029	2/0.90	3	Whole fruit	0.29, 0.21 (0.25)	FR-0385
V-25427-D Tehama County, CA, 2003 (French Prune)	300 EC	0.45 0.45	1029 1029	2/0.90	3	Whole fruit	0.37, 0.27 (0.32)	FR-0385
V-25427-E Polk County, OR, 2003 (Moyer)	300 EC	0.44 0.45	1085 1104	2/0.89	4	Whole fruit	0.24, 0.20 (0.22)	FR-0385
V-25427-F Fresno County, CA, USA, 2003 (French Prune)	300 EC	0.44	1150	2/0.88	4	Whole fruit	0.17, 0.18 (0.18)	FR-0385
		0.44	1169					
		0.89 0.90	1160 1178	2/1.8	4	Whole fruit	0.61, 0.54 (0.58)	
V-27220-A Fresno County, CA, USA, 2004 (French Prune)	300 EC	0.44 0.45	1169 1188	2/0.89	3	Whole fruit	0.29, 0.34 0.27, 0.50 (0.35)	FR-0559

Berries and other small fruit

Strawberry

Ten supervised trials on strawberries were conducted in the US in 1987 and 1989 (Mitten, 1991; Reference FR-11-0345). Two additional trials were carried out in 1991 (Lai, 1992; Reference FR-21-0346). Eleven of the trials matched the current GAP in the US for strawberries (applications at 0.22 to 0.45 kg ai/ha for a total of 0.9 kg ai/ha per season and a PHI of 2 days). At several sites, replicate trials were carried out to study the effect of application rate and PHI on the residues in harvested samples. Mature samples were collected two days after the last application and immediately stored frozen at -20 °C until analysis. Samples from the trials were analysed within this storage stability period with method RM-22-4. Concurrent recoveries from fortified samples analysed together with field samples ranged from 81-115%, mean = 98.3%, with RSD of 8.2% (n=27).

Table 39 Residues of fenpropathrin in strawberries resulting from supervised trials in the United States

Strawberry	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total/ kg ai/ha	days		mg/kg	
GAP, USA	300 EC	0.22- 0.45	935- 2800	2/0.9	2			
M391 T-7125 Watsonville, CA, USA, 1987 (Muir)	300 g/L	0.34	2245	0.34	1 3	Berries	1.2, 1.2 (1.2) 1.1, 1.2 (1.2)	FR-0345
		0.34	2245	2/0.68	1 3	Berries	1.3, 1.2 (1.3) 1.1, 1.1 (1.1)	
M391 T-7126 Watsonville, CA USA, 1987 (Selva)	300 g/L	0.34	2245	0.34	1 3	Berries	0.77, 0.87 (0.82) 0.69, 0.65 (0.67)	FR-0345
		0.34	2245	2/0.68	1 3	Berries	0.68, 0.58 (0.63) 0.56, 0.62 (0.59)	
1714-89-7279 Watsonville, CA, USA, 1989 (Selva)	300 g/L	0.11	1403	0.11	2	Berries	0.43, 0.40 (0.42)	FR-0345
		0.11	1403	2/0.22	2	Berries	0.10, 0.12 (0.11)	
		0.11	1403	3/0.34	2	Berries	0.08, 0.06 (0.07)	
		0.11	1403	4/0.44	2	Berries	0.11, 0.09 (0.10)	
		0.22	1403	0.22	2	Berries	0.78, 0.76 (0.77)	
		0.22	1403	2/0.44	2	Berries	0.35, 0.27 (0.31)	
		0.22	1403	3/0.66	2	Berries	0.22, 0.17 (0.20)	
		0.22	1403	4/0.89	2	Berries	0.12, 0.26 (0.19)	
		0.45	1403	0.45	2	Berries	1.2, 1.1 (1.2)	
		0.45	1403	2/0.90	2	Berries	0.72, 0.54 (0.63)	FR-0345
		0.45	1403	3/1.35	2	Berries	0.35, 0.35 (0.35)	
		0.45	1403	4/1.8	2	Berries	0.43, 0.53 (0.48)	
		0.90	1403	0.90	2	Berries	2.6, 2.8 (2.7)	
		0.90	1403	2/1.8	2	Berries	0.90, 1.3 (1.1)	
		0.90	1403	3/2.7	2	Berries	0.84, 0.87 (0.86)	
		0.90	1403	4/3.6	2	Berries	1.1, 0.74	

Strawberry	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total/ kg ai/ha	days		mg/kg	
							(0.92)	
1714-89-7281 Rosa, LA USA, 1989 (Chandler)	300 g/L	0.45	1440	0.45	2	Berries	0.83, 0.86 (0.85)	FR-0345
		0.45	1440	2/0.89	2	Berries	0.46, 0.50 (0.48)	
1714-89-7282 Bonsall, CA, USA, 1989 (Chandler)	300 g/L	0.45	2806	0.45	2	Berries	0.68, 1.0 (0.84)	FR-0345
		0.45	2806	2/0.90	2	Berries	0.59, 0.71 (0.65)	
		0.45	2806	3/1.3	2 29	Berries	0.43, 0.39 (0.41) 0.05, 0.05 (0.05)	
		0.45	2806	4/1.8	0	Berries	0.55, 0.56 (0.56)	
					1		0.55, 0.44 (0.50)	
					2		0.52, 0.55 (0.54)	
					3		0.45, 0.40 (0.43)	
					4		0.60, 0.57 (0.59)	
					7		0.41, 0.38 (0.40)	
					14		0.14, 0.13 (0.14)	
					21		0.09, 0.10 (0.10)	
							0.03, 0.04 (0.04)	
1741-89-7283 Fresno, CA, USA, 1989 (Chandler)	300 g/L	0.45	1403	2/0.90	2	Berries	0.63, 0.74 (0.69)	FR-0345
1741-89-7285 Santa Maria, CA, USA, 1989 (Muir)	300 g/L	0.45	2806	0.45	2	Berries	1.5, 1.4 (1.5)	FR-0345
		0.45	2806	2/0.90	2	Berries	1.2, 1.2 (1.2)	
		0.45	2806	3/1.3	2	Berries	0.72, 0.76 (0.74)	
		0.45	2806	4/1.8	2	Berries	0.56, 0.66 (0.61)	
1741-89-7287 Grand Rapids, MI, USA, 1989 (Not reported)	300 g/L	0.11	1225	1/0.11	2	Berries	0.07	FR-0345
		0.22	1225	2/0.22	2	Berries	0.16	
		0.45	1225	1/0.45	2	Berries	0.28	
		0.90	1225	2/0.90	2	Berries	0.63	

Strawberry	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total/ kg ai/ha	days		mg/kg	
		0.11	1225	2/0.22	2	Berries	0.09	
		0.22	1225	2/0.44	2	Berries	0.23	
		0.45	1225	2/0.90	2	Berries	<u>0.48</u>	
		0.90	1225	2/1.8	2	Berries	1.1	
1714-89-7427 Vancouver, WA, USA, 1989 (S. Huxson)	300 g/L	0.45	1403	0.45	2	Berries	0.21, 0.20 (0.21)	FR-0345
		0.45	1403	2/0.90	2	Berries	0.29, 0.49 (<u>0.39</u>)	
1714-89-7428 Gales Creek, OR, USA, 1989 (Benton)	300 g/L	0.45	1403	0.45	29	Berries	0.04, 0.03 (0.04)	FR-0345
		0.45	1403	2/0.90	0	Berries	0.38, 0.44 (0.41)	
					1		0.36, 0.47 (0.42)	
					2		0.36, 0.34 (0.35)	
					3		0.38, 0.37 (<u>0.38</u>)	
					4		0.28, 0.35 (0.32)	
					7		0.29, 0.32 (0.31)	
					14		0.19, 0.17 (0.18)	
					21		0.12, 0.11 (0.12)	
					28		0.23, 0.14 (0.19)	
V-1020A Wilson, NC, USA, 1991 (Chandler)	300 g/L	0.45	963	0.45	2	Berries	0.16, 0.18 (0.17)	FR-21-0346
		0.45	963	2/0.90	2	Berries	0.29, 0.22 (<u>0.26</u>)	
V-1020B Watsonville, CA, USA, 1991 (Selva)	300 g/L	0.45	935	0.45	2	Berries	0.29, 0.37 (0.33)	FR-21-0346
		0.45	935	2/0.90	2	Berries	0.59, 0.51 (<u>0.55</u>)	

Raspberry

Seven supervised trials on raspberries were conducted in the USA in 2005 (Samoil, 2007; Reference FR-0560) with higher rate and shorter PHI than the current GAP (applications at 0.22 to 0.34 kg ai/ha for a total of 0.67 kg ai/ha per season and a PHI of 3 days). Mature samples were collected 2–3 days after the last application and immediately stored frozen at -20 °C until analysis. Samples from the trials were analysed within this storage stability period 216 days with method RM-22-4. Concurrent recoveries ranged from 74–110%, mean = 98.3%, with SD of 8.2% (n=10).

Table 40 Residues of fenpropathrin in raspberries resulting from supervised trials in the United States

Raspberry	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		mg/kg	
GAP, USA	2.4 EC (300g ai/l; 2.4lb ai/gal)	0.22- 0.34	935- 2800	0.67	3			
CA*62 Corralitos, CA, USA, 2005 (Isabel)	300 g/L	0.45	505	2/0.9	2	Berries	1.0, 1.2 (1.1)	FR-0560
MI06 Holt, MI USA, 2005 (Heritage)	300 g/L	0.46 0.47	206	2/0.93	2	Berries	5.5, 6.1 (5.8)	FR-0560
NC12 Bridgetown, NJ USA, 2005 (Mandarin)	300 g/L	0.44 0.45	271	2/0.89	2	Berries	4.1, 7.1 (5.6)	FR-0560
NC14 Fletcher, NC USA, 2005 (Canby)	300 g/L	0.48	290	2/0.96	3	Berries	1.4, 1.9 (1.7)	FR-0560
OR07 Aurora, OR USA, 2005 (Marion)	300 g/L	0.49 0.46	411 383	2/0.95	2	Berries	4.7, 3.3 (4.0)	FR-0560
OR08 Aurora, OR USA, 2005 (Willamette)	300 g/L	0.47 0.49	393 402	2/0.96	2	Berries	1.8, 1.7 (1.8)	FR-0560
OR09 Aurora, OR USA, 2005 (Willamette)	300 g/L	0.47	393	2/0.94	2	Berries	2.3, 1.8 (2.1)	FR-0560

Grapes

Twenty five supervised trials were conducted on grapes in the USA during 1983-2001. (Fujie, 1990; Fujie, 1992, Green, 2002; FR-01-0308, FR-01-0335, FR-01-0374). All trials were within the US GAP on grapes. Duplicate samples of mature fruits were collected 21 days after the last application.

All samples were analysed using analytical method RM-22-4 with an LOQ of 0.01 mg/kg within the storage stability period. Concurrent recoveries were within the acceptable range of 70 to 120%.

Table 41 Residues of fenpropathrin in grapes resulting from supervised trials in the United States

GRAPES	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/l)	kg ai/ha	Water (l/ha)	No./ Total kg ai/ha	days		mg/kg	
GAP, USA for grapes	300 EC (300g ai/l)	0.11- 0.45	234- 1870	2/ 0.9	21			
GRAPE								
M335 T-5952 Fresno, CA, USA, 1983 (Thompson Seedless)	300 g/L	0.112	1019	2/0.336	25	Bunches	0.15, 0.13	FR-01-0308
		0.224	1019					
		0.112	1019	2/0.224	25	Bunches	0.07, 0.12	
		0.112	1019					
R187 T-6077, Fresno, CA, USA, 1984 (Thompson Seedless)	300 g/L	0.224	1001	4/0.896	21	Bunches Raisins* Wet pomace Dry pomace Juice Raisin waste	0.31; 0.43 (0.37) 0.45 0.49 2.45 0.02; 0.03 0.03, 0.02**	FR-01-0308 * dried 15 days in field ** repeat analysis
R187 T-6078, Madera, CA, USA, 1984 (Thompson Seedless)	300 g/L	0.224	1403	4/0.896	21	Bunches Raisins* Wet pomace Dry pomace Juice Raisin waste	0.60; 0.90 (0.75) 1.52 0.90 4.90 0.09; 0.09 0.13, 0.13**	FR-01-0308 * dried 15 days in field ** repeat analysis
R187 T-6079, Delano, CA, USA, 1984 (Centennial)	300 g/L	0.224	2338	2/0.448	14	Bunches	0.49, 0.64	FR-01-0308
R187 T-6081, Santa Rosa, CA, USA, 1984 (Cabernet Sauvignon)	300 g/L	0.224	935	0.224	95	Fruit	0.27, 0.16	FR-01-0308
R187 T-6409, Fresno, CA, USA, 1985 (Thompson Seedless)	300 g/L	0.224	1019	4/0.896	1	Fruit	0.49, 0.35 0.43*	FR-01-0308 * washed under tap water
		0.224	1019	4/0.896	7	Fruit	0.72; 0.57	
		0.224	1019	4/0.896	14	Fruit	0.48; 0.40	
		0.224	1019	4/0.896	21	Fruit	0.42; 0.62 (0.52)	
		0.224	1019	4/0.896	28	Fruit	0.44, 0.58 (0.51)	
		0.224	1019	4/0.896	35	Fruit	1.1; 0.56 (0.83)	
R187 T-6410, CA, USA, 1985 (Thompson Seedless)	300 g/L	0.056	1871	4/0.224	21	Bunches	0.05; 0.16	FR-01-0308
		0.112	1871	4/0.448	21	Bunches	0.15; 0.10	
		0.224	1871	4/0.896	21	Bunches	0.45; 0.44 (0.45)	
		0.448	1871	4/1.792	21	Bunches	0.81; 1.5 (1.2)	
R187 T-6411, Santa Maria, CA, USA, 1985 (Chardonnay)	300 g/L	0.224	1871	0.224	21	Bunches	0.34, 0.21	FR-01-0308
		0.224	1871	2/0.448	21	Bunches	0.19; 0.96 (0.58)	
		0.224	1871	3/0.672	21	Bunches	1.0; 0.77 (0.89)	

GRAPES	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/l)	kg ai/ha	Water (l/ha)	No./ Total kg ai/ha	days		mg/kg	
		0.224	1871	4/0.896	21	Bunches	1.6; 1.3_ (1.5)	
R187 T-6412, Fresno, CA, USA, 1985 (Thompson Seedless)	300 g/L	0.224	1019	4/0.896	21	Bunches Raisins* Wet pomace Dry pomace Juice Raisins – off grade* Raisin waste Wine	1.0; 0.48 (0.74) 0.52 0.85 1.2 < 0.01 0.81 3.23 < 0.01	FR-01-0308 * dried 33 days in field
R187 T-6413, Ukiah, CA, USA, 1985 (Caignane)	300 g/L	0.224	1403	4/0.896	21	Bunches Juice Wine	3.3; 2.9 (3.1) < 0.01; 0.01 < 0.01; < 0.01	FR-01-0308
R187 T-6414, Prosser, WA, USA, 1985 (Chardonnay)	300 g/L	0.224	935	0.224	1	Bunches	1.8; 2.8	FR-01-0308
		0.224	935	0.224	7	Bunches	2.0; 1.9	
		0.224	935	0.224	14	Bunches	1.8; 1.6	
		0.224	935	0.224	21	Bunches	1.5; 1.3	
		0.224	935	0.224	28	Bunches	1.0; < 0.01	
		0.224	935	0.224	35	Bunches	0.94; 0.10	
R187 T-6415, Fredonia, NY, USA, 1985 (Concord)	300 g/L	0.224	935	4/0.896	1	Bunches	1.3; 0.82	FR-01-0308
					7		0.90; 0.92	
					14		0.47; 0.87	
					21		0.81; 1.4 (1.1)	
					28		0.80; 0.66	
					35		0.89; 0.91	
R187 T-6416, Wooster, OH, USA, 1985 (Concord)	300 g/L	0.056	935	4/0.224	21	Bunches	0.08; 0.22	FR-01-0308
		0.112	935	4/0.448	21	Bunches	0.16; 0.38	
		0.224	935	4/0.896	21	Bunches	1.4; 0.72 (1.1)	
		0.448	935	4/1.792	21	Bunches Wet pomace Dry pomace Juice	2.4; 2.8 (2.6) 3.8; 1.8 11; 10 0.01; < 0.01	
R187 T-6417, Fennville, MI, USA, 1985 (Concord)	300 g/L	0.224	926	0.224	21	Bunches	0.26; 0.16	FR-01-0308 * washed, frozen
		0.224	926	2/0.448	21	Bunches	1.5; 1.0 (1.3)	
		0.224	926	3/0.672	21	Bunches	1.2; 1.8 (1.5)	
		0.224	926	4/0.896	21	Bunches	1.5; 1.5 (1.5) 1.0*	
R187 T-6725, Phelps, NY, USA, 1986 (Catawba)	300 g/L	0.224	935	4/0.896	19	Bunches	1.4; 0.81 (1.1)	FR-01-0308
R187 T-6726, Fennville, MI, USA, 1986 (Concord)	300 g/L	0.224	926	4/0.896	14	Bunches	1.6, 1.2	FR-01-0308
R187 T-6728, Fresno, CA, USA, 1986	300 g/L	0.224	1001	4/0.896	21	Bunches	0.93; 1.1 (1.0) 0.87*	FR-01-0308 * washed under

GRAPES	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/l)	kg ai/ha	Water (l/ha)	No./ Total kg ai/ha	days		mg/kg	
(Thompson Seedless)								tap water
R187 T-6731, Hughson, CA, USA, 1986 (Chenin Blanc)	300 g/L	0.224	935	4/0.896	21	Bunches Wine Juice Wet pomace Dry pomace Stems	1.3; 1.5 (1.4) < 0.01 < 0.01 0.99 2.6 1.2	FR-01-0308
R187 T-6829, Soledad, CA, USA, 1986 (Cabernet Sauvignon)	300 g/L	0.224	935	4/0.896	21	Bunches	5.7; 5.4 (5.6)	FR-01-0308
		0.224	935	4/0.896	21	Wet pomace Dry pomace Juice Wine	4.1 9.4 0.06 < 0.01	
R187 T-6835, Prosser, WA, USA, 1986 (Chardonnay)	300 g/L	0.414	1796	4/1.656	1	Bunches	1.0; 0.98	FR-01-0308 *duplicate analysis
		0.414	1796	4/1.656	7	Bunches	3.2; 1.9	
		0.414	1796	4/1.656	14	Bunches	1.6; 1.8	
		0.414	1796	4/1.656	21	Bunches Juice Wine Dry pomace	1.2; 1.3 (1.3) 0.04; 0.05; 0.04 < 0.01; < 0.01; < 0.01 4.2, 4.0*	
		0.414	1796	4/1.656	28	Bunches	0.72; 1.6	
		0.414	1796	4/1.656	35	Bunches	1.7; 2.3	
		0.414	1796	4/1.656	35	Bunches	1.7; 2.3	
1714/90/GRA T-7544, Hughson, CA, USA, 1990 (Chenin Blanc)	300 g/L	0.224	224	4/0.896	21	Bunches	0.70; 0.92 (0.81)	FR-01-0335
1714/90/GRA T-7545, Fresno, CA, USA, 1990 (Thompson Seedless)	300 g/L	0.224	234	4/0.896	21	Bunches	0.53; 0.53 (0.53)	FR-01-0335 * sampled in field or processing plant ** duplicate analysis of one sample
		0.448	234	4/1.792	21	Bunches* Raisins*	0.75; 0.93 (0.84) 3.3; 3.1	
		0.448	234	4/1.792	21	Bunches * Raisins * Hydrated Raisin Raisin Waste Juice Wet pomace Dry pomace	0.53 3.3, 3.1** 2.4, 2.3** 6.0, 6.0 ** 0.13, 0.12** 1.1, 1.1** 2.0, 2.1**	
V-22939-A 22939/FR-0374, Madera, CA, USA, 2001 (Thompson Seedless)	300 g/L	0.465	935	2/0.964	21	Fruit	1.7; 1.6 (1.7)	FR-0374
		0.499	1010					
V-22939-B 22939/FR-0374, Fresno, CA, USA, 2001 (Thompson	300 g/L	0.460	926	2/0.927	21	Fruit	1.7; 1.5 (1.6)	FR-0374
		0.467	935					

GRAPES	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/l)	kg ai/ha	Water (l/ha)	No./ Total kg ai/ha	days		mg/kg	
Seedless)								
V-22939-B 22939/FR-0374, Fresno, CA, USA, 2001 (Thompson Seedless)	300 g/L	0.924 0.939	926 945	2/1.863 ,	21	Fruit	2.9; 2.7	FR-0374

Assorted Tropical and Subtropical Fruits – Edible Peel

Olives

Three supervised trials were conducted on olives in the USA during 2005 (Samoil, 200; Reference FR-0561). Each trial consisted of one treated and one untreated plot, except CA64, which had two treated plots. At each trial, two applications of fenpropathrin 2.4 EC were made 7 days apart to treated plots at the rate of 0.45 to 0.48 kg ai/ha for a total of 0.9 kg ai/ha. In one plot for trial CA64, two applications at 5× rate (2.3 kg ai/ha for a total of 4.64 kg ai/ha) were made, to provide samples for processing into olive oil.

Samples of mature olive fruits were taken 7–8 and 14–15 days after the last treatment. Pits were removed from the olives. Samples were immediately frozen at -20 °C until analysis in 200 days. The samples were analysed for residues of fenpropathrin using the method “Determination of fenpropathrin in olive, Revision 0”, a method based on method RM-22-4. The method was validated with an LOQ of 0.02 mg/kg for olives. Concurrent recoveries obtained during sample analysis ranged from 69 to 103% for olive fruit without pits. Note that apparent residues at levels of 0.01–0.03 mg/kg were seen in all control samples of olives without pits. The report explained that this should have no adverse effect on the data for treated olives since the residue levels obtained were at least 40× the magnitude of those from the controls.

Table 42 Residues of fenpropathrin in olives resulting from supervised trials in the United States

OLIVES	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./ Total kg ai/ha	days		mg/kg	
GAP, USA	300 EC (2.4 lb ai/gal)	0.22- 0.34	935	2/0.9	7			
CA63 Davis, CA, USA, 2005 (Manzanillo)	300 EC	0.45 0.45	776 786	2/0.9	7	Olive fruit, pitted	2.1, 2.3 (2.2)	FR-0561
					14	Olive fruit, pitted	1.8, 1.6 (1.7)	
CA64 Orange Cove, CA, USA, 2005	300 EC	0.46 0.46	963 945	2/0.92	7	Olive fruit, pitted	1.8, 2.0 (1.9)	FR-0561
					14	Olive fruit,	1.5, 2.3	

OLIVES	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./ Total kg ai/ha	days		mg/kg	
(Manzanillo)						pitted	(1.9)	
CA65 Healdsburg, CA, USA, 2005 (Leccino)	300 EC	0.48	505	2/0.95	8	Olive fruit, pitted	3.7, 3.4 (3.6)	FR-0561
		0.47	496		15	Olive fruit, pitted	1.2, 2.3 (1.8)	

Fruiting Vegetables, Cucurbits

Cucumber

Six supervised trials on cucumber were conducted in the USA in 1994 and 1996 (Samoil, 1999; Reference FR-0556). The EC formulation of fenpropathrin containing 300 g/L active ingredient, was applied 3 to 5 times to treated plots at the rate of 0.21 to 0.24 kg ai/ha for a total application of 0.7 to 1.1 kg ai/ha per plot. Applications were made at weekly intervals. At maturity, 6 to 8 days after the last application, samples were harvested and immediately frozen at -20 °C. Samples were analysed within the storage stability period with RM-22-2 (LOQ of 0.01 mg/kg). Concurrent recoveries from fortified samples analysed together with the field samples ranged from 66–115% (mean = 89%, RSD= 13.8%, n=15).

Table 43 Residues of fenpropathrin in cucumber resulting from supervised trials in the United States

CUCUMBER	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total/ kg ai/ha	days		mg/kg	
GAP, USA	300 EC	0.22- 0.34	187- 935	0.9	7			
94-FL50 Gainesville, FL, USA, 1994 (Comet II)	300 EC	0.22 0.24 0.24	289 283 292	3/0.7	7	Whole fruit	0.05, 0.06, 0.04 (0.05)	FR-0556
94-OH*14 Fremont, OH USA, 1994 (Carolina)	300 EC	0.22 0.22 0.22	631 631 636	3/0.7	8	Whole fruit	< 0.01, < 0.01 (< 0.01)	FR-0556
96-TX*37 Weslaco, TX, USA, 1996 (Napoleon)	300 EC	0.22	281	4/0.9	7	Whole fruit	< 0.01, 0.01 (0.01)	FR-0556
96-GA*26 Tifton, GA, USA, 1996 (Straight Eight)	300 EC	0.22	281	4/0.9	7	Whole fruit	0.01, < 0.01 (0.01)	FR-0556
96-SC*16 Charleston, SC USA, 1996 (Regal)	300 EC	0.22	222	4/0.9	7	Whole fruit	< 0.01, < 0.01 (< 0.01)	FR-0556
96-OH*28	300 EC	0.22	742 (3x)	5/1.1	7	Whole fruit	< 0.01,	FR-0556

CUCUMBER	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total/ kg ai/ha	days		mg/kg	
Fremont, OH USA, 1996 (Score)			757 461				< 0.01 (< 0.01)	
96-MD02 Salisbury, MD USA, 1996 (Thunder)	300 EC	0.22 0.22 0.21 0.21	215 220 212 213	4/0.86	7	Whole fruit	0.03, 0.02 (0.03)	FR-0556
96-IN02 Lafayette, IN USA, 1996 (SMR 58)	300 EC	0.22 0.21 0.22 0.22	315 306 314 314	4/0.87	7	Whole fruit	< 0.01, < 0.01 (< 0.01)	FR-0556

Melon

Ten supervised trials on melons (cantaloupe) were conducted in the USA in 1994 (Green, 1995; Reference FR-0375). The EC formulation of fenpropathrin containing 300 g/L active ingredient, was applied 4 times to treated plots at the rate of 0.22 kg ai/ha for a total application of 0.9 kg ai/ha per plot. Applications were made at weekly intervals using tractor mounted boom sprayers or CO₂-powered backpack sprayers. At maturity, 7 days after the last application, samples were harvested and immediately frozen at -20 °C. In two trials, samples were also collected 1, 3, and 14 days, and on days 3 and 7 after application. In two trials samples of treated melons were sectioned, separating the rind from the flesh. The samples were stored up to 107 days before analysis. Samples were analysed by method RM-22-4, which was validated to an LOQ of 0.01 mg/kg. Concurrent recoveries from fortified samples analysed together with the field samples ranged from 82–98% (mean = 90%, RSD= 4.3%, n=16).

Table 44 Residues of fenpropathrin in melon resulting from supervised trials in the United States

MELON	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		(mg/kg)	
GAP, USA	300 EC	0.22- 0.34	187- 935	0.9	7			
V-10868-B Ripon, CA USA, 1994 (Magnum)	300 EC	0.22 0.45	281- 290	2/0.88 4/1.8	7 7	Melon	0.2, 0.23 (0.22) 0.51, 0.64 (0.58)	FR-0375
V-10868-C Ripon, CA USA, 1994 (Magnum)	300 EC	0.22	281	4/0.88	1 3 7 14	Melon	0.24, 0.40 (0.32) 0.45, 0.35 (0.40) 0.31, 0.23 (0.27) 0.23, 0.25	FR-0375

MELON	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		(mg/kg)	
							(0.24)	
V-10868-D Uvalde, TX USA, 1994 (Explorer)	300 EC	0.22	281	4/0.88	7	Melon	0.10, 0.14 (0.12)	FR-0375
V-10868-E Uvalde, TX USA, 1994 (Explorer)	300 EC	0.22 0.45	271 281	4/0.88 4/1.8	7	Melon	0.14, 0.15 (0.15) 0.24, 0.21 (0.23)	FR-0375
V-10868-F Somerton, AZ USA, 1994 (Top Mark)	300 EC	0.22	281	4/0.88	7	Melon	0.12, 0.12 (0.12)	FR-0375
V-10868-G Fishers, Indiana USA, 1994 (Iroquois)	300 EC	0.22 0.45	131 140	4/0.88 4/1.8	7 7	Melon	0.26, 0.28 (0.27) 0.46, 0.34 (0.40)	FR-0375
V-10868-H Cory, Colorado USA, 1994 (Mission Hybrid)	300 EC	0.22	281	4/0.88	7	Melon	0.08, 0.10 (0.09)	FR-0375
V-10868-I Hawkinsville, GA, USA, 1994 (Schoons)	300 EC	0.22	187	4/0.88	3 7	Melon	0.16, 0.07 (0.12) 0.06, 0.08 (0.07)	FR-0375
V-10868-J Germansville, PA USA, 1994 (Ball 1776)	300 EC	0.25 0.49	187 215	4/1.0 4/2.0	7 7	Whole melon Rind Centre Melon	0.17, 0.23 (0.20) 0.39, 0.36 (0.38) < 0.01, < 0.01 (< 0.01) 0.30, 0.21	FR-0375
V-10868-L Dos Palos, CA USA, 1994 (Iron Horse)	300 EC	0.22	187	4/0.88	7	Melon Rind Centre	0.20, 0.29 (0.25) 0.28, 0.35 (0.32) < 0.01, < 0.01 (< 0.01)	FR-0375

Squash

Seven supervised trials on summer squash were conducted in the US in 1994 and 1996 (Samoil, 1999; Reference FR-0557). The EC formulation of fenpropathrin containing 300 g/L active ingredient, was applied 4 times to treated plots at the rate of 0.2 to 0.25 kg ai/ha for a total application of 0.85 to 0.98 kg ai/ha per plot. Applications were made at weekly intervals. Samples were analysed with

method RM-22-2 (LOQ of 0.01 mg/kg). Concurrent recoveries from fortified samples analysed together with the field samples ranged from 80–116% (mean = 97%, RSD= 11.8%, n=11).

Table 45 Residues of fenpropathrin in squash resulting from supervised trials in the United States

SQUASH	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./ Total kg ai/ha	days		mg/kg	
GAP, USA	300 EC (2.4 lb ai/gal)	0.22- 0.34	187- 935	2/0.9	7			
94-NJ33 Bridgeton, NJ, USA, 1994 (Lemon Drop)	300 EC	0.22	496 - 533	4/0.89	7	Summer squash	0.01, 0.02 (0.02)	FR-0557
94-FL75 Gainesville, FL, USA, 1994 (Dixier Hybrid)	300 EC	0.25 0.25 0.25 0.23	309	4/0.98	7	Summer squash	0.03, 0.04 (0.04)	FR-0557
94-WA*61 Moxee, WA, USA, 1994 (Early Straightneck)	300 EC	0.22	369	4/0.89	8	Summer squash	< 0.01, < 0.01 (< 0.01)	FR-0557
96-OH*10 Fremont, OH, USA, 1996 (Zucchini Select Hybrid)	300 EC	0.21 0.22 0.22 0.20	730 739 739 917	4/0.85	7	Summer squash	< 0.01, < 0.01 (< 0.01)	FR-0557
96-CA26 Fremont, CA, USA, 1996 (Chefini)	300 EC	0.21 0.20 0.23 0.25	243 243 290 243	4/0.89	6	Summer squash	0.01, 0.03 (0.02)	FR-0557
96-MD01 Salisbury, MD, USA, 1996 (Tigress)	300 EC	0.21 0.21 0.22 0.21	421 421 430 421	4/0.85	7	Summer squash	0.04, 0.02 (0.03)	FR-0557
96-IN01 Lafayette, IN, USA, 1996 (Black Beauty)	300 EC	0.22 0.21 0.22 0.21	318 309 318 309	4/0.86	7	Summer squash	< 0.01, < 0.01 (< 0.01)	FR-0557

Fruiting Vegetables other than Cucurbits

Tomato

Nine supervised trials conducted on tomatoes in the USA in 1993 (Green, 1994; Reference FR-41-0360). Treated plots received 4 applications of an EC formulation containing 300 g/L fenpropathrin, at the rate of 0.22 kg ai/ha, for a total application of 0.88 kg ai/ha. Mature tomato samples were

collected 3 days after the last application and for decline trials, samples were also collected 1, 14, and 21 days after the last application. Upon collection, samples were stored at -20 °C until analysis. All samples from this study were analysed within this validated storage interval. Residues of fenpropathrin were determined by method RM-22-4 with modifications. Concurrent recoveries from fortified samples analysed together with the field samples were within the acceptable range of 70 to 120%.

Table 46 Residues of fenpropathrin in tomato resulting from supervised trials in the United States

TOMATO	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		mg/kg	
GAP, USA	300 EC (2.4 lb ai/gal)	0.17- 0.22	234 - 1122	0.9	3			
V-1032-A Stanislaus County, CA, USA, 1992 (UC-82)	300 EC	0.22	327	6/1.34	3	Whole fruit	0.34, 0.61 (0.48)	FR-41-0360
		0.22	327					
		0.22	532					
		0.22	529					
		0.22	527					
		0.22	532					
		1.0	327	6/6.0	3	Whole fruit	2.9, 2.5 (2.7)	
		1.0	327					
		1.0	532					
		1.0	529					
		1.0	527					
		1.0	532					
V-10633-A San Joaquin County, CA, USA, 1993 (Roma VF)	300 EC	0.22	701	4/0.88	3	Whole fruit	0.24, 0.18 (0.21)	FR-41-0360
V-10633-B San Joaquin County, CA, USA, 1993 (UC-82B)	300 EC	0.22	468	4/0.88	3	Whole fruit	0.22, 0.32 (0.27)	FR-41-0360
		0.45	468	4/1.8	3	Whole fruit	0.49, 0.49 (0.49)	FR-41-0360
V-10633-C Merced County, CA, USA, 1993 (Apex 1000)	300 EC	0.22	468	4/0.88	1	Whole fruit	0.38, 0.46 (0.42)	FR-41-0360
					3		0.30, 0.29 (0.30)	
					7		0.28, 0.27 (0.28)	
					14		0.19, 0.24 (0.22)	
V-10633-D Fayette County, OH, USA, 1993 (Heinz 9035))	300 EC	0.22	281	4/0.88	3	Whole fruit	0.64, 0.46 (0.55)	FR-41-0360
V-10633-E Ottawa County, MI, USA, 1993 (Rio Grande)	300 EC	0.22	302	4/0.88	3	Whole fruit	0.04, 0.06 (0.05)	FR-41-0360
		0.22	286					
		0.22	320					
		0.22	296					
		0.45	295	4/1.8	3	Whole fruit	0.21, 0.13 (0.17)	FR-41-0360
	0.45	293						
	0.45	314						
	0.45	290						
V-10633-F	300 EC	0.22	217	4/0.88	3	Whole fruit	0.12, 0.10	FR-41-0360

TOMATO	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		mg/kg	
Hamilton County, IN, USA, 1993 (Beefmaster)		0.22 0.22 0.22	211 238 238				(0.11)	
V-10633-G Hunterdon County, NJ, USA, 1993 (Better Boy)	300 EC	0.22 0.22 0.22 0.22	311 306 306 306	4/0.88	1	Whole fruit	0.21, 0.21 (0.21)	FR-41-0360
					3		0.18, 0.20 (0.19)	
					7		0.14, 0.13 (0.14)	
					14		0.09, 0.08 (0.09)	
V-10633-H Barnwell County, SC, USA, 1993 (Celebrity)	300 EC	0.22	281	4/0.88	3	Whole fruit	0.08, 0.07 (0.08)	FR-41-0360
V-10633-I Hidalgo County, TX, USA, 1993 (Florade)	300 EC	0.22	281	4/0.88	3	Whole fruit	0.15, 0.21 (0.18)	FR-41-0360

Peppers

A total of ten supervised trials on peppers (six on bell and four on non-bell) were conducted in the US in 1996 and 1998 (Samoil, 2001; Reference FR-0558). The application rates corresponded to US GAP (0.22 kg ai/ha with a total application of 0.9 kg ai/ha per season, but samples were taken at 2 and 4 days instead of the 3 day PHI. Samples of mature peppers were immediately frozen at -25 °C to -15 °C until analysis. Samples were analysed for residues of fenpropathrin using a modification of method RM-22-4. All recoveries were conducted concurrently with the analyses of treated samples and storage stability samples. Non-bell peppers fortified with fenpropathrin at 0.02, 0.10, 0.50, and 10 mg/kg showed respective recoveries of 100-130%, 95%, 92%, and 88-104%. Bell peppers were fortified with fenpropathrin at 0.02, 0.10, 0.40, 0.50 and 1.0 mg/kg and recoveries were 100–107%, 97–101%, 94–106% and 98%, respectively.

Table 47 Residues of fenpropathrin in peppers resulting from supervised trials in the United States

PEPPER	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		mg/kg	
GAP, USA	300 EC (2.4 lb ai/gal)	0.17- 0.22	234 - 1122	0.9	3			
BELL PEPPER								
96-CA*09 Spence County, Salinas, CA, USA, 1996 (Cal Wonder)	300 EC	0.22 0.22 0.22 0.22	430 440 440 430	4/0.88	2	Whole fruit	0.37, 0.36 (0.37)	FR-0558

PEPPER	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		mg/kg	
96-CA*10 East Alisal, Salinas, CA, USA, 1996 (Cal Wonder)	300 EC	0.22 0.22 0.22 0.22	440 430 440 439	4/0.88	2	Whole fruit	0.31, 0.36 (<u>0.34</u>)	FR-0558
96-FL12 NW71st St, Gainesville, FL, USA, 1996 (Capistrano)	300 EC	0.22 0.22 0.22 0.22	290 290 281 281	4/0.88	2	Whole fruit	0.64, 0.70 (<u>0.67</u>)	FR-0558
98-GA*10 Goat Rd., Tifton, GA, USA, 1998 (Camelot)	300 EC	0.22 0.22 0.22 0.22	290 281 281 281	4/0.88	2	Whole fruit	0.40, 0.33 (<u>0.37</u>)	FR-0558
98-MD02 Nanticoke Rd, Salisbury, MD, USA, 1998 (King Arthur)	300 EC	0.22 0.22 0.22 0.22	496 505 505 505	4/0.88	3	Whole fruit	0.48, 0.51 (<u>0.50</u>)	FR-0558
98-SC*12 Savannah Hwy, Charleston, SC, USA, 1998 (Keystone Resistance Giant)	300 EC	0.22 0.22 0.22 0.22	374 374 374 393	4/0.88	4	Whole fruit	0.08, 0.11 (<u>0.10</u>)	FR-0558
NON-BELL PEPPER								
96-CA*09 Spence County, Salinas, CA, USA, 1996 (Jalapeno)	300 EC	0.23 0.22 0.22 0.22	430 440 440 430	4/0.89	2	Whole fruit	0.24, 0.23 (<u>0.24</u>)	FR-0558
96-CA*10 East Alisal, Salinas, CA, USA, 1996 (Jalapeno))	300 EC	0.23 0.22 0.22 0.22	440 430 440 439	4/0.89	2	Whole fruit	0.36, 0.43 (<u>0.40</u>)	FR-0558
96-GA*05 Goat Rd., Tifton, GA, USA, 1996 (Mesilla)	300 EC	0.22 0.22 0.22 0.22	281 281 281 281	4/0.88	2	Whole fruit	0.32, 0.29 (<u>0.31</u>)	FR-0558
96-TX*04 Weslaco, TX, USA, 1996 (Sonora Anaheim)	300 EC	0.22 0.22 0.22 0.22	281 281 281 281	4/0.88	2	Whole fruit	0.33, 0.43 (<u>0.38</u>)	FR-0558

Pulses

Soya beans

Eight supervised trials on soya beans were conducted in Brazil in 2010 and 2013 (Tomaz, 2010; Reference: FR-0570 and Lopez, 2013; Reference: FR-0571). The EC formulation of fenpropathrin containing 300 g/L active ingredient, was applied to treated plots at the rate of 0.045 kg ai/ha from 15

to 45 days prior to harvest. At maturity, 15, 30 and 45 days after the last application, samples were harvested, threshed manually, cleaned, sieved, air-dried and stored frozen at -20 °C. Samples were analysed within 30 days of harvest by GC-ECD (2010 trials) and LC-MS/MS (2013 trials) (LOQ of 0.01 mg/kg). Concurrent recoveries were within the acceptable range of 70–120%, with RSD below 20%.

Table 48 Residues of fenpropathrin in soya beans resulting from supervised trials in Brazil

SOYA BEAN	Application			DLA	Sample	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	No.	days		mg/kg	
GAP, Brazil	300 g/L EC	0.045	1	30			
Pereiras/SP Brazil, 2010 (BRS 232)	300 EC	0.045	1	15 30 45	Seeds	< 0.01 <u>< 0.01</u> < 0.01	FR-0570
Iracemópolis/SP Brazil, 2010 (BRS 245)	300 EC	0.045	1	15 30 45	Seeds	< 0.01 <u>< 0.01</u> < 0.01	FR-0570
Cambará/PR Brazil, 2010 (BRS 245)	300 EC	0.045	1	15 30 45	Seeds	< 0.01 <u>< 0.01</u> < 0.01	FR-0570
Santa Mariana/PR Brazil, 2010 (BRS 245)	300 EC	0.045	1	15 30 45	Seeds	< 0.01 <u>< 0.01</u> < 0.01	FR-0570
Trial C01 Ibiporã/PR Brazil, 2012-2013 (Potência)	300 EC	0.045	1	15 30 45	Seeds	< 0.01 <u>< 0.01</u> < 0.01	FR-0571
Trial C53 Câdido Mota/SP Brazil, 2012-2013 (Potência)	300 EC	0.045	1	30	Seeds	<u>< 0.01</u>	FR-0571
Trial C56 Jataizinho/PR Brazil, 2012-2013 (Força)	300 EC	0.045	1	15 30 45	Seeds	< 0.01 <u>< 0.01</u> < 0.01	FR-0571
Trial C109 Dourados/MS Brazil, 2012-2013 (Potência)	300 EC	0.045	1	30	Seeds	<u>< 0.01</u>	FR-0571

Oilseeds

Cotton

Thirty-two supervised trials on cotton were conducted in the USA in 1983-1989, using a higher number of applications compared to the GAP in the USA (up to 0.9 kg ai/ha and a PHI of 21 days) (Fujie, 1990Reference: FR-01-0306). The EC formulation of fenpropathrin containing 300 g/L active ingredient, was applied to treated plots at the rates of 0.112-0.448 kg ai/ha from 18 to 35 days prior to harvest. Samples were analysed within 12–18 months of harvest. Concurrent recoveries from control

samples fortified with fenpropathrin at levels of 0.1 and 0.2 mg/kg were within the acceptable range of 70–120%, with RSD below 20%.

Table 49 Residues of fenpropathrin in cotton fuzzy seed resulting from supervised trials in the USA

COTTON	Application				DAT	Commodity	Residue mg/kg	Reference
Trial Country, year (Variety)	Formulation (g ai/l)	kg ai/ha	Water (l/ha)	No/Total kg ai/ha	days			
GAP, USA	300 EC	0.22- 0.45		0.9	21			
R194 M335-6023 Bard, California, USA, 1983 (DPL-61)	300 EC	0.224	281	7/1.79	35	Fuzzy seed	< 0.01	FR-01-0306
R194 M335-6023 Bard, California, USA, 1983 (DPL-61)	300 EC	0.448	281	8/3.58	35	Fuzzy seed	< 0.01	FR-01-0306
R194 M335-6024 Arizona, USA, 1983 (Delta Pine 70)	300 EC	0.112	117	8/0.896	33	Fuzzy seed	< 0.01, < 0.01 (< 0.01)	FR-01-0306
R194 M335-6069* Yuma, Arizona, USA, 1984 (DPL-90)	300 EC	0.224	47	10/2.24	18	Fuzzy seed	0.01, < 0.01 (< 0.01)	FR-01-0306
R194 M335-6070 Donna, Texas, USA, 1984 (McNair 220)	300 EC	0.224	94	10/2.02	21	Fuzzy seed	0.02, 0.02 (0.02)	FR-01-0306
R194 M335-6071 Kingsville, Texas, USA, 1984 (GP-3774)	300 EC	0.224	23-94	10/2.24	18	Fuzzy seed	0.03, 0.03 (0.03)	FR-01-0306
R194 M335-6072A Bard, California, USA, 1984 (DPL-90)	300 EC	0.224	94	10/2.24	21	Fuzzy seed	< 0.01, < 0.01 (< 0.01)	FR-01-0306
R194 M335-6073 Fresno, California, USA, 1984 (Acala-SJ-5)	300 EC	0.224	430-702	10/2.24	21	Fuzzy seed	0.01, < 0.01 (0.01)	FR-01-0306
R194 M335-6074 Holtville California, USA, 1984 (Delta Pine 61)	300 EC	0.224	234	10/2.46	34	Fuzzy seed	0.33, 0.18 (0.26)	FR-01-0306
R194 M335-6075 Tifton, Georgia, USA, 1984	300 EC	0.224	99	10/2.24	21	Fuzzy seed	< 0.01, < 0.01 (< 0.01)	FR-01-0306

COTTON	Application				DAT	Commodity	Residue mg/kg	Reference
Trial Country, year (Variety)	Formulation (g ai/l)	kg ai/ha	Water (l/ha)	No/Total kg ai/ha	days			
(McNair 220)								
R194 M335-6076 Greenville, Mississippi, USA, 1984 (Stoneville 825)	300 EC	0.224	94	10/2.24	20	Fuzzy seed	0.33, 0.25 (0.29)	FR-01-0306
R194 R194-6418 Fresno, California, USA, 1985 (SJ-5)	300 EC	0.224	281	10/2.24	3	Fuzzy seed	0.12, 0.14 (0.13)	FR-01-0306
					10		0.08, 0.21 (0.15)	
					14		0.03, 0.02 (0.03)	
					21		0.11, 0.13 (0.12)	
					28		< 0.01, < 0.01 (< 0.01)	
					35		< 0.01, < 0.01 (< 0.01)	
R194 R194-6419 Blythe, California, USA, 1985 (SJ-5)	300 EC	0.112	281	10/1.12	21	Fuzzy seed	< 0.01, 0.03 (0.02)	FR-01-0306
R194 R194-6419 Blythe, California, USA, 1985 (DPL-90)	300 EC	0.224	281	10/2.24	21	Fuzzy seed	0.01, < 0.01 (0.01)	FR-01-0306
R194 R194-6419 Blythe, California, USA, 1985 (DPL-90)	300 EC	0.448	281	10/4.48	21	Fuzzy seed	< 0.01, 0.06 (0.04)	FR-01-0306
R194 R194-6420* Maricopa, Arizona, USA, 1985 (Pima S-2)	300 EC	0.224	94	10/2.24	21	Fuzzy seed	0.02, 0.02 (0.02)	FR-01-0306
R194 R194-6421* Yuma, Arizona, USA, 1985 (DPL-90)	300 EC	0.224	47	10/2.24	21	Fuzzy seed	< 0.01, < 0.01 (< 0.01)	FR-01-0306
R194 R194-6422 College station, Texas USA, 1985 (DPL-41)	300 EC	0.224	42	10/2.24	21	Fuzzy seed	0.09, 0.07 (0.08)	FR-01-0306
R194 R194-6423 Kingsville, Texas, USA, 1985 (SP-37)	300 EC	0.224	47	10/2.24	3	Fuzzy seed	3.2, 3.4 (3.3)	FR-01-0306
					7		0.95, 0.56 (0.76)	
					14		0.19, 0.21 (0.20)	

COTTON	Application				DAT	Commodity	Residue mg/kg	Reference
Trial Country, year (Variety)	Formulation (g ai/l)	kg ai/ha	Water (l/ha)	No/Total kg ai/ha	days			
					21		0.02, 0.02 (0.02)	
					28		< 0.01, 0.02 (0.02)	
					35		< 0.01, < 0.01 (< 0.01)	
R194 R194-6424 Donna, Texas USA, 1985 (McNair 220)	300 EC	0.224	94	10/2.24	21	Fuzzy seed	< 0.01, < 0.01 (< 0.01)	FR-01-0306
R194 R194-6425 Tifton Georgia USA, 1985 (Coker 304)	300 EC	0.224	94	10/2.24	22	Fuzzy seed	< 0.01, < 0.01 (< 0.01)	FR-01-0306
R194 R194-6426 ArkansasUSA, 1985 (Stoneville 825)	300 EC	0.224	47	10/2.24	30	Fuzzy seed	0.02, < 0.01 (0.02)	FR-01-0306
R194 R194-6427 Greenville Mississippi USA, 1985 (DPL-41)	300 EC	0.112	94	10/1.12	21	Fuzzy seed	0.08, 0.09 (0.09)	FR-01-0306
R194 R194-6427 Greenville Mississippi USA, 1985 (DPL-41)	300 EC	0.224	94	10/2.24	3	Fuzzy seed	0.56, 0.47 (0.52)	FR-01-0306
					14		0.47, 0.46 (0.47)	
					21		0.32, 0.40 (0.36)	
					36		0.30, 0.31 (0.31)	
R194 R194-6427 Greenville Mississippi USA, 1985 (DPL-41)	300 EC	0.448	94	10/4.48	7	Fuzzy seed	1.2	FR-01-0306
R194 R194-6715 Greenville Mississippi USA, 1986 (Acala SJ-5)	300 EC	0.224	281	8/1.792	21	Fuzzy seed	1.3, 1.1, 0.95, 0.77 (1.03)	FR-01-0306
R194 R194-6716 Case Grande, Arizona USA, 1986 (DPL-41)	300 EC	0.224	281	8/1.792	20	Fuzzy seed	0.59, 0.62 , 0.47, 0.43 (0.53)	FR-01-0306
R194 R194-6717 Greenville, Mississippi	300 EC	0.224	94	8/1.792	21	Fuzzy seed	0.08, 0.05 (0.07)	FR-01-0306

COTTON	Application				DAT	Commodity	Residue mg/kg	Reference
Trial Country, year (Variety)	Formulation (g ai/l)	kg ai/ha	Water (l/ha)	No/Total kg ai/ha	days			
USA, 1986 (Delta Pine 90)								
R194 R194-6718 Kleberg County, Texas USA, 1986 (GP 3774)	300 EC	0.224	47	8/1.792	21	Fuzzy seed	0.08, 0.06 (0.07)	FR-01-0306
R194 R194-6967Fresno, California USA, 1987 (GC 510)	300 EC	0.224	19	8/1.792	21	Fuzzy seed	0.22, 0.32 (0.27)	FR-01-0306
R194 R194-6968 Glen Alan, Mississippi USA, 1987 (DPL-50)	300 EC	0.224	19	8/1.792	21	Fuzzy seed	< 0.01, < 0.01 (< 0.01)	FR-01-0306
R194 R194-7376 Greenville, Mississippi USA, 1989 (DPL-41)	300 EC	0.336	47	5/1.68	21	Fuzzy seed	0.02, 0.02 (0.02)	FR-01-0306
R194 R194-7377 Grangerburg, Alabama USA, 1989 (McNair 220)	300 EC	0.336	468	5/1.68	21	Fuzzy seed	0.01, 0.01 (0.01)	FR-01-0306
R194 R194-7378 Rosa, Louisiana USA, 1989 (DPL-41)	300 EC	0.336	468	5/1.68	20	Fuzzy seed	0.29, 0.27 (0.28)	FR-01-0306
R194 R194-7379 Proctor, Arkansas USA, 1989 (DPL-41)	300 EC	0.336	468	5/1.68	21	Fuzzy seed	0.04, 0.07 (0.06)	FR-01-0306
R194 R194-7380 Fresno, California USA, 1989 (GC 510)	300 EC	0.336	47	5/1.68	21	Fuzzy seed	0.07, 0.05 (0.06)	FR-01-0306
R194 R194-7381* Greenville, Mississippi USA, 1989 (DPL-41)	300 EC	0.336	9	5/1.68	21	Fuzzy seed	0.03, 0.02 (0.03)	FR-01-0306
R194 Eakly, Oklahoma USA, 1989 (Paymater 145)	300 EC	0.336	9	5/1.68	21	Fuzzy seed	0.03, 0.04 (0.04)	FR-01-0306

*Tree Nuts**Almonds and pecans*

A total of ten supervised trials on tree nuts, five on almonds and five on pecans have been conducted in the US in 2003, following the GAP for tree nuts (Green, 2004; References FR-0382 and FR-0383). Two applications of fenpropathrin 2.4 EC were made to each treated plot at the rate of 0.45 (1×) or 0.90 kg ai/ha (2×). Applications were made at 7-day intervals. Samples of almond hulls as well as almond and pecan nutmeats were collected 3 days after the last application. At one site, samples were also collected at 1, 7, and 14 days after the last application.

All samples were immediately frozen and stored at -20 °C until analysis. Residues of fenpropathrin in nutmeats were analysed using method RM-22-4. The method was modified for analysis of a 10 gram (instead of a 20 gram) sample. The LOQ remained 0.01 mg/kg. Recoveries obtained for the fortified almond and pecan nutmeat samples were between 83.5 and 119%, and 70.9 and 89.6%, respectively.

Table 50 Residues of fenpropathrin in tree nuts resulting from supervised trials in the United States

TREE NUTS	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		mg/kg	
GAP, USA	300 EC (2.4 lb ai/gal)	0.22- 0.45	468 - 3740	0.9	3			
ALMONDS								
V-25398-A Glen County, CA, USA, 2003 (Non-Pareil)	300 EC	0.45 0.45	935 1038	2/0.9	3	Nutmeat	< 0.01, < 0.01 (<u>< 0.01</u>)	FR-0382
	300 EC	0.89 0.90	935 1029	2/1.8	3	Nutmeat	0.02, 0.02 (0.02)	
V-25398-B Stanislaus County, CA, USA, 2003 (Carmel)	300 EC	0.45 0.45	1160 1132	2/0.9	3	Nutmeat	< 0.01, < 0.01 (<u>< 0.01</u>)	FR-0382
V-25398-C Kern County, CA, USA, 2003 (Sornora)	300 EC	0.45 0.46	945 963	2/0.91	3	Nutmeat	0.02, 0.03 (<u>0.03</u>)	FR-0382
V-25398-D Fresno County, CA, USA, 2003 (Non-Pareil)	300 EC	0.46 0.45	945 935	2/0.91	1	Nutmeat	0.01, < 0.01 (<u>< 0.01</u>)	FR-0382
					3	Nutmeat	0.01, < 0.01 (<u>< 0.01</u>)	
					7	Nutmeat	< 0.01, < 0.01 (<u>< 0.01</u>)	
					14	Nutmeat	< 0.01, < 0.01 (<u>< 0.01</u>)	
V-25398-E Tulare County, CA, USA, 2003 (Carmel)	300 EC	0.45 0.45	945 945	2/0.9	3	Nutmeat	< 0.01, < 0.01 (<u>< 0.01</u>)	FR-0382
PECANS								
V-25401-A	300 EC	0.44	1160	2/0.87	3	Nutmeat	< 0.01,	FR-0383

TREE NUTS	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		mg/kg	
Saluda County, SC, USA, 2003 (Cape Fear)		0.43	1132				< 0.01 (<u>< 0.01</u>)	
		0.90 0.90	1169 1160	2/1.8	3	Nutmeat	< 0.01, < 0.01 (<u>< 0.01</u>)	
V-25401-B Tift, GA, USA, 2003 (Summer)	300 EC	0.45 0.45	1188 1197	2/0.9	3	Nutmeat	0.01, < 0.01 (<u>0.01</u>)	FR-0383
V-25401-C Drew County, AR, USA, 2003 (Stewart)	300 EC	0.46 0.46	1169 1188	2/0.92	3	Nutmeat	0.02, 0.02 (<u>0.02</u>)	FR-0383
V-25401-D Burleson County, TX, USA, 2003 (Cheyenne)	300 EC	0.43 0.44	1394 1394	2/0.87	1	Nutmeat	0.02, 0.05 (0.04)	FR-0383
					3	Nutmeat	0.02, 0.02 (0.02)	
					7	Nutmeat	0.02, 0.10 (<u>0.06</u>)	
					14	Nutmeat	< 0.01, < 0.01 (<u>< 0.01</u>)	
V-25401-E Hockley County, TX, USA, 2003 (Western Schley)	300 EC	0.46 0.45	1197 1188	2/0.91	3	Nutmeat	0.04, 0.05 (<u>0.05</u>)	FR-0383

Coffee Beans

Six supervised trials on coffee were conducted in Brazil in 2013 (Gravena, 2013. Reference FR-0572). Two applications of fenpropathrin EC containing 300 g ai/L were made to each treated plot at the rate of 0.12 kg ai/ha (1×). Applications were made at 14-day intervals. Samples of coffee berries were collected 14 days after the last application. In two trials, samples were also collected at 7 and 21 days after the last application. All samples were collected manually and packed in polyethylene bags for further processing. The berries were immediately sent to the experimental station and maintained at room temperature until completion of drying. Samples were dried by opening the bags throughout the day and night, except during rainy days when the packages were sealed. After drying, the coffee beans were placed in a manual peeler and at least 1 kg of coffee beans were packed, labelled, and stored frozen at -20 °C until analysis.

All samples were analysed LC-with LC-MS/MS (LOQ of 0.01 mg/kg) within 30 days following harvest. Average recovery from control samples fortified with fenpropathrin at levels of 0.01 and 0.5 mg/kg ranged from 79–116% (n=13; mean=98%; and RSD = 15.3%).

Table 51 Residues of fenpropathrin in coffee beans resulting from supervised trials in Brazil

COFFEE	Application	DAT	Sample	Residue	Reference

Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No.	days		mg/kg	
GAP, Brazil	300 g/L EC	0.12		2	14			
Trial 01 Irai de Minas/MG Brazil, 2012 (IAPAR 59)	300 g/L EC	0.12	301 303	2	7 14 21	Coffee beans	< 0.01 <u>0.01</u> < 0.01	FR-0572
Trial 02 Indianópolis, MG Brazil, 2012 (Catuai Amarelo)	300 g/L EC	0.12	308 294	2	14	Coffee beans	< 0.01	FR-0572
Trial 03 Rolândia/PR Brazil, 2012 (Novo Mundo)	300 g/L EC	0.12	285 297	2	7 14 21	Coffee beans	< 0.01 <u>< 0.01</u> < 0.01	FR-0572
Trial 04 Bebedouro/SP Brazil, 2012 (Novo Mundo)	300 g/L EC	0.12	298 304	2	14	Coffee beans	< 0.01	FR-0572
Trial 05 Monte Azul Paulista/SP Brazil, 2012 (Novo Mundo)	300 g/L EC	0.12	293 308	2	14	Coffee beans	< 0.01	FR-0572
Trial 06 Araguari/MG Brazil, 2012 (Novo Mundo)	300 g/L EC	0.12	302 303	2	14	Coffee beans	<u>0.02</u>	FR-0572

Tea

All supervised trials on tea were conducted in India during 2002-2004 (Lavakumar, 2003 and 2006; References 1424604-2974-2002; 1424604-4190-2004). All trials were according to the GAP for tea in India, which consists of one application of fenpropathrin at the rate of 0.06 kg ai/ha and a PHI of 7 days.

In ten trials, the collected tea leaves were processed into black tea. In six trials this was achieved through withering, crush/tear/curl process, oxidation and drying while in other trials by machine drying.

In two additional trials, the collected tea leaves were air-dried to prepare green tea. Since growing conditions and application rate/method for black tea and green tea are equivalent with the only difference being in processing methods, results from both sets of trials can be combined to support an MRL and STMR for tea, green and black.

Residues of fenpropathrin were analysed by a method based on RM-22-4 with an LOQ of 0.05 mg/kg.

Table 52 Fenpropathrin residues in black and green tea from supervised trials in India

TEA	Application	DAT	Residues1	Reference

Country, month/year, season (variety)	Form	kg ai/ha	Water l/ha	No.	days	mg/kg	
GAP in India	300 g/L EC	0.05 – 0.06	200 – 400	1	7		
BLACK TEA							
Valparai, India January 2002 Season I (UPASI-9)	300 g/L EC	0.06	400	1	0 1 3 5 7 10 14	2.74 1.75 1.17 0.61 <u>0.17</u> < 0.05 < 0.05	04-2974-2002
Valparai, India September 2002 Season II (UPASI-9)	300 g/L EC	0.06	400	1	0 1 3 5 7 10 14	2.69 1.69 1.10 0.61 <u>0.18</u> < 0.05 < 0.05	04-2974-2002
Valparai, India May 2003 Season III (UPASI-9)	300 g/L EC	0.06	400	1	0 1 3 5 7 10 14	2.22 1.45 0.91 0.49 <u>0.14</u> < 0.05 < 0.05	04-2974-2002
Valparai, India January 2002 Season I (UPASI-9)	300 g/L EC	0.12	400	1	0 1 3 5 7 10 14	5.47 3.13 2.24 1.04 0.37* < 0.05 < 0.05	04-2974-2002
Valparai, India September 2002 Season II (UPASI-9)	300 g/L EC	0.12	400	1	0 1 3 5 7 10 14	5.24 3.02 2.22 1.08 0.36* < 0.05 < 0.05	04-2974-2002
Valparai, India May 2003 Season III (UPASI-9)	300 g/L EC	0.12	400	1	0 1 3 5 7 10 14	4.40 2.57 1.89 0.86 0.30* < 0.05 < 0.05	04-2974-2002
Valparai, India January 2004 Fourth Season (UPASI-9)	300 g/L EC	0.06	400	1	0 1 3 5 7 10 14	0.85 0.50 0.17 < 0.05 <u>< 0.05</u> < 0.05 < 0.05	04-2974-2002
Valparai, India January 2004 Fourth Season (UPASI-9)	300 EC	0.12	400	1	0 1 3 5 7 10 14	1.62 0.93 0.30 < 0.05 < 0.05 < 0.05 < 0.05	04-2974-2002
Gudalur, India June 2004 (Mixed clones)	300 EC	0.06	450	1	0 7 10 14	2.22 <u>0.14</u> < 0.05 < 0.05	Submission by Government of India to JMPR 1993

TEA	Application				DAT	Residues ¹	Reference
Country, month/year, season (variety)	Form	kg ai/ha	Water l/ha	No.	days	mg/kg	
Tocklai, India November 2005 (Mixed clones)	300 EC	0.06	400	1	0 7 10	12.0 <u>1.38</u> 0.12	Submission by Government of India to JMPR 1993
GREEN TEA							
Valparai, India January 2004 Fourth Season (UPASI-9)	300 g/L EC	0.06	400	1	0 1 3 5 7 10 14	1.96 1.32 0.83 0.45 <u>0.13</u> < 0.05 < 0.05	04-4190-2004
Valparai, India January 2004 Fourth Season (UPASI-9)	300 g/L EC	0.12	400	1	0 1 3 5 7 10 14	4.20 2.43 1.55 0.90 0.29* < 0.05 < 0.05	04-4190-2004

Animal feeds

The trial conditions are described under the corresponding food commodities.

Cottonseed hull

Samples were analysed within 12–18 months of harvest. Concurrent recoveries from control samples fortified with fenpropathrin at levels of 0.1 and 0.2 mg/kg were within the acceptable range of 70–120%, with RSD below 20%.

Table 53 Residues of fenpropathrin in cottonseed hulls resulting from supervised trials in the USA

COTTON	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (l/ha)	No/Total kg ai/ha	days		mg/kg	
GAP, USA	300 EC	0.22-0.45		0.9	21			
R194 M335-6070 Donna, Texas, USA, 1984 (McNair 220)	300 EC	0.224	94	9/2.02	21	Hulls	0.02, 0.02 (0.02)	FR-01-0306
R194 M335-6071 Kingsville, Texas, USA, 1984 (GP-3774)	300 EC	0.224	94	10/2.24	18	Hulls	0.03, 0.02 (0.03)	FR-01-0306
R194 R194-6427 Greenville Mississippi USA, 1985	300 EC	0.448	94	110/4.48	7	Hulls	1.0	FR-01-0306

COTTON	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (l/ha)	No/Total kg ai/ha	days		mg/kg	
(DPL-41)								

Almond hulls

All samples were immediately frozen and stored at -20 °C until analysis. Residues of fenpropathrin in hulls were analysed using method RM-22-4. The method was modified for analysis of a 10 gram (instead of a 20 gram) sample. The LOQ remained 0.01 mg/kg. Recoveries obtained for the fortified almond hull samples were between 90.3 and 115%, with an average recovery of 103%.

Table 54 Residues of fenpropathrin in almond hulls resulting from supervised trials in the USA

TREE NUTS	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		mg/kg	
GAP, USA	300 EC (2.4 lb ai/gal)	0.22- 0.45	468 - 3740	3/0.9	3			
V-25398-A Glen County, CA, USA, 2003 (Non-Pareil)	300 EC	0.45 0.45	935 1038	2/0.9	3	Hulls	2.4, 2.9 (<u>2.7</u>)	FR-0382
	300 EC	0.89 0.90	935 1029	2/1.8	3	Hulls	7.2, 7.3 (7.3)	
V-25398-B Stanislaus County, CA, USA, 2003 (Carmel)	300 EC	0.45 0.45	1160 1132	2/0.9	3	Hulls	2.7, 3.1 (<u>2.9</u>)	FR-0382
V-25398-C Kern County, CA, USA, 2003 (Sornora)	300 EC	0.45 0.46	945 963	2/0.91	3	Hulls	3.2, 2.9 (<u>3.1</u>)	FR-0382
V-25398-D Fresno County, CA, USA, 2003 (Non-Pareil)	300 EC	0.46 0.45	945 935	2/0.91	1	Hulls	4.7, 3.2 (3.9)	FR-0382
					3	Hulls	3.5, 3.4 (<u>3.5</u>)	
					7	Hulls	4.3, 2.7 (3.5)	
					14	Hulls	2.1, 4.5 (3.3)	
V-25398-E Tulare County, CA, USA, 2003 (Carmel)	300 EC	0.45 0.45	945 945	2/0.9	3	Hulls	4.3, 2.9 (<u>3.6</u>)	FR-0382

FATE OF RESIDUES IN STORAGE AND IN PROCESS

Processing studies on plums, tomato, olives, oranges, cottonseed and tea were conducted and are summarized in this section.

Processing orange into juice, oil, and other fractions

Orange samples from the following supervised trials treated at 1× or 2× the GAP application rate were sent for processing after harvest: Trials T-6389, T-6390, T-6640, T-6730, and T-6966 (Reference FR-01-0307). The study determined the magnitude of residues in raw unwashed and washed orange samples, peel, pulp, wet and dried peel, molasses, oil and juice.

Duplicate samples were harvested 1–7 days after the last application, and sent to the processor. Samples were stored frozen for up to 9 months before analysis by GC-ECD (method RM-22-4). Orange oil samples were extracted into hexane, partitioned with acetonitrile and extracts purified through alumina oxide (method RM-22-2).

Samples were variously washed by hand, separated into pulp and peel, and processed into wet and dried peel, molasses, oil and juice. Fruit was washed in an in-line foam washer with soap and rinsed with water. Juice was extracted using an automated FMC in-line extractor and the sample passed through a juice finisher with 200 mesh, the juice being collected in a holding tank.

The resulting oil, water, peel, frit emulsion from the FMC extractor was passed through an in-line FMC finisher with 200 mesh screen and collected in a holding tank. This emulsion was heated to 49°C and appropriate enzymes added. The emulsion was then centrifuged to separate out the oil. The peel, rag, frit and seeds (together called as peel) from the juice and oil finishers were deposited in a peel hopper. This was further processed by being shredded and combined with 0.3% dehydrated lime slurry.

The slurry was then pressed (4.82 bar) and molasses collected from the press. The remaining pulp from the press was dried to approximately 8% moisture content and passed through a cyclone separator and cooler and collected. Dry fines were collected from the bottom of the cyclone separator. Procedural recoveries were 77-115% (mean 95% ± 7% RSD, n=57) at fortification levels of 0.1–2.0 mg/kg.

Table 55 Residues of fenpropathrin in orange processed fractions

Processed Fractions	Residues (mg/kg)	Average (mg/kg)	Processing factor	Reference (Trial No.)
Orange RAC	0.43, 0.51	0.47		R195 T-6390
Juice		< 0.01	< 0.02	
Oil		37	79	
Molasses		< 0.01	< 0.02	
Wet peel		0.27	0.57	
Dried peel		0.74	1.6	
Dried fines of peel		0.66	1.4	
Orange (RAC)	0.04, 0.05	0.045		R195 T-6966
Washed fruit	0.02, 0.03	0.025	0.6	
Juice	< 0.01, < 0.01	< 0.01	< 0.2	
Oil	0.99, 0.94	0.97	22	
Molasses	0.01, 0.01	0.01	0.2	
Wet peel	0.04, 0.03	0.035	0.8	
Dried peel	0.13, 0.11	0.12	2.7	
Dried fines of peel	0.16, 0.16	0.16	3.6	
Orange (RAC)	0.22, 0.11	0.17		R195 T-6604

Processed Fractions	Residues (mg/kg)	Average (mg/kg)	Processing factor	Reference (Trial No.)
Pulp		< 0.01	< 0.06	
Peel		0.47	2.8	
Orange (RAC)	0.18, 0.18	0.18		R195 T-6730
Washed fruit		0.13	0.72	
Orange (RAC)	0.09, 0.19	0.14		R195 T-6389
Pulp		< 0.01	< 0.07	
Peel		0.40	2.9	

Processing plums to dried plums/prunes

Plums from trial V-25427-F [treated twice with 1.8 kg ai/ha (2x) per application] were processed to dried plums (Reference FR-0385) by drying in a dehydrator at 187°C for approximately 18 hours. After drying, samples were frozen and analysed within 176 days of harvest of fresh plums.

In another study, fresh plum samples from Trial V-27220-A treated at the GAP rate of 0.9 kg ai/ha were dried in a drying tunnel at about 185 °C (Reference FR-0559) for approximately 18 hours. The dried prunes were pitted by hand, bagged and frozen at -20 °C until analysis within 195 days from harvest of fresh samples. The period of storage was within the demonstrated stability of plums under frozen conditions.

All samples were analysed for fenpropathrin using a modification of method RM-22-4 which is described under analytical methods. Concurrent recoveries of fortified samples ranged from 70–120%.

Table 56 Residues of fenpropathrin in fresh and dried plums

Matrix	Residues (mg/kg)	Average (mg/kg)	Processing factor	Reference (Trial No.)
Plum (RAC)	0.61, 0.54	0.58		FR-0385
Dried plum	0.59, 0.59	0.59	1.0	
Plum (RAC) – prior to processing	0.22, 0.23	0.23		FR-0559
Dried plum	0.58, 0.57	0.58	2.6	
Average			1.8	

Processing tomatoes to pomace, juice, paste and other fractions

The magnitude of residues in raw unwashed and washed tomatoes, canned and pre-canned tomatoes, wet and dry pomace and juice was determined following applications of a 2.4 EC at 1.5× GAP rate (Reference FR-21-0344). Duplicate samples of mature green fruit were sent to the processor. Certain samples were gas-ripened or vine ripened before being sampled for analysis. Samples were stored at -20 °C for up to 5 months before analysis by GC-ECD (method RM-22-4), as described above in section 3. Gas ripened fruit was held in an atmosphere of 0.008–0.015%v/v ethylene at 20 °C for 3–6 days before being sampled. Samples were variously washed in chlorine solution, waxed and/or processed into pre-canned and canned tomatoes, wet and dry pomace, juice and paste. The canning process involved washing in chlorine solution followed by a water rinse, peeling by placing in 20%

sodium hydroxide (lye) solution at 79 °C, rinsing in water, dipping in 1% citric acid solution and further rinsing. Once tomatoes were placed in the can hot (100°C) tomato juice was added and the can was sealed and heated for 55 minutes at 100 °C. Procedural recoveries were 83–112% (with the exception of one sample of green fruit at 130%) at fortification levels of 0.01–0.2 mg/kg.

In another study, the magnitude of residues in raw tomatoes, wet and dry pomace, juice and paste were determined following applications of a 2.4 EC at 7.5× GAP rates to commercial crops (Reference FR-41-0360). Duplicate samples of mature green fruit were harvested 3 days after the last application and sent to the processor. Samples were stored at -20 °C and were analysed within 5 months by GC-ECD (method RM-22-4). Samples were hand sorted and defective fruit were discarded as cannery waste. The tomatoes were then soaked in a 0.5% lye solution for 3 minutes at 54 °C and rinsed using a high pressure spray for 30 seconds. Samples were then batch steam peeled at 5.6–7 bar pressure and processed through a pulp finisher machine where the pulp/pomace was separated from juice. The wet pomace was dried to produce dry pomace and the juice concentrated in a batch vacuum concentrator to approximately 30–32% solids. 1% salt was added to the resulting paste and the temperature was raised to 90 °C before the paste was sealed in cans. The cans were subject to heating at 100 °C for 20 minutes in a water batch before being cooled under running tap water. Juice was reconstituted from the paste with water, salt and ascorbic acid, heated to 90 °C, canned, and the cans treated as for canned paste.

Procedural recoveries were 94–105% (with the exception of two samples of dry pomace at 78 and 118%) at fortification levels of 0.1 mg/kg.

Table 57 Residues of fenpropathrin in tomato processed fractions

Processed Fractions	Residues (mg/kg)	Average (mg/kg)	Processing factor	Reference (Trial No.)
Tomato gas ripe	0.07, 0.07	0.07		FR-21-0344
Washed fruit	0.03, 0.04	0.035	0.5	
Unwashed, pre-canned (RAC)		0.13		
Washed, pre-canned		0.07	0.54	
Pre-canning		< 0.01	< 0.08	
Canned		< 0.01	< 0.08	
Canning waste	0.95, 1.1	1.0	7.7	
Tomato gas ripe	0.12, 0.06	0.09		FR-21-0344
Washed fruit	0.07, 0.11	0.09	1	
Raw tomato (RAC)		0.14		
Washed, pre-canned		0.12	0.86	
Processed, pre-canned		0.01	0.07	
Canned		< 0.01	< 0.07	
Canning waste	0.83, 0.81	0.82	5.9	
Tomato gas ripe	0.05, 0.13	0.09		FR-21-0344
Washed gas ripe	0.04, 0.08	0.06	0.7	
Raw tomato (RAC)		0.13		
Washed fruit		0.14	1.1	
Processed pre-canned		0.02	0.15	
Canned		< 0.01	< 0.08	
Canning waste	1.2, 1.3	1.25	9.6	
Tomato gas ripe (RAC)	0.13, 0.03	0.08		FR-21-0344
Washed and waxed	0.05, 0.06	0.055	0.69	

Processed Fractions	Residues (mg/kg)	Average (mg/kg)	Processing factor	Reference (Trial No.)
Tomato gas ripe (RAC)	0.20, 0.10	0.15		FR-21-0344
Washed and waxed	0.08, 0.18	0.13	0.87	
Ripe fruit (RAC)	2.5, 2.9	2.7		FR-41-0360
Fruit from processor	1, 1.1	1.05	0.4	
Wet pomace	9.7, 11	10.4	3.9	
Dry pomace	51, 45	48	18	
Tomato paste	0.83, 0.81	0.82	0.3	
Tomato juice	0.12, 0.13	0.125	0.05	
OVERALL AVERAGE PROCESSING FACTORS				
Tomato gas ripe (RAC)				
Washed and waxed			0.78	
Washed gas ripe			0.73	
Washed, pre-canned			0.83	
Pre-canned			0.1	
Canned			0.08	
Canning waste			12	
Tomato (RAC)				
Wet pomace			3.9	
Dry pomace			18	
Tomato paste			0.3	
Tomato juice			0.05	
Tomato, canned			0.08	

Processing olive fruit to oil

Samples for processing to olive oil were obtained from trial CA64 where olive plants were treated with fenpropathrin at approximately 5× the GAP application rate (Samoil, 2007; Reference FR-0561). After harvest, the fruits were pitted and delivered at ambient temperature to the processing facility, where the olives were processed into oil. The samples were cleaned of extraneous materials and then subjected to cold water wash and crushed. After crushing, the olives were pressed by hydraulic press to produce olive oil, which was then subjected to bleaching. Processed olive oil was stored at -20 °C until analysis. The storage period was recorded as 167 days. Storage stability samples fortified with 0.2 mg/kg fenpropathrin were stored under the same conditions as the field samples and were analysed after 202 days. Recoveries ranged from 85–93% for olive oil.

Residues of fenpropathrin were determined by a method based on RM-22-4. The method was validated using store-bought oil. Recoveries ranged from 94–103%. The LOQ for olive oil was determined to be 0.02 mg/kg.

Table 58 Residues of fenpropathrin in olive oil

Processed Fractions	Residues (mg/kg)	Processing factor	Reference (Trial No.)
Olive fruit (RAC)	14.63		Trial CA64
Olive oil	15.60	1.07	(FR-0561)

Processing cottonseed to oil, meal and soapstock

Samples for processing to cottonseed oil into meal, oil and soapstock were obtained from trials M335-6070, M355-6071 and R194-6427 where cotton plants were treated with fenpropathrin using a range of application rates and timings (Fujie, 1990; Reference FR-01-0360). A Carver impact huller was used to obtain the fractions (kernels and hulls). The kernels were flaked in a Ferrell-Ross "flake-n-roll" to 0.02 mm thickness. The flakes were washed three times with hexane at a temperature of approximately 145 °C. This extraction process took 3 hours. The oil was recovered with a precision laboratory evaporator. During this process the oil reached a maximum temperature of 75 °C. Warm air was forced through the extractor to dissolve the cotton seed flakes. The oil was refined by the following steps:

1. NaOH was added to the oil while it was stirred at 250 rpm at a temperature of 20–24 °C for 15 minutes.
2. The oil was heated to 63–67 °C for 12 minutes and the stirring reduced to 70 RPM.
3. The oil was then allowed to settle for 60 minutes at a temperature of 60–65 °C.
4. The oil was refrigerated overnight or at least for 12 hours.
5. After refrigeration the oil was filtered to obtain the refined oil and soapstock fractions.

The processed samples were stored at -20 °C until analysis. Recoveries ranged from 78–99% for processed products.

Fenpropathrin residues were determined by gas chromatography using an electron capture detector (Fujie, 1990; Reference FR-01-0306). Recoveries ranged from 94–103%. The LOQ for the method was 0.01 mg/kg. Table 59 summarizes the results of the cottonseed processing study.

Table 59 Residues of fenpropathrin in cottonseed products

Processed Fractions	Residues (mg/kg)					Processing factors						Mean processing factor	Reference (Trial No.)
	M335-6070		M335-6071		R194-6427								
Cottonseed (RAC)	0.02	0.02	0.03	0.03	1.2								(FR-01-0306)
Meal	0.01	0.01	0.01	0.03	0.09	0.5	0.5	0.33	1.0	0.08	0.5		
Crude oil	0.06	0.06	0.07	0.06	2.3	3.0	3.0	2.3	2.0	1.9	2.4		
Refined oil	0.06	0.06	0.10	0.07	2.6	3.0	3.0	3.3	2.3	2.2	2.8		
Soapstock	0.01	0.01	0.04	0.05	1.6	0.5	0.5	1.3	1.7	1.3	1.1		

The processing factors are summarised in table 60.

Table 60 Summary of results of processing

RAC/processed fraction	Residues					Fenpropathrin					
						Processing factors					PF, best estimate
RAC:Whole orange	0.47	0.045	0.17	0.18	0.14	-					
Juice	< 0.01	< 0.01				< 0.02	< 0.22				< 0.02
Oil	37	0.97				78.7	21.56				50.1

RAC/processed fraction	Residues					Fenpropathrin					
						Processing factors					PF, best estimate
Wet peel	0.27	0.035	0.47		0.4	0.6	0.78	2.76		2.86	2.82
Dried peel	0.74	0.12				1.6	2.67				2.1
Dried fines of peel	0.66	0.16				1.4	3.56				2.5
Pulp			< 0.01		< 0.01			0.06		0.07	0.065
RAC: Plum	0.58	0.23									
Dried plum	0.59	0.28				1.0	1.2				2.6
RAC: Tomato	0.07	0.09	0.09	1.05	0.4						
Washed fruit	0.035	0.09	0.06			0.50	1.0	0.67	0.00		0.67
Pre-canned RAC	0.13	0.14	0.13								
Washed pre-canned	0.07	0.12	0.14			0.54	0.86	1.08			0.86
Canned	< 0.01	< 0.01	< 0.01			0.077	0.071	0.077			< 0.075
Canning waste	1.0	0.82	1.25			8	6	10			8
Wet pomace				10.4	3.9				9.9	9.8	9.8
Dry pomace				48	18				46	45.0	45
Tomato paste				0.82	0.3				0.78	0.75	0.77
Tomato juice				0.125	0.05				0.12	0.1	0.12
RAC: Tomato	0.08	0.15									
Washed and waxed	0.055	0.13									0.78
RAC: olives	14.63										
Olive oil, bleached	15.6					1.1					1.1
RAC: Cottonseed	0.02	0.02	0.03	0.03	1.2						
Meal	0.01	0.01	0.01	0.03	0.09	0.5	0.5	0.33	1	0.075	0.5
Crude oil	0.06	0.06	0.07	0.06	2.3	3	3	2.3	2	1.9	2.3
Refined oil	0.06	0.06	0.10	0.07	2.6	3	3	3.3	2.3	2.2	3
Soapstock	0.01	0.01	0.04	0.05	1.6	0.5	0.5	1.3	1.7	1.3	1.3

RESIDUES IN ANIMAL COMMODITIES

Livestock Feeding Studies

Lactating dairy cattle

Lactating Holstein cows were orally administered technical grade fenpropathrin (purity 92.5%) via gelatin capsules for 28 consecutive days (Fujie, 1986; Reference FR-61-0174). Treatment levels were equivalent to 0, 25, 75 and 250 ppm fenpropathrin based upon the daily average food consumption of 18.54 kg/cow. Each dose was administered in two equal portions at the morning and evening milkings. Each treatment group consisted of four animals, except the control group which consisted of 2 animals. Controls animals were administered empty gelatin capsules only.

Milk samples were collected from individual animals in two portions corresponding to the morning and evening milkings on the days -1, 0, 3, 5, 8, 12, 16, 20, 24 and 28. Milk from days 26 and 27 (250 ppm and control groups) were pooled and processed to obtain pasteurized milk, milk fat solids (cream), milk sugars and protein concentrate. At day 28, three animals from each treatment group were sacrificed and samples of liver, kidney, fat and muscle were collected and frozen for analysis. Administration of fenpropathrin then ceased and the animals received untreated diet. At day 31, the remaining animal from each dose group and one of the control animals were sacrificed and tissues sampled accordingly.

Samples of control milk were fortified with fenpropathrin, stored and analysed to confirm the stability of fenpropathrin over the sample storage period (78 days). An average recovery of 105% indicated stability of fenpropathrin in milk samples. Milk samples in the study were stored from 37 to 73 days at -20 °C prior to analysis.

All samples were analysed for fenpropathrin using method RM-22A-1 within the test period of storage stability. Residues of fenpropathrin in the milk reached a plateau after three days. Average residues in the whole milk of the four cows of each group on Day 3 were 0.04, 0.17, and 0.33 mg/kg for the three dose levels.

Fenpropathrin residues in milk and tissues from animals sacrificed at days 28 and 31 are summarized in Tables 61–63. Residue levels were found to be dose related.

Table 61. Residues of fenpropathrin in milk (mg/L) following administration of fenpropathrin to dairy cows for periods of up to 28 days followed by a 3 day depuration phase

Dose Level (mg/kg)	Day/residue (mg/kg)														Mean
	-1	0	1	3	5	8	12	16	20	24	26	27	28	31	
25	< 0.01	< 0.01	0.02	0.05	0.05	0.04	0.06	0.04	0.04	0.05	na	na	0.05	-	
	< 0.01	< 0.01	0.02	0.03	0.03	0.04	0.03	0.04	0.03	0.04	na	na	0.03	< 0.01	
	< 0.01	< 0.01	0.02	0.04	0.03	0.04	0.03	0.03	0.04	0.03	na	na	0.05	-	
	< 0.01	< 0.01	0.02	0.04	0.04	0.05	0.06	0.04	0.04	0.05	na	na	0.04	-	
Mean	< 0.01	< 0.01	0.02	0.04	0.04	0.04	0.05	0.04	0.04	0.04	na	na	0.04		0.04 ^a
75	< 0.01	< 0.01	0.03	0.09	0.09	0.08	0.07	0.08	0.08	0.09	na	na	0.12	-	
	< 0.01	< 0.01	0.07	0.16	0.11	0.13	0.18	0.17	0.22	0.18	na	na	0.16	0.02	
	< 0.01	< 0.01	0.06	0.14	0.10	0.12	0.10	0.07	0.10	0.09	na	na	0.06	-	
	< 0.01	< 0.01	0.13	0.28	0.19	0.18	0.35	0.18	0.17	0.21	na	na	0.16	-	
Mean	< 0.01	< 0.01	0.07	0.17	0.12	0.13	0.18	0.13	0.14	0.14	na	na	0.13	0.02	0.13 ^a
250	< 0.01	< 0.01	0.16	0.33	0.30	0.29	0.34	0.22	0.42	0.26	0.30	0.32	0.31	-	
	< 0.01	< 0.01	0.15	0.30	0.28	0.36	0.35	0.32	0.23	0.21	0.28	0.28	0.24	0.04	
	< 0.01	< 0.01	0.17	0.34	0.38	0.31	0.37	0.33	0.36	0.34	0.41	0.39	0.37	-	
	< 0.01	< 0.01	0.14	0.36	0.37	0.40	0.44	0.45	0.34	0.46	0.45	0.34	0.35	-	
Mean	< 0.01	< 0.01	0.16	0.33	0.33	0.34	0.38	0.33	0.34	0.32	0.36	0.33	0.32	0.04	0.32 ^a

na –not sampled for analysis; ^a Mean 1 – 28 days

Table 62 Maximum residues of fenpropathrin in tissue samples following administration of fenpropathrin to dairy cows for periods of up to 28 days

Dose level (mg/kg)	Tissue residue (mg/kg)			
	Muscle	Kidney	Liver	Fat
25	0.04	0.05	< 0.01	0.44
75	0.12	0.06	< 0.01	1.7
250	0.33	0.20	0.01	4.1

Table 63 Mean residues of fenpropathrin in tissue samples following administration of fenpropathrin to dairy cows for periods of up to 28 days followed by a 3 day depuration phase

Dose level (mg/kg)	Tissue residue (mg/kg)							
	Muscle		Kidney		Liver		Fat	
	After 28 days	3 day depuration	After 28 days	3 day depuration	After 28 days	3 day depuration	After 28 days	3 day depuration
25	0.02	0.01	0.03	0.01	< 0.01	< 0.01	0.33	0.31
75	0.06	0.10	0.04	0.06	< 0.01	< 0.01	1.0	0.83
250	0.20	0.12	0.16	0.14	0.01	< 0.01	3.8	2.6

Milk from sampling days 26 and 27 of the highest dose group were pooled and processed to obtain pasteurized milk, milk fat solids (cream), milk sugars and protein concentrate. The results show that pasteurization does not significantly reduce fenpropathrin residues in milk. Fenpropathrin concentrates in milk fat by a factor of about 10× (from mean of 0.34 mg/kg in whole milk to 3.7 mg/kg in milk fat). There is no concentration of fenpropathrin in milk sugars and protein concentrate (Table 64).

Table 64 Residues of fenpropathrin in milk fractions

Fraction	Residue (mg/kg)
Whole Milk	0.34 ^a
Pasteurized milk	0.25
Milk fat	3.7
Non-fat milk portion	< 0.01
Milk sugar	< 0.01
Protein concentrate	0.02

^a Mean residue in combined days 26 and 27 milk samples from highest dose.

Laying Hens

Groups of 20 White Leghorn laying hens were fed a diet containing technical grade fenpropathrin (purity 94.5%) at a nominal concentration of 0, 2.5, 7.5 and 25 ppm (measured concentrations: 2.45, 7.10 and 23.6 ppm) for a period of 28 days (Fujie, 1986; Reference FR-61-0175). The number and weight measurements of body weight and feed consumption were taken at regular intervals during the study.

At study termination, all birds were examined macroscopically. Composite samples of liver, gizzard, fat and muscle were prepared for analysis. Eggs from days 1, 2, 4, 7, 21 and 28 were analysed as whole eggs minus shell. Stability samples of treated diet were taken for analysis during the study. To determine the stability of fenpropathrin in stored eggs, control samples were fortified with fenpropathrin, stored and analysed at a time interval greater than the storage interval for eggs from the feeding study. An average recovery of 92% of fenpropathrin was obtained after 156 days of storage at -20 °C. All samples were analysed within 30 days of collection and storage.

Eggs and tissues were analysed for fenpropathrin. The method involved extraction with hexane/acetone, acetonitrile/hexane partitioning, silica gel chromatography and analysis by GC-ECD. Residues were found in the eggs only at the highest feeding level. A level of 0.02 mg/kg was reached on the seventh day and remained essentially constant until the end of the study. Average levels of fenpropathrin in the fat reached 0.02, 0.05 and 0.14 mg/kg for the three feeding levels. Residues in all tissues except fat were below the lower limit of determination, 0.01 mg/kg, at the end of the study. Metabolites could only be detected in liver after dosing with 25 ppm were TMPA (0.05 mg/kg) and PBA-glycin (0.03 mg/kg). The distribution of residues between white and yolk was not studied.

Fenpropathrin residues in eggs and poultry tissues from hens sacrificed at day 28 are summarized in Tables 65 and 66. Residue levels were found to be dose related.

Table 65 Residues of fenpropathrin in eggs (mg/kg) following administration of fenpropathrin to laying hens for a period of 28 days

Dose Level (mg/kg)	Day/Residue (mg/kg)						
	-1	1	4	7	14	21	28
2.5	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Mean	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
7.5	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Mean	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
25	< 0.01	< 0.01	< 0.01	0.02	0.02	0.01	0.02
	< 0.01	< 0.01	< 0.01	0.02	0.01	0.02	0.02
Mean	< 0.01	< 0.01	< 0.01	0.02	0.02	0.02	0.02

Table 66 Residues of fenpropathrin in tissue samples following administration of fenpropathrin to laying hens for 28 days

Dose level	Tissue residue (mg/kg)			
	Muscle	Gizzard	Liver	Fat
2.5	< 0.01	< 0.01	< 0.01	0.02
	< 0.01	< 0.01	< 0.01	0.02
	< 0.01			0.02
Mean	< 0.01	< 0.01	< 0.01	0.02
7.5	< 0.01	< 0.01	< 0.01	0.06
	< 0.01	< 0.01	< 0.01	0.05
	< 0.01			0.05
Mean	< 0.01	< 0.01	< 0.01	0.05
25	< 0.01	< 0.01	< 0.01	0.16
	< 0.01	< 0.01	< 0.01	0.14
	< 0.01			0.12
Mean	< 0.01	< 0.01	< 0.01	0.14

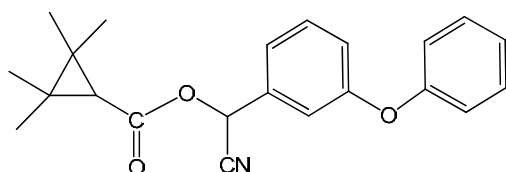
APPRAISAL

Fenpropathrin is a type II pyrethroid insecticide and acaricide used for the control of a variety of arthropods including aphids, worms, moths, beetles, mites, spiders, thrips, flies, fleas and other pests in agriculture.

Fenpropathrin was first evaluated by JMPR in 1993 when an ADI of 0–0.03 mg/kg bw was established and a number of MRLs recommended. In 2006 MRL for tea was recommended. The compound was re-evaluated for toxicology within the periodic review programme in 2012 when the Meeting reaffirmed the ADI of 0–0.03 mg/kg bw and established an ARfD of 0.03 mg/kg bw.

The Forty-fifth Session of the CCPR scheduled fenpropathrin for periodic re-evaluation of residues by the 2014 JMPR. Data to support proposed Codex MRLs on a number of commodities and on animal products were submitted for review.

The structural formulae and IUPAC name of fenpropathrin are:

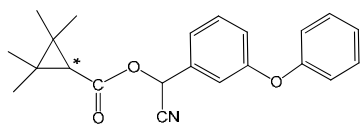


(*RS*)- α -cyano-3-phenoxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate.

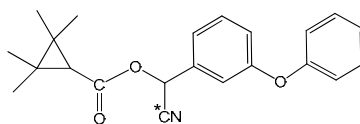
METABOLISM AND ENVIRONMENTAL FATE

The metabolism of fenpropathrin has been investigated in apple, tomato, beans, cotton, cabbage, lactating goat and laying hens. The crops selected represent those for which supervised trials have been provided.

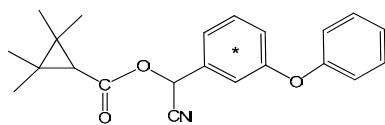
The fate and behaviour of fenpropathrin in plants, animals and soil were investigated using either [cyclopropyl-1-¹⁴C]-fenpropathrin, [benzyl-¹⁴C]-fenpropathrin, [phenoxyphenyl-¹⁴C]-fenpropathrin or [cyano-¹⁴C]-fenpropathrin (all with radiochemical purity >98%).



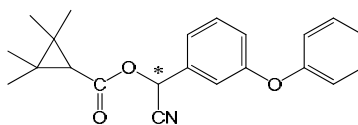
[cyclopropyl-1-¹⁴C]-fenpropathrin



[cyano-¹⁴C]-fenpropathrin



[phenoxyphenyl-¹⁴C]-fenpropathrin



[benzyl-¹⁴C]-fenpropathrin

The chemical and code names and structure of the major degradation compounds, referred to hereunder, are:

Compound Name	Structure	Found in:
2'-or 4'-OH-Fenpropathrin [α -cyano-3-(2'- or 4'-hydroxyphenoxy)benzyl 2,2,3,3-tetramethylcyclopropanecarboxylate]		Plant, animal, soil
2'- or 4'-OH-Fenpropathrin-CH ₂ OH [α -cyano-3-(2'- or 4'-hydroxyphenoxy)benzyl-2-hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylate]		Plant, animal
CONH ₂ -Fenpropathrin [α -carbamoyl-3-phenoxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate]		Soil, water plant
COOH-Fenpropathrin [α -carboxy-3-phenoxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate]		Soil, plant
Desphenyl-Fenpropathrin [α -cyano-3-hydroxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate]		Animal, soil, plant
Fenpropathrin-CH ₂ OH [α -cyano-3-phenoxybenzyl 2-hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylate]		Plant, animal
Fenpropathrin-COOH [α -cyano-3-phenoxybenzyl 2-carboxy-2,3,3-trimethylcyclopropanecarboxylate]		Animal
Fenpropathrin-(CH ₂ OH) ₂ [α -cyano-3-phenoxybenzyl 2,2-dihydroxymethyl-3,3-dimethylcyclopropanecarboxylate]		Plant
2'- or 4'-OH-Fenpropathrin-(CH ₂ OH) ₂ [α -cyano-3-(2'- or 4'-hydroxyphenoxy)benzyl 2,2-dihydroxymethyl-3,3-dimethylcyclopropanecarboxylate]		Plant
PB aldehyde (PBald) [3-phenoxybenzaldehyde]		Plant, animal, soil
PB alcohol (PBAlc) [3-phenoxybenzyl alcohol]		Plant, soil
PBacid [3-phenoxybenzoic acid]		Plant, animal, soil, water

Compound Name	Structure	Found in:
2'- or 4'-OH-PBalc [3-(2'- or 4'-hydroxyphenoxy)benzyl alcohol]		Plant
2'- or 4'-OH-PBacid [3-(2'- or 4'-hydroxyphenoxy)benzoic acid]		Plant, animal, soil
3-OH-Bacid [3-hydroxy-benzoic acid]		Animal, soil
TMPA [2,2,3,3-tetramethylcyclopropane-carboxylic acid]		Plant, animal, soil, water
TMPA-CH2OH [2-hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylic acid]		Plant, animal
TMPA-lactone [5,6,6-trimethyl-3-oxabicyclohexan-2-one]		Plant
TMPA-CH2OH lactone [5-hydroxymethyl-6,6-dimethyl-3-oxabicyclohexan-2-one]		Plant, animal
TMPA carboxamide [2,2,3,3-tetramethylcyclopropane-carboxamide]		Water
TMPA-COOH [2-carboxy-2,3,3-trimethylcyclopropanecarboxylic acid]		Plant, animal

Animal metabolism

Laboratory animals

The toxicological evaluation fenpropathrin was carried out by the 2012 JMPR. Absorption by rats was rapid and excretion was almost complete (97%) within 48 hours. About 56% of the administered dose was found in urine and 40% in faeces after 48 hours. The amount of radioactivity excreted via expired air was 0.005%. The low residues found in blood, liver, kidney, fat, muscle and brain 24 hours after dosing depleted rapidly over the following 7 days to barely detectable levels, and less than 1.5% of the administered dose remained in the body 8 days after treatment. The highest residue was found in the fat. About 29–53% of the parent compound was detected in the faeces and no parent compound was detected in the urine. The predominant urinary metabolites derived from the acid moiety were identified as TMPA–glucuronide and TMPA-CH₂OH (*trans*). Other metabolites identified were

TMPA-COOH (*trans*) and TMPA-CH₂OH-lactone in free form or as the glucuronide. The major urinary metabolites derived from the alcohol moiety were PBacid in free form and as the glycine conjugate, 4'-OH-PBacid-sulfate and 2'-OH-PBacid-sulfate. The urinary metabolites from the alcohol moiety were similar to those from other pyrethroids. The major faecal metabolite was identified as CH₂OH *trans*-fenpropathrin, followed by COOH *trans*-fenpropathrin, 4'-OH-fenpropathrin and 4'-OH,CH₂OH *trans*-fenpropathrin. Fenpropathrin and TMPA were the major components of ¹⁴C in tissues. No sex-related differences in tissue distribution were observed.

Lactating goats

Two lactating goats per group were dosed for five consecutive days via capsules with either [phenoxyphenyl-¹⁴C]-fenpropathrin or [cyclopropyl-1-¹⁴C]-fenpropathrin at a rate equivalent to 50 ppm. Milk samples were collected at the morning and afternoon and urine and faeces once a day.

The mean average total radioactivity recovered following dosing with both labelled compounds was about 65.8% with 40% recovered in the urine and 25% in the faeces.

Excretion via milk was a minor route with radioactivity accounting for approximately 0.15% of the applied phenoxyphenyl labelled and approximately 0.087% for the cyclopropyl labelled compound. Total radioactive residues in the milk reached a steady state by the evening milking on the third day.

Following the treatments with 50 ppm [phenoxyphenyl-¹⁴C]-fenpropathrin, the maximum total residue in milk was 0.25 mg/L. The major residue components were the parent fenpropathrin 78% TRR, (0.02 mg/kg). At around the plateau, the average concentration of the parent compound was 0.05 mg/kg (29%TRR) and PBacid-glycine 0.076 mg/kg (46%TRR). The other metabolites were < 10% TRR.

At sacrifice, the average residues were: in fat: fenpropathrin (0.50 mg/kg, 78%TRR), all metabolites were present at lower than 5%TRR; in muscle: fenpropathrin (0.011 mg/kg, 45%TRR), PBacid-glycine (22.4% TRR), PBacid (10.9% TRR) the other metabolites were below 3%; in liver: fenpropathrin (0.014 mg/kg, 3.2% TRR), PBacid-glycine (20%TRR), PBacid (14% TRR), 4'-OH-PBacid (11%TRR), the other metabolites were below 10% TRR; in kidney: fenpropathrin (0.01 mg/kg, 1.24%TRR), PBacid-glycine (39%TRR), PBacid (38%TRR) and the other metabolites were below 10%.

After the goats were administered with [cyclopropyl-1-¹⁴C]-fenpropathrin, the average total residue in milk was 0.11 mg/L. The parent compound in milk amounted to maximum 70% TRR (0.086 mg/kg). All metabolites amounted to maximum 4% TRR.

At sacrifice, the average residues were: in fat: fenpropathrin (0.55 mg/kg, 81% TRR), all metabolites were present at lower than 3%TRR; in muscle: fenpropathrin (0.005 mg/kg, 11.3% TRR), TMPA-CH₂OH-lactone (19.4% TRR) andTMPA-CH₂OH (11%TRR); in liver: fenpropathrin

0.011 mg/kg (2.45% TRR), TMPA (18.2%TRR), TMPA-CH₂OH (15.8%TRR) and TMPA-CH₂OH lactone (12.5% TRR); in kidney: fenpropathrin 0.0076 mg/kg (1.48% TRR), TMPA-CH₂OH-lactone (40.8% TRR) and TMPA-CH₂OH (14.5% TRR) .

The other metabolites in muscle, liver and kidney were below 5%TRR.

Laying hens

Fenpropathrin, labelled in either the cyclopropyl or the phenoxyphenyl ring was administered in capsules to laying hens daily for 10 days at a nominal rate of either 0.5 or 5 mg/kg body weight.

The recovery of total radioactivity from excreta, eggs and tissues was between 75 and 84% of the total applied dose. Between 98.9 and 99.6% of the recovered activity was found in the faeces irrespective of the label.

Approximately 0.05% of the applied phenoxyphenyl- and 0.2% of the cyclopropyl-labelled compound was found in the eggs. At about the 6th or 7th day of the study residue levels in the eggs reached a plateau of about 0.05 and 0.2 mg/kg fenpropathrin equivalent for the two doses of the phenoxyphenyl-labelled and about 0.2 and 0.5 mg/kg for those of the cyclopropyl-labelled compounds.

In case of high dose group treated with phenoxyphenyl-labelled compound, the average concentration of parent fenpropathrin amounted to 31% of TRR (0.043 mg/kg) in eggs and all identified metabolites were present at less than 10%TRR. In breast and thigh muscle, the average proportions of residues (%TRR) were: parent compound 19% (0.02 mg/kg), PBacid (22%), 3-OH-BAcid (13%). In liver and kidney the average percentage distributions of TRR were, respectively: parent (0.98%, 2.11%; 0.014 mg/kg, 0.096 mg/kg), 3-OH-BAcid (29%, 35%), 4-OH-PBacid (16%, 26%) and PBacid (14.7%, 8.7%). The other metabolites were <10% TRR in all tissues.

In case of cyclopropyl label the average concentration of parent fenpropathrin amounted to 9.8% TRR (0.038 mg/kg) in eggs, and one major metabolite, TMPA-CH₂OH, was present at 11.7% TRR. All other metabolites amounted to <10% TRR and their concentrations were < 0.03 mg/kg. In muscle, the average proportions of residues (%TRR) were: parent compound 6% (0.033 mg/kg), TMPA-CH₂OH (16%), TMPA (15.7%), TMPA-CH₂-OH-lactone (12.3%. All other metabolites amounted to <10% TRR and their concentrations were < 0.03 mg/kg. In liver and kidney the average percentage distributions of TRR were, respectively: parent (1.2%, 0.04 mg/kg; 5.1%; 0.24 mg/kg), TMPA (26.4%, 47.5%), TMPA-CH₂OH (14.7%, 7.7%), TMPA-CH₂-OH-lactone (14.8%, 5.3%), TMPA-COOH (11.4%, 15.3%). The other metabolites were present at < 1.8%TRR.

The metabolic pattern in fat was similar in case of both labels. The fat contained maximum 0.90 mg/kg total residue of which the parent compound amounted to 64%TRR (0.58 mg/kg). The most prominent metabolite was TMPA (14%TRR), the other metabolites occurred at < 6% TRR.

In summary, the major biotransformation reactions of fenpropathrin in animals consisted of oxidation at the methyl groups of the acid moiety and at the 2'- or 4'-positions of the alcohol moiety, cleavage of the ester and ether linkages and conjugation of the resultant carboxylic acids and alcohols.

The parent compound was detected in milk, eggs and tissues, and it was the main residue in fat about 80%TRR. The major metabolites >10% TRR following the treatment with phenoxyphenyl-labelled fenpropathrin were PBacid-glycine, PBacid and 3-OH-BAcid, and after dosing with cyclopropyl-labelled compound the major metabolites were TMPA-CH₂OH, TMPA, TMPA-CH₂-OH-lactone and TMPA-COOH.

Plant metabolism

Apples

One apple tree was treated 3 times with [cyclopropyl-1-¹⁴C]-fenpropathrin and [benzyl-¹⁴C]-fenpropathrin at a rate equivalent to 0.45 kg ai/ha. Samples were collected 14 days after the final application. Un-extractable residues ranged from 3% (both labels in fruit) to 8% (benzyl label in leaves).

Practically, the entire residue found in the fruit (92-94% TRR) was present as the parent compound. The parent compound was also the major component in the rest of the plant (61–66%TRR). All metabolites were < 5%TRR.

Tomatoes

Greenhouse-grown tomato plants were treated four times, 7–8 days apart, with [cyclopropyl-1-¹⁴C]-fenpropathrin and [benzyl-¹⁴C]-fenpropathrin at rates equivalent to 0.224 kg ai/ha. Fruit and leaves were extracted at harvest 19 DALT).

The total radioactive residues were 0.1 mg/kg and 0.4 mg/kg in fruits after treatments with benzyl and cyclopropyl labelled compound and consisted of the parent compound in about 66% of the benzyl label and 28% of conjugated metabolites. Their proportion was about the opposite for cyclopropyl label. The non-extractable residues were between 5.5% and 6.7%. Because of their low level, the radioactivity could not be fully characterised.

In tomato plants, the parent compound was present in 36–39% of TRR (0.1-0.04 mg/kg). Of the identified metabolites only fenpropathrin-(CH₂OH)₂ was present in free form (2.7–3.1% TRR). Numerous other metabolites were in conjugated forms. Non-extractable residues amounted to 7.4–9.3%.

In another study where tomato plants were treated in greenhouse four times with [cyclopropyl-1-¹⁴C]-fenpropathrin and [phenoxyphenyl-¹⁴C]-fenpropathrin at a rate equivalent to 0.224 kg ai/ha. Fruits and plant materials, sampled 3 days after last application, contained the parent

compound in 96–98% of TRR. Polar metabolites amounted to 1.3% of TRR. The surface rinses contained 98–99% of the parent compound determined in the fruits.

Beans

Pinto bean plants grown in greenhouse were treated three times with [cyclopropyl-1-¹⁴C]-fenpropathrin and [benzyl-¹⁴C]-fenpropathrin at a rate equivalent to 0.224 kg ai/ha. Samples were collected 15 days after the final application. Leaves and plant parts contained 98.8%, bean pods and seeds contained 1.1% and 0.1% of the residue. In beans treated with benzyl- and cyclopropyl-labelled compound, the residue in seed was composed of the parent compound (4.1% and 0.1% of TRR), conjugated metabolites (61–51% TRR) free metabolites (17–4%TRR) and un-extractable residues 18.2%–45% TRR. The bean leaves contained 46.7% parent compound and conjugates of PBald (19.5%TRR) after treatment with benzyl labelled compound. After treatment with cyclopropyl-labelled compound the residue composed of 46.4% parent fenpropathrin and conjugates of TMPA-CH₂OH (16.7%TRR). The other metabolites were present at < 10% TRR.

Cotton

Two studies were conducted treating cotton plants with [phenoxyphenyl-¹⁴C]-fenpropathrin and [cyclopropyl-1-¹⁴C]-fenpropathrin.

In the first study the plants were treated in greenhouse four times with syringe applying a total of ca. 4.7–4.8 mg ¹⁴C-fenpropathrin. In the leaves at harvest 66 and 111 days after treatments with phenoxyphenyl- or cyclopropyl labelled compound the total remaining radio activity included 70% TRR and 55% TRR parent compound, respectively. Most of the remaining radio activity was tentatively accounted for PBacid (2.% TRR) and trans-TMPA-COOH (11%TRR) mainly in conjugated forms. All other metabolites were below 2%TRR.

Plants grown on soils treated with 0.5 kg/ha of fenpropathrin contained ¹⁴C residues in very low concentration (0.002 mg/kg in leaves and 0.01 mg/kg in bolls), demonstrating limited tendency for translocation.

In the second study outdoor cotton plants were treated four times at a rate equivalent to 0.336 kg ai/ha. Seeds collected 21 day after last treatment contained total radioactivity 1.14 mg/kg and 1.59 mg/kg, while the foliage contained 78.6 mg/kg and 67.7 mg/kg fenpropathrin equivalent after treatment with phenoxyphenyl- and cyclopropyl-labelled fenpropathrin, respectively. The seed, lint and foliage contained the parent fenpropathrin in 93.8%, 96.2% and 69.2% TRR, respectively, after treatment with phenoxyphenyl -labelled compound. The seed contained 12 metabolites each at < 0.005 mg/kg concentration. Following the application of cyclopropyl-labelled fenpropathrin the parent compound amounted to 85.6%, 100% and 67.4% TRR in seed, lint and foliage, respectively. A small number of metabolites were also detected, but not identified.

Cabbage

Cabbage plants were treated on the 3rd-4th leaves with [cyano- ^{14}C]-fenpropathrin), [cyclopropyl- ^{14}C]-fenpropathrin and [phenoxyphenyl- ^{14}C]-fenpropathrin at a rate equivalent to about 0.09 kg ai/ha. The cabbages were sampled immediately after application and at 3, 7, 14, 21, 28, 35 and 42 days after application. The proportions of parent fenpropathrin in 28-day samples after treatment with cyano-, cyclopropyl- and phenoxyphenyl-fenpropathrin were 16.9%, 15.8% and 12.9% of the applied dose, respectively, and it was present at somewhat lower proportion in 48-day samples. The major part of the residue (23-26% AD) composed of the conjugates of 2'-OH-fenpropathrin- CH_2OH , 4'-OH-fenpropathrin- CH_2OH , 2'-OH-fenpropathrin- $(\text{CH}_2\text{OH})_2$ and 4'-OH-fenpropathrin- $(\text{CH}_2\text{OH})_2$ and TMPA- CH_2OH -lactone-conjugate (11%). The other metabolites were present at $\leq 10\%$ TRR after treatments with cyclopropyl- and phenoxyphenyl-labelled compounds.

Most of the recovered radiocarbon was in the treated leaves and less than 1.2% of the applied radiocarbon was found in the untreated shoots indicating that fenpropathrin and its metabolites hardly translocate from the application site to other parts of the plant.

Fate of hydrogen cyanide (HCN) and TMPA in abscised leaves

The fate of HCN and TMPA in abscised leaves of apple, kidney bean, cabbage, mandarin orange, tomato and vine was studied. Two abscised leaves from each plant were placed in 100 mL distilled water containing ^{14}C -TMPA at a concentration of 1.0 ppm. After cultivation for five days the leaves were extracted with methanol:chloroform:water (4:2:1).

TMPA was readily converted in plants to more polar products. The metabolic pathways for TMPA varied dependent upon species of plant. The glucose ester was a main product in apple and vine leaves. In orange, cabbages and bean leaves, the malonylglucoside was mainly formed.

Further on, two abscised cabbage leaves were treated for four hours with distilled water containing K^{14}CN and then transferred to K^{14}CN -free distilled water. The study demonstrated that if hydrogen cyanide were liberated during the hydrolysis of fenpropathrin, it would be rapidly converted to natural products.

Summary of plant metabolism

Metabolism of fenpropathrin has been studied in apples, tomatoes, beans, cotton and cabbage.

The general pattern of degradation in all the plant studies include break of the ester linkage to produce 3-phenoxybenzoic acid (PBacid) and the corresponding alcohol (PBalc) and aldehyde (PBald). From the acid side of the molecule, the main metabolite is TMPA which can give rise to TMPA- CH_2OH and TMPA- CH_2OH lactone. PBacid can be hydroxylated at various positions on the phenoxy ring to produce, 2'-or 4'-OH-PBacid.

The majority of radioactivity was found in leaf samples. Low levels of radioactivity were found in fruit/beans. The parent fenpropathrin amounted to the major part of the residue. Fenpropathrin and its metabolites hardly translocate from the application site to other parts of the plant.

Environmental fate

In soil

Studies on the metabolism of fenpropathrin in aerobic soil carried out with [phenoxyphenyl-¹⁴C]-fenpropathrin demonstrated that fenpropathrin is degraded in the soil by a combination of photochemical and microbial processes. After 365 days, 18.4% of the dose remained as parent with accumulated volatiles accounting for 59.9% (99.8% of which was CO₂) and un-extractable residues for 17.8%. Metabolism proceeds via cleavage of the ester bonds, hydroxylation, and hydrolysis of the cyano group to CONH₂ and COOH groups. Metabolites included desphenyl-fenpropathrin, 4'-OH-fenpropathrin, phenoxybenzoic acid, and CONH₂-fenpropathrin, which was further degraded to COOH-fenpropathrin. The estimated half-life was about 4 weeks in moist soil (70–75% field capacity) and 16 weeks in a dryer soil with 16% water content.

Photodegradation studies were carried out with fenpropathrin labelled with ¹⁴C in the cyano group, the phenoxyphenyl ring or C-1 position of the cyclopropyl ring. Irradiation greatly enhanced degradation of the fenpropathrin. The main degradation product under irradiation with all three labels was CONH₂-fenpropathrin which reached a maximum in the three soils after 5–7 days during the 14-day exposition.

Fenpropathrin is moderately stable in soil under aerobic condition. The photolysis increased the degradation of the surface residues.

Hydrolytic degradation

Fenpropathrin is stable to hydrolysis in water at pH 5 and pH 7 but it is hydrolysed at a moderate rate at pH 9.

Rotational crops

No study was submitted on rotational crops.

Methods of analysis

Analytical methods have been developed for determination of residues of fenpropathrin in plant and animal matrices. In general, the methods involve solvent extraction, clean-up by either silica gel or Florisil column, GLC using electron capture detection. Additional purification using gel permeation chromatography (GPC) was performed for oily matrices. The main variations depending on the substrates are on extraction and clean-up procedures. Fruits and vegetables may be homogenized with

water, shaken with acetone, and extracted with dichloromethane, using NaCl to minimize emulsification. After drying with anhydrous sodium sulphate and clean-up by silica gel column chromatography, the solvent is evaporated at $< 40^{\circ}\text{C}$ and the residue dissolved in acetone before determination by gas chromatography with electron capture detection. Other extraction procedures involve direct extraction of the homogenized material suspension in water or homogenization with methanol instead of water. The methods were generally validated at 0.01 mg/kg LOQ level. The RSD of recoveries was $< 20\%$

A multi residue method (DFG S19) was validated for the determination of fenpropathrin in plant materials of high water content and acidic plant matrices applying GC-MS detection (m/z 181 and 265 for quantification and 125, 152, 209 and 349 for confirmation).

In a supervised trial on soya bean, the residues were determined with LC-MS/MS utilising the transition of m/z 350 $\rightarrow$ 125. The LOQ was 0.01mg/kg.

Stability of residues in stored analytical samples

The stability of fenpropathrin residues in commodities under frozen conditions has been investigated in apples, orange, cotton, pears, grapes, tomato and its processed products as well as in products of animal origin.

Fenpropathrin was shown to be stable at least for the indicated periods (month) in: apple, orange, cotton, pears and grapes (12);cucumber (8); grape juice, dry pomace (14) wet pomace (12), hydrated raisins and raisin waste (11); melons (6); non-bell peppers (10); olives, olive oil (~ 7); orange oil and orange dried peel (11); raspberries (7); squash (7.5); strawberries (6); tomato (6), tomato paste (5), tomato juice (5) and wet and dry tomato pomace (5) tomato waste (5).

The residues were stable in eggs for 5 months, and milk and kidney at least for 2.5 months.

Definition of the residue

Livestock animal metabolism studies were conducted on lactating goats (50 ppm in feed) and laying hens (0.5 and 5 mg/kg body weight) applying [phenoxyphenyl- ^{14}C]- and [cyclopropyl-1- ^{14}C]-fenpropathrin.

In milk, at around the plateau at 3–5 days, the parent compound amounted to the major part of residues 28% TRR, (0.02 mg/kg) and 66% TRR, (0.086 mg/kg), respectively. The major metabolites were PBacid-glycine (46% TRR) from phenoxy label and all other metabolites were below 3%TRR from cyclohexyl label.

Following the treatments with 50 ppm [phenoxyphenyl- ^{14}C]-fenpropathrin the average residues of the parent fenpropathrin amounted to 0.50 mg/kg (78%TRR) in fat, 0.011 mg/kg (45% TRR) in muscle, 0.14 mg/kg (3.2% TRR) in liver and 0.01 mg/kg (1.2% TRR) in kidney. The major

metabolites in these tissues were PBacid-glycine (20–39% TRR), PBacid (11–38% TRR) and 4'-OH-PBacid (11% TRR). The other metabolites were present at lower than 10% TRR.

After the goats were administered with [cyclopropyl-1-¹⁴C]-fenpropathrin, the average residues comprised of the parent compound 0.55 mg/kg (81% TRR) in fat, 0.005 mg/kg (11% TRR) in muscle, 0.11 mg/kg (2.5% TRR) in liver and 0.0076 mg/kg (1.5% TRR) in kidney. The major metabolites in these tissues were TMPA-CH₂OH-lactone (19–41% TRR), TMPA (18%TRR), TMPA-CH₂OH (11–16%TRR). The other metabolites were below 5%TRR.

In hens, the parent fenpropathrin was a major residue 0.043 mg/kg (31%TRR) and 0.038 mg/kg (9.7%TRR) in eggs following treatments with benzyl- and cyclopropyl-labelled compounds. The only metabolite exceeding 10% TRR was TMPA-CH₂-OH from cyclopropyl label. Following dosing with benzyl label, the average concentration of parent compound was 0.43 mg/kg (29%TRR) in fat, 0.029 mg/kg (1.6% TRR) in muscle, 0.096mg/kg (7%TRR) in kidneys and 0.014 mg/kg (1% TRR) in liver. After dosing with cyclopropyl label, the average concentration of parent compound was 0.033 mg/kg, (1.55%TRR) in muscle, 0.21 mg/kg (10% TRR) in kidneys, 0.036 mg/kg (2% TRR) in liver. The main metabolites from hens dosed with the benzyl-labelled compound were 3-OH-Bacid (4–29%TRR) or 4'-OH-PBacid (4–16% TRR and PBacid 3.2–24% TRR). While from the cyclopropyl-labelled group the TMPA (6–26% TRR) and its CH₂-OH, COOH, CH₂-OH-lactone derivatives (9–41%TRR) were the major metabolites. Several of these metabolites are also formed from other pyrethroid insecticides.

The parent fenpropathrin was the major residue in milk, meat and eggs and it was detected at low concentrations in liver and kidney. The polar metabolites listed above and the minor ones identified are of no toxicological significance.

The Meeting concluded that the parent fenpropathrin is a suitable marker for animal commodities for both enforcement and dietary risk assessment.

As the fenpropathrin residue concentrates in the fat, based on the distribution of residues in various tissues, supported by the logP_{ow} of 6.0 for fenpropathrin, the Meeting concluded that the fenpropathrin residue is fat soluble.

The fate of fenpropathrin residues was studied in apples, beans, cabbages, cotton and tomatoes. The parent fenpropathrin is the major residue in apple fruits (92–94% TRR), tomato fruits (30–66% TRR), in bean leaves (46% TRR), bean seeds (up to 4.1% TRR), in cabbage leaf extract (up to 16% TRR) and cotton seed (up to 94% TRR). The major metabolites were the conjugates of 2'-OH-fenpropathrin-(CH₂OH)₂ and of 4'-OH-fenpropathrin-(CH₂OH)₂ (max 19–22% TRR in cabbage), all other metabolites (1.3–9.8%TRR) were < 10% TRR in case of all labelled compounds.

The nature of metabolites is similar to that goats and hens. The polar metabolites listed above are of no toxicological significance.

The Meeting concluded that the parent fenpropathrin is suitable marker for plant commodities for both enforcement and dietary risk assessment.

Validated analytical methods, suitable for enforcement, are available for detecting fenpropathrin in various matrices.

The Meeting agreed in the following residue definition:

Definition of residue for compliance with MRL and for estimation of dietary intake for animal and plant commodities is the parent fenpropathrin.

The residue is fat soluble.

Results of supervised residue trials on crops

The Meeting received supervised trial data on citrus fruits, pome fruits, stone fruits, berries and small fruits, fruiting vegetable, cucurbits, olives, soya beans, cotton, tree nuts, coffee beans and tea.

The HR values were estimated based on the residues measured in single samples.

Citrus fruits

A total of 31 supervised trials were conducted in the United States on citrus fruits (18 on oranges, 6 on lemons and 7 on grapefruit). The current maximum GAP for citrus in the USA consists of two applications at 0.45 kg ai/ha with a total seasonal rate of 0.90 kg ai/ha and a PHI of 1 day.

The average residues in duplicate composite samples derived from treatments corresponding to US GAP were: in oranges: 0.05, 0.07, 0.12, 0.18, 0.25, 0.26, 0.27, 0.33, 0.33, 0.46, 0.53, 0.96, 1.2 mg/kg; in lemons: 0.51, 0.56, 1.2 mg/kg; in grapefruits: 0.12, 0.18, 0.2, 0.34, 0.34, 0.37, 0.47 mg/kg.

The Meeting noted that the GAP in USA is for citrus fruits, that the residue populations were not significantly different (Kruskal-Wallis H-test) and the median residues were within the 5 times range. Furthermore, the Meeting concluded that the residue data sets for lemons, oranges and grapefruits are suitable for estimation of residue levels for the citrus group. The meeting agreed to combine the datasets which in rank order were: 0.05, 0.07, 0.12, 0.12, 0.18, 0.18, 0.2, 0.25, 0.26, 0.27, 0.33, 0.33, 0.34, 0.34, 0.37, 0.46, 0.47, 0.51, 0.53, 0.56, 0.96, 1.2, 1.2 mg/kg.

The Meeting estimated a maximum residue value of 2mg/kg and, based on the processing factor of 0.065, HR of 0.098 mg/kg and STMR values 0.02 mg/kg for citrus fruit group.

Pome fruits

Forty-seven supervised trials on pome fruit (27 in apples and 20 in pears) were conducted in the USA in 1984-1987, using a higher number of applications and total seasonal rates compared to the GAP in the USA (up to 0.9 kg ai/ha and a PHI of 14 days).

Four trials in apple and pear complied with current US GAP. The residues were: in apple: 0,48, 0.58, 0.88 and 1.1 mg/kg; and in pear: 0.27, 0.3, 1.2 and 1.8 mg/kg.

The Meeting noted that the GAP in USA is for pome fruit, that the residue populations were not significantly different and that the median residues were within the 5 times range.

The Meeting concluded that the residues in apples and pears could be combined: 0.27, 0.3, 0,48, 0.58, 0.88, 1.1, 1.2 and 1.8 mg/kg.

The Meeting noted that in one of the pear samples the residue was 2 mg/kg, and estimated a maximum residue level of 3 mg/kg, HR of 2 mg/kg and STMR of 0.73 mg/kg

The Meeting withdraws its previous recommendation for maximum residue levels of 5 mg/kg

The meeting noted that the short-term intakes of apples and pears for children are 390% and 280% of ARfD, respectively.

There is no alternative GAP available to be considered.

Stone fruits

Supervised trials were carried out in USA on peaches (10), cherries (6) and plums (7) according to US GAP (2×0.45 kg ai/ha, 3 days PHI).

The average residues in duplicate composite samples derived from treatments corresponding to US GAP were: in peach: 0.44, 0.58, 0.65, 0.66, 0.70, 0.71, 0.73, 0.92, 1.0, 1.0 mg/kg; in plums; 0.18, 0.22, 0.23, 0.25, 0.32, 0.35, 0.67 mg/kg; and in cherries: 1.4, 1.5, 1.8, 1.9, 3.3, 3.4 mg/kg.

Since the residue populations are not similar, residue levels were estimated separately for each commodity.

The meeting estimated maximum residue, HR and STMR values for subgroups of: peaches 3 mg/kg, 1.1 mg/kg and 0.71 mg/kg; plums 1 mg/kg, 0.71 mg/kg and 0.25 mg/kg; and cherries 7 mg/kg, 3.53, and 1.85 mg/kg, respectively.

The meeting noted that the short-term intakes of peaches and cherries are 190% and 140% of ARfD for children, respectively.

There is no alternative GAP to be considered.

Berries and other small fruits

Strawberry

Eleven out of 12 trials conducted on strawberry in USA matched the US GAP (applications at up to 0.45 kg ai/ha for a total of 0.9 kg ai/ha per season and a PHI of 2 days).

The average residues in duplicate composite samples derived from treatments corresponding to US GAP were: 0.26, 0.38, 0.39, 0.48, 0.48, 0.55, 0.63, 0.65, 0.69, and 1.2 mg/kg.

The Meeting estimated maximum residue level, HR and STMR values of 2 mg/kg, 1.2 mg/kg and 0.515 mg/kg, respectively.

Raspberry

Seven supervised trials on raspberries were conducted in the USA in 2005 with higher rate and shorter PHI (total 0.9 kg/ha with 2 days PHI) than the current GAP for caneberries (applications at up to 0.34 kg ai/ha for a total of 0.67 kg ai/ha per season and a PHI of 3 days).

As no trial matched the GAP, recommendation cannot be made.

Grape

Twenty five supervised trials were conducted on grapes in the USA during 1983–2001 ($2 \times$ maximum rate of 0.45 kg ai/ha, the maximum seasonal rate of 0.9 kg/ha with 21 days PHI).

The trial data did not match the critical GAP of the USA. As a result no recommendations could be made.

The Meeting withdraws its previous recommendation of 5 mg/kg.

Assorted tropical and subtropical fruits – Edible peel

Olives

Three supervised trials were conducted on olives in the USA during 2005 matching US GAP ($3 \times$ 0.34 kg ai/ha with total seasonal application rates of about 0.9 kg ai/ha and 7 days PHI).

The average residues in pitted olives from two composite samples derived from treatments corresponding to maximum application rates were in rank order: 1.9, 2.2, and 3.6 mg/kg.

Three residue values were not considered sufficient for the estimation of maximum residue levels in olives.

Fruiting Vegetables, Cucurbits

Cucumber

Six supervised trials on cucumber were conducted in the USA in 1994 and 1996, following the GAP in the USA for cucurbit vegetables (applications at the rate of up to 0.34 kg ai/ha at 7 days intervals for a total of 0.9 kg ai/ha/season; PHI is 7 days).

The Meeting noted that the trials were not conducted at maximum GAP. For multiple treatments proportionality could be applied. As a result no recommendations could be made.

Melon

Ten supervised trials on cantaloupe were conducted in the USA in 1994 following the GAP in the USA for cucurbits (applications at the rate of up to 0.34 kg ai/ha for a total of 0.9 kg ai/ha/season; PHI is 7 days).

The Meeting noted that the trials were not conducted at maximum GAP. As the number of applications and or the applied dosage rate differed from maximum GAP, the proportionality could not be applied. As a result no recommendation could be made.

Summer squash

Seven supervised trials on summer squash were conducted in the US in 1994 and 1996, following the GAP in the US for cucurbits (applications at the rate of up to 0.34 kg ai/ha for a total of 0.9 kg ai/ha/season; PHI is 7 days)

The Meeting noted that the trials were not conducted at maximum GAP. As the number of applications and or the applied dosage rate differed from maximum GAP, the proportionality could not be applied. As a result, no recommendation could be made.

*Fruiting vegetables other than Cucurbits**Tomato*

Nine supervised trials conducted on tomatoes in the USA in 1993 matching the US GAP for fruiting vegetables other than cucurbits (applications at the rate of 0.22-kg ai/ha at 7 days intervals but not more often than 7 days, PHI is 3 days) were received.

The average residues in two composite samples derived from treatments corresponding to maximum application rates, in ranked order, were: 0.05, 0.08, 0.11, 0.18, 0.19, 0.21, 0.27, 0.30, and 0.55 mg/kg.

Peppers

Ten supervised trials on peppers (6 on bell and 4 on non-bell) were conducted in the USA in 1996 and 1998. The application rates corresponded to US GAP (0.22 kg ai/ha up to 0.9 kg ai/ha, 3 days PHI), but samples were taken at 2 and 4 days instead of the 3 day PHI.

The average residues in two composite samples were: in Bell pepper: 0.10, 0.34, 0.37, 0.37, 0.50, 0.67 mg/kg; and Chili pepper: 0.24, 0.31, 0.38, 0.40 mg/kg

The Meeting noted that the residues obtained 2–4 days after last application were in a relatively narrow residue range (2×median) leading to lower maximum residue estimate than would be generally expected. Therefore the residue values obtained at day 2 (-33% of PHI) were considered acceptable. (Only one residue data (0.50 mg/kg) was obtained at day 3 and one at day 4 (0.10 mg/kg).

The Mann-Whitney U-test confirmed that the above data could be combined for the estimation of the maximum residue level and STMR. The ranked order of residues from supervised trials on peppers were: 0.10, 0.24, 0.31, 0.34, 0.37, 0.37, 0.38, 0.40, 0.50, and 0.67 mg/kg.

The Meeting noted that the GAP in USA is for fruiting vegetables, other than cucurbits and the residue populations were not significantly different (Kruskal-Wallis H-test) and the median residues were within 5 times range. However, the short-term intake for eggplants would exceed the ARfD by 110% for adults. Consequently, a recommendation for the fruiting vegetables crop group could not be made.

No alternative GAP was available for fruiting vegetables other than cucurbits.

The Meeting therefore agreed to estimate residue levels for individual commodities:

Tomato: maximum residue level of 1 mg/kg, HR of 0.64 mg/kg, STMR of 0.19 mg/kg.

Peppers including chili pepper: maximum residue level of 1 mg/kg, HR of 0.70 mg/kg, STMR of 0.37 mg/kg.

Chili peppers, dried (based on concentration factor of 7): maximum residue level of 7 mg/kg, HR of 4.9 mg/kg mg/kg, STMR of 2.59 mg/kg.

The meeting confirms its previous recommendation for maximum residue level of 1 mg/kg for tomatoes.

Pulses

Soya beans

Eight supervised trials on soya beans were conducted in Brazil in 2010 and 2013, following the GAP in Brazil (one applications at the rate of 0.045 kg ai/ha and a PHI of 30 days). Residues in all samples were below the limit of quantification (< 0.01 mg/kg).

The Meeting estimated a maximum residue and STMR values of 0.01 mg/kg.

Cotton

Thirty-two supervised trials on cotton were conducted in the USA in 1983–1989 with application rates up to 0.9 kg ai/ha and a PHI of 21 days. The current US GAP is up to 0.45 kg ai/ha with a seasonal maximum rate of 0.9 kg/ha and a PHI of 21 days.

As 5–10 applications were made with low dose rates, which do not represent the critical GAP, no recommendations could be made.

The Meeting withdraws its previous recommendation of 1 mg/kg.

Tree nuts

A total of ten supervised trials on tree nuts, 5 on almonds and 5 on pecans have been conducted in the US in 2003, with application rates of 0.45 and 0.9 kg ai/ha at 7 days intervals instead of the minimum 10 days specified on the label.

The following average residue levels in two composite samples were obtained from the trials on almonds and pecans:

Almond nutmeat: < 0.01(4), 0.03 mg/kg; and in pecans: < 0.01, 0.01, 0.02, 0.05, 0.06 mg/kg.

The Meeting noted that the GAP in USA is for tree nuts and the residue populations were not significantly different (Mann-Whitney U-test) and the median residues were within 5 times range. The Meeting agreed to combine the datasets for almonds and pecans which, in ranked order, were: < 0.01(4), < 0.01, 0.01, 0.02, 0.03, 0.05, and 0.06 mg/kg.

As the highest residue in an individual samples was 0.1 mg/kg, the Meeting estimated a maximum residue level of 0.15 mg/kg, HR of 0.1 mg/kg and STMR value of 0.01 mg/kg.

Coffee Beans

Six supervised trials on coffee were conducted in Brazil in 2013 following the current GAP there (two applications at a maximum rate of up to 0.12 kg ai/ha and a PHI of 14 days).

Residues in composite samples, in ranked order, were: < 0.01(4), 0.01, and 0.02 mg/kg.

The Meeting estimated a maximum residue level of 0.03 mg/kg and STMR value of 0.01 mg/kg.

Tea

All supervised trials on tea were conducted in India during 2002-2004. The compound was applied according to the GAP in India (0.05-0.06 kg ai./ha with a PHI of 7 days) and with double rate.

Six trials on black tea and in one trial green tea leaves (0.13 mg/kg) were analysed

The residues in composite samples following application at the GAP rate were: < 0.05, 0.13, 0.14(2), 0.17, 0.18, and 1.38 mg/kg.

The Meeting estimated a maximum residue level for green and black tea of 3 mg/kg and STMR value of 0.14 mg/kg.

The Meeting withdrew its previous recommendation of 2 mg/kg for maximum residue level for tea.

Animal feeds

Almond hulls

The US GAP specifies application rates of up to 0.45 kg ai/ha with a seasonal maximum of 0.9 kg ai/ha at 10 days intervals, and a PHI of 3 days.

In almond hulls, the ranked order of residue concentrations was 2.7, 2.9, 3.1, 3.5, and 3.6 mg/kg. The Meeting estimated a maximum residue level of 10 mg/kg, the highest residue is 3.6 mg/kg and the median residue is 3.1 mg/kg.

Cottonseed hull

The trial conditions did not match US GAP (2×0.45 , max seasonal rate 0.9 mg/kg, PHI 21 days), no recommendations could be made.

Fate of residues during processing

Processing studies were carried out on plums, tomato, olives, oranges, cottonseed and tea.

The processing factors calculated and STMR-P values estimated are summarized below.

Summary of selected processing factors and STMR-P values for fenpropathrin

RAC/processed fraction	Processing factors					PF estimated	STMR-P (mg/kg)
RAC: Whole orange	-						
Juice	< 0.02	< 0.22				< 0.02	0.007
Oil	78.7	21.56				50.1	16.5
Wet peel	0.6	0.78	2.76		2.86	2.82	0.93
Dried peel	1.6	2.67				2.1	0.70
Pulp			0.06		0.07	0.065	0.021
RAC: Plum							
Dried plum	2.56					2.56	0.639
RAC: Tomato							
Canned	0.077	0.071	0.077			< 0.075	0.021
Wet pomace				9.9	9.8	9.8	1.867
Dry pomace				46	45.0	45	8.618
Tomato paste				0.78	0.75	0.77	0.145
Tomato juice				0.12	0.1	0.12	0.023

Note: The residues measured in RAC samples taken at the processing plants are considered as they better reflect the residues in unprocessed commodities than these measured in field samples

There is no concentration of residues in juice and molasses. Residues concentrate in oil (Pf=50.1), and dried peel (Pf=2.1).

The Meeting estimated a maximum residue level of 100 mg/kg and STMR-P of 16.5 mg/kg for citrus oil,

Drying concentrates the residues of fenpropathrin in plums by a factor of 2.6×; The Meeting estimated a maximum residue level of 3 mg/kg, HR-P of 1.85 mg/kg and STMR-P of 0.65 mg/kg for dried plums (or prunes).

As no MRL could be estimated, the Meeting withdrew its previous recommendation of 3 mg/kg for cottonseed oil.

Residues in animal commodities

Estimation of dietary burden

The maximum and mean dietary burdens were calculated using the highest residues or median residues of fenpropathrin estimated at the current Meeting on a basis of the OECD Animal Feeding Table. Only almond hull, citrus pulp and tomato wet pomace can be used as animal feed based on recommended uses. The calculated maximum and mean animal burdens are summarised below

Summary of livestock dietary burdens (ppm of dry matter diet)

	US-Canada		EU		Australia		Japan	
	max	mean	max	Mean	max	mean	Max	mean
Beef cattle	0.09	0.09	0.045	0.045	1.46 ^a	1.46 ^b	0	0
Dairy cattle	0.43	0.04	0.18	0.18	1.46	1.46	0	0
Broilers	0	0	0	0	0	0	0	0
Layers	0	0	0	0	0	0	0	0

^a Suitable for estimating maximum residue levels for milk, meat, fat and edible offal of cattle.

^b Suitable for estimating STMRs for meat, fat and edible offal of cattle.

Farm animal feeding studies

Lactating Holstein cows were orally administered technical grade fenpropathrin (purity 92.5%) via gelatin capsules for 28 consecutive days in two equal portions at the morning and evening milkings. The treatment levels were 0, 25, 75 and 250 ppm fenpropathrin based upon the daily average food consumption.

Residues of fenpropathrin in the milk reached a plateau after three days. Average residues in the whole milk of the four cows of each group on Day 3 were 0.04, 0.17 and 0.33 mg/kg for the three dose levels. On Day 28, these levels were 0.04, 0.13 and 0.32 mg/kg. At the end of the three-day depuration period, residues had fallen to < 0.01, 0.02 and 0.04 mg/kg for the three levels. Pasteurization did not significantly reduce fenpropathrin residues in milk. The residues concentrated in milk fat by a factor of about 10 (from mean of 0.32 mg/kg in whole milk to 3.7 mg/kg in milk fat)

After 28 days of dosing, maximum and (average) residues, expressed in mg/kg, in muscle, kidney, liver and fat were 0.33 (0.2), 0.2 (0.16), 0.01 (0.01), and 4.1 (3.8) mg/kg, respectively, at the maximum 250 ppm dose level. The residues determined after feeding with 25 ppm fenpropathrin in

feed, and the corresponding residues in tissues and milk resulted from the calculated mean and max dietary burden (1.46 ppm) are summarised below.

Dietary burden	Fat		Meat		Liver		Kidney		Milk
	Max	Mean	Max	Mean	Max	Mean	Max	Mean	Mean
25 ppm	0.44	0.33	0.04	0.02	< 0.01	< 0.01	0.05	0.03	0.04
1.46 ppm	0.026	0.018	0.002	0.001	< 0.0006	< 0.0006	0.003	0.002	0.002

Based on the data available the Meeting estimated maximum residue levels of 0.03 mg/kg, HR value of 0.026 mg/kg and STMR value of 0.018 mg/kg for mammalian fat except milk fat.

The Meeting estimated, at the LOQ of 0.01 mg/kg, maximum residue level of 0.01 mg/kg for mammalian meat and edible offal and 0.01 mg/kg for milk. The HR values for meat and edible offal are 0.002 mg/kg and 0.003 mg/kg, respectively

The Meeting estimated STMR values of 0.001 mg/kg for mammalian meat, 0.002 mg/kg for mammalian, edible offal of, and 0.002 mg/kg for milk.

The Meeting withdraws its previous recommendations for cattle meat, edible offal and milk.

Laying hens

Laying hens were dosed at nominal concentrations of 0, 2.5, 7.5 and 25 ppm levels for a period of 28 days. The fenpropathrin residues were below 0.01 mg/kg in case of dose groups 2.5 and 7.5 ppm over the study period. Eggs derived from 25 ppm dose contained 0.02 mg/kg fenpropathrin from day 7. Residues in muscle, gizzard and liver samples were below the LOQ of 0.01 mg/kg in all dose groups. The fenpropathrin residue in fat was 0.02, 0.05 and 0.14 mg/kg for dose groups of 2.5, 7.5 and 25 ppm. Metabolites could only be detected in liver after dosing with 25 ppm were TMPA (0.05 mg/kg) and PBA-glycin (0.03 mg/kg). The distribution of residues between white and yolk was not studied.

Taking into account that poultry feed is not treated with fenpropathrin according to the uses evaluated by the present Meeting, the Meeting estimated maximum residues levels in poultry meat, fat, edible offal and eggs of 0.01 mg/kg*.

The Meeting estimated STMR values of 0 for poultry products

The Meeting withdraws its previous recommendations for poultry products.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI assessment.

Definition of residue for compliance with MRL and for estimation of dietary intake for animal and plant commodities is fenpropathrin.

The residue in fat soluble.

CCN	Commodity Name	Recommended MRL, mg/kg		STMR/STMR-P mg/kg	HR/HR-P mg/kg
		New	Previous		
AM 0660	Almond hulls	10		3.1	3.6
MM 0812	Cattle meat	W	0.5 (fat)		
ML 0812	Cattle milk	W	0.1F		
MO 0812	Cattle, edible offal of	W	0.05		
FS 0013	Cherries	7 ^a		1.85	3.53
HS 0444	Peppers Chili, dried	10		2.59	4.9
FC 0001	Citrus fruits	2		0.02	0.098
OR 0001	Citrus oil, edible	100		16.5	
SB 0716	Coffee beans	0.03		0.01	
SO 0691	Cotton seed	W	1		
OC 0691	Cotton seed oil, crude	W	3		
MO 0105	Edible offal (mammalian)	0.01		0.002	0.003
VO 0440	Egg plant	W	0.2		
PE 0112	Eggs	0.01*	0.01*	0	
VC 0425	Gherkin	W			
FB 0269	Grapes	W	5		
MF 0100	Mammalian fats (except milk fats)	0.03		0.018	0.026
MM 0095	Meat (from mammals other than marine mammals)	0.01		0.001 0.018 (fat)	0.002
ML 0106	Milks	0.01		0.002	
FS 2001	Peaches (including Nectarine and Apricots)	3 ^a		0.71	1.1
VO 0051	Peppers	1		0.37	0.70
FS 0014	Plums (including prunes)	1		0.25	0.71
FP 0009	Pome fruits	3 ^a		0.73	2
PF 0111	Poultry fats	0.01*		0	0
PM 0111	Poultry meat	0.01* (fat)		0	0
PO 0111	Poultry, Edible offal of	0.01*	0.01*	0	0
DF 0014	Prunes	3		0.65	1.85
VD 0541	Soya bean (dry)	0.01		0.01	
FB 0275	Strawberry	2		0.515	1.2
DT 1114	Tea, Green, Black (black, fermented and dried)	3	2	0.14	
VO 0448	Tomato	1		0.19	0.64
TN 0085	Tree nuts	0.15		0.01	0.1
JF 0001	Citrus juice			0.007	
JF 0048	Tomato juice			0.023	
	Tomato canned			0.021	
VW 0448	Tomato paste			0.145	

DIETARY RISK ASSESSMENT

Long-term intake

The evaluation of fenpropathrin resulted in recommendations for MRLs and STMR values for 24 raw and processed commodities. Where data on consumption were available for the listed food commodities, dietary intakes were calculated for the 17 GEMS/Food Consumption Cluster Diets. The results are shown in Annex 3 to the 2014 Report.

The IEDIs in the seventeen Cluster Diets, based on the estimated STMRS were 1-10% of the maximum ADI (0.03 mg/kg bw). The Meeting concluded that the long-term intake of residues of fenpropathrin from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short-term Intake (IESTI) for fenpropathrin was calculated for 24 raw and processed commodities for which maximum residue levels and STMR values were estimated. The results are shown in Annex 4 to the 2014 Report.

For cherries, peaches, and pome fruits the IESTI represented 140%, 180% and 390% of the ARfD of 0.03 mg/kg bw, respectively. No alternative GAP was available. On the basis of information provided to the JMPR it was not possible to conclude that the estimate of short-term intake of fenpropathrin, from the consumption of cherries, peaches and pome fruits, was less than the ARfD.

The other commodities considered by the JMPR were within 0–80% of ARfD. The Meeting concluded that the short-term intake of fenpropathrin when used in ways that have been considered by the MPR is unlikely to present public health concern.

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FR-21-0344	Lai, J. C.	1990	Magnitude of the residue and residue reduction of fenpropathrin in fresh market and canned tomatoes. Chevron Chemical Company Project ID: TSR5680DAN, Sumitomo reference: FR-21-0344 GLP, Unpublished, 8 October 1990.
FR-21-0346	Lai, J. C.	1992	Addendum to Magnitude of the residues of fenpropathrin in strawberries. Valent Dublin Laboratory, CA, USA, Project ID: 1714/89/STBA, Sumitomo reference: FR-21-0346 GLP, Unpublished, 19 March 1992.
FA-41-0040	Lai, J.C., Fujie, G.H.	1984	Determination of fenpropathrin in crops : Method RM-22-4 Chevron Chemical Company, File No. 740/01 Danitol GLP, Unpublished, 10 October 1984.
1424604-2974-2002	Lavakumar, S., <i>et al.</i>	2003	Studies on the three season residues of Fenpropathrin 30% w/w EC in black tea and soil, International Institute of Biotechnology and Toxicology (IIBAT), Tamil Nadu, India, Study No. 04-2974-2002, Report No. 11861, Sumitomo reference: 1424604-2974-2002, GLP, Unpublished, 2003.
1424604-4190-2004	Lavakumar, S., <i>et al.</i>	2004	Studies on the residues of Fenpropathrin 30% w/w EC in tea and soil (fourth season). International Institute of Biotechnology and Toxicology (IIBAT), Tamil Nadu, India, Study No. 04-4190-2004; Sumitomo reference: 1424604-4190-2004, GLP, Unpublished, 2004.

Code	Author(s)	Year	Study Title
1424604-2974-2002	Lavakumar, S., <i>et al.</i>	2003	Studies on the three season residues of Fenpropathrin 30% w/w EC in black tea and soil, International Institute of Biotechnology and Toxicology (IIBAT), Tamil Nadu, India Study No. 04-2974-2002, Report No. 11861, GLP, Unpublished, Sumitomo reference: 1424604-2974-2002, 2003.
1424604-4190-2004	Lavakumar, S., <i>et al.</i>	2004	Studies on the residues of Fenpropathrin 30% w/w EC in tea and soil (fourth season). International Institute of Biotechnology and Toxicology (IIBAT), Tamil Nadu, India Study No. 04-4190-2004; Sumitomo reference: 1424604-4190-2004, GLP, Unpublished, 2004.
FR-0571	Lopez, N.M.R.	2013	Magnitude of residues of fenpropathrin after application of commercial product Danimen 300 EC in soya bean grains. Bioagri Laboratórios Ltda. Study No. 0035.034.137.12 Sumitomo reference: FR-0571, GLP, Unpublished, 29 April 2013.
FM-50-0044	Mikami, N. <i>et al</i>	1985	Metabolism of the Synthetic Pyrethroid Fenpropathrin in Plants. J. Agric. Food Chem., 33: 980-987. Sumitomo reference: FM-50-0044 Non-GLP, Published, 1985.
FM-3-0008	Mikami, N., Sakata, S., Yamada, Y. and Miyamoto, J.	1983	Degradation of fenpropathrin in soil. Sumitomo Chemical Company, Sumitomo reference: FM-30-0008. GLP, Unpublished. 1983.
FR-11-0345	Mitten, M. E.	1991	Magnitude of the residues of fenpropathrin in strawberries. Chevron Chemical Company, Project ID 1714/89/STB Sumitomo reference: FR-11-0345, GLP, Unpublished, 1991.
FR-61-0013	Noble, S.R.	1976	Soil percolation and aqueous die-away studies with WL 41706. Shell Research Report WKGR 0127 76, Sumitomo reference: FR-61-0013, Non-GLP, Unpublished, 1976.
FR-61-0014	Roberts, T.R. Standen, M.E	1976	The degradation of the insecticide WL 41706 in soil under laboratory conditions. Shell Research Report WKGR 0061 76 Sumitomo reference: FR-61-0014, Non-GLP, Unpublished.1976.
FR-0556	Samoil, K. S.	1999	Fenpropathrin: Magnitude of the residue on cucumber. IR-4 Project, USA, Study No. 02502, Sumitomo reference: FR-0556 GLP, Unpublished, 16 July 1999.
FR-0557	Samoil, K. S.	1999	Fenpropathrin: Magnitude of the residue on squash (summer). IR-4 Project, Study No. 02507, Sumitomo reference: FR-0557 GLP, Unpublished, 16 July 1999.
FR-0558	Samoil, K. S.	2001	Fenpropathrin: Magnitude of the residue on pepper. IR-4 Project, USA, Project ID: IR-4 Study No. 02503 Sumitomo reference: FR-0558, GLP, Unpublished, 22 June 2001.
FR-0560	Samoil, K. S.	2007a	Fenpropathrin: Magnitude of the residue on caneberry. IR-4 Project, USA, Report No. IR-4 PR No. 08735 Sumitomo reference: FR-0560, GLP, Unpublished, 2007.
FR-0561	Samoil, K. S.	2007b	Fenpropathrin: Magnitude of the residue on olive. IR-4 Project, USA, Report No. IR-4 PR No. 09374 Sumitomo reference: FR-0561, GLP, Unpublished, 2007.
FM-30-0010	Takahashi, N., Mikami, N.	1983	Hydrolysis of fenpropathrin in aqueous media. Sumitomo Chemical Company, Sumitomo reference: FM-30-0010 Non-GLP, Unpublished, 24 June 1983.
FM-50-0053	Takahashi, N., <i>et. al</i>	1985	Hydrolysis of the pyrethroid insecticide fenpropathrin in aqueous media, J. Pestic. Sci. 16: 113-18, Sumitomo reference: FM-50-0053 Non-GLP, Published.1985.
FM-30-0011	Takahashi, N., Mikami, N., Yamada, H. and Miyamoto, J.	1983	Photodegradation of fenpropathrin in water and on soil and plant foliage. Sumitomo Chemical Company, Sumitomo reference FM-30-0011.GLP, Unpublished. 1983.
FM-50-0055	Takahashi, N., Mikami, N., Yamada, H. and Miyamoto, J.	1985	Photodegradation of the pyrethroid insecticide fenpropathrin in water, on soils and on plant foliage. J. Pestic. Sci. 16: 119-31 Sumitomo reference: FM-50-0055.Non-GLP, Published. 1985.
FM-41-0067	Toia, R.F., Kimmel, E. C. <i>et al</i>	1994	A Metabolism Study with [Cyclopropyl-1- ¹⁴ C]-and [Phenyl-U- ¹⁴ C]-Fenpropathrin on Cotton. PTRL West Project 378,W Sumitomo reference: FM-41-0067, GLP, Unpublished, 3 October 1984.
FR-0570	Tomaz, M. L.	2010	Determination of residues of fenpropathrin in soya bean after application of Danimen 300 EC. Sumitomo Chemical Do Brasil Representações Ltda., Study No. 009 009 09 B, Sumitomo reference: FR-0570, GLP, Unpublished, 15 December 2010.

FLUOPICOLIDE (235)

see also DICHLOBENIL (274)

APPRAISAL

Fluopicolide was first evaluated for residues and toxicological aspects by the 2009 JMPR.

A residue definition of *fluopicolide* was established for compliance with the MRL for both plant and animal commodities, with a residue definition of *fluopicolide* and *2,6-dichlorobenzamide*, measured separately, for dietary risk assessment. A number of maximum residue levels were estimated by the 2009 Meeting.

Dichlobenil was evaluated by the present Meeting for both toxicological and residues aspects. The Meeting established an ADI of 0–0.05 mg/kg bw and ARfD of 0.3 mg/kg bw for 2,6-dichlorobenzamide. The only significant residue of dichlobenil in both plant and animal commodities was 2,6-dichlorobenzamide. The Meeting recommended a residue definition for dichlobenil of 2,6-*dichlorobenzamide* in plant and animal commodities, for both compliance with the MRL and dietary risk assessment.

The Meeting noted that 2,6-dichlorobenzamide residues can arise from use of either fluopicolide or dichlobenil, and that there were a number of crops for which there are fluopicolide use, but no dichlobenil use. The Meeting noted that maximum residue levels were required to accommodate residues of 2,6-dichlorobenzamide arising from use of fluopicolide. See dichlobenil.

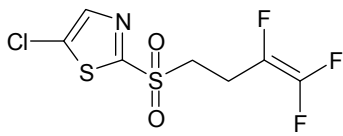
FLUENSULFONE (265)

The first draft was prepared by Dr Michael Doherty, United States Environmental Protection Agency, Washington, DC, USA

EXPLANATION

Fluensulfone is a non-fumigant, fluoroalkenyl nematicide used to control nematode pests in cucurbit vegetables, eggplant, peppers, and tomatoes. Fluensulfone shows activity in multiple nematode physiological systems; however, its specific nematicidal activity is not known at this time. Fluensulfone was considered for the first time for toxicology by the 2013 JMPR and for residues by the 2014 JMPR.

IDENTITY

ISO common name	Fluensulfone (provisionally approved)
Chemical Name	
IUPAC	5-chloro-1,3-thiazol-2-yl 3,4,4-trifluorobut-3-en-1-yl sulfone
CAS	5-chloro-2-[(3,4,4-trifluoro-3-buten-1-yl)sulfonyl]thiazole
CIPAC No.	Not Listed
CAS No.	318290-98-1
Synonyms	MCW-2
Structural Formula	
Molecular formula	C ₇ H ₅ ClF ₃ NO ₂ S ₂
Molecular mass	291.7

Physical and chemical properties

Physical and chemical properties of fluensulfone

Property	Guideline and method	Test material specification and purity	Findings	Reference
Technical Grade Active Ingredient				
Melting point (Technical material)	EEC A1 OECD 102 OPPTS 830.7200	Batch No.: 36372130-291-PF1 96.75%	Mean of two determinations by DSC: 34.8 °C ± 0.5 °C (308.0 K ± 0.5 K)	Weissenfeld M., 2009 R- 23310A
Boiling point (Technical material)	EEC A2 OECD 103 OPPTS 830.7220	Batch No.: 36372130-291-PF1 96.75%	No boiling point determined by DSC, as material decomposed without boiling. Atm. pressure = 99.6 kPa	Weissenfeld M., 2009a R-23310A
Decomposition temperature (Technical material)	EEC A2 OECD 103 OPPTS 830.7220	Batch No.: 36372130-291-PF1 96.75%	The test substance begins to decompose without boiling at 215 °C (capillary method). Atm. pressure = 99.6 kPa	Weissenfeld M., 2009a R-23310A

Property	Guideline and method	Test material specification and purity	Findings	Reference
Physical state and colour	OPPTS 830.6302 OPPTS 830.6303	Batch No.: 36372130-291-PF1 96.75%	Yellow, resin like solid.	Weissenfeld M., 2010 R-23307
Odour	OPPTS 830.6304	Batch No.: 36372130-291-PF1 96.75%	“Specific odour”	Weissenfeld M., 2010 R-23307
Solubility in organic solvents	EEC A6 OECD 105	Batch No.: 36372130-291-PF1 96.75%	Solubilities at 20 °C (shake flask method): Acetone 350.49 g/L Dichloromethane 306.14 g/L Ethyl acetate 350.76 g/L n-Heptane 19.01 g/L Methanol 359.29 g/L n-Octanol 90.42 g/L Xylene 356.18 g/L	Weissenfeld M., 2009d R-23317
Hydrolysis rate at pH 4, 7 and 9	OPPTS 835.2120 OECD Guideline 111.	[thiazole-4- ¹⁴ C]fluensulfone Radiochemical Purity 97.2%	After 5 days at 50 °C and pH 4, 5 and 7, the concentration of [thiazole- ¹⁴ C]fluensulfone comprised ≥ 96.8% of the applied dose in all samples.	Shepler, K 2010 R-23319
Pure Active Ingredient				
Melting, freezing or solidification point	EEC A1 OECD 102 OPPTS 830.7200	Batch No.: 381-022-02 99.1%	Mean of two determinations by DSC: 34.4 °C ± 0.2 °C (307.6 K ± 0.2 K)	Weissenfeld M., 2008a. R-23309
Boiling point	EEC A2 OECD 103 OPPTS 830.7220	Batch No.: 381-022-02 99.1%	Mean of two determinations by DSC: 282.5 °C ± 0.2 °C (555.7 K ± 0.2 K) Atm. pressure = 100.2 kPa	Weissenfeld M., 2008b R-23310
Temperature at which decomposition or sublimation occurs	EEC A2 OECD 103 OPPTS 830.7220	Batch No.: 381-022-02 99.1%	No decomposition or sublimation observed. Boiling point successfully determined.	Weissenfeld M., 2009a R-23310A
Relative density of purified active substance	EEC A3 OECD 109 OPPTS 830.7300	Batch No.: 381-022-02 99.1%	Relative density at 20 °C = 1.876 (Gas comparison pycnometer)	Weissenfeld M., 2008c R-23312
Vapour pressure of purified active substance	EEC A4 OECD 104	Batch No.: 381-022-02 99.1%	Vapour pressure results: 25 °C = 3.0×10^{-2} Pa 35 °C = 1.3×10^{-1} Pa 45 °C = 3.4×10^{-1} Pa (Gas saturation method) Vapour pressure extrapolated from vapour pressure curve was calculated to be 3.0×10^{-2} Pa at 25 °C	Weissenfeld M., 2008d R-23313
Henry's Law constant	Calculation	Batch No.: 381-022-02 99.1%	1.68×10^{-2} Pa m ³ /mol	Weissenfeld M., 2009b R-23313A
Physical state and colour	OPPTS 830.6302 OPPTS 830.6303	Batch No.: 381-022-02 99.1%	White, fine crystalline powder	Weissenfeld M., 2010 R-23307

Property	Guideline and method	Test material specification and purity	Findings	Reference
Odour	OPPTS 830.6304	Batch No.: 381-022-02 99.1%	“Specific odour”	Weissenfeld M., 2010 R-23307
Solubility in water	EEC A6 OECD 105 OPPTS 830.7840	Batch No.: 381-022-02 99.1%	Water solubility at 20 °C = 545.3 mg/L (shake flask method)	Weissenfeld M., 2008e R-23316
n-octanol/water partition coefficient	EEC A8 OECD 117 OPPTS 830.7570	Batch No.: 381-022-02 99.1%	Log P _{ow} = 1.96 (HPLC method)	Weissenfeld M., 2008f R-23318
	Based on solubilities	Not Applicable	Log P _{ow} based on individual solubilities in n-octanol and water: n-Octanol solubility: 90.42 g/L Water solubility: 0.545 g/L Log P _{ow} = Log (90.42/0.545) = 2.2	
Direct phototransformation of purified active substance in water	OPPTS 835.2240 OECD Proposal for a New Guideline Phototransformation of Chemicals in Water (2007)	[thiazole-4- ¹⁴ C]fluensulfone trifluorobutene-1,2- ¹⁴ C]fluensulfone	Aqueous photolysis may contribute to the rapid degradation of fluensulfone in the environment. DT ₅₀ irr. = < 11 hours of continuous sun test irradiation, when extrapolated to environmental conditions results in a DT ₅₀ < 1 day for all latitudes.	Schick, M 2011 R-23322

Fluensulfone is registered as an emulsifiable concentrate (EC) formulation containing 480 g ai/L.

METABOLISM AND ENVIRONMENTAL FATE

Metabolism and environmental fate studies were conducted with [thiazole-4-¹⁴C]fluensulfone and [trifluorobutene-1,2-¹⁴C]fluensulfone (Figures 1 and 2).

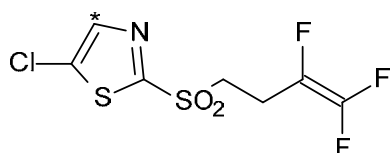


Figure 1 [thiazole-4-¹⁴C]fluensulfone

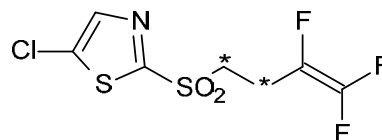
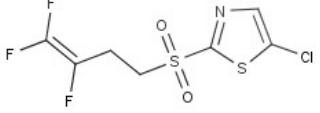
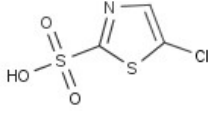
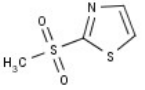
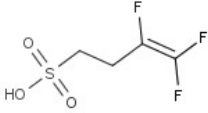
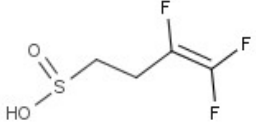
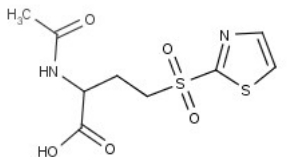
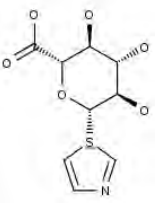


Figure 2 [trifluorobutene-1,2-¹⁴C]fluensulfone

Chemical names, structures, and code names of metabolites and degradation products of fluensulfone are shown below.

Known metabolites and degradation products of fluensulfone

Code Names	Chemical name, molecular formula, molar mass	Structure	Where found
Fluensulfone (parent compound) MCW-2	CAS: 5-chloro-2-(3,4,4-trifluoro- but-3-ene-1-sulfonyl)-thiazole CAS No. 318290-98-1 IUPAC: 5-chloro-1,3-thiazol-2-yl 3,4,4-trifluorobut-3-en-1-yl sulfone $C_7H_5ClF_3NO_2S_2$ 291.70 g/mol		Soil, poultry, rotational crops (minor)
M-3625 Thiazole sulfonic acid TSA	IUPAC: 5-chloro-1,3-thiazole-2- sulfonic acid $C_3H_2ClNO_3S_2$ 199.64 g/mol		Soil, crops, rotational crops, rat (minor)
M-3626 Thiazole methyl sulfone MeS	IUPAC: 2-methylsulfonyl-1,3- thiazole $C_4H_5NO_2S_2$ 163.22 g/mol		Soil, ruminant, rat (minor)
M-3627 Butene sulfonic acid BSA	IUPAC: 3,4,4-trifluorobut-3-ene- 1-sulfonic acid $C_4H_5F_3O_3S$ 190.14 g/mol		Soil, crops, rotational crops, rat (minor)
Butene sulfinic acid	IUPAC: 3,4,4-trifluorobut-3-ene- 1-sulfinic acid $C_4H_5F_3O_2S$ 174.14 g/mol		Rat, ruminant
thiazole mercapturate	IUPAC: 2-acetamido-4-(1,3- thiazole-2-sulfonyl)butanoic acid $C_9H_{12}N_2O_5S_2$ 292.33		Rat
MW-327-I and MW-327-II Thiazole glucuronides (α and β isomers)	Structure not specified $C_9H_{12}NO_6S$ 262.26		Rat

Animal Metabolism

The Meeting received metabolism studies on laboratory animals (2013 Meeting), lactating goats, and laying hens. Note that in the tables below, all data are reported to the level of precision specified in the study reports. All of the studies were conducted with fluensulfone which was radio-labelled,

separately, in the thiazole ring [Thiazole-4- ^{14}C ; Th- ^{14}C] and the ethane bridge between the sulfonyl and trifluorobutene moieties [Trifluorobutene-1,2- ^{14}C ; Bu- ^{14}C].

In animals, fluensulfone is cleaved at the sulfonyl bridge, apparently via glutathione conjugation, to yield products based on the thiazole and trifluorobutene halves of the parent molecule, with each half having the SO_x moiety. No parent fluensulfone was detected in any rat or lactating goat matrix, whereas parent fluensulfone was a major residue ($> 10\%$ of the total radioactive residue, TRR) in poultry fat. In both lactating goat and laying hen, radiolabel from fluensulfone was incorporated into natural products (lactose/glucose in the goat study; sugars, fatty acids, and proteins in the hen study).

Laboratory animals

Fluensulfone was rapidly absorbed at a dose level of 5 mg/kg bw, but the absorption phase was significantly longer at 500 mg/kg bw. The recovery of excreted radiolabel was 94–99% for individual rats. Radiolabel was found mainly in urine (71–83%), faeces (9–11%) and the cage wash (6–16%). One day after dosing with radiolabelled fluensulfone, an average of 0.6–0.8% of the administered radiolabel was in liver and 0.1% in kidney, with 1.7–2.1% in the carcass and 4.5–4.7% remaining in the gastrointestinal tract. By 7 days after treatment with radiolabelled fluensulfone, an average of 0.1–0.2% of the administered radiolabel remained in liver, and 0.03% in kidney.

The majority of the faecal radioactivity was not identified. It consisted of a number of peaks, each representing less than 2% of the administered dose; the only faecal metabolite representing more than 5% of the administered dose was thiazole sulfonic acid. In urine, there were no metabolites common to both radiolabels, which indicate that the initial step is cleavage at the sulfonyl bond. The initial reaction is probably with glutathione to release the trifluorobutene group and conjugation of the thiazole moiety.

Lactating goats

The metabolism of fluensulfone in lactating goats was investigated by LaMar and Quistad (2010, Study R-25458). For each radiolabel position, a single goat was dosed for five days at ca. 29 mg/day (equivalent to 10 ppm in the diet). Milk was collected twice daily and excreta were collected once daily throughout the study. Goats were sacrificed 20–22 hours after the final dosing, at which point tissues, bile, blood, and GI tract (with contents) were collected for analysis.

Total radioactive residues were determined by liquid scintillation counting (LSC) of solubilized tissues (liver, kidney, muscle, blood, bile, and fat), combusted samples (faeces, GI tract), or direct counting (urine). Samples of liver, kidney, and muscle were extracted with twice with acetonitrile:water (ACN:H₂O, 1:1, v/v) and once by ACN (neat) for extraction of residues for characterization and identification. Samples of flank muscle from the butene-label treated goat were additionally extracted with hexane:acetone (4:1, v/v) followed by acetone (neat) prior to alkaline

treatment of the post-extraction solids (PES). Residue analysis for these extracts was by high-performance liquid chromatography (HPLC) and, in some cases, thin-layer chromatography (TLC). For milk, the fat component was separated by centrifugation and extracted with acetone:hexane (1:4, v:v) and then once with acetone. The non-fat milk portion was extracted with acetone to precipitate milk proteins, which were then extracted with acetone:water (1:1, v:v). The resulting extract was added back to the skim milk for direct analysis by HPLC and TLC. Post-extraction solid fractions of all matrices except skim milk and poultry fat underwent further treatment, including alkaline digestion which was followed by acid digestion for liver and kidney (butene label only). Numerous HPLC systems with varying stationary and mobile phases were investigated to achieve satisfactory separation of the radiolabelled components, with detection by UV absorption or mass spectrometry (MS). Identification of metabolites was by co-chromatography with known standards and/or by mass spectral analysis.

The total recovery of radioactivity was 67 and 87% of the AD for the thiazole- and butene-labels, respectively. Low total recovery could be attributed to conversion of radiolabelled material to $^{14}\text{CO}_2$ and the fact that the entire carcass was not analysed for radioactivity. Most of the radioactivity was recovered in urine (38–70% of the AD). The thiazole label excretion in urine was only 53% that of the butene label. Faeces and the gastrointestinal tracts, at sacrifice, contained 14–19% of the AD. Radioactivity in tissues and body fluids accounted for 11% (thiazole-radiolabel) and 3.5% (butene-radiolabel) of the administered dose (Table 1). For both radiolabel positions, residues appeared to plateau in skim milk and reach a steady state of excretion by Day 2 or Day 3 (Tables 2 and 3). It is unclear whether or not residues in milk fat reached a plateau during the treatment phase of the study.

The TRR levels were highest in the liver (0.87–1.6% of AD), followed by kidney (0.1–0.2% of AD). TRR levels in thiazole-labelled muscle samples were about 4 times those found in the butene-labelled muscle samples (ca. 0.22–0.24 mg fluensulfone eq/kg vs. ca. 0.04–0.05 mg fluensulfone eq/kg). Muscle contained 4.2% of the AD for the thiazole label and ca. 1% of the AD for the butene label. Fat had low levels of radioactivity (ca. 0.07–0.13 mg fluensulfone eq/kg (thiazole label) and 0.04–0.07 mg fluensulfone eq/kg (butene label)). Milk contained 1.7% (thiazole label) and 1.1% (butene label) of the administered dose with the thiazole label being excreted at a higher rate than the butene label.

Fluensulfone and the known plant metabolites (i.e. methyl sulfone, thiazole sulfonic acid and butene sulfonic acid) were not identified in any of the goat milk or tissue samples. Thiazole methyl sulfone (25% TRR) and butene sulfinic acid (66% TRR) were identified in urine as major metabolites. No other fluensulfone-related ^{14}C -residues could be identified in any other matrix. Major radiolabelled residues were extensively broken down and appeared to be incorporated into natural products.

Lactose and glucose accounted for the majority of the radioactive residues in liver (ca. 8–9% of the TRR). The majority of the radioactivity, extractable by strong alkaline treatment (24% KOH), was assumed to be composed of a range of other sugars and natural products. Lactose was also the

major residue in skim milk (ca. 46–63% of the TRR). The majority of the remaining radioactivity (31–38% of the TRR) in both skim milk and liver was associated with proteins and amino acids. Radiolabelled glucose was also seen in kidney (13–17% of the TRR). Radioactivity associated with proteins in kidney accounted for 25–36% of the TRR.

Incorporation of radiolabelled residues into triglycerides was apparent in all fat matrices. In milk fat, radioactivity associated with fatty acids accounted for 8 and 31% of the TRR for the butene and thiazole labels, respectively. Fatty acid-radiolabel association in the remaining matrices was for subcutaneous fat (15 and 52% of the TRR), renal fat (24 and 21% of the TRR) and omental fat (13 and 43% of the TRR) for the thiazole and butene labels, respectively. Association of thiazole and butene labels with proteins was 5 and 34% of the TRR in milk fat and 5 and 15% of the TRR in subcutaneous fat.

Radiolabelled residues were also examined in blood with relation to bovine haemoglobin. It was apparent that radioactivity was associated with the globin-protein in haemoglobin in blood.

Table 1 Total radioactive residues (TRRs) of [^{14}C]fluensulfone in tissues, body fluids and excreta

Matrix	[Thiazole-4- ^{14}C]Fluensulfone		[Trifluorobutene-1,2- ^{14}C]Fluensulfone	
	mg eq/kg*	% of admin. dose	mg eq/kg*	% of admin. dose
Tissues and Body Fluids				
Liver	2.623	1.67	0.975	0.87
Kidney	1.402	0.20	0.671	0.10
Skim Milk	0.512	1.40	0.297	0.94
Milk Fat	1.977	0.31	0.531	0.12
Flank Muscle	0.217	Not Reported	0.054	Not Reported
Loin Muscle	0.239	4.2 ^a	0.040	0.93 ^a
Subcutaneous Fat	0.131	0.01 ^b	0.071	0.02 ^b
Omental Fat	0.071	0.04	0.070	0.03
Renal Fat	0.083	0.06	0.043	0.02
Bile	1.412	0.02	0.082	0.00
Blood	0.948	2.76 ^c	0.146	0.47 ^c
Excreta				
Faeces	–	15.66	–	12.05
Gastrointestinal Tract	–	2.93	–	2.08
Urine	–	37.49	–	69.66
Cage Wash	–	0.04	–	0.10
Total Recovery	–	66.85	–	87.39

^a Based on average ^{14}C content, muscle = 50% total goat weight (Luginbuhl, J.M.)

^b Subcutaneous fat was subsampled and does not accurately represent %AD

^c Based on blood = 1/12 total goat weight (Haenlein, G.F.W.)

*Values in mg eq/kg taken from extraction data (sum of fractions) except bile (solubilisation) and blood (combustion)

Table 2 Time course of total radioactive residues (TRRs) of [thiazole-4- ^{14}C]fluensulfone in milk and excreta

		Skim milk		Milk fat		Urine	Faeces	Total
Sampling Time		mg eq/kg	% of AD	mg eq/kg	% of AD	% of AD	% of AD	% of AD
Day 1	AM	n.d.	–	n.d.	–	0	0	0.08
	PM	0.287	0.07	0.526	0.01			
Day 2	AM	0.29	0.11	0.725	0.06	4.73	2.97	7.99

		Skim milk		Milk fat		Urine	Faeces	Total
Sampling Time		mg eq/kg	% of AD	mg eq/kg	% of AD	% of AD	% of AD	% of AD
	PM	0.455	0.1	1.037	0.02			
Day 3	AM	0.397	0.17	1.179	0.05	7.47	3.73	11.58
	PM	0.546	0.13	1.57	0.03			
Day 4	AM	0.462	0.19	1.728	0.03	8.74	2.44	11.56
	PM	0.631	0.13	2.155	0.03			
Day 5	AM	0.436	0.18	1.863	0.03	8.72	3.03	12.05
	PM	0.323	0.07	1.427	0.02			
Day 6	AM	0.512	0.24	1.977	0.04	7.83	3.49	11.6
Total		1.71% (whole milk)				37.49	15.66	54.86

n.d. = not detected.

Table 3 Time course of total radioactive residues (TRRs) of [trifluorobutene-1,2-¹⁴C]fluensulfone in milk and excreta

		Skim milk		Milk fat		Urine	Faeces	Total
Sampling time		mg eq/kg	% of AD	mg eq/kg	% of AD	% of AD	% of AD	% of AD
Day 1	AM	n.d.	—	n.d.	—	0	0	0.12
	PM	0.310	0.11	0.454	0.01			
Day 2	AM	0.122	0.07	0.390	0.01	12.65	2.95	15.80
	PM	0.384	0.11	0.655	0.01			
Day 3	AM	0.152	0.09	0.457	0.01	14.86	2.38	17.48
	PM	0.393	0.12	0.694	0.02			
Day 4	AM	0.145	0.08	0.458	0.01	13.69	1.99	15.91
	PM	0.369	0.12	0.748	0.02			
Day 5	AM	0.119	0.06	0.452	0.01	14.72	2.33	17.15
	PM	0.081	0.02	0.257	0.01			
Day 6	AM	0.297	0.16	0.531	0.02	13.74	2.40	16.32
Total		1.06% (whole milk)				69.66	12.05	82.78

n.d. = not detected.

Table 4 Summary of extraction of radioactive residues from the fluensulfone goat metabolism study

	TRR (mg eq/kg)		% TRR					
			ACN:H ₂ O or Acetone:Hexane		0.1 M KOH 24% KOH 6 N HCl		PES	
Matrix	[Th- ¹⁴ C]	[Bu- ¹⁴ C]	[Th- ¹⁴ C]	[Bu- ¹⁴ C]	[Th- ¹⁴ C]	[Bu- ¹⁴ C]	[Th- ¹⁴ C]	[Bu- ¹⁴ C]
Muscle	0.22-0.24	0.04-0.05	36-37	43-53	54-56	26-30	7.8-9.2	16.7-17.5
Kidney	1.40	0.67	40	48	55	50	5.6	1.9
Liver	2.62	0.98	26	28	72	71	2.0	1.1
Skim milk ^a	0.41	0.26	54	64	37	33	9.0	3.5
Milk fat ^a	3.42	0.33	44	64	34	5	n.d.	n.d.
Subcutaneous fat	0.13	0.07	35	80	57	7	0.8	12.7
Renal fat	0.08	0.04	51	61	35	23	14.5	25.6
Omental fat	0.07	0.07	58	74	37	14	5.6	11.4

^a Milk from Day 6, AM.

n.d. = not detected.

Table 5 Characterization of radioactive residues in kidney

Kidney				
	[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
Fraction	mg eq/kg	%TRR	mg eq/kg	%TRR
TRR (Sum of Fractions)	1.402	100.0	0.671	100.0
Solvent Extractable (ACN:H ₂ O)	0.558	39.8	0.322	48.0
Glucose	0.236	16.8	0.090	13.4
Unknowns ^a	0.243	17.4	0.203	30.1
Digestion of PES				
0.1 M KOH	0.029	2.1	0.029	4.3
24% KOH	0.736	52.5	0.137	20.4
6 N HCl (Rinse & Reflux)	–	–	0.170	25.3
Combined Extracts ^b Acidified and Partitioned with EtAc				
Aqueous Phase	0.689	49.1	0.294	43.8
Unknowns ^c	0.689	49.1	0.294	43.8
EtAc Phase	0.076	5.4	0.042	6.3
Unknowns ^d	0.051	3.7	0.023	3.3
PES	0.079	5.6	0.01	1.9

^a For the thiazole label, there were at least six unknowns which each accounted for 1.1–9.5% of the TRR. For the butene label, there were at least seven unknowns which each accounted for 1.8–9.8% of the TRR.

^b For the thiazole label, combined extracts are 0.1 M KOH and 24% KOH extracts. For the butene label, combined extracts are 0.1 M KOH, 24% KOH and 6 N HCl.

^c For the thiazole label, there were at least three unknowns which each accounted for 7.7–26.0% of the TRR. For the butene label, there were at least four unknowns which each accounted for 1.8–30.7% of the TRR. Further work indicates that these were likely to be natural components.

^d For the thiazole label, there were at least five unknowns which each accounted for 0.2–2.4% of the TRR. For the butene label, there were at least five unknowns which each accounted for 0.3–1.8% of the TRR.

Table 6 Characterization of radioactive residues in liver

Liver				
	[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
Fraction	mg eq/kg	%TRR	mg eq/kg	%TRR
TRR (Sum of Fractions)	2.623	100.0	0.975	100.0
Solvent Extractable	0.673	25.7	0.276	28.3
Glucose	0.232	8.8	0.039	4.0
Lactose	0.208	7.9	0.036	3.7
Unknowns ^a	0.164	6.2	0.123	12.6
Digestion of PES				
0.1M KOH Extract	0.028	1.1	0.042	4.3
24% KOH Extract	1.328	50.6	0.483	49.5
6 N HCl Extract	0.542	20.7	0.163	16.7
Acidification of Combined Extracts ^b & Partition with EtAc				
Aqueous Phase	1.733	66.1	0.636	65.2
Unknowns ^c	1.711	65.3	0.566	58.2
EtAc Phase	0.165	6.3	0.052	5.3
Unknowns ^d	0.122	4.6	0.032	3.2
PES	0.052	2.0	0.011	1.1

^a For thiazole labelled liver, there were at least five unknowns which each accounted for 0.6–1.8% of the TRR. For butene labelled liver, there were at least four unknowns which each accounted for 0.8–5.0% of the TRR.

^b Combined extracts are 0.1 M KOH, 24% KOH and 6 N HCl extracts.

^c For thiazole labelled liver, there were at least six unknowns which each accounted for 1.1–33.1% of the TRR. For butene

labelled liver, there were at least five unknowns which each accounted for 2.1–36.5% of the TRR. Further work indicates that these were likely to be natural components.

^d For thiazole labelled liver, there were at least four unknowns which each accounted for 0.3–2.4% of the TRR. For butene labelled liver, there were at least five unknowns which each accounted for 0.4–1.6% of the TRR.

Table 7 Characterization of radioactive residues in skim milk

	Skim Milk			
	[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
Fraction	mg eq/kg	%TRR	mg eq/kg	%TRR
TRR (Sum of Fractions)	0.409	100.0	0.260	100.0
Solvent extractable	0.221	54.0	0.166	63.8
Lactose	0.187	45.7	0.164	63.1
Unknowns ^a	0.022	5.4	0.002	0.8
Digestion of PES				
Protease	0.151	36.9	0.085	32.7
PES	0.037	9.0	0.009	3.5

^a For the thiazole label, there were two unknowns which each accounted for 2.2–3.2% of the TRR. For the butene label, there was one unknown.

Table 8 Characterization of radioactive residues in milk fat

	Milk Fat			
	[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
Fraction	mg eq/kg	%TRR	mg eq/kg	%TRR
TRR (Sum of Fractions)	3.416	100.0	0.330	100.0
Solvent extractable	1.489	43.6	0.212	64.2
Clean-up on Silica-Gel				
Hexane Fraction	0.004	0.1	0	0
EtAc:Hexane Fraction	1.446	42.3	0.206	62.4
EtAc Fraction	0.039	1.1	0.006	1.8
Saponification of EtAc:Hexane Fraction & Partition with Hexane:H ₂ O and Acidified DCM				
Hexane Phase	0.197	5.8	0.122	37.0
Aqueous Phase	0.181	5.3	0.059	17.9
DCM Phase	1.069	31.3	0.025	7.6
Digestion of PES				
Protease	1.165	34.1	0.015	4.5
24% KOH	0.219	6.4	0	0
Loss ^a	0.543	15.9	0.103	31.2
PES ^b	0	0	0	0

^a Difference in TRR. Loss assumed to be due to conversion to volatile ¹⁴C-residues.

^b No PES remained following digestion with 24% KOH.

Table 9 Characterization of radioactive residues in omental fat

	Omental Fat			
	[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
Fraction	mg eq/kg	%TRR	mg eq/kg	%TRR
TRR (Sum of Fractions)	0.071	100	0.070	100
Solvent extractable	0.041	57.7	0.052	74.3
Clean-up on Silica-Gel				
Hexane Fraction	0.009	12.7	0.044	62.9
EtAc:Hexane Fraction	0.006	8.5	0.005	7.1

Omental Fat				
		[Th- ¹⁴ C]		[Bu- ¹⁴ C]
Fraction	mg eq/kg	%TRR	mg eq/kg	%TRR
EtAc Fraction	0.026	36.6	0.004	5.7
Saponification of Hexane Fraction & Partition with (1) Hexane:H ₂ O and (2) acidified DCM				
Basic Hexane Phase	0	0	0	0
Aqueous Phase	0	0	0.014	20.0
DCM Phase	0.009	12.7	0.030	42.9
Digestion of PES				
0.1 M KOH	0.007	9.9	0.005	7.1
24% KOH	0.019	26.8	0.005	7.1
Combined Alkaline Extracts Acidified and Partitioned with EtAc				
Aqueous Phase	0.021	29.6	–	–
EtAc Phase	0.005	7.0	–	–
PES	0.004	5.6	0.008	11.4

Table 10 Characterization of radioactive residues in subcutaneous fat

Subcutaneous Fat				
		[Th- ¹⁴ C]		[Bu- ¹⁴ C]
Fraction	mg eq/kg	%TRR	mg eq/kg	%TRR
TRR (Sum of Fractions)	0.131	100.0	0.071	100.0
Solvent Extractable (Hexane:Acetone)	0.046	35.1	0.057	80.3
Aqueous Phase	0.010	7.6	0.004	5.6
Clean-up on Silica-Gel of Combined Organic Extracts				
Hexane Fraction	0.020	15.3	0.049	69.0
EtAc:Hexane Fraction	0.008	6.1	0.003	4.2
EtAc Fraction	0.018	13.7	0.005	7.0
Saponification of Hexane Fraction & Partition with (1) Hexane:H ₂ O and (2) Acidified DCM				
Basic Hexane Phase	0	0	0	0
Aqueous Phase	0	0	0.012	16.9
DCM Phase	0.020	15.3	0.037	52.1
Digestion of PES				
0.1 M KOH	0.019	14.5	0.005	7.0
24% KOH	0.055	42.0	–	–
Combined Alkaline Fractions Partitioned with EtAc				
Aqueous Phase	0.061	46.6	–	–
EtAc Phase	0.013	9.9	–	–
PES	0.001	0.8	0.009	12.7

Table 11 Characterization of radioactive residues in renal fat

Renal Fat				
		[Th- ¹⁴ C]		[Bu- ¹⁴ C]
Fraction	mg eq/kg	%TRR	mg eq/kg	%TRR
TRR	0.083	100.0	0.043	100.0
Solvent Extractable	0.042	50.6	0.022	51.2
Clean-up on Silica-Gel				
Hexane Fraction	0.020	24.1	0.014	32.6
EtAc:Hexane Fraction	0.006	7.2	0.004	9.3
EtAc Fraction	0.016	19.3	0.004	9.3
Saponification of Hexane Fraction & Partition with (1) Hexane:H ₂ O and (2) Acidified DCM				
Basic Hexane Phase	0	0	0.002	4.7
Aqueous Phase	0	0	0.003	7.0
DCM Phase	0.020	24.1	0.009	20.9
Digestion of PES				
0.1 M KOH	0.008	9.6	0.004	9.3
24% KOH	0.021	25.3	0.006	14.0
PES	0.012	14.5	0.011	25.6

Table 12 Characterization of radioactive residues in muscle

Fraction	Flank Muscle				Loin Muscle			
	[Th- ¹⁴ C]		[Bu- ¹⁴ C]		[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
TRR (Sum of Fractions)	0.217	100.0	0.054	100	0.239	100.0	0.040	100.0
Solvent extractable								
ACN:H ₂ O Fraction	0.079	36.4	0.023	42.6	0.087	36.4	0.021	52.5
Unknowns ^a	0.073	33.6	0.018	33.4	0.078	32.7	0.023	57.5
Hexane:Acetone	—	—	0.008	14.8	—	—	—	—
Digestion of PES								
0.1 M KOH	0.007	3.2	0.003	5.6	0.009	3.8	0.002	5.0
24% KOH	0.114	52.5	0.011	20.4	0.121	50.6	0.010	25.0
Combined Alkaline Extracts Acidified and Partitioned with EtAc								
Aqueous Phase	0.111	51.2	0.012	22.2	0.119	49.8	0.010	25.0
Unknowns ^b	0.088	40.4	—	—	0.089	37.2	—	—
EtAc Phase	0.010	4.6	0.002	3.7	0.011	4.6	0.002	5.0
PES	0.017	7.8	0.009	16.7	0.02	9.2	0.007	17.5

^a Flank muscle: thiazole = six unknowns, 3.2–12.4% TRR; butene = five unknowns, 3.7–13.0% TRR. Loin muscle:

thiazole = six unknowns, 1.7–20.1% TRR; butene = eight unknowns, 2.5–40.0% TRR. Further work indicates that these were likely to be natural components.

^b Flank muscle: thiazole = six unknowns, 1.8–23.0% TRR. Loin muscle: thiazole = six unknowns, 2.5–20.1% TRR.

In summary, fluensulfone was not a significant residue in goat tissues or milk. Initially the parent compound is cleaved, displacing butene sulfinic acid which is readily excreted in the urine. However, excretion of the butene half of the molecule is not fully effective and the butene moiety is likely broken down to carbon dioxide and enters the normal metabolic pathways. The thiazole half of the molecule is converted to the thiazole methyl sulfone for excretion, but is not excreted as quickly. Therefore, a much larger portion of the thiazole moiety is incorporated into natural products much in the same way as the butene label. Incorporation of radiolabelled residues into glucose and eventually into lactose takes place mainly in the liver, but is also seen in the kidneys. Lactose is then exported to be excreted in milk. Through the citric acid cycle (i.e. oxaloacetate) radiolabelled residues can also be converted and incorporated into fatty acids and a variety of amino acids (e.g. aspartate) then eventually into triglycerides and proteins.

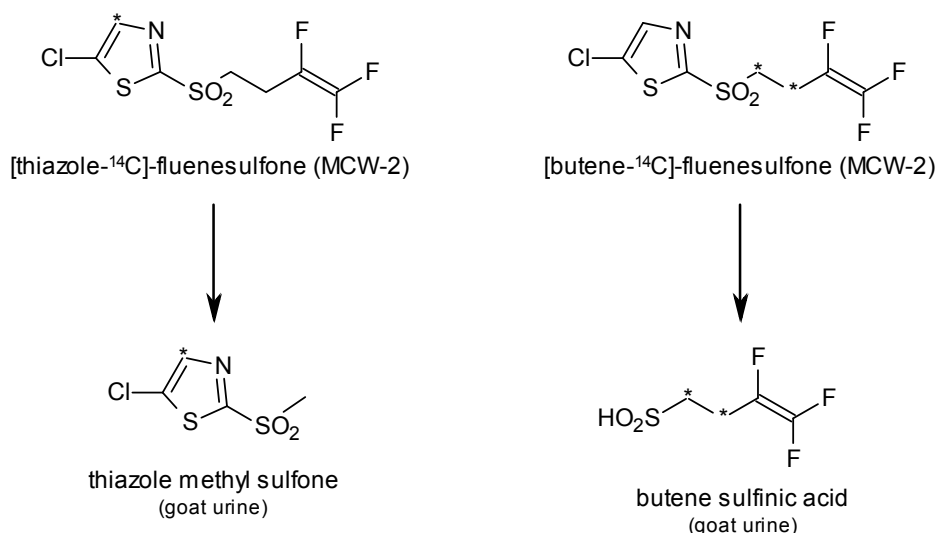


Figure 3 Proposed metabolic pathway of fluensulfone in lactating goat

Laying hens

The metabolism of fluensulfone in laying hens was investigated by LaMar and Quistad (2010, Study R-25454). For each radiolabel position, a group of ten hens was dosed for seven consecutive days at ca. 1.3 mg/bird/day (equivalent to 9.8 ppm in the diet). Eggs were collected twice daily and excreta were collected once daily throughout the study. Hens were sacrificed 20–22 hours after the final dosing, at which point tissues and GI tract (with contents) were collected for analysis.

Tissues, including GI tract with contents, were each homogenized in the presence of dry ice. Total radioactive residues were determined by LSC of solubilized tissues (liver, muscle, and fat) or following combustion (faeces, GI tract). Samples of liver, eggs, and muscle were extracted with twice with ACN:H₂O (1:1, v/v) and once by ACN (neat) for extraction of residues for characterization and identification. Samples of fat were extracted twice with hexane:acetone (4:1, v/v) followed by acetone (neat). Following the extraction procedures, all samples underwent alkaline treatment (KOH in MeOH/H₂O) of the post-extraction solids (PES). Residue analysis for the various extracts was by high-performance liquid chromatography (HPLC) and/or thin-layer chromatography (TLC). Numerous HPLC systems with varying stationary and mobile phases were investigated to achieve satisfactory separation of the radiolabelled components, with detection by UV absorption or refractive index. Identification of metabolites was by co-chromatography with known standards.

In the treated laying hens, 80% (thiazole label) and 79% (butene label) of the AD were recovered. Most of the administered dose was recovered in the excreta and gastrointestinal tracts (76–80%; Table 13). Butene radiolabelled matrices contained consistently higher ¹⁴C-residues when compared to the thiazole radiolabel. For example, only 0.15% of the AD for the thiazole radiolabel was excreted in eggs compared to 1.7% for the butene radiolabel. Differences in the amount of total

radioactive residues between the two radiolabels can be attributed to increased incorporation into natural products for the butene label. Thiazole methyl sulfone and butene sulfonic acid were both identified in the excreta. For both radiolabel positions, residues in eggs steadily increased from Day 1 to Day 8 and did not plateau within the dosing period (Table 14).

In fat samples, solvent extraction (acetone:hexane) released 81–95% of the TRR. Solvent extraction in liver, egg, and muscle was not as efficient, releasing approximately 13–34% of the total radioactivity from the matrix of interest. Use of basic and/or acidic conditions was required to accomplish more quantitative extraction (Table 15).

The TRRs were highest for liver (thiazole label: 0.64 mg eq/kg; butene label: 1.4 mg eq/kg) and egg (thiazole label: 0.29 mg eq/kg; butene label: 4.06 mg eq/kg). Thiazole-labelled omental and subcutaneous fats (0.04 mg eq/kg and 0.08 mg eq/kg, respectively), contained substantially lower radioactivity compared to butene radiolabelled fat (both 0.31 mg eq/kg). Breast and thigh muscle had the lowest amount of radiolabel in comparison to other tissues; however, butene radiolabelled muscle contained significantly higher residues (0.13 mg eq/kg for both thigh and breast muscles), compared to thiazole radiolabelled muscle (both 0.04 mg eq/kg). No tissues other than liver accounted for more than 0.1% of the AD.

Fluensulfone was detected in omental and subcutaneous fat, (0.009 mg/kg and 0.04 mg/kg for thiazole label, 0.02 mg/kg and 0.04 mg/kg for butene label). Trace amounts of fluensulfone may have been present in other matrices but could not be confirmed due to low levels. With the exception of thiazole sulfonic acid in liver (0.02 mg/kg), no other metabolites of fluensulfone were identified in eggs or tissues. Thiazole methyl sulfone and butene sulfonic acid were identified in faeces. The majority of the radioactivity appeared to be incorporated into natural products such as natural sugars (in liver) fatty acids (in eggs, fat and liver) and in proteins. Comparison of extraction of radioactivity from liver samples treated with and without protease enzyme indicates that ca. 0.16 mg/kg (24% TRR) is associated with proteins and/or amino acids. Incorporation of the radioactivity into triglycerides was noted in both fat matrices and in eggs, and accounted for 7% and 27% of the TRR for the thiazole and butene labels, respectively, in eggs and for 7–12% and 79–87% of the TRR for the thiazole and butene labels, respectively, in the fat matrices. In excreta, the identified residues were the methyl sulfone and BSA metabolites; parent fluensulfone was not observed. The tables below summarize the results of the poultry metabolism study.

Table 13 Total radioactive residues (TRRs) in tissues and excreta

Matrix	Thiazole-labelled Fluensulfone		Butene-labelled Fluensulfone	
	mg eq/kg	% of dose	mg eq/kg	% of dose
Tissues				
Liver	0.643	0.3	1.368	0.7
Eggs	0.286	0.15	4.064	1.71
Omental Fat	0.044	0.0	0.311	0.1
Subcutaneous Fat	0.075	0.0	0.311	0.0
Thigh Muscle	0.043	0.0	0.127	0.1

Matrix	Thiazole-labelled Fluensulfone		Butene-labelled Fluensulfone	
	mg eq/kg	% of dose	mg eq/kg	% of dose
Breast Muscle	0.043	0.0	0.117	0.1
Excreta				
Excreta	–	79.4	–	75.8
Gastrointestinal Tract	–	0.2	–	0.5
Total	–	80.1	–	79.0

Table 14 Total radioactive residues (TRRs) in eggs as function of time

Eggs		(Thiazole- ¹⁴ C)-Label		(Butene- ¹⁴ C)-Label	
		mg eq/kg	% of AD	mg eq/kg	% of AD
Day 1	AM ^a	not detected		not detected	
	PM	–		not detected	
Day 2	AM	0.009	0.00	0.006	0.00
	PM	–		0.050	0.00
Day 3	AM	0.019	0.01	0.218	0.11
	PM	–		0.294	0.01
Day 4	AM	0.029	0.02	0.342	0.17
	PM	–		0.661	0.03
Day 5	AM	0.041	0.02	0.486	0.24
	PM	–		–	
Day 6	AM	0.055	0.03	0.578	0.33
	PM	–		–	
Day 7	AM	0.061	0.03	0.684	0.37
	PM	–		–	
Day 8	AM	0.072	0.04	0.745	0.45
	PM	–		–	
Total		n.a.	0.15	n.a.	1.71

– = No egg production.

n.a. = Not applicable.

^a Pre-dosing.

Table 15 Extractability of TRRs in tissues

	TRR (mg eq/kg)		% TRR					
			ACN:H ₂ O or Acetone:hexane		0.1 M KOH 24% KOH 6 N HCl MeOH		PES	
Matrix	[Th- ¹⁴ C]	[Bu- ¹⁴ C]	[Th- ¹⁴ C]	[Bu- ¹⁴ C]	[Th- ¹⁴ C]	[Bu- ¹⁴ C]	[Th- ¹⁴ C]	[Bu- ¹⁴ C]
Liver	0.60	1.17	13	15	73	84	14	0.4
Egg ^a	0.07	0.68	34	5.2	59	67	0	0
Omental fat	0.04	0.31	81	95	n.d.	n.d.	19	5
Subcutaneous fat	0.08	0.32	83	94	n.d.	n.d.	16	7
Thigh muscle	0.04	0.12	29	17	49	65	22	18
Breast muscle	0.04	0.11	22	20	53	65	25	15

^a Day 8, morning collection

n.d. = No data

Table 16 Characterization of radioactive residues in liver

Fraction	Liver			
	[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
	mg eq/kg	%TRR	mg eq/kg	%TRR
TRR (by Sum of Fractions)	0.601	100.0	1.170	100.0
Solvent Extractable	0.078	13.0	0.180	15.4
Thiazole Sulfonic Acid	0.016	2.7	—	—
Unknowns ^a	0.030	5.0	0.164	14.1
Digestion of PES				
0.1 M KOH	0.045	7.5	0.059	5.0
24% KOH	0.334	55.6	0.703	60.1
6 N HCl	0.047	7.8	0.130	11.1
MeOH	0.012	2.0	0.093	7.9
Saponification of Combined Extracts ^b & Partition with (1) Hexane and (2) Acidic DCM				
Basic Hexane Phase	0	0	0	0
Acidic DCM Phase (Analysed by TLC)	0.064	10.6	0.068	5.8
Acidic Aqueous Phase	0.374	62.2	0.917	78.4
Unknowns ^c	0.264	43.9	0.801	68.4
Acidification of Combined Extracts & Partition with Ethyl Acetate				
EtAc Phase	0.203	33.8	0.248	21.2
Loss on Concentration	0.123	20.5	0.175	15.0
EtAc Phase (Analysed by HPLC)	0.080	13.3	0.073	6.2
Unknowns ^d	0.063	10.4	0.044	3.7
Acidic Aqueous Phase	0.235	39.1	0.737	63.0
PES	0.085	14.1	0.005	0.4

^a For the thiazole label, there were at least four unknowns with two having retention times corresponding to the MCW-2 and methyl sulfone metabolite regions. Each unknown accounted for 0.5–2.7% of the TRR. For the butene label, there were at least five unknowns with one having a retention time corresponding to the MCW-2 region. Each unknown accounted for 0.3–11.5% of the TRR.

^b Combined extracts are 0.1 M KOH, 24% KOH, 6 N HCl and MeOH Extracts.

^c For the thiazole label, there were at least six unknowns which each accounted for 3.5–13.1% of the TRR. For the butene label, there were at least 10 unknowns which each accounted for 3.7–22.6% of the TRR.

^d For the thiazole label, there were seven unknowns which each accounted for 0.7–3.2% of the TRR. For the butene label, there were at least seven unknowns which each accounted for 0.3–0.8% of the TRR.

Table 17 Characterization of radioactive residues in eggs

Fraction	Eggs (Day 8, AM)			
	[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
	mg eq/kg	%TRR	mg eq/kg	%TRR
TRR (by Sum of Fractions)	0.071	100.0	0.675	100.0
Solvent Extractable with ACN:H ₂ O	0.024	33.8	0.035	5.2
Hexane Phase (After Partition)	0.005	7.0	0	0
ACN:H ₂ O Phase	0.019	26.8	0.035	5.2
Unknowns ^a	0.014	19.7	0.034	4.9
Solvent Extractable with Hexane:Acetone	0.005	7.0	0.182	27.0
Hexane Phase	—	—	0	0
DCM Phase	—	—	0.164	24.3
Aqueous Phase	—	—	0.018	2.7
Digestion of PES				
0.1 M KOH Extract	0.005	7.0	0.058	8.6
24% KOH Extract	0.037	52.1	0.400	59.3
Acidification of Combined Extracts ^b & Partition with Ethyl Acetate				
EtAc Phase	0.020	28.2	0.202	29.9

Fraction	Eggs (Day 8, AM)			
	[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Unknowns ^c	0.009	12.6	–	–
Aqueous Phase	0.022	31.0	0.256	37.9
H ₂ O Rinse (Following C ₁₈ SPE)	0.011	15.5	0.081	12.0
MeOH Rinse (Following C ₁₈ SPE)	0.011	15.5	0.175	25.9
Unknowns ^d	–	–	0.172	25.4
PES ^e	0	0	0	0

^a For the thiazole label, there were at least seven unknowns which each accounted for 1.4–8.5% of the TRR. For the butene label, there were at least six unknowns which accounted for 0.1–1.8% of the TRR.

^b Combined extracts are the 0.1 M KOH and 24% KOH extracts.

^c For the thiazole label, there were four unknowns which each accounted for 1.4–5.6% of the TRR.

^d For the butene label, there were at least 10 unknowns which each accounted for 1.3–6.4% of the TRR.

^e Following extraction with KOH, no PES remained.

Table 18 Characterization of radioactive residues in omental fat

Fraction	Omental Fat			
	[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
	mg eq/kg	%TRR	mg eq/kg	%TRR
TRR	0.043	100.0	0.312	100.0
Solvent Extractable	0.035	81.4	0.297	95.2
Silica-Gel SPE				
Hexane Rinses	0.005	11.6	0.272	87.2
Combined Hexane: EtAc Eluates and ACN Phases	0.030	69.8	0.025	8.0
HPLC Analysis of the Combined Hexane: EtAc:ACN Fractions				
Fluensulfone	0.009	20.9	0.016	5.1
Unknowns ^a	0.020	46.6	0.002	0.6
Saponification of SPE Hexane Fraction and Partitioning with Hexane:H ₂ O and DCM				
Alkaline Hexane Phase	–	–	0.011	3.5
Acid DCM Phase	–	–	0.237	76.0
Acid Aqueous Phase	–	–	0.024	7.7
PES	0.008	18.6	0.015	4.8

^a For the thiazole label, there were at least two unknowns which each accounted for 4.7 or 41.9% of the TRR. For the butene label, there were at least two unknowns which each accounted for 0.3% of the TRR.

Table 19 Characterization of radioactive residues in subcutaneous fat

Fraction	Subcutaneous Fat			
	[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
	mg eq/kg	%TRR	mg eq/kg	%TRR
TRR (Sum of Fractions)	0.075	100.0	0.324	100.0
Solvent Extractable	0.062	82.7	0.303	93.5
Clean-up on Silica-Gel				
Hexane Rinsing	0.006	8.0	0.257	79.3
Hexane: EtAc:ACN Combined	0.056	74.7	0.048	14.8
HPLC				
Fluensulfone	0.041	54.7	0.037	11.4
M1	–	–	0.010	3.1
Unknowns ^a	0.013	17.4	–	–
Saponification of Hexane Fraction and Partitioning				
Alkaline Hexane Phase	–	–	0.013	4.0
Acid DCM Phase	–	–	0.231	71.3

Fraction	Subcutaneous Fat			
	[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Acid Aqueous Phase	–	–	0.014	4.3
Further Extraction of PES with ACN and ACN:H ₂ O	0.001	1.3	–	–
PES	0.012	16.0	0.021	6.5

^a For the thiazole label, there were at least three unknowns which each accounted for 2.7–12.0% of the TRR. For the butene label, there were no unknowns.

Table 20 Characterization of radioactive residues in muscle

Fraction	Thigh Muscle				Breast Muscle			
	[Th- ¹⁴ C]		[Bu- ¹⁴ C]		[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
TRR (Sum of Fractions)	0.041	100.0	0.121	100	0.036	100.0	0.111	100
Solvent extractable	0.012	29.3	0.020	16.5	0.008	22.2	0.022	19.8
Unknowns ^a	0.008	19.3	0.020	16.5	0.006	16.7	0.015	13.5
Digestion of PES								
0.1 M KOH Extract	0.003	7.3	0.007	5.8	0.002	5.6	0.007	6.3
24% KOH Extract	0.017	41.5	0.072	59.5	0.017	47.2	0.065	58.6
Acidification of Combined Extracts ^b & Partition with EtAc								
EtAc Phase	0.010	24.4	0.015	12.4	0.005	13.9	0.009	8.1
Unknowns ^c	0.010	24.4	–	–	–	–	–	–
Aqueous Phase	0.011	26.8	0.064	52.9	0.014	38.9	0.063	56.8
H ₂ O Rinse (Following C ₁₈ SPE)	–	–	0.019	15.7	–	–	0.019	17.1
MeOH Rinse (Following C ₁₈ SPE)	–	–	0.045	37.2	–	–	0.044	39.6
Unknowns ^d	0.010	24.4	0.041	33.8	0.014	38.8	0.035	31.5
PES	0.009	22.0	0.022	18.2	0.009	25.0	0.017	15.3

^a For the thiazole labelled thigh muscle, there were at least six unknowns with one having a retention time in the region of MCW-2. Each unknown accounted for 2.4–7.3% of the TRR. For the butene labelled thigh muscle, there were at least three unknowns which accounted each for 5.0–5.8% of the TRR. For the thiazole labelled breast muscle, there were at least six unknowns with one having a retention time in the region of MCW-2. Each unknown accounted for 0–11.1% of the TRR. For the butene labelled breast muscle, there were at least three unknowns which each accounted for 1.8–8.1% of the TRR.

^b Combined extracts are the 0.1 M KOH and 24% KOH extracts.

^c For the thiazole labelled thigh muscle, there was only one peak in the fraction.

^d For the thiazole labelled thigh muscle, there were at least four unknowns which each accounted for 2.4–9.8% of the TRR. For the thiazole labelled breast muscle, there were two unknowns each accounting for 19.4% of the TRR.

In summary, the poultry metabolism study indicates that the parent compound is cleaved displacing butene sulfinic acid, while the thiazole moiety is converted to the thiazole methyl sulfone and thiazole sulfonic acid. Both are eliminated in the excreta. Fluensulfone is broken down to carbon dioxide and subsequently enters normal metabolic pathways. Through the citric acid cycle, radiolabelled residues are converted and incorporated into fatty acids and a variety of amino acids, then eventually into triglycerides and proteins (as seen in fat and liver). The thiazole portion of fluensulfone is excreted at a slightly higher rate. The higher levels of butene radiolabel incorporation

into natural products can be mostly attributed to association with fatty acids, which is not as apparent with thiazole radiolabelled tissues.

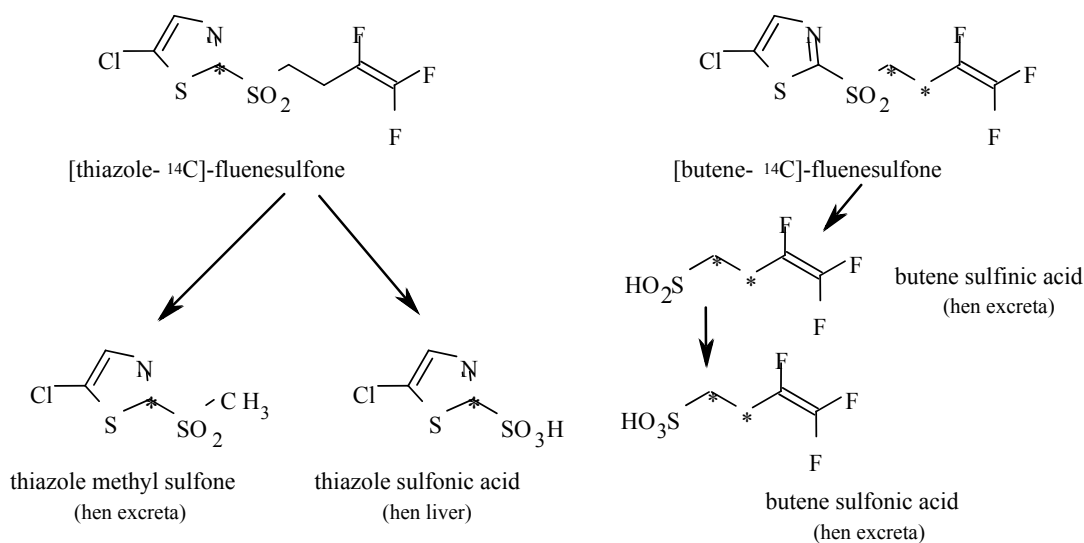


Figure 4 Proposed metabolic pathway of fluensulfone in laying hens

Plant metabolism

The Meeting received studies depicting the metabolism of fluensulfone in tomato, lettuce and potato. All of the studies were conducted with fluensulfone which was radiolabelled, separately, in the thiazole ring and the ethane bridge between the sulfonyl and trifluorobutene moieties.

Fluensulfone was extensively metabolised in all of the studies, with the only major residues being the BSA and TSA metabolites. A few chromatographic fractions had radioactivity in excess of 10% TRR. Investigation of these fractions indicated that the residues were associated with the BSA or TSA metabolites, as salts or other forms of the compounds.

Tomato

The metabolism of fluensulfone was investigated in tomato by Quistad and Bautista (2011, Report OR-25456). For each radiolabel position, an application of fluensulfone, formulated as an emulsifiable concentrate (48% ai), was made to soil at a rate of 4.1 kg ai/ha. Later that same day, tomato seedlings were planted into the treated soil.

Mature tomato fruits were harvested 87 days after treatment. Total radioactive residues were determined by combustion and LSC. Samples were extracted with ACN and ACN:H₂O followed by strong basic hydrolysis for residue characterization and analysis by TLC and/or HPLC. Residue identification was by co-chromatography of the available reference standards. Presence of the known metabolites BSA, MeS, and TSA was confirmed by HPLC-UV, HPLC-MS, and TLC. For unknown

metabolites, the extracts were cleaned up by solid-phase extraction (SPE) with silica-gel columns followed by analytical and semi-preparative HPLC followed by HPLC-MS. No storage stability determination was required as storage time after harvest did not exceed 18 days. Nevertheless a tomato sample containing thiazole labelled residues was reanalysed after 178 days of frozen storage. Fruit was extracted with ACN:H₂O in the same way as the initial fruit sample. HPLC analysis of the combined ACN:H₂O extracts confirmed the stability of the extracts under storage conditions.

Total radioactive residues were higher in samples from the [Bu-¹⁴C]fluensulfone treatment than from the [Th-¹⁴C]fluensulfone treatment (Table 21). Residues were < 0.001 mg eq/kg in the control sample. There was good agreement between total residues based on the sum of extracted fractions and total residues based on the combustion analysis.

Residues were readily extracted with ACN + ACN:H₂O (Table 22), with the follow-up alkaline extraction releasing only an additional 7–8% of the TRR. No fluensulfone was detected in the tomato samples. The identified residues were predominantly the BSA (42% TRR) and TSA (45% TRR) metabolites from the [Bu-¹⁴C] and [Th-¹⁴C] treatments, respectively (Table 23). From the [Th-¹⁴C] treatment, M3, the most abundant unknown metabolite, was considered to be due to the non-retention of TSA, which is a known phenomenon for sulfonic acids. This was confirmed by analysis with and without the presence of matrix which had an effect on peak shape and also by assessing the effects of column loading. Some inter-conversion between M1 and M3 was also noted during these experiments. Further investigation of M1 and M2 suggests that these compounds were salts of TSA. Unresolved radioactivity accounted for 22% of the TRR. From the [Bu-¹⁴C] treatment, Fraction F1-F2 in the combined ACN:H₂O extracts accounted for 27% of the TRR and was found to be mainly due to unretained BSA. Fractions F4 and F5 (2–5% of the TRR) were salts of BSA or related compounds. Unresolved radioactivity accounted for 14% of the TRR.

Table 21 Total radioactive residues (TRRs) in tomato following pre-plant application of [¹⁴C]fluensulfone

	TRR (mg eq/kg) ^a				
	[Th- ¹⁴ C]		[Bu- ¹⁴ C]		Untreated Control
Matrix	sum of fractions	by combustion	sum of fractions	by combustion	
Tomato Fruits	0.256	0.266	0.517	0.516	< 0.001

^a Values determined as sum of extracted and unextracted radiocarbon, except for untreated controls which were determined by combustion

Table 22 Extraction summary for tomato fruits after treatment with [¹⁴C]fluensulfone

	Radioactive Distribution				
	[Th- ¹⁴ C]		[Bu- ¹⁴ C]		
Fraction	mg eq/kg	% TRR	mg eq/kg	% TRR	
ACN:H ₂ O	0.227	88.67	0.472	91.30	
KOH (0.1 M)	0.009	3.52	0.016	3.09	
KOH (24%)	0.011	4.30	0.020	3.87	
PES	0.009	3.52	0.009	1.74	

Total	0.256	100.0	0.517	100.0

Table 23 Radiocarbon detected in tomato fruit following treatment with [^{14}C]fluensulfone

Metabolite/Fraction	Tomato Fruit			
	[Th- ^{14}C]		[Bu- ^{14}C]	
	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	0.256 (0.266) ^a	100.0	0.517 (0.516)	100.0
Solvent Extractable	0.227	88.7	0.472	91.3
Fluensulfone	—	—	—	—
TSA	0.116	45.4	—	—
M1—Form of TSA	0.009	3.5	—	—
M2—Form of TSA	0.007	2.7	—	—
M3—Form of TSA	0.037	14.5	—	—
M4	0.001	0.4	—	—
M8	< 0.001	0.1	—	—
BSA	—	—	0.215	41.6
F1-F2—Form of BSA	—	—	0.137	26.5
F4—Form of BSA	—	—	0.027	5.2
F5—Form of BSA	—	—	0.011	2.1
Unresolved	0.056	21.9	0.074	14.3

^a Values in parentheses determined by combustion

Lettuce

The metabolism of fluensulfone was investigated in lettuce by Quistad and Bautista (2011, Report R-25455). For each radiolabel position, an application of fluensulfone, formulated as an emulsifiable concentrate (48% ai), was made to soil at a rate of 4.1 kg ai/ha. Lettuce seeds were planted prior to application.

Samples of lettuce foliage were collected 49 days (immature) and 64 days (mature) after treatment. Within 23 days of harvest, the harvested lettuce samples were homogenized and the TRRs in each sample were determined by combustion analysis and LSC. Samples were extracted with ACN:H₂O and ACN, followed by strong basic hydrolysis. For thiazole-labelled samples, the extract was analysed by HPLC and TLC. For butene-labelled samples, the extract was filtered and the individual residues were then purified by two different HPLC methods. Purified metabolite (butene sulfonic acid) was then analysed via TLC and LC-MS.

Total radioactive residues were higher in samples from the [Th- ^{14}C]fluensulfone treatment than from the [Bu- ^{14}C]fluensulfone treatment (Table 24). Residues were < 0.001 mg eq/kg in the control sample. There was good agreement between total residues based on the sum of extracted fractions and total residues based on the combustion analysis, although the total by sum of fractions was consistently ca. 87% of that determined by combustion.

Residues were readily extracted with ACN + ACN:H₂O (Table 25), with the follow-up alkaline extraction releasing an additional 5–20% of the TRR. Trace levels of fluensulfone were found in the immature lettuce samples from both radiolabel positions. Major residues were the BSA (24%

TRR/38% TRR, immature/mature) and TSA (68% TRR/71% TRR, immature/mature) metabolites from the [Bu-¹⁴C] and [Th-¹⁴C] treatments, respectively (Table 26). From the [Th-¹⁴C] treatment, the sum of all other fractions (including unresolved) accounted for 24% of the TRR. Further analysis showed that the separated fractions were salts of TSA and/or other forms of TSA. M3 was found to be polar and due to unretained TSA. M1 was found to be a TSA salt or artifact due to chromatography. Metabolites M4, M5 and M6 were also chromatographic artefacts. Unresolved radioactivity accounted for 6–17% of the TRR. From the [Bu-¹⁴C] treatment, the sum of all other fractions (including unresolved) accounted for 46–52% of the TRR. Unresolved radioactivity accounted for 14–24% of the TRR. Further investigation of unidentified fractions showed that most of the fractions were salts or other forms of BSA and/or BSA bound to matrix compounds. Therefore, only BSA is present as a major metabolite with minor amounts of conjugated parent.

Table 24 Total radioactive residues (TRRs) in lettuce following one application of [¹⁴C]fluensulfone

	TRR (mg eq/kg) ^a				
	[Th- ¹⁴ C]		[Bu- ¹⁴ C]		
Matrix	sum of fractions	by combustion	sum of fractions	by combustion	Untreated Control
Immature Lettuce (49 DAT)	5.302	6.092	2.071	2.436	< 0.001
Mature Lettuce (69 DAT)	6.145	7.098	1.290	1.548	< 0.001

^a Values determined as sum of extracted and unextracted radiocarbon, except for untreated controls which were determined by combustion

Table 25 Extraction summary for lettuce after treatment with [¹⁴C]fluensulfone

	Radioactive Distribution							
	Immature Lettuce (49 DAT)				Mature Lettuce (69 DAT)			
	[Th- ¹⁴ C]		[Bu- ¹⁴ C]		[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
Fraction	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
ACN:H ₂ O	4.867	91.8	1.582	76.4	5.831	94.9	1.078	83.6
0.1 N KOH	0.201	3.8	0.156	7.5	0.153	2.5	0.095	7.4
24% KOH	0.209	3.9	0.273	13.2	0.145	2.4	0.103	8.0
PES	0.025	0.5	0.060	2.9	0.016	0.3	0.014	1.1
Total	5.302	100.0	2.071	100.0	6.145	100.0	1.290	100.0

Table 26 Metabolites detected in lettuce following treatment with [¹⁴C]fluensulfone

		Immature Lettuce				Mature Lettuce			
		[Th- ¹⁴ C]		[Bu- ¹⁴ C]		[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
Metabolite		mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	Sum of Fractions	5.30	100.0	2.071	100.0	6.15	100.0	1.29	100.0
	Combustion	6.09		2.436		7.10		1.55	
Solvent Extractable		4.867	91.8	1.582	76.4	5.831	94.9	1.078	83.6
Fluensulfone		0.009	0.2	0.008	0.4	—	—	—	—
TSA		3.572	67.5	—	—	4.34	70.6	—	—
M1—Form of TSA		0.390	7.4	—	—	0.138	2.2	—	—
M2—Form of TSA		0.372	7.0	—	—	0.191	3.1	—	—
M3—Form of TSA		0.091	1.7	—	—	0.024	0.4	—	—
M4		0.041	0.8	—	—	0.057	0.9	—	—

M5	0.050	0.9	—	—	—	—	—	—
M6	—	—	—	—	0.014	0.2	—	—
BSA	—	—	0.492	23.8	—	—	0.485	37.6
F1-F2—Form of BSA	—	—	0.161	7.7	—	—	0.094	7.3
F4—Form of BSA	—	—	0.104	5.0	—	—	0.019	1.5
F5	—	—	0.183	8.9	—	—	0.072	5.5
F6	—	—	0.227	11.0	—	—	0.054	4.2
F7	—	—	0.032	1.5	—	—	0.007	0.5
F11	—	—	0.035	1.7	—	—	0.008	0.6
F12	—	—	0.005	0.2	—	—	0.032	2.5
Unresolved	0.336	6.3	0.293	14.1	1.067	17.4	0.307	23.8

Potato

The metabolism of fluensulfone was investigated in potato by Quistad and Bautista (2011, Report R-25459). For each radiolabel position, an application of fluensulfone, formulated as an emulsifiable concentrate (48% ai), was made to soil at a rate of 4.0 kg ai/ha ([Th-¹⁴C]label) or 4.1 kg ai/ha ([Bu-¹⁴C]label). Potato seed pieces were planted prior to application.

Samples of potato tuber were collected 70 days (immature) and 106 days (mature) after treatment. Within 35 days of harvest, the harvested potato tuber samples were homogenized and the TRRs in each sample were determined by combustion analysis and LSC. Samples were extracted with ACN:H₂O and ACN, followed by strong basic hydrolysis ([Bu-¹⁴C] treatment only) and strong acid extraction (mature samples only). The initial organic extract was analysed by HPLC and TLC.

Total radioactive residues were higher in samples from the [Th-¹⁴C]fluensulfone treatment than from the [Bu-¹⁴C]fluensulfone treatment (Table 27). Residues were < 0.001 mg eq/kg in the control sample. There was good agreement between total residues based on the sum of extracted fractions and total residues based on the combustion analysis.

Residues were readily extracted with ACN + ACN:H₂O (Table 28), with the follow-up alkaline extraction releasing an additional 12–22% of the TRR from the [Bu-¹⁴C] treated samples. For the mature [Bu-¹⁴C] treated sample, the acid extraction released ca. 8% of the TRR. Trace levels of fluensulfone were found in the mature tuber samples from both radiolabel positions (however, this was not confirmed by TLC analysis). Major residues were the BSA (31% TRR/26% TRR, immature/mature) and TSA (63% TRR/65% TRR, immature/mature) metabolites from the [Bu-¹⁴C] and [Th-¹⁴C] treatments, respectively (Table 29). From the [Th-¹⁴C] treatment, the sum of all other fractions (including unresolved) accounted for 25–29% of the TRR. The most abundant unidentified fraction in the combined ACN:H₂O extracts was M3 and accounted for 3–7% of the TRR. Further examination showed this was a polar fraction which was considered to be an artifact resulting from the non-retention of TSA. A small amount of M1 was detected (< 2% of the TRR) which may be attributable to methyl sulfone, but this was not confirmed by TLC analysis. Unresolved radioactivity accounted for 20–21% of the TRR and was attributed to matrix-bound radioactivity causing smearing within the HPLC column or remaining in the pre-column. Further extraction of the PES (8% of the TRR) was not performed due to the low levels of radioactivity compared the amounts that had been

identified. From the [Bu-¹⁴C] treatment, the sum of all other fractions (including unresolved) accounted for 46–50% of the TRR. The most abundant unidentified fraction was the composed fraction F1-F2 in the combined ACN:H₂O extracts which accounted for 16–18% of the TRR. It was found to be mainly due to unretained BSA. Unresolved radioactivity (due to chromatographic artefacts) accounted for 28% of the TRR. Three other fractions F4, F5 and F9, containing < 1% of the TRR, were also resolved but no identification work was carried out due to the very low levels of radioactivity.

Table 27 Total radioactive residues (TRRs) in potato tubers following application of [¹⁴C]fluensulfone

	TRR (mg eq/kg) ^a				
	Thiazole Label		Butene Label		Untreated Control
Matrix	sum of fractions	by combustion	sum of fractions	by combustion	
Immature Potato Tubers (70 DAT)	0.335	0.324	0.225	0.222	< 0.001
Mature Potato Tubers (106 DAT)	0.467	0.436	0.163	0.168	< 0.001

^a Values determined as sum of extracted and unextracted radiocarbon, except for untreated controls which were determined by combustion

Table 28 Extraction summary for potato tubers after treatment with [¹⁴C]fluensulfone

	Radioactive Distribution (mg eq/kg)							
	Immature Potato Tubers (70 DAT)				Mature Potato Tubers (106 DAT)			
	[Th- ¹⁴ C]		[Bu- ¹⁴ C]		[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
Fraction	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
ACN:H ₂ O	0.308	91.94	0.173	76.89	0.428	91.65	0.129	79.14
0.1 N KOH	—	—	0.014	6.22	—	—	0.010	6.13
24% KOH	—	—	0.036	16.00	—	—	0.010	6.13
72% H ₂ SO ₄	—	—	—	—	—	—	0.013	7.98
PES	0.027	8.06	0.002	0.9	0.039	8.35	0.001	0.61
Total	0.335	100.0	0.225	100.0	0.467	100.0	0.163	100.0

Table 29 Radiocarbon detected in potato tubers treated with [¹⁴C]fluensulfone

		Immature Potato Tubers (70 DAT)				Mature Potato Tubers (106 DAT)			
		[Th- ¹⁴ C]		[Bu- ¹⁴ C]		[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
Metabolite/Fraction		mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
TRR	Sum of fractions	0.335	100.0	0.225	100.0	0.467	100.0	0.163	100.0
	Combustion	0.324		0.222		0.436		0.168	
Solvent Extractable		0.308	91.9	0.173	76.9	0.428	91.7	0.129	79.1
Fluensulfone		—	—	—	—	0.005	1.1	0.005	3.1
TSA		0.211	63.0	—	—	0.305	65.3	—	—
M1—Form of TSA		0.005	1.5	—	—	0.009	1.9	—	—
M3—Form of TSA		0.023	6.9	—	—	0.016	3.4	—	—
BSA		—	—	0.069	30.7	—	—	0.042	25.8
F1-F2—Form of BSA		—	—	0.037	16.4	—	—	0.029	17.8
F4—Form of BSA		—	—	< 0.001	< 0.1	—	—	0.002	1.2
F5		—	—	0.001	0.4	—	—	0.002	1.2
F9		—	—	0.002	0.9	—	—	0.002	1.2
Unresolved		0.069	20.6	0.064	28.4	0.093	19.9	0.046	28.2

Overall, the metabolism of fluensulfone in tomato, lettuce, and potato is similar and corresponds well with that observed in rotational crop (see below). An overall summary of the metabolic residue profile from the target crops studies is shown in Table 30, followed by the proposed pathway in Figure 5.

Table 30 Summary of results from metabolism studies with fluensulfone in tomato, lettuce, and potato

	Lettuce				Potato					
	Immature		Mature		Immature		Mature		Tomato	
Fraction/ Compound	% TRR	mg equiv./kg	% TRR	mg equiv./kg	% TRR	mg equiv./kg	% TRR	mg equiv./kg	% TRR	mg equiv./kg
^{[14} C-Butene] Fluensulfone										
TRR, mg/kg	2.4		1.6		0.22		0.17		0.52	
Extract	76	1.6	84	1.1	77	0.17	79	0.13	91	0.47
Parent	0.2	0.009	—	—	—	—	3.1	0.005	—	—
BSA ^a	60 (36)	1.2 (0.75)	60 (22)	0.77 (0.29)	47 (18)	0.11 (0.041)	44 (21)	0.071 (0.035)	68 (21)	0.35 (0.06)
TSA ^b	—	—	—	—	—	—	—	—	—	—
Other	14	0.29	24	0.31	30	0.068	32	0.052	22	0.11
Digest	21	0.43	15	0.20	22	0.05	20	0.033	7.0	0.036
PES ^c	2.9	0.06	1.1	0.014	0.9	0.002	0.61	0.001	1.7	0.009
^{[14} C-Thiazole] Fluensulfone										
TRR, mg/kg	6.1		7.1		0.32		0.44		0.27	
Extract	92	4.9	95	5.8	92	0.31	92	0.43	89	0.23
Parent	0.4	0.008	—	—	—	—	1.1	0.005	—	—
BSA ^a	—	—	—	—	—	—	—	—	—	—
TSA ^b	85 (18)	4.5 (1.4)	77 (6.6)	4.8 (0.41)	71 (8.4)	0.24 (0.028)	71 (5.3)	0.33 (0.025)	67 (21)	0.17 (0.06)
Other	6.3	0.34	17	1.1	21	0.069	20	0.096	22	0.056
Digest	7.7	0.41	4.9	0.30	—	—	—	—	7.8	0.02
PES	0.5	0.025	0.3	0.016	8.1	0.027	8.4	0.039	3.5	0.009

^a Includes fractions shown to be salts and/or chromatographic artefacts of BSA, which are shown parenthetically.

^b Includes fractions shown to be salts and/or chromatographic artefacts of TSA, which are shown parenthetically.

^c Post-extraction solids

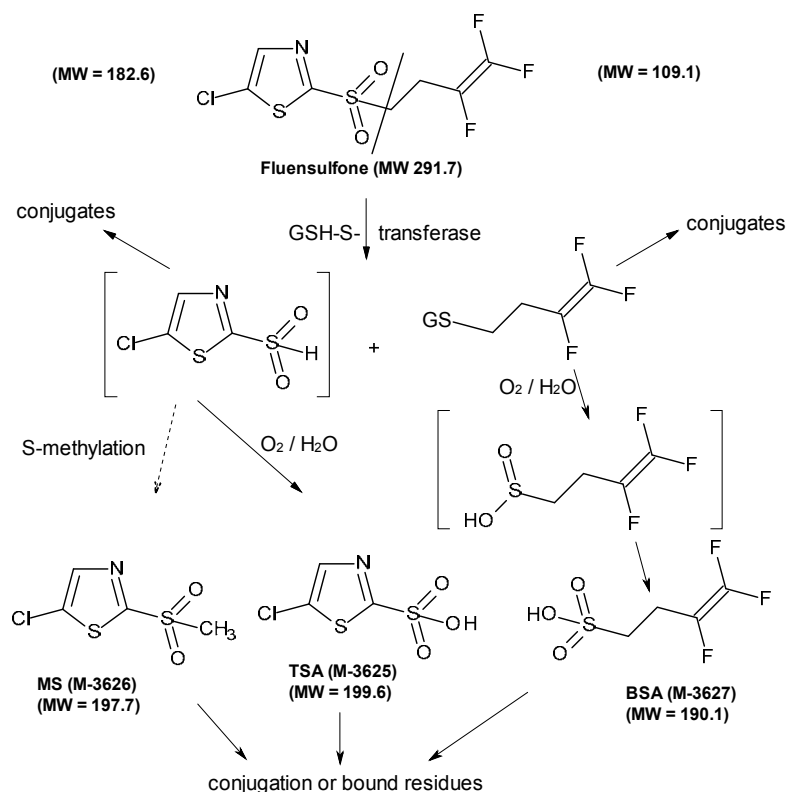


Figure 5 Proposed metabolic pathway of fluensulfone in target and rotational crops

Environmental fate in soil

The Meeting received studies depicting the photodegradation of fluensulfone on soil; aerobic soil metabolism of fluensulfone, TSA, and MeS; and a confined rotational crop study with radish, lettuce, and wheat.

Photolysis

Photolysis of fluensulfone on the surface of soil was investigated by Ponte (2012, Report R-23320) in a study consisting of a preliminary test phase and a definitive test phase. In both phases, [Bu-¹⁴C] or [Th-¹⁴C] labelled fluensulfone was applied to a medium textured sandy loam soil (Table 31) at a rate of 4 kg/ha. The soil was irradiated with artificial sunlight, continuously, for 14 days, corresponding to 32 days of natural sunlight at 38°N. Soils were maintained at 75% of field moisture capacity (at 1/3 bar) and sampled at various time points throughout the irradiation period. The experimental design included a trapping system to capture volatile organic compounds as well as CO₂. Volatilized radiocarbon was released from foam trap plugs by extraction with ACN. Soils were extracted with acetone:water (4:1, v/v). For each sample matrix, TRRs were determined by LSC of trap solution or extract. For determination of residues, soil extracts were analysed by HPLC-UV and co-chromatographed with known standards; confirmation of metabolites was by TLC.

Table 31 Characteristics of the Soil (Northwood, US) Used

Parameter (units)	Value
Source	Northwood, North Dakota, USA
pH in water	6.8
Cation Exchange Capacity (CEC, meq/100 g)	17
Organic Carbon (%)	2
% Moisture at 1/3 bar	21.9
% Moisture at 15 bar	14
% Moisture at 1/10 bar (pF 2.0)	29.3
Bulk Density (disturbed) (gm/cc)	1.02
Sand (%)	63
Silt (%)	16
Clay (%)	21
USDA Textural Classification	Sandy Loam
FAO Textural Class	Medium
Olsen Phosphorus (ppm)	11
Total Nitrogen (Analyser) (%)	0.16
Soluble Salts (mmhos/cm)	0.17
Base Saturation Data	
Cation	Percent / ppm
Calcium	63.2 / 2152
Magnesium	20.4 / 417
Sodium	0.3 / 13
Potassium	3.8 / 253
Hydrogen	12.2 / 21

In irradiated samples, fluensulfone levels decreased from their initial levels to 52% ([Bu-¹⁴C]) or 34% ([Bu-¹⁴C]) of the AR (Tables 32-34).

Table 32 Material balance and metabolism of [Th- and Bu-¹⁴C]fluensulfone soil photolysis (preliminary test)

	[Th- ¹⁴ C] (% AR)				[Bu- ¹⁴ C] (%AR)			
	Incubation Time In Days							
	0	3	6	6	0	3	6	6
Fraction/Identity		(Light)	(light)	(Dark)		(Light)	(Light)	(Dark)
Fluensulfone	92.1	78.4	59.5	84.6	92.8	70.5	62.3	88.3
Other HPLC peaks	0.0	0.4	0.9	0.0	0.6	0.6	0.1	0.4
Other volatiles in EG ^a	NA	1.5	3.5	0.8	NA	1.3	2.2	0.4
¹⁴ CO ₂	NA	6.5	12.3	2.6	NA	1.7	6.9	2.2
Non-extractables	3.2	10.3	16.8	4.9	6.2	21.1	20.1	6.1
Total	95.3	97.1	93.0	92.9	99.6	95.2	91.6	97.4
Mean total ± SD	94.6 ± 2				96 ± 3.4			

^a Ethylene glycol

Table 33 Material balance and metabolism of [Bu-¹⁴C]fluensulfone soil photolysis (definitive test)

	[Bu- ¹⁴ C] (%AR)					
	Incubation Time In Days					
Fraction/Identity	0	2	5	7	9	13
Fluensulfone	91.8	82.9	71.5	74.6	56.2	52.2
Others ^a	0.3	0.2	1.8	1.0	3.9	4.9
Non-extractables	3.7	12.2	20.2	16.0	16.5	19.4

EG Trap	NS	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
NaOH Trap	NS	1.1	< 1.8	3.1	8.2	8.4
ACN Trap	NS	0.6	< 0.9	0.7	2.5	2.6
Total	95.8	96.9	95.9	95.2	87.5	87.4
Mean total $\pm$ SD	93.1 $\pm$ 4.4					

NS = Not Sampled

^a Corresponds to at least six fractions.

Table 34 Material balance and metabolism of [Th-¹⁴C]fluensulfone soil photolysis (definitive test)

	[Th- ¹⁴ C] (%AR)		
	Incubation Time in Days		
Fraction/Identity	0	4	14
Fluensulfone	83.5	78.4	34.4
TSA	0.2	0.3	8.6
Others	0.7	0.8	1.1
Non-extractables	6.2	10.0	16.4
Foam plug trap	1.0	0.4	3.8
2 N H ₂ SO ₄ trap	0.5	0.1	2.7
NaOH trap	4.3	1.3	19.9
Total	96.3	91.3	86.9
Mean total $\pm$ SD	91.5 $\pm$ 4.7		

Aerobic soil metabolism (Fluensulfone)

The metabolism and degradation kinetics of fluensulfone in aerobic soils was investigated by Moser (2011, Report R-27436). In that study, [Bu-¹⁴C] or [Th-¹⁴C] labelled fluensulfone was applied at a rate of 4 kg/ha to six different soils (Table 35). The soils were incubated under dark, aerobic conditions at 20 °C for 120 days. Samples were collected at various time points throughout the study, extracted first with CaCl₂ and then with ACN:H₂O and analysed for residues by LSC, HPLC, and/or TLC. In addition, the Fislis soil underwent Soxhlet extraction and analysis by LSC and TLC.

Table 35 Characteristics of the soils used in the aerobic metabolism study for fluensulfone

Parameter	Value					
Name	Fislis	Hagenthal	Horn	Montesquieu	Senozan	Sevelen
pH (CaCl ₂)	6.75	7.13	7.23	7.36	7.54	7.36
Organic carbon %	2.13	1.65	2.36	1.5	0.61	1.61
CEC (meq/100 g soil)	22.99	18.79	21.72	21.09	13.63	9.09
Soil type	silt loam	silt loam	loam	clay loam	loam	sandy loam
Bulk density [kg/dm ³]	1.08	1.22	0.99	1.18	1.21	1.18
Particle size analyses (mm):						
0.002 (clay) [%]	26.42	24.42	25.11	38.70	20.58	9.51
0.002–0.05 (silt) [%]	65.57	53.51	36.22	31.84	35.25	36.96
0.05–2.0 (sand) [%]	8.01	22.07	38.67	29.46	44.17	53.53
Water holding capacity (g water/100 g soil):						
at pF 1.0	75.62	56.80	73.15	59.76	48.77	58.94
at pF 2.0	39.65	36.88	33.52	33.52	29.97	31.48
at pF 2.5	30.98	25.83	28.47	25.91	19.52	21.45
Biomass determination as mg C/100 g soil (%)						
pre-study	26.9 (1.3)	22.5 (1.4)	15.1 (0.6)	26.9 (1.8)	26.9 (4.4)	18.1 (1.1)
test initiation	28.0 (1.3)	22.5 (1.4)	39.1 (1.7)	22.5 (1.5)	17.0 (2.8)	22.5 (1.4)
mid-study	22.5 (1.1)	22.5 (1.4)	22.5 (1.0)	28.1 (1.9)	17.0 (2.8)	17.0 (1.1)
test end	18.1 (0.8)	28.0 (1.7)	33.6 (1.4)	17.0 (1.1)	17.0 (2.8)	22.5 (1.4)

Table 36 Distribution of applied radioactivity following application of [^{14}C]fluensulfone to six soils

Soil	Day	Percent of applied radioactivity, [Bu- ^{14}C] / [Th- ^{14}C]						
		CaCl ₂ extract	Organic extract	Total extractable	Soil bound	$^{14}\text{CO}_2$	VOC	Total recovered
Fisilis	0	18.8/24.1	68.8/68.5	87.6/92.6	9.8/4.6	NA/NA	NA/NA	97.4/97.2
	2	18.8/22.8	59.7/66.4	78.5/89.2	15.5/8.0	4.6/0.7	< 0.1/< 0.1	98.6/97.9
	7	15.6/27.2	51.5/59.9	67.1/87.1	19.0/12.3	0.6 ^a /1.9	< 0.1/< 0.1	86.7 ^a /101.4
	14	13.2/29.4	33.8/53.9	47.1/83.3	31.5/10.3	39.1/4.3	0.1/< 0.1	117.8 ^b /97.9
	21	11.3/30.5	31.8/50.9	43.1/81.4	29.9/11.3	1.1/5.3	< 0.1/< 0.1	74.1 ^a /98.1
	28	9.6/27.3	27.5/53.0	37.1/80.3	33.4/9.8	29.6/5.1	< 0.1/< 0.1	100.1/95.2
	50	6.8/33.0	12.8/45.3	19.7/78.2	37.8/12.7	44.3/7.4	< 0.1/< 0.1	101.8/98.3
	77	2.6/NS	3.2/NS	5.8/NS	43.9/NS	55.7/NS	< 0.1/NS	105.5/NS
	90	1.9/NS	3.0/NS	4.8/NS	38.8/NS	55.5/NS	< 0.1/NS	99.1/NS
	120	0.5/33.1	1.7/39.6	2.2/72.7	40.1/6.0	11.2/16.8	< 0.1/< 0.1	53.6 ^a /95.5
Hagenthal	0	28.4/30.2	61.8/64.6	90.2/94.9	6.5/3.9	NA/NA	NA/NA	96.7/98.7
	2	22.6/28.6	57.9/60.7	80.5/89.3	12.7/5.9	5.5/0.5	< 0.1/< 0.1	98.7/95.7
	7	18.4/32.8	43.2/55.3	61.5/88.1	23.7/10.4	12.5/1.7	< 0.1/< 0.1	97.8/100.2
	14	12.6/35.9	24.7/47.4	37.3/83.4	31.7/11.3	23.0/3.7	< 0.1/< 0.1	92.0/98.4
	21	15.2/35.4	25.9/45.2	41.0/80.6	26.7/10.5	28.2/5.8	0.1/< 0.1	96.0/96.9
	28	11.5/32.5	21.0/48.2	32.5/80.6	28.4/11.7	39.8/6.3	< 0.1/< 0.1	100.7/98.6
	50	9.2/38.9	10.8/36.3	20.0/75.2	30.1/12.0	36.5/7.1	< 0.1/< 0.1	86.6 ^a /94.3
	77	1.0/NS	1.7/NS	2.7/NS	38.5/NS	36.8/NS	< 0.1/NS	78.0 ^a /NS
	90	0.6/NS	1.2/NS	1.9/NS	37.7/NS	29.7/NS	< 0.1/NS	69.2 ^a /NS
	120	0.6/32.1	1.4/29.2	2.0/61.3	34.7/12.3	52.2/20.3	< 0.1/< 0.1	88.9 ^a /93.9
Horn	0	20.8/23.4	65.8/68.7	86.6/92.1	8.8/5.8	NA/NA	NA/NA	95.5/97.9
	2	17.1/22.7	57.5/66.0	74.6/88.7	18.0/10.9	5.1/0.1	< 0.1/< 0.1	97.7/99.6
	7	14.7/25.7	40.5/58.8	55.2/84.5	27.8/13.4	16.0/0.2	< 0.1/< 0.1	99.0/98.2
	14	10.8/29.9	40.5/50.7	51.4/80.6	28.1/14.4	15.0/0.3	< 0.1/< 0.1	94.6/95.3
	21	10.5/35.9	22.0/47.4	32.5/83.3	30.9/12.9	31.8/10.5	< 0.1/< 0.1	95.2/106.8
	28	7.3/27.4	21.0/51.0	28.3/78.4	34.9/13.3	36.9/8.4	< 0.1/< 0.1	100.2/100.2
	50	3.5/33.4	4.3/43.2	7.8/76.6	41.9/12.4	50.7/9.8	< 0.1/< 0.1	100.4/98.9
	77	0.4/NS	1.6/NS	2.0/NS	37.9/NS	40.9/NS	< 0.1/NS	80.8 ^a /NS
	90	1.3/NS	2.2/NS	3.5/NS	42.2/NS	51.7/NS	0.3/NS	97.6/NS
	120	0.4/27.7	1.6/38.3	2.0/66.0	32.7/14.4	54.6/20.6	< 0.1/< 0.1	89.3 ^a /101.0
Montesquieu	0	28.1/25.5	63.6/66.4	91.7/91.9	6.9/3.8	NA/NA	NA/NA	98.6/95.7
	2	23.5/27.5	59.0/63.4	82.5/90.8	11.8/6.9	2.3/0.4	< 0.1/< 0.1	96.6/98.1
	7	17.9/29.8	47.0/56.9	64.9/86.8	22.5/9.6	8.2/1.2	< 0.1/< 0.1	95.7/97.7
	14	18.2/32.8	36.4/51.4	54.6/84.1	28.3/11.6	15.7/3.4	< 0.1/< 0.1	98.6/99.2
	21	16.3/34.7	33.0/48.6	49.3/83.3	28.9/9.3	23.0/5.0	< 0.1/< 0.1	101.2/97.6
	28	12.4/30.9	28.4/49.5	40.9/80.4	29.4/13.1	27.4/5.1	< 0.1/< 0.1	97.6/98.7
	50	8.8/35.6	13.5/40.3	22.3/75.9	38.6/14.0	36.8/6.5	0.1/< 0.1	97.9/96.5
	77	3.2/NS	3.7/NS	7.0/NS	49.4/NS	45.8/NS	< 0.1/NS	102.1/NS
	90	1.2/NS	2.1/NS	3.2/NS	43.5/NS	46.0/NS	< 0.1/NS	92.7/NS
	120	0.4/35.4	1.5/34.9	1.9/70.3	36.1/11.9	43.8/13.4	0.5/< 0.1	82.3 ^a /95.6
Senozan	0	40.2/40.3	57.4/57.3	97.6/97.6	4.5/2.7	NA/NA	NA/NA	102.0/100.3
	2	33.8/36.2	55.5/57.4	89.3/93.6	8.0/4.4	2.1/0.6	< 0.1/0.2	99.4/98.8
	7	25.9/36.0	45.4/53.3	71.3/89.3	18.9/6.9	8.1/1.6	< 0.1/< 0.1	98.3/97.9
	14	22.9/37.5	35.5/51.8	58.4/89.3	21.5/6.7	31.8/2.6	0.1/< 0.1	111.9 ^b /98.7
	21	17.4/39.7	27.4/48.7	44.8/88.4	32.7/9.1	15.7/0.8	< 0.1/< 0.1	93.2/98.4
	28	15.9/30.3	33.7/54.1	49.6/84.4	25.3/8.7	11.5 ^a /5.5	< 0.1/< 0.1	86.4/98.7
	50	18.6/37.7	27.6/45.6	46.2/83.3	28.8/8.5	23.6/6.0	< 0.1/< 0.1	98.7/97.8
	77	15.0/NS	19.8/NS	34.9/NS	29.3/NS	26.9/NS	< 0.1/NS	91.1/NS
	90	16.7/NS	21.9/NS	38.6/NS	25.0/NS	28.9/NS	< 0.1/NS	92.5/NS
	120	12.7/36.0	13.2/37.8	25.9/73.8	25.9/11.4	28.1 ^a /11.7	0.4/0.1	80.3 ^a /97.0
Sevelen	0	29.1/30.3	63.3/64.3	92.4/94.6	8.0/5.0	NA/NA	NA/NA	100.4/99.6
	2	25.6/29.1	54.1/60.1	79.7/89.3	12.5/7.6	3.7/0.1	< 0.1/< 0.1	95.9/97.0
	7	20.7/30.1	45.0/55.3	65.7/85.4	20.7/10.5	16.1/4.4	< 0.1/< 0.1	102.4/100.3
	14	18.1/32.7	33.6/48.8	51.7/81.5	25.8/12.5	17.6/5.3	< 0.1/< 0.1	95.1/99.3
	21	16.5/33.3	28.0/44.7	44.5/77.9	24.3/12.0	25.0/4.1	< 0.1/< 0.1	93.8/94.0

Soil	Day	Percent of applied radioactivity, [Bu- ¹⁴ C]/ [Th- ¹⁴ C]						
		CaCl ₂ extract	Organic extract	Total extractable	Soil bound	¹⁴ CO ₂	VOC	Total recovered
	28	12.7/32.8	22.0/41.8	34.6/74.6	29.7/15.2	30.6/7.3	< 0.1/< 0.1	94.9/97.2
	50	8.2/31.8	9.2/34.9	17.4/66.7	33.1/12.9	35.1/16.5	< 0.1/< 0.1	85.6 ^a /96.0
	77	3.4/NS	3.0/NS	6.4/NS	33.7/NS	50.4/NS	< 0.1/NS	90.5/NS
	90	1.8/NS	2.3/NS	4.1/NS	36.9/NS	53.3/NS	< 0.1/NS	94.3/NS
	120	1.0/25.4	1.4/23.2	2.5/48.5	32.6/14.6	52.0/30.3	< 0.1/< 0.1	87.1 ^a /93.5

^a Due to losses of ¹⁴CO₂

^b The KOH trap was probably mistakenly exchanged for another sample (cf. day 21).

NS = No Sample

Table 37 Formation of major soil metabolites of fluensulfone in six aerobic soils

Soil	Day	Percent of applied radioactivity			
		Fluensulfone ([Bu- ¹⁴ C]/ [Th- ¹⁴ C])	BSA	TSA	MeS
Filsis	0	86.4/83.2	1.2	9.4	0.0
	2	77.1/74.5	1.4	14.7	0.0
	7	48.6/45.6	18.5	41.5	0.0
	14	35.4/27.1	11.6	53.8	2.4
	21	30.4/23.8	12.7	53.1	4.6
	28	23.2/17.6	13.9	59.3	3.4
	50	7.9/5.6	11.8	68.5	4.2
	77	2.1/NS	3.7	NS	NS
	90	1.9/NS	2.9	NS	NS
	120	1.0/0.0	1.2	72.7	0.0
Hagenthal	0	90.2/87.6	0.0	7.3	0.0
	2	76.2/73.1	4.4	16.2	0.0
	7	47.0/47.3	14.6	40.8	0.0
	14	26.0/20.0	11.1	61.0	2.4
	21	22.3/11.7	18.7	64.4	4.5
	28	15.9/10.5	16.7	64.6	5.5
	50	5.5/2.3	14.5	69.7	3.2
	77	0.9/NS	1.8	NS	NS
	90	0.6/NS	1.2	NS	NS
	120	0.7/0.0	1.3	61.3	0.0
Horn	0	81.9/81.8	4.7	10.3	0.0
	2	70.0/70.6	4.6	18.0	0.0
	7	47.1/37.9	8.1	46.6	0.0
	14	37.1/16.3	14.3	58.8	5.5
	21	16.3/9.8	16.2	68.7	4.9
	28	8.9/5.8	19.4	68.8	3.8
	50	1.3/0.0	5.7	76.6	0.0
	77	0.5/NS	1.2	NS	NS
	90	0.7/NS	2.4	NS	NS
	120	0.5/0.0	1.2	66.0	0.0
Montesquieu	0	90.0/86.8	1.7	5.2	0.0
	2	79.6/86.0	2.9	4.8	0.0
	7	50.1/56.7	14.9	30.0	0.0
	14	40.1/30.9	14.5	53.2	0.0
	21	32.0/33.4	17.3	49.9	0.0
	28	12.7/19.0	28.1	57.2	4.2
	50	8.3/5.5	14.1	65.0	5.4
	77	2.4/NS	4.6	NS	NS
	90	1.2/NS	2.0	NS	NS
	120	0.9/0.0	1.0	70.3	0.0
Senozan	0	97.6/93.4	0.0	4.2	0.0
	2	89.3/90.0	0.0	3.6	0.0

Soil	Day	Percent of applied radioactivity			
		Fluensulfone ([Bu- ¹⁴ C]/ [Th- ¹⁴ C])	BSA	TSA	MeS
	7	62.6/59.8	8.7	29.6	0.0
	14	48.8/55.0	9.7	34.3	0.0
	21	31.0/36.1	13.8	48.4	3.9
	28	32.9/32.4	16.7	48.4	3.6
	50	21.1/19.8	25.1	59.7	3.8
	77	8.7/NS	26.2	NS	NS
	90	7.6/NS	31.0	NS	NS
	120	2.2/0.0	23.8	73.8	0.0
Sevelen	0	92.4/89.8	0.0	4.8	0.0
	2	75.0/82.4	4.7	6.9	0.0
	7	48.3/37.1	17.4	48.3	0.0
	14	34.4/21.2	17.3	57.8	2.5
	21	NS/NS	NS	NS	NS
	28	15.1/8.7	19.5	58.5	7.5
	50	2.5/2.1	14.9	58.4	6.2
	77	0.8/NS	5.6	NS	NS
	90	0.6/NS	3.5	NS	NS
	120	0.4/0.0	2.1	48.5	0.0

NS = No Sample

Table 38 Summary of half-life estimates for fluensulfone and BSA in six soils

Soil	Fislis	Hagenthal	Horn	Montesquieu	Senozan	Sevelen
Type	Silt loam	Silt loam	Loam	Clay loam	Loam	Sandy loam
Model	SFO ^a	SFO	SFO	SFO	SFO	SFO
Residue	Half-life (days)					
Fluensulfone	10.5	7.6	7.2	11.1	16.5	7.1
BSA	19.7	22.6	21.9	17.8	^b	26.3

^a SFO = Single First-Order

^b Degradation rate of BSA could not be calculated due to insufficient data points

Aerobic soil degradation rate (TSA)

The degradation rate of TSA metabolite of fluensulfone in three aerobic soils was investigated by Brands (2011, R-28470). In that study, Fislis, Horn, and Sevelen soils (see fluensulfone aerobic soil metabolism section above) were treated with the test substance at a nominal concentration of 3.2 mg/kg dry soil and incubated, in the dark, for 150 days. Duplicate samples were collected at nine time points throughout the study, extracted with ACN:H₂O (50:50, v/v), and analysed by HPLC-MS/MS.

Overall procedural recoveries from freshly fortified soils ranged from 95–112% of the applied amount (Table 39) and levels of TSA showed a slow decline over the incubation period (Table 40). Single first-order half-life estimates for TSA were 560 days for Fislis soil, 450 days for Horn soil, and 230 days for Sevelen soil.

Table 39 Procedural recovery of TSA from soils fortified at 3.2 mg/kg

	Fislis		Horn		Sevelen	
Sample time ID	TSA (mg/kg)	Recovery (% of nominal)	TSA (mg/kg)	Recovery (% of nominal)	TSA (mg/kg)	Recovery (% of nominal)
7	3.25	101	3.39	105	3.48	108
15	3.4	106	3.29	102	3.43	107
28	3.4	106	3.33	104	3.48	108
42	3.59	112	3.55	110	3.13	97
60	3.18	100	3.03	95	3.13	98
91	3.14	98	3.26	102	3.14	98
120	3.32	104	3.12	98	3.45	108
150	3.45	107	3.36	105	3.17	99

Table 40 Levels of TSA in three soils incubated for 150 days, normalized to Time 0

Time (days)	Fislis	Horn	Sevelen
0	100, 100	100, 100	100, 100
7	109, 106	98, 101	88, 105
15	101, 96	95, 108	87, 97
28	93, 94	92, 100	83, 96
42	90, 96	90, 94	86, 89
60	85, 82	76, 83	74, 77
91	93, 94	82, 90	64, 81
120	88, 86	88, 90	67, 74
150	83, 88	163 ^a , 0 ^a	57, 70

^a Unexpected results were not used for calculation of DT₅₀ and DT₉₀

Aerobic soil degradation rate (MeS)

The degradation rate of MeS metabolite of fluensulfone in three aerobic soils was investigated by Brands (2011, R-28472). In that study, Fislis, Horn, and Sevelen soils (see fluensulfone aerobic soil metabolism section above) were treated with the test substance at a nominal concentration of 0.4 or 0.04 mg/kg dry soil and incubated, in the dark, for 120 days. Duplicate samples were collected at eight time points throughout the study, extracted with ACN:H₂O (50:50, v/v), and analysed by HPLC-MS/MS.

Overall procedural recoveries from freshly fortified soils ranged from 95–112% of the applied amount (Table 41) and levels of MeS showed a slow decline over the incubation period (Table 42). Single first-order half-life estimates for MeS were 41 days for Fislis soil, 28 days for Horn soil, and 30 days for Sevelen soil.

Table 41 Procedural recovery of MeS from soils fortified at 0.4 or 0.04 mg/kg

	Fislis		Horn		Sevelen	
Sample time ID	MeS (mg/kg)	Recovery (% of nominal)	MeS (mg/kg)	Recovery (% of nominal)	MeS (mg/kg)	Recovery (% of nominal)
7	0.393	98	0.338	85	0.357	89
15	0.406	102	0.361	90	0.366	92
28	0.469	117	0.408	102	0.349	87
42	0.0418	104	0.0435	108	0.0369	92
60	0.0395	98	0.0403	100	0.0418	104

	Fisli		Horn		Sevelen	
Sample time ID	MeS (mg/kg)	Recovery (% of nominal)	MeS (mg/kg)	Recovery (% of nominal)	MeS (mg/kg)	Recovery (% of nominal)
91	0.0391	97	0.0395	99	0.0378	94
120	0.0441	110	0.0393	100	0.0415	103

Table 42 Levels of MeS in three soils incubated for 150 days, normalized to Time 0

Time (days)	Fisli	Horn	Sevelen
0	100, 100	100, 100	100, 100
7	95, 111	85, 78	78, 83
15	84, 89	57, 67	62, 60
28	61, 67	47, 42	37, 44
42	48, 54	36, 34	39, 35
60	38, 41	23, 24	26, 30
91	22, 23	14, 14	17, 17
120	14, 16	8, 7	10, 10

Anaerobic soil metabolism

No anaerobic soil metabolism studies were provided.

Confined rotational crop studies

The fate of fluensulfone as relates to rotational crops was investigated by Quistad *et al.* (2011, Report OR-25457). In that study, radish, lettuce and wheat were planted into test boxes that had been treated with [Bu-¹⁴C] or [Th-¹⁴C] labelled fluensulfone at a rate of ca. 4 kg/ha. The crops were planted into the test boxes at 30, 120, and 360 days after treatment (390 days for lettuce due to crop failure at the 360-day plant-back interval). At each plant-back interval (PBI), samples consisted of radish roots and radish tops; lettuce leaves; and wheat forage, grain, hay, and straw.

After harvest, the samples were homogenized and an aliquot was taken for TRR determination by combustion and LSC. In addition, samples were extracted initially with ACN:H₂O (1:1, v/v) and ACN, followed by strong basic and, in some cases, acidic hydrolysis. Extracted residues were analysed by TLC and/or HPLC (UV and radio-chromatography via fraction collection and LSC) for residue determination. Multiple TLC systems and HPLC conditions were used to obtain adequate separation and analysis.

Total radioactive residues in the rotational crops are shown in Table 43. There was generally good agreement between TRR by combustion and TRR by sum of fractions. The TRR levels from the [Th-¹⁴C] treatments were higher from than those from the [Bu-¹⁴C] treatments and tended to show a plateau between the 120-day and 360/390-day PBIs that was not seen with the [Bu-¹⁴C] treatments.

The ACN:H₂O solution was able to extract the majority of the TRR from all of the rotational crop matrices (Tables 44–47). Analysis of the extracts showed levels of fluensulfone in radish (Tables 48–51), lettuce (Tables 52–55), and wheat (Table 56–63) samples from at least one PBI/radiolabel position/sample matrix combination. In all cases, the occurrence of fluensulfone was ≤ 5.1% TRR and

was higher in samples from the [Bu-¹⁴C] treatments. The major residues in all matrices were TSA and BSA from the [Bu-¹⁴C] and [Th-¹⁴C] treatments, respectively. Across PBIs, levels of BSA generally declined in terms of both absolute concentration and in terms of %TRR. Absolute levels of TSA generally declined with increasing PBI, especially between the 30-day and 120-day PBIs, but increased in terms of %TRR over the course of the study. Numerous minor metabolites and/or fractions were observed. Most of these were attributed to salts or chromatographic artifacts of TSA or BSA.

Table 43 Summary of TRRs in rotational crops at each plant-back interval

Crop	TRRs ^a (mg eq/kg)					
	[Th- ¹⁴ C]			[Bu- ¹⁴ C]		
PBI	30 DAT	120 DAT	360 DAT	30 DAT	120 DAT	360 DAT
Radish (foliage)	5.26 (5.76)	1.76 (1.90)	2.86 (3.58)	0.48 (0.54)	0.47 (0.51)	0.04 (0.04)
Radish (roots)	0.79 (0.83)	0.44 (0.45)	0.38 (0.39)	0.15 (0.19)	0.10 (0.08)	0.01 (0.01)
Lettuce (immature)	0.65 (0.58)	0.71 (0.81)	0.13 (0.13) ^b	0.31 (0.33)	0.05 (0.04)	0.05 (0.05) ^b
Lettuce (mature)	0.57 (0.60)	0.33 (0.34)	0.34 (0.33)	0.20 (0.21)	0.05 (0.04)	0.02 (0.01)
Wheat (forage)	16.6 (19.2)	2.96 (3.42)	3.33 (3.19)	2.53 (3.17)	0.71 (0.84)	0.06 (0.07)
Wheat (straw)	18.6 (18.5)	3.52 (3.85)	6.64 (7.26)	1.94 (1.99)	0.30 (0.31)	0.17 (0.16)
Wheat (grain)	0.36 (0.36)	0.30 (0.32)	0.34 (0.35)	0.17 (0.17)	0.06 (0.07)	0.04 (0.04)
Wheat (hay)	27.0 (26.4)	9.40 (9.29)	10.8 (11.3)	4.50 (4.35)	0.37 (0.36)	0.13 (0.14)

^a Values in parenthesis obtained by direct combustion

^b 390 day lettuce samples since 360-day lettuce was replanted because of crop failure

DAT = Days after treatment.

Table 44 Extraction summary for radish foliage and roots after soil treatment with [¹⁴C]fluensulfone

Crop	Radish							
Matrix	Foliage				Roots			
¹⁴ C Label	[Th- ¹⁴ C]		[Bu- ¹⁴ C]		[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
Fraction	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
PBI	30 DAYS							
ACN:H ₂ O Extract	5.166	98.2	0.452	94.4	0.723	91.2	0.126	86.3
0.1 N KOH Extract	0.053	1.0	0.010	2.1	0.011	1.4	0.008	5.5
24% KOH Extract	0.033	0.6	0.013	2.7	0.023	2.9	0.008	5.5
PES	0.011	0.2	0.004	0.8	0.036	4.5	0.004	2.7
Total	5.263		0.479		0.793		0.146	
PBI	120 DAYS							
ACN:H ₂ O Extract	1.709	97.3	0.436	93.4	0.382	87.4	0.079	76.0
0.1 N KOH Extract	0.021	1.2	0.006	1.3	0.009	2.1	0.006	5.8
24% KOH Extract	0.020	1.1	0.023	4.9	0.026	5.9	0.015	14.4
Combined 0.1 N KOH & 24% KOH Extracts, Acidified and Partitioned with EtAc								
EtAc Phase	–	–	–	–	–	–	0.002	1.9
Aqueous Phase	–	–	–	–	–	–	0.019	18.3
PES	0.006	0.3	0.002	0.4	0.020	4.6	0.004	3.8
Total	1.756		0.467		0.437		0.104	
PBI	360 DAYS							
ACN:H ₂ O Extract	2.842	99.4	0.031	81.6	0.365	96.3	0.006	50.0
0.1 N KOH Extract	0.007	0.2	0.001	2.6	0.001	0.3	< 0.001	0.0
24% KOH Extract	0.010	0.4	0.005	13.2	0.009	2.4	0.005	41.7
PES	0.001	0.0	0.001	2.6	0.004	1.1	0.001	8.3
Total	2.860		0.038		0.379		0.012	

Table 45 Extraction summary for immature and mature lettuce after soil treatment with [^{14}C]fluensulfone

Crop	Lettuce							
Matrix	Immature				Mature			
^{14}C Label	[Th- ^{14}C]		[Bu- ^{14}C]		[Th- ^{14}C]		[Bu- ^{14}C]	
Fraction	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
PBI	30 DAYS							
ACN:H ₂ O Extract	0.495	76.5	0.221	71.1	0.494	87.4	0.137	67.2
Acidification with HCl & Partition with EtAc								
EtAc Phase	0.196	30.3	–	–	–	–	–	–
Aqueous Phase	0.299	46.2	–	–	–	–	–	–
0.1 N KOH Extract	0.038	5.9	0.030	9.6	0.023	4.1	0.017	8.3
24% KOH Extract	0.099	15.3	0.055	17.7	0.043	7.6	0.041	20.1
Combined 0.1 N KOH & 24% KOH Extracts, Acidified and Partitioned with EtAc								
EtAc Phase	–	–	0.018	5.8	0.016	2.8	0.010	4.9
Aqueous Phase	–	–	0.067	21.5	0.050	8.8	0.048	23.5
PES	0.015	2.3	0.005	1.6	0.005	0.9	0.009	4.4
Total	0.647		0.311		0.565		0.204	
PBI	120 DAYS							
ACN:H ₂ O Extract	0.622	87.6	0.021	46.7	0.299	90.3	0.021	46.7
0.1 N KOH Extract	0.031	4.4	0.004	8.9	0.012	3.6	0.004	8.9
24% KOH Extract	0.045	6.3	0.014	31.1	0.018	5.4	0.014	31.1
72% H ₂ SO ₄ Extract	0.007	1.0						
Combined 0.1 N KOH & 24% KOH Extracts, Acidified and Partitioned with EtAc								
EtAc Phase	–	–	0	0.0	–	–	0.000	0.0
Aqueous Phase	–	–	0.018	40.0	–	–	0.016	35.6
PES	0.005	0.7	0.006	13.3	0.002	0.6	0.006	13.3
Total	0.710		0.045		0.331	99.9	0.045	
PBI	390 DAYS ^a							
ACN:H ₂ O Extract	0.119	89.5	0.030	60.0	0.331	97.6	0.030	60.0
0.1 N KOH Extract	0.004	3.0	0.004	8.0	0.002	0.6	0.004	8.0
24% KOH Extract	0.009	6.8	0.013	26.0	0.005	1.5	0.013	26.0
Combined 0.1 N KOH & 24% KOH Extracts, Acidified and Partitioned with EtAc								
EtAc Phase	0.002	1.5	0.003	6.0	–	–	–	–
Aqueous Phase	0.011	8.3	0.014	28.0	–	–	–	–
PES	0.001	0.8	0.003	6.0	0.001	0.3	0.003	6.0
Total	0.133		0.050		0.339		0.050	

^a 390-day lettuce samples because of crop failure of 360-day lettuce samples.Table 46 Extraction summary for wheat forage and straw after soil treatment with [^{14}C]fluensulfone

Crop	Wheat							
Matrix	Forage				Straw			
^{14}C Label	[Th- ^{14}C]		[Bu- ^{14}C]		[Th- ^{14}C]		[Bu- ^{14}C]	
Fraction	mg eq/kg	% TRR	mg eq/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
PBI	30 DAYS							
ACN:H ₂ O Extract	15.594	94.1	2.240	88.5	15.626	84.2	1.035	53.5
0.1 N KOH Extract	0.300	1.8	0.073	2.9	0.906	4.9	0.195	10.1
24% KOH Extract	0.538	3.2	0.184	7.3	1.443	7.8	0.350	18.1
Combined 0.1 N KOH & 24% KOH Extracts, Acidified and Partitioned with EtAc								
EtAc Phase	–	–	–	–	0.507	2.7	0.079	4.1
Aqueous Phase	–	–	–	–	1.842	9.9	0.466	24.1
72% H ₂ SO ₄ Extract	–	–	–	–	0.441	2.4	0.283	14.6
PES	0.147	0.9	0.033	1.3	0.153	0.8	0.072	3.7
Total	16.579		2.530		18.569		1.935	
PBI	120 DAYS							
ACN:H ₂ O Extract	2.809	94.8	0.642	90.0	2.966	84.3	0.195	64.4
0.1 N KOH Extract	0.079	2.7	0.024	3.4	0.094	2.7	0.041	13.5

Crop	Wheat							
Matrix	Forage				Straw			
¹⁴ C Label	[Th- ¹⁴ C]		[Bu- ¹⁴ C]		[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
Fraction	mg eq/kg	% TRR	mg eq/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
24% KOH Extract	0.058	2.0	0.038	5.3	0.331	9.4	0.042	13.9
Combined 0.1 N KOH & 24% KOH Extracts, Acidified and Partitioned with EtAc								
EtAc Phase	–	–	0.005	0.7	0.081	NR	0.013	NR
Aqueous Phase	–	–	0.057	8.0	0.344	NR	0.070	NR
72% H ₂ SO ₄ Extract	0.012	0.4	0.007	1.0	0.126	3.6	0.025	8.3
PES	0.005	0.2	0.002	0.3	0.126	3.6	0.025	8.3
Total	2.963		0.713		3.517		0.303	
PBI	360 DAYS							
ACN:H ₂ O Extract	3.157	94.9	0.039	65.0	6.051	91.2	0.102	61.8
0.1 N KOH Extract	–	–	0.003	5.0	0.259	3.9	0.011	6.7
24% KOH Extract	–	–	0.014	23.3	0.229	3.5	0.032	19.4
Combined 0.1 N KOH & 24% KOH Extracts, Acidified and Partitioned with EtAc								
EtAc Phase	–	–	–	–	0.094	1.4	0.010	6.1
Aqueous Phase	–	–	–	–	0.394	5.9	0.033	20.0
PES	0.169	5.1	0.004	6.7	0.097	1.5	0.020	12.1
Total	3.326		0.060		6.636		0.165	

Table 47 Extraction summary for wheat hay and grain after soil treatment with [¹⁴C]fluensulfone

Crop	Wheat							
Matrix	Hay				Grain			
¹⁴ C Label	[Th- ¹⁴ C]		[Bu- ¹⁴ C]		[Th- ¹⁴ C]		[Bu- ¹⁴ C]	
Fraction	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
PBI	30 DAYS							
ACN:H ₂ O Extract	18.891	70.0	2.127	47.3	0.263	73.3	0.109	63.7
0.1 N KOH Extract	5.321	19.7	1.478	32.8	0.034	9.6	–	–
24% KOH Extract	2.224	8.2	0.741	16.5	–	–	–	–
Combined 0.1 N KOH & 24% KOH Extracts, Acidified and Partitioned with EtAc								
EtAc Phase	1.177	4.4	0.249	5.5	0.003	0.8	–	–
Aqueous Phase	6.368	23.6	1.970	43.8	0.059	16.6	–	–
72% H ₂ SO ₄ Extract	0.479	1.8	–	–	–	–	–	–
PES	0.089	0.3	0.154	3.4	–	–	–	–
Total	27.004		4.500		0.359		0.173	
PBI	120 DAYS							
ACN:H ₂ O Extract	7.185	76.5	0.225	60.3	0.247	83.4	0.023	39.0
0.1 N KOH Extract	1.288	13.7	0.060	16.1	0.017	5.7	0.006	10.2
24% KOH Extract	0.715	7.6	0.068	18.2	0.003	1.0	0.001	1.7
Combined 0.1 N KOH & 24% KOH Extracts, Acidified and Partitioned with EtAc								
EtAc Phase	0.282	3.0	0.009	2.4	–	–	–	–
Aqueous Phase	1.721	18.3	0.119	31.9	–	–	–	–
72% H ₂ SO ₄ Extract	0.209	2.2	0.020	5.4	–	–	–	–
PES	0.209	2.2	0.020	5.4	0.029	9.8	0.029	49.2
Total	9.397		0.373		0.296		0.059	
PBI	360 DAYS							
ACN:H ₂ O Extract	7.768	71.7	0.070	54.7	0.260	76.0	0.013	34.2
0.1 N KOH Extract	1.166	10.8	0.011	8.6	0.022	6.4	0.006	15.8
24% KOH Extract	1.230	11.4	0.026	20.3	–	–	–	–
Combined 0.1 N KOH & 24% KOH Extracts, Acidified and Partitioned with EtAc								
EtAc Phase	0.594	5.5	0.001	8.6	0.008	2.3	–	–
Aqueous Phase	1.802	16.6	0.026	20.3	0.014	4.1	–	–
PES	0.671	6.2	0.021	16.4	0.060	17.5	0.019	50.0
Total	10.835		0.128		0.342		0.038	

Table 48 Summary of [Th-¹⁴C]fluensulfone labelled residues in radish foliage

Fraction/residue	30-Day PBI		120-Day PBI		360-Day PBI	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	5.263	100.0	1.756	100.0	2.860	100.0
Solvent Extractable	5.166	98.2	1.709	97.3	2.842	99.4
Fluensulfone	0.010	0.2	—	—	—	—
Thiazole Sulfonic Acid	4.726	89.8	1.624	92.5	2.558	89.4
M1—Form of TSA	0.113	2.2	0.073	4.2	—	—
M3—Form of TSA	0.034	0.6	—	—	—	—
Unresolved	0.258	4.9	0.012	0.7	0.283	9.9

Table 49 Summary of [Th-¹⁴C]fluensulfone labelled residues in radish roots

Fraction/residue	30-Day PBI		120-Day PBI		360-Day PBI	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	0.793	100.0	0.437	100.0	0.379	100.0
Solvent Extractable	0.723	91.2	0.382	87.4	0.365	96.3
Thiazole Sulfonic Acid	0.687	86.6	0.355	81.2	0.341	90.0
M1—Form of TSA	—	—	0.004	0.9	—	—
M3—Form of TSA	0.004	0.5	0.001	0.2	0.004	1.1
Unresolved	0.020	2.5	0.016	4.2	0.019	5.0

M1: This fraction consists of two compounds with the major part of this identified a salt of TSA.

M3: This polar radioactive fraction was always observed as a possible artifact due to the non-retention of TSA.

Alkaline and acid digestion released additional small amounts of radiolabelled compounds.

Table 50 Summary of [Bu-¹⁴C]fluensulfone labelled residues in radish foliage

Fraction/residue	30-Day PBI		120-Day PBI		360-Day PBI	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	0.479	100.0	0.467	100.0	0.038	100.0
Solvent Extractable	0.452	94.4	0.436	93.4	0.031	81.6
Fluensulfone	0.015	3.1	0.024	5.1	0.001	3.0
Butene Sulfonic Acid	0.315	65.8	0.328	70.2	—	—
F1-F2—Form of BSA	0.016	3.4	0.002	0.4	0.002	5.1
F4—Form of BSA	0.017	3.5	—	—	—	—
F5	0.005	1.0	0.015	3.2	0.001	3.6
F6	0.001	0.2	—	—	0.002	5.3
F7	—	—	—	—	0.005	13.2
F9	—	—	—	—	0.001	3.2
Unresolved	0.059	12.3	0.068	14.6	0.013	34.2

Table 51 Summary of [Bu-¹⁴C]fluensulfone labelled residues in radish roots

Fraction/residue	30-Day PBI		120-Day PBI		360-Day PBI	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	0.146	100.0	0.104	100.0	0.012	100.0
Solvent Extractable	0.126	86.3	0.079	76.0	0.006	50.0
Fluensulfone	0.002	1.5	—	—	—	—
Butene Sulfonic Acid	0.058	40.0	0.041	39.4	—	—
F1 - F2—Form of BSA	0.009	6.2	0.002	2.3	0.001	8.3
F5	0.007	4.8	0.005	4.5	—	—
F7	0.006	3.8	—	—	—	—
Unresolved	0.034	23.3	0.025	24.0	0.005	41.7

Table 52 Summary of [Th-¹⁴C]fluensulfone labelled residues in immature lettuce

Fraction/residue	30-Day PBI		120-Day PBI		390-Day PBI	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	0.647	100.0	0.710	100.0	0.133	100.0
Solvent Extractable	0.495	76.5	0.622	87.6	0.119	89.5
Fluensulfone	0.005	0.8	0.004	0.6	—	—
Thiazole Sulfonic Acid	0.183	28.3	0.401	56.5	0.086	64.7
M1—Form of TSA	0.122	18.9	0.113	15.9	0.013	9.8
M2—Form of TSA	—	—	0.009	1.3	—	—
M3—Form of TSA	0.007	1.1	0.004	0.6	0.003	2.3
Unresolved	0.179	27.7	0.091	12.8	0.016	12.0

Table 53 Summary of [Th-¹⁴C]fluensulfone labelled residues in mature lettuce

Fraction/residue	30-Day PBI		120-Day PBI		360-Day PBI	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	0.565	100.0	0.331	100.0	0.339	100.0
Solvent Extractable	0.494	87.4	0.299	90.3	0.331	97.6
Fluensulfone	0.006	1.1	—	—	—	—
Thiazole Sulfonic Acid	0.237	41.9	0.184	55.6	0.279	82.3
M1—Form of TSA	0.113	20.0	0.086	26.0	0.004	1.2
M3—Form of TSA	0.002	0.4	0.004	1.2	—	—
Unresolved	0.134	23.7	0.025	7.6	0.046	13.6

M1: This fraction consists of two compounds with the major part of this identified as a salt of TSA.

M3: This polar radioactive fraction was always observed as a possible artifact due to the non-retention of TSA.

Alkaline & acid digestion: Unresolved metabolites were released especially with 24% KOH. A major component appeared to be TSA.

Table 54 Summary of [Bu-¹⁴C]fluensulfone labelled residues in immature lettuce

Fraction/residue	30-Day PBI		120-Day PBI		390-Day PBI	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	0.311	100.0	0.045	100.0	0.050	100.0
Solvent Extractable	0.221	71.1	0.021	46.7	0.030	60.0
Fluensulfone	0.016	5.1	—	—	0.002	4.0
Butene Sulfonic Acid	0.004	1.2	0.001	2.2	—	—
F1-F2—Form of BSA	0.006	1.9	0.006	13.3	0.005	10.0
F4—Form of BSA	< 0.001	0.1	0.002	4.4	—	—
F5	0.002	0.5	—	—	—	—
F6	0.003	1.2	—	—	—	—
F7	0.002	0.6	0.001	1.3	0.001	2.0
F9	0.010	3.2	0.001	2.2	—	—
F10	< 0.001	0.1	—	—	—	—
Unresolved	0.176	56.6	0.009	20.0	0.019	38.0

Table 55 Summary of [Bu-¹⁴C]fluensulfone labelled residues in mature lettuce

Fraction/residue	30-Day PBI		120-Day PBI		360-Day PBI	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	0.204	100.0	0.045	100.0	0.017	100.0
Solvent Extractable	0.137	67.2	0.027	60.0	0.008	47.1
Fluensulfone	0.004	2.0	—	—	—	—
Butene Sulfonic Acid	0.018	8.8	—	—	—	—
F1-F2—Form of BSA	0.011	5.4	0.016	35.6	0.002	11.8

	30-Day PBI		120-Day PBI		360-Day PBI	
Fraction/residue	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
F5	0.001	0.6	—	—	—	—
Unresolved	0.100	49.0	0.010	21.9	0.006	35.3

Table 56 Summary of [Th-¹⁴C]fluensulfone labelled residues in wheat forage

	30-Day PBI		120-Day PBI		360-Day PBI	
Fraction/residue	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	16.579	100.0	2.963	100.0	3.326	100.0
Solvent Extractable	15.594	94.1	2.809	94.8	3.157	94.9
Thiazole Sulfonic Acid	12.241	73.8	2.254	76.0	2.737	82.3
M1—Form of TSA	0.521	3.1	0.104	3.5	0.011	0.3
M3—Form of TSA	0.147	0.9	—	—	0.036	1.1
Unresolved	2.237	13.5	0.449	15.2	0.376	11.3

Table 57 Summary of [Th-¹⁴C]fluensulfone labelled residues in wheat hay

	30-Day PBI		120-Day PBI		360-Day PBI	
Fraction/residue	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	27.004	100.0	9.397	100.0	10.835	100.0
Solvent Extractable	18.891	70.0	7.185	76.5	7.768	71.7
Thiazole Sulfonic Acid	15.571	57.7	6.221	66.2	5.771	53.3
M1—Form of TSA	0.496	1.8	0.227	2.4	0.084	0.8
M3—Form of TSA	0.413	1.5	0.167	1.8	0.542	5.0
Unresolved	2.348	8.7	0.498	5.3	1.313	12.1

Table 58 Summary of [Th-¹⁴C]fluensulfone labelled residues in wheat grain

	30-Day PBI		120-Day PBI		360-Day PBI	
Fraction/residue	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	0.359	100.0	0.296	100.0	0.342	100.0
Solvent Extractable	0.263	73.9	0.247	83.4	0.260	76.0
Thiazole Sulfonic Acid	0.120	33.4	0.171	57.8	0.195	57.0
M1—Form of TSA	0.047	13.1	0.011	3.7	0.029	8.5
M3—Form of TSA	0.018	5.0	0.009	3.0	0.009	2.6
Unresolved	0.078	21.7	0.056	18.9	0.027	7.9

Table 59 Summary of [Th-¹⁴C]fluensulfone labelled residues in wheat straw

	30-Day PBI		120-Day PBI		360-Day PBI	
Fraction/residue	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	18.569	100.0	3.517	100.0	6.636	100.0
Solvent Extractable	15.626	84.2	2.966	84.3	6.051	91.2
Thiazole Sulfonic Acid	10.712	57.7	2.196	62.4	5.060	76.3
M1—Form of TSA	0.364	2.0	0.031	0.9	0.056	0.8
M3—Form of TSA	0.273	1.5	—	—	0.401	6.0
Unresolved	4.268	23.0	0.727	20.7	0.484	7.3

M1 & M3: Fraction not confirmed by TLC analysis leading to the suggestion to be a chromatographic artifact of TSA.

Alkaline and acid digestion, especially with 24% KOH, liberated several, unresolved metabolites that could not be identified. A major component appeared to be TSA.

Table 60 Summary of [Bu-¹⁴C]fluensulfone labelled residues in wheat forage

Fraction/residue	30-Day PBI		120-Day PBI		360-Day PBI	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	2.530	100.0	0.713	100.0	0.060	100.0
Solvent Extractable	2.240	88.5	0.642	90.0	0.039	65.0
Fluensulfone	0.028	1.1	0.021	2.9	—	—
Butene Sulfonic Acid	1.377	54.4	0.427	59.9	0.002	3.2
F1-F2—Form of BSA	0.090	3.6	0.026	3.6	0.009	15.0
F4—Form of BSA	0.020	7.9	0.016	2.2	—	—
F5	0.069	2.7	0.038	5.3	0.001	1.7
F6	0.035	1.4	—	—	—	—
F7	0.004	0.1	—	—	—	—
F9	0.041	1.6	0.015	2.1	—	—
F10	0.019	0.8	—	—	—	—
Unresolved	0.374	14.8	0.066	9.3	0.024	40.0

Table 61 Summary of [Bu-¹⁴C]fluensulfone labelled residues in wheat hay

Fraction/residue	30-Day PBI		120-Day PBI		360-Day PBI	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	4.500	100.0	0.373	100.0	0.128	100.0
Solvent Extractable	2.127	47.3	0.225	60.3	0.070	54.7
Fluensulfone	0.016	0.4	0.004	1.1	—	—
Butene Sulfonic Acid	1.223	27.2	0.095	25.5	—	—
F1-F2—Form of BSA	0.059	1.3	0.033	8.8	0.021	16.4
F4—Form of BSA	0.050	1.1	0.006	1.7	0.001	0.8
F5	0.077	1.7	0.006	1.6	0.002	1.4
F6	—	—	—	—	0.001	0.6
F7	—	—	—	—	0.001	0.8
F9	—	—	0.003	0.8	0.002	1.6
F10	—	—	—	—	—	—
Unresolved	0.700	15.6	0.064	17.2	0.037	28.9

Table 62 Summary of [Bu-¹⁴C]fluensulfone labelled residues in wheat grain

Fraction/residue	30-Day PBI		120-Day PBI		360-Day PBI	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	0.173	100.0	0.059	100.0	0.038	100.0
Solvent extractable	0.109	63.0	0.023	39.0	0.013	34.2
F1-F2—Form of BSA	0.007	4.0	0.011	18.6	0.004	10.5
unresolved	0.102	59.0	0.012	20.3	0.009	23.7

Table 63 Summary of [Bu-¹⁴C]fluensulfone labelled residues in wheat straw

Fraction/residue	30-Day PBI		120-Day PBI		360-Day PBI	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
TRR	1.935	100.0	0.303	100.0	0.165	100.0
Solvent Extractable	1.035	53.5	0.195	64.4	0.102	61.8
Fluensulfone	0.004	0.2	—	—	—	—
Butene Sulfonic Acid	0.272	14.1	0.031	10.2	0.012	7.0
F1-F2—Form of BSA	0.189	9.8	0.095	31.4	0.016	9.7
F5	0.007	0.4	—	—	0.002	1.0
F9	0.007	0.3	—	—	0.002	1.0
Unresolved	0.546	28.2	0.068	22.4	0.069	41.8

F3: Suggested to be BSA. Due to matrix effects the retention time of BSA is changing considerably leading to chromatographic artefacts.

F4 & F5: The HPLC-MS analysis of these fractions gave a high molecular weight which suggests that it is possibly a natural product.

Field rotational crop studies

No field rotational crop studies were provided.

Field dissipation studies

No field dissipation studies were provided.

RESIDUE ANALYSIS

Summary of analytical methods

Methods for the analysis of fluensulfone and its breakdown products BSA, MeS, and TSA are generally the same for plant, animal, and soil matrices, though there are some differences in the extraction of BSA and TSA in eggs and milk and in the clean-up steps for fatty samples. The methods are summarized in Table 64.

Table 64 Overview of the analytical methods submitted for fluensulfone and its major metabolites

Method ID (Report)	Matrix	Analyte	Extraction	Clean-up	Separation/ Analysis/LOQ
1997W (R-23334 and R-28495) Equivalent to 2061W	Plant commodities	Fluensulfone + MeS	ACN+H ₂ O (1+1, v/v)	Filtration	LC-MS/MS Fluensulfone m/z 292 [M+H] ⁺ → 166 (q) m/z 292 → 89 (c) m/z 292 → 59 (c) LOQ: 0.01 mg/kg MeS m/z 198 [M+H] ⁺ → 120 (q) m/z 198 → 122 (q) LOQ: 0.01 mg/kg
		BSA + TSA	ACN+H ₂ O (1+1, v/v)	C ₋₁₈ SPE + filtration	LC-MS/MS BSA m/z 189 [M-H] ⁻ → 81 (q) m/z 189 → 80 (c) LOQ: 0.01 mg/kg TSA m/z 198 [M-H] ⁻ → 82 (q) m/z 198 → 118 (c) LOQ: 0.01 mg/kg
10M04006-01-VMPL (R-28495)	Plant commodities	MeS	ACN+H ₂ O (1+1, v/v)	Filtration	LC-MS/MS m/z 198 [M+H] ⁺ → 120 (q) m/z 198 → 122 (q) LOQ: 0.01 mg/kg
11M03036-01-VMPL (R-23489)	Plant commodities	Fluensulfone	ACN+H ₂ O (1+1, v/v)	Filtration	LC-MS/MS m/z 292 [M+H] ⁺ → 166 (q) m/z 292 → 89 (c) m/z 292 → 59 (c) LOQ: 0.01 mg/kg
		MeS	ACN+H ₂ O (1+1, v/v)	Orange: filtration Wheat grain and peanut:	LC-MS/MS m/z 198 [M+H] ⁺ → 120 (q) m/z 198 → 122 (q) LOQ: 0.01 mg/kg

Method ID (Report)	Matrix	Analyte	Extraction	Clean-up	Separation/ Analysis/LOQ
				partition against hexane + filtration	
		BSA + TSA	ACN+H ₂ O (1+1, v/v)	C-18 SPE + filtration	LC-MS/MS BSA m/z 189 [M-H] ⁻ → 81 (q) m/z 189 → 80 (c) LOQ: 0.01 mg/kg TSA m/z 198 [M-H] ⁻ → 82 (q) m/z 198 → 118 (c) LOQ: 0.01 mg/kg
11M03036-01-VMAT (R-28512)	Animal commodities	Fluensulfone + MeS	ACN+H ₂ O (1+1, v/v)	Fat: filtration All others: partition against hexane	LC-MS/MS Fluensulfone m/z 292 [M+H] ⁺ → 166 (q) m/z 292 → 89 (c) LOQ: 0.01 mg/kg MeS m/z 198 [M+H] ⁺ → 120 (q) m/z 198 → 125 (c) LOQ: 0.01 mg/kg
		BSA + TSA	Liver, kidney, meat, fat: ACN+H ₂ O (1+1, v/v) Eggs, milk: ACN	C-18 SPE + filtration	LC-MS/MS BSA m/z 189 [M-H] ⁻ → 81 (q) m/z 189 → 80 (c) LOQ: 0.01 mg/kg TSA m/z 198 [M-H] ⁻ → 82 (q) m/z 198 → 118 (c) LOQ: 0.01 mg/kg
2049W (R-23339)	Soils	Fluensulfone + MeS	ACN+H ₂ O (1+1, v/v)	Filtration	LC-MS/MS Fluensulfone m/z 292 [M+H] ⁺ → 166 (q) m/z 292 → 89 (c) LOQ: 0.01 mg/kg MeS m/z 198 [M+H] ⁺ → 135 (q) m/z 198 → 93 (c) LOQ: 0.01 mg/kg
		BSA + TSA	ACN+H ₂ O (1+1, v/v)	C-18 SPE + filtration	LC-MS/MS BSA m/z 189 [M-H] ⁻ → 81 (q) m/z 189 → 80 (c) LOQ: 0.01 mg/kg TSA m/z 198 [M-H] ⁻ → 82 (q) m/z 198 → 118 (c) LOQ: 0.01 mg/kg

Plant materials

For plant matrices, Method 1997W (equivalent to Method 2061W) has been developed as a suitable enforcement method by J. Marin (2010, Report R-23334) for fluensulfone, BSA, and TSA, and expanded by A. Witte (2011, Report R-28495) to include MeS. The method underwent independent validation for all four analytes by R. Bacher (2011, Report R-27478).

Residues are extracted from 10 g of sample matrix using ACN:H₂O (1:1, v/v, 50 mL) by shaking for five minutes followed by centrifugation. The extract is then split, with one aliquot for

analysis of fluensulfone/MeS and a second aliquot for analysis of BSA/TSA. Quantification of all residues is by comparison with external, matrix-matched standards.

For fluensulfone (all matrices) and MeS (non-grain/non-oily matrices), an aliquot of the extract is filtered (0.45 μm) and analysed, without further clean-up, by LC-MS/MS in positive ion spray mode. For MeS in grain and oily matrices, the extract is salted out with NaCl; the separated ACN phase is dried with MgSO_4 and an aliquot is partitioned against hexane to remove co-extracted material. The ACN phase is collected, evaporated to dryness, and the residues are reconstituted in ACN:H₂O (1:1, v/v). The resulting extract is filtered (0.45 μm) and analysed by LC-MS/MS in positive ion spray mode. Chromatography for all extracts is done on a reverse-phase C₋₁₈ column maintained at 45 °C using a gradient mobile phase that transitions from 0.1% formic acid in ACN (95%) + 0.1% formic acid in H₂O (5%) to 0.1% formic acid in H₂O (100%). Ion transitions $[\text{M}+\text{H}]^+$ for fluensulfone are m/z 292 $\rightarrow$ 166 for quantitation (q), m/z 292 $\rightarrow$ 89 for confirmation (c), and m/z 292 $\rightarrow$ 59 (c). Ion transitions for MeS are m/z 198 $\rightarrow$ 120 (q) and the corresponding chlorine isotope transition (m/z 200 $\rightarrow$ 122).

For BSA and TSA (all matrices), a 6 mL aliquot of the initial extract is concentrated to 3 mL and cleaned-up on a C₋₁₈ SPE cartridge. Eluate from the cartridge is brought to volume and analysed by LC-MS/MS in negative ion mode. Chromatography is by reverse-phase C₋₁₈ column maintained at 45 °C using a gradient mobile phase that transitions from 0.05% formic acid in ACN (95%) + 0.05% formic acid in H₂O (5%) to 0.05% formic acid in H₂O (100%). Ion transitions $[\text{M}-\text{H}]^-$ for BSA are m/z 189 $\rightarrow$ 81 (q) and m/z 189 $\rightarrow$ 80 (c). Ion transitions for TSA are m/z 198 $\rightarrow$ 89 (q) and m/z 198 $\rightarrow$ 118).

Linearity data were provided and showed linear responses ($R^2 \geq 0.992$ and generally > 0.998) from 0.2–20 ng/mL. In a few studies, linearity was demonstrated over a broader range, including up to 100 ng/mL. Accuracy and precision data, in the form of spike and recovery studies, were provided for cucumber, lemon, melon, orange flesh, peanut, pepper, tomato and wheat grain. Samples were spiked at either 0.01 or 1 mg/kg of fluensulfone, BSA, MeS, or TSA (separately). Mean recoveries for each analyte at each spiking level and for each matrix were within the generally accepted range of 70–120%. Of the 860 method validation analyses, ten had recoveries outside of the generally accepted range of 70–120%: 53% and 54% for TSA in peanut, 174% for BSA in wheat grain, and seven recoveries ranging from 121% to 170% for fluensulfone, MeS, or TSA in orange flesh or lemon. In all cases, relative standard deviations were $\leq 20\%$.

Testing of fluensulfone and the two sulfonic acid metabolites, BSA and TSA, through the FDA PAM multiresidue method protocols was conducted by T. Ballard (2012, Report R-29565) for non-fatty foods. The compounds showed poor sensitivity, poor recovery, and/or poor chromatography. Overall, the results indicate that the FDA PAM multiresidue protocols are not suitable for the detection or enforcement of fluensulfone, BSA, or TSA residues in non-fatty foods.

Animal materials

A residue analytical method for the analysis of fluensulfone and its BSA, MeS, and TSA metabolites in animal matrices has been developed by Witte (2011, Report R-28512). It is identical to the method for those analytes in plant matrices described above, with the exception of the extraction of BSA and TSA from eggs and milk. For those matrices, extraction is accomplished with ACN (neat). The method was successfully validated by R. Bacher (2012, Report R-29562).

As with the plant method, extracts (except eggs and milk) are split for separate analysis of fluensulfone and MeS or BSA and TSA. For fluensulfone/MeS, the only clean-up is filtration whereas for BSA and TSA, there is clean-up by C₁₈ SPE and filtration. The LC-MS/MS conditions and ions are the same as described above for plants. The method showed linear response from 0.2–20 ng/mL for fluensulfone and MeS, and from 0.2–15 ng/mL for BSA and TSA. Recoveries from 900 spike-and-recovery analyses for all three analytes ranged from 71–109%, with the exception of four analyses, all for fluensulfone: 122% in pork meat, 154% in kidney, and 164% for liver, and egg.

Soil

A residue analytical method for the analysis of fluensulfone and its BSA, MeS, and TSA metabolites in animal matrices has been developed by Marin (2010, Report R-23339, Method 2049W). It is identical to the method for those analytes in plant matrices described above. The method was successfully validated by R. Barker (2012, Report R-29564).

As with the plant method, extracts are split for separate analysis of fluensulfone and MeS or BSA and TSA. For fluensulfone/MeS, the only clean-up is filtration whereas for BSA and TSA, there is clean-up by C₁₈ SPE and filtration. The LC-MS/MS conditions and ions are the same as described above for plants.

Mean recoveries ranged from 78 to 114% for all analyses, with relative standard deviations of < 12%.

Stability of residues in stored samples

The stability of fluensulfone, BSA, and TSA in frozen storage has been investigated in tomato, pepper (Korpalski, S. 2011, 09-01858), cucumber and melon (Korpalski, S. 2011, 09-01859). In addition, the stability of those analytes and MeS was investigated in frozen, stored tomato puree and paste (Jones, G.L. 2011, R-23487). No dissipation of any analyte was observed during the storage periods for the various matrices. Stability was demonstrated in tomato raw agricultural commodity (RAC) for at least 469 days (ca. 15 months) and in tomato processed commodities for at least 181 days (ca. 6 months). For pepper, cucumber, and melon, residues were stable for at least 488 days (ca. 16 months).

Table 65 Storage Stability of fluensulfone, TSA and BSA in cucumber

Analyte	Storage Interval days / months	Spiking Level (mg/kg)	Percent of Nominal Spiking Level	
			Procedural Recovery	Stored (% Remaining)
Fluensulfone	0 / 0	0.10	102, 90, 112	–
	91 / 3		100, 106	104, 97, 101
	266 / 9		84, 82	92, 104, 98
	488 / 16		79, 74	88, 86, 92
TSA	0 / 0	0.10	99, 97, 90	–
	91 / 3		101, 97	104, 105, 105
	267 / 9		110, 110	109, 110, 115
	488 / 16		94, 98	97, 100, 102
BSA	0 / 0	0.10	95, 104, 93	–
	91 / 3		100, 97	97, 95, 96
	267 / 9		110, 108	115, 118, 119
	488 / 16		98, 96	96, 105, 105

Table 66 Storage stability of fluensulfone, TSA and BSA in melon

Analyte	Storage Interval days / months	Spiking Level (mg/kg)	Percent of Nominal Spiking Level	
			Procedural Recovery	Stored (% Remaining)
Fluensulfone	0 / 0	0.10	79, 96, 77	–
	91 / 3		100, 103	101, 91, 106
	266 / 9		84, 76	74, 96, 95
	488 / 16		77, 79	91, 98, 98
TSA	0 / 0	0.10	85, 92, 89	–
	91 / 3		93, 97	90, 95, 97
	267 / 9		103, 103	110, 108, 113
	488 / 16		95, 94	89, 93, 89
BSA	0 / 0	0.10	83, 99, 93	–
	91 / 3		81, 90	79, 85, 89
	267 / 9		100, 103	115, 117, 116
	488 / 16		89, 93	93, 99, 94

Table 67 Storage stability of fluensulfone, TSA and BSA in pepper

Analyte	Storage Interval days / months	Spiking Level (mg/kg)	Percent of Nominal Spiking Level	
			Procedural Recovery	Stored (% Remaining)
Fluensulfone	0 / 0	0.10	91, 80, 89	–
	95 / 3		116, 117	113, 116, 119
	263 / 8.5		103, 107	102, 108, 109
	488 / 16		90, 94	96, 98, 106
TSA	0 / 0	0.10	85, 77, 81	–
	95 / 3		103, 101	105, 88, 95
	263 / 8.5		95, 100	94, 108, 101
	488 / 16		92, 89	85, 94, 98
BSA	0 / 0	0.10	76, 74, 78	–
	95 / 3		98, 110	97, 87, 99
	263 / 8.5		94, 102	102, 103, 102
	488 / 16		89, 94	102, 106, 107

Table 68 Storage stability of fluensulfone, TSA and BSA in tomato

Analyte	Storage Interval	Spiking	Percent of Nominal Spiking Level

	days / months	Level (mg/kg)	Procedural Recovery	Stored (% Remaining)
Fluensulfone	0 / 0	0.10	85, 117, 105	–
	92 / 3		97, 94	97, 87, 98
	244 / 8		112, 108	109, 108, 108
	469 / 15		78, 98	96, 99, 100
TSA	0 / 0	0.10	101, 89, 97	–
	92 / 3		88, 84	91, 90, 91
	244 / 8		114, 95	102, 92, 108
	469 / 15		90, 89	94, 95, 93
BSA	0 / 0	0.10	96, 95, 99	–
	92 / 3		88, 89	87, 81, 84
	244 / 8		105, 95	96, 86, 104
	469 / 15		93, 97	96, 106, 98

Table 69 Storage stability of fluensulfone, TSA, MES and BSA in tomato puree

Analyte	Storage Interval days / months	Spiking Level (mg/kg)	Percent of Nominal Spiking Level	
			Procedural Recovery	Stored (% Remaining)
Fluensulfone	27 / 1	0.10	104, 107	103, 108
	89 / 3		101, 100	98, 86
	181 / 6		80, 84	73, 75
TSA	27 / 1	0.10	80, 74	80, 78
	89 / 3		81, 81	81, 88
	181 / 6		72, 73	79, 81
MES	27 / 1	0.10	88, 88	85, 90
	89 / 3		93, 98	100, 82
	181 / 6		89, 86	88, 87
BSA	27 / 1	0.10	89, 98	93, 98
	89 / 3		93, 95	93, 94
	181 / 6		92, 95	98, 99

Table 70 Storage stability of fluensulfone, TSA, MES and BSA in tomato paste

Analyte	Storage Interval days / months	Spiked Level (mg/kg)	Percent of Nominal Spiking Level	
			Procedural Recovery	Stored (% Remaining)
Fluensulfone	90 / 3	0.10	97, 87	79, 74
	181 / 6		94, 92	83, 73
TSA	90 / 3	0.10	62, 63	65, 65
	181 / 6		63, 63	67, 69
MES	90 / 3	0.10	92, 88	89, 86
	181 / 6		82, 88	89, 88
BSA	90 / 3	0.10	73, 80	87, 85
	181 / 6		85, 84	89, 93

USE PATTERN

Table 71 Good agricultural practices (GAPs) proposed for fluensulfone ^a

Crop and/ or Situation	Pests or Group of Pests Controlled	Application Method Kind	Growth Stage & Season	No.	Interval Between Applications (min)	Rate (kg ai/ha, min–max)	PHI (days)
U.S.A Registration							
Cucurbit	Root-knot,	Drip irrigation,	Pre-planting	1	N/A	1.92–2.8	N/A

Crop and/or Situation	Pests or Group of Pests Controlled	Application Method Kind	Growth Stage & Season	No.	Interval Between Applications (min)	Rate (kg ai/ha, min–max)	PHI (days)
vegetables & fruiting vegetables	root-lesion and cyst nematodes	Band application, Broadcast spray	(minimum of 7 days before transplanting) ^b				

^a Representative formulation is an EC containing 480 g/L (4 lb/gal) of fluensulfone.

^b Only outdoor uses are permitted.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received data from supervised residue trials conducted on cucumber, summer squash, cantaloupe, tomato, and pepper conducted in the USA and Canada. For all samples, analyses were conducted for fluensulfone, BSA, and TSA, and analysis of MeS was conducted for at least some samples from each crop. The reports included method validation data, as recoveries from spiked samples at levels reflecting those observed in the field trial samples; dates from critical events during the study, including application, harvest, storage, and analysis; as well as detailed information on the field site and treatment parameters. Analytical reports were sufficiently detailed and included example chromatograms and example calculations. Samples were analysed by Method 1997W (equivalent to Method 2061W) described above. The results are supported by concurrent recoveries ranging, on average for each analyte and crop, from 81–120% for all commodity × analyte combinations except for fluensulfone in dry tomato pomace, which averaged 131% in one study. Samples were stored, frozen, for the following durations: cucumber = 10 months, squash = 10 months, melons = 9 months, pepper = 7 months, and tomato = 7 months. The storage durations are less than or equal to those for which residues have been demonstrated to be stable.

The field trial study designs included control plots. All measured residues from control plots were < 0.01 mg/kg (i.e., < LOQ) and are not included in the summary tables in this evaluation.

Supervised trials for fluensulfone:

Commodity	Crop	Table
Fruiting vegetable, cucurbit	Cucumber (VC 0424)	Table 72
Fruiting vegetable, cucurbit	Summer squash (VC 0431)	Table 73
Fruiting vegetable, cucurbit	Cantaloupe/muskmelon (VC 4199/VC 4239)	Table 74, 75
Fruiting vegetable, other than cucurbit	Pepper (VO 0051)	Table 76
Fruiting vegetable, other than cucurbit	Tomato (VO 0448)	Table 77

Fruiting Vegetables, Cucurbits

Cucumber

Table 72 Residues of fluensulfone, BSA, MeS, and TSA in cucumber following pre-plant treatment.

Location Year [Variety]	Applic. rate, kg ai/ha	Applic. volume, L/ha	Applic. timing	Applic. type	DAT	Residue, mg/kg			
						Fluensulfone	BSA	MeS	TSA
(Reference)									
cGAP - USA	3.84		7 days pre- plantin g	drip irrig., band applic., broadcast spray	–				
Montezuma, Georgia, USA 2010 [Speedway]	4.01	2087	3 days pre- plantin g	drip irrigation	46	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	0.039, 0.058 (0.049)
(09-01859)					49	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	0.056, 0.065 (0.061)
					53	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	0.052, 0.049 (0.051)
					56	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	0.050, 0.085 (0.068)
					60	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	0.039, 0.035 (0.037)
Clermont, Florida, USA 2009 [Marketmore 76]	4.00	2083	3 days pre- plantin g	drip irrigation	70	< 0.01, < 0.01 (< 0.01)	0.012, 0.019 (0.016)	n.a.	0.020, 0.035 (0.028)
(09-01859)					73	< 0.01, < 0.01 (< 0.01)	0.013, < 0.01 (0.011)	n.a.	0.029, 0.014 (0.022)
					77	< 0.01, < 0.01 (< 0.01)	0.012, < 0.01 (0.011)	n.a.	0.028, 0.014 (0.021)
					80	< 0.01, < 0.01 (< 0.01)	< 0.01, 0.012 (0.011)	n.a.	0.013, 0.026 (0.020)
					84	< 0.01, < 0.01 (< 0.01)	< 0.01, 0.022 (0.016)	n.a.	0.020, 0.037 (0.029)
Fresno, California, USA 2009 [Straight Eight]	4.00	2083	3 days pre- plantin g	drip irrigation	78	< 0.01, < 0.01 (< 0.01)	0.048, 0.071 (0.060)	n.a.	0.063, 0.090 (0.077)
(09-01859)					81	< 0.01, < 0.01 (< 0.01)	0.030, 0.032 (0.031)	n.a.	0.050, 0.038 (0.044)
					85	< 0.01, < 0.01 (< 0.01)	0.033, 0.032 (0.033)	n.a.	0.056, 0.046 (0.051)
					88	< 0.01, < 0.01 (< 0.01)	0.035, 0.028 (0.032)	n.a.	0.040, 0.040 (0.040)
					92	< 0.01,	0.034, 0.018	n.a.	0.052, 0.044

Location Year [Variety]	Applic. rate, kg ai/ha	Applic. volume, L/ha	Applic. timing	Applic. type	DAT	Residue, mg/kg			
(Reference)						Fluensulfone	BSA	MeS	TSA
						< 0.01 (< 0.01)	(0.026)		(0.048)
Visalia, California, USA 2010 [Poinsett 76] (AA100708)	4.09	379	7 d pre- plantin g	BCS	70	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
	4.00	9000	7 d pre- plantin g	drip irrigation	70	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
Hobe Sound, Florida, USA 2010 [Impact] (AA100708)	3.87	222	7 d pre- plantin g	BCS	45	< 0.01, < 0.01 (< 0.01)	0.076, 0.064 (0.070)	0.028, 0.029 (0.029)	0.048, 0.040 (0.044)
Athens, Georgia, USA 2010 [Burpleess Hybrid] (AA100708)	4.05	361	7 d pre- plantin g	BCS	71	< 0.01, < 0.01 (< 0.01)	0.010, 0.010 (0.010)	0.011, 0.011 (0.011)	0.081, 0.084 (0.083)
Seven Springs, North Carolina, USA 2010 [Ashley] (AA100708)	4.10	358	7 d pre- plantin g	BCS	41	< 0.01, < 0.01 (< 0.01)	0.085, 0.215 (0.150)	0.021, 0.024 (0.023)	0.408, 0.524 (0.47)
	4.00	9000	7 d pre- plantin g	drip irrigation	41	< 0.01, < 0.01 (< 0.01)	0.353 , 0.084 (0.22)	0.035, 0.042 (0.039)	0.718, 0.500 (0.61)
Raymondville, Texas, USA 2010 [Sweet Slice] (AA100708)	3.72	261	7 d pre- plantin g	BCS	46	< 0.01, < 0.01 (< 0.01)	0.176, 0.164 (0.17)	0.079, 0.071 (0.075)	0.522, 0.507 (0.52)
Thorndale, Ontario, Canada 2010 [Cross Country] (AA100708)	3.81	381	8 d pre- plantin g	BCS	50	< 0.01, < 0.01 (< 0.01)	0.045, 0.081 (0.063)	0.061, 0.075 (0.068)	0.198, 0.258 (0.23)
Portage la Prairie, Manitoba, Canada 2010 [Slicing] (AA100708)	4.11	359	7 d pre- plantin g	BCS	73	< 0.01, < 0.01 (< 0.01)	< 0.01, 0.011 (0.010)	< 0.01, < 0.01 (< 0.01)	0.058, 0.070 (0.064)

Applic. Type BCS = broadcast spray

Residue n.a. = Not analysed

Summer squash

Table 73 Residues of fluensulfone, BSA, MeS, and TSA in summer squash following pre-plant treatment

Location Year [Variety]	Applic. rate, kg ai/ha	Applic. volume, L/ha	Applic. timing	Applic. type	DAT	Residue, mg/kg			
(Reference)						Fluensulfone	BSA	MeS	TSA
cGAP - USA	3.84		7 days pre-planting	drip irrig., band applic., broadcast spray	–				
Porterville, California, USA 2010 [Dark Green Zucchini] (AA100708)	3.82	286	7 d pre-planting	BCS	49	< 0.01, < 0.01 (< 0.01)	0.014, 0.010 (0.012)	< 0.01, < 0.01 (< 0.01)	0.040, 0.028 (0.034)
	4.00	9000	7 d pre-planting	drip irrigation	49	< 0.01, < 0.01 (< 0.01)	0.062, 0.058 (0.060)	< 0.01, < 0.01 (< 0.01)	0.127, 0.079 (0.10)
Dover, Florida, USA 2010 [Enterprise] (AA100708)	4.01	250	7 d pre-planting	BCS	36	< 0.01, < 0.01 (< 0.01)	0.086, 0.078 (0.082)	< 0.01, < 0.01 (< 0.01)	0.117, 0.106 (0.11)
Seven Springs, North Carolina, USA 2010 [Early Prolific Straight neck] (AA100708)	4.02	352	7 d pre-planting	BCS	41	< 0.01, < 0.01 (< 0.01)	0.206, 0.165 (0.19)	0.033, 0.037 (0.035)	0.715, 0.701 (0.71)
Hinton, Oklahoma, USA 2010 [Enterprise] (AA100708)	4.02	256	6 d pre-planting	BCS	61	< 0.01, < 0.01 (< 0.01)	0.196, 0.231 (0.21)	0.023, 0.018 (0.021)	0.366, 0.563 (0.46)
Ephrata, Washington, USA 2010 [Aristocrat] (AA100708)	4.14	389	7 d pre-planting	BCS	62	< 0.01, < 0.01 (< 0.01)	0.256 , 0.237 (0.25)	0.050, 0.045 (0.048)	0.267, 0.237 (0.25)
Berwick, Nova Scotia, Canada 2010 [Payroll] (AA100708)	4.07	363	7 d pre-planting	BCS	38	< 0.01, < 0.01 (< 0.01)	0.222, 0.169 (0.20)	0.016, 0.011 (0.014)	0.292, 0.253 (0.27)
Branchton, Ontario, Canada 2010 [Senator] (AA100708)	3.89	241	6 d pre-planting	BCS	45	< 0.01, 0.017 (< 0.013)	0.065, 0.035 (0.00)	0.013, 0.010 (0.012)	0.063, 0.051 (0.057)
	4.00	9000	6 d pre-planting	drip irrigation	45	< 0.01, < 0.01 (< 0.01)	0.029, 0.060 (0.045)	< 0.01, < 0.01 (< 0.01)	0.038, 0.061 (0.050)
Elm Creek, Manitoba,	3.80	286	7 d pre-planting	BCS	71	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01	< 0.01, < 0.01	0.036, 0.034 (0.035)

Location Year [Variety]	Applic. rate, kg ai/ha	Applic. volume, L/ha	Applic. timing	Applic. type	DAT	Residue, mg/kg			
(Reference)						Fluensulfone	BSA	MeS	TSA
Canada 2010 [Zucchini] (AA100708)			g				(< 0.01)	(< 0.01)	

Applic. Type BCS = broadcast spray

Melons

Table 74 Residues of fluensulfone, BSA, MeS, and TSA in melons following pre-plant treatment

Location Year [Variety]	Applic. rate, kg ai/ha	Applic. volume, L/ha	Applic. timing	Applic. type	DAT	Residue, mg/kg			
(Reference)						Fluensulfone	BSA	MeS	TSA
cGAP - USA	3.84		7 days pre- plantin g	drip irrig., band applic., broadcast spray	–				
Montezuma, Georgia, USA 2009 [Ambrosia Hybrid] (09-01859)	4.00	2087	3 days pre- plantin g	drip irrigation	70	< 0.01, < 0.01 (< 0.01)	0.010, < 0.01 (< 0.01)	n.a.	0.067, 0.053 (0.060)
					73	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	0.045, 0.037 (0.041)
					77	< 0.01, < 0.01 (< 0.01)	< 0.01, 0.010 (< 0.01)	n.a.	0.046, 0.057 (0.052)
					80	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	0.031, 0.031 (0.031)
Fresno, California, USA 2009 [Yuma Grande F1] (09-01859)	4.00	2083	3 days pre- plantin g	drip irrigation	77	< 0.01, < 0.01 (< 0.01)	0.091, 0.089 (0.090)	n.a.	0.144, 0.177 (0.16)
					80	< 0.01, < 0.01 (< 0.01)	0.117, 0.101 (0.11)	n.a.	0.197, 0.215 (0.21)
					84	< 0.01, < 0.01 (< 0.01)	0.070, 0.074 (0.072)	n.a.	0.146, 0.156 (0.15)
					87	< 0.01, < 0.01 (< 0.01)	0.060, 0.060 (0.060)	n.a.	0.128, 0.128 (0.13)
					91	< 0.01, < 0.01 (< 0.01)	0.050, 0.042 (0.046)	n.a.	0.090, 0.109 (0.10)
King City, California, USA 2010 [Hale's Best]	3.93	383	7 d pre- plantin g	BCS	133	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	0.016, 0.018 (0.017)

Location Year [Variety]	Applic. rate, kg ai/ha	Applic. volume, L/ha	Applic. timing	Applic. type	DAT	Residue, mg/kg			
						Fluensulfone	BSA	MeS	TSA
(Reference)									
Jumbo] (AA100708)									
Arroyo Grande, California, USA 2010 [Top Mark] (AA100708)	3.85	365	7 d pre- plantin g	BCS	97	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
Seven Springs, North Carolina, USA 2010 [Hales Best] (AA100708)	4.10	359	7 d pre- plantin g	BCS	66	< 0.01, < 0.01 (< 0.01)	0.061, 0.036 (0.049)	< 0.01, < 0.01 (< 0.01)	0.673, 0.437 (0.56)
Hinton, Oklahoma, USA 2010 [Caravelle] (AA100708)	3.98	253	6 d pre- plantin g	BCS	82	< 0.01, < 0.01 (< 0.01)	0.065 , 0.062 (0.064)	< 0.01, < 0.01 (< 0.01)	0.303, 0.294 (0.30)
Branchton, Ontario, Canada 2010 [Primo] (AA100708)	4.04	253	6 d pre- plantin g	BCS	80	< 0.01, < 0.01 (< 0.01)	0.015, 0.027 (0.021)	< 0.01, < 0.01 (< 0.01)	0.032, 0.050 (0.041)
Portage la Prairie, Manitoba, Canada 2010 [Athena] (AA100708)	4.11	359	7 d pre- plantin g	BCS	91	< 0.01, < 0.01 (< 0.01)	0.034, 0.030 (0.032)	< 0.01, < 0.01 (< 0.01)	0.172, 0.154 (0.16)
Branchton, Ontario, Canada 2010 [Early sweet] (AA100708)	4.02	251	6 d pre- plantin g	BCS	83	< 0.01, < 0.01 (< 0.01)	0.030, 0.020 (0.025)	< 0.01, < 0.01 (< 0.01)	0.102, 0.091 (0.097)
Corning, California, USA 2010 [ACR 215] (AA100708)	4.02	233	7 d pre- plantin g	BCS	92	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)

Applic. Type BCS = broadcast spray

Residue n.a. = Not analysed

In a magnitude-of-the-residue study by Chevalier (2011, Report R-23486), muskmelon were grown under protected conditions in Southern Europe (Greece, Italy, and Spain) in soil that had been treated with fluensulfone by drip irrigation, at a rate of ca. 4.0 kg ai/ha, 7 days prior to transplanting. The melons were harvested at maturity, 62–79 days after application as well as for residue decline (up to 98 days after application), and stored frozen until preparation for analysis. Preparation consisted of separating the melon samples into their pulp and peel components, homogenizing the fractions, and

then storing them frozen prior to analysis. Analysis was by Method 1977W and the equivalent Method 2061W.

Table 75 Residues of fluensulfone, BSA, MeS, and TSA in melon pulp, peel, and whole fruit following treatment by drip irrigation seven days pre-transplant

Location (Region) Year [Variety]	Applic. rate, kg ai/ha	Applic. volume, L/ha	DAT, days	Fraction	Residue, mg/kg				Reference
					Fluen.	BSA	MeS	TSA	
cGAP - USA	3.84		—						
La Palma, Murcia, Spain (Southern Europe—Indoor) 2010 [Cantasapo]	3.94	9000	77	peel	< 0.01	0.01	n.a.	< 0.01	BPL10/237/CL
				pulp	< 0.01	< 0.01	n.a.	< 0.01	
				whole fruit	< 0.01	0.01	n.a.	< 0.01	
Ramonete, Murcia, Spain (Southern Europe—Indoor) 2010 [Gabriel]	3.94	18000	64	peel	< 0.01	< 0.01	n.a.	< 0.01	BPL10/237/CL
				pulp	< 0.01	< 0.01	n.a.	< 0.01	
				whole fruit	< 0.01	< 0.01	n.a.	< 0.01	
La Puebla, Murcia, Spain (Southern Europe—Indoor) 2010 [Sancho]	3.94	18000	63	peel	< 0.01	< 0.01	< 0.01	< 0.01	BPL10/237/CL
				pulp	< 0.01	< 0.01	< 0.01	< 0.01	
				whole fruit	< 0.01	< 0.01	< 0.01	< 0.01	
La Puebla, Murcia, Spain (Southern Europe—Indoor) 2010 [Sancho]	3.94	18000	66	peel	< 0.01	< 0.01	n.a.	< 0.01	BPL10/237/CL
				pulp	< 0.01	< 0.01	n.a.	< 0.01	
				whole fruit	< 0.01	< 0.01	—	< 0.01	
La Puebla, Murcia, Spain (Southern Europe—Indoor) 2010 [Sancho]	3.94	18000	70	peel	< 0.01	< 0.01	n.a.	< 0.01	BPL10/237/CL
				pulp	< 0.01	< 0.01	n.a.	< 0.01	
				whole fruit	< 0.01	< 0.01	—	< 0.01	
La Puebla, Murcia, Spain (Southern Europe—Indoor) 2010 [Sancho]	3.94	18000	73	peel	< 0.01	0.02	n.a.	< 0.01	BPL10/237/CL
				pulp	< 0.01	< 0.01	n.a.	< 0.01	
				whole fruit	< 0.01	0.01	—	< 0.01	
La Puebla, Murcia, Spain (Southern Europe—Indoor) 2010 [Sancho]	3.94	18000	78	peel	< 0.01	< 0.01	< 0.01	< 0.01	BPL10/237/CL
				pulp	< 0.01	< 0.01	< 0.01	< 0.01	
				whole fruit	< 0.01	< 0.01	< 0.01	< 0.01	
Camacici di Sangioanni Lupatoto, Verona, Italy (Southern Europe—Indoor) 2010 [Giusto]	3.94	9000	70	peel	< 0.01	0.20	n.a.	0.15	BPL10/237/CL
				pulp	< 0.01	0.09	n.a.	0.04	
				whole fruit	< 0.01	0.13	n.a.	0.08	
Salizzole, Verona, Italy (Southern Europe—Indoor)	3.94	9000	62	peel	< 0.01	0.25	n.a.	0.46	BPL10/237/CL

Location (Region) Year [Variety]	Applic. rate, kg ai/ha	Applic. volume, L/ha	DAT, days	Fraction	Residue, mg/kg				Reference
					Fluen.	BSA	MeS	TSA	
2010 [Macigno]									
				pulp	< 0.01	0.17	n.a.	0.20	
				whole fruit	< 0.01	0.20	n.a.	0.31	
Camacici di Sangiovanni Lupatoto, Verona, Italy (Southern Europe—Indoor) 2010 [Macigno]	3.94	9000	67	peel	< 0.01	0.03	< 0.01	0.02	BPL10/237/CL
				pulp	< 0.01	0.02	< 0.01	< 0.01	
				whole fruit	< 0.01	0.02	< 0.01	0.01	
Camacici di Sangiovanni Lupatoto, Verona, Italy (Southern Europe—Indoor) 2010 [Macigno]	3.94	9000	70	peel	< 0.01	0.04	n.a.	0.04	BPL10/237/CL
				pulp	< 0.01	0.02	n.a.	< 0.01	
				whole fruit	< 0.01	0.03	—	0.02	
Camacici di Sangiovanni Lupatoto, Verona, Italy (Southern Europe—Indoor) 2010 [Macigno]	3.94	9000	74	peel	< 0.01	0.06	n.a.	0.04	BPL10/237/CL
				pulp	< 0.01	0.02	n.a.	< 0.01	
				whole fruit	< 0.01	0.03	—	0.02	
Camacici di Sangiovanni Lupatoto, Verona, Italy (Southern Europe—Indoor) 2010 [Macigno]	3.94	9000	77	peel	< 0.01	0.11	n.a.	0.08	BPL10/237/CL
				pulp	< 0.01	0.03	n.a.	0.01	
				whole fruit	< 0.01	0.06	—	0.03	
Camacici di Sangiovanni Lupatoto, Verona, Italy (Southern Europe—Indoor) 2010 [Macigno]	3.94	9000	81	peel	< 0.01	0.07	< 0.01	0.05	BPL10/237/CL
				pulp	< 0.01	0.01	< 0.01	0.01	
				whole fruit	< 0.01	0.03	< 0.01	0.02	
Chalkidona, Macedonia, Greece (Southern Europe—Indoor) 2010 [Lavigal]	3.94	9000	79	peel	< 0.01	0.01	n.a.	0.07	BPL10/237/CL
				pulp	< 0.01	< 0.01	n.a.	0.02	
				whole fruit	< 0.01	0.01	n.a.	0.05	
Nea Magnisia, Macedonia, Greece (Southern Europe—Indoor) 2010 [Galia]	3.94	9000	84	peel	< 0.01	0.03	< 0.01	0.15	BPL10/237/CL
				pulp	< 0.01	0.01	< 0.01	0.05	
				whole fruit	< 0.01	0.02	< 0.01	0.10	
Nea Magnisia, Macedonia, Greece (Southern Europe—Indoor) 2010 [Galia]	3.94	9000	87	peel	< 0.01	0.02	n.a.	0.16	BPL10/237/CL
				pulp	< 0.01	< 0.01	n.a.	0.05	
				whole fruit	< 0.01	0.02	—	0.11	
Nea Magnisia, Macedonia,	3.94	9000	91	peel	< 0.01	0.04	n.a.	0.19	BPL10/237/CL

Location (Region) Year [Variety]	Applic. rate, kg ai/ha	Applic. volume, L/ha	DAT, days	Fraction	Residue, mg/kg				Reference
					Fluen.	BSA	MeS	TSA	
Greece (Southern Europe—Indoor) 2010 [Galia]									
				pulp	< 0.01	0.02	n.a.	0.09	
				whole fruit	< 0.01	0.03	–	0.14	
Nea Magnisia, Macedonia, Greece (Southern Europe—Indoor) 2010 [Galia]	3.94	9000	94	peel	< 0.01	0.05	n.a.	0.30	BPL10/237/CL
				pulp	< 0.01	0.01	n.a.	0.09	
				whole fruit	< 0.01	0.03	–	0.19	
Nea Magnisia, Macedonia, Greece (Southern Europe—Indoor) 2010 [Galia]	3.94	9000	98	peel	< 0.01	0.03	< 0.01	0.14	BPL10/237/CL
				pulp	< 0.01	< 0.01	< 0.01	0.05	
				whole fruit	< 0.01	0.02	< 0.01	0.09	
Average Ratio				peel/fruit	–	1.52	–	1.80	–
				pulp/fruit	–	0.62	–	0.50	–

n.a. = not analysed

*Fruiting vegetables, other than Cucurbits**Peppers*

Table 76 Residues of fluensulfone, BSA, MeS, and TSA in pepper following pre-plant treatment

Location Year [Variety]	Applic. rate, kg ai/ha	Applic. volume, L/ha	Applic. timing	Applic. type	DAT	Residue, mg/kg			
						Fluensulfone	BSA	MeS	TSA
(Reference)									
cGAP - USA	3.84		7 days pre-planting	drip irrig., band applic., broadcast spray	–				
Chili Pepper									
Montezuma, Georgia, USA 2009 [Aristotle X3R] (09-01858)	4.01	2087	3 days pre-planting	drip irrigation	53	< 0.01, < 0.01 (< 0.01)	0.019, 0.018 (0.019)	n.a.	0.064, 0.069 (0.067)
					56	< 0.01, < 0.01 (< 0.01)	0.022, 0.020 (0.021)	n.a.	0.073, 0.064 (0.069)
					60	< 0.01, < 0.01 (< 0.01)	0.015, 0.015 (0.015)	n.a.	0.074, 0.069 (0.072)
					63	< 0.01, < 0.01 (< 0.01)	0.016, 0.016 (0.016)	n.a.	0.065, 0.070 (0.068)

Location Year [Variety]	Applic. rate, kg ai/ha	Applic. volume, L/ha	Applic. timing	Applic. type	DAT	Residue, mg/kg			
(Reference)						Fluensulfone	BSA	MeS	TSA
					67	< 0.01, < 0.01 (< 0.01)	0.014, 0.016 (0.015)	n.a.	0.052, 0.058 (0.055)
	4.01	2087	30 days post- plantin g	drip irrigation	20	< 0.01, < 0.01 (< 0.01)	0.230, 0.207 (0.22)	n.a.	0.140, 0.108 (0.12)
					23	< 0.01, < 0.01 (< 0.01)	0.219, 0.202 (0.21)	n.a.	0.125, 0.125 (0.12)
					27	< 0.01, < 0.01 (< 0.01)	0.181, 0.227 (0.20)	n.a.	0.118, 0.147 (0.13)
					30	< 0.01, < 0.01 (< 0.01)	0.184, 0.185 (0.18)	n.a.	0.117, 0.110 (0.11)
					34	< 0.01, < 0.01 (< 0.01)	0.165, 0.206 (0.186)	n.a.	0.090, 0.099 (0.095)
Clermont, Florida, USA 2009 [Patriot] (09-01858)	4.02	2088	3 days pre- plantin g	drip irrigation	65	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	< 0.01, < 0.01 (< 0.01)
					68	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	< 0.01, < 0.01 (< 0.01)
					72	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	< 0.01, < 0.01 (< 0.01)
					75	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	< 0.01, < 0.01 (< 0.01)
					79	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	0.011, < 0.01 (0.010)
	4.02	2088	36 days post- plantin g	drip irrigation	26	< 0.01, < 0.01 (< 0.01)	0.032, 0.030 (0.031)	n.a.	< 0.01, < 0.01 (< 0.01)
					29	< 0.01, < 0.01 (< 0.01)	0.027, 0.033 (0.030)	n.a.	< 0.01, < 0.01 (< 0.01)
					33	< 0.01, < 0.01 (< 0.01)	0.027, 0.021 (0.024)	n.a.	< 0.01, < 0.01 (< 0.01)
					36	< 0.01, < 0.01 (< 0.01)	0.019, 0.018 (0.019)	n.a.	< 0.01, < 0.01 (< 0.01)
					40	< 0.01, < 0.01 (< 0.01)	0.020, 0.021 (0.021)	n.a.	< 0.01, < 0.01 (< 0.01)
Porterville, California, USA 2010 [Fresno] (AA100707)	3.98	301	7 d pre- plantin g	BCS	83	< 0.01, < 0.01 (< 0.01)	0.049, 0.031 (0.040)	< 0.01, < 0.01 (< 0.01)	0.037, 0.033 (0.035)
	3.93 + 2.0	297 + 9000	7 d pre- plantin g + 40	BCS + drip irrigation	43	< 0.01, < 0.01 (< 0.01)	0.360, 0.392 (0.38)	< 0.01, < 0.01 (< 0.01)	0.137, 0.156 (0.15)

Location Year [Variety]	Applic. rate, kg ai/ha	Applic. volume, L/ha	Applic. timing	Applic. type	DAT	Residue, mg/kg			
(Reference)						Fluensulfone	BSA	MeS	TSA
			d post plant.						
					46	< 0.01, < 0.01 (< 0.01)	0.301, 0.252 (0.28)	< 0.01, < 0.01 (< 0.01)	0.147, 0.139 (0.14)
					51	< 0.01, < 0.01 (< 0.01)	0.282, 0.363 (0.32)	0.011, < 0.01 (0.010)	0.180, 0.155 (0.17)
					53	< 0.01, < 0.01 (< 0.01)	0.265, 0.318 (0.29)	0.010, < 0.01 (< 0.01)	0.118, 0.166 (0.14)
					56	< 0.01, < 0.01 (< 0.01)	0.226, 0.155 (0.19)	< 0.01, < 0.01 (< 0.01)	0.174, 0.145 (0.16)
Oviedo, Florida, USA 2010 [Sweet Banana] (AA100707)	3.90	279	7 d pre- plantin g	BCS	50	< 0.01, < 0.01 (< 0.01)	0.201 , 0.167 (0.18)	< 0.01, < 0.01 (< 0.01)	0.092, 0.086 (0.089)
	3.92 + 2.0	280 + 9000	7 d pre- plantin g + 40 d post plant.	BCS + drip irrigation	3	< 0.01, < 0.01 (< 0.01)	0.250, 0.223 (0.24)	< 0.01, < 0.01 (< 0.01)	0.092, 0.076 (0.084)
Hinton, Oklahoma, USA 2010 [Tam Jalapeno] (AA100707)	4.03	539	7 d pre- plantin g	BCS	101	< 0.01, < 0.01 (< 0.01)	0.044, 0.037 (0.041)	< 0.01, < 0.01 (< 0.01)	0.224, 0.274 (0.25)
	4.08 + 2.0	546 + 9006	7 d pre- plantin g + 40 d post plant.	BCS + drip irrigation	53	< 0.01, < 0.01 (< 0.01)	0.092, 0.084 (0.088)	0.011, < 0.01 (0.010)	0.389, 0.367 (0.38)
Sweet Pepper									
Fresno, California, USA 2009 [Baron] (09-01858)	4.00	2088	3 days pre- plantin g	drip irrigation	102	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	< 0.01, 0.010 (< 0.01)
					105	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	< 0.01, < 0.01 (< 0.01)
					109	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	< 0.01, < 0.01 (< 0.01)
					112	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	< 0.01, < 0.01 (< 0.01)
					116	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	< 0.01, < 0.01 (< 0.01)
	4.00	2088	38 days post- plantin g	drip irrigation	61	< 0.01, < 0.01 (< 0.01)	0.041, 0.040 (0.041)	n.a.	0.021, 0.016 (0.019)
					64	< 0.01,	0.025, 0.032	n.a.	0.013, 0.017

Location Year [Variety]	Applic. rate, kg ai/ha	Applic. volume, L/ha	Applic. timing	Applic. type	DAT	Residue, mg/kg			
(Reference)						Fluensulfone	BSA	MeS	TSA
						< 0.01 (< 0.01)	(0.029)		(0.015)
					68	< 0.01, < 0.01 (< 0.01)	0.029, 0.026 (0.028)	n.a.	0.013, 0.015 (0.014)
					71	< 0.01, < 0.01 (< 0.01)	0.027, 0.021 (0.024)	n.a.	0.015, 0.014 (0.015)
					75	< 0.01, < 0.01 (< 0.01)	0.019, 0.018 (0.019)	n.a.	0.010, 0.011 (0.011)
Porterville, California, USA 2010 [California Wonder] (AA100707)	3.87	342	7 d pre- plantin g	BCS	104	< 0.01, < 0.01 (< 0.01)	0.067, 0.072 (0.070)	< 0.01, < 0.01 (< 0.01)	0.027, 0.027 (0.027)
	4.01	9000	7 d pre- plantin g	drip irrigation	104	< 0.01, < 0.01 (< 0.01)	0.073, 0.062 (0.068)	< 0.01, < 0.01 (< 0.01)	0.080, 0.067 (0.074)
	3.9 + 2.0	345 + 9000	7 d pre- plantin g + 40 d post plant.	BCS + drip irrigation	63	< 0.01, < 0.01 (< 0.01)	0.063, 0.055 (0.059)	< 0.01, < 0.01 (< 0.01)	0.031, 0.027 (0.029)
Oviedo, Florida, USA 2010 [Green Bell] (AA100707)	3.97	284	7 d pre- plantin g	BCS	63	< 0.01, < 0.01 (< 0.01)	0.225, 0.239 (0.23)	0.014, 0.012 (0.013)	0.150, 0.188 (0.17)
	3.94 + 2.0	282 + 9108	7 d pre- plantin g + 40 d post plant.	BCS + drip irrigation	16	< 0.01, < 0.01 (< 0.01)	0.379, 0.372 (0.38)	0.012, 0.010 (0.011)	0.169, 0.170 (0.17)
Seven Springs, North Carolina, USA 2010 [Jupiter] (AA100707)	4.02	351	7 d pre- plantin g	BCS	73	< 0.01, < 0.01 (< 0.01)	0.055, 0.054 (0.055)	< 0.01, < 0.01 (< 0.01)	0.438, 0.483 (0.46)
	3.99 + 2.00	349 + 6279	7 d pre- plantin g + 40 d post plant.	BCS + drip irrigation	26	< 0.01, < 0.01 (< 0.01)	0.116, 0.141 (0.13)	< 0.01, < 0.01 (< 0.01)	1.056, 1.285 (1.2)
					29	< 0.01, < 0.01 (< 0.01)	0.109, 0.129 (0.12)	< 0.01, < 0.01 (< 0.01)	0.915, 1.030 (0.97)
					33	< 0.01, < 0.01 (< 0.01)	0.166, 0.115 (0.14)	< 0.01, < 0.01 (< 0.01)	1.144, 0.853 (1.0)
					35	< 0.01, < 0.01 (< 0.01)	0.131, 0.052 (0.092)	< 0.01, < 0.01 (< 0.01)	1.012, 0.515 (0.76)
					40	< 0.01, < 0.01 (< 0.01)	0.099, 0.092 (0.096)	< 0.01, < 0.01 (< 0.01)	0.898, 0.933 (0.92)
Hinton, Oklahoma,	4.10	534	7 d pre- plantin	BCS	108	< 0.01, < 0.01	0.086, 0.078 (0.082)	< 0.01, < 0.01	0.341, 0.349 (0.34)

Location Year [Variety]	Applic. rate, kg ai/ha	Applic. volume, L/ha	Applic. timing	Applic. type	DAT	Residue, mg/kg			
(Reference)						Fluensulfone	BSA	MeS	TSA
USA 2010 [XR3 Camelot Sweet Bell] (AA100707)			g			(< 0.01)		(< 0.01)	
	4.11 + 2.00	535 + 9002	7 d pre- plantin g + 40 d post plant.	BCS + drip irrigation	64	< 0.01, < 0.01 (< 0.01)	0.150, 0.160 (0.16)	< 0.01, < 0.01 (< 0.01)	0.507, 0.424 (0.47)
Thorndale, Ontario, Canada 2010 [Revolution] (AA100707)	3.84	384	7 d pre- plantin g	BCS	63	< 0.01, < 0.01 (< 0.01)	0.065, 0.060 (0.063)	< 0.01, < 0.01 (< 0.01)	0.286, 0.274 (0.28)
	3.85+ 2.00	385 + 900	7 d pre- plantin g + 40 d post plant.	BCS + drip irrigation	46	< 0.01, < 0.01 (< 0.01)	0.268, 0.244 (0.26)	0.016, 0.017 (0.017)	0.313, 0.348 (0.33)
Portage la Prairie, Manitoba, Canada 2010 [California Wonder] (AA100707)	4.07	355	7 d pre- plantin g	BCS	102	< 0.01, < 0.01 (< 0.01)	0.049, 0.047 (0.048)	< 0.01, < 0.01 (< 0.01)	0.169, 0.162 (0.17)
	4.14+ 2.00	362 + 9000	7 d pre- plantin g + 40 d post plant.	BCS + drip irrigation	46	< 0.01, < 0.01 (< 0.01)	0.151, 0.166 (0.16)	< 0.01, < 0.01 (< 0.01)	0.172, 0.162 (0.17)
Branchton, Ontario, Canada 2010 [Aristotle] (AA100707)	3.82	239	7 d pre- plantin g	BCS	76	< 0.01, < 0.01 (< 0.01)	0.027, 0.036 (0.032)	< 0.01, < 0.01 (< 0.01)	0.059, 0.073 (0.066)
	4.00	9000	7 d pre- plantin g	drip irrigation	76	< 0.01, < 0.01 (< 0.01)	0.068, 0.077 (0.073)	< 0.01, < 0.01 (< 0.01)	0.114, 0.118 (0.12)
	4.07 + 4.00	254 + 9000	7 d pre- plantin g + 40 d post plant.	BCS + drip irrigation	27	< 0.01, < 0.01 (< 0.01)	0.128, 0.164 (0.15)	< 0.01, < 0.01 (< 0.01)	0.077, 0.075 (0.076)
Portage la Prairie, Manitoba, Canada 2010 [Hungarian Yellow Wax] (AA100707)	4.07	356	7 d pre- plantin g	BCS	102	< 0.01, < 0.01 (< 0.01)	0.141, 0.130 (0.14)	0.012, 0.012 (0.012)	0.198, 0.181 (0.19)
	3.93+ 2.00	344 + 9000	7 d pre- plantin g + 40 d post plant.	BCS + drip irrigation	56	< 0.01, < 0.01 (< 0.01)	0.297, 0.275 (0.29)	0.024, 0.019 (0.022)	0.162, 0.143 (0.15)

Applic. Type BCS = broadcast spray

Residue n.a. = not analysed

Tomatoes

Table 77 Residues of fluensulfone, BSA, MeS, and TSA in tomato following pre-plant treatment

Location Year [Variety]	Applic. rate, kg ai/ha	Applic. volume, L/ha	Applic. timing	Applic. type	DAT	Residue, mg/kg			
(Reference)						Fluensulfone	BSA	MeS	TSA
cGAP - USA	3.84		7 days pre- plantin g	drip irrig., band applic., broadcast spray	–				
Clermont, Florida, USA 2010 [Celebrity] (09-01858)	4.01	2083	3 days pre- plantin g	drip irrigation	73	< 0.01, < 0.01 (< 0.01)	0.044, 0.044 (0.044)	n.a.	0.042, 0.037 (0.040)
					77	< 0.01, < 0.01 (< 0.01)	0.021, 0.014 (0.018)	n.a.	0.014, 0.010 (0.012)
					80	< 0.01, < 0.01 (< 0.01)	0.017, 0.014 (0.016)	n.a.	0.016, 0.012 (0.014)
					84	< 0.01, < 0.01 (< 0.01)	0.012, 0.011 (0.012)	n.a.	0.010, 0.010 (0.010)
					87	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	< 0.01, < 0.01 (< 0.01)
					115	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	0.016, 0.018 (0.017)
Fresno, California, USA 2009 [H 8004 Processing] (09-01858)	4.00	2088	3 days pre- plantin g	drip irrigation	118	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	0.024, 0.030 (0.027)
					122	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	0.023, 0.023 (0.023)
					125	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	0.026, 0.025 (0.026)
					129	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	n.a.	0.022, 0.029 (0.026)
Madera, California, USA 2009 [H 8004 Processing] (09-01858)	3.99	2086	3 days pre- plantin g	drip irrigation	122	< 0.01, < 0.01 (< 0.01)	0.016, 0.018 (0.017)	n.a.	0.029, 0.023 (0.026)
					125	< 0.01, < 0.01 (< 0.01)	0.012, 0.016 (0.014)	n.a.	0.036, 0.043 (0.040)

Location Year [Variety]	Applic. rate, kg ai/ha	Applic. volume, L/ha	Applic. timing	Applic. type	DAT	Residue, mg/kg			
						Fluensulfone	BSA	MeS	TSA
(Reference)									
					129	< 0.01, < 0.01 (< 0.01)	0.010, 0.012 (0.011)	n.a.	0.034, 0.024 (0.029)
					132	< 0.01, < 0.01 (< 0.01)	0.018, 0.014 (0.016)	n.a.	0.034, 0.025 (0.030)
					136	< 0.01, < 0.01 (< 0.01)	0.019, 0.014 (0.017)	n.a.	0.028, 0.020 (0.024)
Corning, California, USA 2010 [AB-3] (AA100707)	4.02	233	7 d pre- plantin g	BCS	114	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
	8.02	232	7 d pre- plantin g	BCS	114	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
Porterville, California, USA 2010 [Rio Grande] (AA100707)	3.92	296	7 d pre- plantin g	BCS	112	< 0.01, < 0.01 (< 0.01)	0.059, 0.114 (0.087)	< 0.01, < 0.01 (< 0.01)	0.036, 0.053 (0.045)
Porterville, California, USA 2010 [Champion] (AA100707)	3.92	344	7 d pre- plantin g	BCS	146	< 0.01, < 0.01 (< 0.01)	0.014, 0.020 (0.017)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
	4.00	9000	7 d pre- plantin g	drip irrigation	146	< 0.01, < 0.01 (< 0.01)	0.024, 0.033 (0.029)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
Livingston, California, USA 2010 [Champion] (AA100707)	3.99	357	7 d pre- plantin g	BCS	126	< 0.01, < 0.01 (< 0.01)	0.035, 0.033 (0.034)	< 0.01, < 0.01 (< 0.01)	0.026, 0.028 (0.027)
King City, California, USA 2010 [Rio Grande] (AA100707)	4.04	395	7 d pre- plantin g	BCS	150	< 0.01, < 0.01 (< 0.01)	0.024, 0.022 (0.023)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
Visalia, California, USA 2010 [Champion] (AA100707)	3.94	357	7 d pre- plantin g	BCS	123	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
Chico, California, USA 2010 [AB-3] (AA100707)	4.02	232	7 d pre- plantin g	BCS	143	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01 (< 0.01)
Arroyo Grande, California, USA 2010	3.64	345	7 d pre- plantin g	BCS	113	< 0.01, < 0.01 (< 0.01)	0.015, 0.010 (0.013)	< 0.01, < 0.01 (< 0.01)	0.018, 0.013 (0.016)

Location Year [Variety]	Applic. rate, kg ai/ha	Applic. volume, L/ha	Applic. timing	Applic. type	DAT	Residue, mg/kg			
(Reference)						Fluensulfone	BSA	MeS	TSA
[Shady Lady] (AA100707)									
Hobe Sound, Florida, USA 2010 [Florida 47] (AA100707)	3.97	228	7 d pre- plantin g	BCS	102	< 0.01, < 0.01 (< 0.01)	0.271, 0.267 (0.27)	< 0.01, < 0.01 (< 0.01)	0.081, 0.100 (0.091)
Dover, Florida, USA 2010 [Tigris] (AA100707)	4.04	215	7 d pre- plantin g	BCS	78	< 0.01, < 0.01 (< 0.01)	0.275 , 0.270 (0.27)	< 0.01, < 0.01 (< 0.01)	0.086, 0.075 (0.081)
Seven Springs, North Carolina, USA 2010 [Homestead] (AA100707)	4.11	359	7 d pre- plantin g	BCS	94	< 0.01, < 0.01 (< 0.01)	0.027, 0.024 (0.026)	< 0.01, < 0.01 (< 0.01)	0.202, 0.141 (0.17)
North Rose, New York, USA 2010 [Mountain Spring] (AA100707)	4.02	329	7 d pre- plantin g	BCS	93	< 0.01, < 0.01 (< 0.01)	0.058, 0.090 (0.074)	< 0.01, < 0.01 (< 0.01)	0.080, 0.115 (0.098)
Thorndale, Ontario, Canada 2010 [Mariana] (AA100707)	3.81	381	7 d pre- plantin g	BCS	101	< 0.01, < 0.01 (< 0.01)	0.085, 0.102 (0.094)	< 0.01, < 0.01 (< 0.01)	0.222, 0.234 (0.23)
Thorndale, Ontario, Canada 2010 [Heinz 3478] (AA100707)	3.75	375	7 d pre- plantin g	BCS	88	< 0.01, < 0.01 (< 0.01)	0.185, 0.211 (0.20)	< 0.01, < 0.01 (< 0.01)	0.281, 0.247 (0.26)
Portage la Prairie, Manitoba, Canada 2010 [Fantastic] (AA100707)	4.07	356	7 d pre- plantin g	BCS	91	< 0.01, < 0.01 (< 0.01)	0.081, 0.095 (0.088)	< 0.01, < 0.01 (< 0.01)	0.067, 0.088 (0.078)
Elm Creek, Manitoba, Canada 2010 [Fantastic] (AA100707)	4.12	360	7 d pre- plantin g	BCS	79	< 0.01, < 0.01 (< 0.01)	0.260, 0.201 (0.23)	< 0.01, < 0.01 (< 0.01)	0.374, 0.290 (0.33)
Branchton, Ontario, Canada 2010 [TSH 18] (AA100707)	3.98	249	7 d pre- plantin g	BCS	83	< 0.01, < 0.01 (< 0.01)	0.076, 0.067 (0.072)	< 0.01, < 0.01 (< 0.01)	0.128, 0.113 (0.12)
Branchton, Ontario, Canada 2010 [TSH 28] (AA100707)	3.86	241	7 d pre- plantin g	BCS	85	< 0.01, < 0.01 (< 0.01)	0.077, 0.055 (0.066)	< 0.01, < 0.01 (< 0.01)	0.082, 0.067 (0.075)

Location Year [Variety]	Applic. rate, kg ai/ha	Applic. volume, L/ha	Applic. timing	Applic. type	DAT	Residue, mg/kg			
(Reference)						Fluensulfone	BSA	MeS	TSA
	4.00	9000	7 d pre-planting	drip irrigation	85	< 0.01, < 0.01 (< 0.01)	0.069, 0.087 (0.078)	< 0.01, < 0.01 (< 0.01)	0.085, 0.106 (0.096)
Branchton, Ontario, Canada 2010 [TSH 25] (AA100707)	4.01	251	7 d pre-planting	BCS	83	< 0.01, < 0.01 (< 0.01)	0.180, 0.165 (0.17)	< 0.01, < 0.01 (< 0.01)	0.161, 0.174 (0.17)
Elm Creek, Manitoba, Canada 2010 [Bush Beefsteak] (AA100707)	3.92	296	7 d pre-planting	BCS	85	< 0.01, < 0.01 (< 0.01)	0.044, 0.039 (0.042)	< 0.01, < 0.01 (< 0.01)	0.060, 0.050 (0.055)

Applic. Type BCS = broadcast spray

Residue n.a. = not analysed

FATE OF RESIDUES IN STORAGE AND PROCESSING

Nature of the residue during processing

High-temperature hydrolysis

High-temperature hydrolysis of fluensulfone, BSA, MeS, and TSA was investigated by G. Morlock (2011, R-28467). In the study, fluensulfone, BSA, MeS, and TSA were spiked into buffered solutions, in duplicate, at a target concentration of 1 mg/L. The spiked solutions were put into conditions, in the dark, simulating pasteurisation (90 °C, pH 4, 20 min.); baking, brewing, boiling (100 °C, pH 5, 60 min); and sterilisation (120 °C, pH 6, 20 min.). Prior to and after processing, an aliquot from each sample was collected and preserved with acetonitrile. Samples were stored for not more than 17 days, refrigerated (ca. 4 °C) prior to analysis. Analysis consisted of diluting an aliquot of sample with water (fluensulfone, MeS) or acetonitrile/water (BSA, TSA) followed by HPLC-MS/MS determination. Procedural recoveries from samples spiked at 0.1 or 1.0 mg/L ranged from 88 to 102% with standard deviations of not more than 11% across all four compounds and at both spiking levels.

Table 78 Results of high-temperature hydrolysis studies with fluensulfone, BSA, MeS, and TSA (G. Morlock, 2011, R-28467)

	pH		Mass, g		
Analyte	Start	End	Start	End	Recovery
50 mM Citric Acid Buffer, 90 °C, 20 min.					
Fluensulfone	4.03	4.01	224.9	224.9	100.0
	4.03	4.00	225.7	225.7	100.0
TSA	4.02	4.00	224.5	224.4	100.0
	4.02	3.99	223.3	223.1	99.9

MeS	4.02	4.00	224.9	223.9	99.5
	4.02	4.00	225.9	225.9	100.0
BSA	4.02	4.00	226.2	226.2	100.0
	4.02	4.00	225.9	225.8	100.0
50 mM Acetic Acid Buffer, 100 °C, 60 min.					
Fluensulfone	5.04	5.04	221.6	221.5	99.9
	5.04	5.04	225.0	224.8	99.9
TSA	5.05	5.04	224.5	224.2	99.99
	5.04	5.03	224.3	224.2	100.0
MeS	5.05	5.04	224.8	224.8	100.0
	5.05	5.04	223.6	223.6	100.0
BSA	5.05	5.04	223.8	223.6	99.9
	5.05	5.03	226.2	225.9	99.9
50 mM Citric Acid Buffer, 120 °C, 20 min.					
Fluensulfone	6.02	6.01	226.4	226.1	99.9
	6.01	6.01	224.8	224.4	99.8
TSA	6.01	6.01	222.5	222.3	99.9
	6.01	6.01	224.9	224.6	99.9
MeS	6.02	6.01	226.4	226.0	99.8
	6.01	6.01	226.4	226.0	99.8
BSA	6.02	6.02	225.8	225.5	99.9
	6.02	6.01	225.1	225.2	100.0

Residues after processing

The fate of fluensulfone and its BSA, MeS, and TSA metabolites during processing of raw tomato into processed tomato products was investigated by Jones (2011, Report AA100707) by Burn and Winner (2012, Report FOZ1001), and by Jones (2013, Report R-29577). In all studies, samples of processed commodities were generated using procedures reflective of commercial practices and all samples were analysed by methods equivalent to Method 1977W or Method 2061W.

In Jones 2011, tomatoes were transplanted into plots that had received treatment with fluensulfone seven days prior to transplanting at a rate of 7.93 or 8.02 kg ai/ha. Tomatoes were harvested at maturity from the second trial only (8.02 kg ai/ha) and processed into juice, puree, and paste using simulated commercial practices. RAC and processed commodities were assayed for fluensulfone, BSA, MeS, and TSA. All residues were < 0.01 mg/kg in all samples except for parent fluensulfone at 0.01 mg/kg in tomato juice. Given the results and that other studies are available, further information on processing practices and derivation of processing factors are not addressed in this evaluation.

In Burn and Winner, tomatoes were transplanted in to plots which had received fluensulfone seven days prior to transplanting at a rate of either 3.93 or 7.86 kg ai/ha. Tomatoes were harvested at maturity and processed into peeled, canned, sundried, juice, puree, paste, wet pomace, and dry pomace processed commodities using simulated commercial practices. Raw tomato and processed tomato samples were analysed by a method equivalent to Method 2061W.

Peeled tomatoes were made steaming tomatoes, with stems removed, in a steamer pot for six minutes. After steaming, the skins were removed manually and the tomato cores were removed with a standard kitchen coring tool.

Canned tomatoes were made by placing portions of the peeled tomatoes into containers, topping the containers off with juice from the whole tomato, fitting lids to the containers, and boiling in a water-filled pot for 45 minutes. The canned tomatoes were then cooled rapidly to prevent overcooking.

Juice, puree, paste, and wet pomace were made as follows: Whole tomato samples were chopped in a food processor at low speed. The resulting homogenate was strained using a press and a gauze bag to remove coarse components (skins, seeds, etc.). The material in the bag made up the wet pomace fraction and the strained liquid made up the juice fraction. An aliquot of juice was concentrated, by rotary evaporation in vacuo at 90 °C, to a moisture content of 86% to form puree, and a separate aliquot of juice was concentrated in the same fashion to a moisture content of 72% to form paste. Dry pomace was made by drying wet pomace in an oven to a moisture content of < 10%.

For sundried tomatoes, whole tomato samples were quartered lengthwise and composited into samples containing at least 1 quarter from 12 different tomatoes. The wedges were laid out onto a tray and lightly covered with salt (NaCl, ca. 10–15 g per kg fresh sample). The samples were dried in the sun over a 5-day period. Samples were checked daily for water content and were considered to be suitably dried when they had dehydrated to < 20% of their original weight.

Residues of fluensulfone were < 0.01 mg/kg in all samples. Quantifiable residues of BSA, MeS, and TSA occurred in RAC tomatoes from at least one of the treatment rates. Residues of BSA and TSA were concentrated, relative to the residues in the RAC, in wet and dry pomace. In addition, BSA showed concentration in sundried tomato. MeS was concentrated in dry pomace from the lower treatment rate only.

Table 79 Residues of fluensulfone, BSA, MeS, and TSA in tomato and processed commodities (Burn and Winner, 2012, Report FOZ1001)

Tomato Commodity	Residue, mg/kg		Processing Factor	
	3.93 kg ai/ha rate	7.86 kg ai/ha rate	3.93 kg ai/ha rate	7.86 kg ai/ha rate
Fluensulfone				
Raw fruit	< 0.01	< 0.01	–	–
Canned	< 0.01	< 0.01	–	–
Dry pomace	< 0.01	< 0.01	–	–
Peeled	< 0.01	< 0.01	–	–
Sundried	< 0.01	< 0.01	–	–
Juice	< 0.01	< 0.01	–	–
Paste	< 0.01	< 0.01	–	–
Puree	< 0.01	< 0.01	–	–
Wet pomace	< 0.01	< 0.01	–	–
BSA				
Raw fruit	0.01	0.03	–	–
Canned	< 0.01	0.01	< 1	0.33
Dry pomace	0.17	0.28	17	9.3
Peeled	< 0.01	0.01	< 1	0.33
Sundried	0.02	0.05	2	1.67
Juice	< 0.01	0.02	< 1	0.67
Paste	0.01	0.03	1	1
Puree	< 0.01	0.02	< 1	0.67
Wet pomace	0.04	0.09	4	3

	Residue, mg/kg		Processing Factor	
	3.93 kg ai/ha rate	7.86 kg ai/ha rate	3.93 kg ai/ha rate	7.86 kg ai/ha rate
MeS				
Raw fruit	0.02	< 0.01	–	–
Canned	< 0.01	< 0.01	< 0.5	–
Dry pomace	0.04	0.05	2	–
Peeled	< 0.01	< 0.01	< 0.5	–
Sundried	< 0.01	0.01	< 0.5	–
Juice	< 0.01	< 0.01	< 0.5	–
Paste	< 0.01	< 0.01	< 0.5	–
Puree	< 0.01	< 0.01	< 0.5	–
Wet pomace	< 0.01	0.02	< 0.5	–
TSA				
Raw fruit	0.01	0.02	–	–
Canned	< 0.01	< 0.01	< 1	< 0.5
Dry pomace	0.06	0.09	6.0	4.5
Peeled	< 0.01	0.01	< 1	0.5
Sundried	0.01	0.02	1	1
Juice	< 0.01	< 0.01	< 1	< 0.5
Paste	0.01	0.01	1	0.5
Puree	< 0.01	< 0.01	< 1	< 0.5
Wet pomace	0.02	0.03	2	1.5

In a second study by Jones (2013, R-29577), tomatoes were planted into plots treated at a rate of either 12 kg ai/ha or 20 kg ai/ha seven days prior to transplanting. Tomatoes were harvested from one plot at the higher treatment rate and processed into juice, puree, paste, and wet and dry pomace using simulated commercial practices. Residues of fluensulfone, BSA, MeS, and TSA were assayed using Method 2061W.

Prior to processing, tomatoes were washed (52–57 °C, 3–5 min), hand culled, and suitable fruits were trimmed of defects and/or off-colour areas. Cleaned tomatoes were chopped to a fine consistency and processed through a pulper/finisher with a 2.4 mm screen. Material passing through the screen underwent a hot-break process and was then passed through a pulper/finisher fitted with a 0.84 mm screen. An aliquot of the material passing through the screen was heated (85–91 °C, 3 min), placed into cans, pressure cooked (121–124 °C, 40–45 seconds), and cooled in a water bath (16–27 °C, 28–32 min). The resulting juice fraction was then placed into frozen storage. Separate aliquots of the juice fraction were vacuum-evaporated to form puree (8–24% solids) and paste (24–30% solids). Puree and paste samples were heated, canned, and cooled, as above for juice (no pressure cooking). Material not passing through the screens was collected as wet pomace, of which an aliquot was dried (54–71 °C, 20–24 hrs) to form dry pomace.

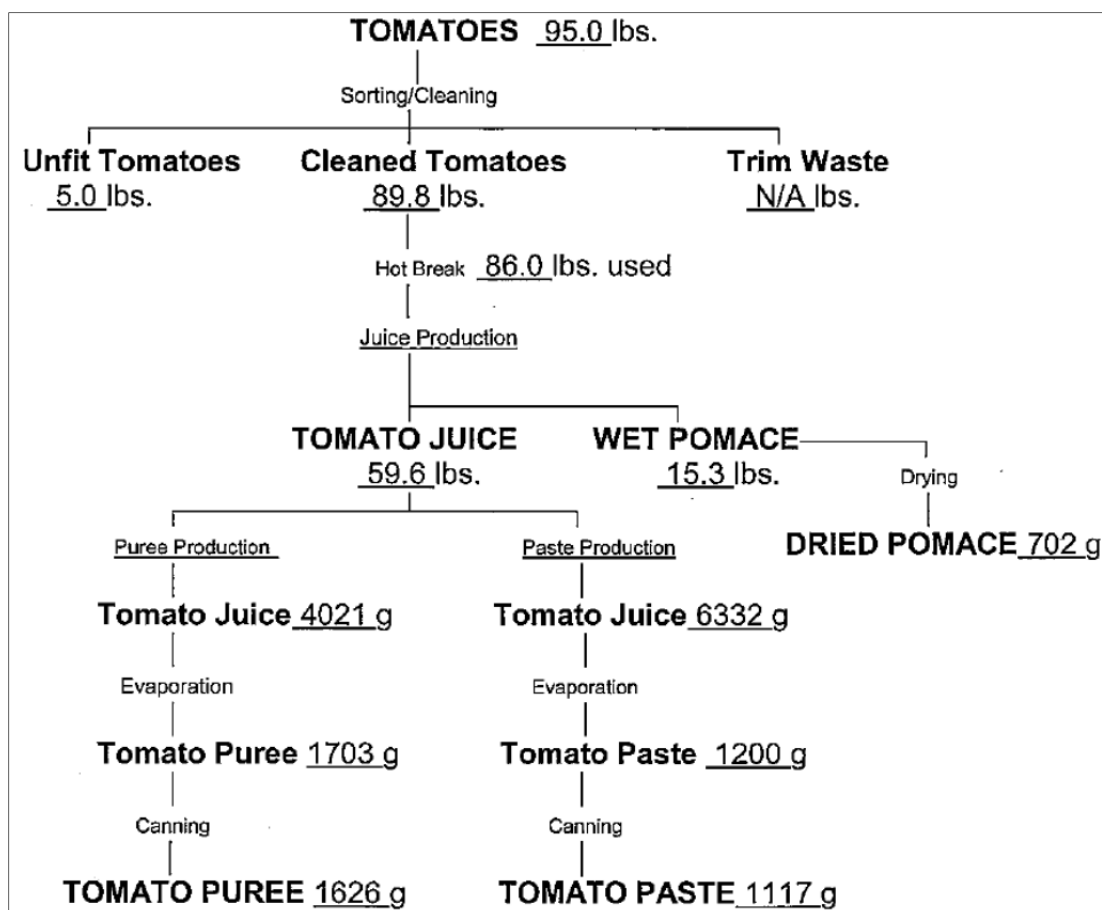


Figure 6 Material balance and processed commodity flowchart for processed tomato commodities (copied without alteration from Jones 2013, Report R-29577)

Residues of fluensulfone and MeS were < 0.01 ppm in all commodities. Residues of BSA and TSA were quantifiable in all commodities and showed concentration, relative to the raw fruit, in dry pomace and paste, and a slight concentration in BSA in puree.

Table 80 Residues of fluensulfone, BSA, MeS, and TSA in tomato and processed commodities (Jones, 2013, Report R-29577)

Tomato Commodity	Average residue, mg/kg	Processing factor
Fluensulfone		
Raw fruit	< 0.01	—
Dry pomace	< 0.01	—
Juice	< 0.01	—
Paste	< 0.01	—
Puree	< 0.01	—
Wet pomace	< 0.01	—
BSA		
Raw fruit	2.17	—
Dry pomace	14.26	6.57
Juice	1.81	0.83
Paste	7.69	3.54
Puree	3.00	1.38
Wet pomace	1.43	0.66
MeS		

Tomato Commodity	Average residue, mg/kg	Processing factor
Raw fruit	< 0.01	–
Dry pomace	< 0.01	–
Juice	< 0.01	–
Paste	< 0.01	–
Puree	< 0.01	–
Wet pomace	< 0.01	–
TSA		
Raw fruit	0.50	–
Dry pomace	1.73	3.46
Juice	0.46	0.92
Paste	1.41	2.82
Puree	0.49	0.98
Wet pomace	0.36	0.72

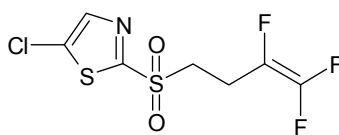
Residues in animal commodities

No feeding studies depicting transfer of residues from animal feeds to animal commodities were provided.

APPRAISAL

Fluensulfone is a non-fumigant nematicide in the fluoroalkenyl class of pesticides. Fluensulfone shows activity in multiple nematicide physiological systems. It was considered for the first time by the 2013 JMPR for toxicology and by the 2014 JMPR for residues. The 2013 JMPR established an ADI of 0–0.01 mg/kg bw and an ARfD of 0.3 mg/kg bw.

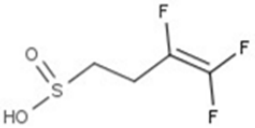
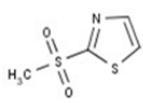
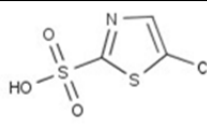
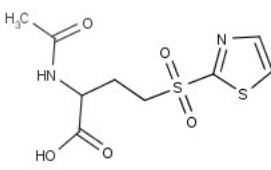
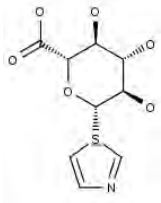
The IUPAC name for fluensulfone is 5-chloro-1,3-thiazol-2-yl 3,4,4-trifluorobut-3-en-1-yl sulfone.



Fluensulfone with ^{14}C radiolabelling in the thiazole ring or in the ethane bridge between the sulfone and trifluorobutene moieties was used in the metabolism and environmental fate studies. In this appraisal, these positions are referred to as the Th and Bu labels, respectively.

The following abbreviations, along with IUPAC names and structures, are used for the metabolites discussed in this appraisal:

BSA	3,4,4-trifluorobut-3-ene-1-sulfonic acid	<chem>OS(=O)(=O)CC/C=C(F)F</chem>

Butene sulfinic acid	3,4,4-trifluorobut-3-ene-1-sulfinic acid	
MeS	2-methylsulfonyl-1,3-thiazole	
TSA	5-chloro-1,3-thiazole-2-sulfonic acid	
Thiazole mercapturate	2-acetamido-4-(1,3-thiazole-2-sulfonyl)butanoic acid	
Thiazole glucuronides (α and β isomers)	Name not specified	

Animal metabolism

The Meeting received studies elucidating the metabolism of fluensulfone in laboratory animals (evaluated by the 2013 Meeting), lactating goats, and laying hens.

In rats, absorption of fluensulfone administered by gavage at 5 mg/kg bw is rapid, with maximal plasma concentrations achieved within 4 hours. At 5 and 500 mg/kg bw, the extent of oral absorption is high (> 80%). Fluensulfone is widely distributed in the body. High concentrations of both butene- and thiazole-labelled material were found in the liver and kidney. The labelled material was rapidly excreted via urine (> 70%), with faecal excretion accounting for no more than 5–13%. Absorbed fluensulfone was extensively metabolized, with almost no unmetabolized parent compound detected. Other than low amounts of thiazole sulfonic acid, no other faecal metabolites were present at levels above 5% of the administered dose. The parent compound probably reacts with glutathione and cleaves, giving rise to thiazole mercapturate, thiazole glucuronide, and butene sulfinic acid, the major urinary metabolites.

In goats dosed for five consecutive days at approximately 28.8 mg/animal/day (10.5 ppm in the diet), most of the recovered radioactivity was in urine and GI tract/faeces, with only 10.7% (Th-14C) or 3.5% (Bu-14C) of the applied dose (AD) accounted for in tissues and body fluids. In excreta, the major identified residues were trifluorobutene sulfinic acid and the MeS metabolite. In other matrices, the highest levels of radioactivity were associated with liver (max. 2.6 mg eq./kg, 1.7% AD), kidney (max 1.4 mg eq./kg, 0.2% AD), and milk fat (2.0 mg eq./kg, 0.31% AD). Seventy-five to

ninety percent of the radioactivity in the goat matrices was extracted with a combination of solvent extraction and alkaline digestion. No BSA, TSA, or fluensulfone was detected in any goat matrix. Radioactivity in milk and tissues was primarily associated with glucose (0.039–0.24 mg/kg; 4–17% TRR), lactose (0.036–0.21 mg/kg; 4–63% TRR), proteins/amino acids (0.015–1.2 mg/kg; 5–37% TRR), and triglycerides/fatty acids (0.009–1.1 mg/kg; 7.6–52% TRR), and the radioactivity appears to be due to incorporation of the radiolabelled carbon.

In hens dosed for seven consecutive days at 1.25 mg/animal/day (9.8 ppm in the diet), total radioactive residues (TRR) in excreta accounted for approximately 80% of the dosed material for both label positions. Total radioactivity in eggs did not plateau within the eight dosing days of the hen study. Radioactive residues in eggs were not identified. Most residues in eggs were associated with aqueous phases in the extraction schemes; however, 0.18 mg eq./kg from the butane label (27% TRR) was extracted with hexane:acetone. Fluensulfone was a major residue (0.009–0.041 mg/kg; 21–55% TRR) in poultry fat; otherwise, parent fluensulfone was not observed in any matrix. Comparison of extraction of radioactivity from liver samples treated with and without protease enzyme indicates that ca. 0.16 mg/kg (24%) of the radioactivity was associated with proteins and/or amino acids and approximately 3% (0.016 mg/kg) was identified as TSA. Incorporation of the radioactivity into triglycerides was noted in both fat matrices and in eggs. In eggs, triglyceride accounted for 7% and 27% of the TRR for the thiazole and butene labels, respectively. In fat matrices, triglycerides accounted for 7–12% and 79–87% of the TRR for the thiazole and butene labels, respectively.

Overall, the animal metabolism studies show that fluensulfone is well absorbed and that the majority (75–90%) of the dosed radioactivity is excreted. Results from rat, goat, and hen studies indicate that fluensulfone is cleaved at the sulfonyl bridge in all three animals; however, the identification of different residues in the studies suggests that there may be different metabolic pathways. In both poultry and goats, fluensulfone can be expected to break down and be incorporated almost entirely into natural products. Based on the residue profile in poultry and the observed incorporation of radioactivity into natural components, the Meeting is of the opinion that the lack of a residue plateau in egg is not of concern.

Plant metabolism

The Meeting received studies depicting the metabolism of fluensulfone in tomato, lettuce, and potato. All of the studies were conducted with fluensulfone which was radiolabelled, separately, in the thiazole ring and the ethane bridge between the sulfonyl and trifluorobutene moieties.

To investigate the metabolism of fluensulfone on tomato, fluensulfone was applied at a rate of 4.07 kg ai/ha to soil. Later that same day, tomato seedlings were planted. Mature fruits were harvested 87 days after treatment. Total radioactive residues in tomato were higher in samples treated with Bu-¹⁴C-labelled material (0.52 mg eq./kg) than those from Th-¹⁴C treatment (0.27 mg eq./kg). The majority (88.7% Th-¹⁴C; 91.3% Bu-¹⁴C) of the radioactivity was extracted with acetonitrile:water

(ACN:H₂O). TSA made up 0.12 mg/kg (45.4% TRR), with an additional 0.06 mg eq./kg (21.2% TRR) as salts/related compounds. BSA occurred at 0.22 mg/kg (41.6% TRR) with salts/related compounds making up 0.13 mg eq./kg (26.5% TRR). No other compounds, including parent fluensulfone, were identified in tomato.

Lettuce seeds were planted into soil and then fluensulfone was applied at a rate of 4.07 kg ai/ha. Samples were collected 49 days and 64 days after application to obtain immature and mature lettuce, respectively. Contrary to the results with tomato, TRR were higher from treatment with Th-¹⁴C-labelled material (6.1–7.1 mg eq./kg) than with Bu-¹⁴C-labelled material (1.5–2.4 mg eq./kg) and were similar in immature and mature foliage. The majority of the radioactivity was extracted with ACN:H₂O, with higher extraction efficiency from samples treated with the Th-¹⁴C label. Following treatment with Th-¹⁴C-labelled material, 3.57 mg/kg and 4.34 mg/kg (67.5% and 70.6% TRR) was identified as TSA in immature and mature leaves, respectively. An additional 1.39 mg eq./kg and 0.41 mg eq./kg (17.8% and 6.6% TRR) in immature and mature leaves, respectively, was determined to be salts and/or other forms of TSA. Following treatment with Bu-¹⁴C-labelled material, BSA occurred at 0.49 mg/kg (23.8% TRR) in immature leaves and at 0.49 mg/kg (37.6% TRR) in mature leaves. As with TSA, salts and other forms occurred for BSA and constituted, in total, 0.75 mg eq./kg (36.0% TRR) in immature foliage and 0.29 mg eq./kg (22.1% TRR) in mature foliage. Fluensulfone occurred at trace levels (0.009 mg/kg, 0.008 mg/kg) in immature lettuce from the Th-¹⁴C and Bu-¹⁴C treatments, respectively. Aside from BSA and TSA, no other metabolites of fluensulfone were identified.

Potato seed pieces were planted just prior to application of fluensulfone to soil at a rate of 4.04 kg ai/ha (Th-¹⁴C) or 4.13 kg ai/ha (Bu-¹⁴C). Immature (70 days after treatment) and mature (106 days after treatment) tubers were harvested and analysed. For immature and mature tubers, respectively, TRR were 0.32 and 0.44 mg eq./kg from the Th-¹⁴C treatment and 0.22 and 0.17 mg eq./kg from the Bu-¹⁴C treatment. Extraction with ACN:H₂O efficiently released residues: 91.9% TRR (Th-¹⁴C, immature tuber), 91.7% TRR (Th-¹⁴C, mature tuber), 76.9% TRR (Bu-¹⁴C, immature tuber), and 79.1% TRR (Bu-¹⁴C, mature tuber). Fluensulfone was found at trace levels (0.005 mg/kg) from both label treatments in mature tubers only. Otherwise, the only identified residues were BSA, TSA, and their salts and/or related compounds. BSA constituted 0.069 mg/kg (30.7% TRR) and 0.042 mg/kg (25.8% TRR) in immature and mature tubers, respectively. Salts and related forms of BSA provided an additional 0.041 mg eq./kg (17.8% TRR; immature tubers) and 0.035 mg eq./kg (21.4% TRR; mature tubers). TSA occurred at 0.21 mg/kg (63.0% TRR) and 0.31 mg/kg (65.3% TRR) in immature and mature tubers, respectively. Salts and related forms of TSA gave an additional 0.028 mg eq./kg (8.4% TRR; immature tubers) and 0.025 mg eq./kg (5.3% TRR; mature tubers).

Fluensulfone was extensively metabolised in all of the studies, with the only major residues being the BSA and TSA metabolites. A few chromatographic fractions contained radioactivity in

excess of 10% TRR. Investigation of these fractions indicated that the residues were associated with the BSA or TSA metabolites, as salts of the sulfonic acids or as related forms of the metabolites. The only major residues in the harvested matrices were the BSA and TSA metabolites and, with the exception of trace levels of fluensulfone in immature lettuce and mature potato, no parent compound was detected.

Environmental fate in soil

Fluensulfone is stable to hydrolysis under accelerated conditions (50 °C, pH 4, 7, 9) but is prone to photolysis [DT₅₀ of 21 days (Th-¹⁴C) or 35 days (Bu-¹⁴C) in soil], showing first-order kinetics. In an aerobic soil metabolism study, major residues following treatment with fluensulfone were BSA, TSA, and MeS, depending on the duration of incubation. Fluensulfone had DT₅₀ estimates ranging from 7 to 17 days across six soils, all following first-order kinetics. BSA formed from fluensulfone generally accumulated for the first ca. 1 month of incubation followed by dissipation (DT₅₀ 18–26 days). Residues of TSA accumulated continuously over the incubation period, reaching maxima of 49–74% of the applied radioactivity at the 120-day sampling. Residues of MeS began to be observed after the first 2–4 weeks of incubation, reaching a maximum of not more than 8% of the applied radioactivity; residues of MeS declined to 0% of the applied radioactivity between the 50- and 120-day sampling times, depending on the soil. In a separate study, the DT₅₀ estimates for the TSA and MeS metabolites under aerobic soil conditions are 421 and 33 days, respectively. Field dissipation studies were not provided.

Confined rotational crop studies were conducted with radish, lettuce, and wheat at plant-back intervals (PBIs) of 30, 120, and 360 or 390 days. Fluensulfone, radiolabelled as either the Th-¹⁴C or Bu-¹⁴C, was applied to soil at a rate of approximately 4 kg ai/ha. Lettuce was replanted at 390 days after application due to crop failure at the 360-day PBI. Following treatment with Th-¹⁴C-labeled material, TRR generally declined sharply from 30 to 120 days and then remained relatively consistent between the 120 and the 360/390-day PBIs. (e.g., wheat hay: 27 mg eq./kg at 30-Day PBI, 9.4 mg eq./kg at 120-Day PBI, 10.8 mg eq./kg at 360-Day PBI) As with primary crops, the major residues were the BSA and TSA metabolites. A low level of the parent compound was observed in lettuce, radish root, radish foliage, and wheat forage, hay, and straw (but not grain). Fluensulfone, when found, was typically 1 to 2 orders of magnitude less than the BSA or TSA residue levels. In all cases, residues of fluensulfone and BSA were not quantifiable after the 120-day PBI whereas residues of TSA persisted at quantifiable levels for at least one year, ranging from 0.13 mg eq./kg (immature lettuce) to 11 mg eq./kg (wheat hay).

Overall, fluensulfone can be expected to dissipate rather rapidly in the environment, with a concomitant increase in residues of BSA, TSA, and, to a much lesser extent, MeS. BSA residues should then decline; however, TSA appears to be stable for an extended period. The Meeting concluded from the soil metabolism and confined rotational crop studies that TSA may accumulate in

soils following repeated uses of fluensulfone and may occur in follow-on crops at plant-back intervals exceeding one year after treatment.

Methods of residue analysis

The Meeting received analytical methods for the analysis of fluensulfone, BSA, MeS, and TSA in plant and animal matrices. The methods are essentially identical for all samples and the LOQ for all matrices and analytes, defined as the lower limit of method validation, is 0.01 mg/kg.

Extraction of residues is accomplished with ACN:H₂O (1:1, v/v) or ACN (BSA and TSA in eggs only); the extract is then split for analysis of fluensulfone and MeS by one set of procedures and for analysis of BSA and TSA by a second set. For fluensulfone and MeS, there is no clean-up of the extract beyond filtration (except hexane partitioning for analysis of MeS in fatty/oily samples). Residues of fluensulfone and MeS are determined by reverse-phase LC-MS/MS in positive ion spray mode. For BSA and TSA, an aliquot of the initial extract is concentrated and then cleaned-up using C-18 SPE. Residues are determined by reverse-phase LC-MS/MS in negative ion spray mode.

The solvent used for extraction is the same as, or very similar to, that used in the metabolism studies and showed adequate extraction efficiency of incurred residues.

Testing of fluensulfone and the two sulfonic acid metabolites, BSA and TSA, through the FDA PAM multiresidue method protocols demonstrated that the compounds showed poor sensitivity, poor recovery, and/or poor chromatography. Overall, the results indicate that the FDA PAM multiresidue protocols are not suitable for the detection or enforcement of fluensulfone, BSA, or TSA residues in non-fatty foods.

Stability of residues in stored analytical samples

The Meeting received data depicting the stability of residues of fluensulfone, BSA, and TSA in tomato, pepper, cucumber, and melon. In addition, the stability of those analytes and MeS was investigated in frozen, stored tomato puree and paste. No dissipation of any analyte was observed during the storage periods for the various matrices. Stability was demonstrated in tomato raw agricultural commodity (RAC) for at least 469 days (ca. 15 months) and in tomato processed commodities for at least 181 days (ca. 6 months). For pepper, cucumber, and melon, residues were stable for at least 488 days (ca. 16 months).

Definition of the residue

Studies depicting the nature of the residues in animals consistently show fluensulfone to be cleaved at the sulfonyl moiety, presumably via glutathione conjugation, resulting in both halves of the molecule having a sulfonyl functional group. With the exception of poultry fat, fluensulfone was not observed in any animal commodity. In livestock, the majority of the radiolabel was excreted. Retained fluensulfone is extensively metabolized, with the radioactivity being associated primarily with sugars,

amino acids, and fatty acids. MeS and butene sulfinic acid were identified in livestock studies, but were observed only in excreta. In the rats, significant residues were thiazole mercapturate, thiazole glucuronide, BSA, TSA, and butene sulfinic acid. MeS, observed in some field trial samples, was not identified in the rat metabolism study.

Based on the livestock metabolism studies, a residue definition potentially suitable for enforcement by the typical criteria is possible only for poultry fat and poultry liver, which were the only matrices in the animal metabolism studies with quantifiable residues of a fluensulfone-specific compound (fluensulfone in fat and TSA in liver). Although fluensulfone was a major residue in poultry fat (up to 55% TRR), the available residue data indicate that quantifiable residues of the parent compound are not expected in plants; thus exposure to fluensulfone via livestock diets is unlikely, making the parent compound an unsuitable marker for enforcement in any livestock commodity. The other potential marker, TSA, occurred only as a minor component in poultry liver (2.7% TRR). Based on the results of the metabolism studies and on the residue profiles observed in crop metabolism studies, confined rotational crop studies, and supervised residue trials, the Meeting determined that a residue definition for livestock commodities is not necessary.

In both plant and rotational crop metabolism studies, fluensulfone appears to follow the same glutathione-mediated pathway observed in livestock; however, in plants quantifiable residues of the BSA and TSA metabolites were consistently observed. Parent fluensulfone was identified only at trace levels in immature lettuce, mature potato, and rotational lettuce, radish foliage, and wheat hay, forage and straw at short PBIs (30 days). In target crops, BSA ranged from 0.071–1.24 mg/kg (43.6–68.1% TRR) and TSA ranged from 0.17–4.75 mg/kg (66.6–85.3% TRR). In rotational crops, BSA was detected in all matrices except wheat grain at the 30-day PBI (0.004–1.4 mg/kg) and in most matrices at the 120-day PBI (0.001–0.43 mg/kg), and was undetected (< 0.001 mg/kg) by the 360/390-day PBI, except wheat straw at 0.012 mg/kg. In contrast, TSA was detected in all rotational crop matrices at all PBIs, ranging from 0.086 mg/kg to 16 mg/kg across all samples.

In crop field trials, fluensulfone was detected in only one sample (summer squash at 0.017 mg/kg). Across all crops, residues of BSA ranged from < 0.01 to 0.27 mg/kg and TSA ranged from < 0.01 to 0.71 mg/kg. MeS ranged from < 0.01 to 0.08 mg/kg and was less than both BSA and TSA in the corresponding sample. In all trials with only pre-plant applications (per GAP), MeS was < 0.01 mg/kg in all samples of cantaloupe, pepper, and tomato. Although MeS was not found in the plant or rotational crop metabolism studies, it was observed in supervised residue trials in cucumber (< 0.01–0.079 mg/kg) and summer squash (< 0.01–0.050 mg/kg).

The Meeting determined that fluensulfone is NOT a suitable marker for compliance with MRLs in crops. Both BSA and TSA are suitable markers based on results of supervised field trials. The confined rotational crop study, however, demonstrates a potential for TSA to carry over into succeeding crops. Therefore, given that quantifiable residues of fluensulfone are not expected in plant commodities, that a separate analysis is required for the analysis of fluensulfone and BSA/TSA, and

that residues of TSA may occur from previous crop cycle treatments with fluensulfone, the Meeting determined that BSA is the most suitable marker for MRL compliance. A validated method exists for analysis of BSA in plant commodities. The Meeting defined the BSA metabolite as the residue definition for compliance in plants.

Regarding the toxicity of the BSA, TSA, and MeS metabolites, the JMPR has concluded that TSA is unlikely to be of any toxicological relevance; data are insufficient at this time to make a definitive toxicological determination regarding the relevancy of BSA and MeS.

For BSA, the JMPR has determined that the ADI and ARfD for fluensulfone could be used as a screening evaluation of exposure to BSA. Based on a comparison of toxicity data between BSA and fluensulfone, the evaluation may be made directly, without a correction for molecular weight. If additional uses are considered in the future, the use of the fluensulfone points of departure to evaluate exposure to BSA may need to be re-evaluated.

For MeS, the JMPR has determined that the IEDI (0.07 µg/kg bw/day) for MeS should be compared to the Cramer class III TTC value of 1.5 µg/kg bw/day and that the IESTI (3.2 µg/kg bw/day)² should be compared to the single-exposure TTC for Cramer class III compounds of 5 µg/kg bw proposed by EFSA. The IESTI is somewhat refined in that for melon, the specific HR (0.01 mg/kg) from melon field trials was used rather than the HR for the fruiting vegetables, Cucurbits group (0.053 mg/kg). On the basis of these comparisons, the Meeting concluded that MeS is not considered to be a relevant metabolite for the crops under consideration. If additional uses are considered in the future, this conclusion may need to be re-evaluated.

Given the residue profile in crops and taking into consideration the available information on the toxicities of the metabolites, the Meeting determined that the residue definition for dietary exposure from crops is BSA. In lieu of BSA-specific toxicological points of departure, dietary intake estimates for BSA should be compared to the ADI and ARfD for fluensulfone, with no correction for molecular weight.

Definition of the residue for compliance with the MRLs and dietary intake for plant commodities: *BSA {3,4,4-trifluorobut-3-ene-1-sulfonic acid}*.

Definition of the residue for compliance with the MRLs and for dietary intake for animal commodities: *Not necessary*

Results of supervised residue trials on crops

Fluensulfone is registered in the USA for use on cucurbit vegetables and on fruiting vegetables. For all crops, the cGAP is an application to the soil at 2.8 kg ai/ha made seven days prior to transplanting

² The estimate of 3.2 µg/kg bw is refined, using the observed HR for melon (0.01 mg/kg) rather than the HR for the fruiting vegetables, Cucurbits group (0.053 mg/kg), which resulted in a maximum dietary intake estimate of 5.3 µg/kg bw.

crops into the field. Application may be made by broadcast spray to the soil, by banded spray, or by drip irrigation. The applied material must be mechanically incorporated 15–20 cm into the soil profile for spray applications or by sufficient volume for drip irrigation application.

The Meeting received supervised residue trial data for cucumber, summer squash, cantaloupe, pepper, and tomato. The trials were conducted in North America (USA and Canada). All trials were conducted at a target application rate of 3.9 kg ai/ha, which reflects a nominal exaggeration of 39% relative to the cGAP. Therefore, the Meeting decided to scale residue values for all analytes from trials otherwise meeting the cGAP to an application rate of 2.8 kg ai/ha. Residues scaled to < 0.01 mg/kg were maintained at < 0.01 mg/kg. Reported values are field trial averages unless otherwise noted.

Residues of fluensulfone were < 0.01 mg/kg in all samples.

Fruiting vegetables, Cucurbits

In cucumber, mean field trial residues of BSA (unscaled) from independent field trials treated 7 days prior to transplant (n = 7) were: < 0.01, 0.01 (2), 0.063, 0.07, 0.17, and 0.219 mg/kg.

Application rates for these trials ranged from 3.72 kg ai/ha to 4.11 kg ai/ha. Scaled to an application rate of 2.8 kg ai/ha, the residues of BSA are: < 0.01 (3), 0.041, 0.045, 0.114, and 0.137 mg/kg.

In summer squash, mean field trial residues of BSA (unscaled) from independent field trials treated 7 days prior to transplant (n = 8) were: < 0.01, 0.05, 0.06, 0.082, 0.186, 0.196, 0.214, and 0.247 mg/kg.

Application rates for these trials ranged from 3.80 kg ai/ha to 4.14 kg ai/ha. Scaled to an application rate of 2.8 kg ai/ha, the residues of BSA are: < 0.01, 0.032, 0.038, 0.051, 0.115, 0.12, 0.133, and 0.149 mg/kg.

In melon, mean field trial residues of BSA (unscaled) from independent field trials treated 7 days prior to transplant (n = 8) were: < 0.01 (3), 0.021, 0.025, 0.032, 0.049, and 0.064 mg/kg.

Application rates for these trials ranged from 3.85 kg ai/ha to 4.11 kg ai/ha. Scaled to an application rate of 2.8 kg ai/ha, the residues of BSA are: < 0.01 (3), 0.013, 0.016, 0.019, 0.030, and 0.040 mg/kg.

Noting that the GAP in the USA is for the cucurbit vegetables crop group, which is equivalent to the Codex fruiting vegetables, Cucurbit group, and that the BSA residue data from cucumbers, summer squash, and melons are not significantly different by the Kruskal-Wallis test, the Meeting determined that the residues from the trials are similar and is estimating a group maximum residue level for fruiting vegetables, Cucurbits based on the following scaled BSA residue data set (n = 23): < 0.01 (7), 0.013, 0.016, 0.019, 0.030, 0.032, 0.038, 0.040, 0.041, 0.045, 0.051, 0.114, 0.115, 0.120, 0.133, 0.137, and 0.149 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg for BSA on fruiting vegetables, Cucurbits; the HR is 0.16 mg/kg (from a single sample) and the STMR is 0.032 mg/kg.

Fruiting vegetables, other than Cucurbits

In chilli pepper, mean field trial residues of BSA (unscaled) from independent field trials treated 7 days prior to transplant (n = 3) were: 0.040, 0.041, and 0.184 mg/kg.

Application rates for these trials ranged from 3.98 kg ai/ha to 4.10 kg ai/ha. Scaled to an application rate of 2.8 kg ai/ha, the residues of BSA are: 0.025, 0.025, 0.116 mg/kg.

In sweet pepper, mean field trial residues of BSA (unscaled) from independent field trials treated 7 days prior to transplant (n = 8) were: 0.048, 0.055, 0.063, 0.070, 0.073, 0.082, 0.136, and 0.232 mg/kg.

Application rates for these trials ranged from 3.84 kg ai/ha to 410 kg ai/ha. Scaled to an application rate of 2.8 kg ai/ha, the residues of BSA are: 0.030, 0.034, 0.041, 0.045 (2), 0.050, 0.083, and 0.146 mg/kg.

In tomato, mean field trial residues of BSA (unscaled) from independent field trials treated 7 days prior to transplant (n = 20) were: < 0.01 (3), 0.013, 0.023, 0.026, 0.029, 0.034, 0.042, 0.072, 0.074, 0.078, 0.087, 0.088, 0.094, 0.173, 0.198, 0.231, 0.269, and 0.273 mg/kg.

Application rates for these trials ranged from 3.64 kg ai/ha to 4.12 kg ai/ha. Scaled to an application rate of 2.8 kg ai/ha, the residues of BSA are: < 0.01 (4), 0.014, 0.016, 0.018, 0.021, 0.026, 0.045, 0.046, 0.049, 0.054, 0.055, 0.061, 0.108, 0.132, 0.140, 0.168, and 0.169 mg/kg;

Noting that the GAP in the USA is for the fruiting vegetables crop group, which is equivalent to the Codex group fruiting vegetables, other than Cucurbits except sweet corn and mushroom, and that the residue data from sweet pepper, chilli pepper, and tomato are not significantly different by the Kruskal-Wallis test, the Meeting determined that the residues from the trials are similar and is estimating a group maximum residue level for fruiting vegetables, other than Cucurbits except sweet corn and mushroom based on the following scaled BSA residue data set: (n = 31): < 0.01 (4), 0.014, 0.016, 0.018, 0.021, 0.025, 0.025, 0.026, 0.030, 0.034, 0.041, 0.045 (3), 0.046, 0.049, 0.050, 0.054, 0.055, 0.061, 0.083, 0.108, 0.116, 0.132, 0.140, 0.146, 0.168, and 0.169 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg for BSA on fruiting vegetables, other than Cucurbits except sweet corn and mushroom; the HR is 0.17 mg/kg (from a single sample) and the STMR is 0.045 mg/kg.

Based on the maximum residue level of fruiting vegetables, other than Cucurbits except sweet corn and mushroom (0.3 mg/kg) and a dehydration factor of 7, the Meeting estimated a maximum residue level of 2 mg/kg for BSA in chilli pepper (dry), an HR of 1.2, and an STMR of 0.32.

Fate of residues during processing

High-temperature hydrolysis

The Meeting received a study investigating the high-temperature hydrolysis of fluensulfone, BSA, MeS, and TSA. Samples of aqueous buffered solutions were spiked with fluensulfone, BSA, MeS, or TSA at ca. 1 mg/L and put under conditions simulating pasteurisation (90 °C, pH 4, 20 min.); baking, brewing, boiling (100 °C, pH 5, 60 min); and sterilisation (120 °C, pH 6, 20 min.). Solutions were analysed by HPLC-MS/MS prior to and after processing. All four analytes were shown to be stable under all three conditions, with overall recoveries ranging from 87 to 118% of the initial concentration.

Residues after processing

The Meeting received data depicting the concentration/dilution of residues during processing of tomato into canned, juice, puree, paste, wet and dry pomace, peeled, and sun-dried processed commodities. Processed commodities were derived using simulated commercial practices. Of the three studies that were submitted, two were suitable for deriving processing factors (in one study, all residues were < 0.01 mg/kg). In those two studies, residues of fluensulfone were < 0.01 mg/kg in all samples and processing factors for the parent compound could not be derived.

Crop	Processed commodity	BSA processing factors	Best processing factor estimate (average)	STMR-P, mg/kg	HR-P, mg/kg
Tomato	RAC	--	--	STMR = 0.045	HR = 0.17
	Canned	0.33	0.33	0.015	0.056
	Dry pomace	6.6, 9.3, 17	11	0.50	1.9
	Peeled	0.33	0.33	0.015	0.056
	Sundried	1.67, 2	1.8	0.081	0.31
	Juice	0.67, 0.83	0.75	0.034	0.13
	Paste	1, 1, 3.54	1.8	0.081	0.31
	Puree	0.67, 1.38	1.0	0.045	0.17
	Wet pomace	0.66, 3, 4	2.6	0.12	0.44

Based on the maximum residue estimate for fruiting vegetables, other than Cucurbits except sweet corn and mushroom (0.3 mg/kg) and the processing factor of 1.8 for both dried tomato and tomato paste, the Meeting recommends a maximum residue level of 0.5 mg/kg for BSA in dried tomato and 0.5 mg/kg for BSA in tomato paste.

Residues in animal commodities

The Meeting has determined that residue definitions for compliance and dietary intake are not necessary for animal commodities and that residues in animal commodities are not expected.

RECOMMENDATIONS

Definition of the residue for compliance with the MRLs and dietary intake for plant commodities: *3,4,4-trifluorobut-3-ene-1-sulfonic acid (BSA)*. Note that for dietary intake, exposure estimates should be compared to the ADI and ARfD for fluensulfone, with no correction for molecular weight.

Definition of the residue for compliance with the MRLs and for dietary intake for animal commodities: *Not necessary*.

Commodity		Recommended MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
CCN	Name	New	Previous		
VC 0045	Fruiting vegetables, Cucurbits	0.3	–	0.032	0.16
VO 0050	Fruiting vegetables, other than Cucurbits except sweet corn and mushroom	0.3	–	0.045	0.17
HS 0444	Peppers chilli, dried	2.1	–	0.32	1.2
VW 0448	Tomato paste	0.5	–	0.081	0.31
DV 0448	Tomato, dried	0.5	–	0.081	0.31

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of BSA were calculated for the 17 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the current Meeting. The ADI for fluensulfone is 0–0.01 mg/kg bw. The calculated IEDIs for BSA were 0–3% of the maximum fluensulfone ADI. The Meeting concluded that the long-term intakes of residues of BSA, when fluensulfone is used in ways that have been considered by the JMPR, are unlikely to present a public health concern.

Short-term intake

The International Estimated Short-Term Intakes (IESTI) of BSA were calculated for food commodities and their processed commodities using HRs/HR-Ps or STMRs/STMR-Ps estimated by the current Meeting. The ARfD for fluensulfone is 0.3 mg/kg bw. The calculated maximum IESTI for BSA was 7% of the fluensulfone ARfD for all commodities. The Meeting concluded that the short-term intake of residues of BSA, when fluensulfone is used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

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Code	Author	Year	Title, Institute, Report reference
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AA100707 R-23487	Jones, GL	2011	Magnitude of the Residue of Fluensulfone in Fruiting Vegetables and Processed Commodities American Agricultural Services, Inc., 404 E. Chatham Street, Cary NC 27511. Makhteshim Chemical Works Report number AA100707, R-23487 GLP, Not published
AA100708 R-23488	Jones, GL	2011	Magnitude of the Residue of Fluensulfone In Cucurbit Vegetables. American Agricultural Services, Inc., 404 E. Chatham Street, Cary NC 27511. Makhteshim Chemical Works Report number AA100708, R-23488 GLP, Not published
09-01858 R-23481	Korpalski, S and Riley, ME	2011	Magnitude of the residue of MCW-2 on fruiting vegetables (2009 trials). Eurofins Agrosience Services, Inc. 150 Industrial Park Drive Forsyth, GA 31029. Makhteshim Chemical Works Eurofin Agrosience Services Report 09-01858. R-23481, GLP, Not published
09-01859 R-23482	Korpalski, S and Riley, ME	2011	Magnitude of the residue of MCW-2 on cucurbit vegetables (2009 trials). Eurofins Agrosience Services, Inc. 150 Industrial Park Drive Forsyth, GA 31029. Makhteshim Chemical Works Eurofin Agrosience Services Report 09-01859. R-23482, GLP, Not published

Code	Author	Year	Title, Institute, Report reference
R-25458	La Mar, J and Quistad, GB	2010	The Metabolism of [^{14}C] MCW-2 (2 Radiolabels) in the Lactating Goat PTRL West, Inc. 625-B Alfred Nobel Drive, Hercules, California 94547, USA Makhteshim Chemical Works PTRL West Project No. 1792W, R-25458 GLP, Not published
R-25454	La Mar, J and Quistad, GB	2010	A Metabolism Study with [^{14}C]MCW-2 (2 Radiolabels) in Laying Hens PTRL West, Inc. 625-B Alfred Nobel Drive, Hercules, California 94547, USA Makhteshim Chemical Works PTRL West Project No. 1793W, R-25454 GLP, Not published
R-23339	Marin, J	2010	Method Validation of an Analytical Method for the Determination of Fluensulfone and its Metabolites in Soil. PTRL West, Inc., 625-B Alfred Nobel Drive, Hercules, California 94547 Makhteshim Chemical Works Ltd PTRL West Project 2049W, R-23339 GLP, Not published.
R-23334	Marin, JE	2010	Method validation of an analytical method for the determination of MCW-2 and its metabolites in plant matrices PTRL West, inc., 625-B Alfred Nobel Drive, Hercules, California 94547 Makhteshim Chemical Works Ltd PTRL West Project 1977W, Report 2, R-23334 GLP, Not published.
R-27436	Moser F	2011	Fluensulfone: Aerobic Degradation in Soils Smithers Viscient AG Seestrasse 21, Postfach CH-9326 Horn, Switzerland Makhteshim Chemical Works, Smithers Viscient AG Study 1026.014.760, R-27436, GLP, unpublished
R-23320	Ponte, M	2012	Photodegradation of [^{14}C]MCW-2 in/on Soil by Artificial Light PTRL West, Inc., 625-B Alfred Nobel Drive, Hercules, CA 94547. Makhteshim Chemical Works, Ltd. PTRL West Report Number 1856W 1, R-23320 GLP, unpublished
R-25456	Quistad, GB and Bautisata, A	2011	A Metabolism Study with [^{14}C]Fluensulfone (MCW-2) (2 Radiolabels) using Tomatoes PTRL West, Inc. 625-B Alfred Nobel Drive, Hercules, California 94547, USA Makhteshim Chemical Works PTRL West Project No. 1787W, R-25456 GLP, Not published
R-25459	Quistad, GB and Bautisata, A	2011	A metabolism study with [^{14}C] MCW-2 (2 radiolabels) using potatoes PTRL West, Inc. 625-B Alfred Nobel Drive, Hercules, California 94547, USA Makhteshim Chemical Works PTRL West Project No. 1785W, R-25459 GLP, Not published
R-25457	Quistad, GB, Bautista, AV, Kovatchev, A and Bronner, K	2011	A Confined Rotational Crop Study with [^{14}C]MCW-2 (2 Radiolabels) using Radish, Lettuce, and Wheat. REPORT BEING FINALISED PTRL West, Inc., 625-B Alfred Nobel Drive Hercules, California 94547, USA Makhteshim Chemical Works PTRL West Project No. 1788W, R-25457 GLP, Not published
R-25455	Quistad, GB, La Mar, J and Bautisata, A	2010	A Metabolism Study with [^{14}C]MCW-2 (2 Radiolabels) using Lettuce PTRL West, Inc. 625-B Alfred Nobel Drive, Hercules, California 94547, USA Makhteshim Chemical Works PTRL West Project No. 1786W, R-25455 GLP, Not published
R-23322	Schick, M	2011	Photodegradation of [^{14}C]MCW-2 in Sterilized pH 7 Buffer by Artificial Sunlight. PTRL West, Inc. 625-B Alfred Nobel Drive Hercules, CA 94547. Makhteshim Chemical Works, Ltd. Report No.: Report no. 1843W-001, R-23322 GLP, Not published
R-23319	Shepler, K	2010	Hydrolysis of [^{14}C]MCW-2 (Fluensulfone) at pH 4, 7 and 9 PTRL West, Inc. 625-B Alfred Nobel Drive Hercules, CA 94547. Makhteshim Chemical Works, Ltd. PTRL West Report No. 1843-001, R-23319 GLP, Not published
R-23309	Weissenfield, M	2008	MCW-2: Determination of the melting point / melting range. Harlan Laboratories Ltd, Zelgliweg 1, 4452 Itingen, Switzerland. Makhteshim Chemical Works Ltd, Harlan Laboratories Study C00347, R-23309 GLP, Not published

Code	Author	Year	Title, Institute, Report reference
R-23310	Weissenfield, M	2008	MCW-2: Determination of the boiling point / boiling range. Harlan Laboratories Ltd, Zelgliweg 1, 4452 Itingen, Switzerland. Makhteshim Chemical Works Ltd, Harlan Laboratories Study C00358, R-23310 GLP, Not published
R-23312	Weissenfield, M	2008	MCW-2: Determination of the relative density. Harlan Laboratories Ltd, Zelgliweg 1, 4452 Itingen, Switzerland. Makhteshim Chemical Works Ltd, Harlan Laboratories Study C00360, R-23312 GLP, Not published
R-23313	Weissenfield, M	2008	MCW-2: Determination of the vapour pressure. Harlan Laboratories Ltd, Zelgliweg 1, 4452 Itingen, Switzerland. Makhteshim Chemical Works Ltd, Harlan Laboratories Study C00382, R-23313 GLP, Not published
R-23316	Weissenfield, M	2008	MCW-2: Determination of water solubility. Harlan Laboratories Ltd, Zelgliweg 1, 4452 Itingen, Switzerland. Makhteshim Chemical Works Ltd, Harlan Laboratories Study C00415, R-23316 GLP, Not published
R-23321	Weissenfield, M	2008	MCW-2: Calculation of dissociation constant. Harlan Laboratories Ltd, Zelgliweg 1, 4452 Itingen, Switzerland. Makhteshim Chemical Works Ltd, Harlan Laboratories Study C00527, R-23321 Not GLP, Not published
R-23310A	Weissenfield, M	2009	MCW-2: Determination of the melting point / melting range and the boiling point / boiling range. Harlan Laboratories Ltd, Zelgliweg 1, 4452 Itingen, Switzerland. Makhteshim Chemical Works Ltd, Harlan Laboratories Study C30773, R-23310A GLP, Not published
R-23313A	Weissenfield, M	2009	MCW-2: Henry's Law constant—Expert statement. Harlan Laboratories Ltd, Zelgliweg 1, 4452 Itingen, Switzerland. Makhteshim Chemical Works Ltd, Harlan Laboratories Study C00516, R-23313A Not GLP, Not published
R-23317	Weissenfield, M	2009	MCW-2: Determination of the solubility in organic solvents. Harlan Laboratories Ltd, Zelgliweg 1, 4452 Itingen, Switzerland. Makhteshim Chemical Works Ltd, Harlan Laboratories Study C00437, R-23317 GLP, Not published
R-23330	Weissenfield, M	2010	MCW-2 Technical: Determination of the Storage Stability (Shelf Life). Harlan Laboratories Ltd, Zelgliweg 1, 4452 Itingen, Switzerland. Makhteshim Chemical Works Ltd, Harlan Laboratories Study C66198, R-23330 (was R-23307) GLP, Not published
R-23489	Witte, A	2011	Validation of an analytical method for the determination of residues of MCW-2 and three metabolites in plant commodities (acidic, dry and fatty). CIP Chemisches Institut Pforzheim GmbH Makhteshim Chemical Works Ltd CIP Study Code: 10M03036-01-VMPL, R-23489 GLP, Not published.
R-28495	Witte, A	2011	Validation of an Analytical Method for the Determination of Residues of MCW-2 metabolite #3626 (methylsulfone) in water containing plant commodities CIP Chemisches Institut Pforzheim GmbH Makhteshim Chemical Works Ltd CIP Study Code: 10M04006-01-VMPL, R-28495 GLP, Not published.
R-28512	Witte, A	2011	Validation of an Analytical Method for the Determination of Residues of MCW-2 and three Metabolites in Food Stuff of Animal Origin CIP Chemisches Institut Pforzheim GmbH Schulberg 17, D-75175 Pforzheim, Germany Makhteshim Chemical Works Ltd Report 11M03036-01-VMAT, R-28512 GLP, Not published

FLUFENOXURON (275)

The first draft was prepared by Ms. Monique Thomas. Pest Management Regulatory Agency, Canada

EXPLANATION

Flufenoxuron is a benzoylurea insect growth regulator with high levels of acaricidal activity. Flufenoxuron kills pest mites and insects through interference with chitin production during cuticle development in mite and insect juvenile stages. Flufenoxuron has limited to no effect on adult mites and insects. It is registered for use in a variety of crops worldwide.

IDENTITY

ISO common name: Flufenoxuron

Chemical name

IUPAC: N-{4-[2-chloro-4-(trifluoromethyl)phenoxy]-2-fluorophenyl}-N'-(2,6-difluorobenzoyl)urea

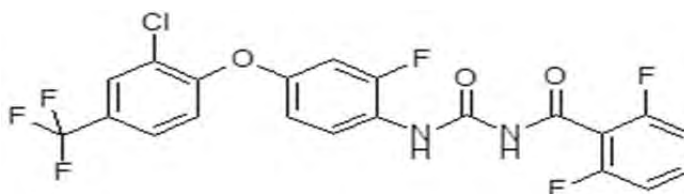
CAS: N-[[[4-[2-chloro-4-(trifluoromethyl)phenoxy]-2-fluorophenyl]amino]carbonyl]-2,6-difluorobenzamide

CAS Registry No: 101463-69-8

CIPAC No.: 470

Synonyms and Trade Name: WL115110, Cascade

Structural formula:



Molecular formula: $C_{21}H_{11}ClF_6N_2O_3$

Molecular mass: 488.8 g/mol

Physical and Chemical Properties

Pure active ingredient, minimum purity 99.0%

Chemical/physical property	Results	Reference	Guidelines
Vapour Pressure (20 °C)	6.52×10^{-9} mPa	FX-390-025 (2000/7000541)	EU Directive 91/414
Melting point	167–172 °C	FX-303-002	
Partition coefficient (25 °C)	pH 4 : log P = 3.99 pH 7 : log P = 4.00 pH 9 : log P = 4.00	FX-301-002 (1988/7000758)	
Relative density (~22 °C)	1.649	Kaestel 2001a, 2001/1019524	OECD Test Guideline 109
Henry's law constant (25 °C)	7.46×10^{-6} Pa.m ³ .mol ⁻¹	FX-390-025 (2000/7000541)	EU Directive 91/414
Physical state, colour, odour	white, crystalline powder with a sourish odour.	Kaestel 2001b, 2001/1009097	EU Directive 91/414
Solubility in water [µg/L] (25 °C)	pH 4 : 1.86 pH 7 : 1.36 pH 9 : 3.69	Langner 1988a, FX-301-002 (1988/7000758)	

Chemical/physical property	Results	Reference	Guidelines
	Flufenoxuron is almost insoluble in water.		
Solubility in organic solvents [g/L] (20 °C ^a)	n-heptane: < 0.01 n-octanol: 1.1 toluene: 3.5 dichloromethane: 16 methanol: 3.5 acetone: 83 ethyl acetate 55 acetonitrile: 6.62	Daum 2001a, FX-301-002 (2001/1017469)	OECD 105
Hydrolysis rate	Hydrolysis has been investigated at 50 °C at pH 5, 7 and 9. At pH 5 and 7: flufenoxuron is hydrolytically stable (half-life > 1 year). At pH 9 : > 87% of flufenoxuron was degraded after 5 days at 50 °C. Degradation was investigated at 60 °C and 70 °C, and half-life extrapolated at 25 °C was found to be 76 days.	Langner 1988a, FX-301-002 (1988/7000758)	
Photochemical degradation	Flufenoxuron decomposes under photolysis into 2,6-difluorobenzamide (maximum 84.5% at DAT 7), with a DT ₅₀ of 4.5 days under continuous irradiation at pH 7 and 22 °C. No counterpart metabolites were observed. Dark samples showed no degradation.	Hassink 2003a, 2003/1000986	
Quantum yield	$\Phi = 1.75 \times 10^{-3}$ for the radiolabelled active ingredient However the DT ₅₀ of the fluoroaniline radiolabelled active substance was poorly determined. For $290 < \lambda < 775$ nm : $\Phi = 2.2 \cdot 10^{-3}$ mol. einstein ⁻¹	Hassink 2003a, 2003/1000986	
Dissociation constant	pKa = 10.1	Camilleri & Langner 1986a, FX-311-002 (1986/7000994)	

^a Purity 99.2%

Technical material; minimum purity 97.4%

Chemical/physical property	Results	Reference	Guidelines
Appearance	white, fine powder with a spicy odour.	Kaestel 2001c, 2001/1009099	
Relative density (20 °C ^a)	1.57	Langer 1988a, FX-301-002 (1988/7000758)	

Chemical/physical property	Results	Reference	Guidelines
Flammability	Not flammable ^b	Van Helvoirt 1990a, FX-330-001 (1990/7001093)	EEC-Directive A-10
Auto-flammability	Not self-igniting ^b	Van Helvoirt 1990b, FX-330-002 (1990/7001094)	EEC-Directive A-16
Explosive properties	Not explosive ^b	Van Helvoirt & Cardinaals 1990a, FX-334-001 (1990/7001095)	EEC-Directive A-14
Oxidising properties	Not oxidising ^b	Van Helvoirt 1990c, FX-356-001 (1990/7001096)	EEC-Directive A-17

^a Purity 97.4%^b Purity 97.6%

Formulation

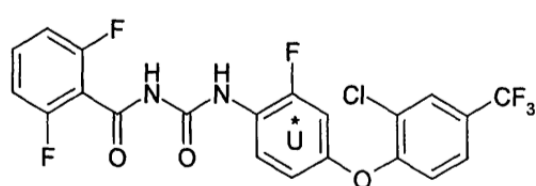
Flufenoxuron is commercially marketed as an emulsifiable concentrate containing 10% flufenoxuron.

Specification

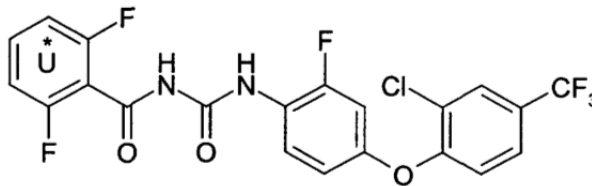
Flufenoxuron has not been evaluated by the Joint Meeting of Pesticide Specifications.

METABOLISM AND ENVIRONMENTAL FATE

The metabolism and distribution of flufenoxuron in plants and animals was investigated using ¹⁴C-labelled test materials as shown below:



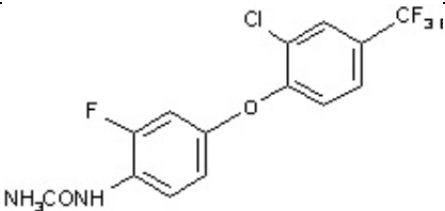
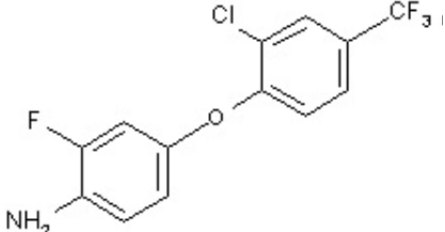
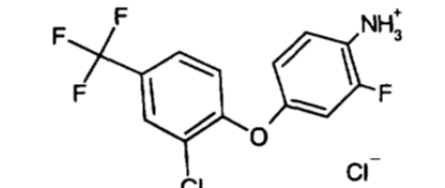
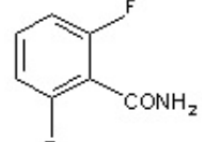
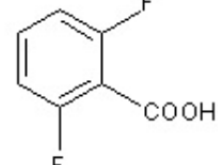
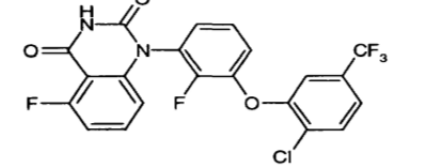
Fluoroaniline-U-¹⁴C



Difluoroamide-U-¹⁴C

Common chemical names, code names and structures of the parent and metabolites are captured below:

Code	Structure	Occurrence
Flufenoxuron WL115110 <i>N</i> -{4-[2-chloro-4-(trifluoromethyl)phenoxy]-2-fluorophenyl}- <i>N'</i> -(2,6-difluorobenzoyl)urea		Rat Lactating goat Laying hen Grape Apple Tomato Chinese cabbage Soil Hydrolysis study

Code	Structure	Occurrence
Reg. No. 4064702 <i>N</i> -{4-[2-chloro-4-(trifluoromethyl)phenoxy]-2-fluorophenyl}urea		Rat Laying hen Hydrolysis study
Reg. No. 241208 4-[2-chloro-4-(trifluoromethyl)phenoxy]-2-fluoroaniline		Rat Laying hen Hydrolysis study
Reg. No. 4064703 (chloride salt of Reg. No. 241208) 4-[2-chloro-4-(trifluoromethyl)phenoxy]-2-fluoroaniline hydrochloride		Hydrolysis study
Reg. No. 102719 2,6 difluorobenzamide		Rat Hydrolysis study
Reg. No. 206925 2,6-difluorobenzoic acid		Rat Hydrolysis study
Reg. No. 4964847 1-{3-[2-chloro-5-(trifluoromethyl)phenoxy]-2-fluorophenyl}-5-fluoro-2,4(1H,3H)-quinazolinedione		Hydrolysis study

Animal metabolism

The Meeting received information on the fate of [^{14}C]flufenoxuron in a lactating goat and laying hens. The lactating goat metabolism study was carried out with [^{14}C]flufenoxuron uniformly labelled at the fluoroaniline ring while the laying hen metabolism study was carried out with uniformly ^{14}C -labelled difluoroamide- and fluoroaniline-flufenoxuron. Metabolism in laboratory animals (rat) was summarised and evaluated by the WHO panel of the 2014 JMPR.

Lactating goat

The metabolism of [^{14}C]flufenoxuron was investigated in one lactating goat, weighing 73 kg, was dosed orally by gavage with 2-fluoroaniline-[U- ^{14}C]-ring-labelled flufenoxuron once daily for 4 consecutive days at a dose level of 10 mg/day equivalent to 10 ppm in the diet. Milk production

ranged from 1.2–2.8 L/day. During the treatment period, milk was collected twice daily while urine and faeces were collected once daily. At sacrifice (within 24 hours after the last dose) samples of liver, kidney, muscle (hind leg and dorsal) and fat (deep body fat store along spine and renal) were collected.

The major route of elimination of the radioactivity was via the faeces which accounted for 18% of the total administered radioactivity (TAR); while urine accounted for 2.5% of the TAR and milk accounted for 8.3% of the TAR. Overall, the tissue burden was low, accounting for < 2% of the TAR. No further justification was provided for the low overall recovery of administered radioactivity (33% of the TAR).

The total radioactive residues (TRRs) were highest in fat (1.6 mg eq/kg), followed by liver (0.37 mg eq/kg), kidney (0.13 mg eq/kg) and muscle (0.076 to 0.1 mg eq/kg).

Table 1 Radioactive residue in organs and tissues

Organ/tissue	TRR [mg eq/kg]
Liver	0.37
Kidney	0.13
Skeletal muscle	
Hind leg	0.076
Dorsal	0.1
Deep body fat	1.6
Renal fat	1.6
Bile	0.26 mg eq/L
Whole blood	0.029 mg eq/L

Milk residues peaked on day 4 (average of 0.27 mg eq/L) with the highest concentrations of radioactivity detected in the cream fraction (accounting for 82–93% of the TRR in whole milk) and the lowest found in whey (1.3–5.7% of the TRR).

Table 2 ¹⁴C-Residues in milk of goat dosed with 10 mg/day of 2-fluoroaniline-[U-¹⁴C]-ring-labelled flufenoxuron

Day	Time (hours) ^a	TRR (mg eq/L)
1	7	0.27
2	23	0.09
	31	0.39
3	47	0.13
	55	0.23
4	71	0.16
	79	0.37
5	95	0.23

^a Time after administration of first dose

Samples of tissues and milk were further analysed for determination of the distribution and composition of the total radioactivity. Liver and kidney samples were homogenised with ethyl acetate/methanol, muscle and fat samples were homogenised with dichloromethane while milk samples were mixed with potassium oxalate, ethanol and diethyl ether, all prior to partitioning with hexane and acetonitrile.

Aliquots of the acetonitrile phases of milk, liver, kidney, muscle and fat were mixed with appropriate amounts of the reference compound and subjected to HPLC/UV analysis.

Table 3 Characterisation of ¹⁴C residues in tissues and milk of goat dosed with 2-fluoroaniline-[U-¹⁴C]-ring-labelled flufenoxuron

Sample	% TRR Initial extraction efficiency	Acetonitrile/ hexane partition		Final recovery in acetonitrile fraction [%]
		% TRR in hexane fraction	% TRR in acetonitrile fraction	
Liver	93.4	3.8	88.5	82.8

Sample	% TRR Initial extraction efficiency	Acetonitrile/ hexane partition		Final recovery in acetonitrile fraction [%]
		% TRR in hexane fraction	% TRR in acetonitrile fraction	
Kidney	91.4	16.1	89.8	82.0
Muscle	66.2	9.8	114.5	63.5
Fat	99.8	1.5	115.3	107.4
Milk	98.3	0.0	81.1	81.1

Extraction of the samples collected recovered 64–107% of the TRR. HPLC/UV analysis of the extracts showed no evidence of cleavage of the flufenoxuron molecule or any metabolism of the parent substance in the goat. Hence, the parent compound remained intact with no other substances being detected in the analytical chromatograms.

The nature of the radioactivity in fat analysed after a 3–4 month storage period at -20°C was examined by HPLC. No differences were noted in the metabolic profile.

Laying Hen

Study 1

Laying hens were dosed once daily for 14 consecutive days with flufenoxuron, uniformly labelled in the difluoroamide or fluoroaniline rings, at 13–14 ppm feed, equivalent to 0.78–0.85 mg/kg bw. Eggs were collected twice daily, in the morning before and in the afternoon after administration, whilst excreta was collected once daily. The animals were sacrificed approximately 23 h after the last dose and the liver, muscle and fat were collected and pooled per dose group. Liquid samples were measured directly by LSC while solid samples were combusted prior to analysis by LSC.

Excreta accounted for 72–78% of the TAR. Although no plateau was reached in eggs during the dosing period (14 days), the radioactivity recovered in eggs amounted to 1.0–1.3% of the TAR (0.5–0.8 mg eq/kg). Among all the tissues analysed, radioactive residues were highest in fat (5.0–5.3 mg eq/kg) followed by liver (0.6–1.1 mg eq/kg) and muscle (0.3–0.4 mg eq/kg). The total recovery of radioactivity was 82% and 77% in the difluoroamide-label and fluoroaniline-label groups, respectively.

Table 4 Recovery of radioactive residues in laying hens

Matrix	Difluoroamide label		Fluoroaniline label	
	% TAR	mg eq./kg	% TAR	mg eq./kg
Excreta	78.41		71.59	
Eggs	0.98		1.28	
Blood	0.02	0.113	0.40	1.269
Liver	0.11	0.577	0.24	1.056
GI-Tract (skin)	0.47	0.504	0.39	0.369
GI-Tract (contents)	0.06	0.252	0.06	0.213
Muscle	0.58	0.326	0.71	0.364
Adipose tissue	1.33	5.041	1.85	5.318
Subtotal organs	2.57		3.65	
Cage wash	0.13	–	0.11	–
Total	82.09	–	76.66	–

Extraction of radioactive residues from tissues and organs was initially performed with methanol, with the extractability ranging from 91–102% and 88–99% of the TRR for the difluoroamide- and fluoroaniline-labels, respectively. While the lowest extractability occurred with the liver from the fluoroaniline-label (88% of the TRR), microwave extraction of the liver post-extraction solid (PES) released another 8% of the TRR. Further characterisation according to polarity of residues was completed by partitioning with ethyl acetate/ water and acetonitrile/iso-hexane (for

fat, eggs and muscle) where the majority of the radioactivity was recovered in the ethyl acetate phase (93–101% and 82–104% of the TRR, difluoroamide- and fluoroaniline-label, respectively).

Table 5 Distribution of ^{14}C -residues in eggs and tissues

Label	Sample	TRR [mg/kg]	ERR ^a [mg/kg] (%TRR)	PES ^b [mg/kg] (%TRR)	Recovery [%] ^c
Difluoroamide	Egg	0.570	0.577 (101.3)	0.006 (1.0)	102.3
	Fat	5.041	5.088 (100.9)	0.011 (0.2)	101.1
	Liver	0.577	0.586 (101.5)	0.010 (1.7)	103.2
	Muscle	0.326	0.297 (91.3)	0.002 (0.7)	92.0
	Excreta ^d	4.082	4.065 (99.6)	0.018 (0.4)	100.0
Fluoroaniline	Egg	0.794	0.770 (97.1)	0.022 (2.8)	99.9
	Fat	5.318	5.258 (98.9)	0.038 (0.7)	99.6
	Liver	1.056	0.930 (88.1)	0.144 (13.6) ^e	101.7
	Muscle	0.364	0.341 (93.7)	0.014 (3.8)	97.5
	Excreta ^d	3.757	3.676 (97.8)	0.081 (2.2)	100.0

^a ERR = Extracted Radioactive Residue (methanol)

^b PES = Post-Extraction Solid

^c Sum of all extracts and the residue

^d Calculated value from ERR and PES

^e Another 7.9% of the TRR was extracted by microwave treatment

For the difluoroamide-label, the parent was the only analyte identified in eggs, muscle, fat and liver ranging from 0.28 mg eq/kg (86.5% of the TRR, muscle) to 4.6 mg eq/kg (91.4% of the TRR, fat).

For the fluoroaniline-label, while the parent compound and the metabolite Reg. No. 4064702 were both identified in the eggs, muscle, fat and liver, the parent accounted for the majority of the TRRs (70–91%). The lowest level of parent was found in muscle with 0.30 mg eq/kg and the highest level in fat with 4.8 mg eq/kg. In eggs and liver, Reg. No. 4064702 was present at 0.10 mg eq/kg and 0.13 mg eq/kg (12.0% and 12.6% of the TRR), respectively. Reg. No. 4064702 was detected in muscle and fat as a minor metabolite amounting to 0.02 mg eq/kg and 0.05 mg eq/kg, respectively (5.5% and 1.0% of the TRR). The formate derivative of Reg. No. 241208 was released from the PES of liver after microwave treatment in the presence of formic acid/acetonitrile. The radioactivity associated with this derivative amounted to 0.04 mg eq/kg (3.3% TRR). The Meeting could not confirm whether the metabolite Reg. No. 241208 is an actual in-vivo metabolite or an artefact formed during microwave treatment.

Table 6 Identification of total radioactive residues in eggs and tissues

Label	Structure	Eggs [mg/kg] (%TRR)	Muscle [mg/kg] (%TRR)	Fat [mg/kg] (%TRR)	Liver [mg/kg] (%TRR)
Difluoroamide	Flufenoxuron	0.514 (90.3)	0.282 (86.5)	4.610 (91.4)	0.597 (103.5)
Fluoroaniline	Flufenoxuron	0.620 (78.2)	0.302 (82.9)	4.840 (91.0)	0.738 (69.9)
	Reg. No. 4064702	0.095 (12.0)	0.020 (5.5)	0.051 (1.0)	0.133 (12.6)

Label	Structure	Eggs [mg/kg] (%TRR)	Muscle [mg/kg] (%TRR)	Fat [mg/kg] (%TRR)	Liver [mg/kg] (%TRR)
	Reg. No. 241208	—	—	—	0.035 ^a (3.3)

^a Amount of Reg. No. 241208 released from liver PES by microwave treatment

Study 2

Five groups (groups 1–5 and 7–10) of three laying hens each (White Leghorn hybrids, 1.4–2.1 kg) were dosed orally by gavage with 2-fluoroaniline-[U-¹⁴C]-ring-labelled flufenoxuron once daily for seven consecutive days at a dose level of 10 mg/kg feed (corresponding to 0.5 mg/kg bw). One group of six untreated hens (group 6) served as a background control group.

Excreta from every group of hens was combined and sampled at 24 h intervals during administration. Eggs of individual hens were collected daily before each administration. After the final administration, before sacrifice, eggs and excreta were collected. Hens were sacrificed 22 hours after the final dosage or 2, 9, 16, and 34 days after the last dose to investigate the depuration behaviour of the flufenoxuron, liver, kidney, muscle (composite of breast and thigh muscle), gizzard (without lining and contents), heart, omental fat and skin were sampled. The content of the gizzard was added to the residual carcass.

On average, 26% of the TAR was excreta-related with eggs, sampled from 0–166 h after the first administration, accounting for 5% of the TAR. At sacrifice, the highest amount of radioactive residues was detected in fat (47% of the TAR), followed by skin (12% of the TAR), muscle (4% of the TAR), liver (2% of the TAR), kidney (0.3% of the TAR), heart and gizzard. The recovery of radioactivity amounted to 96% of the TAR.

Table 7 Balance of radioactivity (means of 15 hens) after seven daily doses of 0.5 mg/kg [¹⁴C]-flufenoxuron sacrificed 22 hr after final dosage

Matrix	% TAR	mg eq./kg
Excreta	25.3	
Cage wash	0.6	
Eggs	4.7	6.0 egg yolks 0.02 egg whites
Liver	1.5	2.28
Kidney	0.3	1.26
Muscle	4.1	3.9
Fat	47.0	14.3
Skin	11.9	3.9
Heart	0.2	0.86
Gizzard		0.44
Total	95.6	

Table 8 Extractability of radioactivity from different organs of laying hens

Organ/tissue	TRR [mg/kg]	Acetone extract		Unextracted residue		% Recovery (extracted + unextracted)
		[mg eq/kg]	%TRR	[mg eq/kg]	%TRR	
Liver ^a	2.28	2.22	97.0	0.22	9.5	106
Kidney ^a	1.26	1.14	90.0	0.16	12.5	102
Muscle	0.38	0.38	102.0	0.03	8.1	110
Gizzard	0.44	0.41	94.2	0.05	11.7	106
Heart	0.86	0.83	96.5	0.09	10.2	107

Organ/tissue	TRR [mg/kg]	Acetone extract		Unextracted residue		% Recovery (extracted + unextracted)
		[mg eq/kg]	%TRR	[mg eq/kg]	%TRR	
Fat	14.3	12.94	90.5	0.03	0.2	91
Skin + adj. fat	3.9	3.58	91.9	0.02	0.6	92

^a After incubation at 37 °C.

Extraction of the samples collected recovered > 90% of the TRR. HPLC/UV analysis of the extracts showed that flufenoxuron was not extensively metabolized and accounted for the majority of the radioactivity in egg yolks, liver, kidney, muscle, gizzard and heart while it was the only compound detected in fat and skin. Hydrolysis of the benzoyl urea bond resulted in the formation of fluoroaniline-label specific minor metabolites Reg. No. 4064702 and Reg. No. 241208.

The metabolite Reg. No. 4064702 was detected in yolks, liver, kidney, muscle, gizzard, and heart at 6–22% of the TRR. The minor metabolite Reg. No. 241208 was only detected in liver and kidney ($\leq 4\%$ of the TRR). The Meeting noted that liver and kidney were the only matrices that were incubated for 16 hours at 37 °C in 0.07 M phosphate buffer at pH 7.5 prior to extraction. However, the Meeting could not confirm that this metabolite is an artefact of the analytical procedure.

Additionally, one to three unknown radioactive fractions in yolks, liver and kidney extracts were detected (all below 10% of the TRR). In liver, three unknown fractions were characterized that were less polar than the reference items. The unknown fraction from yolks and kidney had a similar TLC-behaviour.

Table 9 Identification of total radioactive residues in eggs and tissues (%TRR)

Identity	Yolks	Liver	Kidney	Muscle	Gizzard	Heart	Fat	Skin
Flufenoxuron	84.9	66.3	59.6	90.4	89.5	90.3	98.2	97.2
REG. NO. 4064702	6.3	10.4	21.6	9.3	10.2	9.3	n.d.	n.d.
REG. NO. 241208	n.d.	3.7	2.5	n.d.	n.d.	n.d.	n.d.	n.d.
Total Identified	91.2	80.4	83.7	99.7	99.7	99.6	98.2	97.2
Total characterized (number of fractions)	8.8 (1)	2.9–7.6 (3)	8.9 (1)	–	–	–	–	–
Total Unidentified	–	4.0	7.4	0.3	0.3	0.4	1.8	2.8
TOTAL	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

The depuration study indicated that the radioactivity in egg yolks and muscle decreased steadily up to day 34. In kidney and liver, the decrease in radioactivity was more prominent from day 16 to day 34 of the depuration phase yet in fat, the decrease in radioactivity occurred most rapidly from day 2 to day 9 and from day 16 to day 34. These results demonstrated that radioactive residues are not retained in eggs, organs and tissues after cessation of dosing.

Overview of metabolism in livestock

In the lactating goat metabolism study, the parent flufenoxuron remained intact and was the only residue identified in milk and tissues.

In the laying hen metabolism studies, while flufenoxuron was the predominant residue in eggs and organs/tissues, cleavage of the benzoyl urea bond was observed to a limited extent resulting in the formation of minor metabolites Reg. No. 4064702 (eggs and tissues) and Reg. No. 241208 (liver and kidney).

Plant metabolism

Metabolism studies on tomato, apple, grape and Chinese cabbage were made available to the Meeting.

Grape

Grape-vine plants, variety Muller-Thurgau, grown outdoor and protected with plastic covers after application, were separately treated with [difluorobenzamide- $U-^{14}C$]- and [fluoroaniline-ring- $U-^{14}C$]flufenoxuron formulated as flowable concentrate formulations. Two foliar sprays were made during fruit development at a rate of 0.04 kg ai/ha/application with a 40-day retreatment interval. Mature samples of stalks and fruit (from grape clusters) were collected 28–29 days after last treatment (DAT), while leaves were collected at 15 DAT (immature) and 28–29 DAT (mature).

Total radioactive residues (TRRs) in leaves from the applications using difluorobenzamide-label were 2.7 mg eq/kg at 15 DAT and declined to 1.8 mg eq/kg at 29 DAT (Table 10). TRRs in mature fruit and stalks (29 DAT) were 0.014 mg eq/kg and 0.16 mg eq/kg, respectively. TRRs in leaves from applications using the fluoroaniline-label declined from 2.3 mg eq/kg at 15 DAT 1.4 mg eq/kg at 28 DAT. TRRs in mature fruit and stalks (28 DAT) were 0.012 mg eq/kg and 0.11 mg eq/kg, respectively. Overall, the distribution of radioactivity was relatively similar among both radiolabels.

Homogenized samples of fruit, leaf and stalk samples were extracted three times with methanol (MeOH) and twice with water. After each extraction step, the liquid phase was separated from the post extraction solid (PES) by centrifugation. The extracts and the dried PES were analysed by combustion for the determination of the residual radioactive residues. Aliquots of the MeOH extracts were evaporated to dryness, reconstituted in water and partitioned three times with hexane followed by three partition steps of the remaining aqueous phase with ethyl acetate. Aliquots of the organic and aqueous phases were analysed by LSC. Residues were identified by reverse-phase HPLC-UV and the identity of parent flufenoxuron was confirmed by HPLC with a Polymeric Reversed Phase (PRP-1) column.

As reported in Tables 10 and 11, approximately 95–97% of the TRR (0.012–2.6 mg eq/kg) was extracted from the grape matrices for both radiolabels. In all fruit, leaf and stalk samples (both labels), parent flufenoxuron was the only compound identified at 50–97% of the TRR (0.007–2.2 mg eq/kg). Polar unknowns comprised up to 40–46% of the TRR in mature grape samples (0.005–0.006 mg eq/kg) for both radiolabels. Unextracted residues in all leaf, fruit and stalk samples comprised $\leq 5\%$ of the TRR (< 0.11 mg eq/kg), resulting in overall accountabilities of 99–100%.

The only metabolic reaction observed in grape matrices was the breakdown of flufenoxuron to mainly polar metabolites.

Table 10 Total radioactive residues (TRR) in grape matrices following two foliar applications of [^{14}C]-flufenoxuron at 0.08 kg ai/ha/season

Matrix	DAT (days)	TRR ^a (mg eq/kg)	Distribution of radioactive residues							
			MeOH		Water		Extracted radioactivity ^b		PES ^c	
			Mg eq/kg	% TRR	Mg eq/kg	% TRR	Mg eq/kg	% TRR	Mg eq/kg	% TRR
[difluorobenzamide-U- ¹⁴ C]flufenoxuron										
Immature Leaves	15	2.673	2.551	95.4	0.022	0.8	2.573	96.2	0.100	3.7
Mature Leaves	29	1.819	1.763	96.6	0.011	0.6	1.774	97.2	0.045	2.5
Mature Fruit	29	0.014	0.014	95.8	< 0.0005	0.8	0.014	96.6	< 0.0005	3.4
Mature Stalks	29	0.163	0.17	96.3	0.001	0.5	0.158	96.8	0.005	3.2
[fluoroaniline-ring-U- ¹⁴ C]flufenoxuron										
Immature Leaves	15	2.285	2.153	94.2	0.023	1.0	2.176	95.2	0.108	4.7
Mature Leaves	28	1.424	1.353	95.0	0.012	0.8	1.365	95.8	0.059	4.2
Mature Fruit	28	0.012	0.012	94.3	< 0.0005	0.8	0.012	95.1	0.001	4.9
Mature Stalks	28	0.106	0.100	94.5	0.001	0.8	0.101	95.3	0.005	4.7

^a TRR = sum of extracted and unextracted radioactivity (PES)

^b Methanol extract and water extract combined

^c PES = post extraction solids (solids remaining after extraction)

Table 11 Summary of identified and characterized radioactivity extracted from [difluorobenzamide- ^{14}C]-flufenoxuron -treated immature and mature leaves, grapes and stalks

Compound	Leaves (15 DAT)		Leaves (29 DAT)		Grapes (29 DAT)		Stalk (29 DAT)	
	Mg eq/kg	%TRR	Mg eq/kg	%TRR	Mg eq/kg	%TRR	Mg eq/kg	%TRR
Flufenoxuron	2.305	86.2	1.763	96.9	0.007	49.7	0.157	96.3
Characterised ^a	0.269	9.9	0.011	0.6	0.006	46.9	0.001	0.5
Total extracted ^b	2.574	96.1	1.774	97.5	0.014	96.6	0.158	96.8
Total unextracted	0.100	3.7	0.045	2.5	< 0.001	3.4	0.005	3.2
Accountability ^c	2.674	99.8	1.819	100.0	0.014	100.0	0.163	100.0

^a Total characterised consisted of polar unknowns, each accounting for <10% of the TRR and < 0.005 mg eq/kg

^b Total extracted = MeOH and Aqueous extracts

^c Accountability = (total extracted + total unextracted)/TRR

Table 12 Summary of identified and characterized radioactivity extracted from [U-Aniline- ^{14}C]-flufenoxuron treated immature and mature leaves, grapes and stalks

Compound	Leaves (15 DAT)		Leaves (29 DAT)		Grapes (29 DAT)		Stalk (29 DAT)	
	Mg eq/kg	%TRR	Mg eq/kg	%TRR	Mg eq/kg	%TRR	Mg eq/kg	%TRR
Flufenoxuron	2.153	94.2	1.353	95.0	0.007	54.6	0.100	94.5
Characterised ^a	0.023	1.0	0.012	0.8	0.005	40.5	0.001	0.8
Total extracted ^b	2.176	95.2	1.365	95.8	0.012	95.1	0.101	95.3
Total unextracted	0.108	4.7	0.059	4.2	0.001	4.9	0.005	4.7
Accountability ^c	2.284	99.9	1.424	100.0	0.013	100.0	0.106	100.0

^a Total characterised consisted of polar unknowns, each accounting for < 10% of the TRR and < 0.005 mg/kg

^b Total extractable = MeOH and Aqueous extracts

^c Accountability = (total extracted + total unextracted)/TRR

Apple

Flufenoxuron uniformly labelled in the fluoroaniline ring, formulated as a dispersible concentrate, was sprayed on 10 apple trees (var. Cox's Orange Pippin), maintained in glasshouses. The trees were treated during fruit development, when fruit was approximately 20 mm in diameter, with a single application at a rate of 0.1 kg ai/hL. Samples of immature fruit were harvested 0 days (4 h post-treatment) and 46 days after treatment (DAT), and mature fruit samples were collected at 99 DAT.

Immediately after harvest, all fruit samples (0, 46 and 99 DAT) were washed sequentially with acetonitrile (ACN) and hexane. Total radioactivity was determined in the washes and the fruit by combustion/LSC. At DAT 99 four additional apples were harvested; two of these were not washed and used for autoradiography while two other apples were washed as described above and separated into peel, pulp and seeds prior to analysis.

For the characterization/identification of residues, only the surface washes from the 0 and 99 DAT samples were prepared. Washed fruit samples were extracted three times with ACN:water (7:3, v/v) and centrifuged. The supernatants were combined and partitioned twice with dichloromethane (DCM). The DCM fraction was dried under a stream of nitrogen, and resuspended in DCM for TLC analysis. For HPLC-UV analysis, the DCM was removed under nitrogen and the residue was resuspended in ACN:water (7:3, v/v). The identity of parent flufenoxuron was confirmed by HPLC with a Polymeric Reversed Phase (PRP-1) column.

Total radioactive residues (TRRs) in immature fruit were 2.6 mg eq/kg (0 DAT) and declined to 0.16 mg eq/kg (46 DAT) and 0.06 mg eq/kg (99 DAT), likely due to the increasing size of the fruit as it matured (Table 13). The surface washes accounted for 77–96% of the TRRs with unextracted residues comprising 0.3–2.5% of the TRR (0.001–0.008 mg eq/kg), resulting in accountabilities of 93–100%. Parent flufenoxuron was the only compound identified in the surface washes (74–93% of

the TRRs; 0.043–2.4 mg eq/kg) and fruit extracts (3–16% of the TRRs; 0.01–0.08 mg eq/kg). There were no residues other than the parent that were identified in the apple samples.

For the two apples separated into peel, pulp and seeds and those subjected to autoradiography, the same trend was observed whereby the majority of the radioactivity remained on the peel with limited translocation of the TRRs into the pulp and the seeds.

Table 13 Total radioactive residues (TRR) in apples following a single foliar application of [^{14}C]-flufenoxuron at 100 mg ai/L

Matrix	DAT	TRR ^a (mg/kg)	Distribution of radioactive residues					
			Surface wash (ACN+ Hexane)		Fruit Extract		PES ^b	
			mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Immature fruit	0	2.551	2.457	96.3	0.094	3.7	0.008	0.3
Immature fruit	46	0.163	0.146	89.4	0.017	10.6	0.000	0.0
Mature fruit	99	0.055	0.043	77.0	0.013	23.0	0.001	2.5

^a TRR = sum of extracted and unextracted radioactivity (PES)

^b PES = post extracted solids (solids remaining after extraction)

Table 14 Summary of characterization and identification of ^{14}C -residues in apple fruit following a single foliar application of [U-Aniline- ^{14}C]-flufenoxuron at 100 mg ai/L

	Immature Fruit (0 DAT)		Mature Fruit (99 DAT)	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Flufenoxuron	2.462	96.5	0.054	90.9
Total extracted ^a	2.551	100.0	0.054	90.9
Total unextracted	0.008	0.3	0.001	2.5
Accountability ^b	2.559	100.3	0.055	93.4

^a Total extracted = surface wash + fruit extract

^b Accountability = (total extracted + total unextracted)/TRR

Tomato

[U-Aniline- ^{14}C]flufenoxuron mixed in approximately equal proportions with [aniline- ^{15}N] and formulated as an emulsifiable concentrate, was applied as a single broadcast foliar application to tomato plants (var. Moneymaker) maintained in an outdoor uncovered enclosure. The application was made during fruit development at a rate of 0.125 kg ai/ha and tomato fruit was harvested at 0 and 28 days after treatment (DAT).

Tomato fruit samples were surface washed with ACN:water (7:3, v/v) five times prior to extraction. The washed fruit were homogenized and extracted once with ACN:water (7:3, v/v) and filtered. The surface washes and extracts were diluted with water and partitioned three times with DCM. The organic phase was concentrated, then analysed by thin-layer chromatography (TLC). Radioactivity in surface washes and extracts were determined by LSC while the unextracted solid residues were subjected to combustion analysis.

Total radioactive residue (TRR) in/on tomato fruit declined from 0.38 mg eq/kg on day 0, to 0.17–0.2 mg eq/kg by day 28 (Table 15). The total extracted residues (surface washes and fruit extracts) from 0 DAT to 28 DAT, accounted for 94–99% of the TRR (0.16–0.38 mg eq/kg), mainly from the surface wash ($\geq 94\%$ of the TRRs). Unextracted residues comprised 1.1–5.8% of the TRR (0.004–0.012 mg eq/kg). Only sample II (28 DAT; 0.2 mg eq/kg) underwent identification/characterization of TRRs. Radio- TLC of the extracted radioactivity showed the parent flufenoxuron as the only identified residue, accounting for 91% of the TRR (0.18 mg eq/kg).

Table 15 Total radioactive residues (TRR) in tomato fruit following a single application of [U-Aniline-¹⁴C]-flufenoxuron at 0.125 kg ai/ha

Matrix	DAT	TRR ^a (mg eq/kg)	Distribution of radioactive residues					
			Surface wash (ACN+ Water)		Fruit Extract		PES ^b	
			mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Tomato Sample	0	0.38	0.374	98.0	0.003	0.9	0.004	1.1
Tomato Sample I	28	0.17	0.160	95.4	0.001	0.5	0.007	4.1
Tomato Sample II	28	0.20	0.185	93.8	0.001	0.4	0.012	5.8

^a TRR = sum of extracted and unextracted radioactivity (PES)^b PES = post extracted solids (total unextracted)Table 16 Summary of characterization and identification of ¹⁴C residues in tomato fruit following a single foliar application of [U-Aniline-¹⁴C]-flufenoxuron at 0.125 kg ai/ha

Compound	Tomato Sample (0 DAT)		Tomato Sample I (28 DAT)		Tomato Sample II (28 DAT)	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Flufenoxuron	NR	NR	NR	NR	0.182	91.0
Total Extracted	0.376	98.9	0.164	95.9	0.188	94.2
Total Unextracted	0.004	1.1	0.007	4.1	0.012	5.8
Accountability ^a	0.380	100	0.171	100	0.200	100

NR = Not reported. Identification/Characterization of residues was only performed on the 28 DAT Tomato Sample II.

^a Accountability = Total extracted + total unextracted

Chinese cabbage

[U-Aniline-¹⁴C]flufenoxuron mixed in approximately equal proportions with [aniline-¹⁵N] flufenoxuron, formulated as an emulsifiable concentrate, was applied as a single foliar application at a rate equivalent to 0.10 kg ai/ha to Chinese cabbage plants (grown outdoors) during leaf development. Cabbage plants were harvested at 0 and 28 days after treatment (DAT).

Samples of wrapper leaves were surface washed with ACN:water (7:3, v/v) five times prior to extraction. The washed leaves were homogenized, extracted (once for 0 DAT samples and twice for 28 DAT samples) with ACN:water (7:3, v/v) and filtered. The surface washes and extracts were diluted with water and partitioned three times with DCM. The organic phase was concentrated, then analysed by thin-layer chromatography (TLC). Radioactivity in surface washes and extracts were determined by LSC while the unextracted solid residues were subjected to combustion analysis.

Total radioactive residue (TRR) in/on cabbage wrapper leaves declined from 5.5–7.0 mg eq/kg (average 6.3 mg eq/kg) on day 0, to 0.33–0.36 mg eq/kg (average 0.35 mg eq/kg) by day 28. The total extracted residues (surface washes and extracts from the washed leaves) accounted for 94–97% of the TRR (0.32–6.8 mg eq/kg). However, at 0 DAT, the surface wash represented the majority of the extractable residues while at the 28 DAT, the leaf extracts accounted for much of the extractable radioactivity. Unextracted residues comprised 2.6–5.9% of the TRR (0.01–0.21 mg eq/kg). Only the 28-DAT sample (0.36 mg eq/kg) underwent identification/characterization of residues. Radio- TLC of the extracted radioactivity showed the parent flufenoxuron as the only identified residue, accounting for 93% of the TRR (0.34 mg eq/kg).

Table 17 Total radioactive residues (TRR) in Chinese cabbage leaves following a single foliar application of [U-Aniline-¹⁴C]-flufenoxuron at 0.1 kg ai/ha

Matrix	DAT	TRR ^a (mg eq/kg)	Distribution of radioactive residues					
			Surface wash (ACN+ water)		Leaf Extract		PES ^b	
			mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% T2.8RR
Cabbage	0	6.3	5.3	84.0	0.8	13.2	0.2	2.8
Cabbage	28	0.35	0.07	18.9	0.27	75.9	0.01	5.2

All values reported as mean of replicate samples

^a TRR = sum of extracted and unextracted radioactivity (PES)

^b PES = post extracted solids (total unextracted)

Table 18 Summary of characterization and identification of ¹⁴C residues in Chinese cabbage leaves following a single foliar application of [U-Aniline-¹⁴C]-flufenoxuron at 0.1 kg ai/ha

Compound	Cabbage sample (0 DAT)		Cabbage sample (28 DAT)	
	mg eq/kg	% TRR	mg eq/kg	% TRR
Flufenoxuron	NR	NR		92.5
Total Extracted		97.2		94.8
PES		2.8		5.2
Accountability ^a		100		100

NR = Not reported. Identification/Characterization of residues was only performed on cabbage (28 DAT) sample.

^a Accountability = Total extracted + PES

Overview of metabolic pathway in plants

The grape, apple, tomato and cabbage metabolism studies indicated that parent flufenoxuron was the only residue identified in all the tested primary crop matrices, and was found at > 90% of the TRR except grapes, where parent accounted for 50% of the TRR. No other metabolites were identified and no other residues were characterized (other than polar unknowns). The metabolism data (including autoradiography of the apple samples) indicated that the majority of radioactivity remained on the leaves or surface of the fruit, with limited translocation.

Environmental fate

The Meeting received information on, aerobic and anaerobic degradation in soil, photolysis on soil and pH based degradation. In conformance with the FAO manual 2009, only studies on aerobic degradation in soil were considered for the current evaluation. The fate and behaviour of flufenoxuron in the environment was investigated using amide-[U-¹⁴C]-ring-labelled-flufenoxuron.

Study 1

The rate of aerobic degradation of flufenoxuron in three soils at a temperature of 20 °C was investigated [Goodyear and Gross, 2001, ENV 01-030]. Samples of test soils (50 g dry weight equivalent at 45% maximum water holding capacity (MWHC)), sieved to 2 mm were dispensed into flasks and treated with amide-[U-¹⁴C]-ring-labelled-flufenoxuron at a nominal rate of 0.15 µg ai/g, corresponding to a field application rate of 150 g ai/ha. The treated soils were incubated in the dark at 20 ± 2 °C with moistened carbon dioxide free air drawn through the flasks for up to 120 hours. Volatiles in effluent air were trapped successively in ethanediol, 2% paraffin in xylene and 2 M sodium hydroxide. Samples were taken at 0, 2, 7, 14, 30, 59, 90 and 120 days after treatment (DAT). Trapping solutions were sampled and replaced on the same days.

Flufenoxuron was the only residue present at each sampling interval. The flufenoxuron concentration decreased steadily in all three soil types. At day 0, flufenoxuron represented > 99% TAR and decreased to 22–52% TAR after 120 days of incubation.

Table 19 Recovery of radioactivity and distribution of metabolites after application of amide-¹⁴C-flufenoxuron to Chapel Hill Farm soil and incubation under aerobic conditions [%TAR]

Soil Name				SK 960087		
Source				Chapel Hill Farm, Empingham, Rutland, UK		
UK Particle Size Distribution						
Sand % (2000–63 µm)				33		
Silt % (63–2 µm)				35		
Clay % (< 2 µm)				32		
Textural Class				Clay loam		
Organic carbon (%)				2.7		
Organic matter (%)				4.7		
pH in H ₂ O				8.0		
pH in 1 M KCl				7.2		
CEC (mEq/100 g)				19.6		
Water Holding Capacity pF0 (0.001 bar)%				56.6		
Water Holding Capacity pF2.5 (0.33 bar)%				25.0		
Microbial biomass (µg C/g)						
—start of study				526.5		
—end of study				500.1		
Day	Flufenoxuron	Unresolved	CO ₂	Other volatiles	Water soluble	Total % recovery
0	100.0	0.4	NA	NA	ND	102.7
2	95.1	0.7	0.3	ND	ND	99.4
7	91.1	0.3	1.4	ND	ND	97.6
14	89.5	ND	2.8	ND	ND	100.5
30	83.1	ND	5.5	ND	ND	97.9
59	70.2	0.3	10.1	ND	1.7	97.2
90	58.6	0.2	18.5	ND	2.3	98.6
120	51.6	0.4	23.2	ND	2.1	98.5

ND =Not detected

NA = Not applicable

CO₂ is the sum of the radioactivity in the 2M sodium hydroxide traps

Other volatiles is the sum of the radioactivity in the ethanediol and 2%paraffin in xylene traps

Table 20 Recovery of radioactivity and distribution of metabolites after application of amide-¹⁴C-flufenoxuron to Newhaven soil and incubation under aerobic conditions [%TAR]

Soil Name				SK 15556090		
Source				Newhaven Cottage, Hartington Upper Quarter, Derbyshire, UK		
UK Particle Size Distribution						
Sand % (2000–63 µm)				23		
Silt % (63–2 µm)				57		
Clay % (< 2 µm)				20		
Textural Class				Clay loam		
Organic carbon (%)				4.5		
Organic matter (%)				7.8		
pH in H ₂ O				6.7		
pH in 1 M KCl				6.2		
CEC (mEq/100 g)				17.4		
Water Holding Capacity pF0 (0.001 bar)%				107.7		
Water Holding Capacity pF2.5 (0.33 bar)%				48.5		
Microbial biomass (µg C/g)						
—start of study				604.8		
—end of study				654.1		
Day	Flufenoxuron	Unresolved	CO ₂	Other volatiles	Water soluble	Total %recovery
0	101.0	0.5	NA	NA	ND	103.2
2	96.5	0.7	1.4	ND	ND	101.9
7	85.6	0.3	5.4	ND	1.1	100.8
14	69.1	ND	13.3	ND	0.6	97.1
30	56.2	0.1	21.9	ND	1.5	97.4

Soil Name				SK 15556090		
Source				Newhaven Cottage, Hartington Upper Quarter, Derbyshire, UK		
59	37.6	0.2	34.7	ND	1.8	98.4
90	24.7	0.1	46.0	ND	2.2	99.5
120	22.0	0.1	52.5	ND	1.9	101.6

ND =Not detected

NA = Not applicable

CO₂ is the sum of the radioactivity in the 2 M sodium hydroxide traps

Other volatiles is the sum of the radioactivity in the ethanediol and 2%paraffin in xylene traps

Table 21 Recovery of radioactivity and distribution of metabolites after application of Amide-¹⁴C-flufenoxuron to Baylam soil and incubation under aerobic conditions [%TAR]

Soil Name				PT 103		
Source				Baylam, Ipswich, Suffolk, UK		
UK Particle Size Distribution						
Sand % (2000–63 µm)				71		
Silt % (63–2 µm)				14		
Clay % (< 2 µm)				15		
Textural Class				Sandy loam		
Organic carbon (%)				1.4		
Organic matter (%)				2.4		
pH in H ₂ O				5.5		
pH in 1 M KCl				4.9		
CEC (mEq/100 g)				8.3		
Water Holding Capacity pF0 (0.001 bar)%				47.6		
Water Holding Capacity pF2.5 (0.33 bar)%				16.2		
Microbial biomass (µg C/g)						
—start of study				434.9		
—end of study				226.1		
Day	Flufenoxuron	Unresolved	CO ₂	Other volatiles	Water soluble	Total %recovery
0	99.1	0.5	NA	NA	ND	100.6
2	96.2	0.7	1.2	ND	ND	101.8
7	82.1	0.2	6.1	ND	0.8	98.4
14	73.4	0.2	10.8	ND	0.7	98.1
30	60.3	0.2	19.5	ND	1.5	100.2
59	50.5	0.2	24.3	ND	1.8	96.4
90	46.8	0.3	30.1	ND	1.5	98.7
120	38.3	0.1	36.4	ND	ND	97.4

ND =Not detected

NA = Not applicable

CO₂ is the sum of the radioactivity in the 2M sodium hydroxide traps

Other volatiles is the sum of the radioactivity in the ethanediol and 2% paraffin in xylene traps

Table 22 Dissipation times of flufenoxuron

Parameter	SK 960087	SK 15556090	PT 103
DT ₅₀ (days)	124	36	64
DT ₉₀ (days)	432	191	449
R2 (correlation coefficient)	0.998	0.997	0.997

Study 2

The aerobic degradation rate of 2-fluoroaniline-[U-¹⁴C]-ring-labelled-flufenoxuron was investigated in two types of soils, while the degradation of the flufenoxuron-derived soil metabolite, ¹⁴C-Reg. No. 4064702, was investigated in four different soils [Stephen and Ebert, 2003, 83303]. Samples of test soils (40% MWHC) were dispensed into glass bottles, treated at a nominal rate of 0.05 mg ai/kg,

corresponding to a field application rate of 40 g ai/ha, and incubated in the dark at 20 ± 2 °C. Samples were taken at 0, 3, 7, 14, 30, 57, 91 and 119 days after treatment (DAT).

Table 23 Soil characteristics

Soil Name	Bruch West	Li35B	LUFA2.2	LUFA 3.
Particle Size (Pipette method) (%)				
2000 to ≥ 1000 μm	0.2	1.0	0.5	0.5
1000 to ≥ 500 μm	2.7	6.5	2.3	2.1
500 to ≥ 250 μm	16.3	29.6	25.4	5.2
250 to ≥ 100 μm	39.0	30.0	45.6	26.8
100 to ≥ 50 μm	17.8	8.6	6.7	22.1
50 to ≥ 2 μm	23.1	17.0	1504	37.9
< 2 μm	0.8	7.4	4.1	5.4
Textural Class	Loamy sand	Loamy Sand	Loamy sand	Sandy loam
TOC (Coulometric titration) (%)	2.29	0.89	2.38	2.38
TC (Coulometric titration) (%)	3.90	0.90	2.38	4.00
pH in H_2O	7.0	7.1	6.6	8.1
pH in CaCl_2	7.8	6.3	5.9	7.4
CEC (mval Ba/100 g dry weight)	14.0	7.4	9.7	19.6
Water Holding Capacity (g/100 g dry weight)	30.4	27.7	39.4	42.8
Total Nitrogen (%)	0.16	0.10	0.19	0.22
Ammonium-N (Ion chromatography) (mg/100 g dry weight)	0.3	0.3	0.5	0.4
Nitrate-N (Ion chromatography) (mg/100 g dry weight)	0.20	0.10	0.09	1.8
Microbial biomass (OxiTop method) (mg C/100 g dry weight)	30.2	16.2	36.1	67.8
Ratio Microbial biomass / TOC (calculated) (%)	1.3	1.8	1.5	2.8

The amounts of extractable residues, in both soils treated with radiolabelled flufenoxuron, decreased to $\leq 50\%$ of the total applied radioactivity while the bound residues increased to 38.8–43.8% TAR. No formation of CO_2 was noted. Following treatment of four soils with the metabolite Reg. No. 4064702, less than 25.5% TAR was extractable after 119 days of incubation. The bound residues reached levels of 65–80% TAR at the end of the incubation period. In two of the soils, slow mineralization was observed, reaching 2–8% TAR at the end of incubation while in the other two soils, there was no evidence of CO_2 evolution.

When treated with radiolabelled flufenoxuron, the metabolite Reg. No. 4064702, representing the fluoroaniline moiety after cleavage of the flufenoxuron molecule, was identified by chromatographic comparison with the ^{14}C -labelled reference compound. The highest concentration of this metabolite was reached after 30 days of incubation, accounting for 4.1–8.3% TAR. Flufenoxuron decreased to 45.8–51.0% TAR in the soil after 119 days. While other peaks could be observed in the chromatograms at 119 DAT, all were below the limit of quantification (0.001 mg/kg). The DT_{50} was calculated to be in the range of 115–122 days (Table 24).

In all four tested soils treated with Reg. No. 4064702, the metabolite degraded relatively quickly reaching $< 50\%$ TAR after 57 days of incubation and 16–24% TAR after 119 days. According to the chromatograms of the extracts from sampling days 57, 91 and 119, two degradation products were detected and present at concentrations close to the LOQ. One of these appeared to be the metabolite Reg. No. 241208, DT_{50} values were in the range of 47–59 days (Table 24).

Analyte	Soil	DT ₅₀ (days)	DT ₉₀ (days)
Flufenoxuron ^a	Bruch West	122	407
	Li35B	115	381
Reg. No. 4064702 ^b	Bruch West	57	190
	Li35B	56	186
	LUFA 2.2	59	196
	LUFA 3A	47	156

^b Degradation rates obtained using a 3 compartment model

Residues in Rotational Crops

METHODS OF RESIDUE ANALYSIS

The meeting received analytical method descriptions and validation data for flufenoxuron in plant and animal commodities and in soil and water. For the majority of the methods, residues of flufenoxuron are measured by HPLC-MS/MS with two specific mass transitions or with HPLC-UV at 254–260 nm for each analyte. All methods were validated at the determined LOQs, ranging from 0.01–0.05 mg/kg depending on the matrix. A summary of the analytical methods for plant and animal commodities and environmental media is provided below.

A number of scientific papers report the validation of the QuEChERS multiresidue method using GC-MS/MS for flufenoxuron in various plant commodities.

[illegible]

P1907-G	Apple, citrus, soya bean, wheat grain	Flufenoxuron CL 359882, CL 932338, CL 211558	methanol, water and HCl		HPLC- MS/MS 0.01 mg/kg	2010/1051669
Not specified	Tea Leaves	Flufenoxuron	Aqueous acetone	Liquid/liquid partitioning with hexane followed by ACN.	HPLC-UV 0.8 mg/kg	FX-790-025
Not specified	Infused tea	Flufenoxuron	Precipitation with acetone and lead acetate	Liquid/liquid partitioning with hexane.	HPLC-UV 0.8 mg/kg	FX-790-026
P1340G	Green tea	Flufenoxuron, CL 932338, CL 211558, CL 359882	Methanol/ water/HCl		HPLC- MS/MS 0.01 mg/kg	2007/300204
	Green tea	Flufenoxuron	Methanol/ water/HCl		HPLC- MS/MS 0.1 mg/kg	2008/1042807 Independent laboratory validation

Table 26 Overview of flufenoxuron analytical methods for animal matrices

Method	Matrix	Analyte(s)	Extraction	Clean-up	Detection, LOQ	Reference
Enforcement methods						
SAMS 458-1	Fat	Flufenoxuron	Warm acetone/hexane (20:80 v/v) and sodium sulphate	Partitioning with ACN and clean-up by RP HPLC.	HPLC-UV 0.01 mg/kg	FX-245-003
	Fat	Flufenoxuron	Methylene chloride and sodium sulphate	Clean-up by SPE and RP HPLC.	HPLC-UV 0.05 mg/kg	FX-245-009 Independent laboratory validation
SAMS 486-1	Milk	Flufenoxuron	Diethyl ether and hexane	Partitioning with ACN followed by clean up with NP HPLC.	HPLC-UV 0.1 µg/L	FX-245-004
	Milk	Flufenoxuron	Potassium oxalate and ethanol followed by diethyl ether and hexane	Partitioning with ACN followed by clean up with NP HPLC.	HPLC-UV 0.01 mg/kg	FX-245-008 Independent laboratory validation
Data generation methods						
DFG S 19	Milk, meat, eggs	Flufenoxuron	Aqueous acetone		HPLC- MS/MS 0.01 mg/kg	2002/5004112 (not sufficiently validated)
SAMS 457-2	Fat, whole blood, muscle, kidney, liver, bone marrow	Flufenoxuron	Sodium sulfate, hexane and ACN	Liquid/liquid partitioning and clean-up by NP or RP HPLC	HPLC-UV 0.03 mg/kg	FX-245-002 (1989/7000985)
SAMS 457-1	Liver	Flufenoxuron	Acetone/hexane (20:80 v/v) and sodium sulphate	Partitioning with water/ACN (20:80 v/v).	HPLC-UV LOQ not reported	FX-245-010
Not specified	Blood	Flufenoxuron	Warm acetone	Partitioning with hexane.	HPLC-UV LOQ not reported	FX-245-006
SAMS 492-1	Eggs	Flufenoxuron	Boiling acetone and hexane	Partitioning with ACN followed by clean-up with	HPLC-UV 0.01 mg/kg	FX-245-005

				NP HPLC.		
01791.PCC	Liver of poultry	REG. NO. 4064702	Acetonitrile	Partitioning with n-hexane and clean-up by RP semi-preparative HPLC	HPLC-UV 0.03 mg/kg	FX-705-006

Plant materials

Method RLA 12675 involves extraction of flufenoxuron residues from finely chopped fruit with dichloromethane. An aliquot of the extract is evaporated to dryness, reconstituted in cyclohexane, and then partitioned with water. An aliquot of the cyclohexane phase is taken, evaporated to dryness, reconstituted in acetonitrile:methanol:water (58:10:32, v/v/v), and analysed by LC-MS/MS using the positive ionization mode monitoring ion transitions m/z 498→158 (for quantitation) and m/z 498→141 (for confirmatory purposes).

The applicability of the method was confirmed in an independent laboratory by Schulz (2004, 2004/1000759) and Saha (2008, 2008/7012012). In both laboratories, parent flufenoxuron was analysed with a validated LOQ of 0.05 mg/kg (Table 27).

Table 27 Independent laboratory recovery results for method RLA 12675

Matrix	Fortification level [mg/kg]	No. of tests	Range of Recoveries [%]	Mean Recovery [%]	SD
Tomato	0.05	5	78–92	83	5.7
	0.5	5	78–88	82	4.3
Grapes	0.05	5	86–98	90	4.8
	0.5	5	87–93	90	2.7
Wheat grain	0.05	5	84–92	87	2.9
	0.5	5	79–87	83	3.2
Wheat forage	0.05	5	81–85	83	1.8
	0.5	5	84–89	86	1.8
Wheat straw	0.05	5	75–83	78	3.2
	0.5	5	78–89	83	3.9
Rape seed	0.05	5	80–90	88	5.8
	0.5	5	87–97	93	3.8
Orange fruit	0.05	5	69–81	74	5
	0.5	5	73–86	81	6
Melon fruit	0.05	5	82–93	85	5
	0.5	5	82–95	89	5

Tea

An independent laboratory method validation was conducted by Marin, (2008, 2008/1042807) to determine the validity of the procedure P 1340G to analyse flufenoxuron in tea (green).

Table 28 Independent laboratory recovery results of method P 1340G

Matrix	No. of tests	Fortification level [mg/kg]	Transition ($m/z=489 \rightarrow 158$) ^a			Transition ($m/z=489 \rightarrow 141$)		
			Range of Recoveries [%]	mean [%]	SD [+/–]	Range of Recoveries [%]	mean [%]	SD [+/–]
Tea (green)	5	0.1	65–81	75	7	63–80	72	7
	5	10	76–88	80	5	73–85	78	4

^a Used for quantitation

Animal materials

Method SAMS 486-1 involves treating a sample of milk with potassium oxalate solution and ethanol followed by extraction with diethyl ether and hexane. The diethyl ether is removed by evaporation and the hexane is partitioned with acetonitrile. The acetonitrile is exchanged for a mixture of hexane, ethanol and acetic acid and cleaned-up by normal phase HPLC. Residues of flufenoxuron are determined by reverse-phase HPLC with UV detection at 254 nm.

The applicability of the method was confirmed by an independent laboratory by Skorczynski (1997, RES 97-027) where parent flufenoxuron was analysed with a validated LOQ of 0.01 mg/kg (Table 29).

Table 29 Independent laboratory recovery results of method SAMS 486-1

Matrix	Fortification level [mg/kg]	No. of tests	Range of Recoveries [%]	Mean Recovery [%]
Milk	0.01	2	84–85	85
	0.02	2	89–110	100

Method SAMS 458-1, involves extracting residues of flufenoxuron in fat with methylene chloride and sodium sulphate, followed by evaporation to dryness, then re-dissolving the residue in hexane followed by solid-phase extraction clean-up and reversed-phase HPLC-clean-up. Determination of residues of flufenoxuron is performed by normal-phase HPLC with UV-detection at 254 nm.

The applicability of the method was confirmed in an independent laboratory by Skorczynski (1997, RES 97-037) where parent flufenoxuron was analysed with a validated LOQ of 0.05 mg/kg (Table 30).

Table 30 Independent laboratory recovery results of method SAMS 458-1

Matrix	Fortification level [mg/kg]	No. of tests	Range of Recoveries [%]	Mean Recovery [%]
Beef fat	0.05	2	75–93	84
	0.1	2	77–81	79

STABILITY OF PESTICIDES IN STORED SAMPLES

Plant materials

The storage stability of flufenoxuron has been investigated in various plant matrices for a storage period of up to 36 months.

The storage stability of flufenoxuron was investigated in cottonseed, orange, grape and apple for up to 36 months (Gillard, D.F., 1993a, FX-326-004), in lettuce for 27 months (Edwards, 2000?a, 2000/1021958; Farrell, 1996a, FX-726-003) and in watermelon peel and pulp for 25–26 months (Edwards, 2000b, 2000/1021961; Edwards, 2000c, 2000/1021962).

Homogenised samples were weighed into glass jars and fortified individually at levels of 0.1 mg/kg (cottonseed, orange, grape and apple) and 0.5 mg/kg (lettuce, watermelon peel and pulp). After fortification, the solvent was allowed to evaporate. In addition, untreated samples of each sample material were prepared for control and recovery experiments. Subsequently the jars were closed and stored deep frozen until analysis (except for the day 0 samples). At each sampling interval, 2–3 fortified and two control samples were removed from the deep-freezer. Subsequently, one of the control samples of each sample material was freshly fortified with flufenoxuron to determine the concurrent recoveries. Fortification levels were at the same magnitude as the spiked storage samples.

The grape and apple samples were analysed according to the Analytical Method SAMS 423-3. Briefly, flufenoxuron is extracted from the samples by blending with methylene chloride and sodium sulfate. An aliquot of the extract was concentrated to dryness and cleaned up by solid phase extraction prior to HPLC analysis.

The cottonseed and orange samples were analysed according to the Analytical Method SAMS 454-1 where flufenoxuron was extracted by blending with acetone:hexane (v:v) and sodium sulphate. An aliquot of the extract was concentrated to dryness, reconstituted in acetonitrile and partitioned with hexane. The acetonitrile phase was cleaned up by solid phase extraction prior to HPLC analysis.

Lettuce samples were analysed using Analytical Method RLA 12466.00V while watermelon peel and pulp samples were analysed using Analytical Method RLA 12482.00V. No description was provided of either method.

No significant degradation of the flufenoxuron residues was observed in cottonseed, orange, grape and apple up to 36 months of storage, in lettuce for 27 months and watermelon (pulp and peel) for up to 26 months (Table 31).

Table 31 Storage stability of flufenoxuron in plant commodities fortified with flufenoxuron at 0.1 mg/kg

Matrix	Storage Period (months)	Residue levels in stored samples ^a (%)			Procedural recoveries ^a (%)
		Individual Values	Mean	RSD (%)	Individual Values
Cottonseed	0	126.0, 125.8, 132.0	128	2.8	125.2
	3	107.8, 87.0	98	—	97.4
	6	92.0, 95.0, 81.0	89	8.2	80.0
	12	123.2, 113.2, 130.0	122	6.9	126.4
	14	—	—	—	—
	18	100.8, 104.0, 113.5	106	6.2	121.2
	24	91.2, 104.4, 86.8	94	9.7	110.4
	36	84.2, 79.8, 77.0	80	4.5	81.0
Orange	0	93.2, 112.0, 111.2	105	10.1	116.8
	4	102.8, 114.8, 110.4	109	5.6	119.6
	6	113.2, 113.2, 118.4	115	2.6	116.0
	12	—	—	—	—
	14	126.8, 105.6	117	—	112.8
	18	106.0, 115.4, 120.0	114	6.3	106.6
	24	95.0, 90.8, 84.8	91	5.7	89.6
	36	85.2, 97.2, 87.6	90	7.1	97.6
Grape	0	109.0, 114.0, 109.0	111	2.6	115.0
	3	120.0, 128.5, 106.5	119	9.4	107.0
	6	105.0, 107.0, 106.0	106	0.9	101.0
	12	—	—	—	—
	14	110.0, 109.5	110	—	128.5
	18	119.5, 127.0, 127.0	125	3.5	127.5
	24	116.0, 118.5, 128.5	121	5.5	94.5
	36	81.5, 93.0, 111.0	95	15.6	89.5
Apple	0	105.5, 98.5	103	—	101.5
	3	119.5, 115.0	118	—	98.0
	6	104.0, 106.5, 121.0	111	8.3	95.5
	12	—	—	—	—
	14	114.0, 124.0	119	—	121.0
	18	129.0, 123.5, 127.5	126	2.2	111.0
	24	79.0, 98.0, 104.5	94	14.1	88.5
	36	110.8, 109.4, 105.8	109	2.4	103.5
Lettuce	0	108, 107	108	—	110
	1	94, 93	94	—	106
	3	74, 96	85	—	98

Matrix	Storage Period (months)	Residue levels in stored samples ^a (%)			Procedural recoveries ^a (%)
		Individual Values	Mean	RSD (%)	Individual Values
	14	89, 85	87	—	104
	18	96, 87	92	—	88
	27	87, 85	86	—	90
Watermelon peel	0	108, 107	108	—	107
	1	101, 92	97	—	107
	3	88, 85	87	—	92
	14	106, 102	104	—	109
	18	83, 85	84	—	89
	26	69, 80	75	—	101
Watermelon pulp	0	109, 108	109	—	109
	1	104, 102	103	—	110
	3	105, 97	101	—	109
	14	99, 97	98	—	98
	18	88, 89	89	—	95
	26	79, 92	86	—	78

^a Reported as a function of the nominal (0.1 mg/kg) spiking level

Animal Materials

For animal matrices, the storage stability of flufenoxuron has been investigated for a storage period of up to 12 months (Lewis 1993a, FX-326-003).

The freezer stability of flufenoxuron in various animal matrices was investigated over a period of one year (53 weeks). Untreated bovine muscle, liver, kidney, fat and milk and hen skin, egg yolk and egg white samples were fortified with radiolabelled flufenoxuron at three different concentrations. The samples were stored at -19 °C. All beef and hen skin samples were analysed according to the Analytical Method SAMS 457-2 with a slight modification for extracting heparin-treated blood. Egg yolk and egg white samples were analysed according to the Analytical Method SAMS 492-1. Cow's milk samples were analysed based on the Analytical Method SAMS 486-1. Residues were analysed by TLC, HPLC-UV and LSC.

No significant degradation of the flufenoxuron residues was observed in any of the animal matrices except egg whites where a steady decline was noted by 53 weeks (31% dissipation).

Table 32 Storage stability of flufenoxuron in animal commodities

Commodity	Fortification Level (mg/kg)	Recoveries in stored samples ^a (%)				
		Duration (weeks)				
		0	4	13	26	53
Bovine muscle	0.107	83	61	67	62	61
	1.09	84	62	72	63	66
	11.3	83	63	67	63	68
Bovine liver	0.108	87	75	79	76	84
	1.08	83	73	71	79	88
	10.9	89	75	85	83	87
Bovine kidney	0.108	88	79	—	70	77
	1.10	89	73	—	76	78
	11.3	88	72	—	77	81
Bovine fat	0.106	98	103	92	94	92
	1.04	98	104	95	99	90
	10.9	97	102	92	94	91
Cow's milk	0.0556	88	86	85	90	87
	0.56	91	90	80	78	89
	5.91	88	88	84	84	86
Bovine blood	0.0547	82	80	67	68	83
	0.578	79	77	68	65	84
	5.94	79	81	72	71	84

Commodity	Fortification Level (mg/kg)	Recoveries in stored samples ^a (%)				
		Duration (weeks)				
		0	4	13	26	53
Hen skin	0.109	97	95		85	92
	1.11	96	92	—	80	90
	11.5	96	90		89	89
Egg yolk	0.109	95	94		83	89
	1.09	92	94	—	89	87
	11.8	89	87		81	82
Egg white	0.110	96	NA	62	70	65
	1.14	93	81	63	80	64
	11.6	94	82	71	66	76

^a Reported as total percent applied radioactivity, are a function of the specified fortification level.

USE PATTERN

Flufenoxuron is a new benzoylurea type of acaricide/insecticide which inhibits chitin biosynthesis in nymphal mites and caterpillars. Flufenoxuron is registered in Brazil and in Japan as an emulsifiable concentrate (EC).

The Meeting received the registered label from Brazil for orange, apple and from Japan for tea in the original languages as well as the English translations.

Table 33 List of uses of flufenoxuron

Crop	Country	Application			Application rate per treatment			PHI [days]
		Method	No. per season	Application interval [days]	kg ai/hL	Water L/ha	kg ai/ha	
Orange	Brazil	spray	2	30	0.003–0.005	—	—	15
Apple	Brazil	spray	—	—	0.01	1200–2000	—	35
Tea	Japan	spray	2	7–14	0.0025	2000–4000	0.05–0.1	7

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Residue levels were reported as measured. Application rates were always reported as flufenoxuron equivalents. When residues were not quantifiable they are shown as below the LOQ, e.g., < 0.01 mg/kg.

Application rates and spray concentrations have generally been rounded to two significant figures.

All samples were analysed using either an HPLC-UV method (254 nm) which involved extraction with DCM and sodium sulphate and a validated LOQ of 0.05 mg/kg or an HPLC/MS/MS method involving extraction with methanol/water/HCl and partitioning in water cyclohexane with an LOQ of 0.01 mg/kg.

Laboratory reports included method validation including batch recoveries at fortification levels bracketing the LOQ, 10× LOQ and 100× LOQ. Dates of analyses or duration of residue sample storage were also provided. These storage intervals were covered by the storage stability studies on parent in high oil, high acid and high water content matrices.

Field reports provided data on the sprayers used and their calibration, plot size, residue sample size and sampling date. Although trials included control plots, no control data are recorded in the tables as residues in control samples were below the method LOQ. Residue data are recorded unadjusted for % recovery. Where multiple samples were taken from a single plot or where multiple analyses were conducted for the same sample, the average value is reported, with the individual

values captured in brackets. Residues from trials conducted according to critical GAP are underlined and have been used for the estimation of maximum residue levels, STMR and HR values.

Crop	Field/Greenhouse	Treatment Type	Countries	Table
Orange	Field	Foliar spray	Brazil, Greece, Italy, South Africa	34
Apple	Field	Foliar spray	Chile	35
Tea	Field	Foliar spray	Japan	36

Oranges

Eleven supervised residue trials were conducted during 1986, 2007 and 2011 on oranges grown in Brazil and treated once or twice with a soluble concentrate (SL) or dispersible concentrate (DC) formulation of flufenoxuron at rates of 0.003–0.005 kg ai/hL. Oranges were harvested 0–28 days following the last application. In the 1986 trials, residues are reported in peel and pulp based on ratios of 37% peel and 63% pulp. For the remaining trials, the residues are reported on a whole fruit basis.

Five additional orange trials, involving application of a DC formulation, were also conducted in Greece (two; 1996), Italy (one, 1996) and South Africa (two; 1989). The number of applications and treatment rates for Greece, Italy and South Africa were 2× 0.005 kg ai/hL, 2× 0.008 kg ai/hL and 1× 0.005 kg ai/hL, respectively. Oranges were harvested 0–28 days following the last application and separated into peel and pulp prior to analysis.

Table 34 Residues of flufenoxuron in oranges following foliar spray with SL or DC formulations

Trial location, Country, Year (Variety)	Application					Growth Stage	DAT, days	Matrix	Flufenoxuron Residues ^a (mg/kg)	Ref
	Form	kg ai/ha	kg ai/hL	no.	RTI, days					
cGap Brazil	100 g/L		0.003–0.005	2	30		15			
Mogi-Mirim-SP, Brazil, 1986 (Pera Coroa)	SL 50 g/L	0.04	0.003	1	NA	Ripening	3	Peel	0.11	I/BER/RE/CI-01185
							3	Pulp	< 0.02	
							3	Whole fruit ^b	0.05	
							7	Whole fruit ^b	0.05	
							7	Peel	< 0.02	
							7	Pulp	0.03	
							15	Whole fruit ^b	0.06	
Mogi-Mirim-SP, Brazil, 1986 (Pera Coroa)	SL 50 g/L	0.04	0.005	1	NA	Ripening	15	Whole fruit ^b	< 0.02	I/BER/RE/CI-01185
							15	Peel	0.03	
							15	Pulp		
								Whole fruit ^b		
							3	Peel	0.2	
							3	Pulp	< 0.02	
							3	Whole fruit ^b	0.09	
Mogi-Mirim-SP, Brazil, 1986 (Pera Coroa)	SL 50 g/L	0.04	0.005	1	NA	Ripening	7	Whole fruit ^b	0.2	I/BER/RE/CI-01185
							7	Peel	< 0.02	
							7	Pulp	0.09	
							15	Whole fruit ^b	0.2	
							15	Whole fruit ^b	< 0.02	
							15	Peel	0.09	
							15	Pulp		
Palea Korinthos, Greece, 1996 (Navalline)	DC	0.08–0.11	0.005	2	151	BBCH 85–89	14	Peel	0.23	96-651-01
							14	Pulp	< 0.05	
							14	Whole fruit ^c	0.11	
							28	Whole fruit ^c	0.27	
							28	Peel	< 0.05	
Palea Korinthos, Greece, 1996 (Navalline)	DC	0.08–0.11	0.005	2	151	BBCH 85–89	28	Pulp	0.13	
							28	Pulp		

Trial location, Country, Year (Variety)	Application					Growth Stage	DAT, days	Matrix	Flufenoxuron Residues ^a (mg/kg)	Ref
	Form	kg ai/ha	kg ai/hL	no.	RTI, days					
								Whole fruit ²⁾		
Drosia Chalkidas,, Greece, 1996 (Navalline)	DC	0.07–0.11	0.005	2	153	BBCH 85–89	14 14 14 28 28 28	Peel Pulp Whole fruit ^c Peel Pulp Whole fruit ^c	0.61 < 0.05 0.25 0.43 < 0.05 0.31	96-651-02
Castellanita Marina, Italy, 1996 (Navalin)	DC 50 g/L	0.07	0.008	2	14	BBCH 85–87	0 0 0	Peel Pulp Whole fruit ^b	0.33 < 0.05 0.15	96-556-01
							7 7 7	Peel Pulp Whole fruit ^b	0.36 < 0.05 0.16	
							14 14 14	Peel Pulp Whole fruit ^b	0.28 < 0.05 0.14	
							21 21 21	Peel Pulp Whole fruit ^b	0.31 < 0.05 0.15	
							28 28 28	Peel Pulp Whole fruit ^c	0.16 < 0.05 0.09	
South Africa, 1989 (Navel)	DC	NS	0.003	1	NA	Ripening	0 0 0 7 7 7 13 13 13 28 28 28	Peel Pulp Whole fruit ^c Peel Pulp Whole fruit ^c Peel Pulp Whole fruit ^c Peel Pulp Whole fruit ^c	0.26 0.01 0.04 0.24 < 0.01 0.07 0.45 < 0.01 0.10 0.33 < 0.01 0.08	FX-710-005
South Africa, 1989 (Navel)	DC	NS	0.005	1	NA	Ripening	0 0 0 7 7 7 13 13 13 28 28 28	Peel Pulp Whole fruit ^c Peel Pulp Whole fruit ^c Peel Pulp Whole fruit ^c Peel Pulp Whole	0.43 0.01 0.13 0.51 < 0.01 0.15 0.46 < 0.01 0.09 0.48 < 0.01 0.12	FX-710-005

Trial location, Country, Year (Variety)	Application					Growth Stage	DAT, days	Matrix	Flufenoxuron Residues ^a (mg/kg)	Ref
	Form	kg ai/ha	kg ai/hL	no.	RTI, days					
								fruit ^c		
Santo Antonio de Posse, Brazil, 2007 (Pera)	DC	0.06	0.003	2	NS	BBCH 88	0 5 10 15 20	Whole fruit	0.05 (0.05, 0.05) 0.06 (0.06, 0.06) 0.05 (0.04, 0.05) 0.07 (0.05, 0.04) 0.09, 0.07) 0.06 (0.05, 0.06)	EC-CD-BRUA/1100-06
Uberlandia, Brazil, 2007 (Natal)	DC	0.06	0.003	2	NS	BBCH 83	0 6 10 15 20	Whole fruit	0.12 (0.11, 0.12) 0.10 (0.10, 0.10) 0.11 (0.10, 0.11) 0.10 (0.09, 0.10) 0.07 (0.07, 0.06)	EC-CD-BRVA/1100-06
Pirassununga, Brazil, 2007 (Valência)	DC	0.06	0.003	2	NS	BBCH 87	15	Whole fruit	0.05 (0.05, 0.04)	EC-R-BRUB/1100-06
Estiva Gerbi, Brazil, 2007 (Valência)	DC	0.06	0.003	2	NS	BBCH 88	15	Whole fruit	0.08 (0.09, 0.06)	EC-R-BRUC/1100-06
Santo Antonio de Posse, Brazil, 2011 (Natal)	DC	0.10	0.005	2	30 ^d	BBCH 87	0 7 15 21	Whole fruit	0.13 (0.16, 0.13, 0.12, 0.12) 0.14 (0.18, 0.14, 0.13, 0.13) <u>0.13</u> (0.13, 0.13, 0.13, 0.12) 0.12 (0.15, 0.10, 0.10, 0.11)	G100555
Sao Sebastiao, da Amoreira, Brazil, 2011 (Navelina)	DC	0.10	0.005	2	30 ^d	BBCH 81	15	Whole fruit	<u>0.09</u>	G100557
Tamarana, Brazil, 2011 (Folha murcha)	DC	0.10	0.005	2	30 ^d	BBCH 85	15	Whole fruit	<u>0.16</u> (0.20, 0.14, 0.14, 0.15)	G100631
Jaboticabal, Brazil, 2011 (Pera)	DC	0.08	0.002	2	30 ^d	BBCH 83	0 7 15 20	Whole fruit	0.05 0.02 0.03 < 0.01	G100697
Jaboticabal, Brazil, 2011 (Pera)	DC	0.10	0.005	2	30 ^d	BBCH 81	0 7 15 21	Whole fruit	0.13 0.10 <u>0.11</u> 0.10	G100737

SL: Soluble concentrate

DC: Dispersible concentrate

^a When there are 2 residues per PHI, the replicate samples from the same plot where there are 4 residues per PHI, these represent four replicate samples from two plots.

^b Calculated using a ratio of 37% peel and 63% pulp^c Calculated using the exact peel and pulp samples^d Determined based on dates of application*Apples*

Fourteen supervised residue trials were conducted in Chile during the 1988–1990 growing seasons on apples treated once, early season, with flufenoxuron at rates of 0.01–0.02 kg ai/hL and harvested 35–150 days following application.

Table 35 Residues of flufenoxuron in apples following foliar spray

Trial location, Country, Year (Variety)	Application					Growth Stage	DAT, days	Flufenoxuron Residues (mg/kg)	Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.				
cGap Brazil	100 g/L		0.01	1200– 2000	1		35		
San Fernando, Chile, 1988 (variety not specified)	NS	0.22	0.01	2200	1	prior to bloom	35	0.45	UCH- SF
							77	< 0.05	
							113	< 0.05	
							126	< 0.05	
	NS	0.33	0.015	2200	1	prior to bloom	35	0.52	
							77	< 0.05	
							113	< 0.05	
							126	< 0.05	
	NS	0.44	0.02	2200	1	prior to bloom	35	0.43	
							77	0.08	
							113	< 0.05	
							126	< 0.05	
Requinoa, Chile, 1988 (variety not specified)	NS	0.18	0.01	1800	1	10–15 mm diameter fruit	15	0.28	UCH- R
							36	0.07	
							49	< 0.05	
							85	< 0.05	
							98	< 0.05	
	NS	0.36	0.02	1800	1	10–15 mm diameter fruit	15	0.46	
							36	0.17	
							49	0.09	
							85	0.12	
							98	0.05	
Teno, Chile, 1988 (Red King Oregon)	NS	0.16	0.01	1560	1	Pink bud	150	< 0.05	CHA 187001
		0.23	0.02				150	< 0.05	
		0.31	0.02				150	< 0.05	
Chimbarongo- Rosselot, Chile, 1988 (Granny-spur)	NS	0.35	0.01	3500	1	90% bloom	148	< 0.05	CHA 187002
		0.52	0.02				148	< 0.05	
		0.70	0.02				148	< 0.05	
Chimbarongo- Donoso, Chile, 1988 (Red King Oregon)	NS	0.86 & 0.64	0.02 & 0.015	4280	2	Pink bud 7–8mm fruit	127	< 0.05	CHA 187003
Chimbarongo- Donoso, Chile, 1988 (Granny-spur)	NS	0.44	0.01		1	50% bloom		< 0.05	
		0.64	0.02				146	< 0.05	
		0.86	0.02					< 0.05	
Olivar-Alto, Chile, 1988 (Red-spur)	NS	0.32	0.01	3180	1	Pink bud 10% bloom	145	< 0.05	CHA 187004
		0.48	0.02				145	< 0.05	
		0.64	0.02				145	< 0.05	
Pelequen, Chile, 1988	NS	0.59	0.02	3950	1	8 mm fruit	125	< 0.05	CHA 187005
		0.40	0.01			95% bloom	141	< 0.05	

Trial location, Country, Year (Variety)	Application					Growth Stage	DAT, days	Flufenoxuron Residues (mg/kg)	Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.				
Red King Oregon)		0.59	0.02			95% bloom	141	< 0.05	
		0.79	0.02			95% bloom	141	< 0.05	
								< 0.05	
Molina, Chile, 1988 (Red King Oregon)	NS	0.16	0.01	1650	1	80% bloom	139	< 0.05	CHA 187006
		0.25	0.02					< 0.05	
		0.33	0.02					< 0.05	
Curico, Chile, 1988 (Granny spur)	NS	0.57	0.01	5680	1	100% petal	138	< 0.05	CHA 187007
Curico Chile, 1988 (Richared)	NS	0.85	0.02	5680	1	50% petal	138	< 0.05	
		1.14	0.02					< 0.05	
Rosario, Chile, 1988 (Richared Delicious)	NS	0.44	0.01	4360	1	Fruit set 2 mm fruit	133	< 0.05	CHA 187008
		0.65	0.02					< 0.05	
		0.87	0.02					< 0.05	
Tinguiririca, Chile, 1988 (Red King Oregon & Red Dpur)	NS	0.77	0.01	5140	1	Fruit set	131	< 0.05	CHA 187009
		1.03	0.02					< 0.05	
Requinoa, Chile, 1990 (Red Spur)	DC 100 g/L	0.42	0.05	8320	1	Developing fruit 10 mm	6	0.24	FX- 711-028
							20	0.07	
							41	0.04	
							55	0.02	
							harvest	<0<0.01	
	DC 100 g/L	0.62	0.08	8320	1	Developing fruit 10 mm	6	0.32	
							20	0.23	
							41	0.16	
							55	0.03	
							harvest	<0<0.01	

NS: Not specified

Pears

Two trials were conducted on a same site located in Chile, during the 1990 growing season, on pears treated once early season with a DC formulation at rates of 0.2–0.38 kg ai/ha and harvested 0–60 days following application.

Table 36 Residue decline of Flufenoxuron in pears following foliar spray with DC Formulation

Trial location, Country, Year (Variety)	Application					Growth Stage	DAT, days	Flufenoxuron Residues (mg/kg)	Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.				
Requinoa, Chile, 1990 (Red Spur)	DC 100 g/L	0.2	NS	NS	1	Developing fruit	0	1.0	FX- 711- 028
							6	0.45	
							20	0.3	
							27	0.13	
							41	0.03	
							60	< 0.01	
	DC 100 g/L	0.38	NS	NS	1	Developing fruit	0	2.62	
							6	0.82	
							20	0.42	
							27	0.30	
							41	0.07	
							60	0.03	

NS: Not specified

Melons

Melons, grown in Brazil during the 2010 season, received four applications of a DC formulation of flufenoxuron at 0.01 kg ai/hL, with 4–7 day re-treatment intervals, and harvested 0–10 days following the last application. For some trials, residues in pulp and peel were determined from whole fruit, based on a ratio of 40% peel and 60% pulp.

Table 37 Residues of flufenoxuron in field grown melons following foliar spray with DC formulation

Location, year (Variety)	Application						Growth Stage	DAT, days	Matrix	Flufenoxuron Residues (mg/kg) ^b	Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days					
Ponta Grossa, Brazil, 2010 (Redondo Gaúcho)	DC 100 g/L	0.10	0.01	1000	4	4	89	10	Whole Fruit	0.07	G080414
Assai, Brazil, 2010 (Híbrido Louis)	DC 100 g/L	0.10	0.01	1000	4	4	77	10	Whole Fruit	0.03	G080415
Mossoro, Brazil, 2010 (Galia)	DC 100 g/L	0.10	0.01	1000	4	4	81	10	Whole Fruit	0.03	G080416
Anhembi, Brazil, 2010 (Casca de Carvalho)	DC 100 g/L	0.10	0.014	700	4	NS	83	0	Peel	0.20	EC-CD- BRUC/1099- 06
								0	Pulp	0.08	
								0	Whole Fruit ^a	0.13	
								1	Peel	0.16	
								1	Pulp	0.06	
								1	Whole Fruit ^a	0.10	
								3	Peel	0.17	
								3	Pulp	0.08	
								3	Whole Fruit ^a	0.12	
								5	Peel	0.15	
								5	Pulp	0.03	
								5	Whole Fruit ^a	0.08	
								7	Peel	0.08	
								7	Pulp	< 0.01	
								7	Whole Fruit ^a	0.04	
								10	Peel	0.10	
Icapui, Brazil, 2010 (Amarelo Gold Mine)	DC 100 g/L	0.10	0.014	700	4	NS	80	0	Peel	0.16	EC-CD- BRUC/1099- 06
								0	Pulp	0.01	
								0	Whole Fruit ^a	0.07	
								1	Peel	0.14	
								1	Pulp	0.01	
								1	Whole Fruit ^a	0.06	
								3	Peel	0.09	

Location, year (Variety)	Application						Growth Stage	DAT, days	Matrix	Flufenoxuron Residues (mg/kg) ^b	Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days					
								3	Pulp	0.01	
								3	Whole Fruit ^a	0.04	
								5	Peel	0.07	
								5	Pulp	< 0.01	
								5	Whole Fruit ^a	0.03	
								7	Peel	0.17	
								7	Pulp	< 0.01	
								7	Whole Fruit ^a	0.07	
								10	Peel	0.1	
								10	Pulp	0.02	
								10	Whole Fruit ^a	0.05	
Anhembi, Brazil, 2010 (Casca de Carvalho)	DC 100 g/L	0.10	0.014	700	4	NS	83	3	Peel	0.17	EC-R- BRUA/1099- 06
								3	Pulp	0.01	
								3	Whole Fruit ^a	0.07	
								7	Peel	0.09	
								7	Pulp	< 0.01	
Mossoro, Brazil, 2010 (Amarelo Gold Mine)	DC 100 g/L	0.10	0.014	700	4	NS	80	7	Whole Fruit ^a	0.04	EC-R- BRUB/1099- 06
								3	Peel	0.14	
								3	Pulp	< 0.01	
								3	Whole Fruit ^a	0.06	
								7	Peel	0.1	
Anhembi, Brazil, 2010 (Imperial)	DC 100 g/L	0.10	0.014	700	4	NS	84	7	Pulp	< 0.01	EC-R- BRUB/11099- 06
								3	Peel	0.19	
								3	Pulp	0.18	
								3	Whole Fruit ^a	0.18	
								7	Peel	0.11	
Lodrina, Brazil, 2010 (Lovis)	DC 100 g/L	0.10	NS	NS	4	4	85	0	Peel	0.46	G100218
									Pulp	< 0.01	
									Whole Fruit	0.12	
							79	3	Peel	0.54	
									Pulp	< 0.01	
									Whole Fruit	0.09	
							75	7	Peel	0.33	

Location, year (Variety)	Application						Growth Stage	DAT, days	Matrix	Flufenoxuron Residues (mg/kg) ^b	Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days					
Ibipora, Brazil, 2010 (Lovis)	DC 100 g/L	0.10	NS	NS	4	4	61	0	Peel	< 0.01	G100219
									Whole Fruit	0.07	
									Peel	0.73	
							57	3	Pulp	< 0.01	
									Whole Fruit	0.2	
									Peel	0.27	
							51	7	Pulp	< 0.01	
									Whole Fruit	0.08	
									Peel	0.28	
							87	3	Pulp	< 0.01	
									Whole Fruit	0.06	
									Peel	0.55	
Senador Canedo, Brazil, 2010 (Gaúcho)	DC 100 g/L	0.10			4	4	87	3	Pulp	0.02	G100220
									Whole Fruit	0.21	
Mossoro, Brazil, 2010 (Goldex)	DC 100 g/L	0.10			4	4	79	3	Peel	0.29	G100221
									Pulp	< 0.01	
									Whole Fruit	0.06	

NS: Not specified

^a Calculated assuming a weight ratio of 40% peel and 60% pulp^b Residues < LOQ were assumed to be at 0.01 mg/kg

Tomato

Tomatoes, grown in Spain during the 2008 season and maintained under protective cover, were treated twice with a DC formulation of flufenoxuron at 0.12–0.13 kg ai/ha and 14 day re-treatment interval. Mature tomatoes were harvested 0–7 days following the last application.

During the 2008 season, field tomatoes grown in Brazil were treated with four applications of a DC formulation of flufenoxuron at 0.01 kg ai/hL and a 7-day re-treatment interval. Tomatoes were harvested 0–7 days following the last application.

Table 38 Residues of flufenoxuron in tomatoes following spray with DC formulation

Trial location, Country, Year (Variety)	Application						Growth Stage	DAT, days	Flufenoxuron Residues (mg/kg)	Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days				
Conil de la Frontera, Spain, 2008 (Caramba) Protected	DC 100 g/L	0.12– 0.13	0.01	1180– 1271	2	14	84 85– 86	0	0.067	AF/12238/PM/1
								3	0.093	
								5	0.084	
								7	0.089	
Conil de la Frontera, Spain, 2008 (Bond)	DC 100 g/L	0.13	0.01	1282– 1288	2	14	64 71	7	0.07	AF/12238/PM/2

Trial location, Country, Year (Variety)	Application						Growth Stage	DAT, days	Flufenoxuron Residues (mg/kg)	Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days				
Protected										
Los Palacios Y Villafranca, Spain, 2008 (V1) Protected	DC 100 g/L	0.12–0.13	0.01	1215–1340	2	14	72 73	7	0.09	AF/12238/PM/3
Zaragoza, Spain, 2008 (Caramba) Protected	DC 100 g/L	0.12–0.13	0.01	1243–1300	2	14	77 81	0	0.16	AF/12238/PM/4
								3	0.21	
								5	0.32	
								7	0.24	
Santa Engracia, Spain, 2008 (Caramba) Protected	DC 100 g/L	0.12–0.13	0.01	1200–1286	2	14	75 77	7	0.1	AF/12238/PM/5
Remolinos, Spain, 2008 (Caramba) Protected	DC 100 g/L	0.13	0.01	1282–1299	2	14	73 87–89	7	0.23	AF/12238/PM/6
Cambados, Spain, 2008 (Disie) Protected	DC 100 g/L	0.13	0.01	1270–1294	2	14	71 73	7	0.1	AF/12238/PM/7
Paende San Vicente Meis, Spain, 2008 (Caramba) Protected	DC 100 g/L	0.12–0.13	0.01	1230–1298	2	14	71 72	7	0.14	AF/12238/PM/8
Ponta Grossa, Brazil, 2008 (Raissa) Field	DC 100 g/L	0.10	0.01	1000	4	7	80	0	0.08	EC-CD-BRTA/1162-06
								3	0.08	
								5	0.07	
								7	0.08	
								10	0.06	
Santo Antonio de Posse, Brazil, 2008 (Bonus F1) Filed	DC 100 g/L	0.10	0.01	1000	4	7	85	0	0.38	EC-CD-BRUA/1162-06
								3	0.37	
								5	0.36	
								7	0.37	
								10	0.21	
Araguari, Brazil, 2008 (Alambra) Field	DC 100 g/L	0.10	0.01	1000	4	7	83	3	0.07	EC-R-BRVA/1162-06
								7	0.08	
Ipiranga, Brazil, 2008 (Raissa) Field	DC 100 g/L	0.10	0.01	1000	4	7	78	3	0.05	EC-R-BRTB/1162-06
								7	0.04	
Santo Antonio de Posse, Brazil, 2011 (Italiano)	DC 100 g/L	0.10	0.01	1000	4	7	89	3	0.18	G100074
								7	0.19	

Trial location, Country, Year (Variety) Comprido) Field	Application						Growth Stage	DAT, days	Flufenoxuron Residues (mg/kg)	Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days				
Senador Canedo, Brazil, 2011 (Carolina) Field	DC 100 g/L	0.10	0.01	1000	4	7	84	3 7	0.02 0.02	G100075

Tea

Eleven trials conducted on tea, grown in Japan during the 2005–2007 growing seasons, were treated twice or five times with an EC formulation of flufenoxuron at a rate of 0.0025 kg ai/hL. The re-treatment intervals were not specified in the study report. Tea leaves were harvested 1–14 days following application. On the day of sampling, tea leaves were processed in accordance with the standard green tea processing procedure and individually tightly sealed in a tea can prior to transportation

Table 39 Residues of flufenoxuron in tea (green) following spray with EC formulation

Trial location, Country, Year (Variety)	Application					Growth Stage ^a	DAT, days	Matrix ^c	Flufenoxuron Residues (mg/kg)	Ref
	Form	g ai/ha	0.0025 kg ai/hL	Water, L/ha	no					
cGAP Japan	EC	50–100	25	2000–4000	2/7–14 day RTI		7			
Kagoshima, Japan, 2005 (Okumidori)	EC (10%)	100	25	4000	2	1.0, 2.5	7	Tea (green)	<u>4.58</u>	217858
Shizuoka, Japan, 2005 (Yabukita)	EC (10%)	100	25	4000	2	0.5–1.5, 2.0–3.0	7	Tea (green)	<u>6.02</u>	
Kyoto, Japan, 2005 (Kanayamidori)	EC (10%)	100	25	4000	2	2.0, 3.0	7	Tea (green)	<u>6.23</u>	
Mie, Japan, 2005 (Sayamakaori)	EC (10%)	100	25	4000	2	1.5, 3.0	7	Tea (green)	<u>11.8</u>	
Fukuoka, Japan, 2005 (Meiryoku)	EC (10%)	100	25	4000	2	1.0–1.5, 2.5–3.0	7	Tea (green)	<u>3.95</u>	
Myazaki, Japan, 2005 (Fuushun)	EC (10%)	100	25	4000	2	2.0, 3.0	7	Tea (green)	<u>2.48</u>	
Shizuoka, Japan, 2005 (Yabukita)	EC (10%)	100	25	4000	2	0.5–1.5, 2.0–3.0	7	Tea (green)	<u>2.37</u>	
Japan, 2007 (Okumidori)	EC (10%)	100	25	4000	5	4.0–5.0 ^b	1	Tea (green)	12	P/B 1340G
						3.0–4.0 ^b	7	Tea (green)	6.4	
						2.0–3.0 ^b	14	Tea (green)	3.1	
Japan, 2007 (Yabukita)	EC (10%)	100	25	4000	5	4.0 ^b	1	Tea (green)	14	
						3.0 ^b	7	Tea (green)	5.9	

Trial location, Country, Year (Variety)	Application					Growth Stage ^a	DAT, days	Matrix ^c	Flufenoxuron Residues (mg/kg)	Ref
	Form	g ai/ha	0.0025 kg ai/hL	Water, L/ha	no					
						1.0 ^b	14	Tea (green)	2.4	
Japan, 2007 (Komakage)	EC (10%)	100	25	4000	5	3.5 ^b	1	Tea (green)	13	
						3.5 ^b	7	Tea (green)	6.1	
						2.0 ^b	14	Tea (green)	1.4	
Japan, 2007 (Yabukita)	EC (10%)	100	25	4000	5	4.0–5.0 ^b	1	Tea (green)	19	
						3.5–4.0 ^b	7	Tea (green)	7.4	
						1.5–2.0 ^b	14	Tea (green)	2.7	

^a Leaf stage

^b Leaf stage at last application

^c On the day harvested, tea leaves were processed in accordance with the standard green tea processing procedure and individually tightly sealed in a tea can prior to transportation

FATE OF RESIDUES IN STORAGE AND PROCESSING

Nature of residue during processing

The hydrolysis of flufenoxuron under processing conditions was investigated by Hassink, J. (2003/1000985). [Difluorobenzamide-ring-U-¹⁴C]-flufenoxuron and [fluoroaniline-ring-U-¹⁴C]-flufenoxuron were diluted in sterile buffered aqueous solution at a nominal concentration of 1 µg ai/L, which corresponds to approximately 50% of the water solubility (at pH 4). Incubation was done at three representative sets of hydrolysis conditions: 90 °C, pH 4 for 20 minutes (pasteurisation); 100 °C, pH 5 for 60 minutes (baking, brewing and boiling) and 120 °C, pH 6 for 20 minutes (sterilisation).

Parent compound and potential hydrolysis products were identified and quantified by HPLC-UV.

Thin layer chromatography was used for confirmation of the identity of the test item by co-chromatography with the non-labelled reference item. Material balances were established for each set of hydrolysis conditions.

Table 40 Hydrolysis of flufenoxuron under simulated processing conditions

Hydrolysis Conditions	Incubation time (min)	[difluorobenzamide-ring-U- ¹⁴ C]- flufenoxuron		[fluoroaniline-ring-U- ¹⁴ C]-flufenoxuron	
		Analyte	% applied radioactivity	Analyte	% applied radioactivity
Pasteurization: pH 4, 90 °C	20	Flufenoxuron	90.0	Flufenoxuron	92.7
		Unidentified	2.6		
		Total Recovery	92.6	Total Recovery	92.7
Baking, boiling, brewing procedure (pH 5, 100 °C)	60	Flufenoxuron	66.3	Flufenoxuron	
		Reg. No. 102719	32.0	Reg. No. 4064703 or Reg. No. 241208	4.3
		Unidentified	1.9	Unidentified	2.9
		Total Recovery	104.3	Total Recovery	95.7
Sterilization (pH 6, 120 °C)	20	Flufenoxuron	83.5	Flufenoxuron	62.7
		Reg. No. 102719	7.6	Reg. No. 4064702	3.8
		Reg. No. 206925	9.2	Reg. No. 4064703 or Reg. No. 241208	9.5

		Reg. No. 4964847	3.5	Reg. No. 4964847	9.2
		Unidentified	0.2	Unidentified	2.9
		Total Recovery	105.4	Total Recovery	88.1

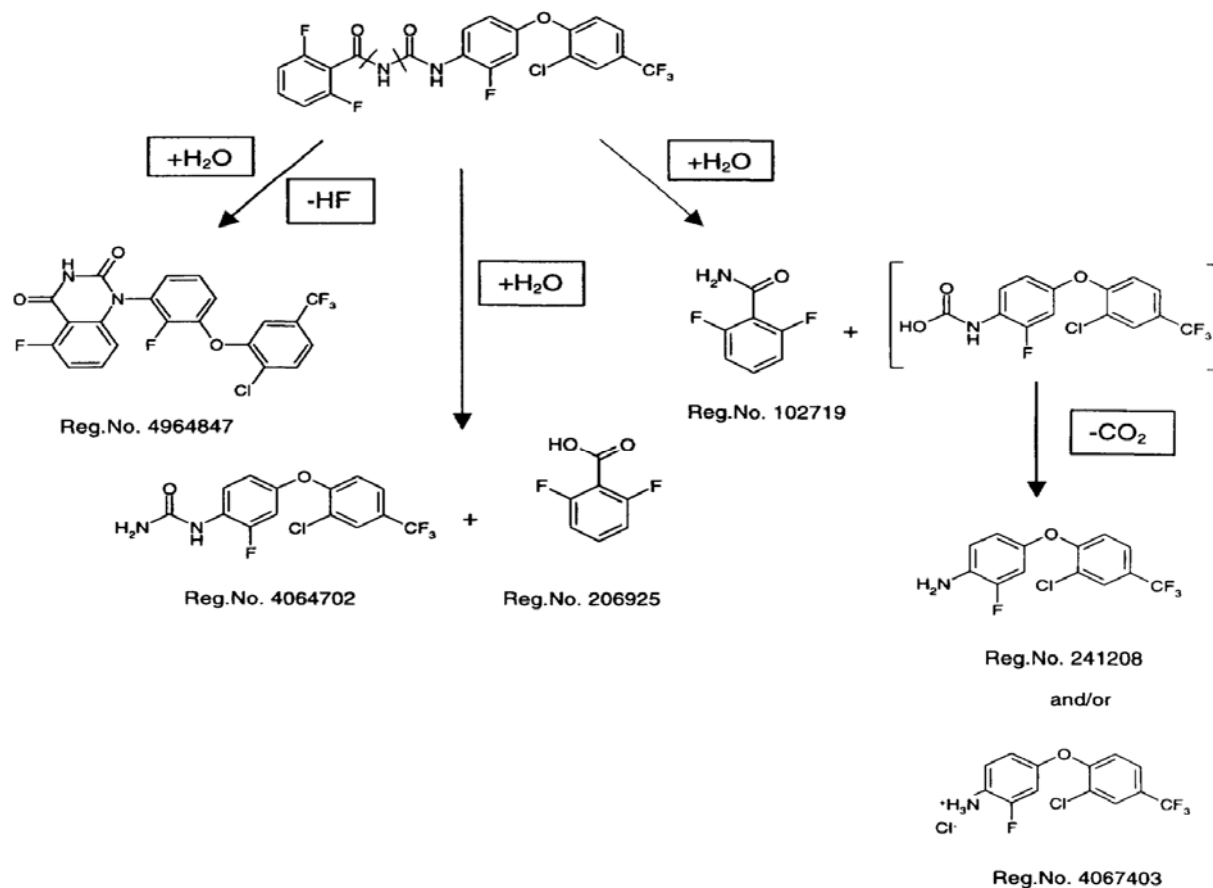


Figure 1 Pathway of hydrolysis of flufenoxuron

Residues after processing

The fate of flufenoxuron during processing of raw agricultural commodity (RAC) was investigated in tomatoes and tea using commercial processing procedures. As a measure of the transfer of residues into processed products, a processing factor (PF) was used, defined as:

$$PF = \frac{\text{Total residue in processed product (mg/kg)}}{\text{Total residue in raw agricultural commodity (mg/kg)}}$$

If residues in the RAC were below the LOQ, no processing factor could be derived. In case of residues below the LOQ, but above the LOD in the processed product, the numeric value of the LOQ was used for the calculation. If residues in the processed product were below the LOD, the numeric value of the LOQ was used for the calculation but the PF was expressed as “less than” (e.g. < 0.5).

A summary of all processing factors for flufenoxuron relevant for the estimation of maximum residue levels of the dietary intake is given in Table 41.

Tomato

Study 1

During the 2002 growing season, four field trials were conducted in field tomatoes in different representative growing areas in southern France and Spain (Smalley R., 2003a; 2003/1004346). The treatment program consisted of three foliar applications of a 100 g/L DC formulation of flufenoxuron at a rate of 0.62 kg ai/ha/application. The tomatoes were harvested 6–7 days following the last application, washed and processed according to commercial practices into pomace, juice, sauce and canned tomatoes.

All samples were analysed for flufenoxuron according to method RLA 12675. The limit of quantitation (LOQ) was 0.05 mg/kg.

Samples of processed commodities were stored for up to 600 days prior to analysis of flufenoxuron which is within the demonstrated storage interval for the parent compound in high oil, high acid and high water content matrices.

In the following table, the residues found in processed products are summarised.

Table 41 Residues of flufenoxuron in processed tomato commodities and calculation of processing factors

Location, year, reference (variety)	No.	kg ai/ha	Sample	DAT (days)	Flufenoxuron									
					Residues (mg/kg)				PF ^a				Median PF	
S. France and Spain, 2002, 2003/1004346 (Avalon, Ercol�, Perfect Peel, Select Peel)	3	0.62	Tomato	6–7	0.26	1.23	0.41	0.74	–				–	
			Tomato before processing		0.14	0.72	0.25	0.51	0.54	0.59	0.61	0.69	0.60	
			Washed tomatoes		0.23	0.44	0.29	0.42	0.88	0.36	0.71	0.57	0.63	
			Wash water		< 0.05	0.46	n.a.	0.07	0.19	0.37	n.a. ^b	0.09	0.19	
			Wet pomace		0.43	0.89	0.43	0.96	1.65	0.72	1.05	1.30	1.18	
			Raw juice		0.09	0.49	0.15	0.33	0.35	0.40	0.37	0.45	0.39	
			Tomato juice		0.07	0.36	0.13	0.22	0.27	0.29	0.32	0.30	0.30	
			Waste puree		1.75	3.53	1.86	2.21	6.73	2.87	4.54	2.99	3.76	
			Tomato puree		0.20	0.59	0.23	0.65	0.77	0.48	0.56	0.88	0.67	
			Blanching water		< 0.05	< 0.05	< 0.05	< 0.05	0.19	0.04	0.12	0.07	0.10	
			Peels		1.54	6.61	1.92	3.52	5.92	5.37	4.68	4.76	5.02	
			Peeled tomatoes		< 0.05	< 0.05	< 0.05	< 0.05	0.19	0.04	0.12	0.07	0.10	
			Canned tomatoes ^c		< 0.05	0.17	0.05	0.08	0.19	0.14	0.12	0.11	0.13	

^a Processing factor (PF) = residue in processed fraction / residue in RAC

^b n.a. = sample was not analysed

^c Raw juice is also added to peeled tomatoes during the process of canning

Study 2

A processing study was conducted in Southern France to determine the residue levels of flufenoxuron and the metabolites Reg. No. 4064702, Reg. No. 102719, Reg. No. 206925, Reg. No. 241208, Reg. No. 4064703 and Reg. No. 4964847 in tomato RAC and processed fractions including canned tomatoes, tomato puree and tomato juice (Ertus, C., 2013; ANADIAG).

The treatment program consisted of a single foliar application of a 100 g/L formulation of flufenoxuron at a rate of 0.5 kg ai/ha made 7 days prior to harvest at the growth stage BBCH 89.

The tomatoes were washed and processed according to the following commercial practices:

For juice production, approximately 8 kg of tomatoes were roughly cut and blended. The mixture obtained was filtered through a fine sieve to separate the juice from peels and seeds. The Brix degree and the pH of the raw juice were determined and the raw juice was heated to 95 °C for 5 minutes. Subsamples of pasteurized juice were stored frozen until analysis.

For production of tomato puree, a juice sample was concentrated under vacuum and gentle heating to 60 °C until reaching a Brix degree of 12. The obtained puree was pasteurized by heating to 95 °C for 5 minutes in a can.

For canning, approximately 2 kg of tomatoes were submerged for about 60 seconds in boiling water and transferred into cold water for 20 seconds to crack the peel. After peeling, subsamples were transferred into glass preserving jars, which were filled up with water, closed and submitted to sterilization. After cooling, canned tomatoes were blended and subsamples were stored frozen until analysis.

All samples were analysed for flufenoxuron and the metabolites according to a method adapted from 'Flufenoxuron: Development and Validation of an Analytical Method for Determination and Confirmation of BAS 307I (Flufenoxuron) and its Degradates in Grape—PTRL Europe Study No.P/B1206G'. Briefly, residues were extracted using a mixture of methanol, water and HCl. The centrifuged extract was analysed by LC/MS/MS. The limit of quantification (LOQ) was reported to be 0.01 mg/kg.

Samples of processed commodities were stored for up to 33 days prior to analysis of flufenoxuron and all metabolites which is within the demonstrated storage interval for the parent compound in high oil, high acid and high water content matrices.

In the following table, the residues found in processed products are summarised.

Table 42 Residues of Flufenoxuron and associated metabolites in processed tomato commodities and calculation of processing factors

Location, year, reference (variety)	N o.	kg ai/ha	Sample	DAT(days)	Residues (mg/kg)							PF ^a
					Flufenoxuron	Reg. No. 40647 02	Reg. No. 1027 19	Reg. No. 2069 25	Reg. No. 2412 08	Reg. No. 4647 03	Reg. No. 49648 47	
S. France, 2012, ANADI AG (Rio Grande)	3	0.5	Tomato	6-7	0.28	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	–
			Fresh tomatoes		0.18	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.64
			Canned tomatoes		< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.04
			Tomato puree		0.08	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.28
			Tomato juice		0.04	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.14

^a Only processing factors for flufenoxuron were reported as residues of all other metabolites were not detected above the limit of quantitation (0.01 mg/kg).

Tea

Two residue trials were conducted in 1990 in Kyoto and Kagoshima, Japan (Class, T., 2007a). Flufenoxuron was applied either once or twice at a rate of 0.0025 kg ai/hL and leaves were harvested 7–14 days following application.

An infusion was prepared from the treated dried green tea leaves by adding 360 mL boiling water to 6 g tea and leaving to stand for five minutes.

Dried tea (green) leaves and tea infusion were analysed using the HPLC-UV method titled Pesticide Residue Analysis for Crop (Shell Corporation, 1996). In summary, the method involves extracting flufenoxuron residues with acetone, in the presence of zinc acetate. The filtrates were extracted with ethyl acetate and subjected to hexane/acetonitrile partition after which the acetonitrile phase is purified using a florisil cartridge prior to HPLC–UV analysis. The limit of quantitation for both tea leaves and tea infusion was reported as 0.02 mg/kg, each.

Samples of tea leaves and tea infusion were stored for up to 108 days prior to analysis of flufenoxuron which is within the demonstrated storage interval for parent in high oil, high acid and high water content matrices.

Table 43 Determination of transfer of residues from treated dried tea (green) leaves to tea infusion

	Treatment times	DAT ^a	Residues in dried tea (green) leaves (mg/kg)		Residues in tea infusion (mg/kg)		% transfer from dried tea (green) leaves to tea infusion
			Found	Mean	Found	Mean	
Kyoto	1	7	6.66, 6.26	6.46	0.04, 0.04	0.04	0.62
	1	14	5.57, 5.18	5.36	0.03, 0.03	0.03	0.56
	2	7	7.98, 7.89	7.94	0.06, 0.05	0.06	0.76
	2	14	6.33, 5.96	6.14	0.04, 0.03	0.04	0.65
Kagoshima	1	7	7.75, 7.57	7.65	0.05, 0.05	0.05	0.65
	1	14	4.09, 4.08	4.08	0.03, 0.03	0.03	0.74
	2	7	7.24, 7.19	7.22	0.05, 0.05	0.05	0.69
	2	14	3.63, 3.53	3.58	0.02, 0.02	0.02	0.56
Median transfer							0.65

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

For the estimation of residues of flufenoxuron in animal matrices lactating cow and laying hen feeding studies were submitted to the Meeting.

Lactating dairy cattle

Residues of flufenoxuron in lactating cows were investigated by Gilland Pack (1993). Flufenoxuron was administered orally to fifteen lactating Friesian dairy cows (three cows/control and treatment group and a depuration group consisting of three cows), between 4–7 years of age and in the weight range of 466–602 kg, for 90 consecutive days. Based on a feeding rate of approximately 20 kg/day, dose levels were 1.75, 5.25 and 17.5 mg/kg feed corresponding to 0.07, 0.21 and 0.7 mg/kg bw/day. Animals were dosed twice daily at half of the daily rate at each milking. The average daily milk production ranged from 13–21 kg starting 14 days prior to dosing up to the last day of dosing. The fat content of milk samples taken on days 7, 28, 56 and 90 of the study ranged from 2.5–4.3%.

Milk was collected twice daily. At the end of the dosing period, two composite milk samples were taken for processing to pasteurized milk, cream, skimmed milk and acid whey.

Three animals from the highest dose level group and one control animal were monitored for flufenoxuron residues over a 40-day depuration phase. Milk from these animals was sampled during the depuration period.

Approximately one day after the last day of dosing, the animals, with the exception of the cows of the depuration group, were sacrificed and liver, kidney, composite muscle (pectoralis/adductor muscle of thigh) and perirenal fat (perirenal/omental) were collected for analysis.

Analysis of the samples of milk and milk products was performed using the Analytical Method SAMS 486-1 with minor modifications. The limit of quantitation of this method for flufenoxuron in milk and milk products was reported to be 0.001 mg/L based on acceptable concurrent recoveries. Analysis of the bovine tissues was carried out in duplicate according to a slightly modified version of the Analytical Method SAMS 457-2. The lowest limit of method validation for flufenoxuron in animal tissues is 0.10 mg/kg for muscle, liver and kidney and 0.30 mg/kg for fat based on acceptable concurrent recoveries.

Residues of flufenoxuron in milk are presented in the following table:

Table 44 Residues of Flufenoxuron in milk after administration of Flufenoxuron at 1.75, 5.25 or 17.5 mg/kg feed/day

Study Day	Mean residues for flufenoxuron (mg/L)			
	Group A ^a control	Group B ^a 1.75 mg/kg diet	Group C ^a 5.25 mg/kg diet	Group D ^b 17.5 mg/kg diet
-3	< 0.001	< 0.001	< 0.001	< 0.001
-1	< 0.001	< 0.001	< 0.001	< 0.001
1	< 0.001	< 0.001	< 0.001	0.003 (0.003, 0.002, 0.003, 0.003, 0.005, 0.002)
2	< 0.001	0.02 (0.025, 0.023, 0.025)	0.07 (0.059, 0.077, 0.068)	0.21 (0.099, 0.12, 0.25, 0.35, 0.29, 0.13)
4	< 0.001	0.08 (0.091, 0.075, 0.084)	0.21 (0.27, 0.24, 0.13)	0.71 (0.73, 0.66, 0.75, 0.87, 0.83, 0.41)
7	< 0.001	0.13 (0.11, 0.12, 0.15)	0.35 (0.45, 0.47, 0.18)	1.70 (1.6, 1.4, 1.7, 1.5, 1.9, 1.6)
10	0.001 (< 0.001, < 0.001, 0.002)	0.16 (0.14, 0.083, 0.27)	0.64 (0.72, 0.79, 0.40)	2.20 (2.1, 1.9, 2.5, 1.9, 2.6, 2.1)
14	0.001 (< 0.001, 0.001, 0.002)	0.27 (0.18, 0.33, 0.31)	0.70 (0.54, 0.92, 0.54)	2.63 (2.6, 2.6, 3.3, 2.9, 2.5, 1.9)
21	0.001 (< 0.001, 0.001, 0.001)	0.34 (0.32, 0.30, 0.41)	0.89 (0.81, 1.3, 0.57)	3.53 (4.6, 4.1, 4.7, 2.1, 3.1, 2.6)
28	0.001 (< 0.001, < 0.001, 0.001)	0.437 (0.39, 0.41, 0.51)	1.33 (1.4, 1.6, 1.0)	3.57 (4.3, 3.1, 3.7, 3.4, 4.0, 2.9)
35	0.0013 (< 0.001, < 0.001, 0.003)	0.460 (0.40, 0.49, 0.49)	1.37 (1.4, 1.6, 1.1)	3.72 (4.5, 2.6, 4.2, 3.9, 4.4, 2.7)
42	0.025 (0.002, 0.002, 0.07)	0.420 (0.40, 0.42, 0.44)	1.20 (1.1, 1.4, 1.1)	3.98 (4.4, 4.0, 4.4, 3.4, 3.8, 3.9)
56	0.003 (< 0.001, 0.002, 0.007)	0.493 (0.47, 0.51, 0.50)	1.57 (1.6, 1.8, 1.3)	5.02 (5.6, 4.6, 5.0, 5.1, 4.9, 5.0)
70	0.001 (< 0.001, < 0.001, 0.001)	0.58 (0.62, 0.56, 0.57)	1.80 (2.0, 1.8, 1.8)	5.45 (5.1, 5.8, 5.3, 5.8, 5.3, 5.4)
86	0.001 (0.001, 0.001, < 0.001)	0.64 (0.60, 0.75, 0.56)	1.53 (1.5, 1.6, 1.5)	5.47 (96.2, 4.7, 5.0, 6.2, 4.8, 5.9)
90	0.001 (0.002, 0.001, < 0.001)	0.64 (0.77, 0.64, 0.51)	1.90 (2.2, 2.0, 1.5)	5.53 (6.5, 5.6, 5.4, 5.1, 4.4, 6.2)
Depuration Phase				
91	0.001 (0.001, 0.001, < 0.001)	—	—	5.13 (4.1, 4.2, 5.6, 5.4, 4.5, 6.9)
92	< 0.001	—	—	4.63 (4.7, 4.0, 5.2)
94	< 0.001	—	—	4.67 (3.9, 3.7, 6.4)
97	< 0.001	—	—	3.27 (3.2, 2.1, 4.5)

Study Day	Mean residues for flufenoxuron (mg/L)			
	Group A ^a control	Group B ^a 1.75 mg/kg diet	Group C ^a 5.25 mg/kg diet	Group D ^b 17.5 mg/kg diet
101	0.002	–	–	2.60 (2.7, 1.7, 3.4)
106	< 0.001	–	–	2.07 (2.1, 1.1, 3.0)
112	< 0.001	–	–	1.43 (1.7, 0.59, 2.0)
118	< 0.001	–	–	1.20 (1.3, 0.69, 1.7)
124	< 0.001	–	–	0.95 (1.1, 0.45, 1.3)
130	< 0.001	–	–	0.92 (8.84, 0.61, 1.3)

^a Mean values of three animals

^b Mean values of six animals, three animals during depuration phase

Pasteurised milk, cream, skimmed milk and acid whey were prepared from pooled control milk (group A) and from pooled milk from the high-dose group (D) at the end of the 90 day dosing period. The mean results of the analyses of those milk products for both treatment groups are summarised in Table 45.

Table 45 Mean residues of Flufenoxuron in milk products after 90 days of dosing.

Product	Mean residues of flufenoxuron in milk products [mg/L]	
	Group A (Control)	Group D (17.5 mg/kg diet)
Raw Milk	0.001	5.5
Pasteurized Milk	0.012	6.4
Cream	0.007	28
Skimmed Milk	< 0.001	0.18
Acid Whey	< 0.001	0.06

Analysis of milk obtained from the high dose group showed that pasteurization had no effect on the levels of flufenoxuron in milk. Residues in cream were concentrated by a factor of 1.5. In skimmed milk and acid whey, residues were reduced compared to the raw milk.

The mean residue levels and individual sample residues in tissues are summarized in Table 46 for each treatment group.

Table 46 Residues of Flufenoxuron in bovine tissues collected 90 days following the last administered dose

Tissue type	Residues of Flufenoxuron in tissues [mg/kg]				
	Group A ^a control	Group B ^b 1.75 mg/kg diet	Group C ^b 5.25 mg/kg diet	Group D ^b 17.5 mg/kg diet	Group D ^b day 40 depuration
Muscle	< 0.03 (< 0.03, < 0.03)	0.14 [0.08, 0.20 (0.14) 0.12, 0.08 (0.10) 0.28, 0.08 (0.18)]	0.66 [0.67, 0.49 (0.59) 0.24, 0.38 (0.31) 1.2, 0.91 (1.1)]	1.63 [1.7, 1.7 (1.7) 1.8, 1.9 (1.9) 1.4, 1.3 (1.4)]	0.25 [0.28, 0.39 (0.34) 0.03, 0.06 (0.04) 0.27, 0.48 (0.37)]
Liver	< 0.03 (< 0.03, < 0.03)	0.74 [0.70, 0.83 (0.77) 0.83, 0.79 (0.81) 0.68, 0.64 (0.66)]	2.17 [2.2, 2.3 (2.3) 1.8, 2.1 (2.0) 2.2, 2.3 (2.3)]	8.70 [9.0, 11.0 (9.8) 7.5, 7.6 (7.6) 8.3, 9.3 (8.8)]	0.92 [1.0, 1.4 (1.2) 0.23, 0.28 (0.25) 1.0, 1.6 (1.3)]

Tissue type	Residues of Flufenoxuron in tissues [mg/kg]				
	Group A ^a control	Group B ^b 1.75 mg/kg diet	Group C ^b 5.25 mg/kg diet	Group D ^b 17.5 mg/kg diet	Group D ^b day 40 depuration
Kidney	< 0.03 (< 0.03, < 0.03)	0.34 [0.40, 0.24 (0.32) 0.43, 0.44 (0.44) 0.31, 0.23 (0.27)]	1.60 [(1.8, 1.1 (1.4) 1.1, 1.0 (1.1) 2.7, 2.1 (2.4)]	4.30 [5.4, 3.0 (4.2) 7.2, 4.0 (5.6) 3.2, 2.9 (3.1)]	0.71 [1.5, 1.0 (1.3) 0.19, 0.14 (0.17) 0.92, 0.40 (0.66)]
Subcutaneous Fat	< 0.03 (< 0.03, < 0.03)	0.84 [1.1, 2.9 (2.0) 0.37, 0.42 (0.39) 0.16, 0.12 (0.14)]	9.27 [13, 13 (13) 3.6, 4.0 (3.8) 7.4, 15 (11)]	8.70 [5.1, 9.9 (7.5) 18, 11 (15) 4.1, 3.1 (3.6)]	3.467 [6.9, 4.8 (5.9) 0.77, 0.42 (0.60) 2.0, 5.8 (3.9)]
Peritoneal Fat	0.04 (0.04, 0.04)	2.31 [3.4, 6.9 (5.2) 1.3, 0.86 (1.1) 0.69, 0.56 (0.62)]	17.27 [22, 20 (2.1) 6.4, 7.2, (6.8) 25, 23 (24)]	29.33 [44, 47 (45) 28, 27 (28) 14, 16 (15)]	7.63 [12, 12 (12) 2.6, 2.2, (2.4) 9.2, 7.7 (8.5)]

^a Only one control animal was sacrificed at the end of the 90-day dosing period

^b Mean of three animals; for each animal two separate analyses were conducted for each tissue

Laying hen

Seventy five white female Leghorn laying hens (three subgroups per control and treatment group, five hens per subgroup; the depuration group consisted of three subgroups of five hens/subgroup), aged 8–9 months and weighing 1303–1896 g, were dosed orally (gavage) once daily with flufenoxuron for 50 consecutive days at 1, 3 and 10 mg/kg feed, based on a daily consumption of 150 g/bird/day, equivalent to 0.15, 0.45 and 1.5 mg/kg bw/day. The average lay efficiency was 0.5 egg/hen/day.

Eggs were collected daily and separated into yolks and whites then pooled by subgroup (of five hens), resulting in three replicate daily samples per treatment group.

Fifteen animals from the top dose group and ten animals from the control group were monitored for flufenoxuron residues over a 40-day depuration phase. Eggs from these animals were sampled during the depuration period.

One day after the last day of dosing (day 40), the birds, with the exception of those of the depuration group, were sacrificed and samples of skin, muscle (breast, leg and thigh pooled), fat and liver (total organs) were taken separately for analysis and subdivided into two subsamples per subgroup.

The egg and tissue samples were analysed for residues of flufenoxuron using Analytical Method SAMS 492-1 and Analytical Method SAMS 457-2, respectively. The limit of quantitation for flufenoxuron is 0.25 mg/kg in egg yolk and 0.05 mg/kg in egg white and tissues based on acceptable concurrent recoveries. The liver samples were also analysed for the metabolite Reg. No. 4064702 using the HPLC/UV Analytical Method 01791.PCC with a limit of quantitation 0.03 mg/kg.

Egg white and yolk samples were analysed separately. Unless otherwise identified, in the majority of cases, no residues of flufenoxuron in egg white exceeded 0.05 mg/kg. The mean residue levels and range of residues in egg yolks are summarized in Table 47.

Table 47 Residues of Flufenoxuron in egg yolks.

Study Day	Mean (range) residues for flufenoxuron in egg yolks (mg/kg)			
	Group A (control)	Group B (1 mg/kg diet)	Group C (3 mg/kg diet)	Group D (10 mg/kg diet)
–3	< 0.25	< 0.25	< 0.25	< 0.25
–1	< 0.25	< 0.25	< 0.25	< 0.25
1	< 0.25	< 0.25	< 0.25	< 0.25
2	< 0.25	< 0.25	0.28(< 0.25–0.30)	0.40 (0.27–0.47)
4	0.27	< 0.25	0.63 (0.44–1.0)	1.87 (1.25–2.30)

Study Day	Mean (range) residues for flufenoxuron in egg yolks (mg/kg)			
	Group A (control)	Group B (1 mg/kg diet)	Group C (3 mg/kg diet)	Group D (10 mg/kg diet)
7	< 0.25	0.54 (0.45–0.70)	2.07 (1.74–2.30)	5.35 (4.07–6.99)
14	< 0.25	1.32 (1.02–1.70) ^a	4.02 (3.18–4.83)	10.23 (7.82–12.61)
21	< 0.25	2.08 (1.65–2.66)	4.92 (3.34–5.76)	15.43 (12.65–20.80)
28	0.47 (< 0.25–0.88)	2.14 (1.78–2.36)	4.70 (4.53–5.58)	17.18 (13.05–20.22)
35	0.49 (< 0.25–0.89)	2.26 (1.37–3.19)	6.22 (5.45–7.37)	19.56 (15.39–26.42)
42	0.62 (< 0.25–1.64)	2.68 (2.46–2.84)	6.06 (4.11–6.96)	25.19 (23.17–28.02)
50	0.25 (< 0.25–0.27)	3.09 (2.57–3.81)	8.01 (7.23–8.58)	28.03 (22.62–31.78)
Depuration phase				
51	0.34 (< 0.25–0.43) ^b	–	–	32.53 (24.52–39.55) ^c
52	< 0.25 ^b	–	–	30.74 (17.31–40.76) ^c
54	< 0.25 ^b	–	–	37.73 (27.21–46.64) ^c
57	< 0.25 ^b	–	–	18.45 (14.94–20.78) ^c
61	0.35 (< 0.25–0.45) ^b	–	–	13.54 (6.74–19.24) ^c
66	< 0.25 ^b	–	–	10.48 (9.48–11.54) ^c
72	< 0.25 ^b	–	–	9.46 (5.34–11.66) ^c
78	< 0.25 ^b	–	–	4.57 (3.01–5.67) ^c
84	< 0.25 ^b	–	–	2.73 (1.90–3.59) ^c
90	< 0.25 ^b	–	–	2.27 (2.07–2.47) ^c

^a The sample of Group B collected on Day 14 was the only egg sample in which Flufenoxuron was also detected in egg white (group mean residue of 0.13 mg/kg, but detected only in one out of three subgroups (subgroup B2)); in each other egg white sample analysed (Day –3 to Day 50), the Flufenoxuron concentration was below 0.05 mg/kg

^b During the depuration period, only the eggs of one control subgroup (5 hens) were analysed

^c During the depuration period, only the eggs of 15 hens at the highest dose level were analysed

The mean and range of residue concentrations of flufenoxuron in hen tissues are listed in Table 48.

Table 48 Residues of Flufenoxuron in tissues

Tissue type	Mean (range) residues of Flufenoxuron in tissues [mg/kg]				
	Group A	Group B (1 mg/kg diet)	Group C (3 mg/kg diet)	Group D (10 mg/kg diet)	Group D Day 40 depuration
Liver	0.09	0.49 (0.31–0.59)	2.30 (0.17–3.89)	2.94 (1.20–4.28)	0.99 (0.48–1.85)
Muscle	0.63 (0.42–0.85)	0.19 (0.16–0.25)	0.69(0.52–0.85)	2.29 (1.92–2.50)	0.33 (0.15–0.53)
Skin	< 0.05	2.02 (1.56–2.67)	6.39 (5.90–7.23)	21.58 (13.24–26.21)	2.94(1.83–3.84)
Fat	0.27 (0.25–0.29)	5.57 (5.49–5.76)	16.9 (15.14–19.22)	69.4 (59.04–77.40)	13.7 (7.95–19.56)

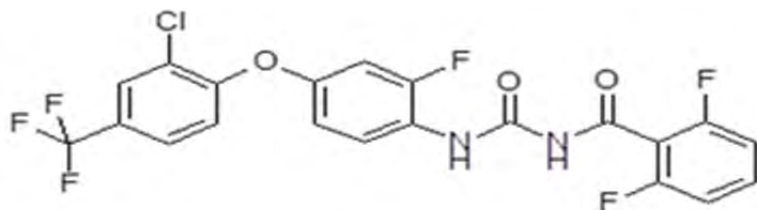
Table 49 Residues of Reg. No. 4064702 in liver

Mean (range) residues of Reg. No. 4064702 in Liver [mg/kg]				
Group A (Control)	Group B (1 mg/kg diet)	Group C (3 mg/kg diet)	Group D (10 mg/kg diet)	Group D Day 40 depuration
< 0.03	0.04 (< 0.02–0.04)	0.18 (0.054–0.37)	0.20 (0.10–0.36)	0.03 (0.028–0.036)

APPRAISAL

Flufenoxuron is a benzylurea insect growth regulator used to kill mites and insects, through interference with chitin production during cuticle development in mite and insect juvenile stages, on various orchard crops, fruiting vegetables and tea. It was considered for the first time by the 2014 JMPR for toxicology and residues.

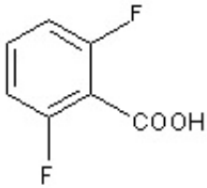
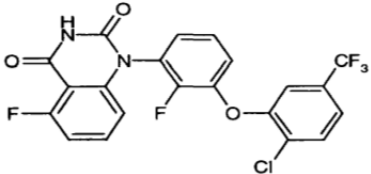
The Meeting received information on physical chemical properties, livestock and plant metabolism, environmental fate, analytical methods, storage stability, supervised residue trials, use patterns, processing and livestock feeding.



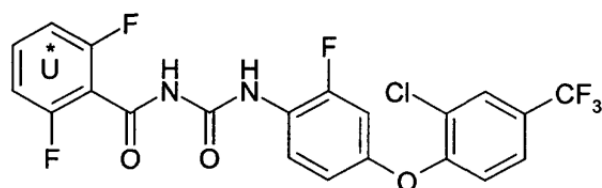
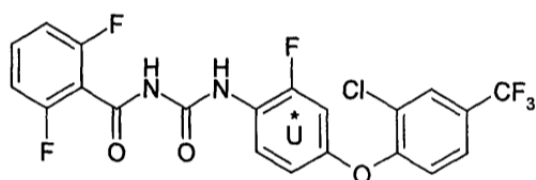
The IUPAC name of flufenoxuron is N-{4-[2-chloro-4-(trifluoromethyl)phenoxy]-2-fluorophenyl}-N'-(2,6-difluorobenzoyl)urea and the CA name is N-[[[4-[2-chloro-4-(trifluoromethyl)phenoxy]-2-fluorophenyl]amino]carbonyl]-2,6-difluorobenzamide.

Common chemical names, code names and structures of the parent and metabolites are captured below:

Code	Structure	Occurrence
Flufenoxuron WL 115110		Rat Lactating goat Laying hen Grape Apple Tomato Chinese cabbage Soil Hydrolysis study
Reg. No. 4064702		Rat Laying hen Soil Hydrolysis study
Reg. No. 241208		Rat Laying hen Hydrolysis study
Reg. No. 4064703 (chloride salt of Reg. No. 241208)		Hydrolysis study
Reg. No. 102719		Rat Hydrolysis study

Code	Structure	Occurrence
Reg. No. 206925		Rat Hydrolysis study
Reg. No. 4964847		Hydrolysis study

Flufenoxuron uniformly labelled in either the fluoroaniline or difluoroamide rings was used in the metabolism and environmental fate studies.



Fluoroaniline U-¹⁴C

Difluoroamide U-¹⁴C

Animal metabolism

Information was available on the metabolism of flufenoxuron in laboratory animals, lactating goats and laying hens.

Metabolism studies in rats demonstrated that unchanged flufenoxuron accounted for the majority of the total applied radioactivity (TAR) in faeces, with minor metabolites (less than 1% of the TAR) identified as 2-amino-5-(2-chloro- α,α,α -trifluoro-p-tolyoxy)-3-fluorophenol (Reg. No. 4110959), Reg. No. 4064702, Reg. No 241208, Reg. No 102719 and Reg. No. 206925. For organs and tissues, parent flufenoxuron was the main component observed.

In the lactating goat metabolism study, one goat received four daily doses of 2-fluoroaniline-[U-¹⁴C]-ring-labelled flufenoxuron at a rate equivalent to 10 ppm in the diet (10 mg/day). The animal was sacrificed 24 hours after administration of the last dose. While the majority of the radioactivity was excreted via the faeces (18% of the TAR) and urine (2.5% of the TAR), milk and tissues accounted for $\leq 10\%$ of the TAR. Total recovered radioactivity was low (33%). No explanation for the low recovery was evident.

The total radioactive residues (TRRs) were highest in fat (1.6 mg eq/kg), followed by liver (0.37 mg eq/kg), kidney (0.13 mg eq/kg) and muscle (0.076 to 0.1 mg eq/kg). Milk residues peaked on day 4 (average of 0.27 mg eq/L) with the highest concentrations of radioactivity detected in the cream fraction (accounting for 82–93% of the TRR in whole milk) and the lowest found in whey (1.3–5.7% of the TRR). Following solvent extraction, residue extractabilities were 66–100%. In milk

and all tissues sampled, the flufenoxuron molecule remained intact with no other metabolites being detected.

Two studies on metabolism in laying hens were available. In the first study, the laying hens received 14 daily doses of flufenoxuron, uniformly labelled in the difluoroamide or fluoroaniline rings, at 13–14 ppm in the feed. The animals were sacrificed approximately 23 h after the last dose. Excreta accounted for 72–78% of the TAR. No plateau was reached in eggs during the dosing period (14 days); however, the radioactivity in eggs and tissues amounted to 1.0–1.3% and 2.6–3.6% of the TAR, respectively. Among all the tissues analysed, radioactive residues were highest in fat (5.0–5.3 mg eq/kg) followed by liver (0.6–1.1 mg eq/kg) and muscle (0.3–0.4 mg eq/kg). The total recovery of radioactivity was 82% and 77% in the groups administered the difluoroamide- and fluoroaniline-labelled flufenoxuron, respectively.

Solvent extraction released 91–102% and 88–99% of the TRRs for the difluoroamide- and fluoroaniline-label, respectively. While the lowest extractability occurred in the liver of the fluoroaniline-labelled study (88% of the TRR), microwave extraction of the liver post-extraction solid (PES) sample released another 8% of the TRR. For the difluoroamide-label, the parent was the only analyte identified in eggs, muscle, fat and liver ranging from 0.28 mg eq/kg (86.5% of the TRR, muscle) to 4.6 mg eq/kg (91.4%, fat).

For the fluoroaniline-label, the parent compound accounted for the majority of the TRRs (70–91%) in the eggs, muscle, fat and liver. The lowest level of parent was found in muscle (0.30 mg eq/kg) with the highest observed in fat (4.8 mg eq/kg). In eggs and liver, Reg. No. 4064702 was present at 0.10 mg eq/kg and 0.13 mg eq/kg (12.0% and 12.6% of the TRR, respectively) while in muscle and fat, Reg. No. 4064702 was a minor metabolite amounting to 0.02 mg eq/kg and 0.05 mg/kg, respectively (5.5% and 1.0% of the TRR). The formate derivative of Reg. No. 241208 was released from the PES of liver after microwave treatment in the presence of formic acid/acetonitrile. The radioactivity associated with this derivative amounted to 0.04 mg eq/kg (3.3% TRR). The Meeting could not confirm whether the metabolite Reg. No. 241208 is an actual in-vivo metabolite or an artefact formed during microwave treatment.

In the second study, laying hens received seven consecutive daily doses of flufenoxuron uniformly labelled in the fluoroaniline ring at a rate equivalent to 10 ppm in the diet. Hens were sacrificed 22 hours following administration of the last dose. To investigate the depuration behaviour of flufenoxuron, four groups of three laying hens each were sacrificed at 2, 9, 16, and 34 days after the last administration.

On average, 26% of the TAR was excreta-related with eggs, sampled from 0–166 h after the first administration, accounting for 5% of the TAR. At sacrifice, the highest amount of radioactive residues was detected in fat (47% of the TAR), followed by skin (12% of the TAR), muscle (4% of the TAR), liver (2% of the TAR), kidney (0.3% of the TAR), heart and gizzard (combined 0.2% TAR). The recovery of radioactivity amounted to 96% of the TAR.

Solvent extraction (including incubation of liver and kidney samples at 37 °C) released 91–102% of the TRRs). The parent compound was the predominant analyte detected in yolks, liver, kidney, muscle, gizzard and heart while it was the only compound detected in fat and skin. The metabolite Reg. No. 4064702 was detected in yolks, liver, kidney, muscle, gizzard, and heart at 6–22% of the TRR, while the minor metabolite Reg. No. 241208 was only detected in liver and kidney at ≤ 4% of the TRR, the only matrices that were incubated for 16 hours at 37 °C in 0.07M phosphate buffer at pH 7.5 prior to extraction

In the depuration study, radioactivity in egg yolks decreased steadily from a mean of 0.02 mg eq./kg, 2 days after cessation of dosing to 0.006 mg eq/kg on depuration day 34. Similarly, radioactivity in muscle decreased from 0.28 mg eq/kg to 0.06 mg eq/kg, during this same interval. In kidney and liver, the decrease in radioactivity was more prominent from day 16 to day 34 of the depuration phase (kidney; 0.48 mg eq/kg to 0.17 mg eq/kg and liver; 0.89 mg eq/kg to 0.42 mg eq/kg) yet in fat, the decrease in radioactivity occurred most rapidly from day 2 to day 9 (13.18 mg eq/kg to 6.00 mg eq/kg) and from day 16 to day 34 (4.6 mg eq/kg to 1.97 mg eq/kg). These results

demonstrate that radioactive residues are not retained in eggs, organs and tissues after cessation of dosing.

In both laying hen studies, the metabolic pattern was comparable with unchanged flufenoxuron accounting for the majority of the radioactivity, representing $\geq 60\%$ of the TRRs in eggs and tissues. The minor metabolites Reg. No. 4064702 (eggs and tissues) and Reg. No. 241208 (liver and kidney), resulting from the cleavage of the benzoyl urea bond, were also observed to a limited extent ($\leq 12\%$ of the TRRs; except in the kidney where Reg. No. 4064702 represented 22% of the TRRs).

The Meeting concluded that in the lactating goat metabolism study, the parent flufenoxuron remained intact and was the only residue identified in milk and all tissues. In the laying hen metabolism studies, while flufenoxuron was the predominant residue in eggs and tissues, cleavage of the benzoyl urea bond was observed to a limited extent resulting in the formation of the metabolites Reg. No. 4064702 (eggs and tissues) and Reg. No. 241208 (liver and kidney), which were also identified in the rats.

Plant metabolism

The Meeting received metabolism studies for flufenoxuron following foliar applications of either [difluorobenzamide- U - ^{14}C]- or 2-fluoroaniline-[U - ^{14}C]-ring-flufenoxuron to grape, apple, tomato and Chinese cabbage.

Two foliar sprays were made to grape vines, grown outdoor and protected with plastic covers after application, during fruit development; at a rate of 0.04 kg ai/ha/application, with a 40-day retreatment interval, resulting in a total rate of 0.08 kg ai/ha. Immature leaves were collected at 15 DAT (days after last treatment) while mature leaves, stalks and fruit (from grape clusters) were harvested 28–29 DAT. TRRs in leaves declined from 2.3–2.7 mg eq/kg at 15 DAT to 1.4–1.8 mg eq/kg at 29 DAT. TRRs in mature fruit and stalks were 0.012–0.014 mg eq/kg and 0.11–0.16 mg eq/kg, respectively. Solvent extraction released approximately 95–97% of the TRR (0.012–2.6 mg eq/kg) from the grape matrices. Flufenoxuron was the only compound identified in all fruit, leaf and stalk samples (50–97% of the TRR; 0.007–2.2 mg eq/kg). Polar unknowns comprised up to 40–46% of the TRR in mature grape samples (0.005–0.006 mg eq/kg) with unextracted residues in all leaf, fruit and stalk samples accounting for $\leq 5\%$ of the TRR (< 0.11 mg eq/kg).

Ten apple trees, maintained in glasshouses, were sprayed with flufenoxuron, uniformly labelled in the fluoroaniline ring. A single application of the dispersible concentrate was made to trees, during fruit development, at a rate of 0.01 kg ai/hL. Samples of immature fruit were harvested 0 days (4 h post-treatment) and 46 days after treatment (DAT), and mature fruit samples were collected at 99 DAT. TRRs in immature fruit were 2.6 mg eq/kg (0 DAT) and declined to 0.16 mg eq/kg (46 DAT) and 0.06 mg eq/kg (99 DAT). The radioactivity in the combined acetonitrile and hexane surface washes decreased with increasing DAT, from 96% of the TRRs at 0 DAT to 77% of the TRRs at 99 DAT, with a corresponding increase in TRRs in fruit extracts (3.7% TRR at 0 DAT to 23% of the TRRs at 99 DAT), demonstrating limited translocation. The parent flufenoxuron accounted for the majority of the TRRs in surface washes (74–93%; 0.043–2.4 mg eq/kg) and in fruit extracts (3–16% of the TRRs; 0.01–0.08 mg eq/kg).

A single broadcast foliar application of 2-fluoroaniline-[U - ^{14}C]-ring-flufenoxuron, formulated as an emulsifiable concentrate, was made to tomato plants, maintained outdoor, during fruit development at a rate of 0.125 kg ai/ha. Tomato fruit was harvested at 0 and 28 DAT. TRRs in/on tomato fruit declined from 0.38 mg eq/kg on day 0, to 0.2 mg eq/kg by day 28. The total extracted residues (ACN:water surface washes and fruit extracts) from 0 DAT to 28 DAT, accounted for 94–99% of the TRR (0.16–0.38 mg eq/kg), mainly from the surface wash ($\geq 94\%$ of the TRRs). Flufenoxuron was the only identified residue in the mature tomato sample (91% of the TRRs).

2-Fluoroaniline-[U - ^{14}C]-ring-flufenoxuron, formulated as an emulsifiable concentrate, was applied once to Chinese cabbage plants, grown outdoor, during leaf development, as a foliar application, at a rate equivalent to 0.10 kg ai/ha. Cabbage plants were harvested at 0 and 28 DAT.

TRRs in/on cabbage wrapper leaves declined from 6.3 mg eq/kg on day 0 to 0.35 mg eq/kg by day 28. At 0 DAT, the surface wash represented the majority of the extracted residues (84% of the TRRs; 5.3 mg eq/kg) while at the 28 DAT, the leaf extracts accounted for a greater fraction of the extractable radioactivity (76% of the TRRs; 0.27 mg eq/kg). The parent flufenoxuron was the only identified residue in mature cabbage leaves (93% of the TRRs).

The Meeting concluded that the metabolism of flufenoxuron in grape, apple, tomato and Chinese cabbage is consistent among all crops, where parent flufenoxuron remained intact. No other metabolites were identified and no other residues were characterized (other than polar unknowns). The Meeting agreed that the majority of radioactivity remained on the leaves or surface of the fruit, with limited translocation.

Environmental fate in soil

The Meeting received information on aerobic degradation in soil.

In these studies, the fluoroaniline-specific metabolite, Reg. No. 4064702, was the only metabolite identified, reaching a maximum concentration after 30 days of incubation (4.1–8.3% TAR). The predominant residue, flufenoxuron, decreased to 45.8–51.0% TAR in the soil after 119 days, resulting in a calculated DT_{50} for flufenoxuron of 115–122 days. Considering the persistence of flufenoxuron, it is desirable that confined rotational crop and field accumulation studies be submitted.

Methods of residue analysis

The Meeting received analytical methods for the analysis of flufenoxuron in plant and animal commodities. The basic principle for plant commodities employs extraction by homogenisation with dichloromethane, methanol/water/HCl or acetone followed by partitioning with water/cyclohexane. For animal matrices, flufenoxuron residues are extracted by homogenization with various non-polar organic solvents followed by liquid partitioning and/or clean-up by normal-phase or reverse-phase HPLC prior to analysis. Residues of flufenoxuron are measured by HPLC-MS/MS with two specific mass transitions or with HPLC-UV at 254–260 nm. The applicability of the proposed enforcement methods was confirmed in various independent laboratories where parent flufenoxuron was analysed with validated LOQs of 0.05 mg/kg for plant and animal commodities and eggs, and 0.01 mg/kg for milk.

The multiresidue method DFG S-19 was tested, for the analysis of flufenoxuron in animal matrices only, and found to be unsuitable.

A number of scientific papers report the validation of the QuEChERS multi-residue method using GC-MS/MS for flufenoxuron in various plant commodities.

The Meeting concluded that the available enforcement analytical methods are suitable for determining residues of flufenoxuron in plant and animal commodities with LOQs, ranging from 0.01–0.05 mg/kg depending on the matrix.

Stability of residues in stored analytical samples

Based on the storage stability data submitted, the Meeting concluded that no significant dissipation of flufenoxuron residues was observed in cottonseed, orange, grape, and apple after 36 months of storage, in lettuce after 27 months and in watermelon (pulp and peel) after 26 months.

The Meeting agreed that no degradation of flufenoxuron residues was observed in animal matrices stored for up to 53 months of storage, except egg whites, where flufenoxuron residues were determined to be stable for up to 4 months.

Definition of the Residue

In the lactating goat metabolism study, flufenoxuron was the only residue identified in tissues and milk with no other metabolites detected. Similarly, in the laying hen metabolism studies, flufenoxuron

accounted for the majority of the radioactivity in eggs, muscle, fat, liver and kidney (60–104% of the TRRs).

Therefore, the Meeting recommends the residue definition for compliance with MRL for animal commodities as flufenoxuron.

The Log K_{ow} of flufenoxuron is 4. In the goat metabolism study, highest levels of the parent compound were observed in fat and cream, while in the laying hen metabolism studies, the highest concentrations of flufenoxuron were observed in the fat ($\leq 98\%$ of the TRRs). These findings were supported by the livestock feeding studies, where the average ratio for cream/skim milk was ≥ 155 and ≥ 112 for egg yolks/egg whites. Further to this, residues in fat were 24–30-fold higher than those in muscle.

In light of this, the Meeting concluded that the residue is fat soluble.

The metabolite Reg. No. 4064702 was also identified in laying hen muscle, fat, liver, kidney and eggs (1–22% of the TRRs) with the highest levels observed in liver and kidney. The minor metabolite Reg. No. 241208 was also observed but only in liver and kidney (2.5–3.7% of the TRRs) which were the only matrices that were subject to microwave extraction (liver only) or incubation at 37 °C in 0.07M phosphate buffer at pH 7.5 prior to extraction (liver and kidney), and hence considered a potential artefact of the analytical procedure.

The toxicity of the minor metabolite Reg. No. 241208, found in laying hen matrices, was considered to be covered by toxicity studies on flufenoxuron since this metabolite was seen in the rat. The metabolite Reg. No. 4064702, also observed in eggs and tissues of laying hens, and observed in the rat, was determined to be more acutely toxic than the parent flufenoxuron based on the LD₅₀. However, according to the poultry feeding study, residues of this metabolite in liver are not expected to exceed 0.04 mg/kg at the lowest feeding level of 1 ppm. Hence, as there are no poultry feed items derived from the proposed crops, the dietary exposure to this metabolite from poultry matrices is unlikely.

The Meeting recommends the residue for dietary intake for animal commodities as parent only.

The fate of flufenoxuron in plants was investigated following foliar application to tomato, apple, grape and Chinese cabbage. In all plant commodities tested, flufenoxuron was the predominant residue accounting for > 90% of the TRR, with the exception of grape, where flufenoxuron accounted for 50% of the TRR. No other metabolites were identified and no other residues were characterized (other than polar unknowns).

According to the hydrolysis study, simulating typical processing conditions (pH 4, 5 and 6 with 90 °C, 100 °C and 120 °C for 20, 60 and 20 minutes), flufenoxuron was degraded to various metabolites including: Reg. No. 102719 (8–32%), Reg. No. 4064702 (4%) and Reg. No. 4964847 (4–9%). All metabolites, except Reg. No. 4064702 and Reg. No. 4964847 are considered to be covered by toxicity studies on flufenoxuron, since they were seen in the rat. The absorption, distribution, metabolism and excretion studies in rat demonstrated that Reg. No. 4064702 was more acutely toxic than the parent flufenoxuron. Conversely, no toxicity information is available on metabolite Reg. No. 4964847, Reg. No. 4064702. Nevertheless, in the tomato processing study where these metabolites were measured in juice, purée and canned tomatoes, none were detected (< 0.01 mg/kg)

The Meeting recommended the following residue definition for flufenoxuron:

Definition of the residue for compliance with MRL and for estimation of dietary intake for plants and animal commodities: *flufenoxuron*

The Meeting considers the residue fat soluble.

Results of supervised residue trials on crops

The Meeting received supervised residue trials from Brazil, Europe and South Africa where flufenoxuron was applied to oranges, apples, pears, melons and tomatoes and Japanese trials on tea.

Oranges

The critical GAP in Brazil for flufenoxuron on oranges is up to two foliar applications of 0.005 kg ai/hL, with a re-treatment interval of 30 days and a PHI of 15 days. Sixteen supervised field trials were conducted in Greece, Italy, South Africa and Brazil.

Four of the trials were conducted in Brazil according to the critical GAP. Residues in whole oranges at the 15-day PHI were: 0.09, 0.11, 0.13 and 0.16 mg/kg.

Five trials in Brazil were conducted at 2×0.002 – 0.003 kg ai/hL with a PHI of 15 days, representing 0.4 – $0.6 \times$ the critical GAP in Brazil. The residues in whole fruit were 0.03, 0.05, 0.07, 0.08 and 0.10 mg/kg. The Meeting agreed to use the proportionality approach to scale the residues at the 15-day PHI according to an application rate of 0.005 kg ai/hL. The rank order of scaled residues in whole fruit was ($n = 5$): 0.08 (2), 0.13 (2), and 0.17 mg/kg.

When combining all the residue data, residues in whole oranges were 0.08 (2), 0.09, 0.11, 0.13 (3), 0.16 and 0.17 mg/kg and residues in pulp were 0.03, 0.04, 0.05 (3), 0.06, 0.08 (2) and 0.10 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg, and a median residue of 0.13 mg/kg for residues of flufenoxuron in whole oranges. For orange pulp, the Meeting estimated an STMR of 0.05 mg/kg.

Apples

The Brazil critical GAP for flufenoxuron on apples is a single foliar application at 0.01 kg ai/hL and a PHI of 35 days.

Three trials on apples were available from Chile where trees were treated at 0.015–0.02 kg ai/hL with PHIs of 35–36 days, representing 1.5 – $2 \times$ the critical GAP in Brazil. The residues in the fruit were 0.17, 0.43 and 0.52 mg/kg. The Meeting agreed to use the proportionality approach to scale the residues at the PHIs of 35–36 days according to the application rate of 0.01 kg ai/hL. The ranked order of the scaled residues were: 0.08, 0.22 and 0.35 mg/kg ($n=3$).

In two trials conducted in Chile in accordance with Brazilian GAP, flufenoxuron residues were 0.07 and 0.45 mg/kg.

The Meeting concluded that the number of trials available was insufficient to estimate a maximum residue level for residues of flufenoxuron in apples.

Melons

There is no GAP in Brazil for flufenoxuron on melons; therefore, the Meeting could not recommend a maximum residue level.

Tomatoes

There is no GAP in Brazil for flufenoxuron on tomatoes; therefore, the Meeting could not recommend a maximum residue level.

Tea

The critical GAP for flufenoxuron in Japan for tea is up to two foliar applications of 0.025 kg ai/hL at a re-treatment interval of 7–14 days and a PHI of 7 days.

In seven of the eleven trials conducted in Japan and matching the critical GAP, residue levels in tea (green) were: 2.37, 2.48, 3.95, 4.58, 6.02, 6.23 and 11.8 mg/kg.

The Meeting estimated a maximum residue level of 20 mg/kg and a STMR of 4.58 mg/kg for residues of flufenoxuron in tea, green, black (black, fermented and dried).

Fate of residues during processing

Nature of residues

The Meeting received information on the hydrolysis of flufenoxuron uniformly labelled in the fluoroaniline and difluoroamide rings where typical processing conditions were simulated (pH 4,5 and 6 with 90 °C, 100 °C and 120 °C for 20, 60 and 20 minutes).

In duplicate samples of sterile buffer solution flufenoxuron (accounting for 63–93% of the TAR) was seen to hydrolyse to various metabolites, however, none accounted for greater than 10% of the TAR, with the exception of Reg. No. 102719, present at 32% of the TAR, following hydrolysis conditions simulating baking, boiling and brewing procedures.

Level of residues

The Meeting also received information on the fate of flufenoxuron residues during the processing of the raw agricultural commodities like tomato to juice, pomace, puree and canned tomatoes and tea to infusion. While the magnitude of the residues of Reg. No. 102719 in tea infusion was not elucidated, in the tomato processing study, the residues of flufenoxuron metabolites, including Reg. No. 102719, Reg. No. 4064702 and Reg. No. 4964847 in all processed tomato commodities were below the LOQ (0.01 mg/kg). However, in the absence of a critical GAP in Brazil for flufenoxuron on tomatoes, the tomato processing study was not relied upon to derive processing factors, STMR-P values and to estimate maximum residue levels for tomato processed commodities.

The processing factor obtained in the tea processing study and the estimated STMR-P value for the dietary intake calculation is summarized below:

Raw agricultural commodity	STMR, mg/kg	Processed commodity (food)	Processing factor	STMR-P (mg/kg)
Tea (green)	6.02	Infusion	0.0065 (median)	0.04

Residues in animal commodities

Farm animal feeding studies

The Meeting received information on the residue levels arising in animal tissues and milk when dairy cows were fed flufenoxuron for 90 days at levels equivalent of 1.75, 5.25 and 17.5 ppm in the diet. Three animals from the highest dose level group were monitored for flufenoxuron residues over a 40-day depuration phase.

At the lowest dose tested, flufenoxuron residues in milk increased steadily over the 90-day period, from 0.0243 mg/kg on day 2 to 0.64 mg/kg on day 90, however, in the mid and high dose groups, residues seemed to plateau on day 56 (at 1.6 mg/kg) and day 70 (at 5.5 mg/kg), respectively. Analysis of milk obtained from the high dose group showed that pasteurization had no effect on the levels of flufenoxuron in milk. Residues in cream were concentrated by a factor of 1.5. In skimmed milk and acid whey, residues were lower than those of raw milk.

In all tissues tested, except subcutaneous fat, residues of flufenoxuron were dose dependant, increasing with increasing dose. In subcutaneous fat, residues were 0.8 mg/kg, 9.3 mg/kg and 8.7 mg/kg, in the low, mid and high dose groups, respectively.

The residue depuration study demonstrated that flufenoxuron residues in milk decreased slowly within the 40 days of cessation of dosing, from 5.1 mg/kg on day 91 to 0.9 mg/kg on day 130. In tissues, flufenoxuron residues decreased on average by 80% by day 130.

The Meeting also received information on the residues in tissues and eggs of laying hens when dosed with flufenoxuron for 50 days at levels equivalent to 1, 3, 10 ppm in the diet. Fifteen animals from the top dose group were monitored for flufenoxuron residues over a 40-day depuration phase.

At all feeding levels, no residues of flufenoxuron in egg white exceeded 0.05 mg/kg. However, residues in egg yolks did not reach a plateau but rather increased steadily over the 50-day period.

Residues of flufenoxuron in liver, muscle, skin and fat increased with increasing feeding level.

During the depuration study, flufenoxuron residues in egg yolks decreased more rapidly than in cattle milk over the same duration, from 32.5 mg/kg on day 51 to 2.3 mg/kg on day 90.

Farm animal dietary burden

As there is no information on citrus dry pulp, the only potential cattle feed item derived from the proposed crops and there are no poultry feed items, the Meeting did not calculate farm animal dietary burdens.

Therefore, the Meeting estimated maximum residue levels of 0.05* mg/kg for flufenoxuron in meat (from mammals other than marine mammals), edible offal (mammalian), mammalian fat (except milk fats) and 0.01* mg/kg for milks. STMRs and HRs for dietary intake estimation are 0 mg/kg for meat (from mammals other than marine mammals), edible offal (mammalian), mammalian fat (except milk fats) and milks.

The Meeting did not estimate maximum residue levels, STMRs or HRs for poultry matrices.

The residue in animal commodities is considered fat soluble.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI assessment.

Definition of the residue (for compliance with the MRL and for estimation of dietary intake, animal and plant commodities): *flufenoxuron*

The residue is fat soluble.

CCN	Commodity Name	MRL mg/kg	STMR or STMR-P mg/kg	HR or HR-P mg/kg
FC 0004	Oranges, Sweet, Sour	0.4	0.13	–
	Orange pulp	–	0.05	–
DT 1114	Tea, Green, Black (black, fermented and dried)	20	4.58	–
	Tea infusion	–	0.04	–
MO 0105	Edible offal (mammalian)	0.05*	0	–
MM0095	Meat (from mammals other than marine mammals)	0.05*	0	–
MF 0100	Mammalian fats (except milk fats)	0.05*	0	–
ML 0106	Milks	0.01*	0	–

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intake (IEDI) for flufenoxuron was calculated based on the recommendation for STMRs for raw and processed commodities (tea infusion) in combination with consumption data for corresponding food commodities. These results are shown in Annexe 3.

The IEDI of the 17 GEMS/Food cluster diets, based on the estimated STMRs represented 0% of the maximum ADI of 0.04 mg/kg bw, expressed as flufenoxuron. The Meeting concluded that the

long-term intake of flufenoxuron residues from uses considered by the Meeting is unlikely to present a public health concern.

Short-term intake

No ARfD was considered necessary. The Meeting concluded that the short-term intake of flufenoxuron residues from uses considered by the Meeting is unlikely to present a public health concern.

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FLUOPYRAM (243)

The first draft was prepared by Mr David Lunn, Food Safety Authority, Wellington, New Zealand

EXPLANATION

Fluopyram, a pyridylethylamide broad spectrum fungicide was evaluated for the first time by the 2010 JMPR, where an ADI of 0–0.01 mg/kg bw and an ARfD of 0.5 mg/kg bw were established, residue definitions were proposed and maximum residue levels were recommended for a limited number of uses where GAP information was available. New GAP and supporting information were evaluated by the 2012 JMPR and a number of additional maximum residue levels were recommended.

The 2010 JMPR also established residue definitions for fluopyram:

- For plant products (compliance with MRLs and dietary intake assessment): *fluopyram*
- For animal products (compliance with MRLs): *sum of fluopyram and 2-(trifluoromethyl)benzamide, expressed as fluopyram*
- For animal products (dietary intake assessment): *sum of fluopyram, 2-(trifluoromethyl)benzamide and the combined residues N-{(E)-2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethenyl}-2-trifluoromethyl benzamide and N-{(Z)-2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethenyl}-2-trifluoromethyl benzamide, all expressed as fluopyram.*

The 45th Session of the CCPR (2013) listed fluopyram for further evaluation by the 2014 JMPR for additional MRLs and the current Meeting received new GAP information and new supporting residue information from the manufacturer for plum, peach, apricot, raspberry, onion, leek, Brussels sprouts, cabbage, cauliflower, melon, lettuce, asparagus and oilseed rape.

New residue information was also provided for watermelon and currants but because no GAP information was available for these crops, these data were not evaluated by the Meeting.

The Meeting also considered residue information provided to the 2010 JMPR for peach, raspberry, onion, leek, Brussels sprouts, cabbage, cauliflower, melon, lettuce and oil seed rape.

USE PATTERNS

Information on GAP in a number of countries in Europe, North America, Southern Africa, Peru, China and South Korea was provided to the Meeting for foliar applications to a range of fruit, vegetable, nut and oil seed crops. This information is summarised in Table 1.

Table 1 Registered uses of fluopyram-SC formulations (including co-formulations with tebuconazole, trifloxystrobin and triadimenol)

Crop	Country	Application					Max/season		PHI (days)	Remarks:
		method	kg ai/ha	kg ai/hL	water L/ha (min)	interval (days)	no	kg ai/ha		
Stone fruits [Group 3]										
Apricot	Greece	spray	0.1	0.0067	1500	14	3	0.4	3	
Apricot	Greece	spray	0.15	0.01	1500	7–14	2	0.4	3	
Apricot	Italy	spray	0.1	0.0067	1500	14	3	0.3	3	
Apricot	Italy	spray	0.15	0.01	1500	7–14	2	0.3	3	
Apricot	Spain	spray	0.076		500–1500	7	2		7	
Apricot	Spain	spray	0.14		500–1500		1		7	
Apricot	Turkey	spray		0.005			2		14	To full flowering

Crop	Country	Application					Max/season		PHI (days)	Remarks:
		method	kg ai/ha	kg ai/hL	water L/ha (min)	interval (days)	no	kg ai/ha		
Nectarine	Greece	spray	0.1	0.0067	1500	14	3	0.4	3	
Nectarine	Greece	spray	0.15	0.01	1500	7–14	2	0.4	3	
Nectarine	Italy	spray	0.1	0.0067	1500	14	3	0.3	3	
Nectarine	Italy	spray	0.15	0.01	1500	7–14	2	0.3	3	
Nectarine	Spain	spray	0.076		500–1500	7	2		7	
Nectarine	Spain	spray	0.14		500–1500		1		7	
Peach	Greece	spray	0.1	0.0067	1500	14	3	0.4	3	
Peach	Greece	spray	0.15	0.01	1500	7–14	2	0.4	3	
Peach	Italy	spray	0.1	0.0067	1500	14	3	0.3	3	
Peach	Italy	spray	0.15	0.01	1500	7–14	2	0.3	3	
Peach	Spain	spray	0.076		500–1500	7	2		7	
Peach	Spain	spray	0.14		500–1500		1		7	
Peach	Turkey	spray		0.005		10–14			3	
Plum	Greece	spray	0.1	0.0067	1500	14	3		3	
Plum	Switzerland	spray	0.14	0.0088			2		21	
Berries and other small fruits [Group 004]										
Small berries ^a	Namibia	spray	0.25	0.083	300–1000	10–14	2		3	
Small berries ^a	South Africa	spray	0.25		300–1000	10–14	2		3	
Bulb vegetables										
Garlic	Greece	spray	0.2		300–800		1		7	
Garlic	Spain	spray	0.2		300–800		1		7	
Leek	Germany	spray	0.2		200–700		1		21	
Leek	Switzerland	spray	0.2				1		21	
Onion (bulb)	Germany	spray	0.1		200–800	7	2		7	
Onion (bulb)	Greece	spray	0.2		300–800		1		7	
Onion (bulb)	Spain	spray	0.2		300–800		1		7	
Onion (bulb)	Peru	spray	0.12						7	
Brassica vegetables										
Brussels sprouts	Germany	spray	0.18		300–800	14	2		14	
Brussels sprouts	Switzerland	spray	0.18				2		14	
Cabbage, head	Switzerland	spray	0.18				2		14	Head brassicas
Cabbage, head	Germany	spray	0.18		300–800	14	2		14	Head brassicas
Broccoli	Germany	spray	0.18		300–800	14	2		14	Flowerheads
Broccoli	Switzerland	spray	0.18				2		14	Flowerheads
Cauliflower	Germany	spray	0.18		300–800	14	2		14	
Cauliflower	Switzerland	spray	0.18				2		14	
Kohlrabi	Switzerland	spray	0.18				2		14	
Fruiting vegetables, Cucurbits										
Melon	South Korea	spray	0.225	0.0125	1800	10	3		3	
Melon (field)	Mexico	spray	0.14			7	4		7	
Pumpkin	Mexico	spray	0.14			7	4		7	
Squash	Mexico	spray	0.14			7	4		7	
Squash (indoor)	Turkey	spray		0.006		10			3	
Water melon	Mexico	spray	0.14			7	4		7	
Watermelon	Canada	spray	0.25		200	7–14		0.5	0	
Watermelon	USA	drip	0.25			5–10		0.5	7	

Crop	Country	Application					Max/season		PHI (days)	Remarks:
		method	kg ai/ha	kg ai/hL	water L/ha (min)	interval (days)	no	kg ai/ha		
Watermelon	China	spray	0.094				2		7	
Watermelon	South Korea	spray	0.225	0.0125	1800	10	3		14	
Watermelon (field)	USA	spray	0.25			7–14		0.5	0	
Watermelon (indoor)	USA	spray	0.25			7–14		0.5	3	
Leafy vegetables										
Lettuce	Netherlands	spray	0.25			7	2		7	
Lettuce	South Africa	spray	0.25		200–1000	10	2		7	
Lettuce	Switzerland	spray	0.2				2		14	
Lettuce	Namibia	spray	0.25		200–1000	10	2		7	
Rucola	Netherlands	spray	0.25			7	2		7	
Stalk and Stem vegetables										
Asparagus	Greece	spray	0.15		1000	10	2			Pre-senescence
Asparagus	Italy	spray	0.15	0.015	1000	10–14	2			Pre-senescence
Asparagus	Switzerland	spray	0.2				2			Pre-senescence
Oilseeds										
Oil seed rape	France	spray	0.125		200–400		1		56	To BBCH 73
Oil seed rape	Germany	spray	0.125		200–400		1			To BBCH 69
Oil seed rape	Hungary	spray	0.125		200–400		2		56	To BBCH 73
Oil seed rape	UK	spray	0.125			14	2	0.25	56	
Oil seed rape	Ukraine	spray	0.113		200–400		2		30	

^a Blackberry, raspberry, blueberry and gooseberry

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting reviewed supervised field trial information provided to the 2012 JMPR and received new information on supervised field trials involving applications of fluopyram to the following crops.

Crop Group	Commodity	Country	Table No.
Stone fruit	Apricots	Europe	2
	Peaches	Europe	3
	Plums	Europe	4
Berries and other small fruit	Currants	Europe	5
	Raspberries	Europe, USA	6–7
Bulb vegetables	Bulb onions	Europe, USA	8–9
	Leek	Europe	10
	Broccoli	Europe	11–12
Brassicas	Brussels sprouts	Europe	13
	Cabbage, head	Europe	14
	Cauliflower	Europe	15
Fruiting vegetables, cucurbits	Melons (except watermelons)	Europe, Australia, USA	16–19
	Watermelons	Europe	20
Leafy vegetables	Lettuce	Europe, USA	21–23
Stalk and stem vegetables	Asparagus	Europe	24
Oilseeds	Oilseed rape	Europe, USA	25–26
Animal feeds	Oilseed rape forage	Europe	27

The supervised trials were well documented with laboratory and field reports. Laboratory reports included method validation including procedural recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables unless residues in control samples exceeded the LOQ. Residue data are recorded unadjusted for recovery.

When multiple applications were made to a crop, the application rate, spray concentration and spray volume were not always identical from one application to the next. If the variation was small, only the final values for application rate, concentration and spray volume were recorded. For larger variations all values were recorded.

Intervals of freezer storage between sampling and analysis were recorded for all trials and were covered by the conditions of the freezer storage stability studies reviewed by the 2010 JMPR.

Results from replicated field plots are presented as individual values and have not been corrected for concurrent method recoveries unless indicated. When residues were not detected they are shown as below the LOQ (e.g. < 0.01 mg/kg). Residues and application rates have been rounded to two significant digits (or if close to the LOQ, rounded to one significant digit). Average values have been calculated from the residue results prior to rounding and the results from trials conducted according to the maximum GAP and used for the estimation of maximum residue levels have been (underlined).

In addition to the description and details of the field trials and analytical methods, each report includes a summary of the method validation, procedural recoveries, and in most cases, concurrent recoveries in stored frozen samples.

In the trials, where multiple analyses are conducted on a single sample, the average value is reported and where duplicate samples have been analysed, both the individual results and the average value have been reported. Where results from separate plots with distinguishing characteristics such as different formulations, varieties or treatment schedules were reported, results are listed for each plot, and the highest value has been used in calculations of MRLs and STMRs.

Stone fruits

Apricots

Results from supervised trials from Europe on apricots were provided to the Meeting. In the trials, fluopyram (SC formulations) were applied twice at 0.21–0.25 kg ai/ha or three times at 0.125 kg ai/ha. In all trials, the treatments were made at 6–8 day intervals to mature, full-sized trees, as foliar sprays using knapsack atomiser or airblast single-nozzle sprayers to apply 1000–1500 L water/ha.

Samples of at least 1.1 kg fruit or 24 fruit (without stalks) were frozen within 24 hours of sampling and stored at –18 °C for up to 582 days before whole fruit analysis for fluopyram using LC/MS/MS Method 00984. The reported LOQ was 0.01 mg/kg and the mean recovery rates in samples spiked with 0.01–1.0 mg/kg fluopyram ranged from 81–111%.

Table 2 Fluopyram residues in apricots from trials in North and South Europe involving foliar applications of fluopyram (SC formulations)

APRICOT Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
GAP: Greece	2	0.15	0.01	1500		3		7–14 day interval

APRICOT Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
France (S), 2008 St Rémy de Provence (Bergeron)	3	0.125	0.0125	1000	Fruit	–0 0 3 7 10	0.17 0.3 0.22 0.22 0.14	08-2055 08-2055-01
Italy, 2008 Bologna (Bella di Imola)	3	0.125	0.0083	1500	Fruit	–0 0 3 7 10	0.04 0.09 0.07 0.04 0.05	08-2055 08-2055-02
France, 2012 Rognonas (Bergarouge)	2	0.228 (0.083)	0.017	1375 (500)	Fruit	–0 0 3 7 14 21 28	0.18 0.31 0.26 <u>0.33</u> 0.2 0.12 0.14	12-2158 12-2158-01
Spain, 2012 Puigverd de Lleida (Pink-cot)	2	0.249 (0.083)	0.017	1500 (500)	Fruit	–0 0 3 7 14 21 27	0.21 0.8 0.54 <u>0.58</u> 0.48 0.26 0.19	12-2158 12-2158-02
Italy, 2012 Morelli (Aurora)	2	0.208 (0.083)	0.017	1250 (500)	Fruit	–0 0 3 7 14 21 28	0.5 0.98 0.93 <u>0.95</u> 0.48 0.21 0.17	12-2158 12-2158-03
Italy, 2012 Cerignola (Orange rubis)	2	0.249 (0.083)	0.017	1500 (500)	Fruit	–0 0 3 7 14 21 28	0.44 0.73 0.41 <u>0.43</u> 0.37 0.23 0.25	12-2158 12-2158-04
Germany, 2012 Mülheim-Kärlich (Bergeron)	2	0.208 (0.083)	0.017	1250 (500)	Fruit	–0 0 3 7 13 21 27	0.13 0.41 <u>0.38</u> 0.21 0.19 0.16 0.14	12-2046 12-2046-01
Austria, 2012 Scharten (Golden Rich)	2	0.2	0.017	1200	Fruit	–0 0 3 7 14 21	0.07 0.22 0.15 <u>0.16</u> 0.15 0.08	12-2046 12-2046-03
Austria, 2012 Poysbrunn (Bergeron)	2	0.249 (0.083)	0.017	1500 (500)	Fruit	–0 0 3 7	0.2 0.61 <u>0.46</u> 0.42	12-2046 12-2046-04

APRICOT Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Germany, 2013 Mühlheim-Kärlich (Kyoto)	2	0.208 (0.083)	0.017	1250 (500)	Fruit	–0	0.08	13-2005 13-2005-01
						0	0.21	
						3	<u>0.2</u>	
						7	0.11	
						14	0.11	
						21	0.07	

Values in parenthesis: application parameter per meter crop height

Peach

Results from supervised trials from Europe on peaches were provided to the Meeting. Some of the trials conducted in 2006 and 2007 were also provided to the 2010 JPPR. In the trials, fluopyram (SC formulations) were applied twice at 0.21–0.25 kg ai/ha or three times at 0.125 kg ai/ha. In all trials, the treatments were made at 7–10 day intervals to mature, full-sized trees, as foliar sprays using knapsack atomiser or airblast single-nozzle sprayers to apply 900–1500 L water/ha (with lower rates of 300–750 L/ha in three trials).

Samples of at least 1.3 kg fruit or 24 fruit (without stalks) were frozen within 24 hours of sampling and stored at –18 °C for up to 527 days before whole fruit analysis for fluopyram using LC/MS/MS Method 00984. The reported LOQ was 0.01 mg/kg and the mean recovery rates in samples spiked with 0.01–1.0 mg/kg fluopyram ranged from 79–115%.

Table 3 Fluopyram residues in peaches from trials in North and South Europe involving foliar applications of fluopyram (SC formulations)

PEACH Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
GAP: Greece	2	0.15	0.01	1500		3		7–14 day interval
France, 2006 Montfavet (Spring White)	2	0.25	0.025	1000	Fruit	–0	0.18	RA-2591/06 R 2006 0602/8
						0	0.41	
						1	0.34	
						3	<u>0.36</u>	
						7	0.26	
						14	0.22	
Italy, 2006 Bologna (Redhaven)	2	0.25	0.02	1250	Fruit	–0	0.19	RA-2591/06 R 2006 0603/6
						0	0.39	
						1	0.31	
						3	<u>0.28</u>	
						7	0.21	
						14	0.18	
Spain, 2006 Alfarp (Sherman)	2	0.25	0.025	1000	Fruit	–0	0.22	RA-2591/06 R 2006 0600/1
						0	0.45	
						1	0.3	
						3	<u>0.26</u>	
						7	0.19	
						14	0.13	
France, 2006 Montjoire (Redwing)	2	0.25	0.025	1000	Fruit	–0	0.09	RA-2591/06 R 2006 0374/6
						0	0.28	
						1	0.28	
						3	<u>0.28</u>	
						7	0.2	
						14	0.18	

PEACH Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
France, 2010 Montfavet (Spring White)	2	0.249	0.017	1500	Fruit	-0 0 3 7 14 21	0.15 0.31 <u>0.2</u> 0.19 0.15 0.09	10-2098 10-2098-01
Spain, 2010 La Fortesa -Piera (Maycrest)	2	0.208	0.017	1250	Fruit	-0 0 3 7 14 21 28	0.33 0.98 <u>0.73</u> 0.48 0.3 0.26 0.28	10-2098 10-2098-02
Italy, 2010 Canosa di Puglia (Fayette)	2	0.249	0.017	1500	Fruit	-0 0 3 7 14 21	0.18 0.28 0.27 <u>0.31</u> 0.22 0.17	10-2098 10-2098-03
Italy, 2010 San Martino (Flaminia)	2	0.208	0.017	1250	Fruit	-0 0 3 7 14 21	0.17 0.62 0.53 <u>0.63</u> 0.36 0.35	10-2098 10-2098-04
France, 2006 Morancé (Elegan lady)	3	0.124	0.0124	1000	Fruit	-0 0 3 8 10 14	0.1 0.16 0.15 0.13 0.11 0.06	RA-2581/06 0362-06
Italy, 2006 Bologna (Red Haven)	3	0.124	0.01	1250	Fruit	-0 0 3 7 10 14	0.09 0.25 0.2 0.19 0.11 0.09	RA-2581/06 0487-06
Spain, 2006 La Fortesa (Royal Glory)	3	0.124	0.01	1200	Fruit	-0 0 3 7 10 14	0.24 0.35 0.34 0.3 0.19 0.25	RA-2581/06 0488-06
Portugal, 2006 Fazendas de Almeirim (Royal Gene)	3	0.124	0.0166	750	Fruit	-0 0 3 7 10 14	0.08 0.13 0.11 0.06 0.08 0.04	RA-2581/06 0489-06
France, 2007 Montjoire (Redwing)	3	0.125	0.01	1250	Fruit	-0 0 3 7 10 14	0.13 0.28 0.24 0.16 0.11 0.13	RA-2585/07 0225-07

PEACH Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Spain, 2007 La Fortesa (Spring lady)	3	0.125	0.001	1300	Fruit	-0 0 2 7 9 14	0.24 0.5 0.33 0.25 0.18 0.28	RA-2585/07 0496-07
Italy, 2007 San Martino (Elegan lady)	3	0.125	0.0125	1000	Fruit	-0 0 3 7 10 14	0.21 0.51 0.27 0.27 0.34 0.22	RA-2585/07 0497-07
Greece, 2007 Neokastro (Luande)	3	0.125	0.0178	700	Fruit	-0 0 3 7 10 14	0.21 0.43 0.26 0.19 0.14 0.18	RA-2585/07 0498-07
Germany, 2008 Meckenbeuren (Red Haven)	3	0.137 0.125 0.125	0.01 0.01 0.01	1367 1250 1250	Fruit	-0 0 3 7 10	0.13 0.25 0.16 0.14 0.09	08-2054 08-2054-01
Germany, 2008 Bodman-Ludwigshafen (Edelstädter)	3	0.125	0.042	300	Fruit	-0 0 3 7 11	0.3 0.5 0.47 0.39 0.25	08-2054 08-2054-02
France, 2010 Valleres (Pavie Sanguine)	3	0.125	0.0139	900	Fruit	-0 0 3 7 10 14	0.1 0.17 0.09 (0.21 ^a) 0.16 0.14 0.09	10-2256 10-2256-01
Germany, 2010 Neustadt (Benedikt)	3	0.125	0.025	500	Fruit	-0 0 3 7 10 14 21	0.04 0.09 0.05 (0.07 ^b) 0.02 0.04 0.03 0.02	10-2256 10-2256-02

Studies RA-2581, RA-2585 were also provided to the 2010 JMPR [2010 JMPR Fluopyram Evaluation: Table 90]

Values in parenthesis: application parameter per meter crop height

^a After 7 days cold storage

^b After 3 days cold storage

Plums

Results from supervised trials from Europe on plums were provided to the Meeting. In these trials, fluopyram (SC formulations) were applied twice at 0.25 kg ai/ha, or three times at 0.125 kg ai/ha. In all trials, the treatments were made at 6–8 day intervals to mature, full-sized trees, as foliar sprays using knapsack atomiser or airblast single-nozzle sprayers to apply 1000–1500 L water/ha. At two sites, 1–2 fluopyram sprays were also applied to separate plots at different times over flowering and to immature fruit.

Samples of at least 1 kg fruit or 24 fruit (without stalks) were frozen within 24 hours of sampling and stored at -18 °C for up to 524 days before whole fruit analysis for fluopyram using

LC/MS/MS Method 00984. The reported LOQ was 0.01 mg/kg and the mean recovery rates in samples spiked with 0.01–1.0 mg/kg fluopyram ranged from 74–107%.

Table 4 Fluopyram residues in plums from trials in North and South Europe involving foliar applications of fluopyram (SC formulations)

PLUM Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
GAP: Greece	3	0.1	0.0067	1500		3		
Spain, 2007 Andalucia (Red Beauty)	2	0.25 0.268	0.017	1500 1605	Fruit	–0 0 3 7 14	0.06 0.08 0.09 0.09 0.06	RA-2589/07 R 2007 0232/9
Italy, 2007 Bologna (Angeleno)	2	0.25	0.017	1500	Fruit	–0 0 3 7 14	0.03 0.07 0.06 0.08 0.05	RA-2589/07 R 2007 0503/4
Germany, 2007 Tettwang (Presenta)	2	0.25	0.017	1500	Fruit	–0 0 3 7 13	0.04 0.11 0.12 0.07 0.09	RA-2588/07 R 2007 0499/2
Germany, 2007 Moos / Bankholzen (Presenta)	2	0.25	0.025	1000	Fruit	–0 0 3 7 14	0.09 0.24 0.25 0.19 0.16	RA-2588/07 R 2007 0502/6
Germany, 2007 Monheim (Hauszw. Purpurgold)	2	0.25	0.025	1000	Fruit	–0 0 3 7 14	0.07 0.19 0.11 0.11 0.08	RA-2588/07 R 2007 0231/0
Netherlands, 2007 Oosterblokker (Reine Victoria)	2	0.25	0.017	1500	Fruit	–0 0 3 7 14	0.07 0.17 0.13 0.16 0.13	RA-2588/07 R 2007 0501/8
Germany, 2011 Thümmlitz-walde (Hanita)	2	0.209 (0.084)	0.017	1250 (500)	Fruit	–0 0 3 7 14 21	0.046 0.11 0.11 0.08 0.11 0.10	11-2017 11-2017-01
Netherlands, 2011 Zwaagdijk-West (Reine Victoria)	2	0.209–0.225 (0.084– 0.090)	0.0167	1250– 1347 (500– 539)	Fruit	–0 0 3 7 14	0.09 0.23 0.24 0.33 0.08	11-2017 11-2017-02
Germany, 2011 Langenfeld (Katinka)	2	0.263–0.268 (0.088– 0.089)	0.017	1589– 1615 (530– 538)	Fruit	–0 0 3 7 14 21	0.06 0.1 0.1 0.09 0.07 0.07	11-2017 11-2017-03

PLUM Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
United Kingdom, 2011 Royston (Victoria)	2	0.209 (0.084)	0.017	1250 (500)	Fruit	-0 0 3 7 14 21	0.04 0.07 0.1 0.06 0.06 0.05	11-2017 11-2017-04
Germany, 2009 Leisnig Sachsen (Cacaks Schöne)	2	0.12	0.008	1500	Fruit	105 108 112 119	< 0.01 < 0.01 < 0.01 < 0.01	09-2224 09-2224-01a
Germany, 2009 Leisnig Sachsen (Cacaks Schöne)	1	0.12	0.008	1500	Fruit	0 32 35 39 46	0.09 0.04 0.05 0.04 0.02	09-2224 09-2224-01b
Germany, 2009 Leisnig Sachsen (Cacaks Schöne)	2	0.12	0.008	1500	Fruit	0 3 7 14	0.17 0.14 0.12 0.07	09-2224 09-2224-01c
Germany, 2009 Leisnig Sachsen (Cacaks Schöne)	1	0.12	0.008	1500	Fruit	0 3 7 14	0.05 0.07 0.06 0.05	09-2224 09-2224-01d
Netherlands, 2009 Oosterblokker (Rheine Victoria)	2	0.12	0.008	1500	Fruit	119 122 126 133	< 0.01 < 0.01 < 0.01 < 0.01	09-2224 09-2224-02a
Netherlands, 2009 Oosterblokker (Rheine Victoria)	1	0.12	0.008	1500	Fruit	67 70 74 81	< 0.01 < 0.01 < 0.01 < 0.01	09-2224 09-2224-02b
Netherlands, 2009 Oosterblokker (Rheine Victoria)	2	0.12	0.008	1500	Fruit	0 3 7 14	0.02 0.01 0.01 < 0.01	09-2224 09-2224-02c
Netherlands, 2009 Oosterblokker (Rheine Victoria)	1	0.12	0.008	1500	Fruit	0 3 7 14	0.02 0.01 0.02 0.01	09-2224 09-2224-02d
Germany, 2008 Tettnang/Bernau (Presenta)	3	0.125	0.0125	1000	Fruit	-0 0 3 8 14	0.12 0.1 0.11 0.11 <u>0.14</u>	08-2062 08-2062-01
Germany, 2008 Meckenheim (Hauszwetsche)	3	0.125	0.0125	1000	Fruit	-0 0 3 7 14	0.2 0.34 0.18 <u>0.22</u> 0.2	08-2062 08-2062-02
Netherlands, 2008 Zwaadijk (Reine Victoria)	3	0.125	0.0083	1500	Fruit	-0 0 3 7 14	0.09 0.11 <u>0.13</u> 0.12 0.13	08-2062 08-2062-03

PLUM Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Netherlands, 2008 St Jacobiparochie (Reine Victoria)	3	0.125	0.0083	1500	Fruit	-0 0 3 7 14	0.06 0.11 <u>0.1</u> 0.1 0.07	08-2062 08-2062-04
France, 2008 Moissac (Reine Claude)	3	0.125	0.0083	1500	Fruit	-0 0 3 7 14	0.07 0.08 0.12 <u>0.13</u> 0.12	08-2063 08-2063-01
Italy, 2008 Castel Guelfo (President)	3	0.125	0.0083	1500	Fruit	-0 0 3 7 13	0.05 0.08 0.08 <u>0.1</u> 0.06	08-2063 08-2063-02
France, 2009 Steinseltz (Quetches d'Alsace)	3	0.125	0.0125	1000	Fruit	-0 0 3 7 14 21	0.16 0.25 <u>0.2</u> 0.18 0.19 0.17	09-2070 09-2070-02
Germany, 2009 Jork (Fellenberger)	3	0.125	0.0125	1000	Fruit	-0 0 3 7 14 20	0.13 0.2 0.19 0.18 0.14 <u>0.2</u>	09-2070 09-2070-04
Germany, 2009 Laumersheim (Elena)	3	0.125	0.0125	1000	Fruit	-0 0 3 7 13 21	0.1 0.29 0.16 <u>0.18</u> 0.13 0.14	09-2070 09-2070-05
France, 2009 Moissac (Reine Claude)	3	0.125	0.008	1500	Fruit	-0 0 3 7 14 21	0.09 0.11 <u>0.16</u> 0.12 0.12 0.13	09-2071 09-2071-01
Italy, 2009 Mulino di Savignano sul Panaro (Angeleno)	3	0.125	0.008	1500	Fruit	-0 0 3 7 14 21	0.03 0.08 <u>0.08</u> 0.08 0.07 0.06	09-2071 09-2071-02
France, 2010 Innenheim (Quetches d'Alsace)	3	0.126	0.014	900	Fruit	-0 0 3 7 14 21	0.17 0.39 0.27 0.27 0.24 0.24	10-2260 10-2260-01
Poland, 2010 Witoldzin (Wagrowicra)	3	0.126	0.0105	1200	Fruit	-0 0 3 7 14	0.28 0.29 0.28 0.24 0.05	10-2260 10-2260-03

Application rates in brackets are per metre crop height

Berries and other small fruit

Currants

Results from supervised trials from Europe on currants were provided to the Meeting. In the German trials, two applications of fluopyram (SC formulations) were made 7–10 days apart to outdoor currants 0.2 kg ai/ha using knapsack atomiser single-nozzle sprayers to apply 300–1000 L/ha. In the Belgian and the Netherlands trials, currants grown under plastic cover were treated with two applications of 0.2 kg ai/ha, applied 7 days apart using knapsack atomiser or hand lances to apply 1000 L/ha.

Samples of at least 0.5 kg fruit were frozen within 24 hours of sampling and stored at -18 °C for up to 234 days before whole fruit analysis for fluopyram using LC/MS/MS Method 00984. The reported LOQ was 0.01 mg/kg and the mean recovery rates in samples spiked with 0.01–2.0 mg/kg fluopyram ranged from 93–107%. In one trial, higher recovery rates of 122–181% were reported in samples spiked at the lower levels of 0.01–0.1 mg/kg, but were 106–107% in the samples spiked at 1.0 mg/kg.

Table 5 Fluopyram residues in currants from trials in North Europe involving foliar applications of fluopyram (SC formulations)

CURRANT Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Germany, 2009 (Titania)	2	0.2	0.02	1000	Fruit	7 14	0.92 0.53	P 1799 G 926/15390 > 120% Recovery at 0.01, 0.1 mg/kg spikes
Germany, 2010 Ellerhoop (Ben Alder)	2	0.2	0.02	1000	Fruit	0 7 14 21	0.85 0.29 0.35 0.27	10L03113-01 FT-1029
Germany, 2010 Frankfurt (Titania)	2	0.2	0.02	1000	Fruit	0 7 14 21	1.98 1.69 0.99 0.81	10L03113-01 FT-1030
Germany, 2010 Neustadt (Tsema)	2	0.2	0.067	300	Fruit	0 7 14 21	1.18 1.01 0.67 0.60	10L03113-01 FT-1031
Germany, 2010 Freising (Ben Nevis)	2	0.2	0.02	1000	Fruit	0 7 14 21	0.75 0.44 0.42 0.40	10L03113-01 FT-1032
Germany, 2010 Bayern (Titania)	2	0.2	0.02	1000	Fruit	7 14	0.14 0.26	10L03113-01 FT-1033
Germany, 2010 Rheinpfalz (Ometa)	2	0.2	0.02	1000	Fruit	7 14	1.63 1.11	10L03113-01 FT-1034
Germany, 2010 Bavendorf (Titania)	2	0.2	0.02	1000	Fruit	7 14	0.64 0.49	10L03113-01 FT-1035

CURRANT Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Belgium, 2011 Perwez (Rosetta) (protected)	2	0.206 0.212	0.0222 0.0222	927 953	Fruit	-0 0 1 7 14 21	0.37 1.1 1.1 0.72 0.48 0.29	BCS-G402-11 G402-11F-1
Netherlands, 2011 KZ Westwoud (Rovada) (protected)	2	0.2	0.02	1000	Fruit	-0 0 1 3 7 14 21	0.37 0.41 0.41 0.44 0.36 0.29 0.22	PTZ-NLF-11796 11796-1

Raspberries

Results from supervised trials from Europe on raspberries grown in the field and under plastic cover were provided to the Meeting. In the German and French field trials, two applications of 0.2 kg ai/ha fluopyram (SC formulations) were made 7–10 days apart using knapsack atomiser single-nozzle sprayers to apply 300–1000 L/ha. In the Belgian and the Netherlands trials, crops grown under plastic (top) cover were treated twice at 0.2 kg ai/ha, 7 days apart using knapsack atomiser or hand lances to apply 900–1000 L/ha.

Samples of at least 0.5 kg fruit were frozen within 24 hours of sampling and stored at –18 °C for up to 234 days before whole fruit analysis for fluopyram using LC/MS/MS Method 00984. The reported LOQ was 0.01 mg/kg and the mean recovery rates in samples spiked with 0.01–2.0 mg/kg fluopyram ranged from 93–107%. In one trial, higher recovery rates of 122–181% were reported in samples spiked at the lower levels of 0.01–0.1 mg/kg, but were 106–107% in the samples spiked at 1.0 mg/kg.

Table 6 Fluopyram residues in outdoor raspberries from trials in Europe involving foliar applications of fluopyram (SC formulations)

RASPBERRIES Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Germany, 2009 Buxtehude (Tulameen)	2	0.2	0.02	1000	Fruit	7 14	0.26 0.32	P 1799 G FT-908
P 1799 G, 909 Germany, 2009 Vechta-Langforden (Meeker)	2	0.2	0.02	1000	Fruit	7 14	0.81 0.22	P 1799 G FT-909
France, 2009 Juillac (Meeker)	2	0.202 0.209	0.0251 0.0251	804 832	Fruit	0 1 3	1.2 0.95 0.88	RAFR03509 RE09005
France, 2009 Saugon Gironde (Meeker)	2	0.211 0.214	0.0251 0.0252	840 850	Fruit	0 1 3	1.5 0.97 0.87	RAFR03509 RE09006
Germany, 2010 Ellerhoop (Schönemanns)	2	0.2	0.02	1000	Fruit	0 7 14 21	0.5 0.19 0.15 0.12	10L03113-01 GEP-1008

RASPBERRIES Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Germany, 2010 Oder (Polana)	2	0.2	0.02	1000	Fruit	0 7 14 21 28	1.9 0.24 0.14 0.11 0.07	10L03113-01 GEP-1009

Table 7 Fluopyram residues in protected raspberries from trials in Europe involving foliar applications of fluopyram (SC formulations)

RASPBERRIES Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
GAP, S Africa	2	0.25		300–1000		3		10-14 day interval
France, 2010 Juillac Correze (Meeker)	2	0.199 0.213	0.0251 0.0251	792 848	Fruit	0 1 3	2.3 1.6 <u>1.2</u>	RAFR00810 RE10015
France, 2010 Saugon Gironde (Heritage)	2	0.22	0.025	875	Fruit	0 1 3	0.76 1.1 <u>0.69</u>	RAFR00810 RE10016
Belgium, 2011 Perwez (Malling Exploit)	2	0.194 0.207	0.0222 0.0222	873 933	Fruit	–0 0 1 3 7 14 21	0.13 0.58 0.5 <u>0.51</u> 0.36 0.19 0.09	BCS-G401-11 G401-11F-1
Netherlands, 2011 Haaften (Brilliant)	2	0.2	0.02	1000	Fruit	–0 0 1 3 7 14 21	0.49 1.3 0.99 <u>0.7</u> 0.54 0.17 0.17	PTZ-NLI-11797 11797-1
Germany, 2011 Koln (Sugana)	2	0.2	0.02	1000	Fruit	0 7 10 14 21	1.6 0.78 0.28 0.32 0.21	P 2428 G RU 1105
Germany, 2011 Kalsruhe (Tulameen)	2	0.2	0.02	1000	Fruit	14	0.51	P 2428 G RU 1107

Bulb vegetables

Onion, bulb

Results from supervised trials from Europe on bulb onions were provided to the Meeting. Trials conducted in 2006 and 2007 were also provided to the 2010 JMPR. In the trials, one or two applications of 0.1 kg ai/ha or 0.2 kg ai/ha (or in some earlier trials one application of 0.5 kg ai/ha) fluopyram (SC formulations) were made 6–8 days apart, using knapsack single-nozzle or mini-boom sprayers (3–12 nozzles) to apply 200–800 L/ha.

Samples of at least 1.5 kg or 24 bulbs were frozen within 24 hours of sampling, and stored at –18 °C for up to 398 days before analysis for fluopyram using LC/MS/MS Method 00984. The

reported LOQ was 0.01 mg/kg and the mean recovery rates in samples spiked with 0.01–1.0 mg/kg fluopyram ranged from 81% and 106%.

Table 8 Fluopyram residues in bulb onions from trials in North and South Europe, involving foliar applications of fluopyram (SC formulations)

ONION (BULB) Country, year Location (variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
GAP: Germany	2	0.1		200–800		7		7 day interval
France, 2008 Bouafle Ile-de-France (Paille des vertus)	1	0.5	0.17	300	Bulb	160 174	0.01 0.01	08-2161-01
Germany, 2008 Burscheid (Sherpa)	1	0.5	0.17	300	Bulb	124 138	< 0.01 < 0.01	08-2161-02
Spain, 2008 Alginet (Liria)	1	0.5	0.17	300	Bulb	139 153	< 0.01 < 0.01	08-2172-01
Italy, 2008 Bologna Emilia (Density)	1	0.5	0.17	300	Bulb	133 146	< 0.01 < 0.01	08-2172-02
Italy, 2011 Cento (Vaquero)	1	0.2	0.067	300	Bulb	0 7 14 21	0.14 0.04 < 0.01 < 0.01	11-2108-01
Spain, 2011 Gavà (Figueras)	1	0.217	0.0799	271	Bulb	0 7 14 21	0.095 0.03 0.016 < 0.01	11-2108-02
Italy, 2012 Cento (Vaquero)	1	0.2	0.0333	600	Bulb	0 3 7 14 21	0.05 0.015 < 0.01 < 0.01 < 0.01	12-2041-01
Italy, 2012 Maccarese (Centurion F1)	1	0.2	0.0286	700	Bulb	0 3 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	12-2041-02
Italy, 2012 Adrano (Locale Marrone)	1	0.2	0.0286	700	Bulb	0 3 7 14 21	0.039 < 0.01 < 0.01 < 0.01 < 0.01	12-2041-03
Spain, 2012 Alginet (Nicasio)	1	0.2	0.0286	700	Bulb	0 3 7 14 21	0.017 < 0.01 < 0.01 < 0.01 < 0.01	12-2041-04
France, 2011 Layarac (Keepwell)	1	0.2	0.0333	600	Bulb	0 3 7 14 21	0.06 0.028 < 0.01 < 0.01 < 0.01	12-2041-05
France, 2012 Etoile sur Rhone (Spirit)	1	0.2	0.0333	600	Bulb	0 3 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	12-2041-06

ONION (BULB) Country, year Location (variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
France, 2011 Nezel (Bugati) Yellow onion	2	0.1	0.0333	300	Bulb	-0 0 7 14 20	0.012 0.072 0.02 0.011 0.012	11-2028-01
Germany, 2011 Kohlhof (Taresco)	2	0.1	0.0333	300	Bulb	-0 0 7 14 21	< 0.01 0.03 < 0.01 0.01 < 0.01	11-2028-02
Germany, 2011 Burscheid (Sherpa F1)	2	0.1	0.0333	300	Bulb	-0 0 7 14 21	< 0.01 0.047 < 0.01 < 0.01 < 0.01	11-2028-03
UK, 2011 Cambridge (Bennito) White bulb	2	0.106 0.1	0.05 0.05	213 200	Bulb	-0 0 7 14 21	< 0.01 0.039 < 0.01 < 0.01 < 0.01	11-2028-04
Germany, 2012 Burscheid (Sherpa F1)	2	0.1	0.0333	300	Bulb	-0 0 3 7 14 21	< 0.01 0.025 < 0.01 < 0.01 < 0.01 < 0.01	12-2034-01
France (N), 2012 Nézel (Bugatti F1)	2	0.1	0.0333	300	Bulb	-0 0 3 7 14 21	0.018 0.092 0.035 0.03 0.015 0.01	12-2034-02
UK, 2012 Cambridge (Bennito)	2	0.1	0.05	200	Bulb	-0 0 3 7 14 22	< 0.01 0.029 0.031 0.04 0.014 < 0.01	12-2034-03
Belgium, 2012 Viller-Perwin (Summit F1)	2	0.1	0.0222	450	Bulb	-0 0 2 7 14 21	< 0.01 0.026 < 0.01 < 0.01 < 0.01 < 0.01	12-2034-04
France, 2006 Bouafle (Jaunes des Cévennes)	2	0.2	0.04	500	Bulb	-0 0 7 14 21	0.04 0.05 0.02 0.01 0.02	R 2006 0340/1 RA-2567/06
France, 2006 Cailloux sur Fontaines (Rebouillon)	2	0.2	0.033	600	Bulb	-0 0 7 14 21	< 0.01 0.01 < 0.01 < 0.01 < 0.01	R 2006 0342/8 RA-2568/06
Germany, 2006 Langenfeld-Reusrath (Stuttgarter Riesen)	2	0.2	0.0666	300	Bulb	-0 0 7 14 21	< 0.01 0.08 < 0.01 < 0.01 < 0.01	R 2006 0537/4 RA-2567/06

ONION (BULB) Country, year Location (variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Germany, 2006 Kohlhof (Takstar)	2	0.2	0.04	500	Bulb	-0 0 7 14 21	0.02 0.06 0.01 < 0.01 0.01	R 2006 0538/2 RA-2567/06
UK, 2006 Thetford (Red Barron)	2	0.2	0.0666	300	Bulb	-0 0 7 14 20	0.03 0.11 0.04 0.02 0.03	R 2006 0539/0 RA-2567/06
Spain, 2006 Gava (Dulce de Fuentes)	2	0.212 0.2	0.033 0.033	636 600	Bulb	-0 0 7 14 21	< 0.01 0.07 0.01 0.01 < 0.01	R 2006 0540/4 RA-2568/06
Italy, 2006 Bologna (Density 5)	2	0.2	0.04	500	Bulb	-0 0 7 14 21	0.01 0.04 0.01 < 0.01 < 0.01	R 2006 0541/2 RA-2568/06
Greece, 2006 Thiva (Ideal)	2	0.2	0.0334	600	Bulb	-0 0 7 15 21	< 0.01 0.04 < 0.01 < 0.01 < 0.01	R 2006 0542/0 RA-2568/06
Germany, 2007 Langenfeld-Reusrath (Stuttgarter Riesen)	2	0.2	0.0666	300	Bulb	-0 0 7 14 21	< 0.01 0.07 < 0.01 0.01 0.01	R 2007 0038/5 RA-2517/07
France, 2007 Layrac (Daytona)	2	0.2	0.0334	600	Bulb	-0 0 7 14 21	0.01 0.1 0.02 0.02 0.01	R 2007 0040/7 RA-2518/07
Germany, 2007 Vettweiß- Müddersheim (Benito)	2	0.2	0.0666	300	Bulb	-0 0 7 14 20	< 0.01 0.08 < 0.01 < 0.01 0.01	R 2007 0558/1 RA-2517/07
France, 2007 Luzillé (Hybing F1)	2	0.2	0.0334	600	Bulb	-0 0 7 14 21	< 0.01 0.05 < 0.01 < 0.01 < 0.01	R 2007 0560/3 RA-2517/07
Netherlands, 2007 Wieringerwerf (Hybell)	2	0.2	0.025	800	Bulb	-0 0 7 14 21	< 0.01 0.03 0.01 < 0.01 < 0.01	R 2007 0561/1 RA-2517/07
Italy, 2007 Poggio Renatico (Rossa di Toscana)	2	0.2	0.033	600	Bulb	-0 0 7 14 21	< 0.01 0.03 < 0.01 < 0.01 < 0.01	R 2007 0563/87 RA-2518/07

ONION (BULB) Country, year Location (variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Spain, 2007 Alginet (Liria)	2	0.2	0.033	600	Bulb	−0 0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	R 2007 0564/67 RA-2518/07
Portugal, 2007 Francisco / Alcochete (Spring Star)	2	0.2	0.04	500	Bulb	−0 0 7 14 21	< 0.01 0.08 0.02 0.02 0.02	R 2007 0565/47 RA-2518/07
Greece, 2007 Aronas - Katerini (Kozanis)	2	0.2	0.04	500	Bulb	−0 0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	R 2007 0566/27 RA-2518/07

Studies RA-2517, RA-2518, RA-2567, RA-2568 were also provided to the 2010 JMPR [2010 JMPR Fluopyram Evaluation: Table 105]

Results from supervised trials from USA on bulb onions were provided to the Meeting. Trials conducted in 2007 were also provided to the 2010 JMPR. In the trials, one or two applications of 0.25 kg ai/ha fluopyram (SC formulations) were made 5 days apart, using knapsack sprayers or tractor-mounted 4-row boom sprayers to apply about 120–280 L/ha. In the 2009 trials, additional plots were treated with fluopyram plus adjuvant.

Duplicate samples of at least 2 kg dry bulbs were frozen within 1 hour of sampling and stored frozen for up to 267 days before analysis for fluopyram using LC/MS/MS Method 00984. The reported LOQ was 0.01 mg/kg and the mean recovery rates in samples spiked with 0.01–0.5 mg/kg fluopyram ranged from 94% to 106%.

Table 9 Fluopyram residues in bulb onions from trials in USA, involving foliar applications of fluopyram (SC formulations)

ONION (BULB) Country, year Location (variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	mean	
USA, 2007 Germansville, PA (Medium Red)	2	0.251	0.14	179	Bulb	0	0.15, 0.12	0.14	RAGML001 GM043-07HA
USA, 2007 Uvalde, TX (Leona)	2	0.248 0.249	0.198 0.147	125 169	Bulb	0	0.15, 0.19	0.17	RAGML001 GM044-07HA
USA, 2007 Larned, KS (Texas Supersweet)	2	0.241 0.241	0.145 0.148	166 163	Bulb	0	0.15, 0.22	0.19	RAGML001 GM045-07HA
USA, 2007 Sanger, CA (Yellow sweet spanish)	2	0.251 0.247	0.155 0.147	162 168	Bulb	0 1 3 7 10	0.01, 0.05 0.03, 0.02 0.04, 0.03 0.01, 0.03 0.01, 0.02	0.07 0.03 0.03 0.02 0.02	RAGML001 GM046-07DA
USA, 2007 Fresno, CA (Gunnison F1)	2	0.248 0.252	0.18 0.18	138 140	Bulb	0	0.09, 0.11	0.1	RAGML001 GM047-07HA
USA, 2007 Parkdale, OR (Yellow Danver)	2	0.251 0.246	0.206 0.198	122 124	Bulb	0	0.08, 0.04	0.06	RAGML001 GM048-07HA

ONION (BULB) Country, year Location (variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	mean	
USA, 2007 Richland, IA (Yellow No. 1)	2	0.248 0.246	0.16 0.156	155 158	Bulb	0	0.02, 0.01	0.02	RAGML001 GM109-07HA
USA, 2007 Hillsboro, OR (Redwing)	2	0.239 0.25	0.145 0.145	165 172	Bulb	0	0.12, 0.18	0.15	RAGML001 GM110-07HA
USA, 2009 Raymondville, TX (Yellow Granex)	1	0.255	0.135	189	Bulb	0 3 14	0.11, 0.09 0.11, 0.07 0.02, 0.02	0.1 0.09 0.02	RAGMP155 GM052-09HA-A
USA 2009, Raymondville, TX (Yellow Granex)	1	0.256	0.135	190	Bulb	0 3 14	0.1, 0.09 0.07, 0.06 0.01, 0.01	0.09 0.06 0.02	RAGMP155 GM052-09HA-B (with adjuvant)
USA, 2009 Fresno, CA (Gannison F1)	1	0.248	0.0883	281	Bulb	0 3 14	0.07, 0.05 0.06, 0.07 0.28, 0.31	0.06 0.07 0.29	RAGMP155 GM053-09HA-A
USA, 2009 Fresno, CA (Gannison F1)	1	0.247	0.0882	280	Bulb	0 3 14	0.05, 0.02 0.12, 0.06 0.15, 0.13	0.04 0.09 0.14	RAGMP155 GM053-09HA-B (with adjuvant)
USA, 2009 Prosser, WA (South Port)	1	0.251	0.117	215	Bulb	0 3 14	0.01, < 0.01 0.02, 0.02 < 0.01, < 0.01	0.01 0.02 < 0.01	RAGMP155 GM054-09HA-A
USA, 2009 Prosser, WA (South Port)	1	0.258	0.117	220	Bulb	0 3 14	< 0.01, 0.02 0.02, 0.02 < 0.01, < 0.01	0.01 0.02 < 0.01	RAGMP155 GM054-09HA-B (with adjuvant)

Study RAGML001 was also provided to the 2010 JMPR [2010 JMPR Fluopyram Evaluation: Table 106]

Leek

Results from supervised trials from Europe on leeks were provided to the Meeting. In these trials fluopyram (SC formulations) foliar sprays were applied either once or twice (13–14 days apart) at 0.2 kg ai/ha or three times at 0.15 kg ai/ha (21–22 days apart), using knapsack mini-boom sprayers (3–12 nozzles) to apply 300–600 L/ha (200 L/ha in one trial). The trials involving two applications, conducted in 2006 and 2007 were also provided to the 2010 JMPR.

Samples of at least 1 kg or 24 plants (without roots) were frozen within 24 hours of sampling and stored at -18 °C for up to 391 days before analysis for fluopyram using LC/MS/MS Method 00984. The reported LOQ was 0.01 mg/kg and the mean recovery rates in samples spiked with 0.01–2.0 mg/kg fluopyram ranged from 71% to 118%.

Table 10 Fluopyram residues in leeks from trials in North and South Europe, involving foliar applications of fluopyram (SC formulations)

LEEK Country, Year Location (variety)	Application				Matrix	DAT	Residues (mg/kg)	Ref
	no	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
GAP: Germany, Switzerland	1	0.2		200–700		21		
Germany, 2012 Leichlingen (Vermont)	1	0.2	0.067	300	Tops ^a	0 14 21 28 35	0.66 0.013 <u>< 0.01</u> < 0.01 < 0.01	12-2033 12-2033-01, 42799

LEEK Country, Year Location (variety)	Application				Matrix	DAT	Residues (mg/kg)	Ref
	no	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
France, 2012 Soings en Sologne (Callahan)	1	0.2	0.067	600	Tops ^a	0 14 21 28 35	0.3 0.015 <u>< 0.01</u> < 0.01 < 0.01	12-2033 12-2033-02, 41230
UK, 2012 Cambridge (Lyon 2 Prizetaker)	1	0.221	0.1	221	Tops ^a	0 14 21 28 35	0.99 0.093 <u>0.06</u> 0.037 0.045	12-2033 12-2033-03, CB22
Belgium, 2012 Villers-Perwin (Surfer F1)	1	0.2	0.044	450	Tops ^a	0 14 21 28 35	0.68 0.081 <u>0.07</u> 0.032 0.022	12-2033 12-2033-04, 6210
France, 2011 Soings en Sologne (Davincy)	1	0.2	0.033	600	Tops ^a	0 7 14 21	0.6 0.067 0.026 <u>< 0.01</u>	11-2029 11-2029-01
Germany, 2011 Werl-Westönnen (Parton F1)	1	0.2	0.067	300	Tops ^a	0 7 14 21	0.49 0.07 0.034 <u>0.03</u>	11-2029 11-2029-02
Germany, 2011 Langenfeld (Prelina)	1	0.2	0.067	300	Tops ^a	0 7 14 21	0.3 0.045 0.013 <u>< 0.01</u>	11-2029 11-2029-03
Belgium, 2007 Villers-Perwin (Belton)	1	0.2	0.044	450	Tops ^a	0 7 14 21	0.36 0.049 0.02 <u>0.01</u>	11-2029 11-2029-04
France, 2006 Faverolles (Diana)	2	0.2	0.067	300	Tops ^a	-0 0 7 14 21 28	0.18 2.1 1.2 0.67 0.32 0.20	RA-2569/06 R 2006 0343 6
Germany, 2006 Langenfeld-Reusrath (Pandora)	2	0.2	0.067	300	Tops ^a	-0 0 7 14 21 28	0.06 0.68 0.08 0.03 0.02 0.02	RA-2569/06 R 2006 0465 3
UK, 2006 Cambridgeshire (Shelton)	2	0.2	0.067	300	Tops ^a	-0 0 7 15 21 28	0.06 1.1 0.55 0.12 0.06 0.07	RA-2569/06 R 2006 0466 1
Germany, 2006 Bornheim – Sechtem (Amundo)	2	0.2	0.067	300	Tops ^a	-0 0 7 15 22 28	0.27 0.97 0.67 0.23 0.17 0.14	RA-2569/06 R 2006 0468 8

LEEK Country, Year Location (variety)	Application				Matrix	DAT	Residues (mg/kg)	Ref
	no	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
France, 2006 Cailloux sur Fontaines (Ahton)	2	0.2	0.033	600	Tops ^a	-0 0 7 14 21 29	0.12 1.1 0.37 0.28 0.17 0.05	RA-2570/06 R 2006 0344 4
Spain, 2006 Lebrija Sevilla (Shelton)	2	0.2	0.05	400	Tops ^a	-0 0 7 14 21 28	0.02 0.32 0.18 0.07 0.04 0.02	RA-2570/06 R 2006 0469 6
Germany, 2007, Schauernheim (Nobel)	2	0.2	0.033	600	Tops ^a	-0 0 7 14 21 28	0.1 0.41 0.08 0.04 0.03 0.02	RA-2521/07 R 2007 0056 3
France, 2007 Bouafle (St Vistor)	2	0.2	0.04	500	Tops ^a	-0 0 7 14 21 28	0.05 1.6 0.22 0.15 0.09 0.11	RA-2521/07 R 2007 0569 7
Netherlands, 2007 Zwaagdijk-Oost (Roxton)	2	0.2	0.033	600	Tops ^a	-0 0 7 14 21 28	0.07 0.48 0.08 0.05 0.02 0.02	RA-2521/07 R 2007 0570 0
Germany, 2007 Langenfeld-Reusrath (Pandora)	2	0.2	0.067	300	Tops ^a	-0 0 7 14 21 28	0.25 1.7 0.26 0.02 0.01 < 0.01	RA-2521/07 R 2007 0571 9
France, 2007 Castelsarrasin (Porbella)	2	0.2	0.033	600	Tops ^a	-0 0 7 14 21 28	0.35 2.8 0.39 0.31 0.24 0.25	RA-2522/07 R 2007 0057 1
Italy, 2007 Lusia (Sabina)	2	0.2	0.033	600	Tops ^a	-0 0 7 14 21 28	0.06 2.1 0.15 0.16 0.03 0.02	RA-2522/07 R 2007 0572 7
Germany, 2007 Schauernheim (Nobel)	3	0.15	0.25	600	Tops ^a	-0 0 7 14 21 28	0.02 0.46 0.07 0.04 0.03 0.02	RA-2609/07 R 2007 0249/3

LEEK Country, Year Location (variety)	Application				Matrix	DAT	Residues (mg/kg)	Ref
	no	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
France, 2007 Bouafle (St Victor)	3	0.15	0.03	500	Tops ^a	-0 0 7 14 21 28	0.04 1.2 0.24 0.11 0.1 0.05	RA-2609/07 R 2007 0573/5
Netherlands, 2007 Zwaagdijk-Oost (Roxton)	3	0.15	0.025	600	Tops ^a	-0 0 7 14 21 28	0.01 0.24 0.04 0.02 0.01 < 0.01	RA-2609/07 R 2007 0574/3
France, 2007 Castelsarrasin (Porbella)	3	0.15	0.025	600	Tops ^a	-0 0 7 14 21 27	0.24 1.7 0.63 0.52 0.34 0.26	RA-2610/07 R 2007 0250/7

^a Whole plant without root

Studies RA-2521, RA-2522, RA-2569, RA-2570 were also provided to the 2010 JMPR [2010 JMPR Fluopyram Evaluation: Table 104]

Brassica vegetables

Broccoli

Results from supervised trials from USA on broccoli were provided to the 2010 Meeting. In these trials, two foliar applications of 0.24–0.26 kg ai/ha fluopyram (SC 500 formulation), tank-mixed with trifloxystrobin (SC) in 122–186 L of water were applied to broccoli plants 5 days apart using knapsack sprayers or tractor-mounted boom sprayers. Plot sizes in these trials ranged from 53–159 square metres.

Duplicate samples of (at least 12 broccoli heads and stems) were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 448 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ of 0.01 mg/kg).

Table 11 Fluopyram residues in broccoli from supervised trials in USA involving two foliar applications of fluopyram (500 SC formulations)

BROCCOLI Country, year Location (variety)	Application				Matrix	DAT	Fluopyram residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)				mean	
USA, 2008 Raymondville, TX (Green Magic)	2	0.262 0.254	0.141 0.141	186 180	Heads	0	0.88, 1.31	1.1	RAGMP076 GM017-07HA
USA, 2007 Guadalupe, CA (Heritage)	2	0.238 0.254	0.195 0.185	122 137	Heads	0	1.2, 1.2	1.2	RAGMP076 GM018-07HA
USA, 2007 Sanger, CA (Marathon)	2	0.25 0.257	0.184 0.158	136 163	Heads	0 1 3 7 10	0.98, 1.1 0.99, 0.99, 0.95 0.85, 1.08 0.83, 0.83 0.76, 0.52 0.7, 0.63	1.1 0.98 0.97 0.83 0.64 0.66	RAGMP076 GM019-07DA Results from processing study

Study RAGMP076 was also provided to the 2010 JMPR [2010 JMPR Fluopyram Evaluation: Table 111]

Results from supervised trials from Europe on broccoli were provided to the Meeting. These trials were also provided to the 2010 JMPR. In these trials, two applications of fluopyram + tebuconazole (SC 200+200) at rates 0.2 kg ai/ha fluopyram in 300–800 L/water/ha of were made at 14–21 day intervals as foliar sprays using knapsack sprayers with hand-held booms (4–12 flat fan or hollow cone nozzles). Plot sizes in these trials ranged from 50–100 square metres.

Unreplicated samples of 12–20 broccoli heads were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 335 days before analysis for fluopyram and its BZM, PAA and PCA metabolites using LC/MS/MS Method 00984 or Method 00984/M001 (LOQs were 0.01 mg/kg for each analyte).

Table 12 Residues in broccoli from supervised trials in France, Germany, Greece, Italy, Netherlands and Spain involving foliar applications of fluopyram (SC formulations)

BROCCOLI Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
GAP: Germany	2	0.18		300–800		14		14 day interval
France, 2006 Ennemain (Chevalier)	2	0.2	0.067	300		–0	< 0.01	RA-2607/06 0400-06
						0	0.74	
						3	0.41	
						7	0.19	
						14	0.09	
						21	<u>0.14</u>	
Netherlands, 2006 Zwaagdijk-Oos (Ironman)	2	0.2	0.067	300		–0	< 0.01	RA-2607/06 0501-06
						0	0.83	
						3	0.61	
						7	0.12	
						14	<u>0.05</u>	
						21	0.02	
France, 2007 Ennemain (Chevalier)	2	0.2	0.067	300		–0	< 0.01	RA-2525/07 0069-07
						0	0.64	
						3	0.05	
						7	0.02	
						14	0.01	
						21	<u>0.02</u>	
Germany, 2007 Brühl-Schwardorf (Patinon)	2	0.2	0.033	600		–0	< 0.01	RA-2525/07 0586-07
						0	0.04	
						3	< 0.01	
						8	0.02	
						14	<u>< 0.01</u>	
						22	< 0.01	
Spain, 2006 Lebrija (Partenon)	2	0.2	0.05	400		–0	< 0.01	RA-2608/06 0401-06
						0	0.48	
						3	0.04	
						7	0.03	
						14	<u>≤ 0.01</u>	
Italy, 2006 Manfredonia (Olimpia)	2	0.2	0.025	800		–0	0.13	RA-2608/06 0502-06 c=0.02 mg/kg
						0	0.82	
						3	0.51	
						7	0.44	
						14	<u>0.13</u>	
						21	0.05	
Greece, 2007 Aronas (Marathon)	2	0.2	0.04	500		–0	0.04	RA-2526/07 0070-07
						0	0.29	
						3	0.26	
						7	0.19	
						14	<u>0.05</u>	
						21	0.05	

BROCCOLI Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
Spain, 2007 Gavà (Megaton)	2	0.2	0.033	600		-0	0.03	RA-2526/07 0587-07
						0	0.61	
						4	0.36	
						7	0.15	
						14	<u>0.06</u>	
						20	0.06	
Italy, 2007 Manfredonia (Partenon F1)	2	0.2	0.027	750		-0	0.03	RA-2526/07 0588-07
						0	0.39	
						3	0.19	
						7	0.06	
						14	<u>0.03</u>	
						21	0.02	

Studies RA-2525, RA-2526, RA-2607, RA-2608 were also provided to the 2010 JMPR [2010 JMPR Fluopyram Evaluation: Table 112]

Brussels sprouts

Results from supervised trials from Europe on Brussels sprouts were provided to the Meeting. These trials were also provided to the 2010 JMPR. In the trials, two foliar sprays of 0.2 kg ai/ha fluopyram (SC formulations) were applied 14–15 days apart using knapsack or wheelbarrow mini-boom sprayers (3-12 nozzles) to apply 300–800 L/ha.

Samples of at least 1 kg of sprouts were frozen within 24 hours of sampling and stored at –18 °C for up to 253 days before analysis for fluopyram using LC/MS/MS Method 00984. The reported LOQ was 0.01 mg/kg and the mean recovery rates in samples spiked with 0.01–1.0 mg/kg fluopyram ranged from 89% to 110%.

Table 13 Fluopyram residues in Brussels sprouts from trials in North and South Europe, involving foliar applications of fluopyram (SC formulations)

BRUSSELS SPROUTS Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
GAP: Germany	2	0.18		300–800		14		14 day interval
France, 2006 Fontaine l'Etalon (Louis)	2	0.2	0.067	300	Sprouts	-0	< 0.01	RA-2575/06 R 2006 0349/5
						0	0.02	
						7	0.07	
						14	<u>0.04</u>	
						21	0.02	
Germany, 2006 Langenfeld-Reusrath (Genius)	2	0.2	0.067	300	Sprouts	-0	0.04	RA-2575/06 R 2006 0473/4
						0	0.09	
						7	0.1	
						14	<u>0.07</u>	
						21	0.07	
UK, 2006 Saxmundham (Helemus)	2	0.2 0.21	0.067 0.067	300 318	Sprouts	-0	0.05	RA-2575/06 R 2006 0474/2
						0	0.16	
						7	0.13	
						14	<u>0.07</u>	
						21	0.06	
Germany, 2006 Meckenbeuren (Lunet)	2	0.2	0.04	500	Sprouts	-0	< 0.01	RA-2575/06 R 2006 0475/0
						0	0.04	
						7	0.03	
						14	<u>0.01</u>	
						22	0.01	

BRUSSELS SPROUTS Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
France, 2006 Castelsarrasin (Oliver)	2	0.2	0.067	300	Sprouts	-0 0 7 14 21	0.05 0.34 0.12 0.05 <u>0.07</u>	RA-2576/06 R 2006 0350/9
Italy, 2006 Ladispoli (Franklin)	2	0.2	0.025	800	Sprouts	-0 0 7 14 20	0.06 0.2 0.09 <u>0.09</u> 0.07	RA-2576/06 R 2006 0476/9
Germany, 2007 Bornheim-Sechtem (Maximus)	2	0.21 0.2	0.05 0.05	424 400	Sprouts	-0 0 6 14 21	0.05 0.08 0.09 <u>0.04</u> 0.02	RA-2531/07 R 2007 0076/8
France, 2007 Criquebeuf sur Seine (Abaccus)	2	0.2	0.04	500	Sprouts	-0 0 7 13 21	0.06 0.2 0.25 <u>0.14</u> 0.10	RA-2531/07 R 2007 0594/8
Netherlands, 2007 Zwaagdijk-Oost (Cerius)	2	0.2	0.033	600	Sprouts	-0 0 7 14 21	0.03 0.19 0.1 <u>0.04</u> 0.03	RA-2531/07 R 2007 0595/6
UK, 2007 Saxmundham (Maximus)	2	0.2	0.067	300	Sprouts	-0 0 7 15 21	0.02 0.26 0.06 0.03 <u>0.04</u>	RA-2531/07 R 2007 0596/4
France, 2007 Rillieux la Pape (Olivier)	2	0.2	0.04	500	Sprouts	-0 0 7 14 21	0.08 0.1 0.06 <u>0.05</u> 0.05	RA-2532/07 R 2007 0077/6
Italy, 2007 Manfredonia (Lunet)	2	0.2	0.0286	700	Sprouts	-0 0 7 14 22	0.16 0.41 0.2 <u>0.15</u> 0.08	RA-2532/07 R 2007 0597/2

Studies RA-2575, RA-2576, RA-2531, RA-2532 were also provided to the 2010 JMPR [2010 JMPR Fluopyram Evaluation: Table 115]

Cabbage, head

Results from supervised trials from Europe on head cabbages (red, white or round) were provided to the Meeting. In these trials fluopyram (SC formulations) foliar sprays were applied either twice at 0.2 kg ai/ha (14 days apart) or three times at 0.15 kg ai/ha (20–21 day intervals) using knapsack mini-boom sprayers (3–12 nozzles) to apply 300–600 L/ha (800 L/ha in one trial). The trials conducted in 2006 and 2007 and involving two fluopyram applications were also provided to the 2010 JMPR.

Samples of at least 12 units (min 10 kg of whole heads or two opposing quarter-heads) were frozen within 24 hours of sampling and stored at –18 °C for up to 307 days before analysis of heads (plus wrapper leaves) for fluopyram using LC/MS/MS Method 00984. The reported LOQ was 0.01 mg/kg and the mean recovery rates in samples spiked with 0.01–1.0 mg/kg fluopyram ranged from 84% to 110%.

Table 14 Fluopyram residues in cabbage heads from trials in North and South Europe, involving foliar applications of fluopyram (SC formulations)

CABBAGE, HEAD Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
GAP: Germany	2	0.18		300–800		14		14 day interval
France, 2006 Fondettes (Eton F1)	2	0.2	0.033	600	Head ^a	–0 0 3 7 14 21	< 0.01 0.05 0.06 0.02 <u>0.01</u> 0.01	RA-2577/06 R 2006 0354/1
Netherlands, 2006 Noorder-koggenland (Slaudena)	2	0.2	0.04	500	Head ^a	–0 0 3 7 14 21	0.03 < 0.01 0.02 < 0.01 <u>< 0.01</u> < 0.01	RA-2577/06 R 2006 0356/8
Germany, 2006 Werl-Westönnen (Novator)	2	0.2	0.04	500 500	Head ^a	–0 0 3 7 14 21	< 0.01 0.01 < 0.01 < 0.01 <u>< 0.01</u> < 0.01	RA-2577/06 R 2006 0477/7
Germany, 2006 Leichlingen (Latima)	2	0.2	0.067	300	Head ^a	–0 0 3 7 14 21	0.01 0.22 0.09 0.02 <u>0.01</u> < 0.01	RA-2577/06 R 2006 0478/5
France, 2006 Arnas (Count)	2	0.2	0.04	500	Head ^a	–0 0 4 7 14 21	0.02 0.19 0.04 0.04 <u>0.02</u> 0.01	RA-2578/06 R 2006 0357/6
Italy, 2006 Ladispoli (Castello)	2	0.2	0.033	600	Head ^a	–0 0 3 7 14 21	0.01 0.1 0.11 0.06 <u>0.01</u> < 0.01	RA-2578/06 R 2006 0479/3
Germany, 2007 Werl-Westönnen (Lennox)	2	0.2	0.067	300	Head ^a	–0 0 4 8 15 22	< 0.01 0.45 < 0.01 < 0.01 <u>< 0.01</u> < 0.01	RA-2529/07 R 2007 0073/3
France, 2007 Cergy (Lennox)	2	0.2	0.067	300	Head ^a	–0 0 3 7 14 21	0.05 0.34 0.42 0.21 <u>0.08</u> 0.04	RA-2529/07 R 2007 0270/1
Germany, 2007 Leichlingen (Rodima)	2	0.2	0.067	300	Head ^a	–0 0 3 7 14 21	0.08 0.42 0.05 0.03 <u>0.02</u> 0.02	RA-2529/07 R 2007 0589/1

CABBAGE, HEAD Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
UK, 2007 Saxmundham (Auralias)	2	0.2	0.067	300	Head ^a	–0 0 3 7 13 20	0.02 0.14 0.17 0.04 <u>0.04</u> 0.02	RA-2529/07 R 2007 0590/5
Belgium, 2007 Villers-Perwin (Regina F1)	2	0.2	0.036	550	Head ^a	–0 0 3 7 14 21	< 0.01 0.09 0.02 0.02 <u>< 0.01</u> 0.01	RA-2529/07 R 2007 0591/3
Italy, 2007 Ladispoli (Castello)	2	0.2	0.033	600	Head ^a	–0 0 3 7 14 21	0.01 0.12 0.11 0.03 0.01 0.02	RA-2530/07 R 2007 0592/1
Italy, 2012 Chiaromonte Gulfi (Green lunar)	2	0.2	0.025	800	Head ^a	–0 0 3 7 14 21 28	< 0.01 0.15 0.029 0.027 0.02 0.013 0.013	12-2042 12-2042-01
Germany, 2007 Werl-Westönnen (Lennox)	3	0.15	0.05	300	Head ^a	–0 0 4 8 15 22	< 0.01 0.12 < 0.01 < 0.01 < 0.01 < 0.01	RA-2612/07 R 2007 0251/5
UK, 2007 Saxmundham (Auralias)	3	0.15	0.05	300	Head ^a	–0 0 3 7 13 20	0.03 0.08 0.1 0.03 0.04 0.03	RA-2612/07 R 2007 0272/8
Belgium, 2007 Villers-Perwin (Regina F1)	3	0.15	0.027	550	Head ^a	–0 0 3 7 14 21	0.01 0.07 0.01 0.02 0.02 0.02	RA-2612/07 R 2007 0598/0

^a Cabbage heads with wrapper leaves

Studies RA-2529, RA-2530, RA-2577, RA-2578 were also provided to the 2010 JMPR [2010 JMPR Fluopyram Evaluation: Table 110]

Cauliflower

Results from supervised trials from Europe on cauliflower were provided to the Meeting. The 2006 and 2007 trials were also provided to the 2010 JMPR. In these trials, two applications of fluopyram (SC formulations) at rates 0.2 kg ai/ha fluopyram in 300–800 L water/ha of were applied at 13–15 day intervals as foliar sprays using knapsack sprayers with a single solid cone nozzle hand lance or hand-held booms (3–12 flat fan or hollow cone nozzles). Plot sizes in these trials ranged from 66–162 square metres.

Unreplicated samples of 12 cauliflower curds were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 14 months before analysis for fluopyram using LC/MS/MS Method 00984 or Method 00984/M001. The reported LOQ was 0.01 mg/kg.

Table 15 Residues in cauliflowers from supervised trials in France, Germany, Italy, Netherlands, Spain and UK involving two foliar applications of fluopyram (SC formulations)

CAULIFLOWER Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	No	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
GAP: Germany	2	0.18		300-800		14		14 day interval
United Kingdom, 2006 West Row (Optamist)	2	0.212 0.2	0.0666	318 300	Curd	-0 0 3 7 14 21	0.01 0.07 0.07 0.04 <u>0.05</u> 0.02	RA-2571/06 0345-06
Germany, 2006 Langenfeld-Reusrath (Freedom)	2	0.2	0.0666	300	Curd	-0 0 3 7 14 21	< 0.01 0.85 0.18 0.08 <u>0.01</u> < 0.01	RA-2571/06 0471-06
Germany, 2007 Langenfeld-Reusrath (Freedom)	2	0.2	0.0334	600	Plant ^a Curd	-0 0 3 7 14 21	0.15 2.4 0.04 0.02 <u>0.01</u> < 0.01	RA-2525/07 0067-07
Netherlands, 2007 Zwaagdijk-Oost (Speedy)	2	0.2	0.0666	300	Curd	-0 0 3 7 14 21	< 0.01 0.36 0.05 0.01 <u>0.01</u> 0.01	RA-2525/07 0583-07
France, 2006 Graveson (Thalassa)	2	0.2	0.0334	600	Curd	-0 0 3 7 13 21	< 0.01 0.14 0.03 0.01 <u>0.01</u> 0.01	RA-2572/06 0346-06
Italy, 2006 Catania (Violetto Catanese)	2	0.2	0.025	800	Curd	-0 0 3 7 14 20	0.13 0.89 0.48 0.19 <u>0.05</u> 0.01	RA-2572/06 0472-06
France, 2007 Graveson (Thalassa)	2	0.2	0.0334	600	Curd	-0 0 3 7 14 21	< 0.01 0.08 0.06 0.02 < 0.01 <u>0.01</u>	RA-2526/07 0068-07
Spain, 2007 Alginet (Movidick)	2	0.2	0.04 0.025	500 800	Plant ^a Curd	-0 0 3 7 14 21	0.4 1.6 1.6 < 0.01 <u>< 0.01</u> < 0.01	RA-2526/07 0584-07

CAULIFLOWER Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	No	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
Italy, 2007 Ladispoli (Meridien F1)	2	0.2	0.033	600	Curd	–0 0 3 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01 <u>< 0.01</u> < 0.01	RA-2526/07 0585-07
France, 2008 Picardie (Tango)	2	0.2	0.067	300	Curd	–0 0 3 7 14 21	< 0.01 0.1 0.09 0.07 0.03 <u>0.05</u>	08-2064 08-2064-01
Germany, 2008 Langenfeld-Reusrath (Freemont)	2	0.2	0.033	600	Curd	0 14	0.57 <u>0.02</u>	08-2064 08-2064-02
Germany, 2008 Werl-Westonnen (Lecanu)	2	0.2	0.067	300	Plant ^a Curd	–0 0 3 6 14 21	< 0.01 0.87 0.05 0.02 <u>< 0.01</u> < 0.01	08-2064 08-2064-03
Netherlands, 2008 Zwaagdijk-Oost (Fremont)	2	0.2	0.04	500	Plant ^a Curd	0 14	6.5 < 0.01	08-2064 08-2064-04

^aAnalysis of the whole plant, without roots

Studies RA-2525, RA-2526, RA-2571, RA-2572 were also provided to the 2010 JMPR [2010 JMPR Fluopyram Evaluation: Table 114]

Fruiting Vegetables (Cucurbits)

Melons (except watermelon)

Results from supervised trials from Europe on greenhouse melons were provided to the Meeting. In the trials conducted in 2006 and 2007, also provided to the 2010 JMPR, and in the 2008 trials, two applications of 0.3 kg ai/ha fluopyram (SC 500 formulations) in 500–1000 L water/ha were applied at 7 day intervals to mature plants. In the 2010 trials, three foliar sprays of 0.1 kg ai/ha (SC formulations) were applied in 500–1000 L of water/ha at 14-day intervals. Applications were made using knapsack or wheel barrow sprayers with 1–3 solid-cone or hollow-cone nozzles or with mini-booms (2–12 nozzles).

Unreplicated samples of at least 12 fruit were taken from each plot, some of the larger fruit were sub-sampled in the field, with 4/12 segments frozen within 24 hours of sample collection and stored at -18 °C or below for up to 418 days before analysis of whole fruit, peel and pulp for fluopyram using LC/MS/MS Method 00984. The reported LOQ was 0.01 mg/kg and the mean recovery rates in samples spiked with 0.01 to 1.0 mg/kg ranged from 82–108% in whole fruit, 91–114% in peel and 93–133% in pulp.

Table 16 Fluopyram residues in melons from indoor trials in North and South Europe, involving foliar applications of fluopyram (SC formulations)

MELONS Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
France (S), 2006 Carpentras (Melon/ Lunastar)	2	0.3	0.03	1000	Fruit	–0	0.11	RA-2587/06 R 2006 0368/1
					Fruit	0	0.21	
					Fruit	1	0.17	
					Fruit	3	0.19	
					Fruit	7	0.08	
					Peel	3	0.38	
					Pulp	3	0.01	
Spain, 2006 Roquetas de Mar (Melon/ Vulcano)	2	0.3	0.03	1000	Fruit	–0	0.01	RA-2587/06 R 2006 0585/4
					Fruit	0	0.03	
					Fruit	1	0.03	
					Fruit	3	0.02	
					Fruit	7	0.04	
					Peel	3	0.05	
					Pulp	3	< 0.01	
Italy, 2006 Manfredonia (Melon/ Proteo)	2	0.3	0.0375	800	Fruit	–0	0.07	RA-2587/06 R 2006 0586/2
					Fruit	0	0.11	
					Fruit	1	0.06	
					Fruit	3	0.11	
					Fruit	7	0.06	
					Peel	3	0.21	
					Pulp	3	< 0.01	
Germany, 2006 Euskirchen-DomEsch (Melon/ Haon)	2	0.3	0.05	600	Fruit	–0	0.01	RA-2587/06 R 2006 0587/06
					Fruit	0	0.25	
					Fruit	1	0.12	
					Fruit	3	0.17	
					Fruit	7	0.02	
					Peel	3	0.26	
					Pulp	3	< 0.01	
France (S), 2006 Castelsarrasin (Melon/ Edgar)	2	0.3	0.03	1000	Fruit	–0	0.09	RA-2587/06 R 2006 0589/7
					Fruit	0	0.12	
					Fruit	1	0.21	
					Fruit	3	0.16	
					Fruit	7	0.1	
					Peel	3	0.76	
					Pulp	3	0.02	
Portugal, 2006 São Bartolomeu- Lourinhã (Melon/ Jaliscas)	2	0.3	0.03	1000	Fruit	–0	0.06	RA-2587/06 R 2006 0591/9
					Fruit	0	0.12	
					Fruit	1	0.08	
					Fruit	3	0.09	
					Fruit	7	0.06	
					Peel	3	0.16	
					Pulp	3	< 0.01	
Spain, 2007 Sanlúcar de Barrameda (Primal) Muskmelon	2	0.3	0.03	1000	Fruit	–0	0.06	RA-2680/07 R 2007 0877/7
					Fruit	0	0.14	
					Fruit	1	0.09	
					Peel	3	0.23	
					Pulp	3	0.01	
					Fruit	7	0.08	

MELONS Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
France, 2008 Castelsarrasin (Strato) Melon	2	0.3	0.06	500	Fruit	–0	0.02	08-2101 08-2101-01 500SC-1 133% recovery in pulp spiked at 0.01 mg/kg (n=1)
					Fruit	0	0.05	
					Fruit	1	0.05	
					Fruit	3	0.06	
					Fruit	7	0.06	
					Fruit	10	0.06	
					Peel	1	0.22	
					Peel	3	0.24	
					Peel	7	0.18	
					Pulp	1	< 0.01	
					Pulp	3	< 0.01	
					Pulp	7	0.01	
France, 2008 Castelsarrasin (Strato) Melon	2	0.3	0.06	500	Fruit	–0	0.03	08-2101 08-2101-02 500SC-2 133% recovery in pulp spiked at 0.01 mg/kg (n=1)
					Fruit	0	0.09	
					Fruit	1	0.07	
					Fruit	3	0.06	
					Fruit	7	0.07	
					Fruit	10	0.05	
					Peel	1	0.13	
					Peel	3	0.17	
					Peel	7	0.14	
					Pulp	3	< 0.01	
					Pulp	7	< 0.01	
					Pulp	7	0.01	
Spain, 2008 Almerimar (Cleo) Melon	2	0.3	0.03	1000	Fruit	–0	0.05	08-2101 08-2101-03 500SC-1 133% recovery in pulp spiked at 0.01 mg/kg (n=1)
					Fruit	0	0.09	
					Fruit	1	0.12	
					Fruit	3	0.12	
					Fruit	7	0.1	
					Fruit	10	0.12	
					Peel	1	0.25	
					Peel	3	0.31	
					Peel	7	0.22	
					Pulp	1	< 0.01	
					Pulp	3	< 0.01	
					Pulp	7	< 0.01	
Spain, 2008 Almerimar (Cleo) Melon	2	0.3	0.03	1000	Fruit	–0	0.05	08-2101 08-2101-04 500SC-2 133% recovery in pulp spiked at 0.01 mg/kg (n=1)
					Fruit	0	0.09	
					Fruit	1	0.09	
					Fruit	3	0.08	
					Fruit	7	0.08	
					Fruit	10	0.07	
					Peel	1	0.19	
					Peel	3	0.22	
					Peel	7	0.19	
					Pulp	1	< 0.01	
					Pulp	3	< 0.01	
					Pulp	7	< 0.01	

MELONS Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
Spain, 2010 El Esido (Brisa) Melon	3	0.1	0.01	1000	Fruit	–0	0.02	10-2192 10-2192-01
					Fruit	0	0.03	
					Fruit	1	0.03	
					Fruit	3	0.02	
					Fruit	7	0.02	
					Fruit	10	0.02	
Italy, 2010 Manfredonia (Talento Clause) Melon	3	0.1	0.01	1000	Fruit	–0	0.05	10-2192 10-2192-02
					Fruit	0	0.13	
					Fruit	1	0.1	
					Fruit	3	0.12	
					Fruit	7	0.08	
					Fruit	10	0.04	
France, 2010 Chazay d Azergues (Anosta F1)	3	0.1	0.011	900	Fruit	–0	< 0.01	10-2192 10-2192-03
					Fruit	0	< 0.01	
					Fruit	1	< 0.01	
					Fruit	3	< 0.01	
					Fruit	7	< 0.01	
					Fruit	10	< 0.01	
Netherlands, 2010 Hanselersdyk (Haon) Melon	3	0.1	0.02	500	Fruit	–0	0.07	10-2192 10-2192-04
					Fruit	0	0.07	
					Fruit	1	0.06	
					Fruit	3	0.05	
					Fruit	7	0.06	
					Fruit	10	0.07	

Studies RA-2587, RA-2680 were also provided to the 2010 JMPR [2010 JMPR Fluopyram Evaluation: Table 122]

Results from supervised trials from Europe on outdoor melons were provided to the Meeting. These trials, conducted in 2006 and 2007, were also provided to the 2010 JMPR and involved two applications of 0.25–0.3 kg ai/ha fluopyram (SC formulations) in 500–1000 L/water/ha, applied at 7 day intervals to mature plants using knapsack or wheel barrow sprayers with 1–3 solid-cone or hollow-cone nozzles or with mini-booms (3–10 nozzles).

Unreplicated samples of at least 12 fruit were taken from each plot, some of the larger fruit were sub-sampled in the field, with 4/12 segments frozen within 24 hours of being sampled, and stored at -18 °C or below for up to 313 days before analysis of whole fruit, peel and pulp for fluopyram using LC/MS/MS Method 00984. The reported LOQ was 0.01 mg/kg and the mean recovery rates in samples spiked with 0.01 to 1.0 mg/kg ranged from 85–106% in whole fruit, 81–103% in peel and 93–132% in pulp.

Table 17 Fluopyram residues in melons from outdoor trials in North and South Europe, involving foliar applications of fluopyram (SC formulations)

MELONS Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
Greece, 2006 Aronas – Pieria (Melon/ Velos F1)	2	0.3	0.06	500	Fruit	–0	0.17	RA-2587/06 R 2006 0590/0,
					Fruit	0	0.42	
					Fruit	1	0.24	
					Fruit	7	0.23	
					Peel	3	0.5	
					Pulp	3	< 0.01	

MELONS Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
France(N), 2006 Antogny le Tillac (Edgar) Muskmelon	2	0.25	0.025	1000	Fruit	−0	0.08	RA-2588/06 R 2006 0370/3
					Fruit	0	0.23	
					Fruit	1	0.18	
					Fruit	3	0.12	
					Fruit	7	0.04	
					Peel	3	0.21	
					Pulp	3	< 0.01	
Germany, 2006 Monheim (Summer dream F1) Muskmelon	2	0.25	0.05	500	Fruit	−0	0.04	RA-2588/06 R 2006 0592/7
					Fruit	0	0.15	
					Fruit	1	0.15	
					Fruit	3	0.11	
					Fruit	7	0.11	
					Peel	3	0.31	
					Pulp	3	0.01	
France(S), 2006, Scorbe clairvaux (Edgar) Melon	2	0.25	0.0415	600	Fruit	−0	0.03	RA-2589/06 R 2006 0371/1
					Fruit	0	0.07	
					Fruit	1	0.05	
					Fruit	3	0.08	
					Fruit	7	0.07	
					Peel	3	0.22	
					Pulp	3	< 0.01	
Greece, 2006, Epanomi (Sterlina) Melon	2	0.25	0.05	500	Fruit	−0	0.07	RA-2589/06 R 2006 0593/5
					Fruit	0	0.15	
					Fruit	1	0.05	
					Fruit	7	0.08	
					Peel	3	0.14	
					Pulp	3	0.02	
Spain, 2006, Alginet (Charentais) Melon	2	0.25	0.0315	800	Fruit	−0	< 0.01	RA-2589/06 R 2006 0594/3
					Fruit	0	0.05	
					Fruit	1	0.03	
					Fruit	3	0.04	
					Fruit	7	0.04	
					Peel	3	0.06	
					Pulp	3	< 0.01	
Italy, 2006, Bologna (Summerdream) Melon	2	0.25	0.0415	600	Fruit	−0	0.05	RA-2589/06 R 2006 0595/1
					Fruit	0	0.13	
					Fruit	1	0.07	
					Fruit	7	0.05	
					Peel	3	0.07	
					Pulp	3	< 0.01	
Greece, 2007 Aronas – Katerini (Velso) Melon	2	0.25	0.05	500	Fruit	−0	0.1	RA-2508/07 R 2007 0009/1 133% recovery in pulp spiked at 0.1 mg/kg (n=1)
					Fruit	0	0.26	
					Fruit	1	0.18	
					Fruit	3	0.21	
					Fruit	7	0.16	
					Peel	3	0.3	
					Peel	7	0.26	
					Pulp	3	< 0.01	
					Pulp	7	< 0.01	

MELONS Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	no	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
Portugal, 2007, Caniceira- Vale de Cavalos (Branco do Ribatejo) Melon	2	0.25	0.05	500	Fruit	–0	0.03	RA-2508/07 R 2007 0535/2
					Fruit	0	0.1	
					Fruit	1	0.05	
					Fruit	3	0.08	
					Fruit	7	0.05	
					Peel	3	0.15	
					Peel	7	0.13	
					Pulp	3	< 0.01	
					Pulp	7	< 0.01	

Studies RA-2508, RA-2587, RA-2588, RA-25890 also provided to the 2010 JMPR [2010 JMPR Fluopyram Evaluation: Table 123]

Results from supervised trials from Australia on outdoor melons were provided to the Meeting. These trials involved three applications of 0.075, 0.125 or 0.19 kg ai/ha fluopyram (SC 500 formulations) in 300–1000 L/water/ha, made at 5 day intervals to mature plants using motorised knapsack hand-held booms or bike-mounted 5 metre booms.

Unreplicated samples of 3–4 fruit were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 308 days before analysis of whole fruit for fluopyram using LC/MS/MS Method 00984. The reported LOQ was 0.01 mg/kg and the mean recovery rates in samples spiked with 0.01 to 1.0 mg/kg ranged from 80–119%.

Table 18 Fluopyram residues in melons from trials in Australia, involving foliar applications of fluopyram (SC formulations)

MELON Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	No	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
Australia, 2008 Bowen, QLD (Frontier) Rockmelon	3	0.077	0.023	328	Fruit	–0	< 0.01	BCS-0242 BCS-0242- C312/T2
					Fruit	0	0.04	
					Fruit	1	0.06	
					Fruit	3	0.06	
					Fruit	7	0.11	
					Fruit	10	0.02	
					Fruit	21	0.04	
Australia, 2008 Bowen, QLD (Frontier) Rockmelon	3	0.128	0.039	328	Fruit	–0	0.05	BCS-0242 BCS-0242- C312/T3
					Fruit	0	0.25	
					Fruit	1	0.1	
					Fruit	3	0.12	
					Fruit	7	0.06	
					Fruit	10	0.06	
					Fruit	21	0.07	
Australia, 2008 Bowen, QLD (Frontier) Rockmelon	3	0.191	0.058	328	Fruit	–0	0.06	BCS-0242 BCS-0242- C312/T4
					Fruit	0	0.14	
					Fruit	1	0.1	
					Fruit	3	0.14	
					Fruit	7	0.17	
					Fruit	10	0.12	
					Fruit	21	0.11	

MELON Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	No	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
Australia, 2008 Bowen, QLD (Hotshot) Rockmelon	3	0.077	0.023	328	Fruit	-0	0.03	BCS-0242 BCS-0242- C313/T2
					Fruit	0	0.03	
					Fruit	1	0.02	
					Fruit	3	0.03	
					Fruit	7	0.04	
					Fruit	10	0.04	
					Fruit	21	0.05	
Australia, 2008 Bowen, QLD (Hotshot) Rockmelon	3	0.128	0.039	328	Fruit	-0	0.05	BCS-0242 BCS-0242- C313/T3
					Fruit	0	0.08	
					Fruit	1	< 0.01	
					Fruit	3	0.09	
					Fruit	7	0.04	
					Fruit	10	0.08	
					Fruit	21	0.07	
Australia, 2008 Bowen, QLD (Hotshot) Rockmelon	3	0.191	0.058	328	Fruit	-0	0.06	BCS-0242 BCS-0242- C313/T4
					Fruit	0	0.19	
					Fruit	1	0.2	
					Fruit	3	0.13	
					Fruit	7	0.05	
					Fruit	10	0.11	
					Fruit	21	0.11	
Australia, 2009 Waikerie, SA (Hales Bett) Rockmelon	3	0.0736	0.008	960	Fruit	-0	< 0.01	BCS-0252 C373/T2
					Fruit	0	< 0.01	
					Fruit	1	0.01	
					Fruit	3	< 0.01	
					Fruit	7	< 0.01	
					Fruit	10	< 0.01	
					Fruit	24	< 0.01	
Australia, 2009 Waikerie, SA (Hales Bett) Rockmelon	3	0.1226	0.013	960	Fruit	-0	< 0.01	BCS-0252 C373/T3
					Fruit	0	0.01	
					Fruit	1	0.03	
					Fruit	3	< 0.01	
					Fruit	7	0.02	
					Fruit	10	< 0.01	
					Fruit	24	< 0.01	
Australia, 2009 Waikerie, SA (Hales Bett) Rockmelon	3	0.1839	0.019	960	Fruit	-0	0.02	BCS-0252 C373/T4
					Fruit	0	0.03	
					Fruit	1	< 0.01	
					Fruit	3	0.01	
					Fruit	7	0.01	
					Fruit	10	0.02	
					Fruit	24	0.01	
Australia, 2009 Millendon, WA (Sterling) Rockmelon	3	0.073	0.022	332	Fruit	-0	0.01	BCS-0252 C374/T2
					Fruit	0	0.08	
					Fruit	1	0.03	
					Fruit	3	0.06	
					Fruit	7	0.01	
					Fruit	10	0.03	
					Fruit	20	0.03	
Australia, 2009 Millendon, WA (Sterling) Rockmelon	3	0.121	0.036	332	Fruit	-0	0.08	BCS-0252 C374/T3
					Fruit	0	0.1	
					Fruit	1	0.01	
					Fruit	3	0.02	
					Fruit	7	0.03	
					Fruit	10	0.01	
					Fruit	20	0.10	

MELON Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	No	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
Australia, 2009 Millendon, WA (Sterling) Rockmelon	3	0.182	0.055	332	Fruit	-0	0.06	BCS-0252 C374/T4
					Fruit	0	0.14	
					Fruit	1	0.1	
					Fruit	3	0.04	
					Fruit	7	0.08	
					Fruit	10	0.04	
					Fruit	20	0.08	
Australia, 2009 Bowen, QLD (Frontier) Rockmelon	3	0.078	0.02	400	Fruit	-0	0.07	BCS-0252 C401/T2
					Fruit	0	0.06	
					Fruit	1	0.11	
					Fruit	3	0.06	
					Fruit	7	0.06	
					Fruit	10	0.1	
					Fruit	21	0.03	
Australia, 2009 Bowen, QLD (Frontier) Rockmelon	3	0.127	0.032	400	Fruit	-0	0.12	BCS-0252 C401/T3
					Fruit	0	0.55	
					Fruit	1	0.2	
					Fruit	3	0.28	
					Fruit	7	0.1	
					Fruit	10	0.1	
					Fruit	21	0.02	
Australia, 2009 Bowen, QLD (Frontier) Rockmelon	3	0.192	0.048	400	Fruit	-0	0.05	BCS-0252 C401/T4
					Fruit	0	0.19	
					Fruit	1	0.21	
					Fruit	3	0.05	
					Fruit	7	0.07	
					Fruit	10	0.33	
					Fruit	21	0.06	
Australia, 2009 Millendon, WA (Sterling) Rockmelon	3	0.072	0.025	289	Fruit	-0	0.02	BCS-0252 C402/T2
					Fruit	0	0.03	
					Fruit	1	0.03	
					Fruit	3	0.03	
					Fruit	7	0.03	
					Fruit	10	0.02	
					Fruit	19	0.02	
Australia, 2009 Millendon, WA (Sterling) Rockmelon	3	0.121	0.042	289	Fruit	-0	< 0.01	BCS-0252 C402/T3
					Fruit	0	0.07	
					Fruit	1	0.06	
					Fruit	3	0.06	
					Fruit	7	0.05	
					Fruit	10	0.04	
					Fruit	19	0.01	
Australia, 2009 Millendon, WA (Sterling) Rockmelon	3	0.182	0.063	289	Fruit	-0	0.02	BCS-0252 C402/T4
					Fruit	0	0.1	
					Fruit	1	0.04	
					Fruit	3	0.06	
					Fruit	7	0.07	
					Fruit	10	0.05	
					Fruit	19	0.04	

Results from supervised trials from the USA on musk melons were provided to the 2010 Meeting. In these trials, two applications of 0.24–0.26 kg ai/ha (SC 500 formulation) were applied to melon plants either by foliar application or by drip line irrigation. In both plots the interval between applications was 5 days.

In the plots treated by foliar application, CO₂ plot sprayers or backpack sprayers were used to apply 139–172 L/spray mix/ha. In the drip line irrigation treated plots, fluopyram concentrations

equivalent to 16–29 kg ai/100 L were applied at rates of 3.3–6.1 L per emitter per hour, to achieve an application rate equivalent to 0.25 kg ai/ha.

Duplicate samples of mature melons (12 fruit from 12 areas within each plot) were taken from each plot, frozen within 3.5 hours of sampling and held in frozen storage for up to 219 days before being analysed for fluopyram using LC/MS/MS Method GM-001-P07-01 (LOQ 0.01 mg/kg).

Table 19 Fluopyram residues in melons from trials in the USA, involving foliar applications of fluopyram (SC formulations).

MELON Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference
	No	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	mean	
USA, 2007 Sycamore, GA (Edisto 74) Muskmelon	2	0.252 0.246	0.155 0.143	163 172	Fruit	0	0.07, 0.08	0.07	RAGMP082 GM055-07HA/A
USA, 2007 Gardner, ND (Minnesota Midget) Muskmelon	2	0.263 0.262	0.177 0.179	149 146	Fruit	0	0.24, 0.45	0.35	RAGMP082 GM056-07HA/A
USA, 2007 East Bernard, TX (Mainstream) Muskmelon	2	0.248 0.254	0.178 0.176	139 144	Fruit	0	0.35, 0.53	0.44	RAGMP082 GM057-07HA/A
USA, 2007 Orland, CA (Hales Best) Muskmelon	2	0.248 0.251	0.177 0.178	140 141	Fruit	0	0.08, 0.07	0.08	RAGMP082 GM058-07DA/A
					Fruit	1	0.16, 0.1	0.13	
					Fruit	3	0.14, 0.12	0.13	
					Fruit	7	0.15, 0.14	0.14	
					Fruit	10	0.08, 0.13	0.11	
USA, 2007 Sanger, CA (Hales Best Jumbo) Muskmelon	2	0.255 0.251	0.159 0.159	160 158	Fruit	0	0.1, 0.17	0.14	RAGMP082 GM059-07HA/A
USA, 2007 Porterville, CA (Top Mark) Muskmelon	2	0.25 0.25	0.176 0.177	142 141	Fruit	0	0.25, 0.22	0.23	RAGMP082 GM060-07HA/A

Study RAGMP082 was also provided to the 2010 JMPR [2010 JMPR Fluopyram Evaluation: Table 120]

Watermelon

Results from supervised trials from Europe on outdoor watermelons were provided to the Meeting. In these trials, two applications of 0.25 kg ai/ha fluopyram (SC 500 formulations) were applied in 500–1000 L/water/ha at 7 day intervals to mature plants using knapsack mini-booms (1–8 nozzles).

Unreplicated samples of at least 12 fruit were taken from each plot, some of the larger fruit were sub-sampled in the field, with 4–6/12 segments frozen within 24 hours of sampling and stored at -18 °C or below for up to 338 days before analysis of whole fruit, peel and pulp for fluopyram using LC/MS/MS Method 00984. The reported LOQ was 0.01 mg/kg and the mean recovery rates in samples spiked with 0.01 to 1.0 mg/kg ranged from 97–109% in whole fruit, 86–106% in peel and 93–110% in pulp.

Table 20 Fluopyram residues in watermelon from trials in North and South Europe, involving foliar applications of fluopyram (SC formulations)

WATERMELON Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	No	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
Germany, 2008 Meckenbeuren (Crimson sweet) Watermelon	2	0.25	0.05	500	Fruit	–0	0.02	08-2042 08-2042-01
					Fruit	0	0.12	
					Fruit	1	0.12	
					Fruit	3	0.12	
					Peel	3	0.19	
					Pulp	3	< 0.01	
					Fruit	7	0.07	
					Peel	7	0.13	
					Pulp	7	< 0.01	
Germany, 2008 Monheim (Crisby) Watermelon	2	0.25	0.05	500	Fruit	–0	0.02	08-2042 08-2042-02
					Fruit	0	0.14	
					Fruit	1	0.12	
					Fruit	3	0.15	
					Peel	3	0.24	
					Pulp	3	< 0.01	
					Fruit	7	0.11	
					Peel	7	0.18	
					Pulp	7	< 0.01	

*Leafy vegetables**Lettuce, leaf and head*

Results from supervised trials from Europe on field and protected lettuce were provided to the Meeting. Trials conducted on outdoor crops and on indoor lettuce in 2006 and 2007 were also provided to the 2010 JMPR. In these trials, two foliar applications of fluopyram (SC 500 formulations) were applied to head or leaf lettuce (non-hearting) at a rate of 0.25 kg ai/ha in 300–1000 L of water and 7 days apart in the greenhouse trials and in 200–800 L water and 7–10 days apart in the field trials. Application was by knapsack sprayers and hand-held booms with 3–12 flat fan or hollow-cone nozzles.

In the more recent trials on protected lettuce, either two applications of 0.2 kg ai/ha, 7 days apart or one application of 0.3 kg ai/ha were applied in 300–1000 L spray mix/ha using knapsack and mini-boom (3–8 nozzle) sprayers.

Unreplicated samples of at least 12 lettuce heads were taken from each plot, frozen within 24 hours of sampling and stored at –18 °C or below for up to 520 days before analysis for fluopyram using LC/MS/MS Methods 00984 or 00984-M001. The reported LOQ was 0.01 mg/kg and the mean recovery rates in samples spiked with 0.01 to 20.0 mg/kg ranged from 72–120%.

Table 21 Fluopyram residues in outdoor leaf and head lettuce from trials in North and South Europe, involving foliar applications of fluopyram (SC formulations)

LETTUCE Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	No	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
GAP: Netherlands	2	0.25				7		7 day interval
France, 2006 Cergy (Estelle) Butterhead	2	0.25	0.05	500	Leaf	-0 0 3 7 10 13	1.9 5.9 0.87 0.62 0.46 0.34	RA-2592/06 R 2006 0375 4
Germany, 2006 Meckenbeuren (Jiska) Butterhead	2	0.25	0.05	500	Leaf	-0 0 3 7 11 14	0.08 8.2 1.9 0.18 0.05 0.01	RA-2592/06 R 2006 0604 4
Germany, 2006 Langenfeld-Reusrath (Gisela) Butterhead	2	0.25	0.042	600	Leaf	-0 0 3 7 10 14	0.2 4.3 1.3 0.93 0.47 0.30	RA-2592/06 R 2006 0605 2
Netherlands, 2006 Zwaagdijk-Oost (Namia) Butterhead	2	0.25	0.032	800	Leaf	-0 0 3 7 10 14	0.37 1.7 0.71 0.26 0.1 0.02	RA-2592/06 R 2006 0606 0
UK, 2006 Cambridgeshire (Iceburg) Crisphead	2	0.25 0.27	0.125 0.125	200 216	Head	-0 0 3 7 10 14	0.75 7.6 1.2 0.13 0.02 < 0.01	RA-2592/06 R 2006 0607 9
France, 2006 St Jory (Toucan) Butterhead	2	0.25	0.042	600	Leaf	-0 0 3 7 10 14	0.47 2.7 0.61 0.25 0.17 0.09	RA-2593/06 R 2006 0376 2
Spain, 2006 Vilanova del Vallés (Rumina) Butterhead	2	0.25 0.25	0.063 0.042	400 600	Leaf	-0 0 4 7 10 14	0.27 3.5 1.5 0.29 0.07 0.01	RA-2593/06 R 2006 0608 7
Italy, 2006 Andria (Brest) Crisphead	2	0.25	0.036	700	Head	-0 0 4 7 10 14	0.09 0.62 0.02 0.02 0.03 0.02	RA-2593/06 R 2006 0609 5
Italy, 2006 Catania (Trocadero) Butterhead	2	0.25	0.042	600	Leaf	-0 0 3 8 10 14	0.59 1.8 0.92 0.46 0.23 0.1	RA-2593/06 R 2006 0610 9

LETTUCE Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	No	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
Greece, 2006 Katerini/ Paralia (White Boston) Butterhead	2	0.25	0.05	500	Leaf	-0 0 3 8 10 14	2.1 11 6.4 1.7 0.39 0.02	RA-2593/06 R 2006 0611 7
UK, 2007 Cambridgeshire (Elenar) Loose Leaf	2	0.25	0.084	300	Leaf	-0 0 3 6 9 13	036 6.1 0.82 0.18 0.02 < 0.01	RA-2509/07 R 2007 0011/3
Germany, 2007 Meckenbeuren (Nobilan) Butterhead	2	0.27 0.25	0.084 0.084	321 300	Leaf	-0 0 2 7 10 13	0.41 4.5 3.6 0.61 0.44 0.13	RA-2509/07 R 2007 0244/2
Germany, 2007 Langenfeld-Reusrath (Torpedo)	2	0.25	0.042	600	Head	-0 0 3 7 10 14	0.85 6.1 1.1 0.57 0.43 0.33	RA-2509/07 R 2007 0537/9
France, 2007 Puzeaux (Madras)	2	0.25	0.084	300	Head	-0 0 4 7 11 15	< 0.01 6 0.12 0.12 0.04 0.02	RA-2509/07 R 2007 0538/7
Belgium, 2007 Villers-Perwin (Appia)	2	0.25	0.063	400	Head	-0 0 3 7 10 14	0.09 5.9 2.1 0.63 0.42 0.26	RA-2509/07 R 2007 0539/5
Netherlands, 2007 Zwaagdijk-Oost (Lolo Rosso)	2	0.25	0.034	750	Leaf	-0 0 3 7 10 14	0.48 6.5 1.3 0.53 0.16 0.07	RA-2509/07 R 2007 0540/9
Portugal, 2007 Olho Marinho (Faustinas)	2	0.25	0.063	400	Head	-0 0 3 7 10 14	0.31 4.5 1.3 0.48 0.24 0.05	RA-2510/07 R 2007 0012 1
Greece, 2007 Agia Marina (Atraction)	2	0.25	0.063	400	Head	-0 0 3 7 10 14	0.11 7 1.2 0.29 0.1 0.03	RA-2510/07 R 2007 0245 0

LETTUCE Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	No	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
France, 2007 Ouzilly (Santoro)	2	0.25	0.042	600	Head	–0	0.17	RA-2510/07 R 2007 0246 9
						0	6	
						3	0.56	
						7	0.27	
						10	0.14	
						14	0.12	
Italy, 2007 Manfredonia (Antony) Loose Leaf	2	0.25	0.036	700	Leaf	–0	0.8	RA-2510/07 R 2007 0541 7
						0	7.4	
						3	4.8	
						7	5.3	
						10	0.87	
						14	0.98	

Leaf=non-hearting lettuce, whole plant sampled with roots and main stalk removed

Studies RA-2509, RA-2510, RA-2592, RA-2593 were also provided to the 2010 JMPR [2010 JMPR Fluopyram

Evaluation: Table 138]

Results from supervised trials from USA on outdoor lettuce were provided to the Meeting. The trials conducted in 2007 and 2008 were also provided to the 2010 JMPR. In the trials, two foliar applications of fluopyram (SC 500 formulation) were applied to leaf lettuce or head lettuce plants at 0.24–0.26 kg ai/ha in 110–190 L of water, 4–7 days apart, using CO₂ pressurised plot sprayers or knapsack sprayers.

At each site, additional plots were treated with two narrow (3–15 cm) band spray applications of 0.24–0.26 kg ai/ha in 23–180 L of water over the top of the row, the first just after transplanting and the second being 1–5 weeks later, at the 5-leaf growth stage (BBCH 15) with treatment intervals ranging from 7 to 35 days.

In the trials conducted in 2009, a single application of 0.25 kg ai/ha (SC formulations) was applied to leaf lettuce, with and without added surfactant, using CO₂ pressurised plot sprayers or knapsack sprayers to apply 180–300 L spray mix/ha.

Duplicate samples (lettuce leaves or lettuce heads with wrapper leaves) were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 429 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01.

The reported LOQ was 0.01 mg/kg and the mean recovery rates in samples spiked with 0.01 to 10.0 mg/kg ranged from 87–101%.

Table 22 Fluopyram residues in outdoor lettuce from trials in USA, involving foliar applications of fluopyram (SC formulations)

LETTUCE Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference
	No	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	mean	
USA, 2007 Germansville, Pennsylvania (New Red Fire)	2	0.247 0.257	0.143 0.14	173 183	Leaf	0	5.4, 4.4	4.9	RAGMP085-1 GM082-07HA-A
USA, 2008 Belle Glade, Florida (SVR0071)	2	0.254 0.246	0.148 0.14	172 176	Leaf	0	1.9, 0.57	1.2	RAGMP085-1 GM083-07HA-A
USA, 2007 King City, CA (Sunbelt)	2	0.246 0.249	0.168 0.187	146 133	Leaf	0	5.0, 4.4	4.7	RAGMP085-1 GM084-07HA-A
						1	4.3, 5.2	4.8	
						3	0.7, 0.7	0.69	
						7	0.3, 0.3	0.3	
						10	0.08, 0.11	0.1	

LETTUCE Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference
	No	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	mean	
USA, 2007 Corning, CA (Unspecified)	2	0.247 0.248	0.132 0.133	187 187	Leaf	0	5.5, 6.4	5.96	RAGMP085-1 GM085-07HA-A
USA, 2008 Fresno, CA (Salad Bowl)	2	0.25 0.249	0.214 0.215	117 116	Leaf	0	9.5, 8.6	9.05	RAGMP085-1 GM086-07HA-A
USA, 2007 Porterville, CA (Salad Bowl)	2	0.248 0.258	0.15 0.142	165 182	Leaf	0	6.3, 7.2	6.8	RAGMP085-1 GM087-07HA-A
USA, 2007 Germansville, PA (Ithaca)	2	0.26 0.253	0.141 0.138	185 184	Head Trimmed	0 0	1.9, 3 0.06, 0.03	2.5 0.05	RAGMP085-1 GM088-07HA-A
USA, 2008 Belle Glade, FL (Gator)	2	0.254 0.248	0.148 0.139	172 178	Head Trimmed	0 0	1.8, 0.79 0.04, < 0.01	1.3 0.02	RAGMP085-1 GM089-07HA-A
USA, 2007 Sanger, CA (Great Lakes)	2	0.236 0.26	0.183 0.157	129 166	Head Head Head Head Head Trimmed	0 1 3 7 10 0	5.2, 5.4 2.1, 3.8 2.4, 2.1 1.1, 1.3 0.89, 0.7 0.62, 1.5	5.3 3 2.2 1.2 0.8 1.1	RAGMP085-1 GM090-07DA-A
USA, 2007 Corning, CA (Great Lake 659)	2	0.247 0.247	0.132 0.132	187 187	Head Trimmed	0 0	2.4, 2.6 0.17, 0.09	2.5 0.13	RAGMP085-1 GM091-07HA-A
USA, 2008 Fresno, CA (Great Lake 659)	2	0.251 0.249	0.215 0.215	117 116	Head Trimmed	0 0	4.7, 4 0.98, 0.71	4.4 0.85	RAGMP085-1 GM092-07HA-A
USA, 2007 Porterville, CA (Vandenburg)	2	0.25 0.251	0.227 0.226	110 111	Head Trimmed	0 0	0.35, 0.81 0.01, 0.03	0.58 0.02	RAGMP085-1 GM093-07HA-A
USA, 2009, High Springs, FL (Coastal Star)	1	0.253	0.138	184	Leaf Leaf Leaf	0 3 13	7.5, 7.4 2.5, 3.4 0.26, 0.22	7.5 2.9 0.24	RAGMP157 GM058-09HA-A
USA, 2009 High Springs, FL (Coastal Star)	1	0.252	0.138	183	Leaf Leaf Leaf	0 3 13	6.5, 7 2.4, 3.5 0.24, 0.3	6.7 2.9 0.27	RAGMP157 GM058-09HA-B (with adjuvant)
USA, 2009 Hickman, CA (Paris Island Cosmi)	1	0.247	0.0882	280	Leaf Leaf Leaf	0 3 14	2.7, 2.4 2.3, 2.5 0.93, 1.0	2.5 2.4 0.99	RAGMP157 GM059-09HA-A
USA, 2009 Hickman, CA (Paris Island Cosmi)	1	0.246	0.0885	278	Leaf Leaf Leaf	0 3 14	3.3, 3.7 1.9, 1.9 0.31, 0.85	3.5 1.9 0.58	RAGMP157 GM059-09HA-B (with adjuvant)
USA, 2009 King City, CA (Sunbelt)	1	0.247	0.0885	279	Leaf Leaf Leaf	0 3 14	5.8 4.5 1.2, 1.3 0.13, 0.11	5.1 1.3 0.12	RAGMP157 GM060-09HA-A
USA, 2009 King City, CA Sunbelt	1	0.254	0.0885	287	Leaf Leaf Leaf	0 3 14	4.2, 4.1 0.55, 0.49 0.1, 0.13	4.1 0.52 0.12	RAGMP157 GM060-09HA-B (with adjuvant)

Study RAGMP085 was also provided to the 2010 JMPR [2010 JMPR Fluopyram Evaluation: Table 135]

Table 23 Fluopyram residues in indoor head and leaf lettuce from trials in North and South Europe, involving foliar applications of fluopyram (SC formulations)

LETTUCE Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	No	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
GAP: Netherlands	2	0.25				7		7 day interval
France, 2006 Ouzilly (Arcadia) Butterhead	2	0.25	0.0415	600	Leaf	-0 0 3 7 10 14	5.2 11 1.7 <u>1.2</u> 0.86 0.51	RA-2586/06 R 2006 0367 3
France, 2006 Fondettes (Sestice) Butterhead	2	0.25	0.0415	600	Leaf	-0 0 3 7 10 14	0.51 7.3 0.66 <u>0.16</u> 0.12 0.02	RA-2586/06 R 2006 0576 5
Germany, 2006 Leichlingen (Alexandria) Head lettuce	2	0.25	0.0835	300	Head	-0 0 3 7 10 14	3.2 7.2 1.8 <u>1.9</u> 1.8 1.4	RA-2586/06 R 2006 0577 3
Germany, 2006 Meckenbeuren (Natalie) Butterhead	2	0.25	0.0835	300	Leaf	-0 0 3 7 10 14	0.89 4.3 0.99 <u>0.81</u> 0.35 0.33	RA-2586/06 R 2006 0578 1
Spain, 2006 Bigues i Riells (Trocadero) Butterhead	2	0.25 0.25	0.05 0.036	500 700	Leaf	-0 0 3 7 10 14	3.4 8.7 6.2 <u>3.8</u> 2.8 1.6	RA-2586/06 R 2006 0580 3
Italy, 2006 Monopoli (Brest) Head lettuce	2	0.25	0.0415	600	Head	-0 0 3 7 10 14	2.7 9.1 6 4.9 <u>7.7</u> 5.2	RA-2586/06 R 2006 0581 1 (24 small immature heads/1.5-2 kg samples)
Portugal, 2006 Olho Marinho (Gisela) Butterhead	2	0.25	0.05	500	Leaf	-0 0 3 7 10 14	4.6 8.6 9.1 <u>4.4</u> 3.5 1.9	RA-2586/06 R 2006 0583 8
Greece, 2006 Marathonas (Manita) Butterhead	2	0.25	0.05	500	Leaf	-0 0 3 7 10 14	6 22 11 <u>8.4</u> 7 6.5	RA-2586/06 R 2006 0584 6 (13 small heads/1.2-1.8 kg samples)

LETTUCE Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	No	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
France, 2007 St Rémy de Provence (Zandria) Head lettuce	2	0.25	0.025	1000	Head	-0 0 3 7 10 14	1.4 5.1 4 <u>2.5</u> 2.2 1.5	RA-2604/07 R 2007 0247/7
Germany, 2007 Leichlingen (Locarno) Loose leaf	2	0.25	0.0415	600	Leaf	-0 0 3 7 10 14	2 9.4 7.8 <u>2.7</u> 2.1 1.4	RA-2604/07 R 2007 0542/5
Italy, 2007 Manfredonia (Canasta rossa-Holter) Loose leaf	2	0.25	0.028	900	Leaf	-0 0 3 7 10 14	1.9 5.5 2.4 0.83 <u>2.3</u> 1.1	RA-2604/07 R 2007 0543/3
Spain, 2007 Bigues i Riells (Batavia) Loose leaf	2	0.25 0.25	0.05 0.0415	500 600	Leaf	-0 0 3 7 10 14	6.6 11 8.3 <u>7.2</u> 5.5 4.9	RA-2604/07 R 2007 0544/1
Germany, 2007 Leichlingen (Alexandria) Head lettuce	2	0.2	0.033	600	Head	-0 0 3 7 14 21	1.5 5.8 4.7 <u>2.1</u> 0.73 0.51	RA-2620/07 R 2007 0266 3
Netherlands, 2007 Wervershoof (Lolo Rosso) Loose leaf	2	0.2	0.02	1000	Leaf	-0 0 3 7 14 21	0.81 5.5 1.2 <u>0.92</u> 0.44 0.24	RA-2620/07 R 2007 0642 1
France, 2007 Ouzilly (Santoro) Head lettuce	2	0.2	0.033	600	Head	-0 0 3 7 14 21	0.39 3.4 2.3 <u>1.4</u> 0.37 0.07	RA-2620/07 R 2007 0644 8
Germany, 2007 Meckenbeuren (Alexandria) Head lettuce	2	0.2	0.067	300	Head	-0 0 3 7 13 21	0.69 3.5 0.63 <u>0.23</u> 0.19 0.07	RA-2620/07 R 2007 0645 6
Portugal, 2008, Brejenjas (Complice) Head lettuce	1	0.3	0.038	800	Head	0 3 7 11 14	14 11 10 6.4 4.8	08-2080 08-2080-01
France, 2008 Fondettes (Appia) Butterhead	1	0.3	0.05	600	Leaf	0 3 7 10 14	8.2 0.98 0.45 0.23 0.15	08-2080 08-2080-02

LETTUCE Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	No	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
Greece, 2008 Aronas/Katerini (Sanguine "Clause") Head lettuce	1	0.3	0.075	400	Head	0	15	08-2080 08-2080-03
						3	1.8	
						7	0.88	
						10	0.68	
						14	0.31	
Germany, 2008 Meckenbeuren (Mafalda) Butterhead	1	0.32	0.075	426	Leaf	0	7.1	08-2080 08-2080-04
						3	1.9	
						7	0.44	
						10	0.44	
						14	0.16	

Leaf=non-hearting lettuce, whole plant sampled with roots and main stalk removed

Studies RA-2586, RA-2604 were also provided to the 2010 JMPR [2010 JMPR Fluopyram Evaluation: Table 137]

Stalk and stem vegetables

Asparagus

Results from supervised trials from Europe on asparagus were provided to the Meeting. In these trials, two applications of 0.15–0.2 kg ai/ha fluopyram (SC formulations) in 300–800 L/water/ha of were applied at 10–11 day intervals to mature ferns (just prior to senescence) as foliar sprays using knapsack and hand lance or mini-boom (4–12 nozzle) sprayers.

Unreplicated samples of at least 1 kg or 24 spears (with soil removed and in some trials, trimmed to 20 cm length) were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 187 days before analysis for fluopyram using LC/MS/MS Method 00984. The reported LOQ was 0.01 mg/kg and the mean recovery rates in samples spiked with 0.01 to 1.0 mg/kg ranged from 72–99%.

Table 24 Fluopyram residues in asparagus from trials in North and South Europe, involving foliar applications of fluopyram (SC formulations)

ASPARAGUS Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	No	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
GAP: Switzerland	2	0.2					Pre-senescence	
Germany, 2008 Malsch – Neumalsch (Rhapsody)	2	0.2	0.033	600	Spears	209	< 0.01	08-2209 08-2209-01 17-spear sample
France, 2008 Fienvillers (Andreas)	2	0.2	0.067	300	Spears	238	< 0.01	08-2209 08-2209-02
France, 2009 Truyes Centre (Andreas white)	2	0.2	0.067	300	Spears	205	< 0.01	09-2073 09-2073-01
Netherlands, 2009 Hoogerheide Zeeland (Grolim white)	2	0.2	0.025	800	Spears	202	< 0.01	09-2073 09-2073-02
France, 2008 Le Burgaud (Larac)	3	0.15	0.025	600	Spears	164	< 0.01	08-2210 08-2210-01
Italy, 2008 Manfredonia(UC 157)	3	0.15	0.019	800	Spears	255	< 0.01	08-2210 08-2210-02

ASPARAGUS Country, Year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference
	No	kg ai/ha	kg ai/hL	Water (L/ha)			Fluopyram	
France, 2009 Truyes, Centre (Andreas white)	3	0.15	0.05	300	Spears	213	< 0.01	09-2072 09-2072-01
Germany, 2009 Neumalsch (Rhapsody white)	3	0.15	0.025	600	Spears	225	< 0.01	09-2072 09-2072-02
Belgium, 2009 Gembloux (Thiclim white)	2+ 1	0.15 0.15	0.025 0.025	600 639	Spears	202	< 0.01	09-2072 09-2072-03
Netherlands, 2009 Hoogerheide (Grolim white)	3	0.15	0.019	800	Spears	202	< 0.01	09-2072 09-2072-04
France, 2009 Ouzilly (Blackim white)	3	0.15	0.025	600	Spears	188	< 0.01	09-2072 09-2072-05
Spain, 2009 Talayueta (Grolim green)	3	0.15	0.019	800	Spears	183	< 0.01	09-2072 09-2072-06

Oilseeds

Rape seed

Results from supervised trials on winter and summer rape in Europe were provided to the Meeting. The trials conducted in 2006 and 2007 were also provided to the 2010 Meeting. In these trials, two applications of fluopyram (SC 500 formulation or as a 250 SC formulation, in combination with prothioconazole) were applied to rape plants at BBCH 63 (30% flowers open) and again, 14–39 days later, at BBCH 73 (30% pods at full size), as foliar sprays using knapsack sprayers with spray booms (3–12 flat fan nozzles), applying 0.125 kg ai/ha in 200–400 L/water/ha. Plot sizes in these trials ranged from 48–563 square metres.

Unreplicated samples of mature seeds (min 1 kg), pods and seeds (min 0.7 kg) and/or plants (min 2 kg green material, including pods and seeds) were taken from each plot, frozen within 24 hours of sampling and stored at -18 °C or below for up to 15 months before analysis using LC/MS/MS Method 00984 (with minor variations). The reported LOQ was 0.01 mg/kg.

Table 25 Residues in oilseed rape from supervised trials in France, Germany, Italy and UK, involving two foliar applications of fluopyram (SC formulations)

RAPE SEED Country, year Location (variety)	Application					DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)	GS last application		matrix	Fluopyram	
GAP: Ukraine	2	0.113		200–400		30			
Germany, 2006 Werl-Westönnen (Smart) winter rape	2	0.125	0.0415	300	BBCH 73	50	seed	0.11	RA-2609/06 0406-06
France (N), 2006 Beuvraignes (Salomon) winter rape	2	0.125	0.05	250	BBCH 73	66	seed	0.02	RA-2609/06 0408-06

RAPE SEED Country, year Location (variety)	Application					DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)	GS last application		matrix	Fluopyram	
Germany, 2006 Burscheid (Talent) winter rape	2	0.125	0.0415	300	BBCH 73	57	seed	0.04	RA-2609/06 0409-06
United Kingdom, 2006 Royston (Canberra) winter rape	2	0.125	0.0625	200	BBCH 73	-0 0 43	plant plant seed	0.04 1.4 0.1	RA-2609/06 0410-06
France (N), 2007 Braslou (Grizzly) winter rape	2	0.125	0.0416	300	BBCH 73	50	seed	0.09	RA-2616/07 0238-07
Germany, 2007 Burscheid (Elektra) winter rape	2	0.125	0.0416	300	BBCH 73	69	seed	0.08	RA-2616/07 0808-07
Germany, 2007 Werl-Westönnen (Oase) winter rape	2	0.125	0.0416	300	BBCH 73	61	seed	0.11 (c=0.02)	RA-2616/07 0809-07
United Kingdom, 2007 Near Eye (Es Astrid) winter rape	2	0.125	0.0416	300	BBCH 73	57	seed	0.19 (c=0.01)	RA-2616/07 0810-07
France (S), 2006 Latille (Savana) winter rape	2	0.125	0.0415	300	BBCH 73	50	seed	0.06	RA-2610/06 0625-06
Italy, 2006 Bologna (Molino) summer rape	2	0.125	0.038	330	BBCH 73	34	seed	0.07	RA-2610/06 0626-06
France (S), 2007 Gargas (Corail) winter rape	2	0.125	0.0416	300	BBCH 73	59	seed	0.1	RA-2617/07 0239-07
Italy, 2007 Conselice (Belcanto) winter rape	2	0.125	0.0313	400	BBCH 73	40	seed	0.14	RA-2617/07 0811-07
Belgium, 2010 Cortil-Noirmont (Exocet) winter rape	2	0.125	0.063	200	BBCH 78	28	seed	0.61	10-2134 10-2134-01

RAPE SEED Country, year Location (variety)	Application					DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)	GS last application		matrix	Fluopyram	
UK, 2010 Royston (Cabernet) winter rape	2	0.125	0.063	200	BBCH 78	28	seed	0.27	10-2134 10-2134-02
Netherlands, 2010 Slootdrop (Haydn) summer rape	2	0.125	0.042	300	BBCH 73	28	seed	0.35	10-2134 10-2134-03
France (N), 2010 Bouafle (Olindigo) summer rape	2	0.125	0.042	300	BBCH 72	21	seed	0.47	10-2134 10-2134-04
Spain, 2010 Vilobi d'Onyar (Artist-Hybrid) winter rape	2	0.125 0.136	0.042 0.042	300 327	BBCH 78	28	seed	0.38	10-2134 10-2134-05
France (S), 2010 Vouille (Aviator) winter rape	2	0.125	0.042	300	BBCH 78	27	seed	0.14	10-2134 10-2134-06
Spain, 2010 Malaga (Kavel) winter rape	2	0.125	0.063	200	BBCH 72	28	seed	0.25	10-2244 10-2244-01
France (S), 2010 Gontaud de Nogaret (Hybri Star) winter rape	2	0.125	0.05	250	BBCH 79	29	seed	0.46	10-2244 10-2244-02
Germany, 2011 Burscheid (Elektra) winter rape	2	0.125	0.042	300	BBCH 79	48	seed	0.26	11-2003 11-2003-01
Belgium, 2011 Sombreffe (Limone Hybrid) winter rape	2	0.125	0.063	200	BBCH 80	29	seed	0.29	11-2003 11-2003-02
UK, 2011 Royston (Flash Hybrid) winter rape	2	0.125	0.063	200	BBCH 79	28	seed	0.1	11-2003 11-2003-03
France (N), 2011 Chambourg sur Indre (Dynastie) winter rape	2	0.125	0.042	300	BBCH 79	27	seed	0.34	11-2003 11-2003-04

RAPE SEED Country, year Location (variety)	Application					DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)	GS last application		matrix	Fluopyram	
Spain, 2011 La Luisiana (Eswilliams) winter rape	2	0.125	0.042	300	BBCH 72	28	seed	0.38	11-2124 11-2124-01
France (N), 2011 Boulac (Nk Alamir) winter rape	2	0.125	0.042	300	BBCH 79	28	seed	0.33	11-2124 11-2124-02
Italy, 2011 Tarquinia (Hybridstar) winter rape	2	0.125	0.042	300	BBCH 75	28	seed	0.27	11-2124 11-2124-03
Spain, 2011 Llerona (Pacific) winter rape	2	0.125	0.042	300	BBCH 78	28	seed	0.46	11-2124 11-2124-04

Studies RA-2609, RA-2616, RA-2610, RA-2617 were also provided to the 2010 JMPR [2010 JMPR Fluopyram Evaluation: Table 177]

Results from supervised trials from USA on oilseed rape (canola) were provided to the Meeting. These trials were also provided to the 2010 Meeting. In these trials, two applications of 0.242–0.256 kg ai/ha (SC 500 formulation) were applied to canola, 13–14 days apart from flowering as foliar sprays using CO₂ plot sprayers or knapsack sprayers with hand-held spray booms, field sprayers or tractor-mounted spray booms to apply 92–184 L/spray mix/ha. Plot sizes in these trials ranged from 70–210 square metres.

Duplicate samples of rape seed (min 1 kg) were taken from at least 12 areas within each plot, frozen within 3 hours of sampling and held in frozen storage for up to 516 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg.

Table 26 Fluopyram residues in rape seed from supervised trials in USA involving two foliar applications (500 SC formulations)

RAPE SEED Country, year Location (variety)	Application					DALA	Fluopyram residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)	GS last application		Fluopyram	mean	
USA, 2007 Sycamore, GA (Hyola)	2	0.25 0.242	0.184 0.161	136 150	BBCH 88	13	0.12, 0.14	0.13	RAGMP035 GM012-06HA
USA, 2006 Sabin, MN (5630)	2	0.256 0.252	0.16 0.144	160 175	BBCH 86	14	0.09, 0.1	0.1	RAGMP035 GM014-06HA
USA, 2006 Eledridge, ND (Hyola357RR)	2	0.252 0.252	0.187 0.177	135 142	BBCH 79	14	0.11, 0.11	0.11	RAGMP035 GM015-06HA
USA, 2006 Velva, ND (Hyola 357RR Magnum)	2	0.253 0.248	0.209 0.203	121 122	BBCH 80	14	0.22, 0.17	0.2	RAGMP035 GM016-06HA

RAPE SEED Country, year Location (variety)	Application					DALA	Fluopyram residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)	GS last application		Fluopyram	mean	
USA, 2006 Jerome, ID (Phoenix)	2	0.247 0.249	0.143 0.15	173 166	BBCH 84	12	2.8, 3.0	2.9	RAGMP035 GM017-06HA
USA, 2006 Madras, OR (CrackerJack)	2	0.252 0.25	0.215 0.216	117 116	BBCH 89	14	0.44, 0.41	0.43	RAGMP035 GM018-06HA
USA, 2006 Ephrata, WA (47755 / 65037)	2	0.248 0.248	0.14 0.139	177 178	BBCH 72	14	0.09, 0.14	0.11	RAGMP035 GM019-06HA
USA, 2006 Springfield, NE (Invigor 4870 LL)	2	0.249 0.248	0.196 0.195	127 127	BBCH 89	0	1.6, 1.6	1.6	RAGMP035 GM013-06DA
						6	0.2, 0.19	0.2	
						12	0.14 0.15	0.14	
						19	0.12, 0.157	0.14	
						26	0.2, 0.18	0.19	

Studies RAGMP035 was also provided to the 2010 JMPR [2010 JMPR Fluopyram Evaluation: Table 178]

Animal feeds

Rape forage

Results from supervised trials on winter and summer rape in Europe were provided to the Meeting. The trials conducted in 2006 and 2007 were also provided to the 2010 Meeting. In these trials, two applications of fluopyram (SC 500 formulation or as a 250 SC formulation, in combination with prothioconazole) were applied to rape plants at BBCH 63 (30% flowers open) and again, 14–39 days later, at BBCH 73 (30% pods at full size), as foliar sprays using knapsack sprayers with spray booms (3–12 flat fan nozzles), applying 0.125 kg ai/ha in 200–400 L/water/ha. Plot sizes in these trials ranged from 48–563 square metres.

Unreplicated samples of pods and plants (min 2 kg green material, including pods and seeds) were taken from each plot, frozen within 24 hours of sampling and stored at –18 °C or below for up to 15 months before analysis using LC/MS/MS Method 00984 (with minor variations). The reported LOQ was 0.01 mg/kg.

Table 27 Residues in oilseed rape plants from supervised trials in France, Germany, Italy and UK, involving two foliar applications of fluopyram (SC formulations)

OILSEED RAPE FORAGE Country, year Location (variety)	Application					DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)	GS last application		matrix	Fluopyram	
GAP: Ukraine	2	0.113		200–400		30			
Germany, 2006 Werl-Westönnen (Smart) winter rape	2	0.125	0.0415	300	BBCH 73	–0 0 12 41	plant plant plant plant	0.06 1.6 0.41 0.1	RA-2609/06 0406-06
France (N), 2006 Beuvraignes (Salomon) winter rape	2	0.125	0.05	250	BBCH 73	–0 0 29 54	plant plant plant plant	0.06 1 0.12 0.04	RA-2609/06 0408-06

OILSEED RAPE FORAGE Country, year Location (variety)	Application					DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)	GS last application		matrix	Fluopyram	
Germany, 2006 Burscheid (Talent) winter rape	2	0.125	0.0415	300	BBCH 73	-0 0	plant plant	0.07 1.6	RA-2609/06 0409-06
United Kingdom, 2006 Royston (Canberra) winter rape	2	0.125	0.0625	200	BBCH 73	-0 0	plant plant	0.04 1.4	RA-2609/06 0410-06
France (N), 2007 Braslou (Grizzly) winter rape	2	0.125	0.0416	300	BBCH 73	-0 0 14 36	plant plant plant pod	0.27 0.98 0.45 0.3	RA-2616/07 0238-07
Germany, 2007 Burscheid (Elektra) winter rape	2	0.125	0.0416	300	BBCH 73	-0 0 14 49	plant plant plant pod	0.45 0.91 0.36 0.07	RA-2616/07 0808-07
Germany, 2007 Werl-Westönnen (Oase) winter rape	2	0.125	0.0416	300	BBCH 73	-0 0 17 44	plant plant plant pod	0.05 0.75 0.29 0.37	RA-2616/07 0809-07
United Kingdom, 2007 Near Eye (Es Astrid) winter rape	2	0.125	0.0416	300	BBCH 73	-0 0 25 46	plant plant plant pod	0.18 0.91 0.31 0.13	RA-2616/07 0810-07
France (S), 2006 Latille (Savana) winter rape	2	0.125	0.0415	300	BBCH 73	-0 0 11 35	plant plant plant plant	0.23 1.1 0.2 0.09	RA-2610/06 0625-06
Italy, 2006 Bologna (Molino) summer rape	2	0.125	0.038	330	BBCH 73	-0 0 20 27	plant plant plant plant	0.25 1.1 0.1 0.35	RA-2610/06 0626-06
France (S), 2007 Gargas (Corail) winter rape	2	0.125	0.0416	300	BBCH 73	-0 0 10	plant plant plant	1.1 0.95 0.31	RA-2617/07 0239-07
Italy, 2007 Conselice (Belcanto) winter rape	2	0.125	0.0313	400	BBCH 73	-0 0 14 30	plant plant plant pod	0.26 0.87 0.71 0.71	RA-2617/07 0811-07

OILSEED RAPE FORAGE Country, year Location (variety)	Application					DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)	GS last application		matrix	Fluopyram	
Belgium, 2010 Cortil-Noirmont (Exocet) winter rape	2	0.125	0.063	200	BBCH 78	-0	plant	0.59	10-2134 10-2134-01
						0	plant	1.7	
						14	pod	1.7	
						21	pod	1.7	
						14	foliage	0.8	
						21	foliage	0.4	
UK, 2010 Royston (Cabernet) winter rape	2	0.125	0.063	200	BBCH 78	-0	plant	1	10-2134 10-2134-02
						0	plant	2.3	
						14	pod	3.4	
						22	pod	3.1	
						14	foliage	1.4	
						22	foliage	0.89	
Netherlands, 2010 Slootdrop (Haydn) summer rape	2	0.125	0.042	300	BBCH 73	-0	plant	0.68	10-2134 10-2134-03
						0	plant	2.7	
						14	pod	2.5	
						21	pod	2.8	
						14	foliage	0.36	
						21	foliage	0.22	
France (N), 2010 Bouafle (Olindigo) summer rape	2	0.125	0.042	300	BBCH 72	-0	plant	2.1	10-2134 10-2134-04
						0	plant	6.7	
						14	pod	11	
						14	foliage	6.4	
Spain, 2010 Vilobi d'Onyar (Artist-Hybrid) winter rape	2	0.125 0.136	0.042 0.042	300 327	BBCH 78	-0	plant	0.16	10-2134 10-2134-05
						0	plant	1.3	
						14	pod	0.91	
						14	foliage	0.25	
France (S), 2010 Vouille (Aviator) winter rape	2	0.125	0.042	300	BBCH 78	-0	plant	0.78	10-2134 10-2134-06
						0	plant	2.1	
						14	pod	1.8	
						21	pod	1.3	
						14	foliage	0.17	
						21	foliage	0.11	
Spain, 2010 Malaga (Kavel) winter rape	2	0.125	0.063	200	BBCH 72	-0	plant	0.8	10-2244 10-2244-01
						0	plant	2.5	
						14	pod	0.37	
						21	pod	1.7	
						14	foliage	0.24	
						21	foliage	0.16	

OILSEED RAPE FORAGE Country, year Location (variety)	Application					DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)	GS last application		matrix	Fluopyram	
France (S), 2010 Gontaud de Nogaret (Hybri Star) winter rape	2	0.125	0.05	250	BBCH 79	-0 0	plant plant	1.2 3.2	10-2244 10-2244-02
						14 22	pod pod	4 < 0.01	
						14 22	foliage foliage	0.47 0.47	
Germany, 2011 Burscheid (Elektra) winter rape	2	0.125	0.042	300	BBCH 79	-0 0	plant plant	0.34 1.9	11-2003 11-2003-01
						14 21 29	pod pod pod	0.89 0.79 0.91	
						14 21	foliage foliage	0.76 0.77	
Belgium, 2011 Sombrefe (Limone Hybrid) winter rape	2	0.125	0.063	200	BBCH 80	-0 0	plant plant	0.41 1.4	11-2003 11-2003-02
						14 21	pod pod	1 0.96	
						14 21	foliage foliage	0.14 0.1	
UK, 2011 Royston (Flash Hybrid) winter rape	2	0.125	0.063	200	BBCH 79	-0 0	plant plant	0.17 1.5	11-2003 11-2003-03
						14 21	pod pod	0.4 0.44	
						14 21	foliage foliage	0.11 0.14	
France (N), 2011 Chambourg sur Indre (Dynastie) winter rape	2	0.125	0.042	300	BBCH 79	-0 0	plant plant	1 2.6	11-2003 11-2003-04
						14 21	pod pod	2.7 3.1	
						14 21	foliage foliage	0.28 0.23	
Spain, 2011 La Luisiana (Eswilliams) winter rape	2	0.125	0.042	300	BBCH 72	-0 0	plant plant	1.6 3.8	11-2124 11-2124-01
						14 21	pod pod	8.6 7.2	
						14 21	foliage foliage	2.2 2.2	
France (N), 2011 Bouloc (Nk Alamir) winter rape	2	0.125	0.042	300	BBCH 79	-0 0	plant plant	0.94 3	11-2124 11-2124-02
						14 21	pod pod	3.5 4	
						14 21	foliage foliage	0.55 0.43	

OILSEED RAPE FORAGE Country, year Location (variety)	Application					DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)	GS last application		matrix	Fluopyram	
Italy, 2011 Tarquinia (Hybridstar) winter rape	2	0.125	0.042	300	BBCH 75	-0 0	plant plant	0.48 1.3	11-2124 11-2124-03
						14 21	pod pod	2.1 2.3	
						14 21	foliage foliage	0.33 0.27	
						14 21	foliage foliage	0.29 0.016	
Spain, 2011 Llerona (Pacific) winter rape	2	0.125	0.042	300	BBCH 78	-0 0	plant plant	0.48 0.81	11-2124 11-2124-04
						14 21	pod pod	1.9 < 0.01	
						14 21	foliage foliage	0.29 0.016	
						14 21	foliage foliage	0.29 0.016	

Studies RA-2609, RA-2616, RA-2610, RA-2617 were also provided to the 2010 JMPR [2010 JMPR Fluopyram Evaluation: Table 177]

FATE OF RESIDUES IN STORAGE AND IN PROCESSING

Magnitude of the residue in processing

Information was provided to the 2010 JMPR on the residue distribution of fluopyram in peel and pulp (melons), on the effects of trimming, washing and cooking of strawberries, blueberries, cabbage, broccoli, summer squash, lettuce, spinach, Mustard greens, celery and on the effects of simulated commercial processing on residues of fluopyram and metabolites in oranges, apples, plums, grapes, tomatoes, potatoes, sugar beet, soya beans, wheat, maize, peanut, rape seed, cotton seed and sunflower seed.

Processing factors derived by the 2010 JMPR of relevance to the commodities considered for maximum residue levels, dietary intake or livestock dietary burden estimation by the current meeting are summarized below:

Table 28 Summary of selected processing factors for fluopyram

Raw agricultural commodity	Processed commodity	Calculated processing factors ^a	Processing factor (mean or median)
Melons & Watermelon	Pulp	< 0.02, < 0.05, 0.05, < 0.06, < 0.06, < 0.07, < 0.08, < 0.09, 0.09, < 0.11, < 0.13, < 0.13, < 0.13, 0.13, 0.13, < 0.17, < 0.17, < 0.2, < 0.2	< 0.11
Cabbage	Washed heads	0.17, 0.25, < 0.5, < 0.5	< 0.36
	Cooked washed heads	< 0.17, < 0.25, < 0.5, < 0.5	< 0.36
Lettuce	Trimmed heads ^b	0.01, 0.02, 0.02, 0.03, 0.09, 0.34	0.03 (median)
	Trimmed heads ^b	0.02, 0.02, 0.03, 0.05, 0.19, 0.2	0.09
	Washed leaves	0.24, 0.85	0.55
Broccoli	Washed heads	0.8	0.8
	Cooked washed heads	0.62	0.62
Plums	Washed fruit	0.49	0.49
	Dried fruit	1.1	1.1
Rape seed	Oil (crude)	1.0, 1.3, 1.3, 2.1	1.4
	Oil (refined)	0.01, 0.64, 0.83, 1.0, 1.7	0.71
	Meal	0.29, 0.34, 0.67, 0.75, 1.4	0.69

^a Each value represents a separate study where residues were above the LOQ in the RAC. The factor is the ratio of the total

residue in the processed item divided by the total residue in the RAC.

^b Wrapper leaves removed

APPRAISAL

Fluopyram, a pyridylethylamide broad spectrum fungicide was evaluated for the first time by the 2010 JMPR, where an ADI of 0–0.01 mg/kg bw and an ARfD of 0.5 mg/kg bw were established, residue definitions were proposed and maximum residue levels were recommended for a number of uses where GAP information was available. New GAP and supporting information were evaluated by the 2012 JMPR and a number of additional maximum residue levels were recommended.

Residue definitions established by the 2010 JMPR are:

For plant products (compliance with MRLs and dietary intake assessment): *fluopyram*

For animal products (compliance with MRLs): *sum of fluopyram and 2-(trifluoromethyl)benzamide, expressed as fluopyram*

For animal products (dietary intake assessment): *sum of fluopyram, 2-(trifluoromethyl)benzamide and the combined residues N-[(E)-2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethenyl]-2-trifluoromethyl benzamide and N-[(Z)-2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethenyl]-2-trifluoromethyl benzamide, all expressed as fluopyram.*

New GAP information and supporting residue data were provided by the manufacturer for evaluation by the Meeting.

Supervised residue trials on crops

The Meeting received new supervised trial data for foliar applications of fluopyram (SC formulations) on plum, peach, apricot, raspberry, onion, leek, Brussels sprouts, cabbage, cauliflower, melon, lettuce, asparagus and oilseed rape and noted that data for some of these crops had also been provided to the 2010 JMPR. New supervised trial data were also provided for watermelon and currants, but because no GAP information was available for these crops, these data were not considered by the Meeting.

The results from these new trials and those previously reported by the 2010 JMPR and either matching critical GAP or where the results can be proportionally adjusted (scaled) to reflect GAP application rates were used to estimate maximum residue levels, STMRs and HRs for a number of commodities for which GAP information was available. The proportionality approach was used to scale the results from trials where the application rates range from $0.33 \times \text{GAP}$ to $4 \times \text{GAP}$ and where all other parameters matched the critical GAP). Frozen sample storage times in the new trials were within the storage intervals considered acceptable by the 2010 JMPR.

Stone fruit

Results from supervised field trials on apricots, peaches and plums conducted in Europe were considered by the Meeting, including some data for peaches that were also provided to the 2010 JMPR.

Peaches (sub-group 003C)

The critical GAP for apricots is in Greece, up to two applications of 0.15 kg ai/ha, 7–14 days apart with a PHI of 3 days. No trials were available matching this GAP. In trials from Europe where apricots were treated with 2×0.21 –0.25 kg ai/ha fluopyram, residues at 3 DALA (days after the last application) were: 0.16, 0.2, 0.33, 0.38, 0.43, 0.46, 0.58 and 0.95 mg/kg. When proportionally adjusted to the 0.15 kg ai/hL GAP application rate (scaling factors of 0.6–0.75), fluopyram residues in apricots from these trials were: 0.12, 0.14, 0.22, 0.26, 0.27, 0.28, 0.35 and 0.69 mg/kg (n=8).

For peaches, the 2012 JMPR recommended a maximum residue level of 0.4 mg/kg based on trials from Southern Europe proportionally adjusted to the GAP in Turkey (0.005 kg ai/hL, PHI 3 days).

The Meeting was advised that new GAP existed in Europe, with a new critical GAP (Greece) of up to two applications of 0.15 kg ai/ha, 7–14 days apart and a PHI of 3 days. No trials were available matching this new GAP. In trials from Europe where peaches were treated with 2×0.21 –0.25 kg ai/ha fluopyram, residues at 3 DALA were: 0.2, 0.26, 0.28, 0.28, 0.31, 0.36, 0.63 and 0.73 mg/kg. When proportionally adjusted to the 0.15 kg ai/ha GAP application rate (scaling factors of 0.6–0.75), fluopyram residues in peaches from these trials were: 0.12, 0.16, 0.17, 0.17, 0.19, 0.22, 0.45 and 0.53 mg/kg (n=8).

The Meeting noted that the medians of the data sets for apricots and peaches differed by less than 5-fold and agreed to consider a group maximum residue level for peaches (subgroup 003C) since GAP exists in Europe for all crops within this subgroup. In deciding on the data set to use for estimating a group maximum residue level, since a Mann-Whitney U-test indicated that the residue populations for apricots and peaches were not different it was agreed to combine the results to give a data set of 0.12, 0.12, 0.14, 0.16, 0.17, 0.17, 0.19, 0.22, 0.22, 0.26, 0.27, 0.28, 0.35, 0.45, 0.53 and 0.69 mg/kg (n=16).

The Meeting estimated a group maximum residue level of 1 mg/kg, an STMR of 0.22 mg/kg and an HR of 0.69 mg/kg for fluopyram on peaches (subgroup 003C) and to recommend withdrawal of the previous maximum residue level recommendation of 0.4 mg/kg for peach.

Plums (sub-group 003B)

The critical GAP for plums is in Greece, up to three applications of 0.1 kg ai/ha with a PHI of 3 days. In trials from Europe matching the GAP in Greece, residues were: 0.08, 0.1, 0.1, 0.13, 0.13, 0.14, 0.16, 0.18, 0.2, 0.2 and 0.22 mg/kg (n=11).

The Meeting estimated a sub-group maximum residue level of 0.5 mg/kg, an STMR of 0.13 mg/kg and an HR of 0.22 mg/kg for fluopyram on plums (subgroup 003B).

Berries and small fruit

Results from supervised trials on outdoor and protected raspberries conducted in Europe and outdoor raspberries were provided to the Meeting.

Cane berries

The critical GAP for small berries (including blackberries and raspberries) is in South Africa, up to two applications of 0.25 kg ai/ha, 10–14 days apart, with a PHI of 3 days. In trials from Europe where protected raspberries were treated with 2×0.2 kg ai/ha fluopyram, residues at 3 DAT were: 0.51, 0.69, 0.7 and 1.2 mg/kg (n=4).

Since the South African GAP does not exclude use on protected crops the Meeting agreed to use the European trial results on protected raspberries matching the GAP in South Africa to estimate a maximum residue level of 3 mg/kg, an STMR of 0.7 mg/kg and an HR of 1.2 mg/kg for fluopyram on raspberries and agreed to extrapolate these estimations to blackberries.

Bulb vegetables

Results from supervised field trials on bulb onions and leeks in Europe and bulb onions in USA were considered by the Meeting, including some data that were also provided to the 2010 JMPR.

Onion (bulb)

The critical GAP in Northern Europe for bulb onions is in Germany, a maximum of two applications of 0.1 kg ai/ha, with a PHI of 7 days. In trials in Europe matching the GAP in Germany, fluopyram residues in onion bulbs were: < 0.01 (3), < 0.01, 0.01, 0.02, 0.03 and 0.04 mg/kg (n=8).

GAP for bulb onions exists in Southern Europe (Greece and Spain), a maximum of one application of 0.2 kg ai/ha with a PHI of 7 days. In trials in Europe matching this GAP, fluopyram residues in onion bulbs were: < 0.01 (6), 0.03 and 0.04 mg/kg (n=8).

The Meeting agreed to use the results matching the GAP in Germany to estimate a maximum residue level, noting that this would accommodate the GAP in Greece and Spain.

The Meeting estimated a maximum residue level of 0.07 mg/kg, an STMR of 0.01 mg/kg and an HR of 0.04 mg/kg for fluopyram on onion (bulb).

Garlic

The Meeting noted that the GAP for onions in Greece and Spain also applied to garlic and agreed to extrapolate the results of the European onion trials matching this GAP to garlic and estimated a maximum residue level of 0.07 mg/kg, an STMR of 0.01 mg/kg and an HR of 0.04 mg/kg for fluopyram on garlic.

Leek

The critical GAP for leeks is in Germany and Switzerland, a maximum of one application of 0.2 kg ai/ha, with a PHI of 21 days. In trials in Europe matching this GAP, fluopyram residues in leeks were: < 0.01 (4), 0.01, 0.03, 0.06 and 0.07 mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg, an STMR of 0.01 mg/kg and an HR of 0.07 mg/kg for fluopyram on leek.

Brassica vegetables

Results from supervised field trials on broccoli, Brussels sprouts, head cabbage and cauliflower in Europe were considered by the Meeting, including some data that were also provided to the 2010 JMPR.

Broccoli

The critical GAP for broccoli (flowerhead brassicas) is in Germany, a maximum of two foliar applications of 0.18 kg ai/ha, with a PHI of 14 days. In trials on broccoli in Europe matching this GAP, fluopyram residues in broccoli were: < 0.01, < 0.01, 0.02, 0.03, 0.05, 0.05, 0.06, 0.13 and 0.14 mg/kg (n=9).

The Meeting estimated a maximum residue level of 0.3 mg/kg, an STMR of 0.05 mg/kg and an HR of 0.14 mg/kg for fluopyram on broccoli.

Cauliflower

In trials on cauliflowers in Europe matching the GAP in Germany for cauliflower (flower head Brassicas), up to two foliar applications of 0.18 kg ai/ha, with a PHI of 14 days. fluopyram residues were: < 0.01 (4), 0.01 (5), 0.02, 0.05, 0.05 and 0.05 mg/kg (n=13).

The Meeting estimated a maximum residue level of 0.09 mg/kg, an STMR of 0.01 mg/kg and an HR of 0.05 mg/kg for fluopyram on cauliflower.

Brussels sprouts

The critical GAP for Brussels sprouts is in Germany, a maximum of two foliar applications of 0.18 kg ai/ha, with a PHI of 14 days. In trials in Europe matching this GAP, fluopyram residues in Brussels sprouts were: 0.01, 0.04, 0.04, 0.04, 0.04, 0.05, 0.07, 0.07, 0.07, 0.09, 0.14 and 0.15 mg/kg (n=12).

The Meeting estimated a maximum residue level of 0.3 mg/kg, an STMR of 0.06 mg/kg and an HR of 0.15 mg/kg for fluopyram on Brussels sprouts.

Cabbages, head

The critical GAP for head cabbages is in Germany, a maximum of two foliar applications of 0.18 kg ai/ha, with a PHI of 14 days. In trials in Europe matching this GAP, fluopyram residues in cabbage heads were: < 0.01 (4), 0.01, 0.01, 0.01, 0.02, 0.02, 0.04 and 0.08 mg/kg (n=11).

The Meeting estimated a maximum residue level of 0.15 mg/kg, an STMR of 0.01 mg/kg and an HR of 0.08 mg/kg for fluopyram on cabbage, head.

Fruiting vegetables, Cucurbits

Results from supervised field trials on protected melons and on field melons in Europe, USA and Australia were considered by the Meeting, including some data that were also provided to the 2010 JMPR.

Melons (except watermelon)

The critical GAP for melons is in South Korea, up to three applications of 0.225 kg ai/ha, 10 days apart, with a PHI of 3 days. Since this GAP does not exclude use on protected crops, the Meeting agreed to consider the data from trials on protected melons in Europe involving three applications of 0.1 kg ai/ha where residues at 3 DAT were < 0.01, 0.02, 0.07 and 0.12 mg/kg. When the results of three of these trials where residues were found above the LOQ are proportionally adjusted to the South Korean GAP (scaling factor of 2.25), fluopyram residues in these trials are: 0.05, 0.06 and 0.27 mg/kg.

The Meeting agreed that these data were not sufficient to estimate a maximum residue level for fluopyram on melons (except watermelon).

*Leafy vegetables, including brassica leafy vegetables**Lettuce*

The critical GAP for lettuce is in Netherlands, up to two applications of 0.25 kg ai/ha, 7 days apart, with a PHI of 7 days.

In outdoor lettuce trials in Europe matching this GAP, fluopyram residues in head lettuce were 0.12, 0.13, 0.57, and 0.63 mg/kg and in leaf lettuce were 0.18, 0.18, 0.25, 0.26, 0.27, 0.53, 0.61, 0.62 and 0.93 mg/kg.

In protected lettuce matching the GAP in Netherlands, fluopyram residues in head lettuce were 0.23, 1.4, 1.9, 2.1, 2.5 and 7.7 mg/kg and in leaf lettuce were 0.16, 0.81, 0.92, 1.2, 2.3, 2.7, 3.8, 4.4, 7.2 and 8.4 mg/kg.

The Meeting noted that highest residues were in protected lettuce and since the data sets for leaf and head lettuce were not from different populations (Mann Whitney), agreed to use the combined data set for protected head and leaf lettuce to estimate maximum residue levels for leaf and head lettuce.

The combined data set of results from the protected lettuce trials matching the GAP in Netherlands is: 0.16, 0.23, 0.81, 0.92, 1.2, 1.4, 1.9, 2.1, 2.3, 2.5, 2.7, 3.8, 4.4, 7.2, 7.7 and 8.4 mg/kg (n=16).

The Meeting estimated maximum residue levels of 15 mg/kg, STMRs of 2.2 mg/kg and HRs of 8.4 mg/kg for fluopyram on lettuce, head and lettuce, leaf.

*Stalk and stem vegetables**Asparagus*

The critical GAP for asparagus is in Switzerland, up to two applications of 0.2 kg ai/ha, applied to mature ferns prior to senescence, about 6 months or more before the new spears emerge. In four European trials matching this GAP, fluopyram residues in the new spears were all < 0.01 mg/kg.

The Meeting estimated a maximum residue level of 0.01 (*) mg/kg, an STMR of 0 mg/kg and an HR of 0 mg/kg for fluopyram on asparagus.

*Oilseeds**Rape seed*

The critical GAP for oilseed rape is in Ukraine, up to two applications of 0.113 kg ai/ha with a PHI of 30 days. Fluopyram residues in rape seed from European trials matching this GAP were: 0.07, 0.1, 0.14, 0.25, 0.27, 0.27, 0.29, 0.33, 0.34, 0.35, 0.38, 0.38, 0.46, 0.46 and 0.61 mg/kg (n=15).

The Meeting estimated a maximum residue level of 1 mg/kg and an STMR of 0.33 mg/kg for fluopyram on rape seed.

*Animal feeds**Oilseed rape, forage*

The Meeting noted that the GAP for fluopyram in Europe is for use on oilseed rape over the flowering period or at early maturity (up to 30 days before harvest). The use of oilseed rape as a forage crop is normally earlier in the season, up to about BBCH 39, prior to the first application of fluopyram and the Meeting concluded that the reported GAP for fluopyram is not relevant for the use of oilseed rape as an animal forage crop.

Fate of residues during processing

The 2010 JMPR reported that fluopyram was stable under conditions simulating pasteurisation, boiling and sterilisation and also estimated processing factors and STMR-Ps for a range of commodities. Relevant processing factors and STMR-Ps for the commodities considered at this Meeting and used for dietary intake risk assessment or for estimating livestock animal burdens are summarised below.

Summary of relevant processing factors and STMR-P values for fluopyram residues

Raw agricultural commodity	Processed commodity	Processing factors ^a (mean or median)	RAC (mg/kg)		STMR-P (mg/kg)	HR-P (mg/kg)
			STMR	HR		
Cabbage			0.01	0.14		
	Cooked washed heads	< 0.36			0.004	0.05
Plums			0.13	0.22		
	Dried fruit	1.1			0.14	0.24
Rape seed			0.33			
	Oil (refined)	0.71			0.23	
	Meal	0.69			0.23	

^a The processing factor is the ratio of the total residue in the processed item divided by the total residue in the RAC.

Animal commodity maximum residue levels

Cattle

The two new cattle feed commodities considered by the Meeting (rape meal and cabbage leaves) increased these dietary burdens by less than 1.5% and the Meeting agreed that the maximum residue levels, STMRs and HRs estimated by the 2012 JMPR for animal commodities did not need to be revised.

Poultry

The new poultry feed commodity considered by the Meeting (rape meal) increased the maximum dietary burden by less than 1% and increased the mean dietary burden by 11%. The Meeting agreed that the maximum residue levels and HRs estimated by the 2012 JMPR for poultry commodities did not need to be revised.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI assessment.

Definition of the residue for compliance with the MRL and for the estimation of dietary intake for plant commodities: *fluopyram*

Definition of the residue for compliance with the MRL for animal commodities: *Sum of fluopyram and 2-(trifluoromethyl) benzamide, expressed as fluopyram*

Definition of the residue for the estimation of dietary intake for animal commodities: *Sum of fluopyram, 2-(trifluoromethyl)benzamide and the combined residues N-[(E)-2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethenyl]-2-trifluoromethyl benzamide and N-[(Z)-2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethenyl]-2-trifluoromethyl benzamide, all expressed as fluopyram.*

	Commodity	MRL (mg/kg)		STMR or	HR or
CCN	Name	New	Prev	STMR-P	HR-P
FS 2001	Peaches	1		0.22	0.69
FS 0247	Peach	W	0.4		
FS 0014	Plums	0.5		0.13	0.22
DF 0014	Dried plums (prunes)			0.14	0.24
FB 0272	Raspberries, Red, Black	3		0.7	1.2
FB 0264	Blackberries	3		0.7	1.2
VA 385	Onion, Bulb	0.07		0.01	0.04
VA 381	Garlic	0.07		0.01	0.04
VA 384	Leek	0.15		0.01	0.07
VB 0400	Broccoli	0.3		0.05	0.14
VB 402	Brussels sprouts	0.3		0.06	0.15
VB 041	Cabbages, Head	0.15		0.01	0.08
VB 404	Cauliflower	0.09		0.01	0.05
VL 482	Lettuce, Head	15		2.2	8.4
VL 483	Lettuce, Leaf	15		2.2	8.4
VS 621	Asparagus	0.01 (*)		0	0
SO 495	Rape seed	1		0.33	
	Cabbage, washed, cooked			0.004	0.05

	Commodity	MRL (mg/kg)		STMR or	HR or
CCN	Name	New	Prev	STMR-P	HR-P
OC 495	Rape seed oil (edible)			0.23	
OR 495	Rape meal			0.23	

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) for fluopyram were calculated for the food commodities for which STMRs or HRs were estimated and for which consumption data were available. The results are shown in Annex 3.

The International Estimated Daily Intakes of fluopyram for the 17 GEMS/Food regional diets, based on estimated STMRs were 3–20% of the maximum ADI of 0.01 mg/kg bw (Annex 3 to the 2014 Report). The Meeting concluded that the long-term intake of residues of fluopyram from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short-term Intakes (IESTIs) for fluopyram were calculated for the food commodities for which STMRs or HRs were estimated and for which consumption data were available (Annex 4 to the 2014 Report).

For fluopyram the IESTI varied from 0–100% of the ARfD (0.5 mg/kg bw) and the Meeting concluded that the short-term intake of residues of fluopyram from uses considered by the Meeting is unlikely to present a public health concern.

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12-2158	Fargeix, G	2013	Determination of the residues of AE C656948 in/on apricot after spray application of fluopyram SC 500 in the field in southern France, Spain and Italy. Bayer S.A.S., Bayer CropScience, Lyon, France. Bayer CropScience. Report No.: 12-2158. Report includes Trial Nos.: 12-2158-01, 12-2158-02, 12-2158-03, 12-2158-04. Edition Number: M-468035-01-1. Date: 2013-10-28. GLP/GEP: yes, unpublished
13-2005	Bellof, S	2014	Determination of the residues of AE C656948 in/on apricot after spray application of fluopyram SC 500 in Germany. Report No.: 13-2005. Edition Number: M-481905-01-1. Date: 2014-04-30. GLP/GEP: yes, unpublished

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BCS-0242	Radunz, L	2009	Determination of residues of AE C656948 (fluopyram) in rockmelon following three applications of AE C656948 500 SC at rates of 75, 125 or 187.5 g ai/ha, five days apart just before harvest. Bayer CropScience, Eight Mile Plains, QLD, Australia. Bayer CropScience. Report No.: BCS-0242. Report includes Trial Nos.: C312, C313. Edition Number: M-358358-01-1. Date: 2009-10-22. GLP/GEP: yes, unpublished.
BCS-0252	Radunz, L	2010	Determination of residues of AE C656948 (fluopyram) in rockmelons following three applications of AE C656948 500 SC at rates of 75, 125 or 187.5 g ai/ha, five days apart just before harvest. Bayer CropScience, Eight Mile Plains, QLD, Australia. Bayer CropScience. Report No.: BCS-0252. Edition Number: M-371302-01-1. Date: 2010-04-30. GLP/GEP: yes, unpublished.
BCS-G401-11	Loriau, P	2012	Residues of fluopyram and trifloxystrobin in raspberry under plastic umbrella at intervals following two foliar applications of FLU+TFS 500 SC—Belgium, season 2011. Redebel S.A., Saint Amand, Belgium. Bayer CropScience. Report No.: BCS-G401-11. Edition Number: M-433737-01-1. Date: 2012-02-27. GLP/GEP: yes, unpublished
BCS-G402-11	Loriau, P	2012	Residues of fluopyram and trifloxystrobin in red currant under plastic umbrella at intervals following two foliar applications of FLU+TFS 500 SC—Belgium, season 2011. Redebel S.A., Saint Amand, Belgium. Bayer CropScience. Report No.: BCS-G402-11. Edition Number: M-433738-01-1. Date: 2012-03-22. GLP/GEP: yes, unpublished
P 1799 G	Bacher, R	2010	Determination of residues of fluopyram and metabolites in currant and raspberry: Minor crop study 2009. PTRL Europe GmbH, Ulm, Germany. Bayer CropScience. Report No.: P 1799 G. Edition Number: M-431229-01-1. Date: 2010-04-07. GLP/GEP: yes, unpublished
P 2428 G	Bacher, R	2012	Determination of residues of fluopyram and metabolites and tebuconazole in/on raspberry: Minor crop study 2011. PTRL Europe GmbH, Ulm, Germany. Bayer CropScience. Report No.: P 2428 G. Edition Number: M-431230-01-1. Date: 2012-02-20. GLP/GEP: yes, unpublished
PTZ-NLI-11796	Oostingh, C	2012	Amendment no. 1 to report no: PTZ-NLI-11796—Residues of fluopyram + trifloxystrobin in red currant under plastic umbrella at intervals following two foliar applications of fluopyram & trifloxystrobin SC 500. Proeftuin Zwaagdijk, Zwaagdijk, Netherlands. Bayer CropScience. Report No.: PTZ-NLI-11796. Edition Number: M-434301-02-1. Date: 2012-06-08. ...Amended: 2013-03-06. GLP/GEP: yes, unpublished
PTZ-NLI-11797	Oostingh, C	2012	Amendment no. 1 to report no: PTZ-NLI-11797—Residues of fluopyram + trifloxystrobin in red raspberry under plastic umbrella at intervals following two foliar applications of fluopyram & trifloxystrobin SC 500. Proeftuin Zwaagdijk, Zwaagdijk, Netherlands. Bayer CropScience. Report No.: PTZ-NLI-11797. Edition Number: M-434309-02-1. Date: 2012-06-08. ...Amended: 2013-03-06. GLP/GEP: yes, unpublished
RA 2591/06	Diot, R	2007	Determination of the residues of AE C656948 in/on peach after spraying of AE C656948 (500 SC) in the field in (the) Southern France, Spain and Italy. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA 2591/06. Report includes Trial Nos.: R 2006 0374/6, R 2006 0600/, R 2006 0602/8, R 2006 0603/6, R 2006 0603/6. Edition Number: M-290819-01-1. Date: 2007-07-23. GLP/GEP: yes, unpublished
RA-2508/07	Ballesteros, C	2008	Determination of the residues of AE C656948 in/on melon and watermelon after spraying of AE C656948 (500 SC) in the field in Greece, Italy, Portugal and Spain. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2508/07. Report includes Trial Nos.: R 2007 0009/1=0009 - 07, R 2007 0010/5=0010 - 07, R 2007 0535/2=0535 - 07, R 2007 0536/0=0536 - 07. Edition Number: M-302924-01-1. Date: 2008-06-19. GLP/GEP: yes, unpublished.
RA-2509/07	Portet, M	2008	Determination of the residues of AE C656948 in/on head lettuce and lettuce after spraying of AE C656948 (500 SC) in the field in United Kingdom, Germany, Northern France, Belgium and Netherlands. Bayer CropScience, Lyon, France. Bayer CropScience. Report No.: RA-2509/07. Report includes Trial Nos.: R 2007 0011/3=0011 - 07, R 2007 0244/2=0244 - 07, R 2007 0537/9=0537 - 07, R 2007 0538/7=0538 - 07, R 2007 0539/5=0539 - 07, R 2007 0540/9=0540 - 07. Edition Number: M-304280-01-1. Date: 2008-07-01. GLP/GEP: yes, unpublished

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RA-2510/07	Portet, M	2008	Determination of the residues of AE C656948 in/on head lettuce and lettuce after spraying of AE C656948 (500 SC) in the field in Portugal, Greece, Southern France and Italy. Bayer CropScience, Lyon, France. Bayer CropScience. Report No.: RA-2510/07. Report includes Trial Nos.: R 2007 0012/1=0012 - 07, R 2007 0245/0=0245 - 07, R 2007 0246/9=0246 - 07, R 2007 0541/7=0541 - 07. Edition Number: M-304278-01-1. Date: 2008-07-01. GLP/GEP: yes, unpublished
RA-2517/07	Ballesteros, C	2008	Determination of the residues of AE C656948 and tebuconazole in/on onion after spraying of AE C656948 & HWG 1608 (400 SC) in the field in Germany, Northern France and Netherlands. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2517/07. Report includes Trial Nos.: R 2007 0038/5=0038 - 07, R 2007 0558/1=0558 - 07, R 2007 0560/3=0560 - 07, R 2007 0561/1=0561 - 07. Edition Number: M-303586-01-1. Date: 2008-07-03. GLP/GEP: yes, unpublished
RA-2518/07	Ballesteros, C	2008	Determination of the residues of AE C656948 and tebuconazole in/on onion after spraying of AE C656948 & HWG 1608 (400 SC) in the field in Southern France, Italy, Spain, Portugal and Greece. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2518/07. Report includes Trial Nos.: R 2007 0040/7=0040 - 07, R 2007 0563/8=0563 - 07, R 2007 0564/6=0564 - 07, R 2007 0565/4=0565 - 07, R 2007 0566/2=0566 - 07. Edition Number: M-303588-01-1. Date: 2008-07-03. GLP/GEP: yes, unpublished
RA-2519/07	Ballesteros, C	2008	Determination of the residues of AE C656948 and tebuconazole in/on onion, welsh after spraying of AE C656948 & HWG 1608 (400 SC) in the field in Germany and United Kingdom. Bayer CropScience S.A., Lyon, France. Report No.: BCSRA-2519/07. Report includes Trial Nos.: R 2007 0042/3, R 2007 0567/0. Edition Number: M-302330-01-1. Date: 2008-06-04. GLP/GEP: yes, unpublished
RA-2520/07	Ballesteros, C	2008	Determination of the residues of AE C656948 and tebuconazole in/on onion, welsh after spraying of AE C656948 & HWG 1608 (400 SC) in the field in Southern France and Italy. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2520/07. Report includes Trial Nos.: R 2007 0043/1=0043 - 07, R 2007 0568/9=0568 - 07. Edition Number: M-302325-01-1. Date: 2008-06-04. GLP/GEP: yes, unpublished
RA-2521/07	Portet, M	2008	Determination of the residues of AE C656948 and tebuconazole in/on leek after spraying of AE C656948 & HWG 1608 (400 SC) in the field in Germany, Northern France and Netherlands. Bayer CropScience, Lyon, France. Bayer CropScience. Report No.: RA-2521/07. Report includes Trial Nos.: R 2007 0056/3=0056 - 07, R 2007 0569/7=0569 - 07, R 2007 0570/0=0570 - 07, R 2007 0571/9=0571 - 07. Edition Number: M-304288-01-1. Date: 2008-07-17. GLP/GEP: yes, unpublished
RA-2522/07	Portet, M	2008	Determination of the residues of AE C656948 in/on leek after spraying of AE C656948 & HWG 1608 (400 SC) in the field in Southern France and Italy. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2522/07. Report includes Trial Nos.: R 2007 0057/1=0057 - 07, R 2007 0572/7=0572 - 07. Edition Number: M-302775-01-1. Date: 2008-06-13. GLP/GEP: yes, unpublished
RA-2529/07	Uceda, L	2008	Determination of the residues of AE C656948 and tebuconazole in/on round cabbage and red cabbage after spraying of AE C656948 & HWG 1608 (400 SC) in the field in Germany, Northern France, United Kingdom and Belgium. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2529/07. Report includes Trial Nos.: R 2007 0073/3=0073 - 07, R 2007 0270/1=0270 - 07, R 2007 0589/1=0589 - 07, R 2007 0590/5=0590 - 07, R 2007 0591/3=0591 - 07. Edition Number: M-302866-01-1. Date: 2008-06-19. GLP/GEP: yes, unpublished
RA-2530/07	Uceda, L	2008	Determination of the residues of AE C656948 and tebuconazole in/on round cabbage after spraying of AE C656948 & HWG 1608 (400 SC) in the field in Italy. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2530/07. Report includes Trial Nos.: R 2007 0592/1=0592 - 07. Edition Number: M-302078-01-1. Date: 2008-05-30. GLP/GEP: yes, unpublished
RA-2531/07	Portet, M	2008	Determination of the residues of AE C656948 and tebuconazole in/on Brussels sprouts after spraying of AE C656948 & HWG 1608 (400 SC) in the field in Germany, Northern France, Netherlands and United Kingdom. Bayer CropScience, Lyon, France. Bayer CropScience. Report No.: RA-2531/07. Report includes Trial Nos.: R 2007 0076/8=0076 - 07, R 2007 0594/8=0594 - 07, R 2007 0595/6=0595 - 07, R 2007 0596/4=0596 - 07. Edition Number: M-304284-01-1. Date: 2008-07-04. GLP/GEP: yes, unpublished

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RA-2532/07	Portet, M	2008	Determination of the residues of AE C656948 and tebuconazole in/on Brussels sprouts after spraying of AE C656948 & HWG 1608 (400 SC) in the field in Southern France and Italy. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2532/07. Report includes Trial Nos.: R 2007 0077/6=0077 - 07, R 2007 0597/2=0597 - 07. Edition Number: M-303735-01-1. Date: 2008-06-27. GLP/GEP: yes, unpublished
RA-2533/07	Uceda, L	2008	Determination of the residues of AE C656948 and tebuconazole in/on Chinese cabbage after spraying of AE C656948 & HWG 1608 (400 SC) in the field in United Kingdom and Germany. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2533/07. Report includes Trial Nos.: R 2007 0078/4=0078 - 07, R 2007 0599/9=0599 - 07. Edition Number: M-302101-01-1. Date: 2008-06-02. GLP/GEP: yes, unpublished
RA-2534/07	Uceda, L	2008	Determination of the residues of AE C656948 and tebuconazole in/on Chinese cabbage after spraying of AE C656948 & HWG 1608 (400 SC) in the field in southern France and Spain. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2534/07. Report includes Trial Nos.: R 2007 0079/2=0079 - 07, R 2007 0600/6=0600 - 07. Edition Number: M-302044-01-1. Date: 2008-05-30. GLP/GEP: yes, unpublished
RA-2565/06	Cavaille, C	2007	Determination of the residues of AE C656948 and tebuconazole in/on onion, Welsh after spraying of AE C656948 & HWG 1608 (400 SC) in the field in (the) northern France and Germany. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2565/06. Report includes Trial Nos.: R 2006 0337/1, R 2006 0504/. Edition Number: M-292996-01-1. Date: 2007-09-25. GLP/GEP: yes, unpublished
RA-2566/06	Cavaillé, C and Portet, M	2007	Determination of the residues of AE C656948 and tebuconazole in/on onion, Welsh after spraying of AE C656948 & HWG 1608 (400 SC) in the field in (the) Southern France and Italy. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2566/06. Report includes Trial Nos.: R 2006 0339/8, R 2006 0505/6. Edition Number: M-292098-01-1. Date: 2007-09-03. GLP/GEP: yes, unpublished
RA-2567/06	Cavaillé, C and Portet, M	2007	Determination of the residues of AE C656948 and tebuconazole in/on onion after spraying of AE C656948 & HWG 1608 (400 SC) in the field in (the) Northern France, Germany and United Kingdom. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2567/06. Report includes Trial Nos.: R 2006 0340/1, R 2006 0537/4, R 2006 0538/2, R 2006 0539/0. Edition Number: M-293181-01-1. Date: 2007-09-28. GLP/GEP: yes, unpublished
RA-2568/06	Cavaillé, C and Portet, M	2007	Determination of the residues of AE C656948 and tebuconazole in/on onion after spraying of AE C656948 & HWG 1608 (400 SC) in the field in (the) Southern France, Spain, Italy and Greece. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2568/06. Report includes Trial Nos.: R 2006 0342/8, R 2006 0540/4, R 2006 0541/2, R 2006 0542/0. Edition Number: M-293014-01-1. Date: 2007-09-25. GLP/GEP: yes, unpublished
RA-2569/06	Cavaille, C and Portet, M	2007	Determination of the residues of AE C656948 and tebuconazole in/on leek after spraying of AE C656948 & HWG 1608 (400 SC) in the field in (the) Northern France, Germany and United Kingdom. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2569/06. Report includes Trial Nos.: R 2006 0343/6=0343 - 06, R 2006 0465/3=0465 - 06, R 2006 0466/1=0466 - 06, R 2006 0468/8=0468 - 06. Edition Number: M-292101-02-1. Date: 2007-09-03. ...Amended: 2008-09-05. GLP/GEP: yes, unpublished
RA-2570/06	Cavaillé, C and Portet, M	2007	Determination of the residues of AE C656948 and tebuconazole in/on leek after spraying of AE C656948 & HWG 1608 (400 SC) in the field in (the) Southern France and Spain. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2570/06. Report includes Trial Nos.: R 2006 0344/4=0344 - 06, R 2006 0469/6=0469 - 06. Edition Number: M-292082-01-1. Date: 2007-08-31. GLP/GEP: yes, unpublished
RA-2573/06	Cavaillé, C and Portet, M	2007	Determination of the residues of AE C656948 and tebuconazole in/on Chinese cabbage after spraying of AE C656948 & HWG 1608 (400 SC) in the field in (the) Northern France and United Kingdom. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2573/06. Report includes Trial Nos.: R 2006 0347/9=0347 - 06, R 2006 0543/9=0543 - 06. Edition Number: M-292103-01-1. Date: 2007-09-03. GLP/GEP: yes, unpublished

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RA-2574/06	Cavaillé, C and Portet, M	2007	Determination of the residues of AE C656948 and tebuconazole in/on Chinese cabbage after spraying of AE C656948 & HWG 1608 (400 SC) in the field in (the) Southern France and Italy. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2574/06. Report includes Trial Nos.: R 2006 0348/7=0348 - 06, R 2006 0544/7=0544 - 06. Edition Number: M-293182-01-1. Date: 2007-09-28. GLP/GEP: yes, unpublished
RA-2575/06	Cavaillé, C and Portet, M	2007	Determination of the residues of AE C656948 and tebuconazole in/on Brussels sprouts after spraying of AE C656948 & HWG 1608 (400 SC) in the field in (the) Northern France, United Kingdom and Germany. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2575/06. Report includes Trial Nos.: R 2006 0349/5=0349 - 06, R 2006 0473/4=0473 - 06, R 2006 0474/2=0474 - 06, R 2006 0475/0=0475 - 06. Edition Number: M-290883-01-1. Date: 2007-07-25. GLP/GEP: yes, unpublished
RA-2576/06	Cavaillé, C and Portet, M	2007	Determination of the residues of AE C656948 and tebuconazole in/on Brussels sprouts after spraying of AE C656948 & HWG 1608 (400 SC) in the field in (the) Southern France and Italy. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2576/06. Report includes Trial Nos.: R 2006 0350/9=0350 - 06, R 2006 0476/9=0476 - 06. Edition Number: M-290881-01-1. Date: 2007-07-24. GLP/GEP: yes, unpublished
RA-2577/06	Schoening, R, Erler, S and Wolters, A	2007	Determination of the residues of AE C656948 and tebuconazole in/on round cabbage after spraying of AE C656948 & HWG 1608 (400 SC) in the field in Northern France, the Netherlands, and Germany. Bayer CropScience. Report No.: RA-2577/06. Report includes Trial Nos.: R 2006 0354/1=0354 - 06, R 2006 0356/8=0356 - 06, R 2006 0477/7=0477 - 06, R 2006 0478/5=0478 - 06. Edition Number: M-295534-01-1. Date: 2007-11-30. GLP/GEP: yes, unpublished
RA-2578/06	Schoening, R and Wolters, A	2008	Determination of the residues of AE C656948 and tebuconazole in/on round cabbage after spraying of AE C656948 & HWG 1608 (400 SC) in the field in Southern France and Italy. Bayer CropScience. Report No.: RA-2578/06. Report includes Trial Nos.: R 2006 0357/6=0357 - 06, R 2006 0479/3=0479 - 06. Edition Number: M-296522-01-1. Date: 2008-01-18. GLP/GEP: yes, unpublished
RA-2581/06	Cavaille, C	2007	Determination of the residues of AE C656948 and tebuconazole in/on peach after spraying of AE C656948 & HWG 1608 (400 SC) in the field in (the) Southern France, Italy, Spain and Portugal. Bayer CropScience S.A., Lyon, France. BCS. Report No.: RA-2581/06. Report includes Trial Nos.: R 2006 0362/2=0362 - 06, R 2006 0487/4=0487 - 06, R 2006 0488/2=0488 - 06, R 2006 0489/0=0489 - 06. Edition Number: M-290544-01-1. Date: 2007-07-12. GLP/GEP: yes, unpublished
RA-2585/07	Portet, M	2008	Determination of the residues of AE C656948 and tebuconazole in/on peach after spraying of AE C656948 & HWG 1608 (400 SC) in the field in Southern France, Spain, Italy and Greece. Bayer CropScience, Lyon, France. Bayer CropScience. Report No.: RA-2585/07. Report includes Trial Nos.: R 2007 0225/6=0225 - 07, R 2007 0496/8=0496 - 07, R 2007 0497/6=0497 - 07, R 2007 0498/4=0498 - 07. Edition Number: M-304286-01-1. Date: 2008-07-04. GLP/GEP: yes, unpublished
RA-2586/06	Diot, R	2007	Determination of the residues of AE C656948 in/on head lettuce after spraying of AE C656948 (500 SC) in the greenhouse in France, Germany, Spain, Italy, Portugal and Greece. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2586/06. Report includes Trial Nos.: R 2006 0367/, R 2006 0576/5, R 2006 0577/, R 2006 0578/1, R 2006 0580/, R 2006 0581/, R 2006 0583/8, R 2006 0584/6. Edition Number: M-291173-01-1. Date: 2007-08-02. GLP/GEP: yes, unpublished
RA-2587/06	Diot, R	2007	Determination of the residues of AE C656948 in/on melon after spraying of AE C656948 (500 SC) in the greenhouse and in the field in Southern France, Spain, Italy, Germany, Greece and Portugal. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2587/06. Report includes Trial Nos.: R 2006 0368/1, R 2006 0585/4, R 2006 0586/2, R 2006 0587/0, R 2006 0589/7, R 2006 0590/0, R 2006 0591/9. Edition Number: M-291162-01-1. Date: 2007-07-26. GLP/GEP: yes, unpublished.
RA-2588/06	Diot, R	2007	Determination of the residues of AE C656948 in/on melon after spraying of AE C656948 (500 SC) in the field in (the) Northern France and Germany. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2588/06. Report includes Trial Nos.: R 2006 0370/3=0370 - 06, R 2006 0592/7=0592 - 06. Edition Number: M-290921-01-1. Date: 2007-07-26. GLP/GEP: yes, unpublished.

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RA-2588/07	Ballesteros, C and Ratajczak, M	2008	Determination of the residues of AE C656948 in/on plum after spraying of AE C656948 (500 SC) in the field in Germany and Netherlands. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2588/07. Report includes Trial Nos.: R 2007 0231/0=0231 - 07, R 2007 0499/2=0499 - 07, R 2007 0501/8=0501 - 0, R 2007 0502/6=0502 - 07. Edition Number: M-326998-01-1. Date: 2008-12-23. GLP/GEP: yes, unpublished
RA-2589/06	Diot, R	2007	Determination of the residues of AE C656948 in/on melon after spraying of AE C656948 (500 SC) in the field in Southern France, Greece, Spain and Italy. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2589/06. Report includes Trial Nos.: R 2006 0371/1=0371 - 06, R 2006 0593/5=0593 - 06, R 2006 0594/3=0594 - 06, R 2006 0595/1=0595 - 06. Edition Number: M-291434-01-1. Date: 2007-07-26. GLP/GEP: yes, unpublished.
RA-2589/07	Ballesteros, C	2008	Determination of the residues of AE C656948 in/on plum after spraying of AE C656948 (500 SC) in the field in Spain and Italy. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2589/07. Report includes Trial Nos.: R 2007 0232/9=0232 - 07, R 2007 0503/4=0503 - 07. Edition Number: M-302341-01-1. Date: 2008-06-05. GLP/GEP: yes, unpublished
RA-2592/06	Diot, R	2007	Determination of the residues of AE C656948 in/on head lettuce after spraying of AE C656948 (500 SC) in the field in (the) Northern France, Germany, Netherlands and United Kingdom. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2592/06. Report includes Trial Nos.: R 2006 0375/4=0375 - 06, R 2006 0604/4=0604 - 06, R 2006 0605/2=0605 - 06, R 2006 0606/0=0606 - 06, R 2006 0607/9=0607 - 06. Edition Number: M-292048-01-1. Date: 2007-08-28. GLP/GEP: yes, unpublished
RA-2593/06	Diot, R	2007	Determination of the residues of AE C656948 in/on head lettuce after spraying of AE C656948 (500 SC) in the field in Southern France, Spain, Italy and Greece. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2593/06. Report includes Trial Nos.: R 2006 0376/2=0376 - 06, R 2006 0608/7=0608 - 06, R 2006 0609/5=0609 - 06, R 2006 0610/9=0610 - 06, R 2006 0611/7=0611 - 06. Edition Number: M-292050-01-1. Date: 2007-08-28. GLP/GEP: yes, unpublished
RA-2604/07	Portet, M	2008	Determination of the residues of AE C656948 in/on lettuce after spraying of AE C656948 (500 SC) in the greenhouse in Southern France, Germany, Italy and Spain. Bayer CropScience, Lyon, France. Bayer CropScience. Report No.: RA-2604/07. Report includes Trial Nos.: R 2007 0247/7=0247 - 07, R 2007 0542/5=0542 - 07, R 2007 0543/3=0543 - 07, R 2007 0544/1=0544 - 07. Edition Number: M-304274-01-1. Date: 2008-07-04. GLP/GEP: yes, unpublished
RA-2609/06	Schoening, R, Raecker, T and Telscher, M	2007	Determination of the residues of AE C656948 in/on winter rape after spraying of AE C656948 (500 SC) in the field in Germany, Northern France, and the United Kingdom. Bayer CropScience. Report No.: RA-2609/06. Report includes Trial Nos.: R 2006 0406/8=0406 - 06, R 2006 0408/4=0408 - 06, R 2006 0409/2=0409 - 06, R 2006 0410/6=0410 - 06. Edition Number: M-295651-01-1. Date: 2007-12-13. GLP/GEP: yes, unpublished
RA-2609/07	Portet, M	2008	Determination of the residues of AE C656948 and tebuconazole in/on leek after spraying of AE C656948 & HWG 1608 (400 SC) in the field in Germany, Northern France and Netherlands. Bayer CropScience, Lyon, France. Bayer CropScience. Report No.: RA-2609/07. Report includes Trial Nos.: R 2007 0249/3=0249 - 07, R 2007 0573/5=0573 - 07, R 2007 0574/3=0574 - 07. Edition Number: M-304276-01-1. Date: 2008-07-08. GLP/GEP: yes, unpublished
RA-2610/06	Schoening, R, Raecker, T and Telscher, M	2007	Determination of the residues of AE C656948 in/on winter rape and summer rape after spraying of AE C656948 (500 SC) in the field in Southern France and Italy. Bayer CropScience. Report No.: RA-2610/06. Report includes Trial Nos.: R 2006 0625/7=0625 - 06, R 2006 0626/5=0626 - 06. Edition Number: M-295825-01-1. Date: 2007-12-19. GLP/GEP: yes, unpublished
RA-2610/07	Portet, M	2008	Determination of the residues of AE C656948 and tebuconazole in/on leek after spraying of AE C656948 & HWG 1608 (400 SC) in the field in Southern France. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2610/07. Report includes Trial Nos.: R 2007 0250/7=0250 - 07. Edition Number: M-302780-01-1. Date: 2008-06-13. GLP/GEP: yes, unpublished

Code	Author(s)	Year	Title, Institute, Report reference
RA-2612/07	Uceda, L	2008	Determination of the residues of AE C656948 and tebuconazole in/on round cabbage and red cabbage after spraying of AE C656948 & HWG 1608 (400 SC) in the field in Germany, United Kingdom and Belgium. Bayer CropScience S.A., Lyon, France. Bayer CropScience. Report No.: RA-2612/07. Report includes Trial Nos.: R 2007 0251/5=0251 - 07, R 2007 0272/8=0272 - 07, R 2007 0598/0=0598 - 07. Edition Number: M-302872-01-1. Date: 2008-06-19. GLP/GEP: yes, unpublished
RA-2616/07	Schmeer, K and Stuke, S	2008	Determination of the residues of AE C656948 and JAU 6476 in/on winter rape after spraying of AE C656948 & JAU 6476 (250 SC) in the field in Northern France, Germany and the United Kingdom. Report No.: BCSRA-2616/07. Report includes Trial Nos.: R 2007 0238/8=0238 - 07, R 2007 0808/4=0808 - 07, R 2007 0809/2=0809 - 07, R 2007 0810/6=0810 - 07. Edition Number: M-308224-01-1. Date: 2008-09-29. GLP/GEP: yes, unpublished
RA-2617/07	Stuke, S and Schmeer, K	2008	Determination of the residues of AE C656948 and JAU 6476 in/on winter rape after spraying of AE C656948 & JAU 6476 (250 SC) in the field in Southern France and Italy. Report No.: BCSRA-2617/07. Report includes Trial Nos.: R 2007 0239/6=0239 - 07, R 2007 0811/4=0811 - 07. Edition Number: M-306557-01-1. Date: 2008-08-26. GLP/GEP: no, unpublished
RA-2620/07	Schmeer, K and Stuke, S	2008	Determination of the residues of AE C656948 and trifloxystrobin in/on head lettuce and lettuce after spraying of AE C656948 & CGA279202 (500 SC) in the greenhouse in Germany, Netherlands and Southern France. Bayer CropScience. Report No.: RA-2620/07. Report includes Trial Nos.: R 2007 0266/3=0266 - 07, R 2007 0642/1=0642 - 07, R 2007 0644/8=0644 - 07, R 2007 0645/6=0645 - 07. Edition Number: M-308622-01-1. Date: 2008-10-06. GLP/GEP: yes, unpublished
RA-2680/07	Ballesteros, C	2008	Determination of the residues of AE C656948 in/on melon after spraying of AE C656948 (500 SC) in the greenhouse in Spain. Bayer CropScience S.A., Lyon, France. Report No.: RA-2680/07. Report includes Trial Nos.: R 2007 0877/7=0877 - 07. Edition Number: M-303139-01-1. Date: 2008-06-26. GLP/GEP: yes, unpublished.
RA-3577/06	Schoening, R, Billian, P, Erler, S and Wolters, A	2007	Determination of the residues of AE C656948 and tebuconazole in/on round cabbage head and the processed fractions (washings; cooking water; head, cooked; head, washed) after spraying of AE C656948 & HWG 1608 (400 SC) in the field in Northern. Bayer CropScience. Report No.: RA-3577/06. Report includes Trial Nos.: R 2006 0354/1=0354 - 06, R 2006 0478/5=0478 - 06. Edition Number: M-295531-01-1. Date: 2007-11-30. GLP/GEP: yes, unpublished
RA-3609/06	Schoening, R, Raecker, T and Telscher, M	2007	Determination of the residues of AE C656948 in/on winter rape seed and the processed fractions (oil, refined; oil, screwpressed; crude oil; extracted meal; oil, solv. extracted; pomace) after spraying of AE C656948 (500 SC) in the field in. Bayer CropScience. Report No.: RA-3609/06. Report includes Trial Nos.: R 2006 0406/8=0406 - 06, R 2006 0409/2=0409 - 06. Edition Number: M-295666-01-1. Date: 2007-12-13. GLP/GEP: yes, unpublished.
RA-3610-06	Schoening, R, Raecker, T and Telscher, M	2007	Determination of the residues of AE C656948 in/on winter rape and summer rape and the processed fractions (oil, refined; oil, screwpressed; crude oil) after spraying of AE C656948 (500 SC) in the field in Southern France and Italy. Bayer CropScience. Report No.: RA-3610-06. Report includes Trial Nos.: R 2006 0625/7=0625 - 06, R 2006 0626/5=0626 - 06. Edition Number: M-295843-01-1. Date: 2007-12-19. GLP/GEP: yes, unpublished.
RAFR00810	Malet, JC and Allard, L	2012	Mesure du niveau de résidu de fluopyram et de trifloxystrobine, après 2 applications de la préparation F413BCS sur framboisier dans le cadre d'une extension d'usage sur la culture - Residues of fluopyram and trifloxystrobine, after 2 applications of F413BCS in raspberry in support of the registration. Ministère de l'Agriculture et de la Pêche, Paris, France. Bayer CropScience. Report No.: RAFR00810. Edition Number: M-434815-01-1. Date: 2012-06-15. GLP/GEP: yes, unpublished
RAFR03509	Malet, JC and Allard, L	2012	Mesure du niveau de résidu de fluopyram et de trifloxystrobine, après 2 applications de la préparation F413BCS sur framboisier dans le cadre d'une extension d'usage sur la culture - Residues of fluopyram and trifloxystrobine, after 2 applications of F413BCS in raspberry in support of the registration. Ministère de l'Agriculture et de la Pêche, Paris, France. Bayer CropScience. Report No.: RAFR03509. Edition Number: M-434818-01-1. Date: 2012-05-07. GLP/GEP: yes, unpublished
RAGML001	Rupprecht, JK and Fischer, DR	2008	AE C656948 500 SC—Magnitude of the residue in/on dry bulb onions. Bayer CropScience LP, Stilwell, KS, USA. Bayer CropScience. Report No.: RAGML001. Edition Number: M-307690-01-1. EPA MRID No.: 47567021. Date: 2008-09-24. GLP/GEP: yes, unpublished

Code	Author(s)	Year	Title, Institute, Report reference
RAGMP034	Lenz, CA	2008	AE C656948 500 SC: Magnitude of the residue in/on canola processed commodities. Bayer CropScience LP, Stilwell, KS, USA. Bayer CropScience. Report No.: RAGMP034. Edition Number: M-306046-01-1. EPA MRID No.: 47567116. Date: 2008-08-13. GLP/GEP: yes, unpublished
RAGMP035	Tang, Z and Fischer, DR	2008	AE C656948 500 SC—Magnitude of the residue in/on canola. Bayer CropScience LP, Stilwell, KS, USA. Bayer CropScience. Report No.: RAGMP035. Edition Number: M-307681-01-1. EPA MRID No.: 47567013. Date: 2008-09-22. GLP/GEP: yes, unpublished
RAGMP076	Fischer, DR and Harbin, AM	2008	AE C656948 500 SC and trifloxystrobin 500 SC—Magnitude of the residue in/on head and stem brassica (crop subgroup 5A). Bayer CropScience LP, Stilwell, KS, USA. Bayer CropScience. Report No.: RAGMP076. Edition Number: M-307503-01-1. EPA MRID No.: 47567025. Date: 2008-09-17. GLP/GEP: yes, unpublished.
RAGMP079	Dallstream, KA and Fischer, DR	2008	AE C656948 500 SC + pyrimethanil 600 SC—Magnitude of the residue in/on caneberry. Bayer CropScience LP, Stilwell, KS, USA. BCS. Report No.: RAGMP079. Edition Number: M-307677-01-1. EPA MRID No.: 47567034. Date: 2008-09-17. GLP/GEP: yes, unpublished
RAGMP081	Timberlake, BC	2008	AE C656948 500 SC—Magnitude of the residue in/on green onions. Bayer CropScience LP, Stilwell, KS, USA. Bayer CropScience. Report No.: RAGMP081. Edition Number: M-306500-01-1. EPA MRID No.: 47567022. Date: 2008-08-27. GLP/GEP: yes, unpublished
RAGMP082	Murphy, IM and Fischer, DR	2008	AE C656948 500 SC—Magnitude of the residue in/on cucurbit vegetables (crop group 9). Bayer CropScience LP, Stilwell, KS, USA. Bayer CropScience. Report No.: RAGMP082. Edition Number: M-307341-01-1. EPA MRID No.: 47567030. Date: 2008-09-17. GLP/GEP: yes, unpublished
RAGMP083	Fischer, DR and Helfrich, KK	2008	AE C656948 500 SC + trifloxystrobin 500 SC—Magnitude of the residue in/on globe herbs (crop subgroup 19A). Bayer CropScience LP, Stilwell, KS, USA. Bayer CropScience. Report No.: RAGMP083. Edition Number: M-307687-01-1. EPA MRID No.: 47567037. Date: 2008-09-23. GLP/GEP: yes, unpublished.
RAGMP085-1	Fischer, DR and Harbin, AM	2008	AE C656948 500 SC and trifloxystrobin 500 SC—Magnitude of the residue in/on leafy vegetables (crop subgroup 4). Bayer CropScience LP, Stilwell, KS, USA. Bayer CropScience. Report No.: RAGMP085-1. Edition Number: M-307507-02-1. EPA MRID No.: 48239917. Date: 2008-09-17. Amended: 2010-09-20. GLP/GEP: yes, unpublished.
RAGMP155	Miller, AL	2010	AE C656948 500 SC, Folicur 3.6 F and Scala 600 SC—Magnitude of the residue in/on dry bulb onions. Bayer CropScience LP, Stilwell, KS, USA. Bayer CropScience. Report No.: RAGMP155. Report includes Trial Nos.: GM052-09HA, GM053-09HA, GM054-09HA. Edition Number: M-366786-01-1. EPA MRID No.: 48239921. Date: 2010-04-12. GLP/GEP: yes, unpublished
RAGMP157	Timberlake, B and Burkett, E	2010	AE C656948 500 SC, Folicur 3.6 F and Gem 500 SC—Magnitude of the residue in/on lettuce. Bayer CropScience LP, Stilwell, KS, USA. Bayer CropScience. Report No.: RAGMP157. Report includes Trial Nos.: GM058-09HA, GM059-09HA, GM060-09HA. Edition Number: M-366788-01-1. EPA MRID No.: 48239923. Date: 2010-04-12. GLP/GEP: yes, unpublished

GLUFOSINATE-AMMONIUM (158)

First draft prepared by Dr D.J. MacLachlan, Department of Agriculture, Australia

EXPLANATION

Glufosinate-ammonium is a herbicide or desiccant. It was first evaluated for residues and toxicology by the 1991 JMPR and re-evaluated (T, R) by the 2012 JMPR. The ADI of glufosinate-ammonium was 0–0.01 mg/kg bw and the ARfD was 0.0 1mg/kg bw. The compound was listed by the 45th Session of CCPR for the JMPR to consider a revised use-pattern for use on soya bean. The residue definition for compliance with MRL and for estimation of dietary intake (for animal and plant commodities) is the sum of glufosinate, 3-[hydroxy(methyl)phosphinoyl]propionic acid and N-acetyl-glufosinate, calculated as glufosinate (free acid).

For the current evaluation the Meeting received critical data required for the estimation of MRLs for soya beans.

RESIDUE ANALYSIS***Stability of pesticide residues in stored analytical samples***

The 2012 JMPR evaluated data on the storage stability of glufosinate-ammonium residues (and metabolites) in plant commodities that included soya bean. The studies concluded residues of glufosinate, NAG and MPP are stable under frozen conditions for at least 24 months in glufosinate-tolerant soya bean seed and for at least 12 months in glufosinate tolerant soya bean hay. The longest storage interval in the current trials was 5.4 months.

USE PATTERN

In the United States the registered use of glufosinate-ammonium for weed control in/on glufosinate-tolerant soya bean was recently amended. Up-dated information on the corresponding label recommendations and the supported use pattern is given in Table 1.

The critical GAP consists in two spray applications before the beginning of bloom, first at the rate of 0.74 kg ai/ha and then at the rate of 0.59 kg ai/ha with a minimum interval of 5 days between the two applications. The pre-harvest interval is 70 days.

The previous label suggested that it was possible to apply at the beginning of bloom and this point now has been specified since the growth stage at the time of application significantly impacts the residues in mature seed at harvest.

Table 1 GAP information on the use of glufosinate-ammonium in/on glufosinate-tolerant soya bean

Crop	Country	Use	Application			PHI
			Rate kg ai/ha	Spray volume L/ha	No.	
LLSoya bean	United States	Post-emergence and up to, but not including, the bloom growth stage	0.45-0.74 for 1 st 0.45-0.59 for 2 nd (max 1.3 kg ai/ha/season)	94-187 L/ha (> 94 L/ha air)	1-2	70 ^a

^a Do not graze the treated crop or cut for hay. Do not use nitrogen solutions as spray carriers. A silicone-based antifoam agent may be added if needed. Do not apply through any type of irrigation system. Sequential applications should be at least 5 days apart.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on supervised residue trials of foliar treatments of glufosinate-ammonium for soya beans (Oakes 2013 TK011619).

Residues, application rates and spray concentrations have been rounded to two figures. Residue data are recorded unadjusted for percentage recoveries or for residue values in control samples. Where multiple analyses were conducted on a single sample, the average value is reported. Residues from the trials conducted according to critical GAP have been used for the estimation of maximum residue levels, STMR and HR values. Those results are underlined.

Table 2 Summary of sprayers, plot sizes and field sample sizes in the supervised trials

Crop	Location	Year	Sprayer	Plot size (m ²)	Sample size (kg)	Sample to analysis interval (months)
Soya	USA	2012	Backpack sprayer	56 m ²	≈2.3	4.9-5.0
Soya	USA	2012	Backpack sprayer	35 m ²	≈0.45	4.0-4.1
Soya	USA	2012	Backpack sprayer	74 m ²	≈2.3	5.1-5.2
Soya	USA	2012	Backpack sprayer	93 m ²	≈2.3	4.5-4.6
Soya	USA	2012	Tractor mounted sprayer	93 m ²	≈2.3	5.3-5.4
Soya	USA	2012	Tractor mounted sprayer	93 m ²	≈2.3	4.7-4.8

Six residue trials with glufosinate-tolerant soya bean (SYHT0H2 event) were conducted in the USA during the 2012 growing season. In each trial the treated plot received two applications of glufosinate-ammonium at the nominal rates of 0.74 kg ai/ha and 0.59 kg ai/ha, respectively, using an SL formulation. The first application was conducted at the growth stages BBCH 12–13 while the second application was conducted 7 to 15 days later, at the growth stages BBCH 14–59. Duplicate samples of soya bean seed were taken at the earliest commercial harvest which was between 86 and 110 days after the last application. Samples were stored frozen for a maximum of 5.4 months before analysis.

Table 3 Residues in glufosinate-tolerant soya bean (mean of replicate samples). Residues are expressed in glufosinate acid equivalents.

Location, year, variety	N (interval)	kg ai/ha	L/ha	GS (BBCH)	matrix	DAT	glufosinate	MPP	NAG	Total
Richland, IA, USA 2012 SYHT0H2 "Jack"	2 (39)	0.74 ^a 0.59 ^a	178 178	13 17	seed	104	0.074	0.022	0.20	<u>0.30</u>
Bagley, IA, USA 2012 SYHT0H2 "Jack"	2 (7)	0.74 ^b 0.65 ^b	178 178	13 15	seed	98	0.290	0.082	0.89	<u>1.3</u>
Cresco, IA, USA 2012 SYHT0H2 "Jack"	2 (9)	0.67 ^a 0.61 ^a	140 140	13 15-16	seed	97	0.058	0.132	0.17	<u>0.36</u>
Geneva, MN, USA 2012 SYHT0H2 "Jack"	2 (9)	0.74 ^a 0.58 ^a	178 168	12-13 14-15	seed	110	0.037	0.030	0.11	<u>0.18</u>
York, NE, USA 2012 SYHT0H2 "Jack"	2 (10)	0.75 ^c 0.59 ^c	187 178	12-13 49-50	seed	86	0.017	0.022	0.044	<u>0.08</u>
Stewardson, IL, USA 2012 SYHT0H2 "Jack"	2 (15)	0.87 ^a 0.60 ^a	159 140	12-13 59	seed	96	0.070	0.072	0.24	0.38

DAT = days after last application

MPP = 3-methyl-phosphinico-propionic acid; NAG = N-acetyl-L-glufosinate.

^a +ammonium sulphate

^b + NIS 0.25% v/v

^c + NIS 0.25% v/v, 2.54 cm overhead irrigation 1 day after application

Table 4 Residues of glufosinate in soya bean seeds from glufosinate-tolerant soya bean in trials reported by the 2012 JMPR and matching the revised US GAP (note all trial locations included application rates that matched GAP, however, for locations where lower application rates were also studied, only the treatment that gave the highest residue is reported below) Residues are expressed in glufosinate acid equivalents

Location, year variety	Application No ^a	kg ai/ha	L/ha	GS ^b	DAT	Residue (mg/kg) glufosinate		MPP	NAG	Total	Reference
Chula USA 2009 S080120	2 (7)	0.73 0.59	160 160	14	123	< 0.01		0.01	0.03	<u>0.04</u>	RAGLP034 +AMS/NIA
Blackville USA 2009 SG4489NLL	2 (6)	0.73 0.60	160 160	14	111	< 0.01		0.02	0.06	<u>0.08</u>	
Proctor AR USA 2009 SG4489NLL	2 (16)	0.45 0.45	140 140	15	79	0.04		0.02	0.33	<u>0.39</u>	
Greenville USA 2009 S080120	2 (9)	0.73 0.59	130 120	15	105	0.06		0.06	0.28	<u>0.40</u>	RAGLP034 +AMS/NIA
Cheneyville USA 2009 S080120	2 (12)	0.75 0.60	150 150	16	85	0.04		0.02	0.45	<u>0.51</u>	RAGLP034 +AMS
					100	0.04		0.02	0.31	<u>0.37</u>	
					110	0.04		0.03	0.36	<u>0.43</u>	
					120	0.06		0.03	0.32	<u>0.41</u>	
					125	0.06		0.03	0.34	<u>0.43</u>	
Springfield USA 2009 S070139	2 (5)	0.73 0.61	130 130	14	78	< 0.01		0.04	0.07	<u>0.11</u>	RAGLP034 +AMS
					95	0.01		0.06	0.14	<u>0.21</u>	
					104	< 0.01		0.07	0.11	<u>0.18</u>	
					112	0.02		0.05	0.15	<u>0.22</u>	
					118	< 0.01		0.06	0.11	<u>0.17</u>	
Bagley IA USA 2009 S080141	2 (14)	0.45 0.45	160 160	15	76	0.06		0.04	0.99	<u>1.09</u>	RAGLP034 +AMS/NIA
Berkley USA 2009 S080141	2 (8)	0.44 0.45	170 160	14	85	0.02		0.04	0.44	<u>0.50</u>	
Geneva MN USA 2009 S080117	2 (10)	0.73 0.60	170 170	14	98	0.02		0.22	0.51	<u>0.75</u>	RAGLP034 +AMS/NIA
Campbell USA 2009 S080119	2 (11)	0.73 0.60	190 190	14	107	0.02		0.02	0.28	<u>0.32</u>	RAGLP034 +AMS
York NE USA 2009 S070139	2 (11)	0.73 0.59	140 140	14	93	0.04		0.07	0.54	<u>0.65</u>	RAGLP034 +AMS
Gardner USA 2009 S070144	2 (6)	0.74 0.61	140 140	14	98	0.02		0.02	0.14	<u>0.18</u>	RAGLP034 +AMS/NIA
Richland IA USA 2009 S070144	2 (15)	0.46 0.45	180 190	15	102	0.01		0.02	0.16	<u>0.19</u>	
Richwood USA 2009 S0701344	2 (8)	0.74 0.45	160 160	14	106	<0.01		0.02	0.08	<u>0.10</u>	
Marysville USA 2009 S0701344	2 (8)	0.45 0.45	170 160	14	106	0.01		0.05	0.26	<u>0.32</u>	
Clarence USA 2009 S070146	2 (7)	0.74 0.59	190 180	14	104	0.01		0.05	0.26	<u>0.32</u>	RAGLP034 +AMS
Lime Springs USA 2009 S080119	2 (10)	0.77 0.59	200 180	14	127	0.03		0.06	0.36	<u>0.45</u>	RAGLP034 +AMS/NIA
Cherry Grove USA 2009 S080119	2 (12)	0.75 0.46	190 190	14	117	0.04		0.04	0.46	<u>0.54</u>	
Seymour USA 2009 S070141	2 (6)	0.67 0.60	160 180	14	82	0.04		0.04	0.22	<u>0.30</u>	RAGLP034 +AMS

DAT = days after last application

^a interval between treatments

^b Soya beans, BBCH growth stages (Meier, 2001)

14 Trifoliate leaf on the fourth node unfolded.

15 Trifoliate leaf on the fifth node unfolded.

16 Trifoliate leaf on the sixth node unfolded.

APPRAISAL

Glufosinate-ammonium is a herbicide or desiccant. It was first evaluated for residues and toxicology by the 1991 JMPR and re-evaluated (T, R) by the 2012 JMPR. The ADI of glufosinate-ammonium was 0–0.01 mg/kg bw and the ARfD was 0.0 1mg/kg bw. The compound was listed by the 45th Session of CCPR for the JMPR to consider residues in soya bean following a revision to the use-pattern.

The residue definition for compliance with MRL and for estimation of dietary intake (for animal and plant commodities) is the sum of glufosinate, 3-[hydroxy(methyl)phosphinoyl]propionic acid and N-acetyl-glufosinate, calculated as glufosinate (free acid).

For the current evaluation the Meeting received critical data required for the estimation of MRLs for soya beans.

Stability of residues in stored analytical samples

The 2012 JMPR evaluated data on the storage stability of glufosinate-ammonium residues (and metabolites) in plant commodities that included soya bean. The studies concluded residues of glufosinate, NAG and MPP are stable under frozen conditions for at least 24 months in glufosinate-tolerant soya bean seed and for at least 12 months in glufosinate tolerant soya bean hay. The longest storage interval in the current trials was 5.4 months.

Results of supervised residue trials in crops

Soya beans, tolerant

The Meeting received field trials performed in the USA involving glufosinate tolerant soya beans. GAP for USA is for (1) one application pre-planting or pre-emergence at 0.59–0.74 kg ai/ha with additional applications from post-emergence to the early bloom growth stage at 0.45–0.59 kg ai/ha with a maximum seasonal rate of 1.3 kg ai/ha/year or (2) post-emergence only with applications from post-emergence to the early bloom growth stage at 0.41–0.50 kg ai/ha with a maximum seasonal rate of 0.91 kg ai/ha/year. The PHI is 70 days. Post-emergent application leads to higher residues.

The use pattern specifies both a last growth stage for application (before the bloom growth stage) and a PHI. The Meeting noted that the interval between the last application and harvest varies significantly depending on the trial location and the soya bean cultivar and that the growth stage at last application was the critical parameter in determining compliance with GAP rather than the PHI.

In trials previously reported by the 2012 JMPR and new trials made available to the current meeting approximating the revised critical GAP in the USA total residues (glyphosate+NAG+MPP) in soya bean seeds were (n=24): 0.04, 0.08, 0.08, 0.1, 0.18, 0.18, 0.19, 0.22, 0.3, 0.3, 0.32, 0.32, 0.32, 0.36, 0.39, 0.4, 0.45, 0.5, 0.51, 0.54, 0.65, 0.75, 1.09, 1.3 mg/kg. The Meeting estimated a median residue of 0.32 mg/kg for use in calculating livestock dietary burdens and a maximum residue level of 2 mg/kg for soya bean, dry to replace its previous recommendation of 3 mg/kg.

Residues for estimation of dietary intake (glufosinate + $0.1 \times [\text{NAG} + \text{MPP}]$) residues were (n=24): 0.02, 0.02, 0.03, 0.04, 0.05, 0.05, 0.07, 0.08, 0.09, 0.09, 0.09, 0.09, 0.09, 0.10, 0.11, 0.11, 0.13, 0.13, 0.14, 0.14, 0.19, 0.25, 0.35 and 0.39 mg/kg. The Meeting estimated an STMR and HR of 0.09 and 0.39 mg/kg respectively for soya bean (dry) for use in calculation of dietary intake.

Fate of residues during processing

Processing factors reported by the 2012 JMPR are used to calculate median residues for aspirated grain fractions, hulls, meal and oil that are used in calculation of livestock dietary burden and also for oil used in calculation of the IEDI and IESTI for glufosinate-ammonium.

Summary of selected processing factors for glufosinate-ammonium

Raw commodity	Processed commodity	Individual PF	Best estimate PF	Median or STMR _{RAC} (mg/kg)	Median or STMR _{RAC} × PF (mg/kg)
Soya bean	Aspirated grain fraction	2.73 8.89	5.81	0.32	1.86
	Hulls	3.15, 11.4	7.275		2.33
	Meal	1.22	1.22		0.39
	Oil	< 0.11 < 0.12 <0.22 < 0.9	< 0.17	0.09	< 0.015

Animal commodity maximum residue levels

The minor changes to the estimated soya bean and soya bean processed commodity levels do not result in significant differences in livestock dietary burdens. The Meeting agreed it is not necessary to make new estimates and recommendations for livestock commodities.

RECOMMENDATIONS

On the basis of the data obtained from supervised residue trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for (compliance with the MRL and for estimation of dietary intake) for animal and plant commodities: *sum of glufosinate, 3-[hydroxy(methyl)phosphinoyl]propionic acid and N-acetyl-glufosinate, calculated as glufosinate (free acid)*

The residue is not fat soluble.

Table of recommendations.

Commodity		Recommended MRL (mg/kg)		STMR or STMR-P (mg/kg)	HR (mg/kg)
CCN	Name	New	Previous		
VD 0541	Soya bean (dry)	2	3	0.09	-

DIETARY RISK ASSESSMENT**Long-term intake**

The Meeting noted that the reduction in the recommended maximum residue level for soya beans would result in lower IEDIs than those previously estimated for glufosinate-ammonium by the 2012 JMPR. The present meeting concluded that the long-term intake of glufosinate-ammonium resulting from the uses considered by the current JMPR is unlikely to present a public health concern.

Short-term intake

The WHO Panel of the 2012 JMPR established an Acute Reference Dose (ARfD) of 0.01 mg/kg bw for glufosinate-ammonium. The IESTI was calculated for soya bean and related commodities using STMR, STMR-P and HR values estimated by the current Meeting. The IESTIs represented 0 to 10% of the ARfD of 0.01 mg/kg bw. The Meeting concluded that the short-term intake of residues of glufosinate-ammonium resulting from uses that have been considered by the JMPR is unlikely to present a public health concern.

REFERENCES

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
RAGLP034 M-388245-01-1	Fischer, DR	2010	Fischer DR. 2010 Ignite 280 SL–Magnitude of the residue in/on soybean. Bayer CropScience Report No. RAGLP034. Bayer CropScience AG Edition No. M-388245-01-1. Unpublished.
	Meier	2001	Meier U (ed.) 2001 BBCH Monograph: Growth stages of mono- and dicotyledonous plants, Federal Biological Research Centre for Agriculture and Forestry, http://www.bba.de/veroeff/bbch/bbcheng.pdf
TK011619	Oakes	2013	Oakes TL. 2013 Magnitude of the residues in or on herbicide tolerant soybean (Event SYHT0H2) Syngenta Crop Protection, Inc., Greensboro, NC, USA Syngenta, Report No.: TK011619, Edition Number: M-470537-01-1 Date: 2013-11-06, unpublished.

IMAZAMOX (276)

The first draft was prepared by Professor Mi-Gyung Lee, Andong National University, Republic of Korea

EXPLANATION

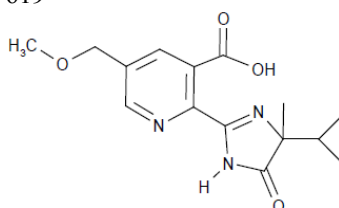
Imazamox is an imidazolinone herbicide used for the control of a wide spectrum of grassy and broadleaf weeds. The mode of action of imazamox is the inhibition of acetohydroxy acid synthase (AHAS), an enzyme involved in the synthesis of essential amino acids (leucine, isoleucine and valine) for the development of the plant.

At the 45th session of the CCPR (2013), it was scheduled for evaluation as a new compound by the current JMPR. Imazamox has been registered in various countries worldwide.

The Meeting received information on physical and chemical properties, animal and plant metabolism, environmental fate, analytical methods, storage stability, use patterns, supervised trials and processing studies.

IDENTITY

ISO common name:	Imazamox
Chemical name	
IUPAC:	(<i>RS</i>)-2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-5-methoxymethylnicotinic acid
CAS:	2-[4,5-dihydro-4-methyl-4-(1-methylethyl)-5-oxo-1 <i>H</i> -imidazol-2-yl]-5-(methoxymethyl)-3-pyridinecarboxylic acid
CAS Registry No.:	114311-32-9
CIPAC No.:	619
Structural formula	



Molecular formula $C_{15}H_{19}N_3O_4$

Molecular mass: 305.3

PHYSICAL AND CHEMICAL PROPERTIES*Purified active ingredient*

Property	Result	Method	References
Melting point:	165.5–167.2 °C	OECD 102	ID-303-001 Coover, 1994a
Relative density:	1.39 at 20 °C	EEC Method A3	ID-301-001 Patel, 1993a
Vapour pressure:	$< 1.33 \times 10^{-5}$ Pa at 25 °C or $< 1.0 \times 10^{-7}$ Torr at 25 °C	US EPA 63-9	ID-306-001 Morelli, 1994a
Volatility:	Henry's Law Constant (k_H) at 25 °C: $< 9.76 \times 10^{-7}$ Pa m ³ mol ⁻¹	Calculation	ID-306-002 Martin, 1997a
Physical state:	Powdered solid at 24.5 °C	Visual inspection, conforming to U.S. EPA registration requirements	ID-301-001 Patel, 1993a

Property	Result	Method	References
		under 40CFR 63-3	
Colour:	White.	ASTM D1535-68	ID-301-001 Patel, 1993a
Odour:	Odourless	ASTM D1292-86	ID-301-001 Patel, 1993a
Solubility in water including effect of pH:	at 25 °C: pH 5: 114 g/L pH 7: > 643 g/L pH 9: > 652 g/L at 10 °C, deionized water: 4.011 g/L at 20 °C, deionized water: 4.413 g/L at 30 °C, deionized water: 4.488 g/L	EEC A.6	ID-310-001 Coover, 1994b
Solubility in organic solvents:	at 25 °C: hexane: 0.0006 g/100 mL solvent methanol: 6.68 g/100 mL solvent acetonitrile: 1.85 g/100 mL solvent toluene: 0.21 g/100 mL solvent acetone: 2.93 g/100 mL solvent dichloromethane: 14.3 g/100 mL solvent ethyl acetate: 1.02 g/100 mL solvent	EEC A.6	ID-310-001 Coover, 1994b
Partition Coefficient:	$K_{ow} = 5.36$ at 25 °C $\log K_{ow} = 0.73$ at 25 °C	US EPA 63-11	ID-315-001 Morelli, 1994b
Hydrolysis rate:	No hydrolysis was observed at pH 4 and pH 7 after 30 days at 50 °C. at pH 9: 70 °C: $t_{1/2} = 1.70$ d 60 °C: $t_{1/2} = 4.17$ d 50 °C: $t_{1/2} = 11.9$ d 25 °C: $t_{1/2} = 192$ d (extrapolated)	EEC C.7	ID-322-002 Holman, 1997b
Photochemical degradation:	at 25 °C: pH 5: $t_{1/2} = 6.8$ h pH 7: $t_{1/2} = 6.7$ h pH 9: $t_{1/2} = 7.1$ h	US EPA 161-2	ID-324-002 An & Ta, 1995a
Dissociation Constant:	$pK_a = 2.3, 3.3, 10.8$ Active substance is the free acid.	US EPA 63-10	ID-320-001 Melcer, 1993a

Technical active ingredient

Property	Result	Method	References
Melting point	166.0–166.7 °C	OECD 102	ID-303-001 Coover, 1994a
Decomposition or sublimation temperature	Decomposition commences ca. 160 °C.	Accelerating rate calorimetry over the temperature range of 50–350 °C	ID-334-001 Patel, 1994a
Physical state:	Powdered solid at 24.5 °C	Visual inspection, conforming to U.S. EPA registration requirements under 40CFR 63-3	ID-301-001 Patel, 1993a
Colour:	Off—white	ASTM D1535-68	ID-301-001 Patel, 1993a
Odour:	Odourless	ASTM D1292-86	ID-301-001 Patel, 1993a
Solubility in water including effect of	at 25 °C: pH 5: 116 g/L	EEC A.6	ID-310-001 Coover,

Property	Result	Method	References
pH:	pH 7: > 626 g/L pH 9: > 628 g/L at 10 °C, deionized water: 3.795 g/L at 20 °C, deionized water: 4.160 g/L at 30 °C, deionized water: 4.193 g/L		1994b
Solubility in organic solvents:	at 25 °C: hexane: 0.0007 g/100 mL solvent methanol: 6.75 g/100 mL solvent acetonitrile: 1.90 g/100 mL solvent toluene: 0.22 g/100 mL solvent acetone: 3.09 g/100 mL solvent dichloromethane: 21.8 g/100 mL solvent ethyl acetate: 1.05 g/100 mL solvent	EEC A.6	ID-310-001 Coover, 1994b

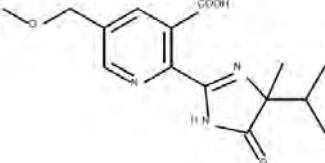
Formulations

The following formulations are commercially available:

Formulation type	Content of imazamox	Other active substances
Aqueous suspension concentrate (SC):	20 g ai/L	560 g ai/L 2,4-D
	17.5 g ai/L	375 g ai/L metazachlor
	25 g ai/L	375 g ai/L metazachlor
	17.5 g ai/L	375 g ai/L metazachlor, 100 g ai/L quinmerac
	6.25 g ai/L	375 g ai/L metazachlor, 125 g ai/L quinmerac
Emulsifiable concentrate (EC):	16.7 g ai/L	250 g ai/L pendimethalin
Soluble concentrate (SL):	40 g ai/L	
	120 g ai/L	
	0.805%	
	33 g ai/L	15 g ai/L imazapyr
	28 g ai/L	600 g ai/L bentazone
	22.4 g ai/L	480 g ai/L bentazone
	20 g ai/L	429 g ai/L bentazone
Water dispersible granule (WG):	70%	
Water dispersible granule (WG):	35%	35% imazethapyr

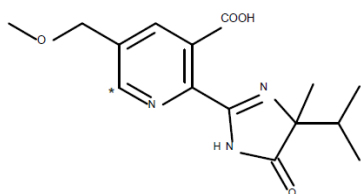
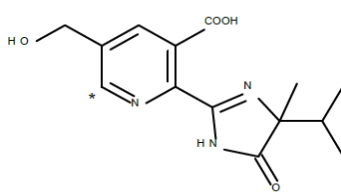
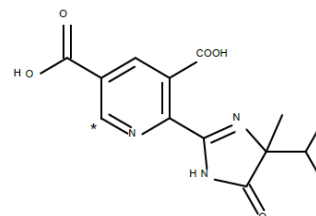
METABOLISM AND ENVIRONMENTAL FATE

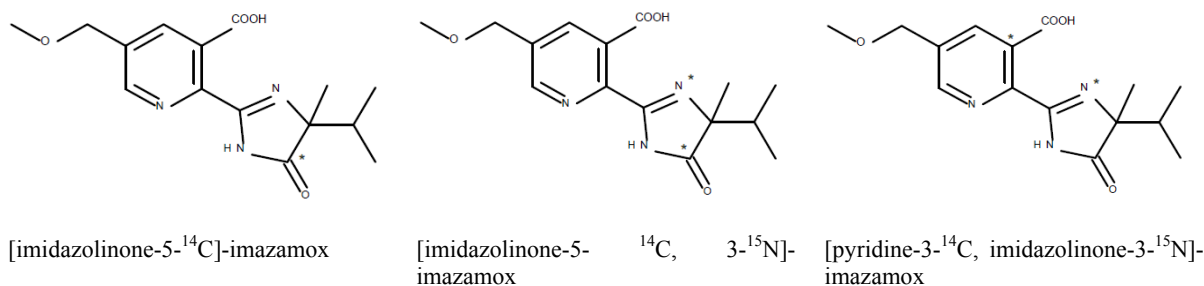
The following links code number and structure or description of the compound appearing in the various metabolism and environmental fate studies.

Code (MW)	Synonyms	IUPAC chemical name	Structure	Found in
CL 299263 (305)	Imazamox; BAS 720 H	(RS)-2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-5-methoxymethylnicotinic acid		rat, goat, hen, oilseed rape, soya bean, pea, alfalfa, maize, wheat, soil, water

Code (MW)	Synonyms	IUPAC chemical name	Structure	Found in
CL 263284 (291)	Reg. No. 4110773; M715H001	5-(hydroxymethyl)-2-(4-isopropyl-4-methyl-5-oxo-2-imazazolin-2-yl) nicotinic acid		oilseed rape, soya bean, pea, alfalfa, maize, wheat
CL 189215 (453.5)	Reg. No. 4110445; M715H002	5-[(β-glucopyranosyl oxy) methyl]-2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl) nicotinic acid		oilseed rape, soya bean, pea, alfalfa, maize, wheat
CL 312622 (305)	Reg. No. 4110542; M720H002	2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-3,5-pyridine-dicarboxylic acid		oilseed rape, soya bean, pea, alfalfa, maize, wheat, soil
CL 354825 (277)	—	5-hydroxy-6-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-nicotinic acid		maize, soil
CL 336554 (323)	—	2-[(1-carbamoyl-1,2-dimethylpropyl) carbamoyl]-5-(methoxymethyl)-nicotinic acid		hydrolysis product at pH 9
CL 152795		2-[(1-carbamoyl-1,2-dimethylpropyl) carbamoyl]-3,5-pyridinedicarboxylic acid		impurity present in dosing solution in animal metabolism study

The metabolism and distribution of imazamox in animals and plants and the fate of imazamox in the environment were investigated using the following radiolabelled compounds (* indicates position of radiolabelling).

[pyridine-6-¹⁴C]-imazamox[pyridine-6-¹⁴C]-CL 263284[pyridine-6-¹⁴C]-CL 312622



Animal metabolism

The Meeting received information on the metabolism of imazamox in ruminants (lactating goats), poultry (laying hens) and laboratory animals (rat). Metabolism studies on rats were reviewed in the framework of toxicological evaluation by the current JMPR and relevant information is summarized below.

Rat

Imazamox administered to rats orally was primarily eliminated as unchanged test compound via the urine and secondly via the faeces. Biliary excretion was not an important route of elimination. Elimination was very efficient and occurred rapidly within 6–48 hours post-dose. In addition, the ratio of enantiomers did not change, demonstrating a similar absorption and excretion rate for the individual enantiomers of imazamox in the rat [Chiu 1995a, ID-440-003; Chiu 1996a, ID-440-004; Thiaener & Lutz 2012a, 2011/1080475].

Lactating goats

Imazamox

Lactating goats were orally given single daily doses of [pyridine-6-¹⁴C]-imazamox for seven consecutive days at a level of either 2.08 or 11.6 ppm in the feed [Johnson 1994a, ID-440-002]. Radiolabelled imazamox was administered orally in gelatin capsules with lactose carrier to lactating goats. Mean feed consumption was 1890 and 1721 g per day for the low and high dose goats during the treatments. The milk, urine, faeces, and blood were collected daily starting one day before the first dosing for each animal group and continued throughout the dosing period. Kidney, liver, leg and loin muscle, and omental fat samples were collected approximately 20 hours after the last dosing. All samples were analysed for radioactivity content.

Samples were analysed by LSC or combustion followed by LSC. The validated detection limit of the radioassay method was approximately 0.01 mg eq/kg for milk, blood, and all tissues. Radioactivity in kidney and urine extracts was characterized using HPLC. Aliquots of the radioactive components isolated from kidney and urine were analysed by GC-MS for confirmation.

During treatment, the total radioactive residues (TRR) in the daily blood and milk samples were less than 0.01 mg eq/kg regardless of the treatment dose levels. The TRR in liver, leg and loin muscle, and omental fat were less than 0.01 mg eq/kg regardless of dose level. The imazamox-derived TRR in the kidney was 0.02 mg eq/kg from the goat dosed at 2.08 ppm and 0.06 ppm from the goat dosed at 11.6 ppm.

Elimination of ¹⁴C radioactivity in the urine accounted for 91.2% and 64.8% of the total dose for the low and high doses, respectively. The radioactivity excreted in the faeces represented 14.8% and 24.0% of the total dose for the low and high doses, respectively. Total recovery of radioactivity in urine and faeces was 106.0% and 88.8% for the low and high doses, respectively.

Radioactive residues from kidney of the high dose goat were extractable (99%) with methanol/water (80:20, v/v). Analysis of the extract by reverse-phase HPLC showed that 89% of the

extractable TRR was attributable to imazamox. The identity of this compound was confirmed by GC-MS. The other radioactivity detected was distributed throughout the chromatogram at levels near background.

That imazamox parent is the only significant compound in tissues is supported by results for urine. Reverse-phase HPLC of goat urine showed 91% of radioactivity was imazamox (confirmed by GC-MS). A radioactive peak containing approximately 5% of the radioactivity recovered from the HPLC column occurred at a shorter retention time than imazamox and the remaining radioactivity occurred as minor components (< 0.5% of radioactivity). The component with a shorter retention time was isolated using HPLC fraction collection, partitioned from the mobile phase using dichloromethane, and reanalysed using HPLC. Of the radioactivity recovered from the HPLC column, 70% was imazamox (confirmed by GC-MS). The most likely explanation for this is that imazamox formed a weak complex with a component in the urine matrix, causing the shorter retention time. This then dissociated during isolation, giving imazamox.

In summary, orally administered imazamox in the goat is mainly excreted in urine. At highly exaggerated dose rates, there were no detectable imazamox-derived residues in milk during and after treatment, nor in tissues except kidney 20 hours after the last dose. The kidney contained 0.02 and 0.06 mg eq/kg total radioactive residue from the low and high dose ^{14}C -treated goats, respectively. This was mostly imazamox. Unaltered parent accounted for most of the excreted residue.

CL 263284

A ruminant metabolism study was conducted with [pyridine-6- ^{14}C]-CL 263284 [Kao 1994a, IA-440-001]. Dose levels for the goats, administered orally in gelatin capsules, were 0, 2.33 or 14.5 mg/kg feed daily for seven days. Samples of blood, milk and excreta were collected daily. After seven days of dosing, the goats were sacrificed (approximately 20 hours after the last dose) and the tissues kidney, liver, muscle and fat were collected. Samples were analysed by LSC or combustion followed by LSC. The validated detection limit of the radioassay method was approximately 0.01 mg eq/kg. Kidney, urine and faeces samples were additionally analysed via HPLC-MS.

TRR levels in most control samples and most tissue samples (muscle, fat and liver) from the low and high dose treated goat and from all milk samples were non-detectable (< 0.01 mg eq/kg). The control sample from kidney showed a residue of 0.01 mg eq/kg and the kidney sample from the high dose goat was at 0.03 mg eq/kg.

Elimination of ^{14}C radioactivity via urine accounted for 14.6% and 18.2% of the total cumulative dose at low dose and high dose, respectively (81.7% and 67.8% via faeces). The recovery of radioactivity in urine and faeces totalled 96.3% and 86.0% at low dose and high dose, respectively.

All milk and all tissue samples contained less than 0.01 mg eq/kg of TRR, the detection limit, except the kidney samples from goat treated at the high dose rate.

Radioactive residues were extracted from kidney using methanol as solvent. The main radioactivity was observed in the extract (91%) and only minor amounts were detected in the PES (9%). The kidney extract was analysed by HPLC to determine the nature of the radioactive residue. The administered compound (CL 263284) was the minor radioactive residue (9% TRR, < 0.01 mg/kg). One further peak was present in the chromatogram (M1; 78% TRR, 0.02 mg eq/kg). M1 was further characterized by treatment with β -glucuronidase, sulfatase or buffer alone. M1 showed to be a very labile component and was easily degraded to CL 263284 under non-enzymatic or enzymatic hydrolysis. Because M1 was converted to CL 263284 in buffer (0.2 M potassium phosphate, pH 6.8) alone, it is likely that M1 was the parent compound weakly coupled with kidney endogenous components. It is suspected that M1 is a salt or conjugate of CL 263284 that is readily dissociated in aqueous solutions such as buffer.

HPLC analysis of the faeces extract of goat treated at high dose showed that 96% of the radioactivity had a retention time corresponding to that of CL 263284 reference standard. HPLC analysis of the faeces extract of goat treated at low dose showed that the major component had a retention time at 9 minutes which is the same as the M1 component found in the high dose kidney.

The M1 component was very labile and was easily converted to CL 263284 under aqueous, acidic conditions or incubated with β -glucuronidase and sulfatase.

The results of this lactating goat study showed that the administered compound was excreted without retention or accumulation in milk. Of the edible tissues only kidney showed detectable residues and these were identified as the administered compound CL 263284 and a labile metabolite that converted to CL 263284 in aqueous solution. This shows a very low transfer of CL 263284-derived residues from animal feed into tissues and milk of lactating ruminants.

CL 312622

Lactating goats were orally treated with a mixture of [pyridine-6- ^{13}C] and [pyrindine-6- ^{14}C]-radiolabelled imazamox metabolite (CL 312622) [Tsaltz 1999a, ID-440-006]. The three goats were dosed for five consecutive days with the nominal dietary equivalent of 0 ppm (control), 3.1 ppm (low dose) and 33 ppm (high dose) in the feed. Milk, urine and faeces samples were collected daily and edible tissues (muscle, fat, liver and kidney) were collected at sacrifice (approximately 22 hours after the last dose). Samples were analysed by LSC or combustion followed by LSC. The specific radioactivity afforded a validated detection limit of 0.006 mg eq/kg CL 312622 equivalents in the tissues, milk, urine and faeces.

TRR levels in all tissue and milk samples were non-detected (< 0.006 mg eq/kg), except kidney (0.025 mg eq/kg) and liver (0.009 mg eq/kg) of goats treated at high dose (33.4 mg/kg feed). Elimination of ^{14}C radioactivity via urine accounted for 8.5% and 7.0% of the total cumulative dose at low dose and high dose, respectively (90.4% and 90.0% via faeces). The recovery of radioactivity in urine and faeces totalled 98.9% and 97.0% at low dose and high dose, respectively.

Radioactive residues were extracted from kidney using acetone/water (3:1, v/v) as solvent. The overall recovery in the extractable portion was $> 100\%$ (by comparison with the TRR obtained by combustion); no radioactivity was observed in the PES. The kidney extract was analysed by HPLC to determine the nature of the radioactive residue. The administered CL 312622 was the predominant radioactive residue (60.0%). The impurity known to be present in the dosing solution (CL 152795) was also found in the extract at 11.0% of TRR.

Minor polar unknowns (total 10.3% of TRR) and non-polar unknowns (total 11.9% of TRR) were also present in the kidney extract. Since these fractions were equivalent to 0.003 mg eq/kg and contained multiple components, no further characterization was attempted.

Radioactive residues were extracted from liver according to a modification of the kidney extraction procedure. The percent extractable radioactivity was 98.7% and the percent TRR in the PES by combustion/LSC analysis yielded residues less than the validated detection limit.

The concentrated extract was analysed by HPLC after solid phase extraction (SPE) clean-up. The administered CL 312622 was present at 38.2% of TRR in the liver. The impurity known to be present in the dosing solution (CL 152795) was also found in the liver extract at 19.0% of the TRR as determined by HPLC. Polar radioactive components were present in the liver extract at 31.2% of the TRR. The majority of that fraction (22.9%) was not retained by the SPE cartridge, indicating it to be very polar material. Some non-polar metabolites were also present at a total concentration of 11.3% of the TRR. Since these fractions were equivalent to 0.003 mg eq/kg or lower and contained multiple components, no further characterization was attempted.

Data on the distribution of the recovered radioactivity from the kidney and the liver extract, as determined by HPLC, are summarized in table below.

Table 1 Distribution of radioactivity in high dose goat kidney and liver goat treated with [^{14}C]-CL 312622 (ID-440-006)

Compound	Kidney (0.025 mg eq/kg)		Liver (0.009 mg eq/kg)	
	% TRR	mg eq/kg	% TRR	mg eq/kg
CL 312622	60.0	0.015	38.2	0.003
CL 152795	11.0	0.003	19.0	0.002

Polar unknowns	10.3	0.003	31.2	0.003
Non-polar unknowns	11.9	0.003	11.3	0.001

Kidney and liver of goat administered with 33.4 mg/kg feed

CL 152795: impurity present in dosing solution

The results of this CL 312622 study showed that the administered compound (CL 312622) and the CL 152795 compound (present as an impurity in the dosing solution) were excreted with minimal retention by the kidney and the liver at the exaggerated dose of 33.4 ppm in the feed. There was no accumulation in any other edible goat tissue or in the milk. There were no residues found in the tissues or milk of the low dose treated goat.

Laying hens

Imazamox

Laying hens were orally dosed with [pyridine-6-¹⁴C]-imazamox [Johnson 1994b, Johnson 1994b, (IA-440-002)]. Feeding levels for each group consisting of eight white Leghorn hens were 0, 2.11 and 10.2 ppm in the feed daily by gelatin capsule for seven consecutive days. Eggs were collected twice daily and blood and the tissues (liver, kidney, muscle, and skin with adhering fat) were collected for analysis approximately 22 hours after the last dose. Samples were analysed by combustion followed by LSC. The validated detection limit of the radioassay method was approximately 0.01 mg eq/kg for eggs, blood and all tissues.

Residues in eggs, blood, skin with adhering fat, muscle, liver, and kidney tissues were all less than 0.01 mg eq/kg. Recovery of ¹⁴C in excreta collected over the 7 day treatment period averaged 85.5% of the total administered dose at 2.11 ppm feeding level and 84.8% at 10.2 ppm feeding level.

The results of this study indicate that imazamox-related residues do not accumulate in eggs or edible tissues of poultry. Orally administered imazamox was mainly eliminated from the hen through excreta. No detectable (< 0.01 mg eq/kg) [¹⁴C]imazamox-derived residues in eggs or edible tissue were found.

CL 263284

A poultry metabolism study was conducted with [pyridine-6-¹⁴C]-radiolabelled imazamox metabolite CL 263284 [Afzal 1994a, IA-440-002]. Hens of each group containing eight animals were dosed orally by gelatin capsules at 0, 2.14 and 10.9 ppm in the feed daily for seven days. Eggs and excreta were collected daily. After seven days of dosing, the hens were sacrificed and the tissues (liver, kidney, muscle and skin with adhering fat) and blood were collected for analysis approximately 22 hours after the last dose. Samples were analysed by combustion followed by LSC. The validated detection limit of the radioassay method was 0.01 mg eq/kg CL 263284 equivalents in the tissues, eggs and excreta.

Total recovery of radioactivity in excreta was 85.3% and 88.6% for the low and high dose, respectively. Residues in all tissues, blood and eggs were less than 0.01 mg eq/kg, the validated detection limit.

The results of this study indicate that the administered CL 263284-related residues do not accumulate in eggs or edible tissues of poultry. Orally administered imazamox was mainly eliminated from the hen through excreta. No detectable (< 0.01 mg eq/kg) ¹⁴C-CL 263284-derived residues in eggs or edible tissue were found.

CL 312622

Laying hens were orally treated with a mixture of [pyridine-6-¹³C] and [pyridine-6-¹⁴C]-radiolabelled imazamox metabolite CL 312622 [Afzal 1999a, ID-440-005]. The eight hens (Hy Line W-36, White Leghorn) by each group were dosed for five consecutive days with 0 ppm feed (control), 0.13 ppm feed (low dose) and 10.5 ppm feed (high dose). Eggs were collected twice daily and bile and the edible tissues (muscle, liver and skin with adhering fat) were collected at sacrifice approximately 22

hours after the last dose. Samples were analysed by combustion followed by LSC. The validated detection limit of the radioassay method was 0.01 mg/kg CL 312622 equivalents in the tissues, eggs and excreta. Detection of CL 312622-related residues in excreta extract was accomplished by HPLC-UV. The radioactivity was quantitated by LSC.

With the exception of high dose composite bile sample (0.018 mg eq/kg), residue in tissues (liver, muscle and skin with adhering fat), eggs and bile were all less than or equal to the validated detection limit (0.006 mg/kg) of the radioassay. Recovery of ^{14}C in excreta collected over the five day treatment period averaged 87.5% and 90.9% of the total administered dose from the low dose (0.13 ppm feed) and high dose (10.5 ppm feed), respectively. These data showed that the administered CL 312622 or derived residues as well as CL 152795 (present as an impurity in the dosing solution) were excreted without retention or accumulation in eggs and edible poultry tissues.

The extremely low residues in eggs and edible tissues precluded further characterization. The high dose day-2 excreta sample was extracted with methanol. The extractability of ^{14}C residue was 91.2%. The extracted ^{14}C residue was analysed by HPLC. The major component of the extractable residue was unchanged CL 312622, accounting for 76.7% of the extractable radioactive residue. The polar dosing solution impurity (CL 152795) accounted for approximately 6.5% of the ERR. All other components were insignificant and no single component exceeded 5% of the TRR.

Table 2 Distribution of radioactivity in high dose hen excreta of day 2 treated with [^{14}C]-CL 312622 (ID-440-005)

Compound	Excreta (9.2 mg eq/kg)	
	% TRR	mg eq/kg
CL 312622	70.0	7.0
CL 152795	5.9	0.6
Non-polar unknowns / Methyl ester	2.0	0.2

Excreta of hen administered with 10.5 ppm.

Plant metabolism

The Meeting received information on the fate of imazamox in oilseed rape, soya bean, pea, maize, wheat and alfalfa.

Oilseed rape

Study 1

A metabolism study was conducted to investigate the amount of imazamox residues and the nature of its degradation products in summer oilseed rape after foliar application [Radzom 2013a, 2011/1281377]. Oilseed rape (imidazolinone herbicide-tolerant variety: Salsa CL) was sown into ten plastic containers (total test area, external size = 2.4 m²) filled with sandy loam soil, which were located in climatic chambers (phytotrons). The phytotrons simulated the natural climatic conditions of a typical rapeseed-growing area.

Under the indoor condition, the crop was treated once with [imidazolinone-5- ^{14}C , 3- ^{15}N]-imazamox at growth stage BBCH 10–18 with a rate of 0.075 kg ai/ha. Labelled ^{14}C , ^{15}N -imazamox and unlabelled ^{12}C -imazamox were mixed at a ratio of 2:1 (^{14}C , ^{15}N -imazamox: ^{12}C -imazamox). Blank formulation (40 g ai/kg blank formulation), adjuvant BAS 160 00 S (1 L/ha) and water was added. The application solution was monitored for purity by HPLC and the isotope pattern was determined by mass spectrometry. The formulation in a spray volume of 216 L/ha was applied with an automatic spray track. Forage samples were taken 12 days after treatment (BBCH 39). At harvest (90 DAT, BBCH 89), mature oilseed rape pods and straw were cut off with scissors. Hull and seed were separated using a thresher. All samples were stored in a freezer at approximately $-18\text{ }^{\circ}\text{C}$ or below. The storage conditions stayed the same until analysis

started and during the whole period of the metabolism study. Extracts were stored in a refrigerator or, for longer periods, in a freezer.

The plant material (forage, straw, hull and seed) were extracted with methanol and water. The combined extracts were measured by LSC. The residual radioactive residues after solvent extraction were combusted. On the other hand, a sequential solubilisation procedure was applied for the PES of rape straw, hull and seed. The residues were extracted with aqueous ammonia solution (1%) and subsequently by treatment with protease (only seed) and with cellulase and macerozyme. The extracts and residues were analysed by LSC.

Methanol extracts of rape forage, straw and hull were directly investigated by LC-MS. Peak assignment in the other samples was done by comparing the metabolite patterns with those of the extracts investigated by MS-analysis and by comparison of the retention times of the identified components (CL 263284, CL 312622) with the ^{14}C -signals of the quantitative and confirmatory HPLC analyses. Additionally, a co-chromatography experiment with the reference items CL 312622 and CL 263284 was performed with the water extract of rape straw. In order to investigate, if cleavage of the imazamox ring systems occurs, reference item dimethylhydantoin was studied with respect to its chromatographic properties to check for similar degradation products. According to retention time comparison, in the HPLC chromatograms of oilseed rape matrices no corresponding signals were detected and therefore no formation of cleavage products was observed.

The TRR of rape forage was 1.004 mg eq/kg, straw 1.100 mg eq/kg, hull 2.448 mg eq/kg, seed 0.149 mg eq/kg, when determined by direct combustion.

Table 3 Total radioactive residues (TRR) in oilseed rape samples following foliar application of [^{14}C , ^{15}N]-imazamox (2011/1281377)

Radioactive residues in treated oilseed rape			
Matrix	Days after treatment	TRR determined by direct combustion (mg eq/kg)	TRR calc. (mg eq/kg)
Forage	12	1.004	0.889
Straw	90	1.100	1.134
Hull	90	2.488	2.527
Seed	90	0.149	0.152

The extractability of rape forage with methanol and water was 97.7% TRR (0.868 mg eq/kg) with the major part of the residues extracted with methanol (0.823 mg eq/kg, 92.6% TRR). The extractability of rape straw and hull with methanol and water was 77.6% TRR (0.879 mg eq/kg) and 78.8% TRR (1.991 mg eq/kg), respectively. The major part of the residues was also extracted with methanol (50.5% TRR in straw and 57.7% TRR in hull). From rape seed, 58.3% TRR (0.089 mg eq/kg) was extracted by solvent extraction (methanol and water), with more radioactivity was extracted with water (34.3% TRR) than with methanol (24.0% TRR).

Table 4 Distribution of residues of [^{14}C , ^{15}N]-imazamox in oilseed rape samples (2011/1281377)

Matrix	Days after treatment	TRR calc. ^a mg eq/kg	Extracts				total		PES	
			methanol extract		aqueous extract		mg eq/kg	%TRR	mg eq/kg	%TRR
			mg eq/kg	%TRR	mg eq/kg	%TRR				
Forage	12	0.889	0.823	92.6	0.045	5.0	0.868	97.7	0.021	2.3
Straw	90	1.134	0.573	50.5	0.307	27.1	0.879	77.6	0.254	22.4
Hull	90	2.527	1.458	57.7	0.533	21.1	1.991	78.8	0.536	21.2
Seed	90	0.152	0.036	24.0	0.052	34.3	0.089	58.3	0.063	41.7

^a TRR calc.: sum of extracted + PES

Table 5 Metabolites detected in oilseed rape matrices following foliar application of [^{14}C , ^{15}N]-imazamox (2011/1281377)

Components	Forage		Straw		Hull		Seed	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Imazamox	0.373	41.9	n.d.		n.d.		n.d.	
CL 263284	0.476	53.5	0.496	43.7	1.776	70.3	0.047	31.0
CL 312622	0.076	8.6	0.296	26.2	0.137	5.4	0.004	2.8
Total identified from extracts	0.925	104.0	0.792	69.9	1.913	75.7	0.051	33.8
Total characterized from extracts	0.063	7.1	0.230	20.3	0.261	10.3	0.037	24.2
Total identified and/or characterized from extracts	0.988	111.2	1.022	90.1	2.174	86.0	0.088	58.0
Unextracted (PES)	0.021	2.3	0.131	11.6	0.323	12.8	0.040	26.5
Grand total	1.009	113.5	1.153	101.7	2.496	98.8	0.146	96.0

Analysis of the methanol extract of rape forage resulted in a pattern of two major and four minor peaks, of which three were identified by direct LC-MS analysis of the methanol extract. One of the two prominent peaks was identified as metabolite CL 263284 and accounted for 0.476 mg eq/kg (53.5% TRR). Parent compound imazamox was the second most abundant component and accounted for 0.373 mg/kg (41.9%). Metabolite CL 312622 was identified at 8.6% TRR. The remaining components were detected at levels below 1.1% TRR and characterised by their chromatographic properties. The same methanol extract was additionally analysed using HPLC that confirmed the metabolite pattern of the quantitative analysis. The respective water extract and the residue obtained after solvent extraction were not further investigated due to their low amounts of radioactive residues. In the extracts, 104.0% of the total radioactive residues were identified and 7.1% were characterised by HPLC analysis or their extractability with water.

Analysis of the methanol extract of rape straw resulted in a pattern of one main peak and seven minor peaks. The main peak was identified as metabolite CL 263284 and accounted for 0.398 mg eq/kg (35.1% TRR). The metabolite CL 312622 was identified at a level of 0.071 mg eq/kg (6.3% TRR). Parent compound imazamox was not detected. The remaining components were detected at levels below 3.7% TRR. HPLC analysis of the water extract of rape straw yielded a pattern of three peaks. The most abundant peak corresponded to the metabolite CL 312622 (0.212 mg eq/kg, 8.7% TRR). The second most abundant component was metabolite CL 263284, which accounted for 0.066 mg eq/kg (5.8% TRR). The PES was further solubilised with ammonia, and a mixture of macerozyme and cellulase. The first solubilisation step with aqueous ammonia solubilised a portion of 0.069 mg eq/kg (6.1% TRR) of the radioactive residues, which may have been incompletely extracted with methanol and water or weakly associated with insoluble plant material. In the subsequent solubilisation step, the NH_4OH residue was incubated with a mixture of macerozyme and cellulase which released 0.045 mg eq/kg (4.0% TRR). Analysis of the ammonia extract resulted in the identification of CL 263284 and metabolite CL 312622, which accounted for up to 1.5% of the TRR. In the supernatant obtained after treatment with macerozyme and cellulase, CL 263284 (1.3% TRR) was identified. In the extracts of rape straw, 66.0% of the total radioactive residues were identified and 14.4% TRR were characterised by HPLC. In the PES, additional 3.9% TRR were identified and 5.9% TRR were characterised by their HPLC elution behaviour.

Analysis of the methanol extract of rape seed hull resulted in a pattern of one main peak and five minor peaks. The main peak was identified as metabolite CL 263284 and accounted for 1.321 mg eq/kg (52.3% TRR). The metabolite CL 312622 was identified at a level of 0.084 mg eq/kg (3.3% TRR). The remaining components were detected at levels below 1.4% TRR and characterised by their chromatographic properties. Likewise, in the water extract again the main constituent was CL 263284 at 0.348 mg eq/kg (13.8% TRR) detected. CL 312622 was identified at 0.049 mg eq/kg (1.6% TRR). The remaining five minor peaks were detected at levels at or below 2.0% TRR and characterised by their chromatographic properties. The residue after solvent extraction was further solubilised with ammonia, and a mixture of macerozyme and cellulase. The first solubilisation step with aqueous ammonia solubilised a portion of 0.114 mg/kg (4.5% TRR) and subsequent solubilisation step with macerozyme and cellulase released 0.082 mg eq/kg (3.2% TRR). Analysis of

the ammonia extract resulted in the identification of CL 263284 and metabolite CL 312622, which accounted for up to 2.4% of the TRR. In the supernatant obtained after treatment with macerozyme and cellulase, CL 263284 (1.8% TRR) was identified. In the extracts of rape hull, 71.0% of the total radioactive residues were identified and 8.3% TRR were characterised by HPLC. In the PES, additional 4.7% TRR were identified and 2.0% TRR were characterised by their HPLC elution behaviour.

Analysis of the methanol extract of rape seed resulted in a pattern of two peaks. The peak at 24.8 min was identified as metabolite CL 263284 and accounted for 0.020 mg eq/kg (13.3% TRR). One polar component eluted at 3.4 min and was detected at 0.017 mg eq/kg (11.1% TRR). In the confirmatory HPLC chromatogram the peak eluting in the polar region was split up into three components each below or equal to 0.009 mg eq/kg (5.9% TRR). For further investigation, the polar fraction was isolated by SPE fractionation and analysed by HPLC. Both chromatograms confirmed that the polar fraction consist of at least two components. In the water extract the main constituent was identified as CL 263284 at 0.027 mg eq/kg (17.8% TRR). CL 312622 was identified at 0.004 mg eq/kg (2.8% TRR). The remaining non-identified components were detected at levels of up to 0.007 mg eq/kg (4.3% TRR). The residue after solvent extraction was further solubilised with ammonia, protease and a mixture of macerozyme and cellulase. The first solubilisation step with aqueous ammonia solubilised a portion of 0.015 mg eq/kg (9.8% TRR) and solubilisation with protease and macerozyme/cellulase released 11.9% TRR and 4.8% TRR, respectively. An aliquot of the concentrated protease solubilisate was analysed and some minor components below or equal to 0.003 mg eq/kg (1.9% TRR) eluting in the polar region were detected. In the ERR, 33.8% of the total radioactive residues were identified and 24.2% were characterised by HPLC. In the PES, additional 9.4% TRR were characterised by HPLC analysis of the protease supernatant. In addition, 14.6% TRR were characterised by solubilisation with aqueous ammonia and a mixture of macerozyme and cellulase.

In order to analyse whether one enantiomer of imazamox and CL 263284 was preferably metabolised in oilseed rape, enantiomer-specific analyses were performed in all four matrices (forage, straw, hull and seed). For the test item and the unlabelled reference item CL 263284 the ratio of enantiomer 1 to enantiomer 2 was found to be approximately 50:50. For the determination of the enantiomer ratio in the different matrices, the parent compound imazamox and metabolite CL 263284 were isolated from the methanol extracts and analysed using HPLC. The enantiomer ratio of imazamox in forage was found to be about 30:70 and the enantiomer ratio of its metabolite CL 263284 was found to be between approximately 40:50 in forage and straw and about 60:40 in hull and seed.

Study 2

A metabolism study was conducted with [pyridine-6-¹⁴C]-imazamox and [pyridine-6-¹³C]-imazamox [McDonnell 1995a, ID-640-003]. The in-life phase of the study was conducted by the EnviroQuest facilities in Minto, Manitoba, Canada in imidazolinone-resistant oilseed rape variety NS 1471. The oilseed rape was maintained under normal outdoor agronomic practices for the region. The test substance was applied as a single post-emergence broadcast spray to a 5.6 m² plot of established oilseed rape seedlings (3- to 4-leaf stage). The application rate was 0.020 kg ai/ha. Plants samples were collected after application on the day of treatment (0 DAT). Seed was collected at maturity (82 DAT). All samples were frozen within 3 hours of the time of sampling and were kept until assay in a freezer, at approximately -20 °C. The crop samples were processed, extracted and analysed within 14 to 42 days of harvest. Plant samples were combusted using an oxidizer and the radioactive residues were assayed by LSC.

The immature oilseed rape foliage sample (0 DAT) was extracted with water/methanol (2:8, v/v). The extract was subjected to radioassay and HPLC analysis. The harvest seeds were processed to oilseed rape oil and the resultant meal. A finely ground seed sample was extracted twice with hexane. The hexane extract was evaporated and the remaining oily residue was presumed to be rape oil, while the air dried PES (post extraction solid) was presumed to be the meal. Radioactive residues in the oil and seed were assayed by LSC.

The TRR in canola foliage (0 DAT) was 2.14 mg eq/kg. The presence of [^{14}C]imazamox (91.9% TRR) was confirmed by comparison of retention time with that of known analytical standard. There were no peaks of radioactivity comprising greater than 2% impurity in the radio-profile.

In the seed, the TRR was below the detectable amount of radioactivity, 0.002 mg eq/kg. The TRR in the canola oil was below the detectable amount of radioactivity (< 0.004 mg eq/kg). No detectable TRR was present in the oil meal.

Study 3

A small plot field study on the metabolic fate of imazamox in oilseed rape was conducted during the oilseed rape-growing season in 1998 [Roman 1999b, ID-640-009]. The field phase of this study was conducted in outdoor fenced-off research plots at EnviroQuest, Minto, Manitoba, Canada where imidazolinone-tolerant oilseed rape variety 45A71 received a single post-emergence treatment with [pyridine-6- ^{14}C]-imazamox. Plot A was treated at a rate of 0.051 kg ai/ha. Plot B was treated at a rate of 0.089 kg ai/ha. The test solution was sprayed evenly over the entire plot of the oilseed rape at the 3 to 4-leaf stage. The spray volume of 280.5 L/ha and the application timing simulated the actual field use conditions.

Oilseed rape plant specimens were sampled directly after the application (0 DAT), foliage was sampled at 22 DAT and the last sampling of seeds and straw took place at 78 DAT. Green plants (0 and 22 DAT) were cut into small pieces and then ground into a fine powder with dry ice. Straw collected at later sampling intervals was first homogenized in dry ice. The seed was manually cleaned of any remaining chaff. The resulting chaff was combined with straw and processed as part of the straw sample. The seed samples were ground into a fine powder. All processed samples were stored frozen until used. The crop samples were processed, extracted, and analysed within 9 to 401 days of harvest. Rape plant and soil samples were combusted and radioactive residues were quantitated by LSC.

Finely ground oilseed rape samples were extracted three times with methanol/water (80:20, v/v) and then once with a solvent mixture of methanol/acetone/water (1:1:1, v/v/v). The radioactive residues in the combined extracts (neutral organo-extractable) were assayed by LSC. The extract was analysed for the radioactive components by HPLC. Oilseed rape seed from plot treated at a 0.089 kg ai/ha was extracted with hexane. The resulting extracts were assayed by LSC.

The remaining non-neutral organo-extractable residues in all crop samples were extracted once with 2% HCl in methanol/water (4:1). The remaining residues in straw samples were further treated with *Penicillium* cellulase and subsequently by 6 N HCl reflux. The extracts and hydrolysates were analysed by combustion.

The TRR in the rape foliage taken at 0 DAT was 2.208 mg eq/kg at a rate of 0.051 kg ai/ha (low rate) and 3.943 mg eq/kg at a rate of 0.089 kg ai/ha (high rate). The level of the radioactive residues, at the low and high rate, declined to 0.040 mg eq/kg and 0.130 mg eq/kg at 22 DAT but increased to 0.088 mg eq/kg and 0.178 mg eq/kg in the straw presumably due to dehydration. The concentration of the TRR in the mature seed (78 DAT) was relatively low as 0.004 mg eq/kg at a low rate and 0.006 mg eq/kg at a high rate.

Table 6 Total radioactive residues (TRRs) in oilseed rape plant, foliage, straw and seed samples after application of [^{14}C]-imazamox

TRRs in treated oilseed rape plant, foliage, straw, seed and soil			
Matrix	Days after treatment	TRR (mg eq/kg)	
		0.051 kg ai/ha	0.089 kg ai/ha
Plant	0	2.208	3.943
Foliage	22	0.040	0.130
Straw	78	0.088	0.178
Seed	78	0.004	0.006

Table 7 Distribution of residues of [¹⁴C]-imazamox in rape samples (ID-640-009)

			Extracts						PES	
Matrix	DAT	TRR calc.	neutral organo-extractable		2% HCl in methanol		total			
		(mg eq /kg)	(mg eq /kg)	(%TRR)	(mg eq /kg)	(%TRR)	(mg eq /kg)	(%TRR)	(mg eq /kg)	(%TRR)
0.051 kg ai/ha										
Plant	0	2.208	2.142	97.00	0.026	1.17	2.168	98.17	0.040	1.83
Foliage	22	0.040	0.037	92.11	0.001	2.56	0.038	94.67	0.002	5.34
Straw	78	0.088	0.067	75.89	0.004	4.60	0.071	80.49	0.017	19.51
Seed	78	0.004	n.d.		n.d.		n.d.			
0.089 kg ai/ha										
Plant	0	3.943	3.840	97.38	0.049	1.24	3.889	98.62	0.055	1.39
Foliage	22	0.130	0.120	92.04	0.006	4.51	0.126	96.55	0.004	3.45
Straw	78	0.178	0.140	78.42	0.007	4.13	0.147	82.55	0.031	17.44
Seed	78	0.006	0.003	56.96	0.000	5.17	0.003	62.13	0.002	37.86

TRR calc.: sum of extracted + PES

Neutral organo-extractable: the extraction was done with 20% aqueous methanol three times followed by methanol/acetone/water once.

n.d.: Not detected, < 0.001 mg eq/kg

In plant, forage, straw and seed, 62.1% to 98.6% of the TRR were extracted with aqueous organic solvent mixtures. The majority (57.0–97.4% TRR) was extracted with 80% aqueous methanol three times and with methanol/acetone/water (1:1:1, v/v/v). Increase in extractability by acidic organic solvent (2% HCl in methanol) was small (1.17–5.17% TRR). The unextracted radioactivity from rape plant, foliage and straw accounted for 1.4 % to 19.5% of the TRR. In the seed, the extractability of the residues was 62.1% (0.003 mg eq/kg) of the TRR. The radioactivity remaining in seed accounted for 37.9% (0.002 mg/kg) of the TRR. In the dry straw, Penicillin cellulose and 6 N HCl reflux released 2.0–2.5% TRR and 7.4–9.4% TRR, respectively. A small extractability by enzyme treatment suggested that the presence of glycosidic linkages of the residue with endocons in the oilseed rape samples is insignificant. Due to the very low residue levels (0.002–0.004 mg eq/kg) in the enzyme hydrolysates of straw sample, no attempts were made to further characterize the nature of the residues.

In rape plant at 0 DAT, residue level of the unchanged imazamox was 1.726 mg /kg or 3.068 mg /kg (78% TRR in both). Imazamox accounted for 21% or 16% TRR in forage (22 DAT), 2% TRR in straw (78 DAT). Imazamox was extensively metabolized in the late growing and matured oilseed rape plants.

Each of the metabolites (CL 189215, CL 263284 and CL 312622) was found in rape at 0 DAT as a minor with none exceeding 2.6% of the TRR. At 22 DAT and 78 DAT, the hydroxymethyl metabolite CL 263284 accounted for 37.4% or 36.0% in forage and 49.6% or 46.4% in straw. Its glucoside CL 189215 and di-acid metabolite CL 312622 were present in very low level (below 8.6% TRR, 0.003 mg eq/kg).

Table 8 Metabolites detected in oilseed rape matrices following foliar application of 0.051 kg ai/ha [¹⁴C]-imazamox

Components	Plant (0 DAT)		Foliage (22 DAT)		Straw (78 DAT)		Seed (78 DAT)	
	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)
Imazamox	1.726	78.2	0.008	21.3	0.002	2.1	n.d.	
CL 189215	0.028	1.3	0.001	3.1	0.002	1.7	n.d.	
CL 263284	0.053	2.4	0.014	37.4	0.044	49.6	n.d.	
CL 312622	0.045	2.0	0.003	8.6	0.001	1.4	n.d.	
Total identified from extracts	1.852	83.9	0.026	70.4	0.049	54.8	n.d.	
Total characterized from extracts	0.290	13.1	0.009	21.7	0.019	21.2	n.d.	
Total identified and/or characterized from extracts	2.142	97.0	0.035	92.1	0.068	76.0	n.d.	

Components	Plant (0 DAT)		Foliage (22 DAT)		Straw (78 DAT)		Seed (78 DAT)	
	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)
2% HCl in aqueous MeOH (unidentified compounds)	0.026	1.17	0.001	2.56	0.004	4.60	n.d.	
Enzyme-released (cellulase)	n.a.	n.a.	n.a.	n.a.	0.002	2.46	n.d.	
6 N HCl Reflux	n.a.	n.a.	n.a.	n.a.	0.008	9.37	n.d.	
Unextracted (PES)	0.040	1.83	0.002	5.34	0.006	6.43	n.d.	
Grand total	2.208	100	0.038	100	0.0088	98.9	0.004	100

n.d.: Not detected

n.a.: Not analysed

Table 9 Metabolites detected in oilseed rape matrices following foliar application of 0.089 kg ai/ha [¹⁴C]-imazamox

Components	Plant (0 DAT)		Foliage (22 DAT)		Straw (78 DAT)		Seed (78 DAT)	
	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)
Imazamox	3.068	77.8	0.020	15.5	0.003	1.7	0.001	12.6
CL 189215	0.045	1.2	0.002	1.7	0.002	1.1	0.001	20.5
CL 263284	0.104	2.6	0.047	36.0	0.083	46.4	0.000	4.6
CL 312622	0.061	1.6	0.004	3.2	0.001	0.8	0.000	2.1
Total identified from extracts	3.278	83.2	0.073	56.4	0.089	50.0	0.002	39.8
Total characterized from extracts	0.561	14.2	0.046	35.7	0.050	28.4	0.002	17.4
Total identified and/or characterized from extracts	3.839	97.4	0.119	92.1	0.139	78.4	0.004	57.2
2% HCl in aqueous MeOH (unidentified compounds)	0.049	1.24	0.006	4.51	0.007	4.13	0.000	5.17
Enzyme-released (cellulase)	n.a.	n.a.	n.a.	n.a.	0.004	2.01	n.a.	n.a.
6 N HCl Reflux	n.a.	n.a.	n.a.	n.a.	0.013	7.40	n.a.	n.a.
Unextracted (PES)	0.055	1.39	0.004	3.45	0.010	5.67	0.002	37.86
Grand total	3.943	100	0.130	100	0.173	97.6	0.006	100

n.a.: not analysed

Soya bean

A small-plot field study was conducted in an outdoor, fenced-off research area during the growing season of soya bean [Chiu 2000b, ID-640-010]. [pyridine-6-¹⁴C]-imazamox was applied as a single post-emergence on soya bean plants var., Hartz 5566 at the three-to-four trifoliate stage. The [pyridine-6-¹⁴C]-imazamox was diluted with non-radiolabelled imazamox and [pyridine-6-¹³C]-imazamox. The radiolabelled imazamox was formulated for application as the ammonium salt in aqueous solution and was sprayed at a rate of 0.39 kg ai/ha. Crop samples were collected at 0, 29, 60, and 153 days after treatment. Plant samples were stored frozen from sampling until analysis for a maximum period of 448 days.

Soya bean matrices were ground with dry ice. The TRR were determined by direct LSC or combustion for all needed samples (plant tissues, non-extractable residues, extracts, fractions from HPLC). The validated detection limit of the radioassay was 0.002 mg eq/kg.

Finely ground soya bean samples were extracted with methanol/water (80:20, v/v) and then partitioned three times with dichloromethane. The unextractable solids were blended with methanol, and the methanol rinse was analysed separately. To determine the potential for residues to accumulate in soya bean oil, the 153 DAT soya bean seed sample was blended with hexane and fractionated. Both fractions, hexane and residues, were assayed by oxidative combustion and LSC analysis.

The PES from soya bean straw and seed (after solvent extraction) were subjected to acid hydrolysis. The PES was stirred in the presence of 2% HCl in methanol/water (1:1) solution. The mixture was filtered. The filtrate was extracted with ethyl acetate if needed (> 0.01 mg eq/kg). The PES was subjected to reflux in the presence of 6 N HCL and subsequently of 1 N NaOH. If needed, the basic filtrate was extracted with ethyl acetate.

The amount of ^{14}C radioactivity present in the extracts and in the post-extraction solids was determined either by direct counting in a liquid scintillation counter or by oxidative combustions of aliquots. Distribution of radioactivity in the extractable fractions of soya bean samples was analysed by HPLC-UV at 254 nm and TLC.

Table 10 Distribution of residues of [^{14}C]-imazamox in soya bean samples (ID-640-010)

			Extracts						PES	
Matrix	DAT	TRR calc.	dichloromethane / methanol extract ^a		aqueous extract ^b		total			
		me eq/kg	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Forage	0	54.90	46.78	85.21	6.62	12.05	53.40	97.26	1.50	2.74
Forage	29	0.56	0.03	5.20	0.47	83.34	0.50	88.54	0.06	11.46
Forage	60	0.13	< 0.01	4.67	0.11	81.25	0.11	85.92	0.02	14.08
Straw	153	0.08	< 0.01	5.22	0.03	35.77	0.04	40.99	0.04	59.01
Seed	153	0.02	< 0.01	5.91	< 0.01	29.39	< 0.01	35.30	0.02	64.70

DAT: Days after treatment

TRR calc: sum of extracted + PES, equal to TRR determined by direct combustion.

^a Residues from three times extraction with dichloromethane, after extraction with aqueous methanol solution (methanol/water, 80:20, v/v). Residue in the methanol rinse (washing of the remaining solids after extracting with the aqueous methanol) was added.

^b Residues in the aqueous layer from extraction of three times with dichloromethane

The largest amount of [pyridine-6- ^{14}C]-imazamox-derived radioactivity appeared in the forage (54.9 mg eq/kg at 0 DAT, 0.56 mg eq/kg at 29 DAT and 0.13 mg/kg at 60 DAT), whereas smaller amounts of residues were noted in the harvest straw and seed, 0.08 mg eq/kg and 0.02 mg eq/kg, respectively.

The TRR in soya bean seed oil extracted with hexane was detectable at 0.002 mg eq/kg. No additional analysis was carried out due to the extremely low radiocarbon content.

Although the radioactive residue in the 0, 29 and 60 DAT forage samples was readily extractable, the extractability of the 153 DAT samples had decreased significantly. By the 153 DAT sampling, the extractability had decreased to 40.99% for straw and 35.30% for seed (8% extractability for seed when hexane was used, < 0.01 mg eq/kg).

The majority of radioactive residues in 0 DAT treated soya bean was extracted into dichloromethane (85.21% of the TRR). In the other samples, smaller amounts of residues were extracted into dichloromethane (excluding residues from methanol rinse): 4.09% TRR in 29 DAT forage, 3.06% TRR in 60 DAT forage, 1.56% TRR in straw and 4.28% TRR in seed.

The unextracted radioactivity in the PES from soya bean straw and seed were 59.01% (0.04 mg eq/kg) and 64.70% of TRR (0.02 mg eq/kg), respectively. By acid and base digestion, 52.96% TRR (0.03 mg eq/kg) and 56.79% TRR (0.01 mg eq/kg) were released from the straw and seed marc, respectively.

Table 11 Metabolites detected in soya bean matrices following foliar application of [^{14}C]-imazamox (ID-640-010)

Components	Forage (0 DAT)		Forage (29 DAT)		Forage (60 DAT)		Straw (153 DAT)		Seeds (153 DAT)	
	(mg eq/kg)	%TRR	(mg eq/kg)	%TRR	(mg eq/kg)	%TRR	(mg eq/kg)	%TRR	(mg)	%TRR
Imazamox	41.81	76.16	0.06	10.69	0.01	5.89	< 0.01	0.44	n/a	n/a
CL 263284	0.28	0.51	0.05	10.07	0.01	7.99	0.01	12.24	n/a	n/a
CL 312622	0.18	0.33	0.04	8.35	0.01	11.07	< 0.01	3.81	n/a	n/a
CL 189215	0.05	0.09	0.20	35.84	0.04	31.87	< 0.01	3.33	n/a	n/a
Total identified from extracts	42.32	77.09	0.35	64.95	0.07	56.82	0.02	19.82	n/a	n/a
Non-polar unknowns	4.42	8.06	0.02	4.71	0.01	6.63	< 0.01	0.43	n/a	n/a
Polar unknowns	0.04	0.07	0.10	17.79	0.03	17.81	< 0.01	5.10	n/a	n/a

Components	Forage (0 DAT)		Forage (29 DAT)		Forage (60 DAT)		Straw (153 DAT)		Seeds (153 DAT)	
	(mg eq/kg)	%TRR	(mg eq/kg)	%TRR	(mg eq/kg)	%TRR	(mg eq/kg)	%TRR	(mg)	%TRR
Total characterized from ERR	4.46	8.13	0.12	22.50	0.04	24.44	< 0.01	5.53	n/a	n/a
Total identified and/or characterized from extracts	46.78	85.22	0.47	87.45	0.11	81.26	0.02	25.35	n/a	n/a
Solvent extract (unidentified compounds)	6.62	12.05	0.01	1.11	< 0.01	4.67	0.05	68.6	n/a	n/a
Unextracted (PES)	1.50	2.74	0.06	11.46	0.02	14.08	0.01	6.05	< 0.01	7.91
Grand total	54.9	100	0.54	100	0.13	100	0.08	100	0.02	100

n/a: Not applicable because some fractions were not analysed due to low residue level (< 0.01 mg/kg)

The parent imazamox declined from 41.81 mg/kg at 0 DAT to 0.06 mg/kg (10.69% TRR) at 29 DAT in forage. The glucoside metabolite CL 189215 increased from 0.05 mg eq/kg (0.09% TRR) at 0 DAT to 0.20 mg eq/kg (35.84% TRR) at 29 DAT. However, CL 263284 and CL 312622, showed a decrease in concentration from 0 DAT to 29 DAT in forage, that is, 0.28 mg eq/kg (0.51%) to 0.05 mg eq/kg (10.07% TRR) for CL 263284 and 0.18 mg eq/kg (0.33% TRR) to 0.04 mg eq/kg (8.35% TRR) for CL 312622, respectively.

In forage at 60 DAT, residue levels of the three components (imazamox, CL 263284 and CL 312622) were very low at 0.01 mg eq/kg (5.89–11.07% TRR) and CL 189215 was found only at 0.04 mg eq/kg, although it accounted for 31.87% TRR. Three non-polar and three polar unknowns were detected but none exceeded 0.01 mg eq/kg or 10% of TRR individually.

In straw and seed at 153 DAT, no or little residues of parent imazamox and the metabolites were found.

Pea

Field peas (variety: Express) were treated post-emergence at the three to four node stage (thirty days after planting) with [pyridine-6-¹⁴C]-imazamox formulated as the ammonium salt at 0.040 kg ai/ha [Chiu 1995c, ID-640-004]. The in-life phase of this study was conducted outdoors in a sandy loam soil. The plots were equipped with removable plastic-covered “All” farms to exclude excessive rainfall, when required.

The whole plants were taken at 0 and 20 DAT. At 61 DAT, foliage/vine, pea, pea shell and pea pod at immature state were sampled. Mature pea and hay were harvested at 84 DAT. All samples were stored in a freezer at approximately –20 °C until used. The crop samples were analysed within 12 to 188 days of harvest. TRR for all samples were analysed by combustion and LSC.

A subsample of the 0 DAT treated plant tissue was extracted twice with methanol/water (8:2, v/v). The PES was air-dried and combusted prior to radioanalysis. Similarly a subsample of the 0 DAT control plant tissue treated with the blank formulation was spiked with [¹⁴C]imazamox and extracted as above.

A subsample of 20 DAT immature pea plant tissue was extracted twice with methanol/water (8:2, v/v). The PES was subjected to microwave extractions with 20% aqueous methanol solution and then 2% HCl in 20% aqueous methanol. Subsequently, reflux with 0.2 N NaOH in 20% aqueous methanol was made.

A subsample of 61 DAT immature pea sample was extracted twice with methanol/water (8:2, v/v). The combined filtrate was concentrated and stored in the refrigerator overnight. A precipitate which formed was removed. The filtrate was further concentrated, followed by precipitation with an equal volume of acetonitrile/methanol (1:1).

A subsample of harvest pea sample (84 DAT) was extracted twice with methanol/water (8:2, v/v). The combined filtrate was concentrated resulting in a thick oily residue. This residue was

washed with acetonitrile/methanol (1:1), resulting in some precipitation. After the acetonitrile/methanol-soluble phase was decanted, the precipitate was reconstituted in water. The water-soluble fraction was concentrated, adjusted to pH 3, and then introduced onto a solid phase extraction (SPE) column.

A subsample of the harvest pea hay sample (84 DAT) was extracted twice with methanol/water (8:2, v/v). The PES was subjected to microwave extractions with 2% HCl in 20% aqueous methanol, and then 0.2 N NaOH in 20% aqueous methanol.

The extracts were assayed by combustion and LSC. HPLC and TLC analysis was made for characterization and identification.

The TRR in whole plant sampled at 0 DAT was determined to be 1.1 mg eq/kg. The TRR in immature pea plants at 20 DAT had decreased to 0.035 mg eq/kg, most probably associated with growth dilution. No detectable residues above the validated limit of the radioanalysis method (0.01 mg eq/kg) were seen in any plant tissues (foliage, pea, shell, and immature pea pod) sampled at 61 DAT. At harvest (84 DAT), the dry pea and pea hay contained small but detectable TRR, 0.01 mg eq/kg and 0.05 mg eq/kg, respectively.

Table 12 TRR in various pea matrices after application of [¹⁴C]imazamox

Matrix	Days after treatment	TRR determined by direct combustion (mg eq/kg)
Whole plant	0	1.12
Whole plant	20	0.035
Foliage/vine	61	< 0.01
Immature pea	61	< 0.01
Immature pea shell	61	< 0.01
Immature pea pod	61	< 0.01
Harvest pea	84	0.01
Harvest pea hay	84	0.05

The radioactive residues from 0 DAT whole pea plant samples with the aqueous methanol solution extracted up to 99% (1.1 mg eq/kg) of the TRR. In whole pea plant fortified with a predetermined amount of [¹⁴C]imazamox, nearly 100% of the TRR was recovered.

From immature green peas (61 DAT) and harvested pea samples (84 DAT), approximately 61% of the TRR (< 0.01 mg eq/kg) and 39.2% of the TRR (0.004 mg eq/kg) were extracted, respectively. No further extractions of the PES were pursued due to the low radioactive residue.

From 20 DAT pea plants and 84 DAT hay samples, 65.4% of the TRR (0.023 mg eq/kg) and 58.4% (0.029 mg eq/kg) of the TRR were extracted respectively. More extractions under acidic and basic condition released nearly all of the remaining residues in the post extraction solids.

Table 13 Distribution of residues of [¹⁴C]imazamox in pea samples

Matrix	DAT	TRR calc.	Extracts, organo-extractable		PES	
		(mg eq/kg)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)
Whole plant	20	0.035	0.023	65.4	0.012	34.6
Hay	84	0.05	0.029	58.4	0.021	41.6
Peas	84	0.01	0.004	39.2	0.006	60.8

TRR calc: sum of extracted + PES

Organo-extractable means the residues extracted with methanol/water (8:2, v/v).

In all pea samples, the parent imazamox, CL 263284, and CL 189215 were found at or below the limit of detection (0.01 mg eq/kg), except hay (0.02 mg eq/kg, 66.3% TRR).

Table 14 Metabolites detected in pea matrices following foliar application of [^{14}C]imazamox

Components	Plant (20 DAT)		Pea (61 DAT)		Pea (84 DAT)		Hay (84 DAT)	
	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)
Imazamox	< 0.01	10.18	< 0.01	30.05	< 0.01	7.82	0.02	66.31
CL 189215	< 0.01	10.98	< 0.01	6.28	< 0.01	8.34	< 0.01	3.02
CL 263284	0.01	43.91	< 0.01	39.91	< 0.01	5.85	< 0.01	13.24
Polar unknown	< 0.01	14.77	< 0.01	5.16	< 0.01	8.57	< 0.01	
Total identified and characterized	0.01	79.84	< 0.01	81.4	< 0.01	30.58	< 0.01	89.57

Alfalfa

The [pyridine-6- ^{14}C]-imazamox was applied once post-emergence to alfalfa (variety, WL 414) [Wu 1998b, ID-640-008]. The test substance (formulated as an ammonium salt) was applied once at a rate of 0.13 kg ai/ha onto four plots: plot B (established crop, breaking dormancy, at the 3rd trifoliate stage), plot D (newly seeded crop, at the 3rd trifoliate stage), plot F (newly seeded crop, late treatment at 5 days after the 1st cut) and plot H (newly seeded crop, late treatment at early bud stage).

The alfalfa forage and hay samples were taken from all four plots at the day of the application (0 DAT), 26 DAT (plots B, D and F), 45 or 74 DAT (1st cut; plots B and D, respectively); 87, 111 or 25 DAT (2nd cut, plots B, D and F, respectively) and 87, 157 or 53 DAT (3rd cut, plots B, D and F, respectively). Mature alfalfa seeds were sampled 74 DAT from plot H.

Forage and hay samples were taken from the three plots (B, D and F). In plot H, forage and seed were sampled at 0 DAT and 74 DAT, respectively. In the all plots, forage was sampled at 0–157 DAT and hay was takes 2–3 days later. All samples were stored frozen at approximately $-20\text{ }^{\circ}\text{C}$.

The TRR in green plants (forage), hay and seeds was determined by combustion and LSC except forage sample collected at 0 DAT. The 0 DAT forage was determined by solvent extraction followed by combustion due to very small size sample and the expected high radioactivity.

Ground alfalfa samples were extracted with methanol/acetone/water (1:1:1, v/v/v). The seed extract was additionally subjected to hexane partition. The PES were re-extracted three times with the acidic extraction solvent (methanol/water/HCl, 40:9:1, v/v/v), except several alfalfa samples due to high extractability and a very low residue level (0.003 mg eq/kg). The residual solids derived from the acidic methanol/water extraction (forage, hay or seed) were treated with cellulase and by 6 N HCl reflux.

The methanol/acetone/water extract of the alfalfa hay was treated with β -glucosidase or with 1 N NaOH for base hydrolysis.

The extracts were assayed by direct LSC or by combustion and characterized by HPLC. Structural characterization of the parent compound and the major metabolites was performed by LC-MS.

The TRR in forage at 0 DAT were 15.28 mg eq/kg (plot B), 10.28 mg eq/kg (plot F), 14.00 mg eq/kg (plot D) and 6.51 mg eq/kg (plot H). At 26 DAT, the radioactive residues declined significantly to 0.34 mg eq/kg (plot B) and 0.67 mg eq/kg (plot D), respectively. The TRR in forage of 1st, 2nd and 3rd cut from the all plots were found at relatively low levels, < 0.01 mg eq/kg to 0.21 mg eq/kg. The TRR levels in hay were three to five times higher than those of forage.

In forage and hay samples, the extracted residues from solvent extraction ranged from 74.9 to 98.8% of the TRR. The most (65.3–98.8%) was derived from methanol/acetone/water solvent extraction. The acidified aqueous methanol (2% HCl in 80% methanol) extracted an additional 1.2 to 14.2% of the TRR (0.001–0.090 mg eq/kg) in the forage and hay. As the result, the residual radioactive residues in PES were 0.001 to 0.252 mg eq/kg. In alfalfa seed, 72.7% TRR was extracted from the organic solvent extractions (15% TRR from acidified aqueous methanol extraction).

The radioactive residues in PES were released by enzyme treatment and acid hydrolyses. The results suggested the presence of glycosidic linkages of the residue with endocons in the alfalfa samples. After cellulase hydrolysis, the PES (greater than 0.05 mg eq/kg) were subjected to acid hydrolysis (6 N HCl reflux). The acid hydrolysis step released about 60% of the radioactive residues (equivalent to 8–9% of TRR or 0.033–0.056 mg eq/kg) while about 40% (equivalent to 6% of TRR or 0.022–0.039 mg eq/kg) remained bound. The results indicated that the labelled carbon has been incorporated into undefined plant constituents.

Table 15 Distribution of TRR following solvent partitioning and extraction of residues of [^{14}C]imazamox in alfalfa matrices

				Extracts						PES	
Plot	Matrix	DAT	TRR (combusted)	methanol/ acetone/ water		acidic methanol/ water		total			
			mg eq/kg	% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg
Plot B	Forage	0	15.28	98.8	15.10	n.e.	n.e.	98.8	15.10	1.2	0.183
	Forage	26	0.34	89.3	0.304	1.2	0.004	90.5	0.308	9.5	0.032
	Forage	45 (1 st cut)	0.21	94.9	0.199	n.e.	n.e.	94.9	0.199	5.1	0.011
	Hay	48 (1 st cut)	0.63	91.5	0.576	3.9	0.025	95.4	0.601	4.6	0.029
	Forage	87 (2 nd cut)	0.03	91.4	0.027	n.e.	n.e.	91.4	0.027	8.6	0.003
	Hay	89 (2 nd cut)	0.12	88.6	0.106	5.0	0.006	93.6	0.112	6.4	0.008
	Forage	129 (3 rd cut)	0.02	96.4	0.019	n.e.	n.e.	96.4	0.019	3.6	0.001
	Hay	131 (3 rd cut)	0.06	86.7	0.052	7.3	0.004	94.0	0.056	6.0	0.004
Plot D	Forage	0	14.00	98.2	13.75	n.e.	n.e.	98.2	13.75	1.8	0.252
	Forage	26	0.67	77.3	0.518	6.4	0.043	83.4	0.559	16.6	0.111
	Forage	76 (1 st cut)	0.07	74.1	0.052	9.7	0.007	83.8	0.059	16.2	0.011
	Hay	78 (1 st cut)	0.35	64.7	0.226	13.8	0.048	78.5	0.274	21.5	0.075
	Forage	111 (2 nd cut)	0.01	74.9	0.007	n.e.	n.e.	74.9	0.007	25.1	0.003
	Hay	113 (2 nd cut)	0.04	70.5	0.028	10.7	0.004	81.2	0.032	18.8	0.008
	Forage	157 (3 rd cut)	< 0.01	n.e.	n.e.	n.e.	n.e.	n.e.	n.e.	n.e.	n.e.
	Hay	160 (3 rd cut)	0.02	68.5	0.014	11.1	0.002	79.6	0.016	20.4	0.004
Plot F	Forage	0	10.28	98.8	10.16	n.e.	n.e.	98.8	10.16	1.2	0.123
	Forage	26 (2 nd cut)	0.18	85.9	0.155	5.0	0.009	90.9	0.164	9.1	0.016
	Hay	28 (2 nd cut)	0.83	80.7	0.670	10.8	0.090	91.5	0.760	8.5	0.071
	Forage	53 (3 rd cut)	0.02	80.2	0.016	5.9	0.001	86.1	0.017	13.9	0.003
	Hay	55 (3 rd cut)	0.08	65.3	0.052	14.2	0.011	78.8	0.063	21.2	0.017
Plot H	Forage	0	6.51	98.2	6.393	n.e.	n.e.	98.2	6.393	1.8	0.117
	Seed	74	0.02	57.7	0.012	15.0	0.003	72.7	0.015	30.0	0.006

Plot B: Breaking dormancy, at 3rd trifoliate stage

Plot D: Newly seeded crop, at 3rd trifoliate stage

Plot F: Newly seeded crop, late treatment at 5 days after 1st cut

Plot H: Newly seeded crop, late treatment at early bud stage

PES: Post extraction solid resulted from methanol/acetone/water extraction followed by acidic methanol/water extraction

n.e.: not extracted

At 0 DAT, the unchanged imazamox parent accounted for the majority of the TRR (79.7–86.6%, 5.64–12.98 mg/kg) in forage from all plots. All other components at 0 DAT, including CL 189215, CL 263284 and CL 312622 were found to be minor (each at < 2% of the total residue). Imazamox was extensively metabolized in alfalfa and accounted for only 3–6% TRR (0.019–0.023 mg/kg) in forage of plot B and D (only data available) at 26 DAT. The metabolites included

CL 189215 (14–20% TRR, 0.048–0.132 mg eq/kg), CL 263284 (14–23% TRR, 0.079–0.095 mg eq/kg) and CL 312622 (18–22% TRR, 0.075–0.119 mg eq/kg) at 26 DAT.

In the 1st and 2nd cut hay (plot B, D and F), imazamox was found at very low level of 2–6% TRR (0.001–0.036 mg/kg). Relatively, the residue levels of the three main metabolites were high: CL 189215 12–31% TRR (0.005–0.208 mg eq/kg), CL 263284 12–15% TRR (0.006–0.105 mg eq/kg) and CL 312622 4–26% TRR (0.002–0.186 mg eq/kg). In seed collected at 74 DAT, residue levels of parent and the three main metabolites were very low at < 0.001 mg eq/kg or 0.001 mg eq/kg.

Table 16 Metabolites detected in alfalfa matrices following foliar application of [¹⁴C]imazamox

Plot	Matrix	DAT	TRR (mg/kg)	Components							
				CL 189215		CL 263284		CL 312622		Imazamox	
				% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Plot B	Forage	0	15.28	1.24	0.189	0.94	0.144	1.60	0.244	84.96	12.98
	Forage	26	0.34	13.98	0.048	23.15	0.079	22.11	0.075	5.52	0.019
	Forage	45 (1 st cut)	0.21	25.66	0.054	18.26	0.038	26.67	0.056	2.65	0.006
	Hay	48 (1 st cut)	0.63	23.85	0.150	14.48	0.091	26.05	0.164	5.52	0.034
	Forage	87 (2 nd cut)	0.03	25.84	0.008	21.08	0.006	13.79	0.004	4.51	0.001
	Hay	89 (2 nd cut)	0.12	30.81	0.037	11.28	0.014	21.67	0.026	1.53	0.002
	Forage	129 (3 rd cut)	0.02	20.15	0.004	24.67	0.005	13.14	0.003	6.40	0.001
	Hay	131 (3 rd cut)	0.06	21.90	0.013	25.06	0.015	20.64	0.012	2.63	0.002
	Forage	0	14.00	0.93	0.130	1.23	0.172	1.56	0.218	82.92	11.61
Plot D	Forage	26	0.67	19.80	0.132	14.11	0.095	17.80	0.119	3.46	0.023
	Forage	76 (1 st cut)	0.07	16.96	0.012	9.49	0.007	13.91	0.010	4.36	0.003
	Hay	78 (1 st cut)	0.35	19.62	0.069	14.73	0.051	20.13	0.070	3.60	0.013
	Forage	111 (2 nd cut)	0.01	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	Hay	113 (2 nd cut)	0.04	11.83	0.005	14.90	0.006	3.96	0.002	2.73	0.001
	Forage	157 (3 rd cut)	< 0.01	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	Hay	160 (3 rd cut)	0.02	16.80	0.003	15.89	0.003	0.71	< 0.001	2.43	< 0.001
	Forage	0	10.28	1.50	0.154	1.93	0.198	1.97	0.203	79.66	8.189
	Forage	26 (2 nd cut)	0.18	25.84	0.047	14.24	0.026	17.98	0.032	4.20	0.008
Plot F	Hay	28 (2 nd cut)	0.83	25.02	0.208	12.72	0.105	22.48	0.186	4.36	0.036
	Forage	53 (3 rd cut)	0.02	15.69	0.003	16.53	0.003	7.03	0.001	1.57	< 0.001
	Hay	57 (3 rd cut)	0.08	14.07	0.011	18.53	0.015	7.81	0.007	3.62	0.003
Plot H	Forage	0	6.51	0.70	0.046	1.02	0.066	0.67	0.044	86.63	5.640
H	Seed	74	0.02	3.00	0.001	7.21	0.001	2.05	< 0.001	2.32	< 0.001

Plot B: Breaking dormancy, at 3rd trifoliate stage

Plot D: Newly seeded crop, at 3rd trifoliate stage

Plot F: Newly seeded crop, late treatment at 5 days after 1st cut

Plot H: Newly seeded crop, late treatment at early bud stage

Table 17 Balance of identified, characterized and unextractable radioactive residues in alfalfa matrices following foliar application of [¹⁴C]-imazamox

Plot	Matrix	DAT	Total Identified		Total Characterized		Total Identified and/or Characterized		Unextracted (PES)		Grand Total	
			% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Plot B	Forage	0	88.74	13.56	12.95	1.981	101.7	15.54	1.2	0.183	102.9	15.72
B	Forage	26	64.76	0.221	28.74	0.097	93.5	0.318	6.5	0.022	100.0	0.340

Plot	Matrix	DAT	Total Identified		Total Characterized		Total Identified and/or Characterized		Unextracted (PES)		Grand Total	
			% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
	Forage	45 (1 st cut)	73.24	0.154	21.66	0.046	94.9	0.200	5.1	0.011	100.0	0.211
	Hay	48 (1 st cut)	69.90	0.439	25.50	0.160	95.4	0.599	4.6	0.029	100.0	0.628
	Forage	87 (2 nd cut)	65.22	0.019	26.18	0.008	91.4	0.027	8.6	0.003	100.0	0.030
	Hay	89 (2 nd cut)	65.29	0.079	28.31	0.034	93.6	0.113	6.4	0.008	100.0	0.121
	Forage	129 (3 rd cut)	64.36	0.013	32.04	0.007	96.4	0.020	3.6	0.001	100.0	0.021
	Hay	131-3 rd cut	70.23	0.042	23.76	0.014	94.0	0.056	6.0	0.004	100.0	0.060
Plot	Forage	0	86.64	12.13	11.55	1.617	98.2	13.75	1.8	0.252	100.0	13.40
D	Forage	26	55.17	0.369	39.02	0.262	94.2	0.631	5.8	0.039	100.0	0.670
	Forage	76 (1 st cut)	44.71	0.032	43.88	0.030	88.6	0.062	11.4	0.008	100.0	0.070
	Hay	78 (1 st cut)	58.08	0.203	35.53	0.125	94.3	0.328	6.4	0.022	100.7	0.350
	Forage	111 (2 nd cut)	n.a.	n.a.	74.9	0.007	74.9	0.007	25.1	0.003	100.0	0.010
	Hay	113 (2 nd cut)	33.42	0.014	50.19	0.020	83.6	0.034	16.4	0.007	100.0	0.041
	Forage	157 (3 rd cut)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	Hay	160-3 rd cut	35.83	0.008	44.78	0.009	80.6	0.017	19.4	0.004	100.0	0.021
Plot	Forage	0	85.66	8.744	13.75	1.414	99.4	10.16	1.2	0.123	100.6	10.28
F	Forage	26 (2 nd cut)	62.26	0.113	28.64	0.052	90.9	0.165	9.1	0.016	100.0	0.181
	Hay	28 (2 nd cut)	64.58	0.535	30.00	0.249	94.6	0.784	5.4	0.045	100.0	0.829
	Forage	53 (3 rd cut)	40.82	0.008	45.28	0.009	86.1	0.017	13.9	0.003	100.0	0.020
	Hay	57 (3 rd cut)	44.03	0.036	41.86	0.034	85.9	0.070	14.1	0.011	100.0	0.081
Plot	Forage	0	89.02	5.796	9.19	0.598	98.2	6.394	1.8	0.117	100.0	6.511
H	Seed	74	14.58	0.004	56.11	0.012	70.7	0.016	25.9	0.005	96.6	0.021

Plot B: Breaking dormancy, at 3rd trifoliate stage

Plot D: Newly seeded crop, at 3rd trifoliate stage

Plot F: Newly seeded crop, late treatment at 5 days after 1st cut

Plot H: Newly seeded crop, late treatment at early bud stage

The metabolite profile of the alfalfa hay based on HPLC analysis shows that the incurred [¹⁴C]imazamox-derived residues in the alfalfa hay are stable when stored frozen for about 13 months. The quantitative distribution of imazamox and metabolites CL 189215, CL 263284, CL 312622 and the very polar components peak in the sample analysed within one month after sampling is similar to those found in the sample analysed at about 13 months after sampling.

Maize

Study 1

A metabolism study of imazamox in maize (imidazolinone-tolerant variety, Pioneer hybrid 3571 IR) was conducted in an outdoor, fenced-off research area on sandy loam soil in Lucama, North Carolina, during the growing season 1996 [Zulalian 1997c, ID-640-006]. The radiolabelled imazamox was a mixture prepared from [pyridine-6-¹⁴C]-imazamox, [pyridine-6-¹³C]-imazamox and non-isotopically labelled imazamox. The radioactive test substance formulated as an aqueous ammonium salt solution was applied as a post-emergence broadcast application 0.13 kg ai/ha to maize plants at the 4–8 leaf growth stage. Samples of maize plants were harvested at 0 DAT (immature maize plant), 14 DAT (immature plant), 30 DAT (early forage), and 62 DAT (late forage). At maturity (100 DAT), collected field samples were separated with fodder (stalks, husks, cobs) and grain. The maize samples were ground with dry ice and stored frozen. The samples were subjected to oxidative combustion and LSC. All control samples were < 0.004 mg eq/kg. The crop samples were analysed within 44 days.

Green plant, forage and fodder samples were extracted three times with methanol/water (80:20, v/v). The PES was rinsed with methanol and the rinsate was combined with the extracts. A grain sample was extracted three times with hexane. The PES was extracted three times with methanol/water (80:20, v/v).

The methanol/water extracts were partitioned three times with dichloromethane. The dichloromethane and aqueous fractions were separated. For the 14 DAT, 30 DAT and 62 DAT forage and 100 DAT fodder samples, the PES (after partitioning with dichloromethane) were extracted once with methanol/water/hydrochloric acid (130:18:2, v/v/v).

TRR in extracts and residual radioactive residues were analysed by combustion and LSC. Distribution of radioactivity in the extracts was analysed by HPLC-UV at 254 nm. Imazamox and the metabolite CL 263284 were isolated and identified by mass spectral analysis. The other components of the residue were identified by comparison of HPLC retention time.

The TRR in immature maize plant at 0 DAT was 8.2 mg eq/kg and rapidly decreased to 0.41 mg eq/kg (14 DAT). At 30 DAT and 62 DAT, the TRR in early and late forage were 0.087 mg eq/kg and 0.078 mg eq/kg, respectively. In maize fodder and grain at maturity (100 DAT), the TRR were 0.047 mg eq/kg and 0.021 mg eq/kg, respectively.

Of the TRR in the various maize substrates, between 48% and 98% (0.014–8.0 mg eq/kg) was extracted with methanol/water (80:20, v/v) and between 2% and 52% (0.006–0.17 mg eq/kg) remained unextracted. Negligible radioactivity (0.4–0.6% of the TRR, < 0.001 mg eq/kg) was extracted from the grain with hexane indicating the imazamox-derived residues did not concentrate in the maize oil.

The additional extraction with acidic extraction solvent (methanol/water/HCl, 80:18:2) released more residues in maize samples up to approximately 56% to 98% of the TRR (0.013–8.0 mg eq/kg). Approximately 2% to 44% (0.006–0.021 mg eq/kg [¹⁴C]imazamox equivalents) remained in the post extraction solids.

Table 18 Distribution of TRR following solvent partitioning and extraction of residues of [¹⁴C]imazamox in maize samples

Matrix	DAT	TRR calc. (mg eq/kg)	Extracts								PES	
			dichloromethane extract (mg eq/kg)	(%TRR)	aqueous extract (mg eq/kg)	(%TRR)	methanol/water/ HCl extract (mg eq/kg)	(%TRR)	total (mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)
Immature plant	0	8.199	3.126	38.1	4.902	59.8			8.028	97.9	0.171	2.1
Immature plant	14	0.409	0.081	19.8	0.281	68.8	0.012	2.9	0.374	91.5	0.035	8.5
Early forage	30	0.087	0.012	14.3	0.061	69.9	0.006	7.0	0.079	91.2	0.008	8.8
Late forage	62	0.078	0.003	4.3	0.055	70.7	0.004	5.6	0.062	80.6	0.015	19.5 ³
Fodder	100	0.047	0.001	2.4	0.021	45.6	0.004	8.2	0.026	56.2	0.021	43.8
Grain	100	0.021	0.004	18.8	0.011	53.1			0.015	71.9 ^a	0.006	27.6
Grain	100	0.021	0.001	4.9	0.012	59.2			0.013	64.1 ^b	0.007	35.3

After extraction with methanol/water (80:20, v/v), followed by partitioning with dichloromethane

TRR calc.: sum of extracted + PES, equal to TRR determined by direct combustion.

Additional radioactivity was released from the PES at 62 DAT as follows. The PES was extracted at elevated temperature and pressure in an ASE 200 Accelerated Solvent Extractor to recover 0.016 mg/kg [¹⁴C]imazamox equivalents (20.5% of the TRR) and only 0.003 mg/kg [¹⁴C]imazamox equivalents (3.8% of the TRR) remained in the PES following this more vigorous procedure. This indicated there were no significant bound residues in the maize forage.

^a Replicate 1, 0.4% (< 0.004 mg/kg) was hexane-soluble.

^b Replicate 2, 0.6% (< 0.004 mg/kg) was hexane-soluble.

The unchanged parent imazamox amounted to 5.3 mg/kg (65% TRR) at 0 DAT plant, 0.073 mg/kg (18% TRR) at 14 DAT plant and less than 0.01 mg/kg (2–9% of the TRR) thereafter.

CL 263284 was identified as the major metabolite in maize, accounting for 0.24 mg eq/kg (3% TRR) at 0 DAT plant, but only 0.007 mg eq/kg (15% TRR) in maize fodder and only 0.004 mg eq/kg (20% TRR) in grain. The residue levels of glucose conjugate (CL 189215), the di-acid (CL 312622) and the hydroxy-acid (CL 354825) were < 0.001–0.003 mg eq/kg (2–5% TRR) in fodder and grain. Many polar and nonpolar compounds (minimum 4 polar and 3 nonpolar) were found at 0.012 mg eq/kg or less (1–14% TRR) individually since 30 DAT.

Table 19 Metabolites detected in maize matrices following post-emergence foliar application of [^{14}C]-imazamox

Components	Plant (0 DAT)		Plant (14 DAT)		Forage (30 DAT)		Forage (62 DAT)		Fodder (100 DAT)		Grain (100 DAT)	
	(mg eq/kg)	(%TR R)	(mg eq/kg)	(%TR R)	(mg eq/kg)	(%TR R)	(mg eq/kg)	(%TR R)	(mg eq/kg)	(%TR R)	(mg eq/kg)	(%TR R)
Imazamox	5.305	64.7	0.073	17.8	0.008	9.2	0.003	3.8	0.001	2.2	0.001	5.0
CL 263284	0.237	2.9	0.093	22.7	0.025	28.7	0.032	41.0	0.007	15.2	0.004	20.0
CL 312622	0.562	6.9	0.029	7.1	0.007	8.0	0.001	1.3	0.001	2.2	0.001	5.0
CL 189215	0.198	2.4	0.024	5.9	0.004	4.6	< 0.001	< 1	0.003	6.5	0.003	15.0
CL 354825	0.486	5.9	0.009	2.2	0.003	3.4	0.002	2.6	< 0.001	< 1	< 0.001	< 1
Total identified	6.788	82.8	0.228	55.7	0.047	53.9	0.038	48.7	0.013	26.1	0.009	45.0
Min. of 4 polar and 3 nonpolar compounds	1.242 (0.055–0.281)	15.2 (0.7–4.4)	0.135 (0.003–0.059)	33.0 (0.7–14.4)	0.024 (0.001–0.012)	27.3 (1.1–13.8)	0.017 (< 0.001–0.009)	21.8 (< 1–11.5)	0.009 (< 0.001–0.004)	19.6 (< 1–8.7)	0.002 (< 0.001–0.001)	10.0 (< 1–5.0)
Total identified and/or characterized	8.030	97.9	0.359	88.8	0.071	81.6	0.055	70.5	0.021	45.7	0.011	55.0

Study 2

The metabolism study of imazamox in maize (Pioneer hybrid 3571 IR, Imidazolinone-tolerant variety) was conducted in an outdoor, fenced-off research area on sandy loam soil in Lucama, North Carolina, during the 1996 growing season [Zulalian 1997d, ID-640-007]. The radio impurity amounting to approximately 3% was identified as the di-acid (CL 312622). The radiolabelled imazamox was a mixture prepared from [pyridine-6- ^{14}C]-imazamox, [pyridine-6- ^{13}C]-imazamox and non-isotopically labelled imazamox. The radioactive test substance was formulated as an aqueous ammonium salt solution and applied as a pre-emergence broadcast treatment (one day after seeding) at a rate of 0.14 kg ai/ha. Samples of maize plants were taken at 14 DAT (immature maize plant), 30 DAT (early maize forage) and 62 DAT (late maize forage). At maturity (112 DAT), the fodder (stalks/husks/cobs) and grain were collected. The samples were ground with dry ice and stored frozen. TRR was analysed by oxidative combustion and LSC. The detection limit was < 0.004 mg eq/kg. The crop samples were analysed within 24 days.

The extraction method with methanol/water (80:20, v/v) and dichloromethane was the same with that of above study. The PES of immature maize plant (14 DAT) was extracted once with methanol/water/hydrochloric acid (80:18:2, v/v/v). A grain sample was extracted three times with hexane. The PES was extracted three times with methanol/water (80:20, v/v). The extracts and post extraction solids were analysed by combustion and LSC. Distribution of radioactivity in the extractable fractions of maize samples was analysed by HPLC-UV detector set at 254 nm. Imazamox and the metabolite CL 263284 were isolated and identified by mass spectral analysis. The other components of the residue were identified by retention times by HPLC.

The TRR in immature maize at 14 DAT was 0.16 mg eq/kg and decreased to 0.027 mg eq/kg in early forage (30 DAT) and further to 0.011 mg eq/kg in late forage (62 DAT). In harvest fodder and grain (112 DAT), the TRR were 0.012 mg eq/kg and 0.010 mg eq/kg, respectively.

In the various maize substrates, 40% to 89% (0.004–0.13 mg eq/kg) of the TRR was extracted with methanol/water, and 17% to 60% (0.005–0.028 mg eq/kg) remained in the post extraction solid

(for 14 DAT maize, including 7% TRR released from acidic solvent extraction). Negligible radioactivity (2.1% of the TRR, < 0.001 mg eq/kg) was extracted from the grain with hexane indicating the imazamox-derived residues did not concentrate in the maize oil.

Since the TRR in the extracts of the 62 DAT and 112 DAT maize samples were insufficient for identification/characterization, the extracts were not carried through the solvent partitioning procedures.

Table 20 Distribution of TRR following solvent partitioning and extraction of residues of [¹⁴C]imazamox in maize samples

Matrix	DAT	TRR calc.	Extracts								PES	
			dichloromethane extract		aqueous extract		methanol/water/HCl extract		total			
		(mg eq/kg)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)
Green plant	14	0.158	0.044	28.0	0.075	47.2	0.011	6.9	0.13	82.1	0.028	17.9
Early forage	30	0.027	0.007	25.5	0.015	57.1			0.022	82.6	0.005	17.3
Late forage	62	0.011							0.009	88.8	0.002	21.2
Fodder	112	0.012							0.005	40.3	0.007	59.7
Grain	112	0.010							0.004	43.6	0.006	56.4

TRR calc.: sum of extracted + PES, equal to TRR determined by direct combustion.

Extracts of grain includes hexane extract, approximately 2% TRR (< 0.001 mg eq/kg).

The unchanged parent imazamox amounted to 0.049 mg/kg (31% TRR) at 14 DAT maize plant and 0.010 mg/kg (37% TRR) at 30 DAT forage. CL 263284 was identified as the major metabolite in maize, accounting for 12% TRR (0.019 mg eq/kg) at 14 DAT plant and 19% TRR (0.005 mg eq/kg) at 30 DAT forage. The identification of imazamox and CL 263284 was confirmed by LC-MS analysis. The residue levels of the metabolites (identified by HPLC retention time) glucose conjugate (CL 189215), the di-acid (CL 312622) and the hydroxy-acid (CL 354825) were between 0.003 (2% TRR) and 0.008 mg eq/kg (5% TRR) at 14 DAT and below < 0.001 mg eq/kg (less than 4% of the TRR) at 30 DAT. The remaining radioactive residue was characterized as multiple polar and nonpolar water-soluble metabolites (0.001–0.016 mg eq/kg, 0.6–10% TRR individually). The only significant components of the residue in maize were imazamox and CL 263284.

Table 21 Metabolites detected in maize immature plant and forage following pre-emergence foliar application of [¹⁴C]imazamox

Components	14 DAT plant		30 DAT forage	
	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)
Imazamox	0.049	31.0	0.010	37.0
CL 263284	0.019	12.0	0.005	18.5
CL 312622	0.008	5.1	0.001	3.7
CL 354825	0.005	3.2	0.001	3.7
CL 189215	0.003	1.9	0.001	3.7
Total identified	0.084	53.2	0.018	66.6
Min. of 4 polar and 3 nonpolar compounds	0.034 (0.001–0.016)	21.4 (0.6–10.1)	0.008 (< 0.001–0.002)	13.41 (< 0.1–7.41)
Total identified and/or characterized	0.118	74.6	0.026	80.01

Wheat

Study 1

Spring wheat (imidazolinone-tolerant variety: AP603 CL) was sowed into ten plastic containers filled with loamy sand soil, which was conducted in climatic chambers, the phytotrons simulated the natural climatic conditions for a typical spring, [Grosshans *et al.*, 2012b, 2012/1064722]. The crop was treated once with [imidazolinone-5-¹⁴C, 3-¹⁵N]-imazamox at a rate of 0.76 kg ai/ha at growth stage BBCH 13–24. The application formulation (including the test item: unlabelled imazamox (2:1 ratio), blank formulation and adjuvant Dash EC) was applied with an automatic spray track at a spray volume of 219 L/ha. Samples of wheat forage and hay were taken 8 days (BBCH 39) after application. Harvest of wheat plants and sampling of straw and grain was accomplished 62 days after application. Eighty wheat plants were thinned out. One half of the plants was minced and frozen as forage, the other half was minced and dried at room temperature. Wheat ears and straw (GS 89) were cut off and the straw was minced. Afterwards, ears were separated into chaff and grain using a thresher. The chaff was mixed to the straw. All samples were stored in a freezer at –18 °C or below. The samples were stored in a refrigerator or in a freezer. Radioassay was performed by combustion and LSC.

The samples were extracted with methanol and water. The residual radioactive residues were extracted twice with 1% ammonia, and then treated with the enzymes in order of macerozyme/cellulase, α -amylase/ β -amylase/amyloglucosidase, and tyrosinase/lactase. After the tyrosinase incubation the PES were extracted once with 1 N HCl, thereafter refluxed with 6 N HCl. The extracts and PES were assayed by LSC. The residues in the extracts were characterized or identified by HPLC. For enantiomer-specific analysis, an aliquot of wheat forage, hay or grain was extracted with methanol and water and then the methanol extract was extracted three times with ethyl acetate, subsequently the ethyl acetate phase partitioned with water (acidified with formic acid to pH 2). The water phase was fractionated using HPLC and the fractions were subjected to enantiomer-specific analysis using HPLC.

There were no major differences between measured TRR and calculated TRR (ERR plus PES). Here, the calculated TRR values were set to 100% TRR for all matrices. For the wheat forage, hay, straw and grain, the TRR were 1.6 mg eq/kg, 8.0 mg eq/kg, 3.2 mg eq/kg and 1.4 mg eq/kg, respectively.

Table 22 Distribution of residues of [¹⁴C]imazamox in wheat samples (2012/1064722)

Matrix	DAT	TRR	TRR cal.	Extracts						PES	
				combined methanol extract		combined aqueous extract		total			
		(mg eq/kg)	(mg eq/kg)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)
Forage	8	1.819	1.602	1.427	89.0	0.055	3.4	1.482	92.5	0.121	7.5
Hay	8	10.09	8.011	6.008	75.0	1.329	16.6	7.337	91.6	0.674	8.4
Straw	62	3.493	3.199	1.962	61.3	0.586	18.3	2.548	79.6	0.651	20.4
Grain	62	1.374	1.412	0.618	43.8	0.608	43.1	1.227	86.9	0.185	13.1

TRR cal.: sum of extracts + PES

The extractability of wheat forage and hay with methanol and water was very high and accounted for 92.5% and 91.6% of the TRR. The major part of the residues was extracted with methanol (forage: 89.0% TRR, hay: 75.0% TRR). The extractability of wheat straw and grain with methanol and water was high and accounted for 79.6% and 86.9% of the TRR. For straw, the major part of the residues was extracted with methanol (61.3% TRR), whereas for grain virtually equal amounts were extracted with methanol and water (methanol 43.8% TRR; water 43.1% TRR).

In wheat forage, analysis of the methanol extract using HPLC resulted in a pattern of six peaks, of which four were identified. The main peak was identified as the parent compound imazamox and accounted for 1.046 mg/kg (65.3% TRR). Metabolite CL 263284 was the second most abundant component and accounted for 0.286 mg eq/kg (17.9% TRR). The metabolites CL 312622 and

CL 189215 were identified at levels of up to 6.7% TRR. In the water extract, the parent compound imazamox was the main constituent at 0.033 mg/kg (2.1% TRR). The remaining components CL 263284, CL 312622 and CL 189215 were identified at levels of up to 0.6% TRR. No signals corresponding to the reference items Reg. No. 60967 (5,5-dimethylimidazolidine-2,4-dione [or 5,5-dimethylhydantoin]; CAS Nr 77-71-4) and Reg. No. 199553 (5-isopropyl-5-methyl-2,4-imidazolidinedione [or iPr-Me-Hydantoin]; CAS-N 5455-35-6)—putative cleavage products of imazamox, imidazoline moiety- were detected in the solvent extracts using HPLC. The residue after solvent extraction was further solubilized. Analysis of the ammonia solubilizate identified imazamox and metabolite CL 263284, which accounted for up to 0.4% of the TRR. In the ERR, 94.5% of the total radioactive residues were identified and 4.1% were characterized. In the PES, additional 3.9% of the TRR were identified and characterized.

In wheat hay, analysis of the methanol extract resulted in a pattern of five peaks, of which four were identified. The parent compound imazamox accounted for 4.056 mg/kg (50.6% TRR). The second most abundant component was metabolite CL 263284, which accounted for 1.689 mg eq/kg (21.1%). The metabolites CL 312622 and CL 189215 were identified at levels of up to 4.2% TRR. In the water extract the same components as in the methanol extract were identified. The parent compound imazamox accounted for 0.910 mg/kg (11.4% TRR). The remaining components CL 263284, CL 312622 and CL 189215 were identified at levels of up to 2.8% TRR. No signals corresponding to the reference items Reg. No. 60967 and Reg. No. 199553 (putative cleavage products of imazamox, imidazoline moiety) were detected in the solvent extracts using HPLC. The ammonia solubilizate contained imazamox and metabolite CL 263284, which accounted for up to 0.4% of the TRR. In the ERR, 96.0% of the total radioactive residues were identified and 1.0% were characterized. In the PES, additional 6.5% of the TRR were identified and characterized.

In wheat straw, the methanol extract showed a pattern of eleven peaks, of which four were identified. The metabolite CL 263284 accounted for 1.041 mg/kg (32.6% TRR). CL 189215 and CL 312622 were 0.394 mg/kg (12.3% TRR) and 0.396 mg/kg (12.4% TRR), respectively. The parent compound imazamox accounted for 6.1% TRR. In the water extract, the same components as in the methanol extract were identified. Likewise, the main constituent was CL 263284 at 0.172 mg eq/kg (5.4% TRR). The remaining components imazamox, CL 189215 and CL 312622 were identified at levels of up to 4.6% TRR. In the solvent extracts up to four non-identified peaks (up to approximately 1% TRR) eluted prior to metabolite CL 189215. It was not possible to assign the peaks to putative cleavage products of imazamox (imidazoline moiety) by HPLC-MS experiments. The ammonia solubilizate contained imazamox, metabolite CL 263284 and CL 312622 at up to 1.2% of the TRR. In the ERR, 78.0% of the TRR were identified and 10.5% were characterized. In the PES, additional 13.7% of the TRR were identified and characterized.

In wheat grain, analysis of the methanol extract of wheat grain resulted in a pattern of eleven peaks, of which three were identified. The parent compound imazamox accounted for 0.577 mg/kg (40.8% TRR) and the metabolites CL 263284 and CL 189215 were identified at levels of up to 3.5% TRR. In the water extract the same components as in the methanol extract were identified. The parent compound imazamox accounted for 0.473 mg/kg (33.5% TRR). The remaining components CL 263284 and CL 189215 were identified at levels of up to 3.0% TRR. In the solvent extracts up to four non-identified peaks (up to approximately 1% TRR) eluted prior to metabolite CL 189215 using HPLC. It was not possible to assign similar peaks to putative cleavage products of imazamox (imidazoline moiety) by HPLC-MS experiments. In the ammonia solubilizate the parent compound imazamox accounted for 0.026 mg/kg (1.8% TRR). In the ERR, 83.4% of the TRR were identified and 5.5% were characterized. In the PES, additional 10.1% of the TRR were identified and characterized.

The ratio of enantiomer 1 to enantiomer 2 for the test item was approximately 1:1. For the parent compound and metabolite CL 263284 isolated from methanol extracts of four matrices (forage, hay, straw and grain), the enantiomer ratio of each compound was found to be approximately 1:1 in all matrices, thus remained unchanged during metabolism of imazamox in wheat.

Table 23 Metabolites detected in wheat matrices following foliar application of [¹⁴C]imazamox (2012/1064722)

Components ^a	Forage (8 DAT)		Hay (8 DAT)		Straw (62 DAT)		Grain (62 DAT)	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Imazamox	1.079	67.3	4.966	62.0	0.251	7.8	1.050	74.4
CL 189215	0.027	1.7	0.411	5.1	0.486	15.2	0.036	2.5
CL 263284	0.295	18.4	1.914	23.9	1.213	37.9	0.092	6.5
CL 312622	0.113	7.0	0.399	5.0	0.544	17.0	n.d.	
Total identified from extracts	1.514	94.5	7.690	96.0	2.494	78.0	1.177	83.4
Total characterized from extracts	0.066	4.1	0.077	1.0	0.337	10.5	0.077	5.5
Total identified and/or characterized from extracts	1.580	98.6	7.767	97.0	2.831	88.5	1.255	88.9
Unextracted (PES)	0.062	3.9	0.523	6.5	0.439	13.7	0.143	10.1
Grand total	1.688	105	8.392	105	3.336	104	1.416	100

^a The extracts (methanol + aqueous) for these samples were combined and analysed by HPLC

n.d.: Not detected

The radioactive residues in wheat samples were initially extracted with methanol and water and analysed by HPLC at 102–126 days (forage), 102–129 days (hay), 45–75 days (straw) and 47–160 days (grain), after sampling. After 324 days (forage), 323 days (hay), 326 days (straw) and 327 days (grain), re-analysis with the methanol and water extract confirmed the initial metabolic patterns. Otherwise, re-extraction from straw and grain was performed 362 days and 336 days, respectively after sampling. The metabolic patterns of the initial analyses remained. During the storage of sample extracts or straw and grain samples over approximately one year, no changes in the metabolic patterns were found.

Study 2

In another wheat study, [pyridine-6-¹⁴C]-imazamox was applied post-emergence to imidazolinone-tolerant wheat (variety: Fidel selection number 2) at a rate of 0.14 kg ai/ha [Johnson 1996b, ID-640-005]. Wheat seeds were planted in a field test site under outdoor condition. Emergence was noted seven days after sowing. The wheat forage, forage of hay stage, straw (including chaff), and grain were harvested 28, 45, 70, and 70 days after treatment, respectively. Additionally, plant samples were taken the same day of treatment (0 DAT). The samples were stored in a freezer (–20 °C). Extracts were stored in a refrigerator or in a freezer. Total radioactive residues were analysed by combustion and LSC

For forage at 0 DAT, 28 and 45 DAT, the radioactive residues were extracted three times with methanol/water (8:2, v/v). The aqueous phase was partitioned three times with methylene chloride.

For wheat grain and straw, residues in the samples were extracted three times with methanol/water (8:2, v/v) and further extracted three times with a mixture of methanol/water/concentrated HCl (8:2:0.2, v/v/v). The PES were extracted with 0.5% t-octylphenoxy-polyethoxyethanol (Triton X-100) and then by 6 N HCl reflux.

Extracts were concentrated, assayed by LSC and characterized/identified by HPLC. Further, imazamox, CL 263284 and CL 312622 were identified by gas chromatography-negative ion chemical ionization mass spectrometric (GC-NICIMS) analysis.

The TRR found in 0 DAT (plant), 28 DAT (forage), 45 DAT (hay stage), straw (70 DAT) and grain (70 DAT) were 5.57 mg eq/kg, 0.102 mg eq/kg, 0.087 mg eq/kg, 0.157 mg eq/kg and 0.067 mg eq/kg, respectively. Most likely plant growth dilution resulted in the rapid decline in residue from the time of application.

The radioactive residues in the 0 DAT, 28 DAT and 45 DAT forage as well as the harvest wheat grain were readily extractable, with 97.4%, 82.3%, 75.6 and 75.2% of the TRR extracted into

methanol/water, respectively. However, only approximately 46% of TRR was extractable from the harvest wheat straw sample. For residual radioactive residues in PES from 28 DAT/45 DAT forage and wheat grain, no further characterization was made (< 0.05 mg eq/kg, 17.7–24.8% TRR).

In straw sample, 91.5% TRR was totally extracted: 46% with aqueous methanol, 6.5% with acidic aqueous methanol, 1% with Triton X-100 and 38% by 6 N HCl reflux. The PES contained only 5.0% (0.008 mg eq/kg) of the TRR.

Table 24 Distribution of residues of [^{14}C]imazamox in wheat samples (ID-640-005)

Matrix	DAT	TRR ^c mg eq/kg	Extracts						PES	
			organic extract		aqueous extract		total		mg eq/kg	%TRR
			mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR		
Plant	0	5.570	5.150	92.5	0.270	4.90	5.42	97.4	0.14	2.6
Forage	28	0.102	0.026	25.6	0.058	56.7	1.084	82.3	0.018	17.7
Forage	45	0.087	0.017	19.6	0.049	56.0	0.066	75.6	0.021	24.4
Grain	70	0.067	0.050 ^d	75.2 ^{**}	n.d.		0.050	75.2	0.017	24.8
Straw	70	0.157	0.083 ^a	52.5 ^a	0.062 ^b	39.0 ^b	0.145	91.5	0.008	5.0

^a Sum of extracted residues with aqueous methanol (46% TRR) and acidic aqueous methanol (6.5% TRR)

^b Sum of extracted residues with X-100 (1% TRR) and 6 N HCl reflux (38% TRR)

^c TRR was determined after combustion.

^d Aqueous methanol

In forage at 0 DAT, the unaltered parent imazamox accounted for 91.8% (5.11 mg/kg) of the TRR. No known metabolites (CL 263284, CL 312622, CL 189215) were detected.

In 28 DAT forage, the concentration of the parent imazamox was 0.042 mg/kg (41.6% TRR) and present in both organic and aqueous phases. There was only one major radioactive peak (imazamox) in the organic phase. In the aqueous phase, there were four peaks (imazamox, CL 263284, CL 312622 and CL 189215). CL 263284 was present at 0.010 mg eq/kg (9.8% TRR). The other two metabolites identified (CL 312622 and CL 189215) were both less than 0.01 mg eq/kg (each < 10% TRR). Several minor components were detected at near background radioactivity.

In 45 DAT forage, the parent imazamox was present at 0.035 mg/kg (39.9% TRR). There was only one major radioactive peak (imazamox) in the organic phase. In the aqueous phase, there were four peaks (imazamox, CL 263284, CL 312622 and CL 189215). The three metabolites were all less than 0.01 mg eq/kg (each < 10% of TRR). Several minor components were detected at near background radioactivity.

In 70 DAT grain, imazamox was present at 0.027 mg/kg (40.3% TRR). Two other components (CL 263284 and CL 189215) were also present at less than 0.01 mg eq/kg (< 10% TRR) each; the di-acid metabolite (CL 312622) was not observed. Several minor components were detected at near background radioactivity.

In 70 DAT straw, the metabolite CL 263284 was present in the largest quantity, at 0.020 mg eq/kg (13.0% TRR), followed by imazamox at 0.015 mg/kg (9.5% TRR), and CL 312622 and CL 189215 at less than 0.01 mg eq/kg (< 10% TRR) each. There were many unknown minor components present at near background levels of radioactivity (< 0.01 mg eq/kg, < 10% TRR each). From extractions with the acidic aqueous methanol, X-100 surfactant and 6 N HCL reflux, the radioactive residues were found at 0.01 mg eq/kg (6.3% TRR), < 0.01 mg eq/kg (1.0% TRR) and 0.06 mg eq/kg (38.0% TRR), respectively. HPLC analysis of the each extract (except surfactant extract due to low radioactivity) showed several minor components at near background radioactivity.

Table 25 Metabolites detected in wheat matrices following foliar application of [^{14}C]imazamox (ID-640-005)

Components	Forage						Grain (70 DAT)		Straw (70 DAT)	
	0 DAT		28 DAT		45 DAT					
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Imazamox	5.11	91.8	0.042	41.6	0.035	39.9	0.027	40.3	0.015	9.5
CL 263284	n.d.		0.010	9.8	0.008	9.7	0.007	10.3	0.020	13.0
CL 312622	n.d.		0.006	6.2	0.004	5.1	n.d.		0.009	5.7
CL 189215	n.d.		0.004	3.5	0.002	2.3	0.002	3.70	0.004	2.8
Total identified from extracts	5.11	91.8	0.062	61.1	0.049	57.0	0.036	54.3	0.048	31.0
Total characterized from extracts	0.31	5.6	0.021	21.4	0.015	18.6	0.014	20.9	0.096	60.5 ^a
Total identified and/or characterized from extracts	5.41	97.4	0.083	82.5	0.064	75.6	0.05	75.2	0.144	91.5
Unextracted (PES)	0.14	2.6	0.018	17.7	0.021	24.4	0.017	24.8	0.008	5.0
Grand total	5.55	100	0.101	100.2	0.085	100	0.067	100	0.152	96.5

^a Sum of the radioactivity not accounted for by the identified compounds in the initial extract and the radioactivity in the dilute acid, X-100 and 6 N acid extracts

PES after extraction with solvents like methanol and water

n.d: not detected

After 4 to 39 days after sampling, the all samples were removed from the combustion and HPLC analyses, except the 0 DAT plant sample. The 0 DAT sample was analysed by HPLC 423 days after sampling. The parent compound imazamox was only significant component in the 0 DAT sample. This indicated that the residue component in the sample was stable for more than 14 months.

Proposed metabolic pathway of imazamox in plants

The metabolism of imazamox was studied in oilseed rape, soya bean, pea, alfalfa, maize and wheat. Application was made on imidazolinone-tolerant varieties, except pea and soya bean. In all the studied crops, [pyridine-6- ^{14}C]-imazamox was treated with post-emergence application (for maize, additionally pre-emergence treatment). In oilseed rape and wheat studies, imidazolinone-labelled imazamox, [imidazolinone-5- ^{14}C , 3- ^{15}N]-imazamox, was also used.

The key step of the metabolism of imazamox was the cleavage of the methyl ether group (demethylation) resulting in metabolite CL 263284. Subsequently, oxidation of the hydroxyl group of CL 263284 generated the dicarboxylic acid metabolite CL 312622, while glycosylation led to the glucose conjugate CL 189215. Both metabolites were formed mostly in very minor amounts. In oilseed rape, soya bean, alfalfa and maize, several polar metabolites were also present. A number of nonpolar metabolites and possibly the hydroxy acid (CL 354825) were found in maize in addition.

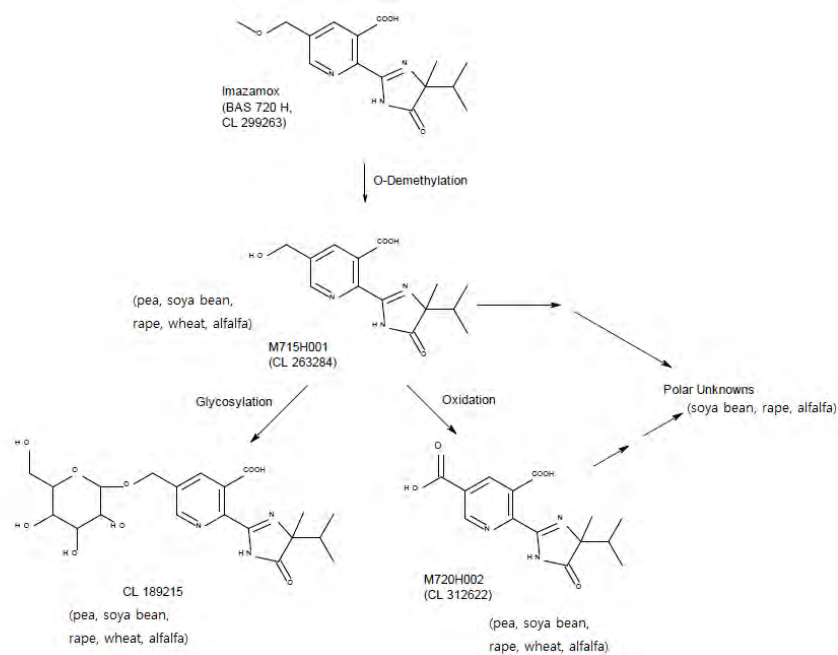


Figure 1 Proposed metabolic pathway of imazamox in pea, soya bean, oilseed rape, wheat and alfalfa

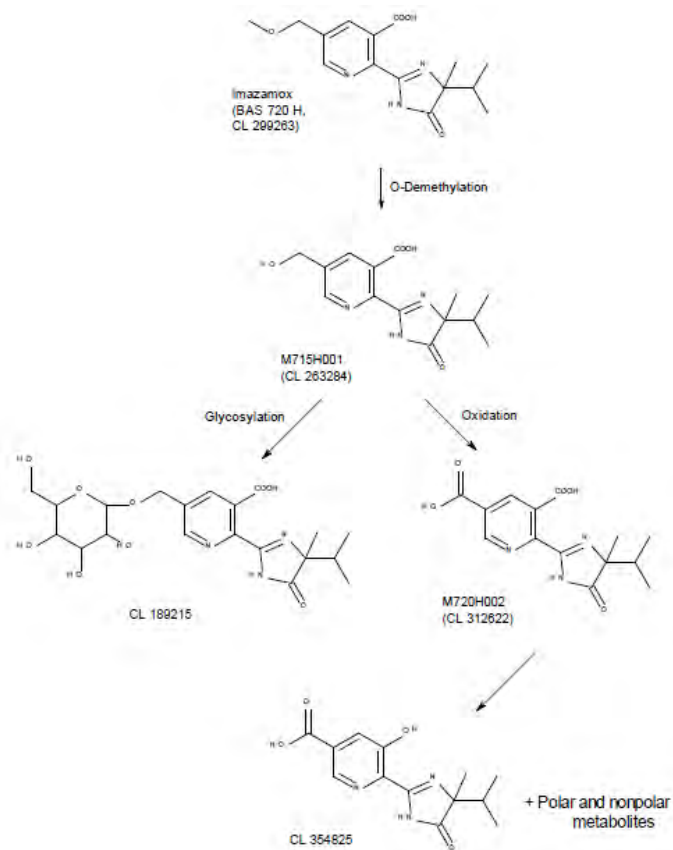


Figure 2 Proposed metabolic pathway of imazamox in maize plant

Environmental in soil

The Meeting received information on aerobic soil degradation, photolysis on soil, hydrolysis, and confined rotational crop studies. The fate and behaviour of imazamox in the environment was investigated using [pyridine-6-¹⁴C]-imazamox, [Imidazolinone-5-¹⁴C]-imazamox, (see Figure 1).

Aerobic degradation in soil—laboratory studies

Study 1

The study was conducted using [pyridine-6-¹⁴C]-imazamox on a New Jersey (sandy loam) soil [Ta 1997a, ID-620-018]. The test compound was applied at a rate of 0.10 kg ai/ha. The soil was maintained aerobically in the dark at 25 ± 1 °C for up to 12 months at 75% of 1/3 bar moisture. The soil characteristics were as follows: % sand, 65; % silt, 22; % clay, 13; CEC (meq/100 g), 9.1; % moisture at 1/3 bar, 14.1; % organic carbon, 1.5; pH in soil:CaCl₂ (1:1), 6.8.

The ethylene glycol and NaOH traps were radioassayed and the foam plugs were extracted with ethyl acetate before being radioassayed. The extracts, after clean up by SPE using C-18 OH and SCX cartridges, were analysed by HPLC and TLC.

Total recoveries of radioactivity ranged from 95.0% to 102% of the total applied radioactivity throughout 1 year of incubation. Imazamox degraded rapidly in soil with an initial half-life of 31 days. The parent compound was initially oxidized to CL 312622 (the di-acid metabolite). The CL 312622 was then converted to a second metabolite CL 354825 (the hydroxy acid metabolite). There were no other metabolites > 5% TAR observed. Imazamox and both metabolites were further mineralized to ¹⁴CO₂ (48.1% TAR over the year) or bound into soil (7.1% TAR at the end of 1 year incubation).

Table 26 Distribution of parent and metabolites (as % TAR) in soil treated with [¹⁴C]imazamox (ID-620-018)

Time	Parent	CL 312622	CL 354825	CO ₂	Bound residue	Others	Total
0 Time	96.3	2.3	ND	ND	1.0	0.0	99.6
1 Day	92.6	4.3	ND	0.8	1.8	0.0	99.5
3 Days	85.4	11.9	ND	1.4	2.3	0.0	101.0
7 Days	71.0	24.0	1.6	2.0	3.0	0.0	101.6
14 Days	59.9	24.3	7.7	3.1	3.4	0.0	98.4
21 Days	59.2	22.0	7.9	4.5	4.3	0.0	97.9
28 Days	50.1	26.2	9.5	6.0	5.3	0.0	97.1
60 Days	33.9	16.8	24.3	12.8	6.2	1.0	95.0
90 Days	29.5	6.6	27.1	21.3	7.3	3.87	95.5
120 Days	20.7	4.2	29.6	27.6	6.7	6.7	95.5
180 Days	17.3	1.3	32.5	33.2	8.9	4.2	97.4
270 Days	15.3	1.1	25.6	42.6	7.9	4.4	96.9
365 Days	15.2	0.2	22.2	48.1	7.1	5.1	97.9

Study 2

The study was conducted using [pyridine-6-¹⁴C]-imazamox on a New Jersey (sandy loam) soil at 25 ± 1 °C for up to 12 months at 75% of 1/3 bar moisture [Ta 1995a, ID-620-008]. The test material was applied at a rate of 0.10 kg ai/ha. The soil characteristics were as follows: % sand, 53.2; % silt, 34.4; % clay, 12.4; CEC (MEQ/100 g), 5.8; % moisture at 1/3 bar, 14.1; % organic carbon, 1.7; pH in soil:CaCl₂ (1:1), 6.6. EDTA (ethylene diamine tetracetic acid-disodium salt) extraction and subsequently followed 0.5 N NaOH extraction recovered the major part of radioactivity in soil.

Total recoveries of radioactivity ranged from 90.5% to 104% of the time0 dose, through 86 days of incubation, and then slowly decreased to 81% of the TAR after 365 days.

Approximately 60% of imazamox was degraded during the first month, 69% by two months, and 77% by 365 days of aerobic incubation. The degradation pattern was biphasic, with an initial half-life of 28 days over the first two months. The DT₉₀ was greater than 365 days.

Imazamox was initially oxidized to CL 312,622 (identified by TLC, HPLC, MS and NMR). The di-acid reached a maximum concentration of approximately 44% of the applied dose at 4–6 weeks after dosing. It then decreased over the remainder of the study and accounted for less than 5% of the applied dose from 6 to 12 months. A second metabolite, CL 354,825 (identified by TLC, HPLC, MS and NMR) increased in concentration over time, and accounted for approximately 55% of the applied dose from 6 to 12 months. Ultimately imazamox and its degradates were mineralized to CO₂.

Table 27 Distribution of parent and metabolites (as % TAR) in soil treated with [¹⁴C]imazamox (ID-620-008)

Time, days	% Parent	% AC 312622	% AC 354825
0	89.9	2.9	1.0
2	93.2	11.9	2.8
4	75.4	18.2	3.0
7	55.7	31.2	6.4
14	45.1	40.8	13.0
21	38.5	42.5	12.0
90	30.5	43.6	16.2
44	24.7	40.6	22.1
58	21.4	33.7	26.0
86	16.3	27.3	35.8
114	16.1	13.8	44.8
177	13.5	3.3	53.5
267	11.8	3.4	55.2
365	12.5	2.9	54.0

Study 3

The rate of degradation of [pyridine-6-¹⁴C]-imazamox was studied on three EU soils (two silt loams [Ta and Lewis 1997a, ID-620-027]. The test material was applied to soil at a rate of 0.075 kg ai/ha. The soil moistures were maintained at approximately 45% of the maximum water holding capacity (MWHC). Soil samples were extracted three times with 0.5 M NaOH solution. The extracts were subjected to acid precipitation. After centrifugation, the humic acid fraction (precipitate) was re-dissolved in 0.5 N NaOH. The supernatant (fulvic acid fraction) was passed through a C-18 cartridge and the radioactivity was eluted with methanol:water (90:10 v/v). The extracts of one of the silt loam soils (Soil A) were further cleaned-up using a silica column. All samples were concentrated prior to chromatography by HPLC and TLC. Unextracted soil residues (humic fraction) were dried and aliquots combusted.

Table 28 Characteristics of soils used (ID-620-027)

Location	Limours (FR) Soil A	Boissy (FR) Soil B	Ponfaverger (FR) Soil C
% Sand	4.7	7.9	14.3
% Silt	79.4	74.7	60.7
% Clay	15.8	17.4	25.1
USDA Textural Class	Silt loam	Silt loam	Silty clay loam
CEC (meq/100 g)	13	12.6	18.3
% Moisture at 1/3 bar	20.4	19.4	22.2
% Organic Carbon	0.8	0.8	1.1
pH in 1:1 soil:water ratio	5.8	6.5	8.1

CEC: cation exchange capacity

Mass balance at 122 days was $\geq 96\%$ ($\geq 95\%$ at all earlier time points).

In the silty clay loam (soil C), at 20 °C, the amount of AC 299,263 decreased from 97% to 4% of applied radioactivity during the 122 day incubation period. CL 312,622, was a major metabolite at especially 14 days and 27 days (42%) but then decreased to 2% at 122 days. CL 354,825 increased to 40% at 66 days then remained at about this level for the remainder of the incubation period.

In one of the silt loams (soil B) at 20 °C, the amount of imazamox decreased from 95% of applied radioactivity to 61% at 27 days. The main degradate was CL 354,825, which increased to 33% at 66 days. CL 312,622 was a minor metabolite reaching its maximum concentration of 8% at 14 days.

In the other silt loam (soil A) at 20 °C, degradation of imazamox was much slower than either of the other soils at the same temperature. Imazamox decreased from 91% TAR at 0 day to 73% TAR at 122 days, CL 354,825 increased to 12% TAR at 122 days but amounts of CL 312,622 were < 1% throughout the incubation period.

In the silty clay loam (soil C) at 10 °C, the amount of imazamox declined from 97% TAR to 12% TAR at 122 days. CL 312,622 was the principal metabolite and increased to 45% TAR at 66 days, then remained at this level to the end of the study (42% at 122 days). Levels of CL 354,825 increased during the study and accounted for 24% at 122 days.

In the silt loam (soil B, Boissy, FR) at 10 °C, the amount of imazamox declined from 96% TAR to 42% TAR at 122 days. CL 312,622 and CL 354,825 were both formed, and accounted for 22% and 19%, respectively after 122 days.

Several minor unidentified degradation products were detected but in total accounted for < 6% of applied radioactivity at any time point. Small amounts of radioactivity were also detected in the non-absorbed fraction from the C-18 cartridges ($\leq 5\%$) and the humic acid fractions ($\leq 4\%$), but these were not characterized.

The calculated first-order DT₅₀ and DT₉₀ values are summarized as follows:

Soil	Temperature (°C)	DT ₅₀ (Days)	DT ₉₀ (Days)
C (Silty clay loam)	20	12	39
B (Silt loam)	20	44	147
A (Silt loam)	20	207	687
C (Silty clay loam)	10	42	140
B (Silt loam)	10	113	375

Imazamox was degraded in this laboratory study at rates dependent upon the soil type and temperature. In most incubation groups two major metabolites (CL 312622 and CL 354825) were present. Radiolabelled residues were also incorporated into soil bound residues (especially the humin fraction, values up to 17%). Up to 24% (1–24%) of the radioactivity was evolved as volatiles, mainly carbon dioxide, demonstrating eventual mineralization of imazamox.

Study 4

The aerobic metabolism of imazamox in soils was conducted using [Imidazolinone-5-¹⁴C]-imazamox [Ta 2012a, 2011/7002438]. Two soils of different textures: a sandy loam soil (Limbergerhof, Rheinland-Pfalz, Germany) and a loam soil (Hunterdon County in New Jersey, USA) were used. The test material was applied at the rate of 0.050 kg ai/ha (0.066 mg ai/kg) and the soil moistures were maintained to approximately 50% MWHC. The soil samples were extracted once with methanol, followed by 50% methanol in water, twice. Soils were further extracted with 0.5 N NaOH solution and then the extract was subjected to acid precipitation.

Table 29 Characteristics of soils used (2011/7002438)

Location	Bruch West (DE)	New Jersey (US)
% Sand	65.7	28
% Silt	23.1	49

Location	Bruch West (DE)	New Jersey (US)
% Clay	11.2	23
USDA Textural Class	Sandy loam	Loam
CEC (meq/100 g)	12.6	7.6
Maximum water holding capacity %	31.5	37.4
% Organic Carbon	1.38	1.16
pH in 1:1 soil:water ratio	8.1	7.1

The average total recovery of the radiolabelled material was 95.5% of the TAR for the entire samples from two soils.

From both soils, organic solvent extractable [^{14}C]-residues decreased from approximately 94.2% to 96.8% TAR at 0 days after treatment to approximately 23.4% to 40.5% TAR at 120 DAT. NaOH extracts increased from 3.2% to 3.4% at 0 DAT to 35.8% to 49.5% TAR at 120 DAT and non-extractable [^{14}C]-residues increased from approximately 0.8% to 1.6% TAR at 0 DAT to 17.7% to 22.1% TAR at 120 DAT. At 120 DAT, the total evolved $^{14}\text{CO}_2$ was 2.5 to 6.8% TAR.

The concentration of [imidazolinone-5- ^{14}C]-imazamox decreased to approximately 28.4% to 38.8% TAR at 120 DAT. There were two major (> 5% TAR) transformation products, CL 312622 and CL 354825 at maximum values of 14.8% to 30.7% TAR, and 18.1% to 28% TAR, respectively. Several minor transformation products were also observed and none of them reached concentration of > 5% TAR. Additional investigations on the potential shift of the isomer ratio of imazamox and its metabolites during soil incubation were performed. The results clearly show that the isomer ratios were almost constant over time.

The DT_{50} values of imazamox were calculated to be 38.1 days and 23.1 days in sandy loam soil (Bruch West, Germany) and loam soil (New Jersey, USA), respectively. Mineralization was observed with levels of CO_2 reaching values of 2.5% to 6.8% TAR after 120 days of incubation.

Proposed degradation pathway of imazamox in soil under aerobic condition

Imazamox was rapidly degraded in soil. Half-life of imazamox was 28–38 days or 12–207 days (depending on soil types and temperatures). In soil, two major metabolites, CL 312622 (di-acid) and CL 354825 (hydroxyl acid) were present. Imazamox was initially oxidized to CL 312,622 (the di-acid metabolite). The CL 312622 was then converted to a second metabolite CL 354,825 (the hydroxy acid metabolite). There were no other metabolites > 5% TAR observed. Parent and both metabolites were further mineralized to $^{14}\text{CO}_2$ or bound into soil.

Photodegradation on soil surface

Study 1

The photolysis of imazamox was conducted using [pyridine-6- ^{14}C]-imazamox [Ta 1995b, ID-620-004]. The test compound was applied to New Jersey (sandy loam) soil (53.2% sand; 34.4 silt; 12.4% clay; 1.7% organic carbon; pH 6.6; CEC 5.8 meq/100 g; 14.1% soil moisture at 1/3 bar) at a rate equivalent to 0.10 kg/ha (40 ppm on a soil weight basis) in photolysis chamber. Samples were irradiated continuously for up to 30 days by light from a xenon-arc lamp (filtered to remove wavelengths less than 290 nm) and maintained at 25 °C and 75% of 1/3 bar moisture during the course of the study. Soil sample was extracted two times with 0.5 M NaOH, then the extract was combusted and analysed by LSC. TLC (normal and reverse phase) and HPLC were used for identification.

Total radioactivity recovered from the irradiated samples ranged between 97.7% TAR and 101.3% TAR (105.5–109.0% TAR in control samples) throughout the experiment. These recoveries demonstrated that volatile degradates were not formed.

The 0.5 M NaOH extracts contained 96% or greater of the radioactivity initially applied to the soil. The residual radioactivity remaining in the soil after extraction ranged from 0.5% TAR to 1.3% TAR in the irradiated samples (0.8–1.2 % TAR in the control samples).

TLC analyses of the soil extracts showed that approximately 68.6% (normal phase TLC) to 73.7% (reverse phase TLC), averaging 71% of the parent compound remained intact at the end of the 30 days irradiation. The control samples were stable throughout the course of the study. The half-life was calculated to be 65 days of continuous irradiation.

There was one major product formed, CL 312,622 (the di-acid metabolite) which accounted for approximately 11.9% TAR (normal phase TLC) to 15.4% TAR (reverse phase TLC), averaging 14% TAR after 30 days. This metabolite was also found in the dark control sample, indicating that this metabolite is not photolytic specific degradate. There were at least ten other unidentified radiolabelled components, each of which represented less than 6% TAR.

Study 2

The photolysis of imazamox was conducted using [pyridine-6-¹⁴C]-imazamox or [imidazolinone-5-¹⁴C]-imazamox [McCall and Blood 2013a, 2012/7003612]. The test compound was applied to a loam soil from New Jersey at a rate of 0.050 kg/ha (0.33 ppm based on an assumed bulk density of 1.5 g/cm³ and 1 cm depth). Samples were irradiated continuously for up to 15 days at 20 °C by light from a xenon-arc lamp filtered to remove wavelengths less than 290 nm. Total irradiance during the duration of the study was 528.56 W/m⁻² which is comparable to the natural sunlight intensity in the spring at 40 ° N latitude. The loam soil characteristics were as follows: 28% sand; 48% silt; 24% clay; 1.3% organic carbon; pH 6.9; CEC 8.9 meq/100 g; 49% a maximum of water holding capacity.

Volatile residues were collected for the irradiated and the dark control samples and analysed at the soil sampling intervals. Each sample was extracted once with 40 mL methanol and two times with 40 mL methanol:water (1:1; v:v) and finally with 40 mL 0.5 N NaOH at each sampling point. The extracts were analysed by HPLC. Reference standards were used for co-chromatography for characterization in conjunction with MS/MS analysis of select samples.

The mass balance for both irradiated and dark controls for the imidazolinone label ranged from 89.3% TRR to 101.2% TAR. The cumulative volatile ¹⁴C-residues from the irradiated and dark control samples were 10.7 %TAR for the light and for the dark controls, 1.2% of the TAR. The mass balance for both irradiated and dark controls for the pyridine label ranged from 98.0% to 102.5% TAR.

In overall, extractabilities of radioactive residues were in the range of 70.7–99.9% TRR.

The DT₅₀ value of imazamox in the irradiated samples was calculated to be approximately 28.4 days (both labels, n=4) and found to be 19.7 days in the dark control samples. One minor product (< 10% TAR) observed in the irradiated samples was the di-acid CL 312,622. It accounted for a maximum level of approximately 8% TAR through the study period in both labels. In the control samples, CL 312622 accounted for a maximum of 41% TRR in the imidazolinone label, and a maximum of 25% TRR in pyridine label through the study period. There were no unique photoproducts of imazamox in soil at 20 °C.

These studies indicated that imazamox on soil surface is photodegraded and any specific metabolite is not formed under irradiation. The half-life of imazamox was 65 days or 28 days and it was slightly longer in dark control condition. One metabolite CL 312622 was present in irradiated soil as a major or minor component; however, it was not specific for photolysis.

Hydrolysis

Test solution containing imazamox at concentration of 8 µg/mL was prepared in pH 4, 7 and 9 buffers and incubated at 50 °C [Holman 1997a, ID-322-002]. The test was conducted for 5 days in the dark under sterile conditions. Additional samples buffered at pH 9 were incubated for up to 24 days at 50 °C, 10 days at 60 °C, and 4 days at 70 °C. Aliquots of the samples were analysed by HPLC.

No degradation of imazamox occurred during the test period of 5 days at 50 °C in pH 4 and pH 7 buffers, indicating that the test compound is stable to hydrolysis in these buffers.

In the sample at pH 9, considerable loss of the compound was observed. For samples at pH 9 and 50 °C, the half-life of imazamox was calculated to be 11.9 days; at 60 °C, 4.17 days; and at 70 °C, 1.70 days. Extrapolating to 25 °C, the half-life is 192 days.

There was one major hydrolysis product confirmed by LC-MS. This was identified as the carbamoyl compound CL 336554, formed from the ring opening at the nitrogen bond.

The study indicated that imazamox is hydrolytically stable at pH 4 and pH 7. However, at pH 9 at 25 °C, it hydrolyses with a half-life of 192 days, forming the hydrolysis product, carbamoyl compound CL 336554.

Metabolites and distribution in succeeding crops

Confined Rotational Crop Study

Study 1

Soya beans (variety, Hutcheson) were treated at the 4–6 leaf stage (sandy loam soil) with [pyridine-6-¹⁴C]-imazamox at a rate of 0.072 kg ai/ha (ammonium salt formulation) [Gatterdam 1994b, ID-640-002]. At 100 days after treatment (100 DAT), the soya bean crop was harvested and winter wheat (variety, Pioneer 2548) was seeded in subplot 1. At 268 DAT, maize (variety, Pioneer 3165), radish (variety, White Icicle) and lettuce (variety, Black-Seeded Simpson) were seeded (subplots 2–4), respectively. Radish and lettuce were planted in subplots 5 and 6 at 420 DAT.

Soya bean foliage was sampled at the day of treatment; fodder and seed were taken at harvest, 100 DAT. The rotational crop commodities wheat forage, maize forage (stalk, ear foliage and cob), radish leaves and lettuce plants were sampled at mid-maturity, 238, 357, 302 and 311 DAT, respectively. Wheat grain, maize seed, radish roots and lettuce plants were taken at harvest, 331, 420, 311 and 335 DAT, respectively. The study was terminated before harvesting the 420 DAT samples (radish and lettuce), thus these samples were not analysed for radioactive residues. Soil samples were taken before treatment, immediately after application, at rotational crop planting and at crop sampling (mid-maturity and harvest). Soil samples were 46 cm in depth (sections of 0–8, 8–15, 15–30, and 30–46 cm) except the pre-treatment and 0 DAT cores which were 30 cm in depth. Plant tissue and soil samples were combusted and ¹⁴C was determined by LSC.

The soil extract was analysed by HPLC-UV. The column was eluted isocratically with a mobile system consisting of acetonitrile/ tetrahydrofuran/formic acid/water (14:2:1:83, v/v/v/v). A liquid scintillation cocktail was added for LSC. The retention time of imazamox analytical standard was compared to radiochromatograms of soil extracts to verify identification of the radioactivity.

Soya bean foliage at 0 DAT contained total radioactive residues averaging 1.70 mg eq/kg. The plants at harvest (100 DAT) were collected but not analysed.

Rotational crops planted at 100 DAT (wheat) and 268 DAT (maize, radish and lettuce) showed TRRs below the limit of detection of 0.01 mg/kg at both mid-maturity and harvest intervals. Further analysis for radish and lettuce planted at 420 DAT was not performed.

Radioactive residues in treated soil (0–8 cm) at 0 DAT averaged 0.03 mg/kg. At the 100 DAT, 268 DAT and 420 DAT rotational crop intervals, there were no detectable residues in soil (< 0.01 mg eq/kg TRR).

Plant and soil samples were stored frozen from sampling until analysis. Sampling to final analysis ranged from 14 to 71 days for crops and from 8 to 147 days for soil.

The stability of the treatment solution was verified in a soil extract sample (0 DAT). Recovery of radioactivity by extraction showed 92.8% was extracted and 7.2% was unextracted. Cartridge clean-up (C₁₈ SPE) of the soil extract yielded two carbon-14 containing residues. The major fraction (approximately 90% of the cleaned up residue) showed one peak by HPLC constituting 97.3% of the recovered radioactivity. This radioactive peak coincided with imazamox on the HPLC-

UV chromatogram. The minor fraction (approximately 10% of the cleaned up residue) was not analysed due to low radioactivity.

Table 30 Total radioactive residues in crops after treatment with [^{14}C]imazamox (ID-640-002)

Matrix (Days after sowing /planting, DAP)	TRR (mg eq/kg)
Soya bean (0 DAT)	1.70
Soya bean (100 DAT)	n.a.
Plant back interval: 100 DAT, seeded	
Wheat forage (138 DAP, 238 DAT)	< 0.01
Wheat grain (231 DAP, 331 DAT)	< 0.01
Plant back interval: 268 DAT, seeded	
Maize forage (89 DAP, 357 DAT)	< 0.01
Maize seed (152 DAP, 420 DAT)	< 0.01
Radish leaves (34 DAP, 302 DAT)	n.a.
Radish roots (43 DAP, 311 DAT)	< 0.01
Lettuce plants (43 DAP, 311 DAT)	< 0.01
Lettuce plants (67 DAP, 335 DAT)	< 0.01
Plant back interval: 420 DAT, planted	
Radish	n.a.
Lettuce	n.a.

DAT: Days after treatment

n.a.: not analysed

Table 31 Total radioactive residues in soil samples following treatment with [^{14}C]imazamox (ID-640-002)

Soil Samples (Days after treatment, DAT)	TRR determined by direct combustion (mg eq/kg)
(0 DAT)	0.03 (0–8 cm); < 0.01 (8–46 cm)
(100 DAT)	< 0.01
Plant back interval: 100 DAT	
Wheat (100, 238, 331 DAT)	< 0.01
Plant back interval: 268 DAT	
Maize (268, 357, 420 DAT)	< 0.01
Radish (268, 302, 311 DAT)	< 0.01
Lettuce (268, 311, 335 DAT)	< 0.01
Plant back interval: 420 DAT	
Radish (420 DAT)	< 0.01
Lettuce (420 DAT)	< 0.01

Study 2

A confined rotational crop study was conducted with two radiolabels-[imidazolinone 5- ^{14}C , 3- ^{15}N]-imazamox and [pyridine-3- ^{14}C , imidazolinone-3- ^{15}N]-imazamox [Funk and Possienke 2013b, 2013/1085609]. The active substance was applied to bare sandy loam soil (USDA scheme) in plastic containers at a rate of 0.075 kg ai/ha using an automatic spray track system. The nature and the level of radioactive residues were investigated in spinach (variety, Corvette F1; immature and mature), white radish (variety, April Cross; root and top) and spring wheat (variety, Thassos; forage, hay, straw and grain). For the imidazolinone label samples from the plant back intervals 31, 119 and 364 days were investigated, while for the pyridine label plant back intervals of 29, 123 and 365 days were used.

Plant samples were harvested at maturity and additional immature spinach samples as well as spring wheat forage samples (in part dried to hay) were taken 23 to 33 days and 50 to 75 days after sowing, respectively. Mature and immature spinach leaves were sampled and the roots remained in the soil. Ripe white radishes were pulled from the soil and separated into the edible parts (root) and the remaining green parts (top). Soil samples were taken after ploughing at the individual plant back intervals and after harvest of the mature crops.

All samples were stored in a freezer at -18°C or below. Prior to extraction and determination of the TRR, sample material was homogenized. Plant matrices with a sufficient level of radioactivity were extracted with solvents, while for samples with low concentrations of radiolabelled compounds the amount of the TRR was obtained only by combustion analysis. Soil samples were directly subjected to combustion.

Aliquots of homogenized plant material were extracted three times with methanol. The residue was further extracted in the same way with water (twice). The TRR included in some spring wheat hay and straw samples radioactive residue of pendimethalin, which was identified as a contamination in this study. The obtained methanol extracts of spring wheat matrices were partitioned three times with ethyl acetate for forage, hay and grain, with cyclohexane for wheat grain. For grain, further partition for the obtained ethyl acetate phase was conducted with n-hexane.

The radioactive residues in the PES (after solvent extraction with methanol and water) were subsequently extracted twice with ammonia. After ammonia extraction, the residues were dried and subsequently solubilized with different enzymes (β -glucosidase and hesperidinase; macerozyme R-10 and cellulase Onozuka R-10; α -amylase, β -amylase and amyloglucosidase). For straw, the macerozyme solubilizes were treated with yeast (fermentation). For grain, water extract derived from the solvent extraction with water two times was precipitated with acetone. The precipitate was treated with protease. The extracts were analysed by LSC and if needed, HPLC.

The calculated TRR of all extracted rotational crop matrices showed no major differences to the TRR values obtained by combustion.

At all plant back intervals, the highest TRR levels were found in spring wheat hay, straw and grain. The concentration of residues in grain decreased significantly after a plant back interval of approximately 365 days: in imidazolinone/pyridine label, respectively, 0.032/0.053 mg eq/kg at one month PBI and 0.002/0.004 mg eq/kg at one year PBI. For spinach and white radish, much lower TRR values (less than 0.01 mg eq/kg) were found in all PBI intervals and both labels.

Table 32 Total radioactive residues in crops after treatment with [^{14}C]imazamox (2013/1085609)

Matrix (Days After Sowing /Planting, DAP)	TRR Combusted (mg eq/kg)		TRR Calc. (mg eq/kg)	
	Imidazolinone label	Pyridine label	Imidazolinone label	Pyridine label
Plant back interval days	31	29	31	29
Spinach (immature) (25/26)	0.006	0.009	0.005	0.008
Spinach (mature) (43/41)	0.003	0.005	0.003	0.004
White radish top (64/67)	0.005	0.006	0.006	0.005
White radish root (64/67)	0.003	0.003	0.003	0.003
Spring wheat forage (52/52)	0.008	0.014	0.008	0.012
Spring wheat hay (52/52)	0.046	0.078	0.042	0.074 ^b
Spring wheat straw (incl. chaff) (115/108)	0.044	0.077	0.040 ^b	0.080 ^b
Spring wheat grain (115/108)	0.032	0.053	0.030	0.051
Plant back interval days	119	123	119	123
Spinach (immature) (26/23)	0.005	0.002	0.004	0.002
Spinach (mature) (42/39)	0.005	0.002	0.004	0.002
White radish top (60/65)	0.009	0.003	0.007	0.002
White radish root (60/65)	0.002	0.002	0.001	0.001
Spring wheat forage (53/50)	0.012	0.004	0.011	0.004
Spring wheat hay (53/50)	0.050	0.023	0.043	0.020
Spring wheat straw (incl. chaff) (97/108)	0.132	0.026	0.111 ^b	0.025
Spring wheat grain (97/108)	0.035	0.019	0.036	0.019
Plant back interval days	364	365	364	365

Matrix (Days After Sowing /Planting, DAP)	TRR Combusted (mg eq/kg)		TRR Calc. (mg eq/kg)	
	Imidazolinone label	Pyridine label	Imidazolinone label	Pyridine label
Spinach (immature) (33/33)	< 0.001	0.004	< 0.001 ^a	0.004
Spinach (mature) (46/48)	< 0.001	0.005	< 0.001 ^a	0.004
White radish top (60/76)	< 0.001	0.006	< 0.001 ^a	0.006
White radish root (60/76)	< 0.001	0.001	< 0.001 ^a	0.001
Spring wheat forage (55/75)	< 0.001	0.008	< 0.001 ^a	0.008
Spring wheat hay (55/75)	0.002	0.052	0.002 ^a	0.051
Spring wheat straw (incl. chaff) (137/129)	0.003	0.034	0.003 ^a	0.033
Spring wheat grain (137/129)	0.002	0.004	0.002 ^a	0.005

DAT: days after treatment

^a TRR combusted. TRR calculated was not determined because of low amount of radioactivity.

^b Including a contamination of pendimethalin

Soil samples (imidazolinone label/pyridine label) were taken at 0/0 day DAT. On the days after ploughing at 31/29, 119/123 and 364/365 DAT (PBIs) and on the days after harvest of mature crops, the each labelled soil samples were taken at 74, 161, 410 DATs in spinach, 95, 179, 424 DATs in white radish and 146, 216, 501 DATs in spring wheat.

In both labels, the residue concentration in the top soil layer decreased rapidly after aging and ploughing, 0.670 mg eq/kg (0 DAT) to 0.019 mg eq/kg (31 DAT) in imidazolinone label and 0.694 mg eq/kg (0 DAT) to 0.050 mg eq/kg (29 DAT) in pyridine label. Then, the level decreased relatively in small ratio: imidazolinone label 0.013 mg eq/kg at 364 DAT and pyridine label 0.018 mg eq/kg at 365 DAT. After harvest of the mature crops, the residue levels in soil were 0.009–0.019 mg eq/kg for the all PBIs of both labels, being remained more or less stable.

The extractability of the radioactive residues with methanol and water ranged for rotational crop matrices from 17.1% to 77.1% TRR in the imidazolinone label and from 17.8% to 77.9% TRR for the pyridine label. In spring wheat grain, low extractability with both solvents was observed (less than 30% TRR). Besides, extractabilities for some forage and hay sample (pyridine label, 123 DTA) were low (less than 30% TRR). The major portions of the radioactive residues were generally extracted with methanol, except for spring wheat grain (all PBIs, both labels), hay (123 DAT, pyridine label) and straw (365 DAT, pyridine label) where similar portions were extracted with methanol and water.

In most cases, higher portions of the radioactive residues extracted with methanol were water soluble, and lower portions were found in the organic fractions (ethyl acetate phase or cyclohexane phase). In some cases, comparable portions were found in the organic phases and in the water phase.

HPLC analysis for all spring wheat matrices and plant back intervals showed a metabolite pattern with only a few peaks. Parent imazamox was identified very small in three matrices: 31 DAT grain (0.002 mg/kg, 6.2% TRR) in imidazolinone label and 29 DAT hay (0.007 mg/kg, 9.1% TRR) and 29 DAT straw (0.002 mg/kg, 2.1%) in pyridine label. The metabolite CL 263284 was found in only two matrices-29 DAT hay (0.002 mg/kg, 2.3% TRR) and 123 DAT straw (0.001 mg/kg, 5.5% TRR) in pyridine label. CL 263284 is characterized by a hydroxyl group, derived from the cleavage of the methyl ether group (demethylation) of the parent. There were degradation products in minor concentrations. These were characterized by HPLC and/or liquid-liquid partition.

		Extracts						PES	
DAT	TRR calc.	Methanol extract		Water extract		Total			
	mg eq/kg	% TRR	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
<i>Spinach (immature)</i>									
31	0.005	0.003	50.1	< 0.001	6.0	0.003	56.1	0.002	43.0
119	0.004	0.003	74.7	< 0.001	2.4	0.003	77.1	0.001	22.9
<i>Spinach (mature)</i>									
31	0.003	0.002	52.7	< 0.001	8.2	0.002	60.9	0.001	39.1
119	0.004	0.002	64.7	< 0.001	3.4	0.002	68.1	0.001	31.9
<i>White radish top</i>									
31	0.006	0.003	53.8	0.001	11.1	0.004	64.9	0.002	35.1
119	0.007	0.004	63.0	0.001	9.7	0.005	72.7	0.002	27.3
<i>White radish root</i>									
31	0.003	0.002	60.3	< 0.001	4.3	0.002	64.6	0.001	35.4
119	0.001	0.001	56.0	< 0.001	0.0	0.001	56.0	0.001	44.0
<i>Spring wheat forage</i>									
31	0.008	0.003 n.a. ^a n.a. ^b	35.5 n.a. ^a n.a. ^b	< 0.001	5.2	0.003	40.7	0.005	59.3
119	0.011	0.005 0.001 ^a 0.004 ^b	48.7 13.9 ^a 33.5 ^b	0.001	6.3	0.006	55.0	0.005	45.0
<i>Spring wheat hay</i>									
31	0.042	0.017 0.007 ^a 0.008 ^b	39.4 16.4 ^a 19.9 ^b	0.001	2.9	0.018	42.3	0.024	57.7
119	0.043	0.018 0.007 ^a 0.012 ^b	42.0 16.3 ^a 28.7 ^b	0.006	14.0	0.024	56.0	0.019	44.0
<i>Spring wheat straw</i>									
31	0.040	0.016 0.011 ^a 0.007 ^b	41.2 28.0 ^a 17.6 ^b	0.003	7.9	0.019	49.2	0.020	50.8
119	0.111	0.056 0.035 ^a 0.020 ^b	50.0 31.7 ^a 17.6 ^b	0.018	16.1	0.074	66.7	0.037	33.3
<i>Spring wheat grain</i>									
31	0.030	0.004 0.001 ^a 0.003 ^b	12.6 3.5 ^a 8.8 ^b	0.004	13.0	0.008	25.5	0.022	74.5
119	0.036	0.004 0.002 ^a 0.002 ^b	10.7 5.4 ^a 5.3 ^b	0.002	6.4	0.006	17.1	0.030	82.9

^a Organosoluble: ethyl acetate used as organic solvent for forage, hay and straw extracts; cyclohexane used as organic solvent for grain extracts.

		Extracts						PES	
DAT	TRR calc.	Methanol extract		Water extract		total			
	mg eq/kg	% TRR	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
<i>Spinach (immature)</i>									
29	0.008	0.004	50.7	0.001	6.2	0.005	56.8	0.004	43.2
123	0.002	0.001	54.0	< 0.001	9.9	0.001	63.9	0.001	36.1
365	0.004	0.003	72.2	< 0.001	5.7	0.003	77.9	0.001	22.1
<i>Spinach (mature)</i>									

		Extracts						PES	
DAT	TRR calc.	Methanol extract		Water extract		total			
	mg eq/kg	% TRR	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
29	0.004	0.002	42.4	< 0.001	8.2	0.002	50.6	0.002	49.4
123	0.002	0.001	45.1	< 0.001	9.8	0.001	54.9	0.001	45.1
365	0.004	0.002	59.6	< 0.001	7.1	0.002	66.7	0.001	33.3
<i>White radish top</i>									
29	0.005	0.003	49.4	0.001	10.0	0.003	59.4	0.002	40.6
123	0.002	0.001	42.5	< 0.001	12.4	0.001	54.9	0.001	45.1
365	0.006	0.004	62.1	0.001	11.4	0.004	73.5	0.002	26.5
<i>White radish root</i>									
29	0.003	0.002	69.6	< 0.001	2.9	0.002	72.5	0.001	27.5
123	0.001	0.001	67.4	< 0.001	2.6	0.001	70.0	< 0.001	30.0
365	0.001	0.001	66.8	< 0.001	4.0	0.001	70.8	< 0.001	29.2
<i>Spring wheat forage</i>									
29	0.012	0.005 0.002 ^a 0.003 ^b	41.2 13.1 ^a 24.8 ^b	< 0.001	3.8	0.005	45.0	0.006	55.0
123	0.004	0.001 n.a. ^a n.a. ^b	24.0 n.a. ^a n.a. ^b	< 0.001	4.8	0.001	28.8	0.003	71.2
365	0.008	0.004 n.a. ^a n.a. ^b	44.4 n.a. ^a n.a. ^b	< 0.001	6.0	0.004	50.4	0.004	49.6
<i>Spring wheat hay</i>									
29	0.074	0.025 0.010 ^a 0.014 ^b	33.7 13.3 ^a 19.4 ^b	0.007	9.8	0.032	43.4	0.042	56.6
123	0.020	0.002 0.001 ^a 0.004 ^{bc}	9.1 ^a 5.6 ^b 18.0	0.002	8.7	0.004	17.8	0.014	67.8
365	0.051	0.020 0.006 ^a 0.011 ^b	39.1 12.5 ^a 21.1 ^b	0.011	21.2	0.030	60.3	0.020	39.7
<i>Spring wheat straw</i>									
29	0.080	0.039 0.026 ^{ad} 0.012 ^b	48.6 32.3 ^a 15.3 ^b	0.010	12.6	0.049	61.3	0.031	38.7
123	0.025	0.006 0.003 ^a 0.003 ^b	23.6 10.1 ^a 12.3 ^b	0.003	11.4	0.009	35.0	0.016	65.0
365	0.033	0.007 0.002 ^a 0.004 ^b	21.5 6.7 ^a 12.4 ^b	0.007	20.5	0.014	42.0	0.019	58.0
<i>Spring wheat grain</i>									
29	0.051	0.008 0.002 ^a 0.006 ^b	15.9 3.9 ^a 11.3 ^b	0.007	13.4	0.015	29.3	0.036	70.7
123	0.019	0.002 0.001 ^a 0.001 ^b	8.7 2.9 ^a 4.4 ^b	0.002	9.3	0.003	18.0	0.015	82.0
365	0.005	0.001 n.a. ^a n.a. ^b	11.8 n.a. ^a n.a. ^b	0.001	16.0	0.001	27.8	0.003	72.2

TRR calc.: sum of extracts + PES

^a Organosoluble: ethyl acetate used as organic solvent for forage, hay and straw extracts; cyclohexane used as organic solvent for grain extracts.

^b Water soluble: ethyl acetate used as organic solvent for forage, hay and straw extracts; cyclohexane used as organic solvent for grain extracts.

^c Radioactive residues in methanol extract presumably underestimated (low dpm values in LSC measurement).

^d Organosoluble as sum of water phase and n-hexane phase derived from ethyl acetate phase

Table 35 Identified or characterized residues in wheat samples—imidazolinone label (2013/1085609)

Designation	Wheat forage 119 DAT		Wheat hay 31 DAT		Wheat hay 119 DAT		Wheat straw 31 DAT		Wheat straw 119 DAT		Wheat grain 31 DAT		Wheat grain 119 DAT	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Imazamox								12.1			0.002	6.2		
Total characterized from extracts	0.006	53.7	0.018	42.0	0.025	59.0	0.017	44.1	0.072	64.4	0.005	17.1	0.006	16.5
Total characterized from PES			0.014	33.5	0.009	20.6	0.010	24.1	0.020	18.0	0.020	67.6	0.025	69.5
Total identified and/or characterized			0.032	75.5	0.034	79.6	0.032 ^a	80.0 ^a	0.092 ^b	82.4 ^b	0.027	90.9	0.031	86.0
Final residue	0.005		0.006	14.5	0.006	14.7	0.009	23.4	0.017	15.6	0.003	9.1	0.004	9.8
Grand total	0.011	100.0	0.038	90.0	0.040	94.3	0.041	103.6	0.109	98.0	0.030	99.9	0.035	95.8

^a For wheat straw (31 DAT), the value included residue concentration of pendimethalin (contaminant), detected at 0.005 mg/kg (12.1% TRR).

^b For wheat straw (119 DAT), the value included residue concentration of pendimethalin (contaminant), detected at 0.035 mg/kg (31.7% TRR).

Table 36 Identified or characterized residues in rotational crops (wheat forage, hay and straw)—pyridine label (2013/1085609)

Designation	Wheat forage 29 DAT		Wheat hay 29 DAT		Wheat hay 123 DAT		Wheat hay 365 DAT		Wheat straw 29 DAT		Wheat straw 123 DAT		Wheat straw 365 DAT	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Imazamox			0.007	9.1	0.001	4.0			0.002	2.1				
CL 263284			0.002	2.3							0.001	5.5		
Total characterized from ERR	0.005	41.7	0.021	28.9	0.006	28.2	0.027	53.7	0.038	46.8	0.007	28.3	0.013	39.6
Total characterized from PES			0.024	31.9	0.006	29.8	0.010	20.7	0.012	14.5	0.005	20.3	0.007	21.5
Total identified and/or characterized			0.055	74.3 ^a	0.013	62.0	0.038	74.4	0.059 ^a	73.5 ²	0.013	54.2	0.020	61.1
Final residue	0.006	55.0	0.014	18.7	0.007	31.8	0.007	14.6	0.014	17.7	0.009	36.7	0.012	35.7
Grand total	0.011	96.7	0.069	93.0 ^a	0.019	93.8	0.045	89.0	0.073 ^a	91.2 ^a	0.022	90.9	0.032	96.9

^a Residue value (0.002 mg/kg, 2.2% TRR) of contaminant pendimethalin was included.

Table 37 Identified or characterized residues in rotational crop (wheat grain)—pyridine label (2013/1085609)

Designation	Wheat grain 29 DAT		Wheat grain 123 DAT	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Imazamox	0.005	10.1	0.001	2.7
CL 263284	0.001	1.4		
Total characterized from extracts	0.004	8.5	0.003	15.0
Total characterized from PES	0.026	50.7	0.010	52.7
Total identified and/or characterized	0.039	77.6	0.013	70.5
Final residue	0.005	10.6	0.002	13.3
Grand total	0.045 ^a	88.2 ^a	0.016 ^b	83.8 ^b

^a Loss of 8.8% TRR during ammonia solubilisation for solid residue was not significant due to low amount of radioactive residues (0.004 mg/kg).

^b Loss of 16.7% TRR during ammonia solubilisation for solid residue was not significant due to low amount of radioactive residues (0.003 mg/kg).

Methanol and water extracts of rotational crop matrices were prepared within periods of 12 to 243 days after sampling for the imidazolinone label and 223 to 463 days after sampling for the pyridine label. For partition phases of the methanol extract, concentrated water extracts and supernatants from water extract incubations (after acetone precipitation and protease treatment, HPLC analysis was conducted within 28 to 458 days after extraction for imidazolinone label and 7 to 329 days after extraction for pyridine label.

With spring wheat grain (imidazolinone label PBI 31 DAT and pyridine label PBI 29 DAT samples), storage stability tests were performed for the water phase of the methanol extract.

PBI 31 DAT wheat grain sample was extracted at 176 days after sampling. The stored water phase of the methanol extract was analysed by HPLC at 220 days after extraction and re-analysed at 647 days (14 months) after extraction. For PBI 29 DAT wheat grain sample, extraction was conducted at 387 days after sampling. The water phase of the methanol extract was analysed by HPLC at 54 days after extraction and re-analysed at 344 days (9 months) after extraction. As results, water phase of the methanol extract was stable for at least 14 months.

PBI 31 DAT and 29 DAT wheat grain samples were re-extracted at 817 days (2.2 years) after sampling and 729 days (2 years) after sampling, respectively. Similar amounts of radioactive residues were extracted compared to the initial extractions (176 days after sampling for PBI 31 DAT and 387 days after sampling for PBI 29 DAT). Further, HPLC analysis for the water phase of the methanol extract showed similar HPLC patterns. Thus, residues in wheat grain frozen stored were stable for about 2 years. From these results, residues in wheat grain over the period of the study were shown to be stable.

In summary, the confined rotational crop studies (radish, lettuce, spinach, maize and wheat) showed that imazamox-derived residues from a previous use or soil treatment do not accumulate in rotational crops; only low levels of radioactive residues were observed in all rotation crop matrices at all analysed plant back intervals.

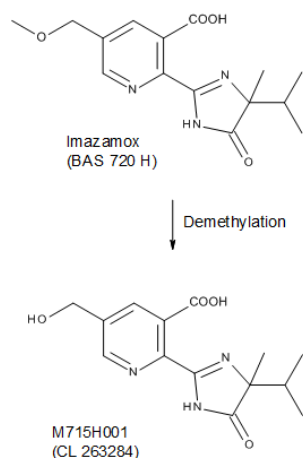


Figure 3 Proposed metabolic pathway of imazamox in rotational crops

Field rotational studies

No data submitted.

RESIDUE ANALYSIS

The Meeting received descriptions and validation data for analytical methods for imazamox and its metabolites in plant or animal matrices.

Good linearity was observed in the standard solutions with linear correlations with coefficients > 0.99. Overall, recovery values were within an acceptable level (70–120%), where the

relative standard deviations were mostly less than 20%. A brief description of the methods is given below.

Table 38 Summary of analytical methods used for the determination of imazamox and its metabolites in various matrices

Method No.	Matrix	Analyte(s)	Detection	LOQ (mg/kg)	Reference
L0188/01	Grapes Green beans Peas (dry) Rice (grain) Sunflower (seed) Rice (straw) Rice (whole plant)	Imazamox CL 263284 CL 189215 CL 312622	LC-MS/MS	0.01	2012/1294678 (validation) 2013/1177533 (validation) 2013/1249356 (ILV)
CEM-236/001	Peas French beans (pods) French beans (whole plant) Maize (grain) Maize (ears, whole plant)	Imazamox CL 263284	HPLC-UV	0.05	ID-244-018 (validation) ID-244-015 (validation) ID-244-016 (validation) ID-244-017 (validation)
M 3076	Oilseed rape (seed)	Imazamox	CE	0.05	ID-244-020 (ILV)
M 2460	Peas (whole pod) Peas (dry seed) Soya bean (seed) Oilseed rape (seed) Peas (vine, hay, straw) Soya bean (forage, straw)	Imazamox CL 263284 combined imazamox + CL 263284	GC-NPD	0.05	ID-244-005 (ILV)
M 3519	Lentil (seed) Lentil (forage)	Imazamox CL 263284 CL 189215 CL 312622	LC-MS, LC-MS/MS	0.05	2004/5000274 (validation) 2002/5004302 (ILV)
M 3515	Orange Grapes Peas Maize (grain) Wheat (grain) In ILV, grapes, maize (grain), and wheat (grain)	Imazamox CL 263284	LC-MS	0.05 for orange, grapes, peas and maize (grain) 0.01 for wheat (grain)	2002/7006089 (ID-244-029) (validation) 2002/5002749 (ILV)
M 3178	Alfalfa (forage + hay) Alfalfa (seed)	Imazamox CL 263284 CL 189215 CL 312622	LC-MS LC-MS/MS	0.1	ID-244-022 (ILV)
M 3098	Wheat (grain) Wheat (straw + hay) Wheat (forage)	Imazamox CL 263284	CE-UV	0.05	ID-244-026 (ILV)
M 2248, M 2248.01	Soya bean (seed) Peanut (nut) Soya bean (foliage) Peanut (foliage) In ILV, soya bean (seed) only	Imazamox	HPLC-UV	0.05	<u>M 2248</u> ID-244-002 (validation) <u>M 2248.01</u> ID-244-001 (validation) ID-244-007 (validation) ID-244-009 (validation) ID-244-008 (validation) ID-244-010 (validation) ID-244-004 (ILV of M 2248.01)
M 2333 (confirmatory method for M 2248)	Soya bean (seed)	Imazamox	LC-MS	0.05	ID-244-003 (validation)
ABC Report 46324	Sunflower (seed) Sunflower (refined oil) Sunflower (meal)	Imazamox CL 263284 CL 312622	LC-MS	0.05	2002/5004111 (validation)
SOP-PA.0288	Sunflower (seed)	Imazamox CL 263284 CL 189215	LC-MS/MS	0.01	2010/1090461 (validation) 2010/1057139 (validation)

Method No.	Matrix	Analyte(s)	Detection	LOQ (mg/kg)	Reference
D0303	Bovine muscle Bovine fat Bovine kidney + liver Bovine milk Poultry egg (validated only in reference 2013/7002842)	Imazamox CL 263284	LC-MS/MS	0.01	2003/5000116 (validation) 2013/7002842 (validation)

Analytical methods for plant matrices

L0188/01

Imazamox and its metabolites CL 312622, CL 263284 and CL 189215 were extracted from plant matrices (grapes, green beans, dry peas, rice grain, sunflower seed, rice straw, rice whole plant) with methanol/water/1 N HCl (60:39:1, v/v/v). In case of high protein samples (e.g. peas and beans), ammonium sulphate was added to the extract. A portion of the extract was centrifuged for 5 min at 4000 rpm, filtered through a disposable syringe filter and an aliquot of the filtrate was diluted to obtain the final volume for LC-MS/MS determination against matrix matched standards. In all matrices tested the mean recovery values were between 80% and 108% and the RSDs were below 12% except one value (23% in 0.01 mg/kg fortified sunflower seed). The LOQ for each analyte was 0.01 mg/kg in all matrices tested [Lehmann 2013a, 2012/1294678; Lehmann 2013b, 2013/1177533].

Table 39 Recovery results of imazamox, CL 312622, CL 263284 and CL 189215 from plant matrices using the method L0188/01

Test substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)		RSD (%)	
Transition				1:00 AM	2	1:00 AM	2
Imazamox	Rice (whole plant)	0.01	5	101.3	107.4	9.1	6.3
		0.1	5	92.3	91	2.5	2.7
	Rice (grain)	0.01	5	95.4	94.4	1.5	2.5
		0.1	5	94.4	95	1.9	0.9
	Rice (straw)	0.01	5	102.5	100.3	6.5	7.8
		0.1	5	87.2	86.9	4	4.2
	Green beans	0.01	5	96.9	95	1.8	1.6
		0.1	5	92.8	92.6	0.5	1.3
	Sunflower (seeds)	0.01	5	95.4	94.8	2.5	4.3
		0.1	5	91.1	92.3	2.8	3.4
	Peas (dry)	0.01	5	89.1	89.5	11.7	12
		0.1	5	86.8	90.5	4.2	7.4
	Grapes	0.01	5	94.1	93.6	3.1	6.7
		0.1	5	85.7	85.5	4.4	4.3
Transition				1:00 AM	2	1:00 AM	2
CL 312622	Rice (whole plant)	0.01	5	98.7	105.5	9	9.1
		0.1	5	92.2	90.8	1.4	1.7
	Rice (grain)	0.01	5	96.7	93.9	1.6	4.9
		0.1	5	94.8	95.1	2.2	1.6
	Rice (straw)	0.01	5	96	100.8	5.3	7.6
		0.1	5	85.7	84.4	3.1	3.8
	Green beans	0.01	5	93.6	91	2.1	4.6
		0.1	5	91.9	92.5	1.5	1.5
	Sunflower (seeds)	0.01	5	92.6	92	6.9	4.4
		0.1	5	89	87.7	3	3.8
	Peas (dry)	0.01	5	92	92	4.5	10.2
		0.1	5	89.7	92.2	4.9	1.5
	Grapes	0.01	5	94.7	101.9	9.1	6.8
		0.1	5	86.6	88.9	3.9	4.5

Test substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)		RSD (%)	
Transition				1	2:00 AM	1	2:00 AM
CL 263284	Rice (whole plant)	0.01	5	95.1	98.8	9.4	8.8
		0.1	5	94.1	93.8	2.5	2.5
	Rice (grain)	0.01	5	99.8	99.5	4.5	3.9
		0.1	5	97.9	96.9	2.9	2.6
	Rice (straw)	0.01	5	101.4	103.2	8.6	9.2
		0.1	5	89.4	89.2	3.2	1.9
	Green beans	0.01	5	94.1	92.4	4.1	1.8
		0.1	5	91.3	91.2	1.6	1.4
	Sunflower (seeds)	0.01	5	89.4	88.4	2.8	3.7
		0.1	5	85	84.7	2.5	3
	Peas (dry)	0.01	5	93.2	94.8	11.8	4.6
		0.1	5	97.3	95.3	2.2	1.9
	Grapes	0.01	5	97.1	93.4	4.4	3.8
		0.1	5	87.8	87.3	3.2	2.9
Transition				1:00 AM	2	1:00 AM	2
CL 189215	Rice (whole plant)	0.01	5	98.6	98.1	8.5	10
		0.1	5	94.7	93.7	1.1	2.6
	Rice (grain)	0.01	5	103.1	107.7	3.3	7.3
		0.1	5	100	98.9	2.8	2.2
	Rice (straw)	0.01	5	101.8	93.5	8.9	8.5
		0.1	5	91.8	90.3	1.4	2.9
	Green beans	0.01	5	89	90.6	2.2	5.4
		0.1	5	89.1	88.7	3.1	3.4
	Sunflower (seeds)	0.01	5	87.7	80.1	2.1	23.3
		0.1	5	88.3	88	1.8	3.6
	Peas (dry)	0.01	5	92.7	89.9	12.2	14.1
		0.1	5	95.8	95.8	4.7	3
	Grapes	0.01	5	102	94.3	8.2	8.2
		0.1	5	88	91.6	2.9	4.3

Transition 1: imazamox, 306→261 m/z; CL 312622, 306→261 m/z; CL 263284, 292→232m/z; CL 189215, 454→292 m/z

Transition 2: imazamox, 306→193 m/z; CL 312622, 306→69 m/z; CL 263284, 292→247 m/z; CL 189215, 454→86 m/z

^a Proposed for quantification

An independent laboratory validation study (ILV) of method L0188/01 for the determination of imazamox, CL 312622, CL 263284 and CL 189215 in plant matrices (grapes, green beans, dry peas, rice grain, sunflower seed, rice straw and rice whole plant) was conducted. In all matrices tested, the mean recovery values were between 76% and 110% (RSD, < 18%). The LOQ for each analyte was 0.01 mg/kg in all matrices tested [Mewis A., 2013a, 2013/1249356].

Table 40 Recovery results of imazamox, CL 312622, CL 263284 and CL 189215 from plant matrices using the Method L0188/01 (independent laboratory validation study)

Test Substance	Crop	Fortification Level (mg/kg)	No. of Tests	Average Recovery (%)		RSD (%)	
Transition				1:00 AM	2	1:00 AM	2
Imazamox	Rice (whole plant)	0.01	5	106	102	8	4
		0.1	5	78	78	12	12
	Rice (grain)	0.01	5	101	97	3	2
		0.1	5	85	84	6	6
	Rice (straw)	0.01	5	99	102	3	1
		0.1	5	86	85	16	16
	Green beans	0.01	5	106	106	7	4
		0.1	5	86	86	8	8

Test Substance	Crop	Fortification Level (mg/kg)	No. of Tests	Average Recovery (%)		RSD (%)	
	Sunflower (seeds)	0.01	5	97	93	5	6
		0.1	5	85	83	6	7
	Peas (dry)	0.01	5	100	99	3	5
		0.1	5	83	84	4	3
Grapes	0.01	5	108	106	6	4	
	0.1	5	92	90	7	7	
Transition				1:00 AM	2	1:00 AM	2
CL 312622	Rice (whole plant)	0.01	5	106	96	6	15
		0.1	5	80	77	11	14
	Rice (grain)	0.01	5	103	101	4	3
		0.1	5	84	88	5	3
	Rice (straw)	0.01	5	103	101	4	3
		0.1	5	82	81	16	16
	Green beans	0.01	5	106	106	5	4
		0.1	5	84	87	7	7
	Sunflower (seeds)	0.01	5	98	94	6	11
		0.1	5	86	83	6	6
	Peas (dry)	0.01	5	102	106	6	3
		0.1	5	83	83	3	4
	Grapes	0.01	5	103	100	7	9
		0.1	5	92	92	6	8
Transition				1:00 AM	2	1:00 AM	2
CL 263284	Rice (whole plant)	0.01	5	109	108	11	6
		0.1	5	80	78	13	11
	Rice (grain)	0.01	5	104	107	3	4
		0.1	5	84	84	8	5
	Rice (straw)	0.01	5	104	101	6	3
		0.1	5	82	81	17	16
	Green beans	0.01	5	102	106	9	8
		0.1	5	83	82	8	8
	Sunflower (seeds)	0.01	5	96	100	11	7
		0.1	5	85	84	8	6
	Peas (dry)	0.01	5	99	106	2	3
		0.1	5	83	84	2	3
	Grapes	0.01	5	108	108	7	5
		0.1	5	94	92	6	7
Transition				1:00 AM	2	1:00 AM	2
CL 189215	Rice (whole plant)	0.01	5	108	94	10	11
		0.1	5	77	76	13	11
	Rice (grain)	0.01	5	104	100	6	9
		0.1	5	86	87	7	10
	Rice (straw)	0.01	5	106	101	8	12
		0.1	5	86	85	15	16
	Green beans	0.01	5	107	107	5	18
		0.1	5	82	82	7	7
	Sunflower (seeds)	0.01	5	99	100	9	11
		0.1	5	81	83	6	5
	Peas (dry)	0.01	5	105	100	2	9
		0.1	5	85	82	4	5
	Grapes	0.01	5	106	110	5	10
		0.1	5	90	89	6	7

Transition 1: imazamox, 306→261 m/z; CL 312622, 306→261 m/z; CL 263284, 292→232m/z; CL 189215, 454→292 m/z

Transition 2: imazamox, 306→193 m/z; CL 312622, 306→69 m/z; CL 263284, 292→247 m/z; CL 189215, 454→86 m/z

^a Proposed for quantification

This method was used in the field residue trials for beans, rice and sunflower seeds, where the procedural recoveries and LOQ levels were acceptable.

CEM-236/001

Imazamox and CL 263284 are extracted from plant matrices (peas, beans pods, whole bean, maize grain, maize ears and maize whole plant) by homogenization with methanol/water/1 M hydrochloric acid (60:39:1, v/v/v). Clean-up of the extract was achieved using liquid/liquid partition and solid phase extraction using a Bond Elut SCX cartridge. Final quantitative determination of residues was by HPLC-UV detection at 254 nm. In all tested matrices, the recovery values were in the range of 72% to 110%. The LOQ for each analyte was 0.01 mg/kg in all matrices tested [Robbins 1996a, ID-244-018; Robbins 1997a, ID-244-015; Robbins 1997b, ID-244-016; Robbins 1997c, ID-244-017].

Table 41 Recovery results of imazamox and CL 263284 from plant matrices using the Method CEM-236/001

Substance	Crop	Fortification level (mg/kg)	No. tests	Range (mg/kg)	Average recovery (%)	RSD (%)
Imazamox	Peas (fresh)	0.05–1.0	5	94–109	101	5.7
	French beans (pods)	0.05–1.0	5	73–99	88	12.5
	French beans (whole plant)	0.05–1.0	5	79–98	90	8
	Maize (grain)	0.05–1.0	5	80–107	93	13.1
	Maize (ears)	0.05–1.0	5	87–104	94	6
	Maize (whole plant)	0.05–1.0	5	82–97	89	6.5
CL 263284	Peas (fresh)	0.05–1.0	5	76–98	89	10.2
	French beans (pods)	0.05–1.0	5	72–82	77	6
	French beans (whole plant)	0.05–1.0	5	72–89	79	10
	Maize (grain)	0.05–1.0	5	91–110	102	6.9
	Maize (ears)	0.05–1.0	5	82–90	86	3.5
	Maize (whole plant)	0.05–1.0	5	75–87	82	5.5

Recovery test was conducted with one test per each of five fortification levels (0.05, 0.10, 0.25, 0.50 and 1.0 mg/kg).

In the field residue trials for beans, peas and rice, this method was used with acceptable procedural recoveries and LOQ levels.

M 3076

An independent laboratory validation study of method M 3076 for the determination of residues of imazamox in canola seed was performed. Residues of imazamox were extracted from oilseed rape seed (canola) with acidic aqueous methanol. Clean-up of the extract was achieved by evaporation, liquid/liquid partition and solid phase extraction using an SCX cartridge. Final quantitative determination of residues was by capillary electrophoresis (CE). The recovery values were in the range of 74% to 85%. The LOQ for imazamox was 0.05 mg/kg in rape seed [Sweeney and Gross 1998a, ID-244-020].

Table 42 Recovery results of imazamox from rape seed using the method M 3076 (independent laboratory validation study)

Crop	Fortification level (mg/kg)	No. of tests	Recovery (%)	Average recovery (%)	RSD (%)
Oilseed rape (seeds)	0.05	2	84, 85	85	
	0.10	2	74, 85	80	
	0.50	2	79, 83	81	

M 2460

Residues of imazamox and CL 263284 are extracted from the plant samples (pea whole pod/dry seed, soya bean seed/forage/straw, rape seed, and pea vine/hay/straw) with acidic methanol-water. The extracts are subjected to suitable clean-up involving solvent partitioning and solid phase extraction. Both compounds are converted to a common methylated product and measured as one chromatographic peak representing a total imazamox-related residue. Measurement of the total imazamox-related residues is accomplished using on-column methylation and GC-NPD. Results were calculated as total imazamox-related residues by the direct comparison of peak heights to those of external standards. In all matrices tested, the recovery values were in the range of 65% to 131%. The LOQ was 0.05 mg/kg for each compound as well as for combined imazamox and CL 263284 in all matrices tested [Safarpour 1995a, ID-244-005].

Table 43 Recovery results of imazamox and CL 263284 from plant matrices using the method M 2460

Test substance	Crop	Fortification level (mg/kg)	No. of Tests	Recovery (%)	Average recovery (%)	RSD (%)
Imazamox + CL 263284	Oilseed rape (seeds)	0.05	2	85, 105	95	
		0.50	2	120, 121	121	
	Pea (vines)	0.05	2	91, 131	111	
		0.50	2	106, 116	111	
	Pea (hay)	0.05	2	90, 91	91	
	Pea (dry seed)	0.05	2	99, 102	101	
	Pea (straw)	0.05	2	65, 103	84	
	Pea (whole pod)	0.05	2	85, 100	93	
	Soya bean (forage)	0.05	2	70, 83	77	
	Soya bean (seed)	0.05	2	82, 84	83	
Imazamox	Soya bean (straw)	0.05	1	98	98	
		0.50	1	105	105	
	Oilseed rape (seeds)	0.05	2	114, 121	118	
	Soya bean (straw)	0.05	2	104, 110	107	
CL 263284	Oilseed rape (seeds)	0.05	2	124, 122	123	
	Soya bean (straw)	0.05	2	91, 94	93	

This method was used in the field residue trials for peas, soya bean and rape seeds, where the procedural recoveries and LOQ levels were acceptable.

M 3519

Imazamox, CL 263284, CL 189215 and CL 312622 were extracted from lentil seed and forage samples using an acidic methanol-water solution. An aliquot of the seed or forage sample extract was diluted with methanol and then passed through an SCX cartridge, which retained the analytes. The SCX cartridge was washed with methanol to remove co-extractives; then the residues were selectively eluted with a water-methanol solution. The eluate was evaporated to dryness and the residues were dissolved in acidic water for analysis. Measurement of the residues was accomplished by LC-MS or LC-MS/MS. In both matrices tested, the mean recovery values were between 75% and 99% (RSD, < 4%). The LOQ for each analyte was 0.05 mg/kg in lentil seed and forage [Nejad 2004a, 2004/5000274].

Table 44 Recovery results of imazamox, CL 263284, CL 189215 and CL 312622 from lentils using the method M 3519

Test substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)	RSD (%)
Imazamox	Lentil (seed)	0.05	5	93	4
		0.5	5	95	2
	Lentil (forage)	0.05	5	94	2
		0.5	5	92	1
CL 263284	Lentil (seed)	0.05	5	98	4
		0.5	5	95	2
	Lentil (forage)	0.05	5	94	2
		0.5	5	93	1
CL 189215	Lentil (seed)	0.05	5	89	3
		0.5	5	99	2
	Lentil (forage)	0.05	5	92	3
		0.5	5	96	3
CL 312622	Lentil (seed)	0.05	5	86	4
		0.5	5	75	2
	Lentil (forage)	0.05	5	95	4
		0.5	5	87	3

An independent laboratory validation of method M 3519 for the determination of imazamox, CL 263284, CL 189215 and CL 312622 residues in lentils (seed and forage) was performed. In both matrices tested, the mean recovery values were between 84% and 109% (RSD, < 19%). The LOQ for each analyte was 0.05 mg/kg in lentil seed and forage [Jordan J.M., 2003a, 2002/5004302].

Table 45 Recovery results of imazamox, CL 263284, CL 189215 and CL 312622 from lentils using the method M 3519 (independent laboratory validation study)

Test substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)	RSD (%)
Imazamox	Lentil (seed)	0.05	5	95	16
		0.50	5	87	7
	Lentil (forage)	0.05	5	84	15
		0.50	5	88	5
CL 263284	Lentil (seed)	0.05	5	109	19
		0.50	5	99	4
	Lentil (forage)	0.05	5	100	9
		0.50	5	87	14
CL 189215	Lentil (seed)	0.05	5	86	7
		0.50	5	93	7
	Lentil (forage)	0.05	5	84	2
		0.50	5	89	6
CL 312622	Lentil (seed)	0.05	5	86	10
		0.50	5	88	6
	Lentil (forage)	0.05	5	85	11
		0.50	5	87	13

This method was used in the field residue trials for lentil, rape seeds and sunflower seeds, where the procedural recoveries and LOQ levels were acceptable.

Homogenised subsamples of straw and seed were additionally extracted according to method M 3519, QuEChERS method and DFG S19 method. For straw samples the extractability with method No. M 3519 (80.2% TRR) and QuEChERS method (64.8% TRR) was comparable to the extractability with methanol and water (77.6% TRR). For seed, method M 3519 extracted about one half (31.0% TRR) and QuEChERS method one third (17.2% TRR) of the radioactive residues extracted with methanol and water (58.3% TRR). For both matrices the DFG S19 method extracted the lowest amounts of radioactive residues with 16.9% TRR for straw and 8.0% TRR for seed. HPLC analyses of the straw and seed extracts obtained from extraction with method M 3519, QuEChERS and DFG S19

method (for straw only) showed a similar metabolite pattern compared to methanol extracts used for metabolism investigation, with CL 263284 as the main component. This data demonstrates that among the tested residues methods method M 3519 has the capability to extract the relevant residues of imazamox.

M 3515

Imazamox and CL 263284 were extracted from plant matrices (orange, grape, pea, maize grain and wheat grain) using an acidic methanol-water solution. For wheat grain, an aliquot of the sample extract was evaporated to dryness, the residue dissolved in acidic water and then passed through a C-18 cartridge, which retained the analytes. The cartridge was washed with water to remove co-extractives; then the residues were downloaded with methanol onto an SCX cartridge, which retained the analytes. For all other plant matrices, an aliquot of the sample extract was diluted with methanol then passed through an SCX cartridge, which retained the analytes. For all plant matrix samples, the SCX cartridge was washed with methanol to remove co-extractives then the residues were eluted with a water-methanol solution. The eluate was evaporated to dryness and the residues were dissolved in water for analysis. Measurement of the residues was accomplished by LC-MS. In all matrices tested, the mean recovery values were between 83% and 109% (RSD, < 12%). The LOQ for each analyte was 0.01 mg/kg in wheat grain and 0.05 mg/kg in all other matrices [Fletcher 2002a, ID-244-029].

Table 46 Recovery results of imazamox and CL 263284 from plant matrices using the method M 3515

Test substance	Crop	Fortification level (mg/kg)	No. of Tests	Average recovery (%)	RSD (%)
imazamox	Peas	0.05	5	94	6.5
		0.5	5	96	3.8
	Grapes	0.05	5	94	5.4
		0.5	5	88	2.0
	Orange	0.05	5	97	2.5
		0.5	5	89	3.7
	Maize (grain)	0.05	5	89	2.7
		0.5	5	91	9.1
	Wheat (grain)	0.01	5	83	3.1
		0.1	5	77	6.3
L 263284	Peas	0.05	5	95	3.4
		0.5	5	91	6.1
	Grapes	0.05	5	95	4.9
		0.5	5	86	3.1
	Orange	0.05	5	106	2.5
		0.5	5	90	3.6
	Maize (grain)	0.05	5	109	3.5
		0.5	5	94	11.6
	Wheat (grain)	0.01	5	90	6.7
		0.1	5	71	4.5

An independent laboratory validation study of method M 3515 for the determination of imazamox and CL 263284 residues in plant matrices (grape, maize grain and wheat grain) was performed. In all matrices tested, the mean recovery values were between 77% and 97% (RSD, < 19%). The LOQ for each analyte was 0.01 mg/kg in wheat grain and 0.05 mg/kg in grape and maize grain [Malinsky 2002a, 2002/5002749].

Table 47 Recovery results of imazamox and CL 263284 from plant matrices using the method M 3515 (Independent laboratory validation study)

Test Substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)	RSD (%)
Imazamox	Grapes	0.05	5	86	9
		0.50	5	89	6
	Maize (grain)	0.05	5	93	4

Test Substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)	RSD (%)
CL 263284	Wheat (grain)	0.50	5	91	3
		0.01	5	97	2
		0.10	5	90	6
	Grapes	0.05	5	78	10
		0.50	5	79	5
	Maize (grain)	0.05	5	94	4
		0.50	5	84	4
	Wheat (grain)	0.01	5	88, 96 ^a	19, 5 ^a
		0.10	5	77	11
		overall	9	85	14

^a When one value of 55% was excluded, the average (n = 4) and the RSD

In the field residue trials for lentil and sunflower seeds, this method was used with acceptable procedural recoveries and LOQ levels.

M 3178

An independent laboratory validation study of the method M 3178 was conducted. Imazamox, CL 263284, CL 189215 and CL 312622 in alfalfa (forage, hay and seeds) were extracted from the samples with acidic water-methanol followed by filtration. For imazamox, CL 263284 and CL 189215, the extracts were cleaned up with precipitation, centrifugation and solid phase extraction (SPE) techniques using RP-102 and SCX columns. For CL 312622, the extracts were cleaned up by filtration with an ISOLUTE filtration column and SPE (C₁₈, QMA and SCX columns). Measurements of the residues were accomplished by reverse-phase HPLC with mass spectrometric detection (LC-MS) and monitoring product ions (LC-MS/MS). LC-MS/MS was used for the analysis of imazamox, CL 263284, CL 189215 and CL 312622 in alfalfa seed samples. The recovery values were between 68% and 109%, except for alfalfa seed at 10 mg/kg fortification level (59–75%). The LOQ for each analyte was 0.1 mg/kg in the all alfalfa matrices [Wickremesinhe and Safarpour 1998a, ID-244-022].

Table 48 Recovery results of imazamox, CL 263284, CL 189215 and CL 312622 from alfalfa using the method M 3178 (independent laboratory validation study)

Test Substance	Crop	Fortification level (mg/kg)	No. of tests	Recovery range (%)	Average recovery (%)	RSD (%)
Imazamox	Alfalfa (forage)	0.1–10	8	73–101	85	12
	Alfalfa (hay)	0.1–10	8	82–102	92	7.5
	Alfalfa (seeds)	0.1–10	8	59 ^a , 84–88	83	12
CL 263284	Alfalfa (forage)	0.1–10	8	80–102	87	11
	Alfalfa (hay)	0.1–10	8	76–88	84	4.9
	Alfalfa (seeds)	0.1–10	8	68 ^a , 84–109	91	13
CL 189215	Alfalfa (forage)	0.1–10	8	74–96	82	11
	Alfalfa (hay)	0.1–10	8	68, 68, 71–82	76	7.9
	Alfalfa (seeds)	0.1–10	8	59 ^a , 70–96	75	13
CL 312622	Alfalfa (forage)	0.1–10	8	77–91	86	5.6
	Alfalfa (hay)	0.1–10	8	72–84	80	5.0
	Alfalfa (seeds)	0.1–10	8	75 ^a –85	78	4.0

Recovery test was conducted with two tests per each of four fortification levels (0.1, 0.2, 0.5 and 10 mg/kg).

^a At a fortification level of 10 mg/kg

This method was used in the residue trials for soya bean and alfalfa with acceptable procedural recoveries and LOQ levels.

M 3098

An independent laboratory validation study of the method M 3098 for the determination of residues of CL 299263 and CL 263284 in wheat hay, forage, straw and grain was conducted. Imazamox and CL 263284 were extracted from wheat forage, hay, straw and grain samples with acidic water-

methanol. Samples were further purified using solid phase extraction (SPE) and liquid-liquid partitioning techniques. Measurement of imazamox and CL 263284 residues was accomplished by capillary electrophoresis (CE) using a high sensitivity- flow cell and a ultra-violet absorbance detector set at 240 nm. In all matrices tested, the recovery values were between 69% and 97%. The LOQ of each analyte was 0.05 mg/kg in wheat matrices [Xu and Nejad 1999a, ID-244-026].

Table 49 Recovery results of imazamox and CL 263284 from wheat using the method M 3098 (independent laboratory validation study)

Test Substance	Crop	Fortification level (mg/kg)	No. of tests	Range of recovery (%)	Average recovery (%)	RSD (%)
Imazamox	Wheat (forage)	0.05, 0.50	4	87–93	90	2.8
	Wheat (hay)	0.05, 0.50	4	75–89	82	7.8
	Wheat (straw)	0.05, 0.50	4	69–89	80	12
	Wheat (grain)	0.05, 0.50	4	72–87	82	8.8
CL 263284	Wheat (forage)	0.05, 0.50	4	85–88	85	2.5
	Wheat (hay)	0.05, 0.50	4	71–97	79	9.8
	Wheat (straw)	0.05, 0.50	4	74–88	81	7.5
	Wheat (grain)	0.05, 0.50	4	72–83	79	6.5

Recovery test was conducted with two tests per each of two fortification levels.

This method was used in residue trials for wheat with acceptable procedural recoveries and LOQ levels.

M 2248, M 2333 (Confirmatory method for M 2248)

M 2248: Imazamox (CL 299263) was extracted from soya bean seeds with acidic aqueous methanol using a Polytron ultrasonic extractor. The extract was cleaned up by liquid/liquid partitioning with methylene chloride, gel permeation chromatography using a Biobead S-X3 column and solid phase extraction using an SCX column. Measurement of the residue was performed by HPLC-UV at 254 nm (HPLC-UV). The mean recovery values were between 82% and 88% (RSD, < 11%). The LOQ was 0.05 mg/kg for all matrices tested [Witkonton 1994b, 1994/7003428; Witkonton 1993a, ID-244-002].

Soya bean seed samples showing positive (> 0.05 mg/kg) residues of imazamox by method M 2248 above were confirmed by LC-MS (method M 2333). The two same extracts prepared in method M 2248 were directly amenable to analysis by LC-MS. The (M+H)⁺ ion of imazamox at m/z 306⁺ was monitored for confirmation [Witkonton *et al.*, 1994a, ID-244-003]. The recovery values were 76% and 79% and the LOQ was 0.05 mg/kg.

Table 50 Recovery results of imazamox from soya bean using the method M 2248 and M 2333

Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)	RSD (%)	Reference
Soya bean (seeds)	0.05	3	88	6.8	ID-244-002 M 2248
	0.10	3	82	11	
	0.50	3	86	10	
	0.05	2	78 (mean of 76 and 79)		ID-244-003 M 2333

M 2248.01

Imazamox (CL 299263) was extracted from the samples (soya bean seed/foilage and peanut/peanut foliage) with acidic aqueous methanol using a Polytron ultrasonic extractor. The extract was cleaned up by liquid/liquid partitioning with methylene chloride and solid phase extraction using an SCX column. Measurement of the residue was performed by HPLC-UV at 254 nm. There was a major modification to the method M 2248.01: exclusion of a sample clean up procedure by gel-permeation chromatography (GPC). The mean recovery values were between 81–115%. The LOQ was

0.05 mg/kg for each matrix [[Minoura and Ohba 1996a, ID-244-007; Minoura and Ohba 1996b, ID-244-008; Minoura and Ohba 1996c, ID-244-009; Minoura and Ohba 1996d, ID-244-010].

Table 51 Recovery results of imazamox from soya bean using the method M 2248.01

Crop	Fortification level (mg/kg)	No. of tests	Range of recovery (%)	Average recovery (%)	RSD (%)	Reference
Soya bean (seeds)	0.05, 0.2	4	100.3–112.4	105.0	5.0	ID-244-007
Soya bean (foliage)	0.05, 0.1	4	87.2–115.4	101.4	11	ID-244-008
Peanut (nut in shell)	0.05, 0.2	4	90.0–109.9	101.8	9.5	ID-244-009
Peanut (foliage)	0.05, 0.1	4	80.6–104.7	94.9	12	ID-244-010

Recovery test was conducted with two tests per each of two fortification levels

An independent laboratory validation study of method M 2248.01 for the determination of imazamox residues in soya bean seed was performed. GPC clean up procedure was included in this study. The recovery values were 75% between 90% and the LOQ (0.05 mg/kg) for soya bean seed was confirmed [Witkonton 1994a, ID-244-004].

Table 66 Recovery results of imazamox from soya bean seed using the method M 2248.01 (independent laboratory validation study)

Crop	Fortification level (mg/kg)	No. of tests	Recovery (%)
Soya bean (seeds)	0.05	2	75, 81
	0.10	2	76, 86
	0.40	2	88, 90

In the field residue trials for peanut, this method was used with acceptable procedural recoveries and LOQ levels.

ABC Report 46324

Imazamox and metabolites CL 263284 and CL 312622 were extracted from sunflower seed and meal samples with an acidified methanol/water solution, filtered and concentrated. The residues were purified on a C₁₈ SPE column eluted with methanol in aqueous ammonium acetate. Residues in refined oil samples were diluted in hexane and extracted by shaking with acidified acetonitrile/water solution. Following phase separation, the lower aqueous acetonitrile layer was collected. The final chromatography analysis of imazamox residues was performed using LC-MS. The mean recovery values were between 75–109% (RSD, < 13%). The LOQ for each analyte was 0.05 mg/kg in sunflower matrices [Johnston 2003a, 2002/500411].

Table 52 Recovery results of imazamox, CL 263284 and CL 312622 from sunflower using the method ABC Report 46324

Test Substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)	RSD (%)
Imazamox	Sunflower (seed)	0.05	3	92	3.3
		0.1	3	90.1	0.8
		0.5	3	92.7	0.5
	Sunflower (refined oil)	0.05	3	94.5	0.7
		0.1	3	96.5	0.8
		0.5	3	107	0.5
	Sunflower (meal)	0.05	3	102	5.1
		0.1	3	87.7	1.9
		0.5	3	79.8	3.5
CL 263284	Sunflower (seed)	0.05	3	90.7	6
		0.1	3	87.9	1.4
		0.5	3	92	0.3
	Sunflower (refined oil)	0.05	3	97.3	0.9
		0.1	3	96.8	0.3

Test Substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)	RSD (%)
	Sunflower (meal)	0.5	3	108	1.1
		0.05	3	89.8	5.9
		0.1	3	82	2.9
		0.5	3	79.6	3.7
CL 312622	Sunflower (seed)	0.05	3	95.4	8.9
		0.1	3	77.8	2.3
		0.5	3	74.6	0.7
	Sunflower (refined oil)	0.05	3	93.6	1.6
		0.1	3	97.3	0.5
		0.5	3	109	0.9
	Sunflower (meal)	0.05	3	88.2	10
		0.1	3	81.2	1
		0.5	3	75.3	5.3

SOP-PA.0288

Imazamox and its metabolites CL 263284 and CL 189215 were extracted from sunflower samples using extraction solution methanol/water/1 M HCl (60:39:1, v/v/v). Final determination was performed using LC-MS/MS determination. The mean recovery values were between 76% and 106% (RSD, < 15%). The LOQ for each analyte was 0.01 mg/kg in sunflower seed [Freitas 2010a, 2010/1057139; Leite 2008a, 2010/1090461]

Table 53 Recovery results of imazamox, CL 263284 and CL 189215 from sunflower using the method SOP-PA.0288

Test substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)		RSD (%)	
Transition				1	2	1	2
Imazamox	Sunflower (seeds)	0.01	6	85	76	8	5
		1.0	5	100	103	4	6
Transition				1	2	1	2
CL 263284	Sunflower (seeds)	0.01	5	82	82	7	9
		1.0	6	106	101	6	8
Transition				1	2	1	2
CL 189215	Sunflower (seeds)	0.01	6	98	94	15	11
		1.0	6	105	106	5	6

Transition 1 (for quantification): imazamox, 306→246 m/z; CL 263284, 292→247m/z; CL 189215, 454→179 m/z

Transition 2 (for confirmation): imazamox, 306→193 m/z; CL 263284, 292→179 m/z; CL 189215, 454→292 m/z

Analytical methods for animal matrices

D0303

Imazamox and CL 263284 were extracted from bovine tissues (liver, kidney and muscle) with acidic methanol-water solution. Following centrifugation, an aliquot of the extract was diluted with methanol and then passed through an SCX cartridge, which retained the analytes. The SCX cartridge was washed with methanol to remove co-extractives; then the residues were selectively eluted with a water-methanol solution. The eluate was evaporated to dryness and the residues were dissolved in water for analysis. Residues from milk and fat were extracted with acetonitrile in hexane. Following solvent partitioning, an aliquot of the acetonitrile extract was diluted with methanol and passed through the SCX cartridge using the same procedure as described for the tissue samples. The final detection was accomplished by LC-MS/MS. In all matrices tested, the mean recovery values were

between 69% and 121% (RSD, 25%). The LOQ for each analyte was 0.01 mg/kg for bovine matrices [Stewart 2003a, 2003/5000116; Gooding 2013a, 2013/7002842].

Table 54 Recovery results of imazamox and CL 263284 from bovine matrices using the method D0303

Test substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery ^a (%)	RSD (%)
Imazamox	Liver	0.01	5	92	25
		0.10	5	93	9
	Kidney	0.01	4	82	7
		0.10	5	73	7
	Muscle	0.01	5	81	6
		0.10	5	87	7
	Fat	0.01	5	95	9
		0.10	4	69	7
	Milk	0.01	5	103	17
		0.10	5	86	8
CL 263284	Liver	0.01	5	116	16
		0.10	5	102	10
	Kidney	0.01	4	106	10
		0.10	5	78	8
	Muscle	0.01	5	98	17
		0.10	5	83	17
	Fat	0.01	5	121 (110, 114, 116, 122 and 144%)	12
		0.10	4	79	19
	Milk	0.01	5	93	6
		0.10	5	93	4

^a Recoveries have been corrected for mean recoveries in control samples. The detected residue concentrations in two control samples were: for imazamox, 0.004 and 0.005 mg/kg in liver, 0.005 and 0.003 mg/kg in kidney, 0.004 and 0.006 mg/kg in muscle, 0.004 and 0.003 mg/kg in fat, and 0.004 and 0.000 mg/kg in milk; for CL 263284, 0.000 and 0.003 mg/kg in liver.

Another validation study for the method D0303 was performed for bovine matrices and egg commodity. In all matrices tested, the mean recovery values were between 70% and 111% (RSD, < 22%).

Table 55 Recovery results of imazamox and CL 263284 from bovine matrices and egg using the method D0303

Test Substance	Crop	Fortification level (mg/kg)	No. of Tests	Recovery range (%)	Average recovery (%)	RSD (%)	No. of tests	Recovery range (%)	Average recovery (%)	RSD (%)
Transition				1				2		
Imazamox	Muscle	0.01	5	71–87	79	9	5	67, 68, 70–84	72	10
		0.1	5	77–84	81	4	5	75–78	77	1
	Kidney	0.01	5	73–90	78	9	5	71–82	76	6
		0.1	5	75–82	79	4	5	70–85	77	9
	Liver	0.01	5	65, 90–107	90	17	5	62, 77–93	78	14
		0.1	5	71–106	89	15	5	68, 81–94	85	13
	Fat	0.01	5	100–117, 126	110	10	5	89–108, 125	105	13
		0.1	5	88–94	89	5	5	82–94	89	5
	Milk	0.01	5	89–109, 126	107	12	5	92–120	106	13
		0.1	5	81–120	93	17	5	84–105	93	9
	Egg	0.01	5	101–111	107	4	5	97–111	104	5
		0.1	5	83–96	89	6	5	79–99	87	9
Transition				1				2		
CL 263284	Muscle	0.01	5	69, 72–86	77	9	5	71–84	80	7

Test Substance	Crop	Fortification level (mg/kg)	No. of Tests	Recovery range (%)	Average recovery (%)	RSD (%)	No. of tests	Recovery range (%)	Average recovery (%)	RSD (%)
	Kidney	0.1	5	77–88	82	6	5	75–79	77	2
		0.01	5	72–82	76	6	5	71–84	77	7
		0.1	5	80–82	81	2	5	79–85	81	3
	Liver	0.01	5	63, 75–103	80	18	5	54, 66, 66, 68, 95	70	22
		0.1	5	73–103	92	13	5	95–101	90	14
	Fat	0.01	5	91–103	96	6	5	89–108	97	7
		0.1	5	84–91	87	3	5	83–90	86	3
	Milk	0.01	5	96–118	108	10	5	101–115, 134	111	12
		0.1	5	72–88	82	8	5	77–97	86	8
	Egg	0.01	5	87–117	100	13	5	92–111, 127	106	13
		0.1	5	93–112	99	9	5	88–115	99	10

Transition 1 used for quantification: imazamox, 306→261 m/z; CL 263284, 292→247m/z

Transition 2 used for confirmation: imazamox, 306→86 m/z; CL 263284, 292→179 m/z

M 2503, M 2463, SOP CEM-236/002, M 2379 and M 3178.01

The methods used for storage stability studies are summarized briefly below.

M 2503: The samples were analysed by method M 2503 which determines the analytes by means of CE-UV. Residues of a soya bean sample aliquot were extracted with acidic water methanol. After filtration of the samples, one aliquot was subjected to solid phase extraction and liquid-liquid partitioning techniques. The final quantitation was performed by CE-UV (240 nm). The method has a limit of quantitation of 0.5 mg/kg for each compound in soya bean.

M 2463: The samples were analysed by Method M 2463 which determines the analytes by means of CE-UV. The validated LOQ of the method is 0.1 mg/kg per compound. Residues of the test compounds were extracted with acidic water-methanol followed by clean-up steps which included precipitation, centrifugation and solid-phase extraction techniques. Measurement of CL 263284 was accomplished by capillary electrophoresis (CE) equipped with a high sensitivity flow cell and a UV detector set at 240 nm.

SOP CEM-236/002: The samples were analysed by CEMAS SOP CEM-236/002 which determines the analytes by means of HPLC-UV. Residues of imazamox and CL 263284 were extracted by maceration with extraction solution (methanol/water/1 M hydrochloric acid). After filtration, extracts were re-suspended in methanol and hydrochloric acid before they were partitioned into dichloromethane. After evaporation to dryness, the residue was reconstituted in methanol and acetonitrile before being extracted into hexane. The hexane layer was evaporated to dryness and the residuum was re-dissolved into 0.2% hydrochloric acid. The extracts were cleaned up by solid phase extraction. The quantitation of residues was performed by HPLC-UV at 254 nm. The method has a limit of quantitation of 0.05 mg/kg for each compound in maize.

M 2379: The samples were analysed by Method M 2379 which determines the analytes by means of CE-UV. Residues of the test compounds were extracted with 1 N HCl/water/methanol solution (1:39:60, v/v/v).

M 3178.01: The samples were analysed by Method M 3178.01 which determines the analytes by means of HPLC-MS. The validated LOQ of the method is 0.1 mg/kg per compound. Residues of the test compounds were extracted with acetone/water/methanol solution (1:1:1, v/v/v) and purified by SPE techniques. Measurement of imazamox, CL 263284, CL 189215 and CL 312622 was accomplished by LC-MS.

RLA 12539, HPLC-MSD and CEM-236

The methods used for determination of incurred residues in supervised field trial samples are summarized briefly below.

RLA 12539 (wheat, ID-730-019): The whole wheat specimens were analysed for residues of imazamox using method RLA 12539 (CE-UV). The validated limit of quantitation was 0.05 mg/kg. The samples were extracted with methanol:water:HCl 1 mol/L (60:39:1, v/v/v). After clean-up by solid phase extraction, the specimen was partitioned with dichloromethane and evaporated to dryness and reconstituted in water. The final determination was performed with CE-UV at 240 nm. Mean recovery of imazamox from fortified untreated wheat whole plant, wheat grain and wheat straw, over the range 0.05 to 1.00 mg/kg, was 87%, 88% and 84%, respectively. Mean recovery of CL 263284 from fortified untreated wheat whole plant, wheat grain and wheat straw, over the range 0.05 to 1.00 mg/kg, was 81%, 77%, and 76%, respectively.

HPLC-MSD method (sunflower, sunflower seed processing, 2002/5004111): Residues of imazamox were extracted from sunflower seed and meal samples with an acidified methanol:water solution, filtered and concentrated. The residues are purified on a C₁₈ SEP column eluted with 25% MeOH in 0.05 M aqueous ammonium acetate and brought to final volume with water. Residues in refined oil samples are diluted in hexane and extracted by shaking with acidified acetonitrile:water solution. Following phase separation, the lower aqueous acetonitrile layer is collected and adjusted to final volume with water. The final analysis of imazamox residues is performed using LC-MSD. At fortification levels of 0.05, 0.1 and 0.5 mg/kg for sunflower seed and 0.05 mg/kg for refined oil and meal, recoveries were in the range of 70.8% and 113% (RSDs, 2–13%).

CEM-236 (alfalfa, ID-731-001): The specimens were analysed for imazamox and its metabolite CL 263284 with method CEM-236 quantifying each relevant analyte with a limit of quantitation of 0.05 mg/kg. For the analysis, alfalfa samples (green matter and hay) were extracted with an acidified methanol/water mixture. After clean-up of the extract by liquid/liquid partition and solid phase extraction residues were determined by UV detection after liquid chromatographic separation (HPLC-UV). In whole plant samples fortified at 0.05 mg/kg with imazamox and CL 263284 each, recovery was 82% and 72%, respectively.

Stability of residues in stored analytical samples

The Meeting received information on freezer storage stability of imazamox and its metabolites in plant commodities. Storage stability study on animal commodity was not submitted. Residues in plant commodities were generally stable for the duration of the studies.

Frozen samples were homogenized and fortified with test compound. The fortified homogenate was stored in deep freezer. After each specified period, a portion of sample was analysed for test compound. The stability results are expressed as average percentage of the nominal fortification and are not corrected for the procedural recoveries. In order to account for possible variations over the time investigated, the mean procedural recovery results are additionally given. The storage conditions, storage periods and percent remaining after each period were summarized in the table below.

Table 56 Stability of imazamox and its metabolites in frozen plant matrices

Matrix	Storage period (month)	Imazamox % remaining	Proc. rec.	CL 263284 % remaining	Proc. rec.	CL 189215 % remaining	Proc. rec.	CL 312622 % remaining	Proc. rec.
Soya bean matrices fortified at 0.1 mg/kg, stored at –20 °C and analysed with Method SOP-PA.0288 [Leite and Alves 2011(a), 2011/1207286]									
Grain	0			107	–	107	–		
	1			101	103	94	108		
	2			92	99	88	106		
	3			104	103	99	107		
	7			75	89	94	103		
	10			111	123	100	124		

Matrix	Storage period (month)	Imazamox % remaining	Proc. rec.	CL 263284 % remaining	Proc. rec.	CL 189215 % remaining	Proc. rec.	CL 312622 % remaining	Proc. rec.
Laminated bean	0			95	—	90			
	1			98	108	86	111		
	2			—	—	—	—		
	3			93	104	99	89		
	7			—	—	—	—		
	10			—	—	—	—		
Defatted meal	0			101	—	92	—		
	1			105	107	85	126		
	2			—	—	—	—		
	3			107	97	97	100		
	7			—	—	—	—		
	10			—	—	—	—		
Toasted defatted meal	0			114	—	98	—		
	1			112	104	88	126		
	2			—	—	—	—		
	3			96	103	111	90		
	7			—	—	—	—		
	10			—	—	—	—		
Oil	0			111	—	106	—		
	1			86	102	83	100		
	2			—	—	—	—		
	3			100	110	93	116		
	7			—	—	—	—		
	10			—	—	—	—		
Soya bean matrices fortified at 0.5 mg/kg, stored at $\leq -15^{\circ}\text{C}$ and analysed with method M 2503 [Bixler and Safarpour 2000(a), ID-720-070]									
Seed	32	119	78	80	74				
	35	134	87	91	79				
	38	124	90	86	85				
	44	124	88	80	79				
Forage	32	91	79	61	74				
	35	109	89	73	74				
	38	105	90	70	81				
	44	100	85	66	77				
Hay	32	92	87	57	75				
	35	112	93	64	79				
	38	106	90	69	83				
	44	107	86	73	84				
Wheat matrices fortified at 0.5 mg/kg, stored at $\leq -15^{\circ}\text{C}$ and analysed with method M 3098 Bibo X. 2002(a), 2002/5004279].									
Grain	0	82	95	71	84				
	3	101	102	83	82				
	6	108	103	97	85				
	12	109	100	89	88				
	18	98	92	81	75				
	24	110	108	98	94				
	37	87	92	88	84				
	48	84	84	68	72				
Straw	0	83	87	70	75				
	3	92	94	78	74				
	6	95	93	81	82				
	12	99	92	79	75				
	18	76	88	62	73				
	24	91	99	81	89				
	37	80	98	84	85				
	48	81	82	61	66				
Hay	0	88)	93	78	80				
	3	95	90	77	78				

Matrix	Storage period (month)	Imazamox % remaining	Proc. rec.	CL 263284 % remaining	Proc. rec.	CL 189215 % remaining	Proc. rec.	CL 312622 % remaining	Proc. rec.
	6	75	91	62	82				
	12	76	98	61	85				
	18	64 (65)	98	57	82				
	24	93	95	76	92				
	37	74	88	70	79				
	48	65 (80)	81	57	75				
Forage	0	80	86	70	77				
	3	90	94	76	71				
	6	90	108	75	85				
	12	88	93	75	83				
	18	79	90	69	82				
	24	88	106	82	91				
	37	91	99	82	95				
	48	72	83	62	68				
Wheat matrices fortified at 0.5 mg/kg, stored at $\leq -10^{\circ}\text{C}$ and analysed with method M 2463 [Nejad 1999(a), IA-730-011].									
Grain	14			91	91	83	90		
	18			102	104	82	98		
	24			112	106	85	103		
Straw	14			92	91	79	78		
	18			93	98	80	89		
	24			92	92	87	77		
Hay	14			82	92	83	92		
	18			75	89	77	95		
	24			84	108	88	93		
Forage	14			79	89	79	85		
	18			97	87	84	95		
	24			70	87	71	90		
Maize matrices fortified at 0.5 mg/kg, stored at $\leq -18^{\circ}\text{C}$ and analysed with method SOP CEM-236/002 [Rawle 2003(a), 2003/1030079]									
Grain	0	86	82	75	72				
	3	85	82	75	71				
	6	80	78	74	76				
	12	71	75	71	71				
	18	87	89	83	90				
	24	83	88	79	92				
Ear	0	86	89	77	75				
	3	84	90	75	88				
	6	95	93	92	93				
	12	69	75	64	74				
	18	79	89	80	88				
	24	86	94	80	90				
Immature plant	0	80	100	75	87				
	3	83	72	77	76				
	6	86	78	81	76				
	12	85	81	80	74				
	18	80	76	77	78				
	24	87	87	80	87				
Rape seed fortified at 0.5 mg/kg, stored at $\leq -18^{\circ}\text{C}$ and analysed with method M 2460 [Bixler and Khunachak 2000 (a), ID-326-022]									
Rape seed	0	74	97						
	3	72	99						
	6	64	87						
	7.5	75	72						
	12	87	108						
	18	67	99						
Peanut matrices fortified at 1 mg/kg, stored at -5 to -25°C and analysed with method M 2379 [Nejad and Xu 2000 (a), IA-740-023]									
Hull	1			87	78	81	78		
	5			122	113	84	79		
	6			86	88	97	96		

Matrix	Storage period (month)	Imazamox % remaining	Proc. rec.	CL 263284 % remaining	Proc. rec.	CL 189215 % remaining	Proc. rec.	CL 312622 % remaining	Proc. rec.
	12			81	84	84	86		
	18			68	85	74	85		
	24			83	93	83	96		
Nutmeat	1			87	98	80	87		
	5			83	92	84	91		
	6			76	85	76	86		
	12			75	93	78	96		
	18			62	84	66	83		
	24			85	81	82	77		
Alfalfa matrices fortified at 1.0 mg/kg, stored at -10 to -35 °C and analysed with method M 3178.01 [Fletcher 2001 (a), ID-326-024]									
Hay	0	79	81	76		69	87	70	68
	1	76	89	76		70	72	65	93
	3	71	94	68		65	78	67	86
	6	70	81	69		60	64	69	83
	12	75	85	82		63	98	79	93
	18	75	92	84		80	91	101	93
Forage	0	59	67	67	82	66	77	81	85
	1	78	85	82	88	80	81	83	94
	3	74	82	75	86	78	82	81	87
	6	78	80	75	74	66	80	77	79
	12	75	81	73	78	68	71	72	84
	18	76	87	78	91	75	75	76	84
Seed	0	58	59	76	78	55	80	71	82
	1	65	72	76	82	61	80	68	79
	3	68	83	79	87	57	81	66	88
	6	74	79	79	87	61	68	75	79
	12	76	90	63	85	59	88	77	91
	18	84	89	74	72	61	63	77	82

Stability of residues in stored analytical samples was investigated for imazamox, CL 263284, CL 189215 and CL 312622.

In acidic methanol water extract and final volume solution (for LC-MS/MS), the four compounds were stable for 3 days and 7 days, respectively in the matrices of rice (whole plant, grain and straw), grapes and sunflower seeds at refrigeration conditions [Lehmann 2013a, 2012/1294678; Lehmann 2013b, 2013/1177533].

Stability of calibration solutions prepared with 1% acetic acid in water was tested at 4 °C, where the four compounds were stable for 27 days [Jordan 2003a, 2002/5004302]. The mixed fortification standard solution in 10% methanol/water was stable up to one month at 4 °C with no more than 8% difference between the response of the one month old and the new fortification solution [Wickremesinha and Safarpour 1998a, ID-244-022].

Imazamox and CL 263284 in methanol or acidified water (calibration solution) were stable for 56 days and 28 days, respectively when stored under refrigeration [Stewart 2003a, 2003/5000116; Gooding 2013a, 2013/7002842]. In the study, extracts of livestock matrices in acidic methanol-water were stable for the time period tested 2 to 6 days.

USE PATTERNS

Imazamox is a systemic herbicide used for the control of grassy and broadleaf weeds. It is formulated as a liquid or granular product either a solo product or in combination with other active substances for use on legume vegetables, pulses, cereal grains, oilseeds and legume animal feeds. It is registered in a number of countries for crops with pre-emergence and post-emergence applications.

The authorized uses relevant to the supervised trials data submitted to the current Meeting and described in English are summarized in the table below.

Table 57 Registered use of imazamox relevant to the residue evaluation by the current Meeting

Crop	Country	Formulation	Application					Remarks
			Growth stage & season (crop)	No.	Water L/ha	Rate, kg ai/ha	PHI, days	
Bean	Brazil	WG 700 g/kg	Post-emergence	1	30–50 100–300	0.028–0.042	43	Add non-ionic surfactant in 0.25% v/v of mixture.
Bean(dry)	Canada	WG 700 g/kg	Early post-emergence BBCH 12–18	1	100	0.020	60	Apply with a nitrogen source and EC surfactant (0.5%, v/v).
Bean (dry)	Canada	SL 20 g/L (bentazone, 429 g/L)	Early post-emergence	1	100	0.020	60	Apply with a nitrogen source. Do not graze the treated crop or cut for hay.
Bean	Chile	WG 700 g/kg	Early post-emergence	1	150–200	0.042–0.056		
Bean (common)	Costa Rica	WG 700 g/kg	Early post-emergence	1	200–300	0.040	43	Add a non-ionic surfactant at 0.25%.
Bean (Winter and spring Faba)	France	EC 16.7 g/L (pendimethalin, 250 g/L)	Pre-emergence (within 5 days after sowing); pre-emergence of the weeds	1 (max every 2 yrs)	100–500	0.075	90	Applied on the soil. Do not roll after treatment.
Bean (Green)	Israel	EC 40 g/L	2–3 leaves			0.024		
Bean (Spring and winter field)	UK	EC 16.7 g/L (pendimethalin, 250 g/L)	Before weed emergence after sowing (pre-emergence of the crop)	1	200–300	0.075		Do not apply once the plumule is less than 13 mm from the soil surface.
Bean (Lima succulent)	USA	SL 120 g/L as free acid (ammonium sulphate)	Early post-emergence (1 st –2 nd trifoliate leaf stage)	1		0.035		A non-ionic surfactant must be added to the spray solution.
Bean, snap	USA	SL 120 g/L as free acid (amm. sulphate)	Post-emergence (at least 1 fully expanded trifoliate leaf and before the bloom stage)	1		0.035		A non-ionic surfactant must be added to the spray solution.
Bean (dry)	USA	SL 120 g/L as free acid (amm. sulfate)	Post-emergence (prior to bloom stage but after dry beans have at least 3 pairs of leaves)			0.035		An adjuvant (a surfactant or a crop oil concentrate) must be added to the spray solution.
Lentil (Clearfield)	Canada	WG 700 g/kg	Early post-emergence (2–6 leaf stage of the crop) BBCH 12–18	1	100	0.015–0.020	60	Apply with EC surfactant (0.5% v/v). Do not graze the treated crop or cut for hay within 20 days of application.
Lentil (Clearfield)	Canada	WG 35% (imazethapyr, 35%)	Early post-emergence (the 2–6 leaf stage of the crop)	1	50–100	0.015	60	Apply with EC surfactant (0.5% v/v). Do not harvest forage or cut hay for forage.

Crop	Country	Formulation	Application					Remarks
			Growth stage & season (crop)	No.	Water L/ha	Rate, kg ai/ha	PHI, days	
Lentil (Clearfield)	Canada	SL 33 g/L (imazapyr, 15 g/L)	Early post-emergence (1–9 node stage) BBCH 12–17	1	50–100	0.020	60	Apply with EC surfactant (0.5% v/v).
Lentil	France	EC 16.7 g/L (pendimethalin, 250 g/L)	Pre-emergence post-sowing of the crop	1	100–500	0.037	63	
Lentil (Clearfield)	USA	SL 120 g/L as free acid (amm. salt)	Post-emergence (2–leaf to prior to flower bud formation)	1		0.035–0.053		A non-ionic surfactant and nitrogen-based fertilizer must be added to the spray solution.
Pea (Field)	AUS	WG 700 g/kg	Early post-emergence	1	min 50	0.032	not req.	WHP: 6 weeks (grazing). Spray adjuvant and liquid ammonium sulfate should be added.
Pea (Succulent shelled peas)	Canada	SL 20 g/L (bentazone, 429 g/L)	Early post-emergence (3 to 6 node stage)	1	100	0.020	40	
Pea (Field)	Canada	WG 350 g/kg (imazethapyr, 35%)	BBCH 11–16	1	50–100	0.015	60	Apply with EC surfactant (0.5% v/v). Field peas treated may be fed to livestock 30 days after application. Do not harvest forage or cut hay for forage.
Pea (Succulent)	Canada	WG 700 g/kg	Early post-emergence (3–6 node stage)	1	100	0.020	40	Must be applied in tank mix combination with bentazone (429 g/ha) and nitrogen source (2 L/ha). Do not graze the treated crop or cut for hay.
Pea (Field)	Canada	WG 700 g/kg	Early post-emergence (3–6 node stage)	1	100	0.020	60	The same with the above
Pea (Field)	Canada	SL 20 g/L (bentazone, 429 g/L)	Early post-emergence (3–6 node stage)	1	100	0.020	60	Apply with a nitrogen source. Do not graze the treated crops or cut for hay.
Pea	Chile	WG 700 g/kg	Early post-emergence	1	150–200	0.042–0.056		–
Pea (canning)	France	EC 16.7 g/L (pendimethalin, 250 g/L)	Pre-emergence (within 3–4 days after sowing)	1 (max every 2 years)	100–500	0.075	63	Applied on the soil. Do not roll after treatment.
Pea (Winter and spring protein)	France	EC 16.7 g/L (pendimethalin, 250 g/L)	Pre-emergence post-sowing (preferred) or post-emergence (2–3 leaf stage of the culture) BBCH 12–13	1 (max every 2 years)	100–500	0.075	63	
Pea (Combining and vining pea)	UK, Ireland	EC 16.7 g/L	Before weed emergence after	1	200–300	0.075		Do not apply once the plumule is less than 13

Crop	Country	Formulation	Application					Remarks
			Growth stage & season (crop)	No.	Water L/ha	Rate, kg ai/ha	PHI, days	
		(pendimethalin, 250 g/L)	sowing					mm from the soil surface.
Pea	Israel	EC 40 g/L	2–3 leaves			0.024		
Pea (English)	USA	SL 120 g/L as free acid (amm. sulfate)	Early post-emergence	1		0.026		A non-ionic surfactant must be added to the spray solution.
Pea (dry) (field pea, southern pea (cow pea))	USA	SL 120 g/L as free acid (amm. sulfate)	Early post-emergence prior to bloom stage but after dry peas have at leaves 3 pairs of leaves	1		0.035		A non-ionic surfactant (or crop oil concentrate) must be added to the spray solution.
Peanut	AUS	WG 700 g/kg	Early post-emergence	1	min 50	0.035		WHP: 4 weeks (grazing) A spray adjuvant and liquid ammonium sulfate should be used. Do not graze or cut for stock food for 4 weeks after application.
Peanut	Israel	EC 40 g/L	3–4 true leaves			0.032		
Peanut	South Africa	SL 120 g/L	Post-emergence	1	200	0.048	50	This must only be used with a non-ionic surfactant (0.2% v/v) and ammonium sulfate solution.
Peanut	Zimbabwe	SL 120 g/L	Early post-emergence	1	200	0.036		Add an adjuvant and ammonium sulfate solution. Do not graze the treated crops or cut for hay within 20 days of application.
Rape (Clearfield winter rape)	UK	SC 17.5 g/L (metazachlor, 375 g/L)	Post-emergence BBCH 10–18	1	100	0.035		EC spreader may be used.
Rape seed (Clearfield canola)	AUS	SL 33 g/L (imazapyr, 15 g/L)	Early post-emergence (2–6 leaf stage) BBCH 12–16	1	Min 70–100	0.0099–0.025		Do not graze or cut for stock food for 5 weeks after application.
Rape (Clearfield canola, Clearfield canola quality <i>Brassica juncea</i>)	Canada	SL 33 g/L (imazapyr, 15 g/L)	Early post-emergence (2–6 leaf stage)	1	50–100	0.020	60	Apply with EC surfactant (0.5% v/v).
Rape (Clearfield canola, Clearfield canola quality <i>Brassica juncea</i>)	Canada	WG 350 g/kg (imazethapyr, 35%)	Early post-emergence BBCH 12–16	1	50–100	0.015	60	Apply with EC surfactant (0.5% v/v). Do not graze treated crops or cut for hay. Do not harvest forage or cut for hay.
Rape (Clearfield canola, Clearfield canola quality <i>Brassica juncea</i>)	Canada	WG 700 g/kg	Early post-emergence BBCH 12–16	1	100	0.020	60	Apply with EC surfactant (0.5% v/v). Do not graze treated crops or cut for hay within 20 days of

Crop	Country	Formulation	Application					Remarks
			Growth stage & season (crop)	No.	Water L/ha	Rate, kg ai/ha	PHI, days	
								application.
Rape (Clearfield)	Chile	WG 700 g/kg	Early post-emergence	1	150–200	0.042–0.056		
Rape (Clearfield canola)	South Africa	SL 40 g/L	Post-emergence (after the 5-leaf stage)	1	200	0.048	30	
Rape (Clearfield canola)	USA	SL 120 g/L as free acid (amm. salt)	Post-emergence	1		0.035		An adjuvant and a nitrogen fertilizer must be added to the spray solution.
Rice (Red, Clearfield)	COL	SL 33 g/L (imazapyr 15 g/L)	Early post-emergence; 1 st application on red rice with 3 leaves	2 Intervals, 8–10 day	min 150	0.050	45	Apply always among co-adjuvant (spreader penetrant).
Rice (Red, Clearfield)	COL	WG 700 g/kg	Early post-emergence; 1 st application on red rice with 3 leaves	2 Intervals, 8–10 day	min 150	0.098	45	Apply always it among silicone co-adjuvants 1%.
Rice (Asian, Clearfield)	Costa Rica	WG 700 g/kg	Post-emergence (two leaf stage of the crop) BBCH 12–14	2 Intervals, 14 day	200–300	0.070	50	Add EC spreader 37.5 (methyl oleate/palmitate).
Rice (Asian, Clearfield)	DOM	WG 700 g/kg	Early post-emergence; BBCH 12–14 (1 st)	2 Intervals, 14 day	200–300	0.081	50	Add EC spreader 37.5 (methyl oleate/palmitate).
Rice (Clearfield)	Greece	SL 40 g/L as free acid (amm. salt)	Post-emergence (two leaf stage of the crop) BBCH 12–22	1	200–400	0.050		Add EC spreader.
Rice (Clearfield)	Greece	SL 40 g/L as free acid (amm. salt)	BBCH 11–14 3–4 leaves at 1 st appl.; BBCH 15–22 at 2 nd appl.	2 Intervals, 21 day	200–400	0.035		Add EC spreader.
Rice (Clearfield)	Spain	SL 40 g/L	Post-emergence	2 Intervals, 14–21 days	200–300	0.035		Rice plant in water or dry planted rice
Rice (Clearfield, Clearfield rice hybrids)	USA	SL 120 g/L as free acid (amm. salt)	Post-emergence (4-leaf rice to rice panicle initiation plus 14 days for Clearfield rice; 4-leaf rice up to rice panicle initiation for Clearfield rice hybrids)	1 or 2		0.035–0.053 (0.087 /season) G or A		For water-seeded and dry/drill-seeded rice. Do not apply to Clearfield rice hybrids after panicle initiation. A crop oil concentrate must be added to the spray solution (1% v/v).
Soya bean	AUS	WG 700 g/kg	Post-emergence	1	min 50	0.035		WHP: 4 weeks (grazing) A spray adjuvant and liquid ammonium sulphate should be used. Do not graze or cut for stock food for 4 weeks after application.

Crop	Country	Formulation	Application					Remarks
			Growth stage & season (crop)	No.	Water L/ha	Rate, kg ai/ha	PHI, days	
Soya bean	Brazil	WG 700 g/kg	Post-emergence	1	30–50 in aerial appl; 100–300 in ground appl	0.042–0.049	70	Use non-ionic adjuvant at 0.25% v/v of mixture. Aerial spraying
Soya bean	Canada	WG 700 g/kg	BBCH 10–14	1	200	0.025	60	Apply with a non-ionic surfactant at a rate of 0.25% v/v and a fertilizer solution. Do not graze the treated crops or cut for hay within 20 days of application.
Soya bean	Canada	SL 20 g/L (bentazone, 429 g/L)	Early post-emergence (cotyledon to 4-leaf stage)	1	100	0.020	60	Apply with a nitrogen source. Do not graze the treated crops or cut for hay within 20 days of application.
Soya bean	Canada	WG 35% (imazethapyr, 35%)	BBCH 11–13 (1–3 true leaf stage after weeds have emerged)	1	50–100	0.015	85	Apply with EC surfactant (0.5% v/v). Do not graze the treated crops or cut for hay. Do not harvest forage or cut hay for forage.
Soya bean	China	SL 40 g/L	Early post-emergence	1	225–450	0.045–0.050		
Soya bean	Costa Rica	WG 700 g/kg	Early post-emergence	1	200–300	0.040	70	Add a non-ionic surfactant (0.25%).
Soya bean	France	SL 40 g/L	Post-emergence (1 trifoliolate leaf up to 5–6 leaf stage of the culture BBCH 12–14)	1	100–300	0.040–0.050	90	
Soya bean	France	SL 40 g/L	BBCH 12–14	2 Interval, 8–10 day	100–300	0.025	90	An adjuvant is used.
Soya bean	South Africa	SL 120 g/L	Post-emergence	1	min. 200	0.048	64	This must be used with a non-ionic surfactant (0.2% v/v). Grazing of crop 64 days after application.
Soya bean	USA	SL 120 g/L as free acid (amm. sulfate)	Early post-emergence; before bloom stage	1		0.035–0.045		
Soya bean	ZIM	SL 120 g/L	Early post-emergence in soya beans up to but before flowering	1	200	0.036		Add an adjuvant and ammonium sulphate to the spray solution. Do not graze the treated crops or cut for hay within 20 days of application.
Sunflower (Clearfield)	Canada	WG 700 g/kg	Early post-emergence (2–8 leaf)	1	100	0.015–0.020	70	Apply with EC surfactant (0.5% v/v). Do not graze treated

Crop	Country	Formulation	Application					Remarks
			Growth stage & season (crop)	No.	Water L/ha	Rate, kg ai/ha	PHI, days	
			BBCH 12–18					crop or cut for straw.
Sunflower (Clearfield)	Canada	WG 350 g/kg (imazethapyr, 35%)	Early post-emergence (2–8 leaves) BBCH 12–18	1	50–100	0.015	60	Apply with EC surfactant (0.5% v/v). Do not harvest forage or cut hay for forage.
Sunflower (Merigold, Clearfield)	Chile	WG 700 g/kg	Early post-emergence	1	150–200	0.042–0.056		
Sunflower (Clearfield)	France	SL 40 g/L	Post emergence (2–6 leaf) BBCH 12–18	1	100–300	0.050	90	Do not add any adjuvant at greater than 0.050 kg ai/ha. At fractionation, 0.025 kg ai/ha with adjuvant for each 1 st and 2 nd application.
Sunflower (Clearfield)	Greece	SL 40 g/L	Post-emergence BBCH 14–16	1	200–300	0.040		+ 0.5 L/ha of EC spreader
Sunflower (Clearfield)	South Africa	SL 33 g/L (imazapyr, 15 g/L)	2–6 leaf BBCH 12–16		150–250	0.033	70	
Sunflower (Clearfield)	Spain	SL 40 g/L	Post-emergence (4–8 pairs of leaves)	1		0.040		+ 0.5 L/ha of EC spreader
Sunflower (Clearfield)	USA	SL 120 g/L as free acid (amm. salt)	Early post-emergence (2–8 leaf) BBCH 12–18	1		0.035		A non-ionic surfactant and nitrogen-based fertilizer must be added to the spray solution.
Wheat (Clearfield)	ARG	WG 700 g/kg	Early post-emergence	1	100–200	0.049–0.070		Add a non-ionic surfactant of 0.25% of the main ingredient into the total volume.
Wheat (Clearfield Plus)	AUS	SL 33 g/L (amm. salt; imazapyr, 15 g/L)	Early post-emergence (3 leaf-1 st node stage) BBCH 13–31,	1	min 70–100	0.012–0.025	not required	Do not graze or cut for stock food for 4 weeks after application. Do not use on CL STL and CL JNZ wheat variety.
Wheat (Clearfield Plus)	Canada	EC 20 g/L (2-4 D of 2-ethyl ester, 560 g/L)	Early post-emergence (3 to 6-leaf stage; prior to flag leaf emergence) BBCH 13–16	1	100	0.020	79	Apply with non-ionic surfactant at a rate of 0.25% v/v. Do not graze the treated crop within 4 days of application.
Wheat (Clearfield)	Canada	SC 120 g/L (amm. salt)	Early post-emergence (2–6 leaf) BBCH 12–16	1	50–100	0.015–0.020	79 (grain, straw)	Apply with non-ionic surfactant at a rate of 0.25% v/v. Do not graze the treated crop within 14 days of application or cut for hay within 42 days of application.
Wheat (Clearfield)	Chile	WG 700 g/kg	Early post-emergence	1	150–200	0.042–0.056		
Wheat (Spring, Clearfield)	USA	SL 120 g/L as free acid (amm. salt)	Post-emergence (4 leaf to prior to jointing)	1		0.13		A molded jug pack contains ammonium salt of imazamox and 2-ethylhexyl ester of MCPA (67.9%). A surfactant and nitrogen-

Crop	Country	Formulation	Application					Remarks
			Growth stage & season (crop)	No.	Water L/ha	Rate, kg ai/ha	PHI, days	
								based fertilizer must be added to the spray solution. Do not forage or graze meat animals on treated areas within 7 days of slaughter. Do not forage or graze dairy animals on treated areas within 7 days after treatment.
Wheat (Winter, Clearfield)	USA	SL 120 g/L as free acid (amm. salt)	Post-emergence at tiller initiation but prior to jointing	1		0.11–0.16		the same with remarks above
Wheat (Spring, Clearfield)	USA	SL 120 g/L as free acid (amm. salt)	Early post-emergence (4-leaf to prior to jointing)	1		0.035–0.044		Add a non-ionic surfactant and nitrogen-based fertilizer. COC or MSO may be substituted for the non-ionic surfactant.
Wheat (Winter, Clearfield)	USA	SL 120 g/L as free acid (amm. salt)	Early post-emergence (after tiller initiation but prior to jointing)	1 or 2		0.035–0.053 (0.070 kg ai/ha/season)		Add a non-ionic surfactant and nitrogen-based fertilizer. COC or MSO may be substituted for the non-ionic surfactant in 2-gene winter wheat. There are no restrictions for feeding or grazing of wheat forage and hay.
Alfalfa (Lucerne)	AUS	WG 700 g/kg	Post-emergence (2 trifoliolate leaf; apply as following cutting or grazing)	1	min 50	0.032–0.035		WHP: 7 days (grazing). Do not graze or cut for stock food for 7 days after application. A spray adjuvant and liquid ammonium sulfate should be added. Do not use the Hasten or Kwickin mix (oil adjuvant) on seedling lucerne.
Alfalfa	Chile	WG 700 g/kg	Early post-emergence	1	150–200	0.042–0.056	n.a.	
Alfalfa	France	EC ^a 16.7 g/L (pendimethalin, 250 g/L)	Post-emergence ^b	1	100–500	0.033 or 0.067 ^b	30	
Alfalfa (Medicago sativa)	Greece	SL 40 g/L (4.22% amm. salt)	^c	1	300	0.050	28	+ 4 kg/ha ammonium sulfate
Alfalfa (established)	Saudi Arabia	SL 40 g/L	^d	1	300	0.048 (newly sown) or 0.060 (established)	35	Addition of nitrogen fertilizer (urea) to spray solution at a rate of 2 kg/ha.

Crop	Country	Formulation	Application					Remarks
			Growth stage & season (crop)	No.	Water L/ha	Rate, kg ai/ha	PHI, days	
Alfalfa (Lucerne)	South Africa	SL 120 g/L	post-emergence	1	min. 200	0.036–0.048	22	This must only be used in a tank mixture with a non-ionic surfactant (0.2% v/v) and ammonium sulfate (2.0% v/v).
Alfalfa	South Africa	SL 40 g/L	Post-emergence	1 or 2	200	0.048	22	Two applications can be made on lucerne per growing season. If necessary, the 2 nd application can be made immediately after any of the early to mid-season cuttings.
Alfalfa	Spain	SL 40 g/L	Early post-emergence (4 true leaves)			0.050		
Alfalfa	USA	SL 120 g/L as free acid (amm. sulfate)	^e	1		0.035–0.053		

In Costa Rica, Guatemala, El Salvador, Nicaragua, and Panama, the same use is approved for common bean.

As well as UK, the same use for field bean is approved in Ireland.

In Cota Rica, Guatemala, El Salvador, Nicaragua, and Panama, the same use is approved for soya bean.

In Cota Rica and El Salvador, the same use is approved for Asian rice (Clearfield).

^a Seed-bearing fodder legumes (violet clover, white clover and crimson clover and of seed-bearing alfalfa: a) on set-up cultures at 0.067 kg ai/ha, in a single application and making sure that the application is carried out at most every 2 years. B) on a young plant culture at 0.033 kg ai/ha, between the 2 leaf and 4–5 trifoliate leaf stages. A second 0.033 kg ai/ha application may prove to be useful in certain cases. Do not exceed a maximum of two applications every 2 years.

^b Applied in post-emergence of the alfalfa: for young cultures, 0.033 kg ai/ha from the 2 trifoliate leaf stage of alfalfa; for cultures set up, a) end of autumn/beginning of winter: 0.033 kg ai/ha b) out of winter (resumption of vegetation growth): 0.033–0.067 kg ai/ha. If an application was carried out at 0.033 kg ai/ha at the end of autumn/beginning of winter, resumption is possible at the end of winter at a dosage of 0.033 kg ai/ha if this is necessary. Notes: Do not apply if the culture has been milled beforehand. At the end of the season between the last cut and the potential application, observe a min. 15 days period

^c 1) New planting: one application after germination or after the 1st cut, when the crop has 2–5 leaves 2) Established planting (from 2nd year): one application during dormancy of the crop or after the 1st cut (as soon as the alfalfa acquires 2–5 new leaves)

^d 1) Newly sown: 2–3 leaf stage 2) Established: after dormant season or 4–5 days after cutting

^e Early post-emergence 1) Seedling: application in the 2nd trifoliate stage or larger. For alfalfa grown for seed, before bud formation. 2) Established: application in the fall, winter, or in the spring to dormant or semi dormant alfalfa, or between cuttings. Any application should be made before significant alfalfa growth or regrowth (3 inches).

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received residue data from supervised field trials conducted on legume vegetables, pulses, cereal grains, oilseed and animal feeds.

Application rates and residue concentrations were reported as imazamox. Residue concentrations were recorded unadjusted for recoveries or for residue values in control samples. Where multiple samples were taken from a single plot, the calculated mean concentration is reported and used for estimation of maximum residue level. When trials were conducted in the same location, with the same or similar varieties, same or similar formulations, and same equipment, and at the same or similar timing, they are not regarded as independent and only one result from these trials was chosen for the estimation of a maximum residue level.

Analytical methods used for determination of imazamox-related residues in field trial samples were well validated through procedural recovery tests or method validation conducted previously.

Concentration of the metabolite CL 263284 was expressed as parent equivalents using conversion factor of 1.048. When calculating sum of parent and CL 263284, for values below LOQ, the LOQ was used for each analyte.

In many of the following trials, plants resistant to imidazolinone pesticides were used. That is expressed as “CL” in their variety name, which means non-GM plants showing the tolerance due to the mutation(s) of endogenous AHAS gene from conventional breeding technique.

Commodity group	Commodity	Table No.
Legume vegetables	Beans	58
Pulses	Peas	59
	Beans (dry)	60
	Peas (dry)	61
	Lentil (dry), imidazolinone-tolerant	62
	Soya bean (dry)	63
Cereals	Rice, imidazolinone-tolerant	64
	Wheat, imidazolinone-tolerant	65
Oilseed crop	Peanut	66
	Rape seed, imidazolinone-tolerant	67
	Sunflower seed, imidazolinone-tolerant	68
Legume animal feeds	Alfalfa	69
	Pea forage and fodder	70
	Soya bean forage and fodder	71
Forage and fodder of cereal grains and grasses	Rice forage and straw, imidazolinone-tolerant	72
	Wheat forage and straw, imidazolinone-tolerant	73, 74
Miscellaneous fodder and forage crops	Rape seed forage, imidazolinone-tolerant	75

Legume vegetables

Beans

Residue field trials on beans were conducted in the European countries and USA during the growing season of 1996, 2011 and 2012. SL or EC formulation was used at various rates of 0.028–0.075 kg ai/ha with a pre- or post-emergence application. Bean seeds or other bean fractions were analysed for the determination of imazamox and the metabolites.

Table 58 Residues of imazamox in beans from supervised field trials

Location Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in beans, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Denmark Røjleskovej 2011 (Flagrano) 404371// L110253 2012/1221518	SL 22.4	0.028	0.027	1	(22-51)	0	w. plant	1.70	< 0.01	1.71	< 0.01	< 0.01
						29	pod w/ seed	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
						29	rest of plant	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
						62	pod w/ seed	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
						62	pod w/o	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
						62	seed	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
						62	rest of plant seeds	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01

Location Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in beans, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Germany Palatinate 2011 (Primel) 404371// L110251 2012/1221518	SL 22.4	0.028	0.028	1	(25)	0 27 27 41 41 41 41	w. plant pod w/ seed rest of plant pod w/ seed pod w/o seed rest of plant seeds	0.48 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.49 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01
Germany Baden- Württemberg 2011 (Flageolet) 404371// L110254 2012/1221518	SL 22.4	0.028	0.028	1	(25)	0 30 30 51 51 51 51	w. plant pod w/ seed rest of plant pod w/ seed pod w/o seed rest of plant seeds	2.10 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.02 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	2.11 < 0.02 0.03 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.01 < 0.01 0.07 < 0.01 0.01 0.02 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01
N-France Loiret 2011 (Nagano) 404371// L110252 2012/1221518	SL 22.4	0.028	0.028	1	(25)	0 28 28 35 35	w. plant pod w/seed rest of plant pod w/seed rest of plant	3.70 < 0.01 < 0.01 < 0.01 < 0.01	0.03 < 0.01 < 0.01 < 0.01 < 0.01	3.73 < 0.02 < 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01 < 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01
N-France Hebuterine 2012 (Flageolet bean: Flambean) 421932// L120366 2013/1361329	EC 16.7	0.075	0.038	1	Pre- emergence	46 74 74	w. plant pod w/ seed rest of plant	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
UK Haddenham 2012 (Flageolet bean: Safari) 421932// L120365 2013/1361329	EC 16.7	0.075	0.038	1	Pre- emergence	48 73 73	w. plant pod w/ seed rest of plant	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
S-France Tarnet Garonne 2011 (Proton) 404371// L110255 2012/1221518	SL 22.4	0.028	0.028	1	(25, 60)	0 28 28	w. plant pod w/seed rest of plant	1.60 < 0.01 < 0.01	< 0.01 < 0.01 0.03	1.61 < 0.02 0.04	< 0.01 0.02 0.08	< 0.01 < 0.01 < 0.01
S-France Tarnet Garonne 2011 (Proton) 404371// L110256 2012/1221518	SL 22.4	0.039	0.039	1	(25, 60)	0 28 28	w. plant pod w/seed rest of plant	0.80 < 0.01 < 0.01	< 0.01 < 0.01 0.01	0.81 < 0.02 0.02	< 0.01 0.02 0.05	< 0.01 < 0.01 < 0.01
S-France Pusignan 2012	EC 16.7	0.075	0.038	1	Pre- emergence	36 64 64	w. plant pod w/ seed rest of plant	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01

Location Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in beans, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
(Flageolet bean: Flajoly) 421932// L120368 2013/1361329												
Greece Thessalonikj 2011 (Etna) 404371// L110257 2012/1221518	SL 22.4	0.028	0.028	1	(25)	0	w. plant	0.79	0.06	0.85	< 0.01	< 0.01
						29	pod w/ seed	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
						29	pod w/o	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
						29	seed rest of	< 0.01	< 0.01	< 0.02	0.01	< 0.01
						29	plant	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
						39	seeds	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
						39	pod w/seed	< 0.01	< 0.01	< 0.02	0.02	< 0.01
						39	pod w/o	< 0.01	< 0.01	< 0.02	0.01	< 0.01
						39	seed rest of	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
							plant seeds					
Italy Minrbio (French bean : n.r.) 1994 94072//1 ID-720-044	SL 120	0.050	0.010	1	Post- emergence	53	Pods	< 0.05	< 0.05	< 0.10		
Italy Sasso Morelli- Imola 1994 (French bean: n.r.) 94072//2 ID-720-044	SL 120	0.050	0.010	1	Post- emergence	58	Pods	< 0.05	< 0.05	< 0.10		
Italy Sabbbiuno 1994 (French bean: Bronco) 94072//3 ID-720-044	SL 120	0.050	0.010	1	Post- emergence	28	Pods	< 0.05	< 0.05	< 0.10		
Italy Granarolo Faentino 1994 (French bean: Masai) 94072//4 ID-720-044	SL 120	0.050	0.010	1	Post- emergence	29	Pods	< 0.05	< 0.05	< 0.10		
Italy Sasso Morelli 1996 (French bean: Twiggi) ID-IT-96- 601// 96-601- 01 ID-720-053	SL 40	0.050	0.010	1	Post- emergence (13)	42	whole plant	< 0.05	< 0.05	< 0.10		
						51	whole plant	< 0.05	< 0.05	< 0.10		
						61	pod w/ seed	< 0.05	< 0.05	< 0.10		
Italy Gaiba 1996 (French bean: Masai) ID-IT-96-	SL 40	0.050	0.010	1	Post- emergence (13)	26	whole plant	< 0.05	< 0.05	< 0.10		
						35	whole plant	< 0.05	< 0.05	< 0.10		
						45	pod w/ seed	< 0.05	< 0.05	< 0.10		

Location Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in beans, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
601// 96-601-02 ID-720-053												
Italy Roncoferraro 2012 (Flageolet bean : Borlotto Lingua di Fuoco) 421932// L120367 2013/1361329	EC 16.7	0.075	0.038	1	Pre- emergence	32 74 74	w. plant pod w/ seed rest of plant	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
Spain La Rioja, 2011 (Beronia) 404371// L110258 2012/1221518	SL 22.4	0.028	0.028	1	(25)	0 27 27 35 35 35 35	w. plant pod w/ seed rest of plant pod w/ seed pod w/o seed rest of plant seeds	0.81 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.83 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01
USA Freeville, NY 1998 (Snap bean: Labrador) 06663// 98-NY23 ID-720-073	SL 11.5%, w/w	0.050		1	Post- emergence	35	pod w/seed	< 0.05				
USA Arlington, WI 1998 (Snap bean: Hystyle) 06663// 98-WI30 ID-720-073	SL 11.5%, w/w	0.050		1	Post- emergence	30	pod w/seed	< 0.05				
USA East Lansing, MI 1998 (Snap bean: Strike) 06663// 98-MI40 ID-720-073	SL 11.5%, w/w	0.050		1	Post- emergence	41	pod w/seed	< 0.05				
USA Clinton, NC 1998 (Snap bean: Provider) 06663// 98-NC21 ID-720-073	SL 11.5%, w/w	0.050		1	Post- emergence	50	pod w/seed	< 0.05				
USA Gainesville, FL	SL 11.5%, w/w	0.050		1	Post- emergence	37	pod w/seed	< 0.05				

Location Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in beans, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
1998 (Snap bean: Podsquad) 06663// 98-FL61 ID-720-073												
USA Kimberly, ID 1998 (Snap bean: Idelite Garden) 06663// 98-ID15 ID-720-073	SL 11.5%,w/w	0.040	0.022	1	Post- emergence	44	pod w/seed	< 0.05				

Mix formulation: SL 22.4 g/L (bentazone 480 g/L); EC 16.7 g (pendimethalin 250 g/L)
w. plant: whole plant without roots

Peas

Residue trials on peas were conducted in EU countries and USA during the growing season of 1995–1998 and 2002–2008. SL or EC formulation was used at various rates of 0.028–0.075 kg ai/ha with a pre- or post-emergence application. Pea seeds or other pea fractions were analysed for the determination of imazamox and the metabolites.

Table 59 Residues of imazamox in peas from supervised field trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage at application (BBCH)	DAT	Portion analysed	Residue in peas, mg/kg		
								Parent	CL 263284	Parent + CL 263284
Germany Ostrau (Ambassador) 2007/2008 B42930// A/GE/H/07/136 2007/1023133	SL 22.4	0.028	0.014	1	Post- emergence (18)	40	seed (green) pods plant w/o pods	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.013	< 0.02 < 0.02 0.02
N-France Caillouet (Solara) 1996 ID-FR-96-610//96- 610-319 ID-720-048	EC 16.7	0.075	0.021	1	Pre- emergence (05–08)	88	seeds (green)	< 0.05	< 0.05	< 0.1
N-France Bourgogne (Baccara) 1996 ID-FR-96-610//96- 610-422 ID-720-048	EC 16.7	0.075	0.031	1	Pre- emergence (00)	105	seeds (green)	< 0.05	< 0.05	< 0.1
N-France Fains (Solara) 1996 ID-FR-96-611//96-	EC 16.7	0.075	0.021	1	Pre- emergence (05–08)	95	seeds (green)	< 0.05	< 0.05	< 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage at application (BBCH)	DAT	Portion analysed	Residue in peas, mg/kg		
								Parent	CL 263284	Parent + CL 263284
611-320 ID-720-051										
N-France Fains (Solara) 1996 ID-FR-96-611//96- 610-423 ID-720-051	EC 16.7	0.075	0.031	1	Pre- emergence (00)	110	seeds (green)	< 0.05	< 0.05	< 0.1
N-France Viesly Nord Pas de Calais (Arabelle) 2007/2008 B42930// A/NF/H/07/133 2007/1023133	SL 22.4	0.029	0.007	1	Post- emergence (51)	43 43 43	seed (green) pods plant w/o pods	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02
N-France Wassigny (Ultimo) 2007/2008 B42930// A/NF/H/07/134 2007/1023133	SL 22.4	0.028	0.007	1	Post- emergence (51)	44 44 44	seed (green) pods plant w/o pods	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02
UK Evesham (Ambassador) 2007/2008 B42930// A/UK/H/07/135 2007/1023133	SL 22.4	0.028	0.007	1	Post- emergence (16-17)	51 48 48	seed (green) pods plant w/o pods	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.010	< 0.02 < 0.02 0.02
S-France Nerac (Carrera) 1996 ID-FR-96-660//96- 660-293 ID-720-049	EC 16.7	0.075	0.023	1	Pre- emergence (00-01)	96	seeds (green)	< 0.05	< 0.05	< 0.1
S-France Chateaufort (Douce Provence) 2007/2008 B42930// A/SF/H/07/137 2007/102333	SL 22.4	0.029	0.007	1	Post- emergence (12)	37 39 39	seed (green) pods plant w/o pods	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.015	< 0.02 < 0.02 0.02
S-France Lagnes (Douce Provence) 2007/2008 B42930// A/SF/H/07/140 2007/102333	SL 22.4	0.027	0.007	1	Post- emergence (14)	29 31 31	seed (green) pods plant w/o pods	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.026	< 0.02 < 0.02 0.04
Italy Emilia-Romagna 1994 (n.r.) 94071//1 ID-720-047	SC 22.7	0.068	0.014	1	Pre- emergence	78	seeds	< 0.05	< 0.05	< 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage at application (BBCH)	DAT	Portion analysed	Residue in peas, mg/kg		
								Parent	CL 263284	Parent + CL 263284
	SL 120	0.050	0.010	1	Post- emergence	59	seeds	< 0.05	< 0.05	< 0.1
Italy Emilia-Romagna (Hunter) 1994 94071//2 ID-720-047	SC 22.7	0.068	0.014	1	Pre- emergence	77	seeds	< 0.05	< 0.05	< 0.1
	SL 120	0.050	0.010	1	Post- emergence	34	seeds	< 0.05	< 0.05	< 0.1
Italy Emilia-Romagna (Hunter) 1994 94071//3 ID-720-047	SC 22.7	0.068	0.014	1	Pre- emergence	83	seeds	< 0.05	< 0.05	< 0.1
	SL 120	0.050	0.010	1	Post- emergence	38	seeds	< 0.05	< 0.05	< 0.1
Italy Emilia-Romagna (n.r.) 1994 94071//4 ID-720-047	SC 22.7	0.068	0.014	1	Pre- emergence	70	seeds	< 0.05	< 0.05	< 0.1
	SL 120	0.050	0.010	1	Post- emergence	53	seeds	< 0.05	< 0.05	< 0.1
Italy Torello (Zaffiro) 1996 ID-IT-96-608// 96-608-01 ID-720-050	SL 40	0.050	0.010	1	Post- emergence (13)	52	seeds (green)	< 0.05	< 0.05	< 0.1
	SL 40	0.075	0.016	1	Post- emergence (13)	52	seeds (green)	< 0.05	< 0.05	< 0.1
Italy Comacchio (Zaffiro) 1995 ID-IT-95-012// 95-012-01 ID-720-052	SL 120	0.050	0.01	1	Post- emergence (21–25)	40	seeds (green)	< 0.05	< 0.05	< 0.1
Italy Masi Torello (Java) 1995 ID-IT-95-012// 95-012-02 ID-720-052	SL 120	0.050	0.01	1	Post- emergence (12–13)	63	seeds (green)	< 0.05	< 0.05	< 0.1
Italy Caleppio di Settala (Pactol) 2007/2008 B42930// A/IT/H/07/138 2007/1023133	SL 22.4	0.028	0.007	1	Post- emergence (24)	72	seed (green)	< 0.01	< 0.01	< 0.02
						72	pod	< 0.01	< 0.01	< 0.02
						72	plant w/o pod	< 0.01	0.029	0.039
Italy Marcallo con	SL 22.4	0.027	0.009	1	Post- emergence	62 62	seed (green) pod	< 0.01 < 0.01	< 0.01 < 0.01	< 0.02 < 0.02

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage at application (BBCH)	DAT	Portion analysed	Residue in peas, mg/kg		
								Parent	CL 263284	Parent + CL 263284
Castone (Starter) 2007/2008 B42930// A/IT/H/07/139 2007/1023133					(25)	62	plant pods w/o	< 0.01	< 0.01	< 0.02
Spain Sarifienna-Huesca (Puyet) 1996 ID-SP-96-609 //96-609-27 ID-720-054	SL 40	0.052	0.017	1	Post- emergence (14-15)	64	seeds	< 0.05	< 0.05	< 0.1
	SL 40	0.073	0.024	1	Post- emergence (14-15)	64	seeds	< 0.05	< 0.05	< 0.1
USA Freeville (Bolero) 1998 06664//98-NY12 ID-720-075	SL 11.5%,w/w	0.050		1	Post- emergence	59	seeds	< 0.05		
USA Salinas (Maestro) 1998 06664//98-CA*38 ID-720-075	SL 11.5%,w/w	0.050		1	Post- emergence	44	seeds	< 0.05		
USA Salinas (Oregon Sugar Pod II 1998 06664//98-CA*39 ID-720-075	SL 11.5%,w/w	0.050		1	Post- emergence	36	pod seeds with	< 0.05		
USA Aurora (Knight) 1998 06664//98-OR19 ID-720-075	SL 11.5%,w/w	0.050		1	Post- emergence	65	seeds	< 0.05		
USA Arlington (Rally) 1998 06664//98-WI16 ID-720-075	SL 11.5%,w/w	0.050		1	Post- emergence	44	seeds	< 0.05		
USA Arlington (Dual) 1998 06664//98-WI17 ID-720-075	SL 11.5%,w/w	0.050		1	Post- emergence	48	seeds	< 0.05		
USA ^a East Lansing (Utrillo) 1998 06664//98-MI24 ID-720-075	SL 11.5%,w/w	0.050		1	Post- emergence	57	seeds	< 0.05		
USA ^a East Lansing	SL 11.5%,	0.050		1	Post- emergence	57	seeds	< 0.05		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage at application (BBCH)	DAT	Portion analysed	Residue in peas, mg/kg		
								Parent	CL 263284	Parent + CL 263284
(Utrillo) 1998 06664//98-MI25 ID-720-075	w/w									
USA Charleston (Wando) 1998 06664//98-SC*03 ID-720-075	SL 11.5%,w/w	0.050		1	Post- emergence	49	seeds	< 0.05		

Mix formulation: SC 22.7 g/L (pendimethalin of 341 g/L); SL 22.4 g/L (bentazone of 480 g/L); EC 16.7 g/L (pendimethalin of 250 g/L)

wp: Whole plant

^a Not independent trials

Pulses

Bean (dry)

Residue field trials on dry beans were conducted in the European countries and USA during the growing season of 1998, 2011 and 2012. SL or EC formulation was used at various rates of 0.028–0.075 kg ai/ha with a pre- or post-emergence application. Bean seeds or other bean fractions were analysed for the determination of imazamox and the metabolites.

Table 60 Residues of imazamox in bean (dry) from supervised field trials

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in bean (dry), mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
N-France Hebuterine 2012 (Flageolet bean : Flam bean) 421932 //L120366 2013/1361329	EC 16.7	0.075	0.038	1	Pre- emergence	108 108 108 108	pod w/seed	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
							pod w/o seed	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
							rest of plant	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
							seeds	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
GermanyHalen 2011 (Broad bean: Fuego) 407230 //L110308 2012/1299407	SL 40	0.040	0.020	1	16	0 116 116	w. plant	3.07	0.04	3.11	< 0.01	< 0.01
							seeds	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
							rest of plant	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
Netherlands Vlagtwedde 2011 (Broad bean: Fuego) 407230 2012/1299407	SL 40	0.040	0.020	1	16	0 115 115	wp w/o root	1.58	0.02	1.60	< 0.01	< 0.01
							seeds	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
							rest of plant	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
UK Haddenham 2012 (Flageolet bean	EC 16.7	0.075	0.038	1	Pre- emergence	97 97 97 97	pod w/seed	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
							pod w/o seed	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
							rest of plant	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
							seeds	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in bean (dry), mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
: Safari) 421932 //L120365 2013/1361329												
UK Warwickshire 2011 (Broad bean: Wizard) 407230 //L110309 2012/1299407	SL	0.040	0.020	1	16	0 117 117	w. plant seeds rest of plant	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
UK Banbury 2011 (Broad bean: Wizard) 407230 //L110310 2012/1299407	SL 40	0.040	0.020	1	16	0 139 139	w. plant seeds rest of plant	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.01	< 0.02 < 0.02 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
S-France Pusignan 2012 (Flageolet bean: Flajoly) 421932 //L120368 2013/1361329	EC 16.7	0.075	0.038	1	Pre- emergence	91 91 91 91	pod w/seed pod w/o seed rest of plant seeds	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01
S-France Tarn Et Garonne 2011 (Irene) 4042844// L110245 2011/1112577	SL 22.4	0.031	0.030	1	25	0 55 55	w. plant seeds pods+ rest5	1.2 < 0.01 < 0.01	< 0.01 0.01 0.05	1.2 0.02 0.06	< 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01
S-France Tarn Et Garonne 2011 (Castel) 4042844// L110247 2011/1112577	SL 22.4	0.033	0.030	1	24-25	0 57 57	w. plant seeds pods+ rest5	0.62 < 0.01 < 0.01	< 0.01 < 0.01 0.01	0.63 < 0.02 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
Italy Roncoferraro 2012 (Flageolet bean : Borlotto Lingua di Fluco) 421932 //L120367 2013/1361329	EC 16.7	0.075	0.038	1	Pre- emergence	92 92 92 92	pod w/seed pod w/o seed rest of plant seeds	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01
Italy Vaccolino 2011 (Broad bean: Aqua Dolce) 407230 //L110314 2012/1299407	SL 40	0.040	0.020	1	16	0 51 51	w. plant seeds rest of plant	2.05 < 0.01 < 0.01	0.04 < 0.01 < 0.01	2.09 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in bean (dry), mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Greece Thessaloniki 2011 (Aqua Dulce) 4042844// L110248 2011/1112577	SL 22.4	0.028	0.03	1	25	0 62 62	w. plant seeds pods+rest 5	1.8 < 0.01 < 0.01	0.03 < 0.01 0.02	1.8 < 0.02 0.03	< 0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01
Spain Zafarraya 2011 (Broad bean: Muchamiel) 407230 //L110313 2012/1299407	SL 40	0.040	0.020	1	16	0 91 91	w. plant seeds rest of plant	2.15 < 0.01 < 0.01	0.45 < 0.01 < 0.01	2.60 < 0.02 < 0.02	0.02 < 0.01 < 0.01	0.02 < 0.01 < 0.01
Spain Malaga 2011 (Broad bean: Muchamiel) Broa407230 //L110315 2012/1299407	SL 40	0.040	0.020	1	16	0 90 90	w. plant seeds rest of plant	1.34 < 0.01 < 0.01	0.29 < 0.01 < 0.01	1.63 < 0.02 < 0.02	0.01 < 0.01 < 0.01	0.01 < 0.01 < 0.01
USA Ereeville, NY 1998 (Red kidney bean: Horizon Light) 06820// 98-NY11 ID-720-071	SL 11.5%	0.050	0.017- 0.035	1	Post- emergence 1st trifoliate stage	76	dry seeds	< 0.05				
USA ^a Prosser 1998 (Othello Pinto) 06820// 98-WA*41 ID-720-071	SL 11.5%	0.050	0.017	1	Post- emergence 1st trifoliate stage	59	dry seeds	< 0.05				
USA ^a Prosser 1998 (Othello Pinto) 06820// 98-WA*42 1998 ID-720-071	SL 11.5%	0.050	0.017	1	Post- emergence 1st trifoliate stage	59	dry seeds	< 0.05				
USA Parlier 1998 (UC 6 Blackeye) 6820//98-CA37 ID-720-071	SL 11.5%	0.050	0.035	1	Post- emergence 1st trifoliate stage	84	dry seeds	< 0.05				
USA ^b Freemont 1998 (Avanti Navy) 06820// 98-OH*09 ID-720-071	SL 11.5%	0.050	0.035	1	Post- emergence 1st trifoliate stage	75	dry seeds	< 0.05				
USA ^b	SL	0.050	0.035	1	Post-	70	dry seeds	< 0.05				

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in bean (dry), mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Freemont 1998 (Avanti Navy) 06820// 98-OH*10 1998 ID-720-071	11.5%				emergence 1st trifoliate stage							
USA ^b Freemont 1998 (Avanti Navy) 06820// 98-OH*11 ID-720-071	SL 11.5%	0.050	0017– 0.035	1	Post- emergence 1st trifoliate stage	68	dry seeds	< 0.05				
USA ^c Scottsbluff 1998 (Berly Great Northern) 06820//98-NE01 ID-720-071	SL 11.5%	0.050	0017– 0.035	1	Post- emergence 1st trifoliate stage	78	dry seeds	< 0.05				
USA ^c Scottsbluff 1998 (Berly Great Northern) 06820// 98- NE02 ID-720-071	SL 11.5%	0.050	0017– 0.035	1	Post- emergence 1st trifoliate stage	78	dry seeds	< 0.05				
USA ^c Scottsbluff 1998 (Berly Great Northern) 06820// 98- NE03 ID-720-071	SL 11.5%	0.050	0017– 0.035	1	Post- emergence 1st trifoliate stage	82	dry seeds	< 0.05				
USA Prosser 1998 (Lima bean: Baby) 06659// 98-WA*40 1998 ID-720--072	SL 11.5%	0.050		1	Post- emergence 1st -2 nd trifoliate stage	76	seeds	< 0.05				
USA ^d Tifton 1998 (Lima bean: Cangreen Bush) 06659// 98-GA*11 ID-720--072	SL 11.5%	0.050		1	Post- emergence 1st -2 nd trifoliate stage	64	seeds	< 0.05				
USA ^d Tifton 1998 (Lima bean: Cangreen Bush) 06659// 98-GA*12 ID-720--072	SL 11.5%	0.050		1	Post- emergence 1st -2 nd trifoliate stage	64	seeds	< 0.05				

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in bean (dry), mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
USA Fermont 1998 (Lima bean: Nemagreen) 06659// 98-OH*08 ID-720--072	SL 11.5%	0.050		1	Post- emergence 1st -2 nd trifoliate stage	74	seeds	< 0.05				
USA Parlier 1998 (Lima bean: Henderson) 06659// 98-CA35 ID-720-072	SL 11.5%	0.070		1	Post- emergence 1st -2 nd trifoliate stage	61	seeds	< 0.05				
USA Parlier 1998 (Lima bean: Mezcla) 06659// 98-CA36 ID-720--072	SL 11.5%	0.070		1	Post- emergence 1st -2 nd trifoliate stage	98	seeds	< 0.05				

Mix formulation: SL 22.4 g/L (bentazone of 480 g/L); EC 16.7 g/L (pendimethalin of 250 g/L)

w. plant: Whole plant without roots

n.a.: Not available; a default LOQ (0.05 mg/kg) for CL263284 was used in calculating sum of imazamox and CL 263284 residues

^a Not independent trials

^b Not independent trials

^c Not independent trials

^d Not independent trials

Pea (dry)

Residue field trials on dry peas were conducted in France during the growing season of 1994 and 1996. SL or EC formulation was used at rates of 0.071–0.075 kg ai/ha with a pre-emergence application. Pea seeds or other pea fractions were analysed for the determination of imazamox and the metabolites.

Table 61 Residues of imazamox in pea (dry) from supervised field trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in pea (dry), mg/kg		
								Parent	CL 263284	Total
N-France Serez 1994 (Solara) 94092// R03.94/F-370 ID-720-042	SL 120	0.075	0.019	1	Pre- emergence (06–07)	127 127	grain straw			< 0.05 ^a < 0.05 ^a
N-France Bataille 1994 (Solara) 94092// R03.94/F-371 ID-720-042	SL 120	0.075	0.019	1	Pre- emergence (06–07)	126 126	grain straw			< 0.05 ^a < 0.05 ^a

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in pea (dry), mg/kg		
								Parent	CL 263284	Total
N-France Bourgogne 1994 (Messire) 94092// R03.94/F-449 ID-720-042	SL 120	0.075	0.025	1	Pre- emergence (06)	117	grain straw			< 0.05 ^a < 0.05 ^a
N-France Marne 1994 (Montana) 94092// R03.94/F-535 ID-720-042	SL 120	0.075	0.023	1	Pre- emergence (00–05)	124	grain straw			< 0.05 ^a < 0.05 ^a
N-France Varennes 1994 (Mesire) 94092// R03.94/F-536 ID-720-042	SL 120	0.075	0.0 23	1	Pre- emergence (00–02)	116	grain straw			< 0.05 ^a < 0.05 ^a
N-France Caillouet- Orgeville 1996 (Solara) ID FR 96 610 //96-610-319 ID-720-048	EC 16.7	0.075	0.021– 0.031	1	Pre- emergence (05–08)	106 106	Pods+haulm seeds (dried)	< 0.05 < 0.05	0.17 < 0.05	0.22 < 0.1
N-France Bourgogne 1996 (Baccara) ID-FR-96-610 //96-610-422 ID-720-048	EC 16.7	0.075	0.031	1	Pre- emergence (00)	126 126	Pods+haulm seeds (dried)	< 0.05 < 0.05	< 0.05 < 0.05	< 0.1 < 0.1
N-France Fains 1996 (Solara) ID FR 96 611 //96-611-320 ID-720-051	EC 16.7	0.075	0.021	1	Pre- emergence (05–08)	117 117	seeds (dried) Pods+haulm	< 0.05 < 0.05	< 0.05 < 0.05	< 0.1 < 0.1
N-France Pomacle 1996 (Carrera) ID FR 96 611 //96-610-423 ID-720-051	EC 16.7	0.071	0.031	1	Pre- emergence (00)	129 129	seeds (dried) Pods+haulm	< 0.05 < 0.05	< 0.05 < 0.05	< 0.1 < 0.1
S-France Nerac 1996 (Carrera) ID FR 96 660 //96-660-293 ID-720-049	EC 16.7	0.075	0.023	1	Pre- emergence (00–01)	120 120	seeds (dried) Pods+haulm	< 0.05 < 0.05	< 0.05 < 0.05	< 0.1 < 0.1

Mix formulation: EC 16.7 g/L (pendimethalin of 250 g/L)

^a Imazamox and CL 263284 were measured as one chromatographic peak representing a total imazamox-related residue.

Lentil

Residue trials were conducted with imidazolinone-tolerant lentils in Canada and USA during the growing season of 2001–2008. SL or WG formulation was used at rates of 0.015–0.021 kg ai/ha with a post-emergence application. Lentil seeds were analysed for the determination of imazamox and the metabolites.

Table 62 Residues of imazamox in imidazolinone-tolerant lentils from supervised field trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in lentil, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Canada Dundurn 2008 (Impact) 327307//R080103 2008/7019226	SL 31.3	0.020	0.019 – 0.020	1	Stem elongation, 7–8 nodes	58	seed	< 0.05	< 0.05	< 0.1		
Canada Portage la Prairie 2008 (CDC Impact) 327307//R080104 2008/7019226	SL 31.3	0.020	0.019 – 0.020	1	Early flower	40 50 60 70 81	seed seed seed seed seed	0.08 0.07 < 0.05 0.06 0.06	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.13 0.11 < 0.1 0.11 0.11		
Canada Dundurn 2008 (Imperial) 327307//R080105 2008/7019226	SL 31.3	0.020	0.019 – 0.020	1	Stem elongation, 7–8 node	58	seed	< 0.05	< 0.05	< 0.1		
Canada Wellwood 2008 (CDC Impact) 327307//R080106 2008/7019226	SL 31.3	0.020	0.019 – 0.020	1	Early bud	59	seed	< 0.05	< 0.05	< 0.1		
Canada Alvena 2008 (Impact) 327307//R080107 2008/7019226	SL 31.3 g/L	0.020	0.020	1	Stem elongation, 6–8 nodes	59	seed	0.06	< 0.05	0.11		
Canada Elm Creek 2001 (RH 44) 66880//2001926 2002-5004457	WG 360	0.015	0.015	1	6 nodes	78	seed	< 0.05	< 0.05	< 0.10	< 0.05	< 0.05
Canada Schuler 2001 (RH 44) 66880//2001927 2002-5004457	WG 360	0.020	0.020	1	3–6 leaf	55	seed	< 0.05	< 0.05	< 0.1	< 0.05	< 0.05
Canada Richmond 2001 (RH 44) 66880//2001928 2002-5004457	WG 360	0.015	0.015	1	6 nodes	48	seed	< 0.05	< 0.05	< 0.1	< 0.05	< 0.05
Canada Delisle 2001 (RH 44) 66880//2001929 2002-5004457	WG 360	0.020	0.020	1	4–6 nodes	62 69	seed seed	< 0.05 0.056	< 0.05 < 0.05	< 0.1 0.11	< 0.05 < 0.05	< 0.05 < 0.05

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in lentil, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Canada Paradise Valley 2001 (RH 44) 66880/2001930 2002-5004457	WG 360	0.015	0.015	1	5–6 nodes	64	seed	< 0.05	< 0.05	< 0.1	< 0.05	< 0.05
Canada Minto 2001 (RH 44) 66880/2001931 2002-5004457	WG 360	0.015	0.015	1	6 nodes	98	seed	< 0.05	< 0.05	< 0.1	< 0.05	< 0.05
Canada Hepburn, SK 2005 (CDC 2464) 138146/R05083 2006/7006733	WG 700	0.020	0.010	1	Full flowering (50% flowers Open)	60	seed	< 0.05	< 0.05	< 0.1		
Canada Hepburn, SK 2006 (Smart) 258736/R06475 2006/7011065	WG 700	0.021	0.010	1	Crop 25 in tall (51–62)	60	seed	< 0.05	< 0.05	< 0.1		
USA Pepin, WI 2005 (CDC 2464) 138146/R05080 2006/7006733	WG 700	0.020	0.011	1	Inflorescence or flower buds visible	60	seed	0.12	< 0.05	0.17		
USA Stutsman, MD 2005 (CDC 2464) 138146/R05081 2006/7006733	WG 700	0.021	0.017	1	First flower petals visible (in petalled form)	60	seed	0.12	< 0.05	0.17		
USA Brown, SD 2005 (CDC 2464) 138146/R05082 2006/7006733	WG 700	0.020	0.017	1	First flowers open	60	seed	0.065	< 0.05	0.11		

Mix formulation: SL 31.3 g/L (imazapyr of 14.2 g/L); WG 360 g/kg (imazethapyr of 350 g/kg)

Soya bean (dry)

Residue trials on soya beans were conducted in EU countries and Canada during the growing season of 1994 and 2007–2011. SL or WG formulation was used at various rates of 0.035–0.071 kg ai/ha with a pre- or post-emergence application. Soya bean seeds were analysed for the determination of imazamox and the metabolites.

Table 63 Residues of imazamox in soya bean (dry) from supervised field trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in soya bean (dry), mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
N-France Piney 2011 (Sigalia)	SC 22.4	0.042	0.021	1	(25–55)	104	seed	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in soya bean (dry), mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
402845// L110243 2011/1112578												
N-France Cigogné 2011 (Protina) 402845// L110244 2011/1112578	SC 22.4	0.042	0.021	1	(69)	69	seed	< 0.01	< 0.01	< 0.02	0.04	< 0.01
N-France ^a Burgundy 2008 (Essor) B89774// A/NF/H/08/48 2008/1034456	SC 22.4	0.043	0.021	1	(13)	90	seed	< 0.01	< 0.01	< 0.02		
N-France ^a Burgundy 2008 (Essor) B89774// A/NF/H/08/49 2008/1034457	SC 22.4 + Dash	0.043	0.021	1	(13)	90	seed	< 0.01	< 0.01	< 0.02		
Germany Limburgerhof 2011 (Merlin) 402845// L110241 2011/1112578	SC 22.4	0.042	0.021	1	(25)	78	seed	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
Germany Lentzke 2011 (Merlin) 402845// L110242 2011/1112578	SC 22.4	0.042	0.021	1	(21–51)	98	seed	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
S-France Saint-Paul-Les- Romans 2007 (Quito) B42941// A/SF/H/07/142 2007/1023134	SC 22.4	0.042	0.021	1	(13)	90	seed	< 0.01	< 0.01	< 0.02		
S-France Saint-Paul-Les- Romans 2007 (Quito) B44111// A/SF/H/07/165 2007/1028359	SC 22.4 + Dash	0.045	0.021	1	(13)	90	seed	< 0.01	< 0.01	< 0.02		
S-France Bevons 2007 (Deka Big) B42941// A/SF/H/07/143 2007/1023134	SC 22.4	0.040	0.021	1	(13)	104	seed	< 0.01	< 0.01	< 0.02		
S-France Bevons 2007	SC 22.4	0.042	0.021	1	(13)	104	seed	< 0.01	< 0.01	< 0.02		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in soya bean (dry), mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
(Deka Big) B4411// A/SF/H/07/166 2007/1023134	+ Dash											
Italy Gorgonzola 2007 (M10, Pioneer) B42941// A/IT/H/07/144 2007/1023134	SC 22.4	0.043	0.011	1	(23–24)	88	seed	< 0.01	< 0.01	< 0.02		
Italy Gorgonzola 2007 (M10, Pioneer) B44111// A/IT/H/07/67 2007/1028359	SC 22.4 + Dash	0.042	0.011	1	(23–24)	88	seed	< 0.01	< 0.01	< 0.02		
Italy Liscate 2007 (B92, Pioneer) B42941// A/IT/H/07/145 2007/1023134	SC 22.4	0.042	0.011	1	(24)	85	seed	< 0.01	< 0.01	< 0.02		
Italy Liscate 2007 (B92, Pioneer) B44111// A/IT/H/07/168 2007/1028359	SC 22.4 + Dash	0.042	0.011	1	(24)	85	seed	< 0.01	< 0.01	< 0.02		
Canada Wallacetown 1994 (n.r.) RES 95-157// 101355 ID-720-015	WG 700	0.035		1	Pre- emergence	119	bean			< 0.05		
		0.070		1	Pre- emergence	119	bean			< 0.05		
		0.035		1	Post- emergence	112	bean			< 0.05		
		0.070		1	Post- emergence	112	bean			< 0.05		
Canada Georgetown 1994 (AC Bravor) RES 95-048// XP94CN07 ID-720-020	WG 700	0.071		1	Post- emergence	106	dry seed			< 0.05		

Mix formulation: SC 22.4 g/L (bentazone of 480 g/L)

In the trials conducted in Canada, imazamox and CL 263284 were measured as one chromatographic peak representing a total imazamox-related residue.

^a Not independent trials

Cereals

Rice

Residue trials were conducted with rice plant water (imidazolinone-tolerance variety) in Italy in 2004–2012. SL formulation was used at various rates of 0.035–0.075 kg ai/ha at BBCH 13–31. In the all trial, the flooding was interrupted a few days before treatment. Applications were carried out on humid soil but without water in the paddy rice. Rice grain was analysed for the determination of imazamox and the metabolites.

Table 64 Residues of imazamox in imidazolinone-tolerant rice from supervised field trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in rice, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Italy 2004 (n.r.) A-49-04-30//n.r. 2004/1025824 2004-1026636	SL 40	0.070	n.r.	1	25–30	n.r.	paddy rice brown rice milled rice	< 0.05 < 0.05 < 0.05				
	SL 40 + Dash	0.050	n.r.	1	25–30	n.r.	paddy rice brown rice milled rice	< 0.05 < 0.05 < 0.05				
	SL 40	0.035	n.r.	2	13–14 25–31 Interval, 15–25 days	n.r.	paddy rice brown rice milled rice	< 0.05 < 0.05 < 0.05				
	SL 40 + Dash	0.035	n.r.	2	13–14 25–31 Interval, 15–25 days	n.r.	paddy rice brown rice milled rice	< 0.05 < 0.05 < 0.05				
Italy Salussola 2011 (Sirio CL) 407231//L110320 2012/1278352	SL 40	0.075	0.038	1	24–25	90	grain	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
	SL 40 + Dash	0.075	0.038	1	24–25	90	grain	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
	SL 40 + Dash	0.038	0.019	2	13–14 24–25 Interval, 13 days	90	grain	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
Italy Zeme 2011 (Sirio CL) 407231// L110321 2012/1278352	SL 40	0.075	0.038	1	25	78	grain	0.01	0.04	0.05	0.02	< 0.01
	SL 40 + Dash	0.075	0.038	1	25	78	grain	0.02	0.05	0.07	0.03	< 0.01

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in rice, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
	SL 40 + Dash	0.038	0.019	2	13–14 25 Interval, 31 days	78	grain	< 0.01	0.03	0.04	0.02	< 0.01
Italy Zibido 2012 (CL 71) 429716// L120440 2012/1272623	SL 40 + Dash	0.075	0.038	1	25	85	grain	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
	SL 40 + Dash	0.038	0.019	2	13, 25 Interval, 37 days	85	grain	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
Italy Salussola 2012 (CL 71) 429716// L120441 2012/1272623	SL 40 + Dash	0.075	0.038	1	25	90	grain	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
	SL 40 + Dash	0.038	0.019	2	13, 25 Interval, 37 days	90	grain	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
Spain ^a Los Palacios 2011 (Sirio) 407231// L110322 2012/1278352	SL 40	0.075	0.038	1	25	84	grain	< 0.01	0.03	0.04	0.02	< 0.01
	SL 40 + Dash	0.075	0.038	1	25	84	grain	< 0.01	0.03	0.04	0.01	< 0.01
	SL 40 + Dash	0.038	0.019	2	13–14, 25 Interval, 18 days	84	grain	< 0.01	0.03	0.04	0.01	< 0.01
Spain ^a Los Palacios 2011 (Sirio) 407231// L110323 2012/1278352	SL 40	0.075	0.038	1	25	84	grain	< 0.01	0.02	0.03	0.01	< 0.01
	SL 40 + Dash	0.075	0.038	1	25	84	grain	< 0.01	0.04	0.05	0.02	< 0.01
	SL 40 + Dash	0.038	0.019	2	13–14, 25 Interval, 18 days	84	grain	< 0.01	0.01	0.02	0.01	< 0.01
Spain ^b Los Palacios 2012	SL 40	0.075	0.038	1	25	77	grain	0.01	0.05	0.06	0.02	< 0.01

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in rice, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
(Sirio) 429716// L120442 2012/1272623	+ Dash											
	SL 40 + Dash	0.038	0.019	2	13, 25 Interval, 20 days	77	grain	< 0.01	0.02	0.03	< 0.01	< 0.01
Spain ^b Los Palacios 2012 (Sirio) 429716 2012/1272623 L120443	SL 40 + Dash	0.075	0.038	1	25	84	grain	0.01	0.04	0.05	0.02	< 0.01
	SL 40 + Dash	0.038	0.019	2	13, 25 Interval, 17 days	84	grain	< 0.01	0.01	0.02	< 0.01	< 0.01

n.r.: Not reported

w. plant: Whole plant without roots

^a Not independent trials^b Not independent trials

Wheat

During the growing season of 1995–1998, residue trials were conducted with wheat of imidazolinone-tolerant variety in France, Italy, Canada and USA. Treatments were made post-emergent with various formulations at rates of 0.020–0.075 kg ai/ha. Wheat grain samples were analysed for the determination of imazamox and the metabolites.

Table 65 Residues of imazamox in imidazolinone-tolerant wheat from supervised residue trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat, mg/kg		
								Parent	CL 263284	Parent + CL 263284
N-France Les Mureaux 1996 (Fidel) ID-FR-96-617// 96-617-323 ID-730-023	SL 40	0.075	0.021	1	25 5 tillers detectable	129	grain	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.018	1	25 5 tillers detectable	129	grain	< 0.05	< 0.05	< 0.1
N-France Pignicourt 1996 (Fidel) ID-FR-96-617// 96-617-465 ID-730-023	SL 40	0.075	0.025	1	25–29 5 to 9 or more tillers detectable	125	grain	< 0.05	< 0.05	< 0.1
	EC	0.067	0.022	1	25–29	125	grain	< 0.05	< 0.05	< 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat, mg/kg		
								Parent	CL 263284	Parent + CL 263284
	16.7				5 tillers detectable- beginning of stem elongation					
N-France Cassignas 1995 (Fidel) ID-FR-95-005// ID-730-034	SL 120	0.075	0.019	1	31 first node detectable	118	grain	< 0.05	< 0.05	< 0.1
N-France Montchauvet 1996 (Fidel) ID-FR-96-616// 96-616-322 ID-730-035	SL 40	0.075	0.021	1	23–29 3 tillers detectable – end of tillering	121	grain	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.028	1	23–29	121	grain	< 0.05	< 0.05	< 0.1
N-France Tilloy-Bellay 1996 (Fidel) ID-FR-96-616// 96-616-464 ID-730-035	SL 40	0.075	0.032	1	25–27 5–7 tillers detectable	146	grain	< 0.05	< 0.05	< 0.10
	EC 16.7	0.067	0.018	1	25–27	146	grain	< 0.05	< 0.05	< 0.1
S-France ^a Cassignas 1996 (Fidel) ID-FR-96-619// 96-619-291 ID-730-022	SL 40	0.075	0.020	1	26–28 6–8 tillers detectable	131	grain	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.018	1	26–28 6–8 tillers detectable	131	grain	< 0.05	< 0.05	< 0.1
S-France ^a Cassignas 1996 (Fidel) ID-FR-96-619// 96-619-292 ID-730-022	SL 40 g/L	0.075	0.020	1	27–29 7 tillers detectable- end of tillering	118	grain	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.018	1	27–29 7 tillers detectable- end of tillering	118	grain	< 0.05	< 0.05	< 0.1
S-France Montchauvet 1995 (Fidel) ID-FR-95-005// 95-005-318 ID-730-034	SL 120	0.075	0.019- 0.025	1	30 beginning of stem elongation	140	grain	< 0.05	< 0.05	< 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat, mg/kg		
								Parent	CL 263284	Parent + CL 263284
S-France Bourgogne 1995 (Fidel) ID-FR-95-005// 95-005-484 ID-730-034	SL 120	0.075	0.019- 0.025	1	30	135	grain	< 0.05	< 0.05	< 0.1
Italy Bologna 1996 (Fidel) ID-IT-96-618// 96-618-01 ID-730-019	SL 40	0.075	0.016	1	25 (5 tillers detectable)	102	grain	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.016	1	25	102	grain	< 0.05	< 0.05	< 0.1
Italy Pisa 1996 (Fidel) ID-IT-96-618// 96-618-02 ID-730-019	SL 40 g/L	0.075	0.016	1	25	103	grain	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.016	1	25	103	grain	< 0.05	< 0.05	< 0.1
Italy Bologna 1996 (Fidel) ID-IT-96-615// 96-615-01 ID-730-020	SL 40	0.075	0.016	1	25 5 tillers detectable	95	grain	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.016	1	25 5 tillers detectable	95	grain	< 0.05	< 0.05	< 0.1
Italy Bologna 1997 (Fidel) ID-IT-97-808// 97-808-01 ID-730-021	SL 40	0.075	0.016	1	29 end of tillering	98	grain	< 0.05	< 0.05	< 0.1
Canada Minto 1997 (SWP 965-001) RES 98-001// XP97CN01 ID-730-015	WG 700 + Merge	0.020	0.020	1	5–6 leaf stage	89	grain	< 0.05	< 0.05	< 0.1
	WG 700 + Merge	0.040	0.040	1	5–6 leaf stage	89	grain	< 0.05	< 0.05	< 0.1
Canada Aberdeen 1997 (SWP 965-001) RES 98-005// XP97CN05 ID-730-016	WG 700 + Merge	0.020		1	3–6 leaf stage	72	grain	< 0.05	< 0.05	< 0.1
	WG	0.040		1	3–6 leaf	72	grain	< 0.05	< 0.05	< 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat, mg/kg		
								Parent	CL 263284	Parent + CL 263284
	700 + Merge				stage					
Canada Saskatoon 1997 (SWP 965-001) RES 98-002// XP97CN02 ID-730-017	WG 700 + Merge	0.020		1	4-6 leaf stage	79	grain	< 0.05	< 0.05	< 0.1
	WG 700 + Merge	0.040		1	4-6 leaf stage	79	grain	< 0.05	< 0.05	< 0.1
Canada Barnwell 1997 (SWP 965-001) RES 98-003// XP97CN03 ID-730-018	WG 700 + Merge	0.020		1	4-6 leaf stage	69	grain	< 0.05	< 0.05	< 0.1
	WG 700 + Merge	0.040		1	4-6 leaf stage	69	grain	< 0.05	< 0.05	< 0.1
Canada Nisku 1997 (SWP 965-001) RES 98-006// XP97CN06 ID-730-028	WG 700 + Merge	0.020		1	6 leaf stage	90	grain	< 0.05	< 0.05	< 0.1
	WG 700 + Merge	0.040		1	6 leaf stage	90	grain	< 0.05	< 0.05	< 0.1
USA Northwood, ND 1998 (NDGRDN01) RES 99-094// XP98ND04 ID-730-038	SC 11.5% + X-77	0.067	0.067	1	5 leaf stage	82	grain	< 0.05	< 0.05	< 0.1
	SC 11.5% + X-77	0.20	0.20	1	5 leaf stage	82	grain	< 0.05	< 0.05	< 0.1
	SC 11.5% + X-77	0.32	0.32	1	5 leaf stage	82	grain	0.053	< 0.05	0.1

Mix formulation: EC 16.7 g/L (pendimethalin of 250 g/L)

PE: pre-treatment

^a Not independent trials

*Oilseed crop**Peanut*

Residue trials were conducted with peanut in Australia and Brazil. Treatments were made with various formulations at rates of 0.056–0.072 kg ai/ha. Peanut samples were analysed for the residues of imazamox and the metabolites.

Table 66 Residues of imazamox in peanut from supervised field trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in peanut, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
AUS Kingaroy 1994 (n.r.) TTR-96-010// ID-720-034	100A	0.072	0.072	1	n.r.	130	nut shell	in	< 0.05			
AUS Kingaroy 1994 (n.r.) TTR-96-011// ID-720-035	WG 700	0.072	0.072	1	n.r.	130	nut shell	in	< 0.05			
AUS Norwin 1993/1994 (n.r.) TTR-96-012//n.r. ID-720-036	SL 120	0.072	0.072	1	n.r.	171	nut shell	in	< 0.05			
Brazil Jaboticabal 2011/2012 (IAC 505) 382673// G100726 2013/3000444	SL 28	0.056	0.028	1	81 79 79	36 43 50	seeds seeds seeds		< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	0.01 0.02 0.05
Brazil Candido Rodrigues 2011/2012 (IAC 505) 382673// G100727 2013/3000444	SL 28	0.056	0.028	1	81 77 77	36 43 50	seeds seeds seeds		< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01
Brazil Santo Antônio de Posse 2011/2012 (Tatu) 382673// G100730 2013/3000444	SL 28	0.056	0.028	1	79	43	seeds		< 0.01	< 0.01	< 0.02	0.07
Brazil Uberlândia 2011/2012 (Tatu) 382673// G100733	SL 28	0.056	0.028	1	79 75 71	36 43 50	seeds seeds seeds		< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in peanut, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
2013/3000444												
Brazil Senador Canedo 2012 (Ranner) 382673// G100734 2013/3000444	SL 28	0.056	0.028	1	75	43	seeds	< 0.01	< 0.01	< 0.02	< 0.01	

Mix formulation: SL 28 g/L (bentazone of 600 g/L)

n.r.: Not reported

Rape seed

Residue trials were conducted with rape plant of imidazolinone-tolerant variety (Imi-res.) in European countries, Argentina and Canada in 1994–1998 and 2005–2009. Treatments were made post-emergent with SL, SC, or WG formulations at various rates of 0.020–0.080 kg ai/ha. Rape seed samples were analysed for residues of imazamox and the metabolites.

Table 67 Residues of imazamox in imidazolinone-tolerant rape seed from supervised residue trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in rape seed, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
N-France Boisset-Les- Prevanches 1996/97 (Imi-res., NW 4602) ID-FR-97-667// 97-667-301 ID-750-009	SL 40	0.073		1	(16–17)	236	seed			< 0.05 ^c		
N-France Mousseaux- Neuville 1996/97 (Imi-res., NW4602) ID-FR-97-667// 97-667-302 ID-750-009	SL 40	0.075		1	(17–18)	221	seed			< 0.05 ^c		
N-France Caillouet-Orgeville 1996 (Imi-res., Pioneer 46A72) ID FR 96 663// 96-663-385 ID-750-006	SL 40	0.075		1	(13–14)	74	seed			< 0.05 ^c		
N-France Herpy 1996 (Imi-res., 46A72) ID FR 96 663// 96-663-469 ID-750-006	SL 40	0.075		1	(12–14)	87	seed			< 0.05 ^c		
Germany Uetza 2005/2006	SC 25	0.050		1	(51)	101	seed	< 0.05	< 0.05	< 0.1		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in rape seed, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
(PS22-1A-VH) 211921// AF/10114/BA/3 2007/1007939												
Germany Uetza 2005/2006 (PS22-1A-VH) 251536// AF/10475/BA/3 2005/2006 2007/1007963	SC 25 + Dash	0.050		1	(51)	101	seed	< 0.05	< 0.05	< 0.1		
Germany Schwienau/Melzingen 2005/2006 (PS22-1A-VH) 211921// AF/10114/BA/4 2007/1007939	SC 25	0.050		1	(51)	100	seed	< 0.05	< 0.05	< 0.1		
Germany Schwienau/Melzingen 2005/2006 (PS22-1A-VH) 251536// AF/10475/BA/4 2007/1007963	SC 25 + Dash	0.050		1	(51)	100	seed	< 0.05	< 0.05	< 0.1		
Germany Euskirchen-Dom Esch 1997/98 (Imi-res., NW 4200) ID-GE-98-403// 98-403-01 ID-750-017	SL 40	0.075	0.019	1	(18) 8 leaves unfolded	254	seed			< 0.05 ^c		
Germany Billingsdorf Flurnummer 1997/98 (Imi-res., NW 4200) ID-GE-98-403// 98-403-02 1997/98 ID-750-017	SL 40	0.077	0.025	1	(17) 7 leaves unfolded	262	seed			< 0.05 ^c		
UK ^a Sowerby, Thirsk 1996 (Imi-res., 45A71) ID UK 96 661// 96-661-01 ID-750-004	SL 40	0.075		1	(13) 3 leaves unfolded	93	seed			< 0.05 ^c		
UK ^b Sessay, Thirsk 1996 (Imi-res., 45A71) ID UK 96 661// 96-661-04 ID-750-004	SL 40	0.075		1	(14) 4 leaves unfolded	93	seed			< 0.05 ^c		
UK ^a Sowerby, Thirsk	SL 40	0.075		1	(13)	93	seed	< 0.05	< 0.05	< 0.05 ^c		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in rape seed, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
1996 (Imi-res., 45A71) ID UK 96 662// 96-662-01 ID-750-005												
UK ^b Sessay, Thirsk 1996 (Imi-res., 45A71) ID UK 96 662// 96-662-04 ID-750-005	SL 40	0.075		1	(14)	93	seed	< 0.05	< 0.05	< 0.05 ^c		
UK Northamptonshire 1996/97 (Imi-res., Pioneer 4601) ID-UK-97-681 97-681-01 ID-750-008	SL 40	0.078		1	(13–16) 3–6 leaves	240	seed			< 0.05 ^c		
	SL 40 + amm. sulfate	0.078		1	(13–16)	240	seed			< 0.05 ^c		
UK Suffolk 1996/97 (Imi-res., Pioneer 4601) ID-UK-97-681// 97-681-02 ID-750-008	SL 40 g/L	0.079		1	(13–16) 3–6 leaves	238	seed			< 0.05 ^c		
	SL 40 + amm. sulfate	0.079		1	(13–16)	238	seed			< 0.05 ^c		
S-France La francaise 2005/2006 (PS22-1A-VH) 211921// AF/10114/BA/1 2007/1007939	SC 25	0.050		1	(61)	78	seed	< 0.05	< 0.05	< 0.1		
S-France La francaise 2005/2006 (PS22-1A-VH) 251536// AF/10475/BA/1 2007/1007939	SC 25 + Dash	0.050		1	(61)	78	seed	< 0.05	< 0.05	< 0.1		
S-France Montauban 2005/2006 (PS22-1A-VH) 211921// AF/10114/BA/2 2007/1007939	SC 25	0.050		1	(53)	83	seed	< 0.05	< 0.05	< 0.1		
S-France	SC	0.050		1	(53)	83	seed	< 0.05	< 0.05	< 0.1		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in rape seed, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Montauban 2005/2006 (PS22-1A-VH) 251536// AF/10475/BA/2 2007/1007939	25 + Dash											
S-France Saint-Brice 1997/98 (Imi-res., NW 4200) ID-FR-98-401// 98-401-297 ID-750-016	SL 40 g	0.075		1	(17-18) 7-8 leaves unfolded	246	seed			< 0.05 ^c		
S-France Martres 1997/98 (Imi.-res., NW 4200) ID-FR-98-401// 98-401-298 ID-750-016	SL 40	0.075		1	(14-17) leaves unfolded	243	seed			< 0.05 ^c		
S-France Yvrac 1997/98 (Imi-re s., NW 4200) ID-FR-98-401// 98-401-299 1997/98 ID-750-016	SL 40	0.075		1	(18-19)	244	seed			< 0.05 ^c		
S-France Martres 1996/97 (Imi-res., NW4602) ID-FR-97-683// 97-683-294 ID-750-007	SL 40	0.075		1	(16-17)	220	seed			< 0.05 ^c		
S-France Cassignas 1996/97 (Imi-res., NW4602) ID-FR-97-683// 97-683-296 ID-750-007	SL 40	0.075		1	(16-18)	217	seed			< 0.05 ^c		
S-France Laroque Timbaut 1996/97 (Imi-res., NW4602) ID-FR-97-683// 97-683-297 ID-750-007	SL 40	0.075		1	(16-17)	212	seed			< 0.05 ^c		
S-France St Quentin De Baron 1996/97 (Imi-res., NW4602) ID-FR-97-683// 97-683-298 ID-750-007	SL 40	0.075		1	(17-18)	210	seed			< 0.05 ^c		
S-France Frespech 1996/97 (Imi- res., NW4602)	SL 40	0.075		1	(16-17)	210	seed			< 0.05 ^c		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in rape seed, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
ID-FR-97-683// 97-683-299 ID-750-007												
Argentina Venado Tuerto 2009 (CL rape: Advanta) 420295// G110284 2012/3001643	WG 700	0.050		1		84	seed	< 0.01	< 0.01	< 0.02	< 0.01	
	WG 700	0.10		1		84	seed	< 0.01	< 0.01	< 0.02	< 0.01	
Argentina San Jeronimo 2009 (CL rape: Advanta) 420295// G110285 2012/3001643	WG 700 g/kg	0.050		1	(14–17)	83	seed	< 0.01	< 0.01	< 0.02	< 0.01	
	WG 700	0.10		1		83	seed	< 0.01	< 0.01	< 0.02	< 0.01	
Canada Estlin 19994 (n.r.) RES 95-113// 101086 ID-750-003	WG 700	0.020		1		83	seed			< 0.05 ^c		
	WG 700	0.040		1		83	seed			< 0.05 ^c		
	WG 700	0.080		1		83	seed			< 0.05 ^c		
	WG 700	0.20		1	(18–19)	83	seed			< 0.05 ^c		
Canada Stirling 1994 (n.r.) RES 95-113// 101088 ID-750-003	WG 700	0.020		1		93	seed			< 0.05 ^c		
	WG 700	0.040		1		93	seed			< 0.05 ^c		
	WG 700	0.080		1		93						
Canada Nisku 1994 (n.r.) RES 95-113// 101089 ID-750-003	WG 700	0.020		1		95	seed			< 0.05 ^c		
	WG 700	0.040		1	Post- emergence	95	seed			< 0.05 ^c		
	WG	0.080		1	Post-emerg.	95	seed			< 0.05 ^c		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in rape seed, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
	700											
Canada Rosthern 1994 (n.r.) RES 95-113// 101057 ID-750-003	WG 700	0.020		1	Post- emergence	86	seed			< 0.05 ^c		
Canada Carman 1994 (n.r.) RES 95-113// 50128 ID-750-003	WG 700	0.020		1	Post- emergence	70	seed			< 0.05 ^c		
	WG 700	0.040		1	Post- emergence	70	seed			< 0.05 ^c		

w. plant: Whole plant without roots

^a Not independent trials

^b Not independent trials

^c Imazamox and CL 263284 were measured as one chromatographic peak representing a total imazamox-related residue.

Sunflower seed

Residue trials on sunflower seed (imidazolinone-tolerance variety) were conducted in European countries, Turkey, Argentina, Canada and USA in 2002–2012. Foliar application was made post-emergent with SL, SC or WG formulation at various rates of 0.015–0.081 kg ai/ha. Rape seed samples were analysed for residues of imazamox and the metabolites.

Table 68 Residues of imazamox in imidazolinone-tolerant sunflower from supervised field trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in sunflower, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
N-France Seebach 2003 (IMA-tol., DK 3900 CL) 166093//FAN/28/03 2004/1000754	SL 40	0.049	0.017	1	(14)	16 38 99	w. plant w. plant seed	0.16 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	0.21 < 0.1 < 0.1		
N-France Thorée les Pins 2003 (IMA-tol., CMS425xRHA426 SF02) 166093//FBM/18/03 2004/1000754	SL 40	0.051	0.017	1	(18)	8 37 98	w. plant w. plant seed	0.09 < 0.05 < 0.05	0.07 < 0.05 < 0.05	0.16 < 0.1 < 0.1		
N-France Pays de la Loire 2012 (Paraiso 1000) 417820//L120331 2013/1003728	SL 25	0.050		1	(18)	0 121 121	w. plant r. plant seed	3.30 < 0.01 < 0.01	0.01 < 0.01 0.02	3.31 < 0.02 0.03	< 0.01 < 0.01 0.07	0.01 < 0.01 < 0.01
	SL	0.050		1	(18)	0	w. plant	2.40	< 0.01	2.41	< 0.01	0.02

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in sunflower, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
	40 + Dash					121 121	r. plant seed	< 0.01 < 0.01	< 0.01 0.01	< 0.02 0.02	< 0.01 0.05	< 0.01 < 0.01
Germany Halen 2011 (IMA-tol., LN 11180CL plus) 407229//L110316 2012/1084183	SL 40	0.050	0.03	1	(18)	0 112 112	w. plant r. plant seed	1.74 < 0.01 < 0.01	< 0.01 < 0.01 0.02	1.75 < 0.02 0.03	< 0.01 < 0.01 0.03	< 0.01 < 0.01 < 0.01
	SL 40 + Dash	0.050		1	(18)	0 112 112	w. plant r. plant seed	1.60 < 0.01 < 0.01	< 0.01 < 0.01 0.02	1.61 < 0.02 0.03	< 0.01 < 0.01 0.03	< 0.01 < 0.01 < 0.01
Germany Manker Brandenburg 2012 (Paraiso 1000) 417820//L120330 2013/1003728	SL 25	0.050		1	(18)	0 118 118	w. plant r. plant seed	2.40 < 0.01 < 0.01	0.01 < 0.01 0.07	2.41 < 0.02 0.08	< 0.01 < 0.01 0.12	< 0.01 < 0.01 < 0.01
	SL 40 + Dash	0.050		1	(18)	0 118 118	w. plant r. plant seed	2.60 < 0.01 < 0.01	0.01 < 0.01 0.07	2.61 < 0.02 0.08	< 0.01 < 0.01 0.13	< 0.01 < 0.01 < 0.01
UK Banbury 2011 (IMA-tol., LN 11180 CL plus) 407229//L110317 2012/1084183	SL 40	0.050	0.03	1	(18)	0 117 117	w. plant r. plant seed	4.32 < 0.01 < 0.01	0.01 < 0.01 0.01	4.33 < 0.02 0.02	< 0.01 < 0.01 0.02	0.01 < 0.01 < 0.01
	SL 40 + Dash	0.050	0.03	1	(18)	0 117 117	w. plant r. plant seed	4.34 < 0.01 < 0.01	< 0.01 < 0.01 0.02	4.35 < 0.02 0.03	< 0.01 < 0.01 0.02	0.01 < 0.01 < 0.01
S-France Tourtres 201 (IMA-tol., LN 11180 CL plus) 407229//L110318 2012/1084183	SL 40	0.050	0.03	1	(18)	0 111 111	w. plant r. plant seed	2.78 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	2.79 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
	SL 40 + Dash	0.050	0.03	1	(18)	0 111 111	w. plant r. plant seed	2.47 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	2.48 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
Italy Chiesuol Del Fosso 2011 (IMA-tol., LN 11180 CL plus) 407229//L110319 2012/1084183	SL 40	0.050	0.03	1	(18)	0 117 117	w. plant r. plant seed	2.96 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	2.97 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
	SL 40 + Dash	0.050		1	(18)	0 117 117	w. plant r. plant seed	3.20 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	3.21 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	0.02 < 0.01 < 0.01
Italy Altredo Emilia Romagna 2012 (Paraiso 1001) 417820//L120333 2013/1003728	SL 25	0.050		1	(18)	0 114 114	w. plant r. plant seed	4.80 < 0.01 < 0.01	0.01 < 0.01 < 0.01	4.81 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	0.02 < 0.01 < 0.01

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in sunflower, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
	SL 40 + Dash	0.050		1	(18)	0 114 114	w. plant r. plant seed	2.90 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	2.91 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
Spain Utrera Andaluca 2012 (Paraiso 1001) 417820//L120332 2013/1003728	SL 25	0.050		1	(18)	0 107 107	w. plant r. plant seed	6.60 < 0.01 < 0.01	< 0.01 < 0.01 0.02	6.61 < 0.02 0.03	< 0.01 < 0.01 0.05	0.01 < 0.01 < 0.01
	SL 40 + Dash	0.050		1	(18)	0 107 107	w. plant r. plant seed	2.70 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	2.71 < 0.02 < 0.02	< 0.01 < 0.01 0.02	< 0.01 < 0.01 < 0.01
Spain Los Palacios 2002 (Mycogene X 81359) 142855//ALO/22/02 2002/1012087	SL 40	0.040	0.013	1	(14)	17 46 103	w. plant w. plant seed	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1		
Spain Utrera 2002 (Mycogene X 81359) 142855//ALO/23/02 2002/1012087	SL 40	0.040	0.013	1	(16)	17 46 110	w. plant r. plant seed	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1		
Spain El Coroni 2002 (Mycogene X 81359) 142855//AYE/16/02 2002/1012087	SL 40	0.040	0.013	1	(15)	14 43 91	w. plant r. plant seed	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1		
Spain Alcala del Rio 2002 (Mycogene X 81359) 142855//AYE/17/02 2002/1012087	SL 40	0.040	0.013	1	(16)	20 36 83	w. plant r. plant seed	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1		
Turkey n.r. (n.r.) 148288// 2002/1020471	SC 33	0.041		1	4-leaf stage	n.r. n.r. n.r.	seed seed seed	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1		
		0.041		1	8-leaf stage	n.r. n.r. n.r.	seed seed seed	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1		
		0.081		1	4-leaf stage	n.r. n.r. n.r.	seed seed seed	0.15 0.11 0.10	0.10 0.07 0.08	0.25 0.18 0.18		
		0.081		1	8-leaf stage	n.r. n.r. n.r.	seed seed seed	0.13 0.08 0.11	0.10 < 0.05 0.07	0.23 0.13 0.18		
Argentina Balcarce Buenos Aires 2007/2008 (f) 2008/2009 ^d	WG 700	0.040		1	V3–V4	106 ^c 105 ^d	seed ^c seed ^d	< 0.01 < 0.01	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in sunflower, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
(n.r.) 310642//G100057 2010/1057139												
	WG 700	0.080		1	V3–V4	106 ^c 105 ^d	seed ^c seed ^d	< 0.01 < 0.01	0.02 0.02	0.03 0.03	0.03 0.04	
Argentina Tandil, Buenos Aires 2007/2008 ^c 2008/2009 ^d (n.r.) 310642//G100062 2010/1057139	WG 700	0.040		1	V3–V4	106 ^c 105 ^d	seed ^c seed ^d	< 0.01 < 0.01	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 0.01	
	WG 700	0.080		1	V3–V4	106 ^c 105 ^d	seed ^c seed ^d	< 0.01 < 0.01	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	
Argentina Realicó, Lapampa 2007/2008 ^c 2008/2009 ^d (n.r.) 310642//G100063 2010/1057139	WG 700	0.040		1	V3–V4	98 ^c 109 ^d	seed ^c seed ^d	0.02 < 0.01	0.04 < 0.01	0.06 < 0.02	0.09 0.02	
	WG 700	0.080		1	V3–V4	98 ^c 109 ^d	seed ^c seed ^d	0.03	0.09	0.12	0.20	
Canada Grey, MB 2007 (Jaguar) 261967// R070268 2008/7008101	WG 350 + Merge	0.015		1	(61–65)	59	seed	0.07	0.06	0.13		
Canada Grant, SK 2007 (Viper) 261967//R070270 2008/7008101	WG 350 + Merge	0.015		1	(65)	58	seed	0.14	0.16	0.30		
Canada St. Claude, MB 2008 (NuSun 8N358) 327308//R080098 2008/7019225	SL 33	0.019		1	Flowering declining (67)	68	seed	0.05	0.19	0.25		
Canada Portage la Prairie, MB 2008 (Viper) 327308// R080099 2008/7019225	SL 33	0.019		1	Full flowering (65)	49 59 70 82 92	seed seed seed seed seed	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.06 0.09 0.06 0.09 0.10	0.11 0.14 0.11 0.14 0.15		
Canada Dundern, SK 2008 (Viper) 327308// R080100	SL 33	0.020		1	Stem elongation (33–39)	70	seed	< 0.05	< 0.05	< 0.1		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in sunflower, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
2008/7019225												
Canada Wellwood, MB 2008 (Jaguar) 327308//R080101 2008/7019225	SL 33	0.020		1	End of flowering (69)	70	seed	< 0.05	0.06	0.11		
Canada Wellwood, MB 2008 (Viper) 327308//R080102 2008/7019225	SL 33	0.019		1	End of flowering (69)	70	seed	< 0.05	0.06	0.11		
USA Pepin, WI 2007 (8H419CL) 261967//R070269 2008/7008101	WG 350 + Merge	0.017		1	(65)	60	seed	0.05	0.17	0.22		
USA McHenry, ND 2007 (DKF38-80CL) 261967//R070271 2008/7008101	WG 350 + Merge	0.017		1	Late flowering	59	seed	< 0.05	< 0.05	< 0.1		
USA Cass, ND 2007 (Jaguar) 261967//R070272 2008/7008101	WG 350 + Merge	0.034		1	Early bud	40 50 60 69 79	seed seed seed seed seed	0.21 0.14 0.085 0.060 0.095	0.10 0.05 0.07 0.06 0.12	0.31 0.19 0.16 0.12 0.21		
USA Fargo, ND 2000 (IMI sunflower hybrid, CMS HA425/RHA426) 07219//ND02 2002/5004111	SL 11.78% + MSO _b	0.056		1	vegetative ,18- 30 cm	100	seed	< 0.05	< 0.05	< 0.1		< 0.05
USA ^a Minot, ND 2000 (IMI sunflower hybrid, CMS HA425/RHA426) 07219//ND03 2002/5004111	SL 11.78% + MSO _b	0.056		1	vegetative ,18- 30 cm	88	seed	< 0.05	< 0.05	< 0.1		< 0.05
USA ^a Minot, ND 2000 (IMI sunflower hybrid, CMS HA425/RHA426) 07219//ND04 2002/5004111	SL 11.78% + MSO _b	0.056		1	Vegetative,33 cm	90	seed	< 0.05	< 0.05	< 0.1		< 0.05
USA Scottsbluff, NE 2000 (IMI sunflower hybrid, CMS HA425/RHA426) 07219//NE01 2002/5004111	SL 11.78% + MSO _b	0.056		1	Vegetative, 33 cm	77	seed	< 0.05	< 0.05	< 0.1		< 0.05

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in sunflower, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
USA Scottsbluff, NE 2000 (IMI + sunflower hybrid, CMS HA425/RHA426) 07219//NE02 2002/5004111	SL 11.78%	0.056		1	Vegetative, 33 cm	76	seed	< 0.05	< 0.05	< 0.1		< 0.05
USA Fort Collins, CO 2000 (IMI + sunflower hybrid, CMS 425/RHA426) 07219//CO02 2002/5004111	SL 11.78%	0.056		1	vegetative ,30– 46 cm	72	seed	< 0.05	< 0.05	< 0.1		< 0.05

Mix formulation: SC or SL 33 g/L (imazapyr of 15 g/L); WG 350 g/kg (imazethapyr 35%)

w. plant: whole plant without roots

r. plant: rest of plant without roots

MSO: methylated seed oil

^a Not independent trials due to the same trial location and close application time two days separate

^b Tank mix with imazapyr

^c Grown in 2007/2008

^d Grown in 2008/2009

Legume animal feeds

Alfalfa

Residue trials on alfalfa were conducted in Greece, Spain and USA in 1996–1999. Foliar application was made post-emergent with SL, SC or WG formulation at rates of 0.067–0.075 kg ai/ha. The forage and hay samples were collected and analysed for residues of imazamox and the metabolites.

Table 69 Residues of imazamox in alfalfa from supervised residue trials

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in alfalfa, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Greece Partheni, Thessaloniki 1999 (Ipati) ID-HE-99-12// 99-12-01 ID-731-023	SL 40	0.075		1	(19)	21 29 42	w. p. ^a w. p. ^a w. p. ^a	< 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1	< 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1
Spain Lerida 1996 (Aragon) ID-SP-96-613// 96-613-24 ID-731-001	SL 40	0.070	0.023	1	4–6 true leaves (14–16)	35 49 56 63 63	w. p. ^a w. p. ^a w. p. ^a w. p. ^a w. p. ^b	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1		
Spain Basanova,	SL 40	0.073		1	5–7 true leaves	7 21	w. p. ^a w. p. ^a	< 0.1 < 0.1	0.27 < 0.1	0.37 < 0.2	0.28 < 0.1	0.68 < 0.1

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in alfalfa, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Almenar 1997(Aragon) ID-SP-97-805// 97-805-04 ID-731-002					(15-17)	28 35 35	w. p. ^a w. p. ^a w. p. ^b	< 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1	< 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1
Spain Basanova, Almenar 1996 (Aragon) ID-SP-96-612// 96-612-25 ID-731-003	SL 40	0.070		1	4-6 true leaves (14-16)	40 40	w. p. ^a w. p. ^b	< 0.1 < 0.1	< 0.1 < 0.1	< 0.2 < 0.2	< 0.1 < 0.1	< 0.1 < 0.1
Spain Castellsera, Lerida 1996 (Aragon) ID-SP-96-612// 96-612-25 ID-731-003	SL 40	0.070		1	5-7 true leaves (15-17)	40 40	w. p. ^a w. p. ^b	< 0.1 < 0.1	< 0.1 < 0.1	< 0.2 < 0.2	< 0.1 < 0.1	< 0.1 < 0.1
USA Northwood, ND 1997 (Vernal) RES 99-006// XP97ND05 ID-731-004	WG 700	0.067		1	10-15 cm early season regrowth	27 27 56 56 99 99	forage hay forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.2 < 0.2 < 0.2 < 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1
USA Kerman, CA 1998 (Cuff) RES 99-048// XP98CA02 ID-731-007	SL 11.5%	0.067		1	post- emergence 25-36 cm plant height	PE PE 0 0 7 7 14 14 20 20 50 50 79 79	forage hay forage hay forage hay forage hay forage hay forage hay forage hay	< 0.1 < 0.1 3.9 6.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 0.32 3.3 0.13 0.48 < 0.1 0.28 0.42 < 0.2 < 0.2 < 0.2 < 0.2 < 0.2 < 0.2	< 0.2 < 0.2 4.6 10 0.25 0.60 < 0.2 0.28 0.14 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 0.15 0.73 < 0.1 0.14 0.36 < 0.1 0.10 0.16 < 0.1 < 0.1 < 0.1 < 0.1	< 0.10 < 0.10 0.21 2.9 0.22 0.73 < 0.1 0.36 0.36 < 0.1 0.16 0.16 < 0.1 < 0.1 < 0.1 < 0.1
USA Barnard, SD 1998 (DeKalb DK 127) RES 99-088// XP98SD01 ID731-009	WG 70.7%	0.068		1	10-20 cm plant height regrowth	26 26 56 56 96 96	forage hay forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.2 < 0.2 < 0.2 < 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1
USA Noblesville, IN 1998 (Blazer) RES 99-086// XP98IN02 ID-731-010	WG 70.7%	0.067		1	5 cm size regrowth	36 36 92 92 155 155	forage hay forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.2 < 0.2 < 0.2 < 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1
USA New Holland,	SL 11.5%	0.067		1	13 cm plant height	22 22	forage hay	< 0.1 < 0.1	< 0.1 < 0.1	< 0.20 < 0.20	< 0.1 < 0.1	< 0.1 0.13

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in alfalfa, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
OH 1998 (Mustang) RES 99-087// XP98OH02 ID-731-013						48 48 80 80	forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1	< 0.20 < 0.20 < 0.20 < 0.20	< 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1
USA Ephrata, WA 1997 (89-31 WL 323 Foundation) RES 99-092// XP97WA03 ID-731-014	SL 11.9%	0.071		1	4 th – 5 th trifoliolate regrowth stage	30 30 66 66 105 105	forage hay forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.2 < 0.2 < 0.2 < 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 0.14 < 0.1 < 0.1 < 0.1 < 0.1
USA Germansville, PA 1997 (WL-322 HQ) RES 98-237// XP97PA01 ID-731-017	SL 11.9%	0.067		1	10–15 cm regrowth	27 27 57 57 89 89	forage hay forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 0.29 < 0.1 < 0.1 < 0.1 < 0.1	< 0.2 0.41 < 0.2 < 0.2 < 0.2 < 0.2	< 0.1 0.58 < 0.1 < 0.1 < 0.1 < 0.1	0.33 0.92 < 0.1 < 0.1 < 0.1 < 0.1
USA Hawkinsville, GA 1997 (Alfagraze) RES 99-093// XP97GA01 ID-731-022	WG 700	0.067		1	6 th –8 th trifoliolate stage	27 27 62 62 125 125	forage hay forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 0.19 < 0.1 < 0.1 < 0.1 < 0.1	< 0.2 0.32 < 0.2 < 0.2 < 0.2 < 0.2	0.16 0.36 < 0.1 < 0.1 < 0.1 < 0.1	0.30 0.49 < 0.1 < 0.1 < 0.1 < 0.1
USA Ephrata, WA 1998 (WH 80) RES 99-049// XP98WA01 ID-731-005	SL 11.5%	0.067		1	early bloom	78	seed	< 0.1	< 0.1	< 0.2	< 0.1	< 0.1
USA Five Points, CA 1998 (Pioneer N58258) RES 99-058// XP98CA04 ID-731-006	WG 700	0.068		1	late bud/early bloom	68	seed	< 0.1	< 0.1	< 0.2	< 0.1	< 0.1
USA Mendota, CA 1998 (SW 14) RES 99-057// XP98CA03 ID-731-008	SL 11.5%	0.067		1	early bud stage, 71 cm tall	75	seed	< 0.1	< 0.1	< 0.2	< 0.1	< 0.1
USA Noblesville, IN 1998 (Pioneer 5454) RES 99-085// XP98IN01 ID-731-011	SL 11.5%	0.067		1	15–18 cm plant post dormancy regrowth	20 20 73 73 165 165	forage hay forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	0.43 0.89 < 0.1 < 0.1 < 0.1 < 0.1	0.56 1.02 < 0.2 < 0.2 < 0.2 < 0.2	0.45 0.80 < 0.1 < 0.1 < 0.1 < 0.1	0.82 1.6 < 0.1 < 0.1 < 0.1 < 0.1
USA Ephrata, WA 1997 (WH 80)	SL 11.9%	0.067		1	Early bud	74	seed	< 0.1	< 0.1	< 0.2	< 0.1	< 0.1

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in alfalfa, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
RES 98-234// XP97WA01 ID-731-012												
USA Fort Dodge, IA 1997 (Cenex/LOL 740) RES 99-041// XP97IA02 ID-731-015	WG 700	0.067		1	3 leaf	21 21 52 52	forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1	< 0.2 < 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1 < 0.1	< 0.1 0.13 < 0.1 < 0.1
USA Kerman, CA 1997 (Cuff 101) RES 99-069// XP97CA01 ID-731-016	SL 11.9%	0.067		1	10–18 cm plant height after regrowth	PE PE 0 0 7 7 14 14 18 18 48 48	forage hay forage hay forage hay forage hay forage hay forage hay	< 0.1 < 0.1 2.2 6.4 < 0.1 0.24 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 0.59 3.3 0.11 0.48 < 0.1 0.11 0.22 < 0.2 < 0.1 < 0.1 < 0.1 < 0.1	< 0.2 < 0.2 2.9 12 0.21 0.81 < 0.2 0.22 0.30 < 0.1 < 0.2 0.11 < 0.2 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 0.82 0.13 0.30 0.23 < 0.1 < 0.1 0.21 < 0.1 < 0.1	
USA Delta, CO 1998 (Vernema) RES 99-083// XP98CO01 ID-731-018	SL 11.5%	0.067		1	1–8 cm plant height, third trifoliate stage	39 39 102 102	forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1	< 0.2 < 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1
USA Payette, ID 1997 (Vernema) RES 98-233// XP97ID01 ID-731-019	WG 700	0.069		1	post- emergence, early bud stage	79- 80	seed	< 0.1	< 0.1	< 0.2	< 0.1	< 0.1
USA Payette, ID 1998 (Vernema) RES 99-099// XP98ID02 ID-731-020	WG 700	0.067		1	post- emergence, early bud stage	73	seed	< 0.1	< 0.1	< 0.2	< 0.1	< 0.1
USA Carlyle, IL 1998 (77917) RES 99-084// XP98IL01 ID-731-021	WG 700	0.067		1	10–25 cm plant height	PE PE 0 0 7 7 14 14 20 20 52 52 80	forage hay forage hay forage hay forage hay forage hay forage hay forage	< 0.1 < 0.1 3.7 5.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 0.11 1.6 0.56 2.0 0.25 0.70 0.15 0.31 < 0.1 < 0.1 < 0.1 < 0.1	< 0.2 < 0.2 3.8 7.4 0.93 2.2 0.38 0.82 0.25 0.46 < 0.2 < 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1 0.52 0.20 0.43 0.20 0.43 0.17 0.20 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 2.6 0.73 2.2 0.54 1.3 0.36 0.64 < 0.1 < 0.1 < 0.1 < 0.1

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in alfalfa, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
						80	hay	< 0.1	< 0.1	< 0.2	< 0.1	< 0.1

^a w. p.: Whole plant, green^b w. p.: Whole plant, dried

PE: Pre-treatment

Pea forage and fodder

Residue trials on pea forage or fodder were conducted in France, Spain and Italy in 1994, 1996 and 2011. EC or SL formulation was used at rates of 0.050–0.075 kg ai/ha.

Table 70 Residues of imazamox in pea forage and fodder from supervised residue trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai/hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in pea forage and fodder, mg/kg		
								Parent	CL 263284	Parent + CL 263284
N-France Caillouet (Solara) 1996 ID-FR-96-610//96- 610-319 ID-720-048	EC 16.7	0.075	0.021	1	Pre- emergence (05–08)	88 106	Pods+haulm	< 0.05	< 0.05	< 0.1
							Pods+haulm	< 0.05	0.17	0.22
N-France Bourgogne (Baccara) 1996 ID-FR-96-610//96- 610-422 ID-720-048	EC 16.7	0.075	0.031	1	Pre- emergence (00)	105 126	Pods+haulm	< 0.05	< 0.05	< 0.1
							Pods+haulm	< 0.05	< 0.05	< 0.1
N-France Fains (Solara) 1996 ID-FR-96-611//96- 611-320 ID-720-051	EC 16.7	0.075	0.021	1	Pre- emergence (05–08)	75 85 95	Whole plant	< 0.05	< 0.05	< 0.1
							Whole plant	< 0.05	< 0.05	< 0.1
							Pods+haulm	< 0.05	< 0.05	< 0.1
N-France Fains (Solara) 1996 ID-FR-96-611//96- 610-423 ID-720-051	EC 16.7	0.075	0.031	1	Pre- emergence (00)	91 100 110 129	Whole plant	< 0.05	< 0.05	< 0.1
							Whole plant	< 0.05	< 0.05	< 0.1
							Pods+haulm	< 0.05	< 0.05	< 0.1
							Pods+haulm	< 0.05	< 0.05	< 0.1
S-France Nerac (Carrera) 1996 ID-FR-96-660//96- 660-293 ID-720-049	EC 16.7	0.075	0.023	1	Pre- emergence (00–01)	77 86 96 120	Whole plant	< 0.05	< 0.05	< 0.1
							Whole plant	< 0.05	< 0.05	< 0.1
							Pods+haulm	< 0.05	< 0.05	< 0.10
							Pods+haulm	< 0.05	< 0.05	< 0.10
Italy Torello (Zaffiro) 1996 ID-IT-96-608// 96-608-01 ID-720-050	SL 40	0.050	0.010	1	Post- emergence (13)	33 42 52	Whole plant	< 0.05	< 0.05	< 0.1
							Whole plant	< 0.05	< 0.05	< 0.1
							Pods+haulm	< 0.05	< 0.05	< 0.1
	SL	0.075	0.016	1	Post-	52	Pods+haulm	< 0.05	< 0.05	< 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai/hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in pea forage and fodder, mg/kg		
								Parent	CL 263284	Parent + CL 263284
	40				emergence (13)					
Spain Sarriena-Huesca (Puyet) 1996 ID-SP-96-609 //96-609-27 ID-720-054	SL 40	0.052	0.017	1	Post- emergence (14–15)	44 53 64	whole plant whole plant pods+haulm	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
	SL 40	0.073	0.024	1	Post- emergence (14–15)	44 53 64	whole plant whole plant pods+haulm	< 0.05 < 0.05 < 0.05	< 0.05 0.06 < 0.05	< 0.1 0.11 < 0.1

Mix formulation: EC 16.7 g/L (pendimethalin of 250 g/L)

Soya bean forage and fodder

Residue trials on soya bean forage or fodder were conducted in France, Germany and Canada in 1994 and 2011. SC or WG formulation was used at rates of 0.035–0.071 kg ai/ha.

Table 71 Residues of imazamox in soya bean forage and fodder from supervised residue trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in soya bean forage and fodder, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
N-France Piney 2011 (Sigalia) 402845// L110243 2011/1112578	SC 22.4	0.042	0.021	1	(25–55)	0 28 104	w. plant w. plant rest	1.5 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	1.5 < 0.02 < 0.02	< 0.01 0.04 < 0.01	< 0.01 < 0.01 < 0.01
N-France Cigogné 2011 (Protina) 402845// L110244 2011/1112578	SC 22.4	0.042	0.021	1	(69)	0 28 69	w. plant w. plant rest	2.1 < 0.01 < 0.01	< 0.01 0.01 0.05	2.11 0.02 0.06	< 0.01 0.14 0.08	< 0.01 < 0.01 < 0.01
Germany Limburgerhof 2011 (Merlin) 402845// L110241 2011/1112578	SC 22.4	0.042	0.021	1	(25)	0 27 78	w. plant w. plant rest	2.2 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	2.2 < 0.02 < 0.02	< 0.01 0.05 0.01	< 0.01 < 0.01 < 0.01
Germany Lentzke 2011 (Merlin) 402845// L110242 2011/1112578	SC 22.4	0.042	0.021	1	(21–51)	0 27 98	w. plant w. plant rest	3.8 < 0.01 < 0.01	< 0.01 < 0.01 0.01	3.9 < 0.02 0.02	< 0.01 0.07 < 0.01	< 0.01 < 0.01 < 0.01
Canada Guelph 1994 (Galt) RES 95-049// XP94CN08	WG 700	0.035		1	Post- emergence	PE 0 17 31 42	forage forage forage forage			< 0.05 1.9 < 0.05 < 0.05 < 0.05		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in soya bean forage and fodder, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
ID-720-019						54	forage			< 0.05		
		0.070		1	Post-emergence	PE 0 17 31 42 54	forage forage forage forage forage			< 0.05 6.6 < 0.05 < 0.05 < 0.05 < 0.05		
Canada Georgetown 1994 (AC Bravor) RES 95-048// XP94CN07 ID-720-020	WG 700	0.035		1	Post-emergence	PE 0 14 31 42 64 106	forage forage forage forage forage straw			< 0.05 3.7 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05		
	WG 700	0.071		1	Post-emergence	PE 0 14 31 42 64 106	forage forage forage forage forage straw			< 0.05 11 0.23 < 0.05 < 0.05 < 0.05 0.06		

Mix formulation: SC 22.4 g/L (bentazone of 480 g/L)

w. plant: whole plant

PE: pre-treatment

Forage and fodder of cereal grains and grasses

Rice forage and straw

Residue trials were conducted with imidazolinone-tolerant rice in Italy and Spain in 2011 and 2012. SL formulation was used at rates of 0.038–0.075 kg ai/ha. The forage and straw were collected and analysed for residues of imazamox and the metabolites.

Table 72 Residues of imazamox in imidazolinone-tolerant rice forage and straw from supervised field trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in rice forage and straw, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Italy Salussola 2011 (Sirio CL) 407231//L110320 2012/1278352	SL 40	0.075	0.038	1	24–25	0 42 90	w. plant w. plant straw	3.74 < 0.01 < 0.01	0.03 < 0.01 < 0.01	3.77 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	0.02 < 0.01 < 0.01
	SL 40 + Dash	0.075	0.038	1	24–25	0 42 90	w. plant w. plant straw	3.42 < 0.01 < 0.01	0.05 < 0.01 < 0.01	3.47 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	0.02 < 0.01 < 0.01
	SL 40 +	0.038	0.019	2	24–25	0 42 90	w. plant w. plant straw	1.91 < 0.01 < 0.01	0.04 < 0.01 < 0.01	1.95 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in rice forage and straw, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
	Dash											
Italy Zeme 2011 (Sirio CL) 407231// L110321 2012/1278352	SL 40	0.075	0.038	1	25	0 25 78	w. plant w. plant straw	1.74 0.01 < 0.01	< 0.01 0.02 0.02	1.75 0.03 0.03	< 0.01 < 0.01 0.01	0.01 < 0.01 < 0.01
	SL 40 + Dash	0.075	0.038	1	25	0 25 78	w. plant w. plant straw	1.68 0.03 < 0.01	0.01 0.04 0.03	1.69 0.07 0.04	< 0.01 < 0.01 0.02	< 0.01 < 0.01 < 0.01
	SL 40 + Dash	0.038	0.019	2	25	0 25 78	w. plant w. plant straw	1.06 0.01 < 0.01	0.02 0.03 0.02	1.08 0.04 0.03	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
Italy Zibido 2012 (CL 71) 429716// L120440 2012/1272623	SL 40 + Dash	0.075	0.038	1	25	0 39 85	w. plant w. plant straw	2.1 < 0.01 < 0.01	0.02 < 0.01 < 0.01	2.1 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
	SL 40 + Dash	0.038	0.019	2	25	0 39 85	w. plant w. plant straw	1.8 0.01 < 0.01	0.02 0.08 < 0.01	1.8 0.09 < 0.02	< 0.01 0.02 < 0.01	< 0.01 < 0.01 < 0.01
Italy Salussola 2012 (CL 71) 429716// L120441 2012/1272623	SL 40 + Dash	0.075	0.038	1	25	0 41 90	w. plant w. plant straw	3.6 0.01 < 0.01	0.04 0.08 < 0.01	3.6 0.09 < 0.02	< 0.01 0.02 < 0.01	< 0.01 < 0.01 < 0.01
	SL 40 + Dash	0.038	0.019	2	25	0 41 90	w. plant w. plant straw	1.5 < 0.01 < 0.01	0.02 < 0.01 < 0.01	1.5 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
Spain ^a Los Palacios 2011 (Sirio) 407231// L110322 2012/1278352	SL 40	0.075	0.038	1	25	0 36 84	w. plant w. plant straw	3.75 < 0.01 < 0.01	0.02 0.02 < 0.01	3.77 0.03 0.02	< 0.01 < 0.01 < 0.01	0.01 < 0.01 < 0.01
	SL 40 + Dash	0.075	0.038	1	25	0 36 84	w. plant w. plant straw	2.22 0.01 < 0.01	0.02 0.03 0.01	2.24 0.04 0.02	< 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01
	SL 40 + Dash	0.038	0.019	2	25	0 36 84	w. plant w. plant straw	2.00 < 0.01 < 0.01	0.02 0.02 0.01	2.02 0.03 0.02	< 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01
Spain ^a Los Palacios 2011 (Sirio)	SL 40	0.075	0.038	1	25	0 36 84	w. plant w. plant straw	2.91 0.01 < 0.01	0.01 0.04 0.02	2.92 0.05 0.03	< 0.01 0.02 0.01	0.01 < 0.01 < 0.01

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in rice forage and straw, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
407231// L110323 2012/1278352												
	SL 40 + Dash	0.075	0.038	1	25	0 36 84	w. plant w. plant straw	2.40 0.01 < 0.01	0.02 0.04 0.02	2.42 0.05 0.03	< 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01
	SL 40 + Dash	0.038	0.019	2	25	0 36 84	w. plant w. plant straw	1.44 < 0.01 < 0.01	0.02 0.02 < 0.01	1.46 0.03 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
Spain ^b Los Palacios 2012 (Sirio) 429716// L120442 2012/1272623	SL 40 + Dash	0.075	0.038	1	25	0 31 77	w. plant w. plant straw	5.1 0.02 < 0.01	0.04 0.04 0.06	5.1 0.06 0.07	< 0.01 < 0.01 0.03	< 0.01 < 0.01 0.01
	SL 40 + Dash	0.038	0.019	2	25	0 31 77	w. plant w. plant straw	1.7 < 0.01 < 0.01	0.02 0.02 0.02	1.7 0.03 0.03	< 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01
Spain ^b Los Palacios 2012 (Sirio) 429716 2012/1272623 L120443	SL 40 + Dash	0.075	0.038	1	25	0 38 84	w. plant w. plant straw	2.9 < 0.01 < 0.01	0.04 0.02 0.04	2.9 0.03 0.05	< 0.01 < 0.01 0.02	< 0.01 < 0.01 0.01
	SL 40 + Dash	0.038	0.019	2	25	0 38 84	w. plant w. plant straw	1.2 < 0.01 < 0.01	0.02 < 0.01 0.02	1.2 < 0.02 0.03	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01

n.r.: Not reported

w. plant: Whole plant without roots

^a Not independent trials^b Not independent trials*Wheat forage and straw*

Residue trials were conducted with imidazolinone-tolerant wheat in France, Italy, Canada and USA. Treatments were made with various formulations at rates of 0.020–0.075 kg ai/ha. The forage and straw were analysed for residues of imazamox and metabolites.

Table 73 Residues of imazamox in imidazolinone-tolerant wheat forage from supervised residue trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat forage, mg/kg		
								Parent	CL 263284	Parent + CL 263284
N-France Les Mureaux 1996 (Fidel) ID-FR-96-617//	SL 40	0.075	0.021	1	25 5 tillers detectable	71 91	whole plant whole plant	< 0.05 < 0.05	< 0.05 < 0.05	< 0.1 < 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat forage, mg/kg		
								Parent	CL 263284	Parent + CL 263284
96-617-323 ID-730-023										
	EC 16.7	0.067	0.018	1	25 5 tillers detectable	71 91	whole plant whole plant	< 0.05 < 0.05	< 0.05 < 0.05	< 0.1 < 0.1
N-France Pignicourt 1996 (Fidel) ID-FR-96-617// 96-617-465 ID-730-023	SL 40	0.075	0.025	1	25–29 5 to 9 or more tillers detectable	0 65 83	whole plant whole plant whole plant	5.12 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	5.17 < 0.1 < 0.1
	EC 16.7	0.067	0.022	1	25–29 5 tillers detectable- beginning of stem elongation	0 65 83	whole plant whole plant whole plant	4.47 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	4.52 < 0.1 < 0.1
N-France Montchauvet 1996 (Fidel) ID-FR-96-616// 96-616-322 ID-730-035	SL 40	0.075	0.021	1	23–29 3 tillers detectable – end of tillering	83	whole plant	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.028	1	23–29	83	whole plant	< 0.05	< 0.05	< 0.1
N-France Tilloy-Bellay 1996 (Fidel) ID-FR-96-616// 96-616-464 ID-730-035	SL 40	0.075	0.032	1	25–27 5–7 tillers detectable	110	whole plant	< 0.05	< 0.05	< 0.10
	EC 16.7	0.067	0.018	1	25–27	110	whole plant	< 0.05	< 0.05	< 0.1
S-France ^a Cassignas 1996 (Fidel) ID-FR-96-619// 96-619-291 ID-730-022	SL 40	0.075	0.020	1	26–28 6–8 tillers detectable	96	whole plant	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.018	1	26–28 6–8 tillers detectable	96	whole plant	< 0.05	< 0.05	< 0.1 <
S-France ^a Cassignas 1996 (Fidel) ID-FR-96-619// 96-619-292 ID-730-022	SL 40 g/L	0.075	0.020	1	27–29 7 tillers detectable- end of tillering	83	whole plant	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.018	1	27–29 7 tillers detectable- end of tillering	83	whole plant	< 0.05	< 0.05	< 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat forage, mg/kg		
								Parent	CL 263284	Parent + CL 263284
Italy Bologna 1996 (Fidel) ID-IT-96-618// 96-618-01 ID-730-019	SL 40	0.075	0.016	1	25 (5 tillers detectable)	0 53 74	whole plant whole plant whole plant	3.34 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	3.39 < 0.1 < 0.1
	EC 16.7	0.067	0.016	1	25	0 53 74	whole plant whole plant whole plant	2.74 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	2.79 < 0.1 < 0.1
Italy Pisa 1996 (. Fidel) ID-IT-96-618// 96-618-02 ID-730-019	SL 40 g/L	0.075	0.016	1	25	0 50 71	whole plant whole plant whole plant	1.61 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	1.66 < 0.1 < 0.1
	EC 16.7	0.067	0.016	1	25	0 50 71	whole plant whole plant whole plant			
Italy Bologna 1996 (Fidel) ID-IT-96-615// 96-615-01 ID-730-020	SL 40	0.075	0.016	1	25 5 tillers detectable	69	whole plant	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.016	1	25 5 tillers detectable	69	whole plant	< 0.05	< 0.05	< 0.1
Italy Bologna 1997 (Fidel) ID-IT-97-808// 97-808-01 ID-730-021	SL 40	0.075	0.016	1	29 end of tillering	70	whole plant	< 0.05	< 0.05	< 0.1
Canada Minto 1997 (SWP 965-001) RES 98-001// XP97CN01 ID-730-015	WG 700 + Merge	0.020	0.020	1	5–6 leaf stage	PE 0 7 14 28 42 56	forage forage forage forage forage forage	< 0.05 1.60 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.1 1.65 0.10 < 0.1 < 0.1 < 0.1 < 0.1
	WG 700 + Merge	0.040	0.040	1	5–6 leaf stage	PE 0 7 14 28 42 56	forage forage forage forage forage forage	< 0.05 3.51 0.10 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.1 3.56 0.15 < 0.1 < 0.1 < 0.1 < 0.1
Canada Aberdeen 1997 (SWP 965-001) RES 98-005// XP97CN05 ID-730-016	WG 700 + Merge	0.020		1	3–6 leaf stage	0 14 28	forage forage forage	1.19 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	1.28 < 0.1 < 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat forage, mg/kg		
								Parent	CL 263284	Parent + CL 263284
	WG 700 + Merge	0.040		1	3–6 leaf stage	0 14 28	forage forage forage	1.94 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	2.07 < 0.1 < 0.1
Canada Saskatoon 1997 (SWP 965-001) RES 98-002// XP97CN02 ID-730-017	WG 700 + Merge	0.020		1	4–6 leaf stage	7 29 56	forage forage forage	0.13 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	0.20 < 0.1 < 0.1
	WG 700 + Merge	0.040		1	4–6 leaf stage	7 29 56	forage forage forage	0.24 < 0.05 < 0.05	0.056 < 0.05 < 0.05	0.36 < 0.1 < 0.1
Canada Barnwell 1997 (SWP 965-001) RES 98-003// XP97CN03 ID-730-018	WG 700 + Merge	0.020		1	4–6 leaf stage	PE 0 7 14 28 42 56	forage forage forage forage forage forage	< 0.05 1.6 0.15 0.080 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.1 1.7 0.23 0.13 < 0.1 < 0.1 < 0.1
	WG 700 + Merge	0.040		1	4–6 leaf stage	PE 0 7 14 28 42 56	forage forage forage forage forage forage	< 0.05 3.2 0.40 0.18 0.055 < 0.05 < 0.05	< 0.05 < 0.05 0.11 0.083 < 0.05 < 0.05 < 0.05	< 0.1 3.3 0.52 0.26 0.11 < 0.1 < 0.1
Canada Nisku 1997 (SWP 965-001) RES 98-006// XP97CN06 ID-730-028	WG 700 + Merge	0.020		1	6 leaf stage	0 14 41	forage forage forage	0.68 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	0.78 < 0.1 < 0.1
	WG 700 + Merge	0.040		1	6 leaf stage	0 14 41	forage forage forage	1.2 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	1.3 < 0.1 < 0.1
USA Northwood, ND 1998 (NDGRDN01) RES 99-094// XP98ND04 ID-730-038	SL 11.5% + X-77	0.067	0.067	1	5 leaf stage	13 47	forage forage	0.12 < 0.05	< 0.05 < 0.05	0.13 < 0.1

Mix formulation: EC 16.7 g/L (pendimethalin of 250 g/L)

PE: Pre-treatment

^a Not independent trials

Table 74 Residues of imazamox in imidazolinone-tolerant wheat straw from supervised residue trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat straw, mg/kg		
								Parent	CL 263284	Parent + CL 263284
N-France Les Mureaux 1996 (Fidel) ID-FR-96-617// 96-617-323 ID-730-023	SL 40	0.075	0.021	1	25 5 tillers detectable	129	straw	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.018	1	25 5 tillers detectable	129	straw	< 0.05	< 0.05	< 0.1
N-France Pignicourt 1996 (Fidel) ID-FR-96-617// 96-617-465 ID-730-023	SL 40	0.075	0.025	1	25–29 5 to 9 or more tillers detectable	125	straw	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.022	1	25–29 5 tillers detectable- beginning of stem elongation	125	straw	< 0.05	< 0.05	< 0.1
N-France Cassignas 1995 (Fidel) ID-FR-95-005// ID-730-034	SL 120	0.075	0.019	1	31 first node detectable	118	straw	< 0.05	< 0.05	< 0.1
N-France Montchauvet 1996 (Fidel) ID-FR-96-616// 96-616-322 ID-730-035	SL 40	0.075	0.021	1	23–29 3 tillers detectable – end of tillering	121	straw	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.028	1	23–29	121	straw	< 0.05	< 0.05	< 0.1
N-France Tilloy-Bellay 1996 (Fidel) ID-FR-96-616// 96-616-464 ID-730-035	SL 40	0.075	0.032	1	25–27 5–7 tillers detectable	146	straw	< 0.05	< 0.05	< 0.10
	EC 16.7	0.067	0.018	1	25–27	146	straw	< 0.05	< 0.05	< 0.1
S-France ^a Cassignas 1996 (Fidel) ID-FR-96-619// 96-619-291 ID-730-022	SL 40	0.075	0.020	1	26–28 6–8 tillers detectable	131	straw	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.018	1	26–28 6–8 tillers detectable	131	straw	< 0.05	< 0.05	< 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat straw, mg/kg		
								Parent	CL 263284	Parent + CL 263284
S-France ^a Cassignas 1996 (Fidel) ID-FR-96-619// 96-619-292 ID-730-022	SL 40 g/L	0.075	0.020	1	27–29 7 tillers detectable– end of tillering	118	straw	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.018	1	27–29 7 tillers detectable– end of tillering	118	straw	< 0.05	< 0.05	< 0.1
S-France Montchauvet 1995 (Fidel) ID-FR-95-005// 95-005-318 ID-730-034	SL 120	0.075	0.019- 0.025	1	30 beginning of stem elongation	140	straw	< 0.05	< 0.05	< 0.1
S-France Bourgogne 1995 (Fidel) ID-FR-95-005// 95-005-484 ID-730-034	SL 120	0.075	0.019- 0.025	1	30	135	straw	< 0.05	< 0.05	< 0.1
Italy Bologna 1996 (Fidel) ID-IT-96-618// 96-618-01 ID-730-019	SL 40	0.075	0.016	1	25 5 tillers detectable	102	straw	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.016	1	25	102	straw	< 0.05	< 0.05	< 0.1
Italy Pisa 1996 (. Fidel) ID-IT-96-618// 96-618-02 ID-730-019	SL 40 g/L	0.075	0.016	1	25	103	straw	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.016	1	25	103	straw	< 0.05	< 0.05	< 0.1
Italy Bologna 1996 (Fidel) ID-IT-96-615// 96-615-01 ID-730-020	SL 40	0.075	0.016	1	25 5 tillers detectable	95	straw	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.016	1	25 5 tillers detectable	95	straw	< 0.05	< 0.05	< 0.1
Italy Bologna 1997 (Fidel) ID-IT-97-808// 97-808-01	SL 40	0.075	0.016	1	29 end of tillering	98	straw	< 0.05	< 0.05	< 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat straw, mg/kg		
								Parent	CL 263284	Parent + CL 263284
ID-730-021										
Canada Minto 1997 (SWP 965-001) RES 98-001// XP97CN01 ID-730-015	WG 700 + Merge	0.020	0.020	1	5–6 leaf stage	42 56 89	hay hay straw	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
	WG 700 + Merge	0.040	0.040	1	5–6 leaf stage	42 56 89	hay hay straw	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
Canada Aberdeen 1997 (SWP 965-001) RES 98-005// XP97CN05 ID-730-016	WG 700 + Merge	0.020		1	3–6 leaf stage	28 36 72	hay hay straw	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
	WG 700 + Merge	0.040		1	3–6 leaf stage	28 36 72	hay hay straw	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
Canada Saskatoon 1997 (SWP 965-001) RES 98-002// XP97CN02 ID-730-017	WG 700 + Merge	0.020		1	4–6 leaf stage	42 56 79	hay hay straw	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
	WG 700 + Merge	0.040		1	4–6 leaf stage	42 56 79	hay hay straw	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
Canada Barnwell 1997 (SWP 965-001) RES 98-003// XP97CN03 ID-730-018	WG 700 + Merge	0.020		1	4–6 leaf stage	42 56 69	hay hay straw	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
	WG 700 + Merge	0.040		1	4–6 leaf stage	42 56 69	hay hay straw	< 0.05 < 0.05 < 0.05	0.098 0.056 < 0.05	0.17 0.11 < 0.1
Canada Nisku 1997 (SWP 965-001) RES 98-006// XP97CN06 ID-730-028	WG 700 + Merge	0.020		1	6 leaf stage	41 56 90	hay hay straw	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
	WG 700 + Merge	0.040		1	6 leaf stage	41 56 90	hay hay straw	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
USA Northwood, ND 1998	SL 11.5% +	0.067	0.067	1	5 leaf stage	47 82	hay straw	< 0.05 < 0.05	< 0.05 < 0.05	< 0.1 < 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat straw, mg/kg		
								Parent	CL 263284	Parent + CL 263284
(NDGRDN01) RES 99-094// XP98ND04 ID-730-038	X-77									

Mix formulation: EC 16.7 g/L (pendimethalin of 250 g/L)

PE: Pre-treatment

^a Not independent trials

Miscellaneous Fodder and Forage crops

Rape seed forage

Residue trials were conducted with imidazolinone-tolerant rape plant in Germany, UK and France. Treatments were made with SC or SL formulation at rates of 0.050–0.077 kg ai/ha. The plant was analysed for residues of imazamox and metabolites.

Table 75 Residues of imazamox in imidazolinone-tolerant rape seed forage from supervised residue trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in rape seed forage, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Germany Uetza 2005/2006 (PS22-1A-VH) 211921// AF/10114/BA/3 2007/1007939	SC 25	0.050		1	(51)	0 13	w. plant r. plant	0.53 < 0.05	< 0.05 < 0.05	0.58 < 0.1		
Germany Uetza 2005/2006 (PS22-1A-VH) 251536// AF/10475/BA/3 2005/2006 2007/1007963	SC 25 + Dash	0.050		1	(51)	0 13	w. plant r. plant	2.20 0.19	< 0.05 0.52	2.25 0.71		
Germany Schwienau/Melzingen 2005/2006 (PS22-1A-VH) 211921// AF/10114/BA/4 2007/1007939	SC 25	0.050		1	(51)	0 12	w. plant r. plant	0.26 < 0.05	< 0.05 < 0.05	0.31 < 0.1		
Germany Schwienau/Melzingen 2005/2006 (PS22-1A-VH) 251536// AF/10475/BA/4 2007/1007963	SC 25 + Dash	0.050		1	(51)	0 12	w. plant r. plant	0.22 0.05	< 0.05 < 0.05	0.27 0.10		
Germany Euskirchen-Dom	SL 40	0.075	0.019	1	(18) 8 leaves	0 145	whole plant whole plant			< 0.05 ^a < 0.05 ^a		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in rape seed forage, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Esch 1997/98 (Imi-res., NW 4200) ID-GE-98-403// 98-403-01 ID-750-017					unfolded	230	whole plant			< 0.05 ^a		
Germany Billingsdorf Flurnummer 1997/98 (Imi-res., NW 4200) ID-GE-98-403// 98-403-02 1997/98 ID-750-017	SL 40	0.077	0.025	1	(17) 7 leaves unfolded	0 154 232	whole plant whole plant whole plant			< 0.05 ^a < 0.05 ^a < 0.05 ^a		
UK Sowerby, Thirsk 1996 (Imi-res., 45A71) ID UK 96 662// 96-662-01 ID-750-005	SL 40	0.075		1	(13)	0 51 63	whole plant whole plant whole plant	3.97 < 0.05 < 0.05	0.06 < 0.05 < 0.05	4.03 < 0.1 < 0.1		
UK Sessay, Thirsk 1996 (Imi-res., 45A71) ID UK 96 662// 96-662-04 ID-750-005	SL 40	0.075		1	(14)	0 51 63	whole plant whole plant whole plant	3.13 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	3.18 < 0.1 < 0.1		
S-France La francaise 2005/2006 (PS22-1A-VH) 211921// AF/10114/BA/1 2007/1007939	SC 25	0.050		1	(61)	0 15	w. plant r. plant	0.68 < 0.05	< 0.05 < 0.05	0.73 < 0.1		
S-France La francaise 2005/2006 (PS22-1A-VH) 251536// AF/10475/BA/1 2007/1007939	SC 25 + Dash	0.050		1	(61)	0 15	w. plant r. plant	0.97 0.12	< 0.05 0.21	1.02 0.33		
S-France Montauban 2005/2006 (PS22-1A-VH) 211921// AF/10114/BA/2 2007/1007939	SC 25	0.050		1	(53)	0 19	w. plant r. plant	0.44 < 0.05	< 0.05 < 0.05	0.49 < 0.1		
S-France Montauban 2005/2006 (PS22-1A-VH) 251536// AF/10475/BA/2 2007/1007939	SC 25 + Dash	0.050		1	(53)	0 19	w. plant r. plant	1.50 0.14	< 0.05 0.28	1.6 0.42		

w. plant: Whole plant without roots

r. plant: Rest of plant without roots that seeds were taken

^a Imazamox and CL 263284 were measured as one chromatographic peak representing a total imazamox-related residue.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

The Meeting received information on effects of heating in water and processing on imazamox residues in soya bean, wheat and sunflower seed.

The processing factors (residue processed commodity divided by residue RAC) were calculated based on the total imazamox residues (sum of imazamox and CL 263284, expressed as imazamox). When the total imazamox residue value of the RAC was below limit of quantification, no processing factor was derived. When the total imazamox residue value of the processed commodity was below limit of quantification, the calculated processing factor is reported with a “less than” (<) symbol.

Effects on the nature of residue

To estimate the degradation behaviour of [pyridine-3-¹⁴C, imidazolinone-3-¹⁵N]-imazamox during industrial processing or household preparation, different processes (pasteurization, baking, brewing, boiling and sterilization) were simulated [Hassink 2012(a), 2011/1286214].

The test item was suspended in aqueous buffer solutions of different pH-values, to give a final concentration of ca. 0.92 µg/mL. Pasteurization was simulated by incubating the test solutions (pH 4) in a rotated round-bottom flask in a water bath for 20 min at 90 °C. For baking, brewing and boiling, the test solutions (pH 5) were treated in an Erlenmeyer flask under reflux at 100 °C for 60 min. To avoid an influence of light, the glassware was wrapped. Sterilization was performed by autoclaving the test solution (pH 6) at about 120 °C for 20 min. The total radioactivity present was determined by LSC. Characterization of the radioactivity in the buffered samples after incubation was performed by HPLC.

The recovery of total applied radioactivity (TAR) was 98.3–103.5%. Only in sterilization test, one replicate test solution showed a breakdown product of 5.8% TAR, the peak split into at least two peaks. Imazamox was stable under the simulation conditions of pasteurization, baking, boiling, brewing and sterilization.

Effects on the residue level

No residues were observed in trials with soya beans treated at exaggerated rates (0.13 kg ai/ha or 0.22 kg ai/ha) and no processing of the raw commodity was undertaken [ID-720-005, ID-720-006].

Wheat

During the 1998 growing season, a field trial was carried out in North Dakota, United States [Rodriguez 1999a, ID-730-038]. Wheat was treated with SL formulation (11.5%) and 0.25% adjuvant X-77. Trial plot received one post-emergence broadcast foliar application at a rate of either 0.20 kg ai/ha or 0.32 kg ai/ha, respectively. The applications were made when the plants were in the 5 leaf growth stage. Replicate wheat straw and grain samples were collected at crop maturity for typical wheat harvest (82 DAT). Wheat grain samples treated with 0.20 kg ai/ha were analysed for residues of imazamox and CL 263284 according to method M 3098 (CE-UV). The validated limit of quantitation was 0.05 mg/kg for imazamox and CL 263284 each. Wheat grain treated with 0.32 kg ai/ha was analysed for imazamox and CL 263284 residues according to method M 3252 (HPLC-MS; method M 3098). The validated limit of quantitation was 0.01 mg/kg for each analyte. Samples were stored frozen for a maximum of 5 months until analysis.

Table 76 Residues of imazamox and its metabolites in processed commodities of wheat

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue, mg/kg		
							Parent	CL 263284	Parent + CL 263284
USA Northwood, ND 1998 (NDGRDN01) RES 99-094// XP98ND04 ID-730-038	SL 11.5% + X-77	0.20	1	5 leaf stage	82	grain	< 0.05	< 0.05	< 0.1
	SL 11.5% + X-77	0.32	1	5 leaf stage	82	Grain ^a	0.053	< 0.05	0.1
						pre- processing grain	0.028 ^b	< 0.01	0.038
						germ	0.075 Pf=2.7	< 0.01	0.085 Pf=2.2
						bran	0.11 Pf=3.9	0.020	0.13 Pf=3.4
						middlings	0.046 Pf=1.6	< 0.01	0.056 Pf=1.5
						shorts	0.090 Pf=3.2	0.013	0.104 Pf=2.6
						flour	0.034 Pf=1.2	< 0.01	0.044 Pf=1.2
						asp. grain fr.	0.029 Pf=1.0	< 0.01	0.039 Pf=1.0

^a Field trial sample^b Mean residue for five subsamples (0.031, 0.030, 0.027, 0.027, 0.026 mg/kg)*Sunflower seed*

During the 2000 growing season, one field trial was carried out in Minot, ND, US. Sunflowers were treated with SL formulation (11.78%) and a formulation containing imazapyr. Trial plot received one foliar tank-mix application at a rate of either 0.056 kg ai/ha or 0.11 kg ai/ha of imazamox. Another one plot received a single foliar application of imazamox alone at 0.14 kg ai/ha. All spray solutions included methylated seed oil (1.0% v/v). The applications were made post-emergence when the plants were 30 to 46 cm tall (6 to 8 leaf growth stage). Replicate sunflower seed RAC samples were harvested at normal crop maturity, 88 days after treatment and processed according to simulated commercial practices into meal and refined oil. Sunflower samples were analysed for residues of imazamox, CL 263284 and CL 312622 using a developed HPLC-MSD method. The validated limit of quantitation was 0.05 mg/kg for each analyte. Samples were kept in frozen storage from collection until analysis for a maximum period of 7 months.

Processing factors were not derived as residue concentrations of sunflower seed (RAC) were all < LOQ.

Table 77 Residues of imazamox and its metabolites in processed commodities of sunflower seeds (2000)

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	No.	Crop growth stage (BBCH) at application	D A T	Portion analysed	Residue, mg/kg				
							Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
USA Minot, ND 2000 (IMI sunflower hybrid, cmS HA425/RHA426) 07219//ND03 2002/5004111	SL 11.78 % + MSO ^a	0.056	1	vegetative ,18–30 cm	88	seed	< 0.05	< 0.05	< 0.1		< 0.05
						meal	< 0.05	< 0.05	< 0.1		< 0.05
						refined oil	< 0.05	< 0.05	< 0.1		< 0.05
	SL 11.78 % + MSO ^a	0.11	1	vegetative ,18–30 cm	88	seed	< 0.05	< 0.05	< 0.1		< 0.05
						meal	0.07	0.07	0.14		< 0.05
						refined oil	< 0.05	< 0.05	< 0.1		< 0.05
	SL 11.78 % + MSO	0.14	1	vegetative ,18–30 cm	88	seed	< 0.05	< 0.05	< 0.1		< 0.05
						meal	0.05	0.06	0.11		< 0.05
						refined oil	< 0.05	< 0.05	< 0.1		< 0.05

^a tank mix with imazapyr

During the 2008 growing season, one field trial was carried out in SK, Canada [Norris 2009(a), 2008/7019225]. Clearfield Sunflowers were treated with a single foliar application of SL formulation (33% imazamox and 15% imazapyr) at 0.10 kg ai/ha. The applications were made broadcast post-emergence targeting stem elongation (BBCH 33-39) to late flowering growth stages (BBCH 69), at 70 days prior to the harvest of mature (dry) seed. The sunflower samples were analysed for residues of imazamox and CL 263284 using method M 3519 (LC-MS/MS) with a limit of quantitation of 0.05 mg/kg. Samples were stored after harvest for a maximum of 157 days prior to analysis.

Table 78 Residues of imazamox and its metabolites in processed commodities of sunflower seeds (2008)

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	No.	Crop growth stage (BBCH) at appl.	D A L A	Portion analysed	Residue, mg/kg		
							Parent	CL 263284	Parent + CL 263284
Canada Dundern, SK 2008 (Viper) 327308//	SL 33	0.10	1	(33–39)	70	seed	0.20 0.26 mean: 0.23	0.24 0.35 mean: 0.30	0.45 0.63 mean: 0.54

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	No.	Crop growth stage (BBCH) at appl.	D A L A	Portion analysed	Residue, mg/kg		
							Parent	CL 263284	Parent + CL 263284
R080100 2008/7019225									
						meal	0.53 0.51 mean: 0.52 Pf=2.3	1.0 1.0 mean: 1.0	1.6 1.5 mean: 1.6 Pf=3.0
						refined oil	< 0.05 < 0.05 Pf=< 0.2	< 0.05 < 0.05	< 0.05 < 0.05 Pf=< 0.2

Residues from sampled two bulks

Mix formulation: SL 33 g/L (imazapyr of 15 g/L).

During the 2007 growing season, one field trial was carried out in ND, USA [Johnston 2009a, 2008/7008101]. Clearfield sunflowers were treated with a single foliar application of WG formulation (35% imazamox and 35% imazethapyr) at 0.075 kg ai/ha of imazamox. The applications were made post-emergence when the sunflowers were at flower buds visible to late flowering growth stages, at 59 days prior to the harvest of mature (dry) seed. Sunflower samples were analysed for residues of imazamox and CL 263284 using method SOP-PA.0288 (LC-MS/MS) with a limit of quantitation of 0.05 mg/kg. Seed samples were stored frozen after harvest or fraction collection for a maximum of 239 days prior to analysis.

Table 79 Residues of imazamox and its metabolites in processed commodities of sunflower seeds (2007)

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	No.	Crop growth stage (BBCH) at appl.	D A L A	Portion analysed	Residue, mg/kg		
							Parent	CL 263284	Parent + CL 263284
USA McHenry, ND 2007 (DKF38-80CL) 261967// 2008/7008101	WG 350 + Merge	0.075	1	Late flowering	59	seed	< 0.05 < 0.05 mean: < 0.05	0.16 0.11 mean: 0.14	0.21 0.17 mean: 0.19
						meal	< 0.05 < 0.05	0.24 0.34 mean: 0.29	0.30 0.41 mean: 0.36 Pf=1.9
						refined oil	< 0.05 < 0.05	< 0.05 < 0.05	< 0.1 < 0.1 Pf=< 0.5

Mix formulation: WG 350 g/kg (imazethapyr 35%)

Table 80 Summary of processing factors for sunflower seed processing

Processed commodity	Imazamox processing factor	Mean or best estimate for imazamox Pf	Sum of imazamox and CL 263284	Mean or best estimate for sum residue Pf
Meal	2.3	2.3	1.9, 3.0	2.5
Refined oil	< 0.2	< 0.2	< 0.2, < 0.5	< 0.5

APPRAISAL

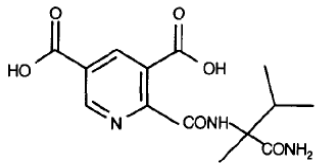
Imazamox is an imidazolinone herbicide registered in many countries to control a wide spectrum of grass and broadleaf weeds. At the Forty-fourth Session of the CCPR (2012)³, it was scheduled for evaluation as a new compound by the 2014 JMPR.

The Meeting received information on the physical and chemical properties, animal and plant metabolism, environmental fate, analytical methods, storage stability, use patterns, supervised trials and processing studies.

The following abbreviated names were used for the metabolites discussed below.

Code (MW)	IUPAC chemical name	Structure
CL 299263 (305) Imazamox; BAS 720 H	(<i>RS</i>)-2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-5-methoxymethylnicotinic acid	
CL 263284 (291) M715H001	5-(hydroxymethyl)-2-(4-isopropyl-4-methyl-5-oxo-2-imazolin-2-yl) nicotinic acid	
CL 189215 (453.5) M715H002	5-[(β-glucopyranosyl oxy) methyl]-2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl) nicotinic acid	
CL 312622 (305) M720H002	2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-3,5-pyridine-dicarboxylic acid	
CL 354825 (277)	5-hydroxy-6-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-nicotinic acid	
CL 336554 (323)	2-[(1-carbamoyl-1,2-dimethylpropyl) carbamoyl]-5-(methoxymethyl)-nicotinic acid	

³ REP12/PR-Appendix XIII

Code (MW)	IUPAC chemical name	Structure
CL 152795	2-[(1-carbamoyl-1,2-dimethylpropyl) carbamoyl]-3,5-pyridinedicarboxylic acid	

Animal metabolism

The Meeting received metabolism studies on imazamox, CL 263284 (hydroxymethyl) and CL 312622 (dicarboxylic acid) orally administered to lactating goats and laying hens.

Imazamox

Lactating goats were orally administered single daily doses of [pyridine-6-¹⁴C]-imazamox, via gelatin capsules, for seven consecutive days at either 2.1 or 12 ppm. Kidney, liver, leg and loin muscle, and omental fat samples were collected approximately 20 hours after the last dosing.

In the goat the imazamox was mainly excreted in urine (92% of the low dose and 65% of the high dose) and via faeces (14.8% of the low dose and 24.0% of the high dose). Unaltered parent accounted for most of the excreted residue. The total radioactive residues in the daily blood and milk samples and tissues (liver, leg, loin, omental fat) were less than 0.01 mg eq./kg regardless of the treatment dose levels. In the kidney, the TRR was 0.02 mg eq./kg at a dose level of 2.1 ppm and 0.06 mg eq./kg at a dose level of 12 ppm. This was mostly imazamox (89% TRR).

CL 263284

A ruminant metabolism study was conducted with [pyridine-6-¹⁴C]-CL 263284. Dose levels for the goats, administered orally in gelatin capsules, were either 2.3 or 15 ppm daily for seven days. Samples of blood, milk and excreta were collected daily. After seven days of dosing, the goats were sacrificed (approximately 20 hours after the last dose) and the tissues kidney, liver, muscle and fat were collected.

Elimination of ¹⁴C radioactivity via faeces accounted for 82% and 68% of the total cumulative dose at the low dose and high dose, respectively. TRR levels in most tissue samples (muscle, fat, liver) from the low and high dose treated goat and all milk samples were < 0.01 mg eq./kg. The kidney showed a residue of 0.03 mg eq./kg at the 15 ppm dose, which comprised minor component CL 263284 (9% TRR, < 0.01 mg/kg) and very labile component M1(salt or conjugate of CL 263284; 78% TRR, 0.02 mg eq./kg).

CL 312622

Lactating goats were orally treated with a mixture of [pyridine-6-¹⁴C, ¹³C]-CL 312622. The three goats were dosed for five consecutive days with either 3.1 or 33 ppm. Milk, urine and faeces samples were collected daily. Edible tissues (muscle, fat, liver and kidney) were collected at sacrifice (approximately 22 hours after the last dose).

About 90% of the total cumulative dose was excreted via the faeces. TRR levels in all tissues and milk samples were < 0.006 mg eq./kg, except kidney (0.025 mg eq./kg) and liver (0.009 mg eq./kg) of goats treated at the high dose level (33 ppm).

In the kidney sample, CL 312622 was the predominant radioactive residue (60% TRR). The impurity (CL 152795) present in the dosing solution was found at 11% of TRR. Minor polar unknowns (total 10% of TRR) and non-polar unknowns (total 12% of TRR) were also present in the kidney extract, composed with fractions of 0.003 mg eq./kg with multiple components. In the liver, CL 312622 was present at 38% of TRR (impurity, CL 152795, 19% TRR) and polar and nonpolar (31% and 11% TRR, respectively) fractions were equivalent to 0.003 mg eq./kg or lower, containing multiple components.

In summary imazamox and its metabolites (CL 263284 and CL 312622) each was mainly excreted unchanged through urine and faeces in lactating goats. There was no accumulation in any other edible goat tissue or in the milk (< 0.01 mg eq./kg) except kidney. Kidney mostly contained the unchanged administered compound.

Metabolism of imazamox in laying hens

Imazamox

Laying hens were orally dosed with [pyridine-6- ^{14}C]-imazamox at a dose level of either 2.1 or 10 ppm for seven consecutive days. Eggs were collected twice daily and blood and the tissues (liver, kidney, muscle, and skin with adhering fat) were collected for analysis approximately 22 hours after the last dose. About 85% of the total dose administered at the low and high dose was in the excreta. Residues in eggs, skin with adhering fat, muscle, liver and kidney tissues were all less than 0.01 mg eq./kg.

CL 263284

A poultry metabolism study was conducted with [pyridine-6- ^{14}C]-CL 263284. Hens were dosed orally at a dose level of either 2.1 or 11 ppm by gelatin capsules for seven days. Eggs and excreta were collected daily. The hens were sacrificed and the tissues (liver, kidney, muscle and skin with adhering fat) were collected approximately 22 hours after the last dose. About 87% of the total dose administered at the low and high dose was eliminated via excreta. Residues in all tissues and eggs were less than 0.01 mg eq./kg.

CL 312622

Laying hens were orally treated with a mixture of [pyridine-6- ^{13}C]-CL 312622 and [pyridine-6- ^{14}C]-CL 312622. Hens were dosed for five consecutive days with a dose level of either 0.13 or 10.5 ppm. Eggs were collected twice daily and the edible tissues (muscle, liver and skin with adhering fat) were collected at sacrifice approximately 22 hours after the last dose.

About 89% of the total dose administered at the low dose and high dose was eliminated via the excreta. Residues in tissues (liver, muscle and skin with adhering fat) and eggs were all 0.006 mg eq./kg or below. There was no retention or accumulation in eggs or edible poultry tissues.

In summary, in laying hens, imazamox, its metabolites (CL 263284 and CL 312622) were mainly eliminated unchanged through excreta. No residues (< 0.01 mg eq./kg) in eggs or edible tissues were found.

Plant metabolism

The Meeting received information on the fate of imazamox in oilseed rape, soya bean, pea, maize, wheat and alfalfa.

The metabolic fate of imazamox in imidazolinone-tolerant oilseed rape was investigated in one indoor and two outdoor studies. In one of the outdoor studies, a mixture of [pyridine-6- ^{14}C]-imazamox and [pyridine-6- ^{13}C]-imazamox was post-emergence treated at 0.02 kg ai/ha. Residues in plant at 0 DAT and seed at 82 DAT were 2.1 mg eq./kg and < 0.002 mg eq./kg, respectively (not further identified for component in plant and seed).

In the other two studies (indoor or outdoor), either [pyridine-6- ^{14}C]-imazamox or [imidazolinone-5- ^{14}C , 3- ^{15}N]-imazamox was applied post-emergence at rates of 0.051–0.089 kg ai/ha (outdoor) or 0.075 kg ai/ha (indoor), respectively. TRR in plant at 0 DAT were 2.2–3.9 mg eq./kg. Residues in forage (12–22 DAT) ranged from 0.04 mg eq./kg to 0.89 mg eq./kg. In straw and seed (78–90 DAT), residues were 0.088–1.1 mg eq./kg and 0.004–0.15 mg eq./kg, respectively. The majority of residues (80–99% TRR) in forage and straw samples were extracted with aqueous methanol or acidic aqueous methanol. Extractability in seeds was relatively low, 58–80% TRR.

In both labels, major components of the residues in forage were imazamox (16–42% TRR) and CL 263284 (36–54% TRR). In straw, major components were CL 263284 (44–50% TRR) and CL

312622 (up to 26% TRR); imazamox, < 2% TRR. In rape seed, major components of the residues were CL 263284 (up to 31%) or CL 189215 (up to 21%); imazamox, present at up to 13%.

The metabolic fate of [pyridine-6-¹⁴C]-imazamox in soya bean under outdoor field was studied with post-emergence treatment at a rate of 0.39 kg ai/ha. TRR in plant at 0 DAT was 55 mg eq./kg. Residues in forage (29–60 DAT) ranged from 0.13 mg eq./kg to 0.56 mg eq./kg. In straw and seed (153 DAT), residues were 0.08 mg eq./kg and 0.02 mg eq./kg, respectively. The majority of residues in forage (86–97%) were extracted with aqueous methanol and dichloromethane. Acidic and/or basic digestion for straw and seed samples extracted up to 53% and 57% of residues, respectively.

A major component of the residue in soya bean forage (29–60 DAT) was CL 189215 (32–36% TRR); imazamox, CL 263284 and CL 312622 each, present at 6–11% TRR. In soya bean seed, CL 263284 was a major component, representing 12% TRR.

Field peas were treated post-emergent, under outdoor growing conditions, with [pyridine-6-¹⁴C]-imazamox at a rate of 0.040 kg ai/ha. TRRs in whole plant at 0 DAT and 20 DAT were 1.1 mg eq./kg and 0.035 mg eq./kg, respectively. At 61 DAT, residues in all of the samples (pea foliage, immature peas, pea shells and pea pods) were < 0.01 mg eq./kg. In pea and pea hay (84 DAT), residues were 0.01 mg eq./kg and 0.05 mg eq./kg, respectively. The aqueous methanol extracted 61–99% of the TRR in whole plant. From the hay and peas, 58% and 39% of the TRR was extracted, respectively.

In pea hay, imazamox was present at 0.02 mg eq./kg (66% TRR) and other components (CL 263284, CL 189215) were negligible ($\leq$ 0.01 mg eq./kg).

Alfalfa was treated, post-emergent, in plots at different ages of crop and timing of application under outdoor conditions with [pyridine-6-¹⁴C]-imazamox at a rate of 0.13 kg ai/ha. TRR in alfalfa plants at 0 DAT was 6.5–15 mg eq./kg. Residues in forage (26 DAT) ranged from 0.18 mg eq./kg to 0.67 mg eq./kg. Residues in forage (1st, 2nd, 3rd cut at 26–111 DAT) and hay (1st, 2nd, 3rd cut at 28–160 DAT) were < 0.01–0.21 mg eq./kg and 0.02–0.83 mg eq./kg, respectively. The majority (65–99% TRR) of residues in forage and hay were extracted with a solvent mixture of methanol/acetone/water.

Residue components in alfalfa forage (26 DAT) were: imazamox, 3–6% TRR and CL 263284, CL 189215 and CL 312622, 14–26% TRR. For forage and hay samples, 1st, 2nd, 3rd cut, composition of the compounds were similar to those in forage at 26 DAT.

The metabolic fate of imazamox on imidazolinone-tolerant maize was investigated in two outdoor studies. Radiolabelled imazamox, either [pyridine-6-¹⁴C]-imazamox or a mixture of [pyridine-6-¹⁴C]-imazamox and [pyridine-6-¹³C]-imazamox was post- or pre-emergence (one day after seedling) treated at rates of 0.13–0.14 kg ai/ha. Residues in forage (14, 30 and 62 DAT) ranged from 0.011 mg eq./kg to 0.41 mg eq./kg. In fodder and grain (100–112 DAT), TRRs were 0.012–0.047 mg eq./kg and 0.010–0.01 mg eq./kg, respectively. 40–98% of the TRR in maize samples were extracted with aqueous methanol (40% TRR in fodder).

Major components of the residues in maize forage were imazamox (18–31% TRR) and CL 263284, (12–23% TRR). In fodder, CL 263284 was present at 15% TRR, < 1–7% TRR in the other components. In grain, major components were CL 263284 (20% TRR) and CL 189215 (15% TRR) with imazamox at low level, < 5% TRR.

The metabolic fate of imazamox on imidazolinone-tolerant spring wheat was investigated under indoor and outdoor conditions. Radiolabelled imazamox, [pyridine-6-¹⁴C]-imazamox or [imidazolinone-5-¹⁴C, 3-¹⁵N]-imazamox was post-emergence applied at rates of 0.14 or 0.76 kg ai/ha. Residues in forage (8–28 DAT) ranged from 0.10 mg eq./kg to 1.6 mg eq./kg. In straw and grain (62–70 DAT), residues were ranged in 0.16–3.2 mg eq./kg and 0.067–1.4 mg eq./kg, respectively. The 53–93% TRR in all wheat samples was extracted with methanol and water or acidic aqueous methanol (53% in straw).

Major components of the residues in spring wheat forage were imazamox (42–67% TRR) and CL 263284 (10–18% TRR). In straw, CL 263284 was most abundant (13–38% TRR). The other

components, imazamox (8–10% TRR), CL 189215 (3–15% TRR) and CL 312622 (6–17% TRR) were found at lower levels. In wheat grain, imazamox was most abundant component (40–70% TRR, 0.027–1.1 mg eq./kg); CL 263284 (7–10% TRR) and CL 189215 (3–4% TRR) were present at relatively low levels and CL 312622 was not detected.

The key step of the metabolism of imazamox in plant was the cleavage of the methyl ether group (demethylation) resulting in metabolite CL 263284. Subsequently, oxidation of the hydroxyl group of CL 263284 generated the dicarboxylic acid metabolite CL 312622, while glycosylation led to the glucose conjugate CL 189215.

In conclusion, in the edible portions of most treated food crops harvested at maturity, no or a very small residue of imazamox or its metabolites are expected to be found. However, for certain crops such as wheat, it is considered that the residues may be detected in wheat grain. In animal feed crops, imazamox, CL 263284, CL 189215 and CL 312622 are expected to be found above the LOQ.

Environmental fate

The Meeting received information on aerobic soil metabolism, photodegradation on the soil surface, hydrolysis and residues in succeeding crops.

Aerobic soil metabolism

Imazamox applied in sandy loam soil degraded rapidly. In three studies, half-life of imazamox was 28–38 days in sandy loam soil treated with imazamox at 0.050–0.10 kg ai/ha. However, in another study, half-life of imazamox treated in combinations of soil type (silty clay loam and silt loam) and temperature (10 °C and 20 °C) was in a range of 12–207 days (DT₉₀, 39–687 days). Imazamox was shown to be moderately persistent in aerobic soil. In all the studies, dicarboxylic acid (CL 312622) and hydroxy acid (CL 354825) metabolites were the major residue components. The parent and both metabolites were ultimately mineralized to CO₂.

Residues in succeeding crops

In a confined rotational crop study, [pyridine-6-¹⁴C]-imazamox was applied once to soya beans at the 4–6 leaf stage, in a sandy loam soil, at a rate of 0.072 kg ai/ha. One-hundred days after treatment, the soya bean crop was harvested. On the day of harvest, winter wheat was seeded into a subplot (100-day plant-back interval; PBI) of the treated field. Maize, radish, and lettuce were seeded into separate subplots at a 268-day PBI. Total radioactive residues were < 0.01 mg eq./kg in all rotational crops.

In another study, radiolabelled imazamox, either [imidazolinone-5-¹⁴C, 3-¹⁵N]-imazamox or [pyridine-3-¹⁴C, imidazolinone-3-¹⁵N]-imazamox were applied to bare sandy loam soil in plastic container followed by sowing spinach, white radish and spring wheat at PBIs of 1 month, 4 month and 1 year.

In spinach and white radish, residues were < 0.01 mg eq./kg at any PBIs. However, in spring wheat samples (forage, hay, straw, grain), total residues (TRR) were: 0.008–0.078 mg eq./kg at one month PBI (grain, 0.032–0.053 mg eq./kg), 0.004–0.132 mg eq./kg at four month PBI (grain 0.019–0.035 mg eq./kg) and < 0.001–0.052 mg eq./kg at one year PBI (grain, 0.002–0.004 mg eq./kg). In wheat grain at one month PBI, residues of imazamox and CL 263284 were 0.001–0.005 mg eq./kg.

In conclusion, residues of imazamox and the metabolites are expected to be less than 0.01 mg eq./kg in succeeding crops.

Photodegradation

Soil photolysis of [pyridine-6-¹⁴C]-imazamox was studied in a sandy loam soil, surface treated at a rate equivalent to 0.10 kg ai/ha and exposed for 30 days to artificial sunlight. The 92% imazamox at time-0 decreased to 74% imazamox at the end of the 30 days irradiation. A half-life of imazamox was calculated to be 65 days. A degradate, dicarboxylic acid CL 312622 (2% TAR in dark control), was formed at about 14% of the total residues after 30 days irradiation.

Imazamox is photodegraded with a half-life of 65 days on the surface of soil. Any degradate specific for photolysis was not found.

Hydrolysis

Imazamox is used for rice production. In the hydrolysis study under different pH conditions, imazamox was stable at pH 4, pH 7 and pH 9.

Methods of analysis

The Meeting received description and validation data for analytical methods for residue analysis of imazamox and its metabolites in various plant and animal commodities.

In general, the methods involved extraction of residues with acidic methanol-water solution, clean-up procedure by mainly solvent partitioning and solid phase extraction, and determination by HPLC-UV or LC-MS/MS. In the case of milk and fat, acetonitrile in hexane is used for the extraction step. Some methods employ capillary electrophoresis-UV or GC-NPD, in case of use of GC-NPD, where combined determination for imazamox-related residues is possible by converting to a common methylated product.

A number of specific methods for plant matrices were found suitable for analysis of imazamox, CL 263284, CL 189215 and CL 312622 with LOQ ranging 0.01–0.05 mg/kg for these analytes except that it was 0.1 mg/kg for alfalfa.

For animal matrices, one method determined by LC-MS/MS was submitted and found suitable for analysis of imazamox and CL 263284 with LOQs of 0.01 mg/kg for bovine matrices and poultry egg.

No multi-residue methods were submitted.

Stability of residues in stored analytical samples

The stability of imazamox and its metabolites during frozen storage of samples was investigated in a range of plant matrices for which supervised residue trials were submitted.

Compounds tested were imazamox, CL 263284, CL 189215 and CL 312622. Each compound was spiked to matrices at 0.1 to 1 mg/kg.

All of the compounds tested were found to be stable (> 70% remaining) at least during the storage periods tested: for imazamox, 3.6 years in soya bean (seed, forage and hay), 4 years in wheat (grain, straw, forage and hay), 2 years in maize (grain, ear and immature plant), 1.5 years in rape seed and 1.5 years in alfalfa (hay, forage and seed); for CL 263284, 3.6 years in soya bean (seed, forage and hay), 10 months in processed soya bean products, 4 years in wheat (grain, straw, forage and hay), 2 years in maize (grain, ear and immature plant) and peanut (hull and nutmeat) and 1.5 years in alfalfa (hay, forage and seed); for CL 189215, 10 months in soya bean seed and processed soya bean products, 2 years in peanut (hull and nutmeat) and 1.5 years in alfalfa (hay, forage and seed); and for CL 312622, 1.5 years in alfalfa (hay, forage and seed).

Definition of the residue

In metabolism studies of imazamox and the metabolites (CL 263284, CL 312622) in goats and hens, the compounds were mostly excreted (85–106%) unchanged through urine and faeces. In hen, no detectable residues were found in eggs or edible tissue. In goat, there were no detectable residues in milk and tissues except kidney. Residue in the kidney was predominantly the administered parent. Therefore, no residues of imazamox and related compounds are expected in livestock tissues, milk or eggs at expected livestock dietary burdens of less than 3 ppm.

The log P_{ow} for imazamox is 0.73 suggesting imazamox residues are not fat soluble.

In confined crop rotation studies, any imazamox related residues in succeeding crops were not detected above more than 0.01 mg/kg.

In primary crops, imazamox is either not found or found at very low levels ($< 30\%$ TRR, < 0.01 mg eq./kg) in non-tolerant food commodities and imidazolinone-tolerant food commodities (tolerant rape seeds, soya bean seeds, pea seeds, alfalfa, tolerant maize seeds). Imidazolinone-tolerant wheat grain, is the only food commodity, where parent is found at significant levels ($40\text{--}74\%$ TRR, $0.027\text{--}1.1$ mg eq./kg). Among the metabolites found, CL 263284 is the most predominant metabolite, although present at low levels in food commodities (up to 31% TRR, maximum 0.092 mg eq./kg). In supervised trials imazamox and CL 263284 are found at levels above the LOQ in food commodities (lentil seeds, sunflower seeds). In feed commodities (tolerant rape forage or straw, soya forage or straw, pea forage or hay, alfalfa forage or hay, tolerant maize forage or fodder, wheat forage, hay or straw), imazamox ($1.5\text{--}67\%$ TRR, $< 0.01\text{--}5.0$ mg eq./kg) and CL 263284 are more prominent ($8\text{--}54\%$ TRR, $< 0.01\text{--}1.9$ mg eq./kg).

Parent and CL 263284 are the only two compounds expected to be found in food commodities. Metabolite CL 263284 can also arise in plant commodities as a result of treatment with imazapic, another imidazolinone herbicide. Since CL 263284 cannot be seen as a marker for imazamox, the Meeting decided to define the residue for enforcement/monitoring as parent only.

Parent and CL 263284 are considered relevant for dietary risk assessment as the toxicity of CL 263284 is similar to that of imazamox in rat. Therefore, the Meeting decided to define the residue for dietary risk assessment as the sum of parent and CL 263284, expressed as imazamox.

The Meeting agreed the following residue definitions:

Definition of the residue for plant and animal commodities for compliance with the MRL: imazamox.

Definition of the residue for plant and animal commodities for the estimation of dietary intake: sum of imazamox and 5-(hydroxymethyl)-2-(4-isopropyl-4-methyl-5-oxo-2-imazazolin-2-yl) nicotinic acid (CL 263284), expressed as imazamox.

Residue is not fat-soluble.

Should imazamox be used on crops other than cereals, pulses and oilseeds, the residue definition may need to be revised.

Results of supervised residue trials on crops

The Meeting received supervised trial data for imazamox on legume vegetables (beans and peas), pulses (bean, pea, lentil and soya bean), cereals (rice and wheat), oil seed crops (peanut, rape and sunflower) and alfalfa.

In below, a maximum residue level was estimated based on residue concentration of imazamox. Total residue means sum of parent compound and CL 263284. Concentration of CL 263284 was expressed as parent equivalents (conversion factor, 1.048).

Legume vegetables

Beans

Residue trials on beans were performed in Denmark, Germany, France, the UK, Greece, Italy, Spain and the USA. The GAP for beans in Chile is a single early post-emergent application at 0.056 kg ai/ha with no PHI specified.

In six trials on French beans conducted in Italy (1×0.050 kg ai/ha at post-emergence) matching the Chile GAP, residues in bean pods with seeds were: parent < 0.05 (6) mg/kg and metabolite < 0.05 (6) mg/kg. Total residues in the bean pods with seeds were ($n=6$): < 0.1 (6) mg/kg.

In other six trials from USA (1×0.050 kg ai/ha at post-emergence) matching the Chile GAP, residue concentrations of imazamox (only measured) were < 0.05 (6) mg/kg in pods with seeds of snap beans.

Three trials conducted in France, the UK and Italy involved an earlier application timing and a higher rate (1×0.075 kg ai/ha at pre-emergence). Residues in pods with seeds of common bean (flageolet bean) were: parent < 0.01 (3) mg/kg, metabolite < 0.01 (3) mg/kg. Total residues in the bean pods with seeds were < 0.02 (3) mg/kg.

The Meeting considered that there was no expectation of residues above LOQ in bean pods with seeds. The Meeting estimated a maximum residue level of 0.05^* mg/kg and an STMR of 0 mg/kg for beans, except broad bean and soya bean (green pods and immature seeds).

Peas

Residue trials on peas were performed in Italy, France, Spain, the UK, Germany and the USA. The GAP for peas in France is for a single pre-emergent application post sowing per every 2 years, treating on soil at 0.075 kg ai/ha with a PHI of 63 days.

In nine trials conducted in Italy and France approximating the French GAP (1×0.068 – 0.075 kg ai/ha at pre-emergence, 70–110 day PHI), residues in pea seeds were: parent < 0.05 (9) mg/kg and metabolite < 0.05 (9) mg/kg. Total residues in pea seeds were ($n=9$): < 0.1 (9) mg/kg.

Another ten trials were conducted in Italy and Spain with post-emergence treatments at 0.050 – 0.052 kg ai/ha in eight trials and 0.073 – 0.075 kg ai/ha in two trials. The total residues were all < 0.1 (10) mg/kg.

The Meeting estimated a maximum residue level of 0.05^* mg/kg and an STMR of 0 mg/kg for peas, shelled (succulent seeds).

Pulses

Bean (dry)

Residue trials on dry beans were performed in France, Germany, Netherlands, the UK, Italy, Greece, Spain and the USA. The GAP for dry bean in the UK is a single pre-emergence application to soil at a rate of 0.075 kg ai/ha.

In four trials from France, the UK and Italy (1×0.075 kg ai/ha at pre-emergence, 91–108 day PHIs), approximating the UK GAP, residues in common bean seeds were: parent < 0.01 (4) mg/kg and metabolite < 0.01 (4) mg/kg. Total residues in seed of common beans were < 0.02 (4) mg/kg. In these trials, the total residues in whole plants harvested early (PHIs of 32–48 days) were < 0.02 mg/kg.

In two trials from the USA applied at a post-emergence timing and at a rate comparable to the UK GAP (1×0.070 kg ai/ha, 61–98 day PHIs), residue concentrations of the parent compound were also below LOQ, < 0.05 (2) mg/kg.

Pea (dry)

The GAP for dry peas in France is a single pre- or post-emergence application every 2 years at a rate of 0.075 kg ai/ha with a PHI of 63 days. A total of ten trials were conducted in France at the GAP rate (1×0.075 kg ai/ha at pre-emergence) but with longer PHIs (106–129 days). The residues measured for each compound or combined total residue were: parent < 0.05 (5) mg/kg, metabolite < 0.05 (5) mg/kg and total residue < 0.1 (10) mg/kg.

Lentil (dry)

Residue trials for lentil were available from Canada and the USA. In Canada, imazamox is approved for use in imidazolinone-tolerant lentils with a single early post-emergent application at a rate of 0.020 kg ai/ha (plus adjuvant) with a PHI of 60 days.

In a total of fourteen trials (1×0.015 – 0.021 kg ai/ha at post- or early post-emergent timings and a 60 day, or shorter, PHI (without use of an adjuvant), residues in imidazolinone-tolerant lentils

were: imazamox < 0.05 (9), 0.056, 0.060, 0.065, 0.12, 0.12 mg/kg and metabolite < 0.05 (14) mg/kg. Total residues were: < 0.1 (9), 0.11 (3), 0.17, 0.17 mg/kg.

Soya bean (dry)

Residue trials on soya bean were performed in France, Germany, Italy and Canada. The GAP for soya bean in Brazil is for a single post-emergence application at a rate of 0.049 kg ai/ha (plus adjuvant) with a PHI of 70 days.

In nine trials conducted in France, Germany and Italy (1×0.042–0.045 kg ai/ha at post- or early post-emergence, 69–104 days of PHI, with or without adjuvant) approximating the Brazilian GAP, residues in soya bean seeds were: imazamox < 0.01 (7) mg/kg and metabolite < 0.01 (7) mg/kg. Total residues were < 0.02 (7) mg/kg. Of which five trials showed no effect of adjuvant on the level of residue in dry seed.

In two trials conducted in Canada at an exaggerated rate of 0.070–0.071 kg ai/ha (single post-emergence application, 106–112 day PHIs), total residues were all < 0.05 (2) mg/kg.

The Meeting estimated a maximum residue level of 0.05* mg/kg and an STMR of 0 mg/kg for bean and pea in dry, respectively.

For lentil (dry), the Meeting estimated a maximum residue level of 0.2 mg/kg and an STMR of 0.1 mg/kg.

For soya bean (dry), the Meeting estimated a maximum residue level of 0.01* mg/kg and an STMR of 0 mg/kg.

Cereals

Rice

Residue trials on imidazolinone-tolerant rice were performed in Italy and Spain. The use pattern in Spain is twice post-emergence treatment at a rate of 0.035 kg ai/ha with an interval of 14–21 days and no PHI, applying on imidazolinone-tolerant rice plant in water or dry planted.

In a total of six trials on imidazolinone-tolerant rice (2×0.035–0.038 kg ai/ha at BBCH 13–14 and BBCH 25–31, 10–12 day interval) from Italy and Spain matching the Spain GAP, residues in rice grain were: imazamox < 0.01 (6) mg/kg and metabolite < 0.01 (3), 0.02, 0.03 (2) mg/kg. Total residues were: < 0.02 (3), 0.030, 0.040 (2) mg/kg.

The Meeting estimated a maximum residue level of 0.01* mg/kg and an STMR of 0.025 mg/kg for rice.

Wheat

Residue trials on imidazolinone-tolerant wheat were performed in France, Italy, Canada and the USA. The GAP for wheat (imidazolinone-tolerant) in Argentina is a single early post-emergence application at 0.070 kg ai/ha with no PHI.

In a total of twelve trials from France (eight trials), Italy (four trials) and USA (one trial) (1×0.067–0.075 kg ai/ha at post-emergence) matching the GAP, residues in imidazolinone-tolerant wheat grain were: imazamox < 0.05 (13) mg/kg and metabolite < 0.05 (13) mg/kg. Total residues were < 0.1 (13) mg/kg.

The Meeting considered that there is no expectation of residues above the LOQ for wheat grain and agreed to estimate a maximum level of 0.05* mg/kg and an STMR of 0.1 mg/kg for wheat.

Oilseeds

Peanuts

Five trials were conducted in Brazil with a single post-emergence application (BBCH 71–79) at a rate of 0.056 kg ai/ha with a PHI of 43–50 days without use of an adjuvant, matching the GAP for peanut in South Africa (1×0.048 kg ai/ha plus adjuvant) at post-emergence with a PHI 50 days. The residues in peanut kernels were: imazamox < 0.01 (5) mg/kg and metabolite < 0.01 (5) mg/kg. Total residues were < 0.02 (5) mg/kg.

Although only a relatively small number of trials were available, there is no expectation of residues above the LOQ in peanuts. The Meeting estimated a maximum residue level of 0.01* mg/kg and an STMR of 0 mg/kg.

Rape seed

Residue trials on imidazolinone-tolerant oilseed rape were conducted in France, Germany, the UK and Canada and Argentina. The GAP for imidazolinone-tolerant rape in Chile is a single early post-emergence application at a rate of 0.056 kg ai/ha with no PHI. In six trials from Germany, France and Argentina (1×0.050 kg ai/ha at post-emergence) matching the Chile GAP, residues in rape seed (imidazolinone-tolerant variety) were: imazamox < 0.01 (2), < 0.05 (4) mg/kg and metabolite < 0.01 (2), < 0.05 (4) mg/kg. Total residues were < 0.02 (2), < 0.1 (4) mg/kg.

In another eighteen trials from France, Germany and the UK, an exaggerated rate of 0.073–0.079 kg ai/ha did not lead to above LOQ (< 0.05 mg/kg) for total residues in seeds of imidazolinone-tolerant oilseed rape.

The Meeting considered that there is no expectation of residues above the LOQ in rape seeds.

The Meeting estimated a maximum residue level of 0.05* mg/kg, an STMR of 0 mg/kg for rape seed.

Sunflower seed

Residue trials on imidazolinone-tolerant sunflower were conducted in France, Germany, UK, Italy, Spain, Argentina, Canada and USA. In Canada, critical GAP for sunflower (imidazolinone-tolerant) is a single early post-emergence application at a rate of 0.015 kg ai/ha with a specified PHI of 60 days and an addition of adjuvant.

A total of six trials conducted in Canada and USA (1×0.015–0.019 kg ai/ha at PHIs of 58–68 days with or without adjuvant) approximated the Canadian GAP. The residues in seeds of imidazolinone-tolerant sunflower were: imazamox < 0.05 (2), 0.05 (2), 0.07, 0.14 mg/kg and metabolite < 0.05, 0.06, 0.10, 0.16, 0.17, 0.19 mg/kg. Total residues were: < 0.1, 0.13, 0.15, 0.22, 0.25, 0.30 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR of 0.19 mg/kg for sunflower seed.

Legume animal feeds

Alfalfa

Residue trials on alfalfa were performed in Greece, Spain and USA. The GAP for alfalfa in France is a single post-emergence application at a rate of 0.067 kg ai/ha with a PHI of 30 days.

In a total of seven trials from Greece, Spain and USA (1×0.067–0.075 kg ai/ha at PHIs of 26–30 days) matching the French GAP, residues in alfalfa forage were as received basis: imazamox < 0.1 (7) mg/kg and metabolite < 0.1 (7) mg/kg. Total residues in alfalfa forage were < 0.2 (7) mg/kg. For alfalfa hay, residues were as received basis: imazamox < 0.1 (5) mg/kg and metabolite < 0.1 (3), 0.19, 0.29 mg/kg. Total residues were < 0.2 (3), 0.32, 0.41 mg/kg in hay.

The Meeting estimated a median residue of 0 mg/kg and a highest residue of 0.2 mg/kg for alfalfa forage.

For alfalfa hay, the Meeting estimated a maximum residue level of 0.1* mg/kg, a median residue of 0.20 mg/kg and a highest residue of 0.41 mg/kg.

Pea vines (green) and pea fodder

Five trials approximating the French GAP for pea (single pre-emergence post-sowing per every 2 years at 0.075 kg ai/ha) were conducted in France with PHIs of 88–110 days. Residues in pea vines, (pods + haulm) on an as received basis, were: imazamox < 0.05 (5) mg/kg and metabolite < 0.05 (5) mg/kg. Total residues in pea vines were < 0.1 mg/kg.

Ten trials approximating the French GAP for field pea (single pre- or post-emergence application per every 2 years at a rate of 0.075 kg ai/ha) were conducted in France with PHIs of 106–120 days. Residues in pea fodder (straw or pods + haulm) on an as received basis, were: imazamox < 0.05 (5) mg/kg and metabolite < 0.05 (4), 0.17 mg/kg. Total residues were < 0.05 (5), < 0.1(4) and 0.22 mg/kg.

The Meeting estimated a median residue of 0.1 mg/kg and a highest residue of 0.1 mg/kg for pea vines. For pea fodder, the Meeting estimated a maximum residue level of 0.05* mg/kg, a median residue of 0.075 mg/kg and a highest residue of 0.22 mg/kg.

Soya bean, forage and fodder

Four trials were conducted in France and Germany approximating the Brazil GAP for soya bean (1×0.049 kg ai/ha). Residues in soya bean forage harvested at 27–28 PHIs were as received basis: imazamox < 0.01 (4) mg/kg and metabolite < 0.01 (4) mg/kg. Total residues were < 0.02 (4) mg/kg. For fodder, residues were as received basis: imazamox < 0.01 (4) mg/kg and metabolite < 0.01 (2), 0.01, 0.05 mg/kg. Total residues were < 0.02 (2), 0.02, 0.06 mg/kg in soya bean fodder.

For soya bean forage, the Meeting estimated a median residue of 0.02 mg/kg and a highest residue of 0.02 mg/kg.

For soya bean fodder, the Meeting estimated a maximum residue level of 0.01* mg/kg, a median residue of 0.02 mg/kg and a highest residue of 0.06 mg/kg.

Forage and fodder of cereal grains and grasses

Rice straw and fodder

In six trials conducted in Italy and Spain matching the Spain GAP (post-emergence treatment on imidazolinone-tolerant rice plant, water or dry planted, 2× 0.035 kg ai/ha, 14–21 day interval), residues in imidazolinone-tolerant rice straw (77–90 day PHIs) were as received basis: imazamox < 0.01 (6); metabolite < 0.01 (3), 0.01, 0.02 (2) mg/kg. Total residues were < 0.02 (3), 0.02, 0.03 (2) mg/kg in straw.

The Meeting estimated a maximum residue level of 0.01* mg/kg, a median residue of 0.02 mg/kg and an highest residue of 0.03 mg/kg in rice straw.

Wheat forage and straw

Trials on imidazolinone-tolerant wheat were conducted in Canada approximating the GAP for imidazolinone-tolerant wheat in Canada (single early post-emergence application at 0.020 kg ai/ha). In Canada, grazing and cutting for hay are not permitted within 4 days and 42 days of application, respectively. Five trials for forage (7, 14 day PHIs) and four trials (42, 56 day PHIs) for hay and five trials (72–90 PHIs) for straw matched the Canadian GAP on feedstuffs.

Residues in imidazolinone-tolerant wheat forage were as received basis: imazamox < 0.05 (2), 0.05, 0.13, 0.15 mg/kg; metabolite < 0.05 (5) mg/kg. Total residues were < 0.1 (2), 0.10, 0.20, 0.23 mg/kg.

Residues in imidazolinone-tolerant wheat hay were as received basis: imazamox < 0.05 (4) mg/kg; metabolite < 0.05 (4) mg/kg. Total residues were < 0.1 (4) mg/kg.

Residues in imidazolinone-tolerant wheat straw were as received basis: imazamox < 0.05 (5) mg/kg; metabolite < 0.05 (5) mg/kg. Total residues were < 0.1 (5) mg/kg.

For wheat forage, the Meeting estimated a median residue of 0.1 mg/kg and a highest residue of 0.23 mg/kg.

For wheat hay and straw, the Meeting estimated a maximum residue level of 0.05* mg/kg, a median residue of 0 mg/kg and a highest residue of 0.1 mg/kg.

Miscellaneous Fodder and Forage crops

Rape seed forage

In four residue trials on imidazolinone-tolerant oilseed rape conducted in France and Germany, matching the GAP of Chile for oilseed rape (imidazolinone-tolerant rape, a single early post-emergence application, 0.056 kg ai/ha), residues in whole plants without roots (12–19 day PHIs) on an as received basis were: imazamox, 0.05, 0.12, 0.14 0.19 mg/kg; metabolite 0.52, < 0.05, 0.21, 0.28 mg/kg. Total residues were: 0.10, 0.33, 0.42 and 0.71, mg/kg.

The Meeting estimated a median residue of 0.38 mg/kg and an highest residue of 0.71 mg/kg for rape forage.

Fate of residues during processing

High temperature hydrolysis

The hydrolysis of [pyridine-3-¹⁴C, imidazolinone-3-¹⁵N]-imazamox was investigated in aqueous buffer solutions. After pasteurization (20 minutes at 90 °C, pH 4), baking/brewing/boiling (60 minutes at 100 °C, pH 5) and sterilization (20 minutes at 120 °C, pH 6), 98–104% of the applied radioactivity remained in the test solutions, where the detectable radioactive component was unchanged imazamox only. Imazamox was stable under such simulated processing conditions.

Processing

Information on processing of wheat and sunflower seed were available for an estimation of maximum residue level and STMR-P for the processed product. Estimated processing factors, maximum residue levels and STMR-Ps are summarized below. A maximum residue level and STMR-P value for processed product were calculated with imazamox residue and STMR value (total residue of imazamox and CL 263284), respectively.

RAC and processed product	RAC		Pf, best estimate (individual Pf)		Processed product	
	Estimated maximum residue level (mg imazamox/kg)	STMR (mg total/kg)	Imazamox	Total residue	Estimated maximum residue level (mg imazamox/kg)	STMR-P total residue (mg total/kg)
Wheat	0.05*	0.1				
Wheat germ			2.7	2.2	0.1	0.22
Wheat bran, unprocessed			3.9	3.4	0.2	0.34
Wheat flour			1.2	1.2	0.06	0.12
Wheat asp. grain fraction			1.0	1.0		0.10
Wheat by products (middling, shorts)			2.4 (1.6, 3.2)	2.1 (1.5, 2.6)		0.21
Sunflower seed	0.3	0.19				
Sunflower seed meal			2.3	2.5 (1.9, 3.0)		0.48
Sunflower refined oil				< 0.5 (< 0.2, < 0.5)		0.095

The Meeting estimated maximum residue levels of 0.1 mg/kg for wheat germ and 0.2 mg/kg for wheat bran, unprocessed.

Residues in animal commodities

Farm animal feeding studies

Information on farm animal feeding studies was not submitted.

Estimation of dietary burdens

Dietary burden calculations for beef cattle and dairy cattle and poultry are provided below. The dietary burdens were estimated using the OECD diets listed in Appendix IX of the 2009 edition of the FAO Manual.

Summary of livestock dietary burden (ppm of dry matter diet)

	US-Canada		EU		Australia		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	0.230	0.178	0.821	0.463	2.37 ^a	1.27 ^c	0.217	0.193
Dairy cattle	0.670	0.404	0.0749	0.0433	1.50 ^b	0.765 ^d	0.256	0.197
Poultry broiler	0.278	0.278	0.179	0.179	0.200	0.200	0.052	0.023
Poultry layer	0.278	0.278	0.75 ^e	0.478 ^f	0.188	0.188	0.072	0.072

^a Highest maximum beef cattle dietary burden suitable for MRL estimates for mammalian meat

^b Highest maximum dairy cattle dietary burden suitable for MRL estimates for milk

^c Highest mean beef cattle dietary burden suitable for STMR estimates for mammalian meat

^d Highest mean dairy cattle dietary burden suitable for STMR estimates for milk

^e Highest maximum poultry dietary burden suitable for MRL estimates for poultry meat and eggs.

^f Highest mean poultry dietary burden suitable for STMR estimates for poultry meat and eggs

Animal commodity maximum residue levels

Maximum estimated dietary burdens for beef cattle and dairy cattle were 1.4 and 1.1 ppm, respectively. These dietary burdens were similar to the dose rates in the metabolism studies in lactating goats (< 0.01 mg eq./kg at 2.1 ppm for imazamox; < 0.01 mg eq./kg at 2.3 ppm for CL 263284). As no residues in cattle meat and milk are expected, the Meeting estimated a maximum residue level of 0.01* mg/kg and HR and STMR of 0 in each animal commodity: milk, meat (mammalian except marine mammals) and edible offal and fat.

In poultry, the maximum dietary burden, 0.75 ppm was 3 times lower than dose rates in metabolism studies in laying hens (< 0.01 mg eq./kg at 2.1 ppm for imazamox and CL 263284 each). The Meeting estimated a maximum residue level of 0.01* mg/kg and HR and STMR of 0 for poultry meat, poultry edible offal, poultry fat and eggs.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for plant and animal commodities for compliance with the MRL: *imazamox*

Definition of the residue for plant and animal commodities for estimation of dietary intake: *sum of imazamox and 5-(hydroxymethyl)-2-(4-isopropyl-4-methyl-5-oxo-2-imazazolin-2-yl) nicotinic acid (CL 263284), expressed as imazamox*

Residue is not fat soluble.

Commodity		Recommended MRL (mg/kg)		STMR or STMR-P (mg/kg)	HR, HR-P, highest residue (mg/kg)
CCN	Name	New	Previous		
VP 0061	Beans, except broad bean and soya bean (green pods and immature seeds)	0.05*		0	
VP 0064	Peas, shelled (succulent seeds)	0.05*		0	
VD 0071	Bean (dry)	0.05*		0	
VD 0072	Pea (dry)	0.05*		0	
VD 0533	Lentil (dry)	0.2		0.10	
VD 0541	Soya bean (dry)	0.01*		0	
GC 0649	Rice	0.01*		0.025	
GC 0654	Wheat	0.05*		0.1	
CF 1210	Wheat germ	0.1		0.22	
CM 0654	Wheat bran, unprocessed	0.2		0.34	
CF 1211	Wheat flour			0.12	
SO 0697	Peanut	0.01*		0	
SO 0495	Rape seed	0.05*		0	
SO 0702	Sunflower seed	0.3		0.19	
AL 1020	Alfalfa fodder	0.1*		0.20	0.41
AL 0072	Pea hay or fodder	0.05*		0.075	0.22
AL 0541	Soya bean fodder	0.01*		0.02	0.06
AS 0649	Rice straw and fodder, dry	0.01*		0.02	0.03
AS 0654	Wheat straw and fodder, dry	0.05*		0	0.1
MO 0105	Edible offal (mammalian)	0.01*		0	0
PE 0112	Eggs	0.01*		0	0
MM 0095	Meat (from mammals other than marine mammals)	0.01*		0	0
MF 0100	Mammalian fats (except milk fats)	0.01*		0	0
ML 0106	Milks	0.01*		0	0
PM 0110	Poultry meat	0.01*		0	0
PF 0111	Poultry fats	0.01*		0	
PO 0111	Poultry, Edible offal of	0.01*		0	0
	Sunflower seed refined oil			0.095	

For calculating animal dietary burden

Commodity		Median residue or STMR-P (mg/kg)	Highest residue or HR-P (mg/kg)
CCN	Name		
AL 1021	Alfalfa forage (green)	0	0.2
AL 0528	Pea vines (green)	0.1	0.1
AL 1265	Soya bean forage (green)	0.02	0.02
	Wheat forage	0.1	0.23
AV 0495	Rape seed forage	0.38	0.71
	Wheat aspirated grain fraction		0.10
	Wheat by products (middling, shorts)		0.21
	Sunflower seed meal		0.48

DIETARY RISK ASSESSMENT

Long-term intake

The ADI for imazamox is 0–3 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for imazamox were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the present JMPR. The results are shown in Annex 3 of the 2014 JMPR Report. The IEDIs were 0% of the maximum ADI. The Meeting concluded that the long-term intake

of residues of imazamox from uses considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The ARfD for imazamox is 3 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for imazamox were calculated for the food commodities for which STMRs or HRs were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4. The IESTIs were 0% of the ARfD for children and the general population.

The Meeting concluded that the short-term intake of residues of imazamox from uses considered by the present Meeting are unlikely to present a public health concern.

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ID-244-029	Fletcher, JS	2002 a	BAS 720 H (CL 299263): Laboratory validation of LC/MS determinative and LC/MS/MS confirmatory method M 3515 for the determination of BAS 720 H and CL 263284 residues in plant matrices BASF Corp. Agro Research, Princeton NJ, United States of America Unpublished
ID-326-024	Fletcher, JS	2001 a	CL 299263 (Imazamox): Freezer storage stability of CL 299263, CL 263284, CL 189215 and CL 312622 residues in alfalfa seed, forage and hay BASF Corp. Agro Research, Princeton NJ, United States of America Unpublished
ID-790-014	Fletcher, JS	2001 b	CL 299263 (Imazamox): Freezer storage stability of CL 299263, (and its metabolites) CL 263284, CL 189215 and CL 312622 residues in alfalfa seed, forage and hay—Report amendment No. 01 BASF Corp. Agro Research, Princeton NJ, United States of America Unpublished
ID-432-001	Foss, JA	1994 b	An oral developmental toxicity (embryo-fetal toxicity / teratogenicity) study with AC 299, 263 in rats Argus Research Laboratories Inc., Horsham PA, United States of America Unpublished
2010/1057139	Freitas & Guimaraes, S	2010 a	Study of Imazapyr and Imazamox residues in sunflower (seed), after treatment with Sweeper 70 DG and Clearsol DF, under field conditions in Argentina BASF SA, Guaratingueta, Brazil Unpublished
2010/1057139	Freitas & Guimaraes, S	2010 a	Study of Imazapyr and Imazamox residues in sunflower (seed), after treatment with Sweeper 70 DG and Clearsol DF, under field conditions in Argentina BASF SA, Guaratingueta, Brazil Unpublished
2013/1085609	Funk, D & Possienke, M	2013 b	Nachbaustudie mit ¹⁴ C-BAS 720 H—Confined rotational crop study with ¹⁴ C-BAS 720 H BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished
2013/1233155	Funk, D & Taraschewski, I	2013 a	Comparative in-vitro-metabolism with ¹⁴ C-BAS 720 H BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished
ID-452-001	Futagawa, H	1995 a	AC 299263 technical: General pharmacological study The Institute of Environmental Toxicology, Kodaira-shi Tokyo 187, Japan Unpublished
2012/1084183	Gabriel, EJ	2013 b	Study on the residue behaviour of Imazamox in sunflower after treatment with BAS 720 06 H and BAS 160 00 S under field conditions in Germany, the United Kingdom, southern France and Italy, 2011 SGS Institut Fresenius GmbH, Taunusstein, Germany Fed. Rep. Unpublished
2012/1272623	Gabriel, EJ	2013 c	Study on the residue behaviour of Imazamox (BAS 720 H) in rice after treatment with BAS 720 06 H, applied with the adjuvant Dash (BAS 160 00 S) under field conditions in Italy and Spain, 2012 SGS Institut Fresenius GmbH, Taunusstein, Germany Fed. Rep. Unpublished
2012/1278352	Gabriel, EJ	2013 a	Study on the residue behaviour of BAS 720 H (Imazamox) in rice after treatment with BAS 720 06 H under field conditions in Italy and Spain, 2011 SGS Institut Fresenius GmbH, Taunusstein, Germany Fed. Rep.

Code	Author(s)	Year	Title, Institute, Report reference
			Unpublished
ID-640-002	Gatterdam, PE	1994 b	Confined accumulation study of carbon-14 labelled CL 299, 263 using radishes, corn, lettuce and wheat as rotational crops American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-416-001	Glaza, SM	1992 a	Dermal sensitization study with AC 299, 263 in guinea pig Hazleton Laboratories America Inc., Madison WI, United States of America Unpublished
ID-416-002	Glaza, SM	1996 a	Dermal sensitization study of AC 299, 263 in guinea pigs—Maximization test Corning Hazleton Inc., Madison WI, United States of America Unpublished
2012/7004251	Gooding, R	2013 a	Validation of BASF analytical method D1102: Method for determination of BAS 720 H and Its metabolites CL 312622 and CL 354825 residues in soil using LC-MS/MS BASF Crop Protection, Research Triangle Park NC, United States of America Unpublished
2013/7002842	Gooding, RF	2013 a	Validation of BASF analytical method D0303: Method for the determination of BAS 720 H (CL 299263) and its metabolite CL 263284 in animal matrices using LC-MS/MS BASF Crop Protection, Research Triangle Park NC, United States of America Unpublished
2012/1064722	Grosshans, F <i>et al.</i>	2012 b	Metabolism of ¹⁴ C-Imazamox in wheat BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished
ID-731-015	Hallman, DC	1999 a	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in established alfalfa after treatment with AC 299263 70DG herbicide in Iowa American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-016	Hallman, DC	1999 b	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in established alfalfa after treatment with AC 299263 1AS herbicide in California American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-017	Hallman, DC	1999 c	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in established alfalfa after early season treatment with AC 299263 1AS herbicide in Pennsylvania American Cyanamid Co., Princeton NJ, United States of America Unpublished
2011/1286214	Hassink, J	2012 a	Imazamox: Hydrolysis at 90 °C, 100 °C and 120 °C BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished
ID-731-014	Higham, JW	1999 a	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 263284, CL 189215 and CL 312632 residues in newly-seeded alfalfa after treatment with AC 299263 1AS herbicide in Washington State American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-022	Higham, JW	1999 b	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in newly-seeded alfalfa after treatment with AC 299263 (70DG) herbicide in Georgia American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-432-002	Hoberman, AM	1995 c	An oral developmental toxicity (embryo-fetal toxicity / teratogenicity) definitive study with AC 299, 263 in rabbits Argus Research Laboratories Inc., Horsham PA, United States of America Unpublished
ID-413-001	Hoffman, GM	1994 a	Acute inhalation toxicity study with AC 299, 263 in rats Bio/dynamics Inc., East Millstone NJ, United States of America Unpublished
ID-322-002	Holman, J	1997 a	Hydrolysis of AC 299, 263 American Cyanamid Co., Ewing NJ, United States of America Unpublished
ID-322-002	Holman, J	1997 b	Hydrolysis of AC 299, 263 American Cyanamid Co., Ewing NJ, United States of America Unpublished
ID-360-001	Humphries, K	1996 a	CL 299, 263—Spectral database (Report amendment 2) American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-440-001	Johnson, DH	1994 b	Metabolic fate of carbon-14 labelled CL 299263 in tissues and eggs of the laying hen American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-440-002	Johnson, DH	1994 a	Metabolic fate of carbon-14 labelled CL 299263 in the milk and edible tissues of the lactating goat American Cyanamid Co., Princeton NJ, United States of America Unpublished

Code	Author(s)	Year	Title, Institute, Report reference
ID-640-005	Johnson, DH	1996 b	CL 299263: Metabolism of carbon-14 labelled CL 299263 in wheat under field conditions—Report amendment number 1 (Amendment number 1 included—in the first place) American Cyanamid Co., Princeton NJ, United States of America Unpublished
2002/5004111	Johnston, R	2003 a	The magnitude of Imazamox (BAS 720 H) and Imazapyr (BAS 693H) residues in sunflower and sunflower processed fractions BASF Corp. Agricultural Products Center, Research Triangle Park NC, United States of America Unpublished
2002/5004111	Johnston, R	2003 a	The magnitude of Imazamox (BAS 720 H) and Imazapyr (BAS 693H) residues in sunflower and sunflower processed fractions BASF Corp. Agricultural Products Center, Research Triangle Park NC, United States of America Unpublished
2002/5004111	Johnston, R	2003 a	The magnitude of Imazamox (BAS 720 H) and Imazapyr (BAS 693H) residues in sunflower and sunflower processed fractions BASF Corp. Agricultural Products Center, Research Triangle Park NC, United States of America Unpublished
2008/7008101	Johnston, RL	2009 a	Magnitude of Imazamox and Imazethapyr residues in sunflower RAC and processed fractions following applications of BAS 724 00 H BASF Agricultural Research Center, Research Triangle Park NC, United States of America Unpublished
2008/7008101	Johnston, RL	2009 a	Magnitude of Imazamox and Imazethapyr residues in sunflower RAC and processed fractions following applications of BAS 724 00 H BASF Agricultural Research Center, Research Triangle Park NC, United States of America Unpublished
2002/5004302	Jordan, JM	2003 a	Independent method validation of BASF analytical method M 3519 entitled BAS 720 H and BAS 685 H: Determination and confirmation of BAS 720 H, CL 263284, CL 189215, CL 312622, BAS 685 H, CL 288511 and CL 182704 residues in lentils BASF Agro Research RTP, Research Triangle Park NC, United States of America Unpublished
2002/5004302	Jordan, JM	2003 a	Independent method validation of BASF analytical method M 3519 entitled BAS 720 H and BAS 685 H: Determination and confirmation of BAS 720 H, CL 263284, CL 189215, CL 312622, BAS 685 H, CL 288511 and CL 182704 residues in lentils BASF Agro Research RTP, Research Triangle Park NC, United States of America Unpublished
IA-440-001	Kao, LM	1994 a	CL 263,284: Metabolism of [¹⁴ C] CL 263,284 in the lactating goat American Cyanamid Co., Princeton NJ, United States of America Unpublished
2013/1235040	Kapp, MD,& Landsiedel, R	2013 a	Reg.No. 4110542 (metabolite of BAS 720 H, Imazamox)—In vitro gene mutation test in CHO cells (HPRT locus assay) BASF SE, Ludwigshafen/Rhein, Germany Fed.Rep. Unpublished
ID-425-002	Kelly, CM	1994 b	90-day dietary toxicity study with AC 299, 263 in purebred beagle dogs Pharmaco LSR Inc., East Millstone NJ, United States of America Unpublished
ID-427-001	Kelly, CM	1995 c	One-year dietary toxicity study with AC 299, 263 in purebred beagle dogs (Volume I and II) Pharmaco LSR Inc., East Millstone NJ, United States of America Unpublished
ID-428-001	Kelly, CM	1995 d	An oncogenicity study with AC 299, 263 in mice (Volume I–V) Pharmaco LSR Inc., East Millstone NJ, United States of America Unpublished
ID-731-009	Kenny, S	1999 a	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in newly seeded alfalfa after treatment with AC 299263 70DG herbicide in South Dakota, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-004	Kenny, SM	1999 a	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in established alfalfa after early season treatment with AC 299263 70DG herbicide in North Dakota American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-005	Kenny, SM	1999 b	CL 299263 (Imazamox): CL 299263, CL 189215, CL 312622 and CL 263284 residues in alfalfa grown for seed production after treatment with AC 299263 1AS herbicide in Washington American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-006	Kenny, SM	1999 c	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in alfalfa grown for seed production after treatment with AC 299263 70DG herbicide in California, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished

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ID-731-007	Kenny, SM	1999 d	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues and their decline over time in established alfalfa after treatment with AC 299263 1AS herbicide in California, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-008	Kenny, SM	1999 e	CL 299263 (Imaxamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in alfalfa grown for seed production after treatment with AC 299263 1AS herbicide in California, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-010	Kenny, SM	1999 f	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in established alfalfa after treatment with AC 299263 70DG herbicide in Indiana, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-011	Kenny, SM	1999 g	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in established alfalfa after early season treatment with AC 299263 1AS herbicide in Indiana, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-013	Kenny, SM	1999 h	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in established alfalfa after treatment with AC 299263 1AS herbicide in Ohio, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-018	Kenny, SM	1999 i	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in newly seeded alfalfa after treatment with AC 299263 1AS herbicide in Colorado, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-020	Kenny, SM	1999 j	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in alfalfa grown for seed production after treatment with AC 299263 70DG herbicide in Idaho, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-021	Kenny, SM	1999 k	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues and their decline over time in established alfalfa after early season treatment with AC 299263 70DG herbicide in Illinois, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-324-003	Knoch, E	1996 a	Determination of the direct phototransformation of AC299263 in a buffered medium at pH 7 Institut Fresenius Chemische und Biologische Laboratorien GmbH, Herten, Germany Fed. Rep. Unpublished
2007/1023133	Kreke, N	2009 a	Residues at harvest of Imazamox and Bentazone in green pea (RAC seed, pods, and plant without pods) following one treatment with BAS 762 01 H (22.4/480 g/L) from eight open field trials in Northern and Southern Europe in 2007/2008 Harlan Laboratories Ltd., Itingen, Switzerland Unpublished
ID-731-012	Kukel, C	1999 a	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in alfalfa grown for seed production after treatment with AC 299263 1AS herbicide in Washington American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-019	Kukel, C	1999 b	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in alfalfa grown for seed production after treatment with AC 299263 70DG herbicide in Idaho American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-435-004	Kumaroo, VP	1994 b	AC 299, 263: Test for chemical induction of chromosome aberration in cultured Chinese hamster ovary (CHO) cells with and without metabolic activation SITEK Research Laboratories, Rockville MD, United States of America Unpublished
2012/1294678	Lehmann, A	2013 a	Validation of BASF method no. L0188/01: Method for the determination of Imazamox (BAS 720 H, Reg.No. 4096483) and its metabolites Reg.No. 4110542 (CL312622), Reg.No. 4110773 (CL263284) and Reg.No. 4110445 (CL189215) in plant matrices BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished
2012/1294678	Lehmann, A	2013 a	Validation of BASF method no. L0188/01: Method for the determination of Imazamox (BAS 720 H, Reg.No. 4096483) and its metabolites Reg.No. 4110542 (CL312622), Reg.No. 4110773 (CL263284) and Reg.No. 4110445 (CL189215) in plant matrices BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished

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2013/1177533	Lehmann, A	2013 b	Amendment No. 1: Validation BASF No. L0188/01—Method for determination of Imazamox (BAS 720 H, Reg.No. 4096483) and its metabolites Reg.No. 4110542 (CL312622), Reg.No. 4110773 (CL263284) and Reg.No. 4110445 (CL189215) in plant matrices BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished
2013/1177533	Lehmann, A	2013 b	Amendment No. 1: Validation BASF No. L0188/01—Method for determination of Imazamox (BAS 720 H, Reg.No. 4096483) and its metabolites Reg.No. 4110542 (CL312622), Reg.No. 4110773 (CL263284) and Reg.No. 4110445 (CL189215) in plant matrices BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished
2010/1090461	Leite, R	2008 a	SOP-PA.0288_E Rev.03—Determination of Imidazolinones residues and its metabolites in several matrices and processed fractions by LC/MS/MS BASF SA, Guaratingueta, Brazil Unpublished
2011/1207286	Leite, R., & Alves, M	2011 a	Investigation study of the storage stability of Imazapyr (BAS 693 H), Imazapic (BAS 715 H) and its metabolites CL 263,284 and CL 189,215 in soya bean and processed fractions BASF SA, Guaratingueta, Brazil Unpublished
2002/5004457	Leonard, R	2003 a	Magnitude of residue of BAS 720 H and BAS 685 H and their related metabolites residues in lentils after treatment with BAS 724 00H BASF Corp. Agricultural Products Center, Research Triangle Park NC, United States of America Unpublished
2002/5002749	Malinsky, DS	2002 a	Independent method validation of BASF analytical method M3515 entitled: BAS 720 H (CL 299263): LC/MS determinative and LC/MS/MS confirmatory method for the determination and confirmation of BAS 720 H and CL 263284 residues in plant matrices BASF Corp. Agro Research, Princeton NJ, United States of America Unpublished
ID-324-001	Mangels, G	1994 a	AC 299,263: Estimation of the photochemical oxidation rate in the atmosphere American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-306-002	Martin, C	1997 a	Henry's Law constant calculation for Imazamox (AC 299, 263) American Cyanamid Co., Princeton NJ, United States of America Unpublished
2012/7003612	McCall, WS & Blood, A	2013 b	BAS 720 H Imazamox: Soil photolysis BASF Crop Protection, Research Triangle Park NC, United States of America Unpublished
ID-640-003	McDonnell, RJ	1995 a	CL 299263: Metabolism of carbon-14 labelled CL 299263 in field grown canola American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-320-001	Melcer, M	1993 a	AC 299, 263: Determination of the dissociation constants American Cyanamid Co., Princeton NJ, United States of America Unpublished
2013/1249356	Mewis, A	2013 a	Independent laboratory validation (ILV) of an analytical method L0188/01 for the determination of BAS 720 H and 3 metabolites in plant matrices Eurofins Agroscience Services GmbH, Niefern-Oeschelbronn, Germany Fed.Rep. Unpublished
2012/1299407	Meyer, M	2013 a	Study on the residue behaviour of Imazamox in broad beans after treatment with BAS 720 06 H under field conditions in Germany, the United Kingdom, The Netherlands, Italy and Spain, 2011 SGS Institut Fresenius GmbH, Taunusstein, Germany Fed. Rep. Unpublished
ID-244-007	Minoura, M & Ohba, K	1996 a	CL 299263 (Imazamox): Validation of HPLC method M 2248.01 for the determination of CL 299263 residues in soya bean, seed Cyanamid Japan Ltd., Tahara Aichi 441-34, Japan Unpublished
ID-244-008	Minoura, M & Ohba, K	1996 b	CL 299263 (Imazamox): Validation of HPLC method M 2248.01 for the determination of CL 299263 residues in soya bean, foliage Cyanamid Japan Ltd., Tahara Aichi 441-34, Japan Unpublished
ID-244-009	Minoura, M & Ohba, K	1996 c	CL 299263 (Imazamox): Validation of HPLC method M 2248.01 for the determination of CL 299263 residues in peanut, nut in shell Cyanamid Japan Ltd., Tahara Aichi 441-34, Japan Unpublished
ID-244-010	Minoura, M & Ohba, K	1996 d	CL 299263 (Imazamox): Validation of HPLC method M 2248.01 for the determination of CL 299263 residues in peanut, foliage Cyanamid Japan Ltd., Tahara Aichi 441-34, Japan Unpublished
ID-720-034	Minoura, M & Ohba, K	1996 a	CL 299263 100A: Residues of CL 299263 in peanut, nut in shell / Kingaroy, Queensland, Australia Cyanamid Japan Ltd., Tahara Aichi 441-34, Japan Unpublished
ID-720-035	Minoura, M &	1996 b	CL 299263 700SG: Residues of CL 299263 in peanut, nut in shell /

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	Ohba, K		Kingaroy, Queensland, Australia Cyanamid Japan Ltd., Tahara Aichi 441-34, Japan Unpublished
ID-720-036	Minoura, M & Ohba, K	1996 c	CL 299263 120A: Residues of CL 299263 in peanut, nut in shell / Norwin, Queensland, Australia Cyanamid Japan Ltd., Tahara Aichi 441-34, Japan Unpublished
ID-306-001	Morelli, D	1994 a	AC 299, 263—Determination of the vapor pressure American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-315-001	Morelli, D	1994 b	AC 299, 263—Determination of the N-octanol/water partition coefficient American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-435-001	Mulligan, E	1995 a	Evaluation of CL 299, 263 in a bacterial / microsome mutagenicity assay American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-470-003	Mulligan, E	1995 c	Microbial mutagenicity plate incorporation assay of CL 263,284 American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-470-004	Mulligan, E	1995 d	Microbial mutagenicity plate incorporation assay of CL 189,215 American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-470-005	Mulligan, E	1995 b	Microbial mutagenicity plate incorporation assay of CL 312,622 American Cyanamid Co., Princeton NJ, United States of America Unpublished
2004/5000274	Nejad, H	2004 a	Validation of BASF analytical method M 3519 (2002) entitled BAS 720 H and BAS 685 H: LC/MS determinative and LC/MS/MS confirmatory method for BAS 720 H, CL 263284, CL 189215, CL 312622, BAS 685 H, CL 288511 CL 182704 in lentils seed, forage BASF Corp. Agro Research, Princeton NJ, United States of America Unpublished
IA-730-011	Nejad, H	1999 a	CL 263,222 (Imazapic): Freezer stability of residues of CL 263,222, CL 263,284 and CL 189,215 in wheat green forage, wheat hay, wheat straw, and wheat grain Centre Analytical Laboratories Inc., State College PA, United States of America Unpublished
IA-740-023	Nejad, H & Xu, B	2000 a	CL 263,222 (Imazapic): Freezer stability of residues of CL 263,222, CL 263,284 and CL 189,215 in peanut hull and nutmeat Centre Analytical Laboratories Inc., State College PA, United States of America Unpublished
2008/7019226	Norris, F	2009 a	The magnitude of Imazamox and Imazapyr residues in Clearfield lentils following application of BAS 723 00 H BASF Agricultural Research Center, Research Triangle Park NC, United States of America Unpublished
2008/7019225	Norris, FA	2009 a	The magnitude of Imazamox and Imazapyr residues in Clearfield sunflower and Clearfield sunflower processed fractions following application of BAS 723 00 H BASF Agricultural Research Center, Research Triangle Park NC, United States of America Unpublished
2008/7019225	Norris, FA	2009 a	The magnitude of Imazamox and Imazapyr residues in Clearfield sunflower and Clearfield sunflower processed fractions following application of BAS 723 00 H BASF Agricultural Research Center, Research Triangle Park NC, United States of America Unpublished
2007/1007939	North, L	2007 a	Study on the residue behaviour of Imazamox, Metazachlor and Quinmerac in oilseed rape following foliar applications under field conditions in Northern and Southern Europe during 2005–2006 Agrisearch UK Ltd., Melbourne Derbyshire DE73 8AG, United Kingdom Unpublished
2007/1007963	North, L	2007 b	Study on the residue behaviour of Imazamox, Metazachlor and Quinmerac in oilseed rape following foliar applications under field conditions in Northern and Southern Europe during 2005–2006 Agrisearch UK Ltd., Melbourne Derbyshire DE73 8AG, United Kingdom Unpublished
ID-301-001	Patel, JA	1993 a	Technical CL 299263—Color, physical state, odor, bulk density, relative density, pH, oxidising / reducing properties American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-301-001	Patel, JA	1993 a	Technical CL 299263—Color, physical state, odor, bulk density, relative density, pH, oxidising / reducing properties American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-301-001	Patel, JA	1993 a	Technical CL 299263—Color, physical state, odor, bulk density, relative density, pH, oxidising / reducing properties American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-301-001	Patel, JA	1993 a	Technical CL 299263—Color, physical state, odor, bulk density, relative density, pH, oxidising / reducing properties American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-334-001	Patel, JA	1994 a	Technical CL 299, 263—Explodability American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-334-001	Patel, JA	1994 a	Technical CL 299, 263—Explodability American Cyanamid Co., Princeton

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ID-334-001	Patel, JA	1994 a	NJ, United States of America Unpublished Technical CL 299, 263—Explodability American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-334-001	Patel, JA	1994 a	Technical CL 299, 263—Explodability American Cyanamid Co., Princeton NJ, United States of America Unpublished
2004/1000754	Perny, A	2004 a	Study on the residue behaviour of Imazamox in Imazamox resistant sunflowers after application of BAS 720 02 H under field conditions in France, in 2003 Anadiag SA, Haguenau, France Unpublished
2011/1281377	Radzom, M	2013 a	Metabolism of ¹⁴ C-Imazamox in rapeseed BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished
2003/1030079	Rawle, NW	2003 a	Freezer stability of AC 299263 and CL 263284 in maize grain, ear and immature whole plant samples CEMAS—CEM Analytical Services Ltd., North Ascot Berkshire SL5 8JB, United Kingdom Unpublished
ID-244-015	Robbins, AJ	1997 a	Validation of CEM Analytical Services Standard Operating Procedure CEM-236 for the determination of residues of AC 299263 and CL 263284 in French bean whole plant samples CEMAS—CEM Analytical Services Ltd., North Ascot Berkshire SL5 8JB, United Kingdom Unpublished
ID-244-016	Robbins, AJ	1997 b	Validation of CEM analytical services standard operating procedure CEM-236 for the determination of residues of AC 299263 and CL 263284 in maize grain samples CEMAS—CEM Analytical Services Ltd., North Ascot Berkshire SL5 8JB, United Kingdom Unpublished
ID-244-017	Robbins, AJ	1997 c	Validation of CEM analytical services standard operating procedure CEM-236 for the analysis of residues of AC 299263 and CL 263284 in maize ears and immature whole plant CEMAS—CEM Analytical Services Ltd., North Ascot Berkshire SL5 8JB, United Kingdom Unpublished
ID-244-018	Robbins, AJ	1996 a	Standard Operating Procedure—The determination of residues of AC 299263 and AC 263284 in crops using HPLC CEMAS—CEM Analytical Services Ltd., North Ascot Berkshire SL5 8JB, United Kingdom Unpublished
ID-730-038	Rodriguez, D	1999 a	CL 299263 (Imazamox): CL 299263 and CL 263, 284 residues in imidazolinone-tolerant spring wheat racs and processed commodities after post-emergence treatment with AC 299263 (1AS) herbicide in North Dakota American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-730-038	Rodriguez, D	1999 a	CL 299263 (Imazamox): CL 299263 and CL 263, 284 residues in imidazolinone-tolerant spring wheat racs and processed commodities after post-emergence treatment with AC 299263 (1AS) herbicide in North Dakota American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-640-009	Roman, Y	1999 b	AC 299263: Metabolism of carbon-14 labelled AC 299263 in field grown oil seed rape/canola American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-244-005	Safarpour, H	1995 a	CL 299263: Validation of GC method M 2460 for the determination of CL 299263 and 263284 residues in soya bean, field pea and canola by Huntingdon Analytical Services American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-750-003	Safarpour, H	1995 a	Imazamox (CL 299263): Residues of total CL 299263 and CL 263284 in canola seed American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-720-071	Salzman, FP	1999 a	Imazamox: Magnitude of the residue on bean (dry) Rutgers State University of New Jersey, North Brunswick NJ, United States of America Unpublished
ID-720-072	Salzman, FP	1999 b	Imazamox: Magnitude of the residue on bean (Lima) Rutgers State University of New Jersey, North Brunswick NJ, United States of America Unpublished
ID-720-073	Salzman, FP	1999 b	Imazamox: Magnitude of the residue on bean (Snap) Rutgers State University of New Jersey, North Brunswick NJ, United States of America Unpublished
ID-720-075	Salzman, FP	1999 a	Imazamox: Magnitude of the residue on pea (succulent) Rutgers State University of New Jersey, North Brunswick NJ, United States of America Unpublished
ID-430-001	Schroeder, RE	1995 a	A two-generation reproduction study with AC 299, 263 in rats (Volume I–IV) Pharmaco LSR Inc., East Millstone NJ, United States of America

Code	Author(s)	Year	Title, Institute, Report reference
2013/1235041	Schulz, M & Mellert, W	2013 a	Unpublished Reg.No. 4110773 (Metabolite of BAS 720 H, Imazamox)—Micronucleus test in bone marrow cells of the mouse BASF SE, Ludwigshafen/Rhein, Germany Fed.Rep. Unpublished
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ID-435-002	Sharma, RK	1993 b	Evaluation of CL 299, 263 in the mammalian cell CHO/HGPRT mutagenicity assay American Cyanamid Co., Princeton NJ, United States of America Unpublished
2002/1020471	Stewart, J	2002 a	Analysis of Imazamox and Imazapyr residues in sunflower samples treated with BAS 723 00 H from efficacy trials in Turkey, 2002—Analytical report of the analysis of BAS 720 H, CL 263284 and BAS 693 H BASF Corp., Research Triangle Park NC, United States of America Unpublished
2003/5000116	Stewart, J	2003 a	Method validation of BASF Analytical Method D0303 entitled Method for the Determination of BAS 720 H (CL 299263) and its metabolite CL 263284 in bovine matrices using LC/MS/MS BASF Agro Research RTP, Research Triangle Park NC, United States of America Unpublished
ID-244-020	Sweeney, RA, & Gross, JR	1998 a	CL 299263: Independent laboratory validation of CE method M 3076 for the determination of residues of CL 299263 in canola seed American Cyanamid Co., Princeton NJ, United States of America Unpublished
2011/7002438	Ta, C	2012 a	Aerobic soil metabolism of ¹⁴ C-Imidazolinone BAS 720 H BASF Crop Protection, Research Triangle Park NC, United States of America Unpublished
ID-620-004	Ta, CT	1995 c	AC 299263: Photodegradation on soil American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-620-008	Ta, CT	1995 a	AC 299263: Aerobic soil metabolism American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-620-018	Ta, CT	1997 a	AC 299263: Aerobic soil metabolism American Cyanamid Co., Ewing NJ, United States of America Unpublished
ID-620-027	Ta, CT & Lewis, CJ	1997 a	AC 299263: Soil degradation study Covance Laboratories, Harrogate North Yorkshire HG3 1PY, United Kingdom Unpublished
2011/1112577	Tandy, R	2012 a	Study on the residue behaviour of Imazamox (BAS 720 H) and Bentazone (BAS 351 H) in dry beans after treatment with BAS 762 01 H in southern Europe during 2011 Eurofins Agroscience Services, Melbourne Derbyshire DE73 8AG, United Kingdom Unpublished
2012/1221518	Tandy, R	2012 a	Study on the residue behaviour of Imazamox (BAS 720 H) and Bentazone (BAS 351 H) in fresh beans after treatment with BAS 762 01 H in Northern and Southern Europe during 2011 Eurofins Agroscience Services, Melbourne Derbyshire DE73 8AG, United Kingdom Unpublished
2012/1266578	Tandy, R	2012 b	Amendment No. 2—Study on the residue behaviour of Imazamox (BAS 720 H) and Bentazone (BAS 351 H) in fresh beans after treatment with BAS 762 01 H in Northern and Southern Europe during 2011 Eurofins Agroscience Services, Melbourne Derbyshire DE73 8AG, United Kingdom Unpublished
2011/1080475	Thiaener, J & Lutz, T	2012 a	Metabolism of ¹⁴ C-Imazamox (BAS 720 H) in rats BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished
2011/1080475	Thiaener, J & Lutz, T	2012 b	Metabolism of ¹⁴ C-Imazamox (BAS 720 H) in rats BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished
2013/1224024	Toledo, F	2013 a	Method development and validation of an analytical method for the determination of BAS 720 H and its 2 metabolites Reg.No 4110603 and Reg.No 4110542 in water (analytical method L0209) SGS Institut Fresenius GmbH, Taunusstein, Germany Fed. Rep. Unpublished
ID-750-017	Trewhitt, JA	2000 a	AC 299263 40 g ai/L SL (SF 09464): At harvest residue study on AC 299263 and CL 263284 in Imi oil seed rape (Germany, 1997–1998)BASF plc, Gosport Hampshire PO13 0AS, United Kingdom Unpublished
ID-440-006	Tsalta, C	1999 a	CL 312622: Metabolism of [¹⁴ C]-CL 312622 in the lactating goat American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-244-022	Wickremesinha, E & Safarpour, H	1998 a	CL 299263 (Imazamox): Independent laboratory validation of HPLC/MSD method M 3178 for the determination of residues of CL 299263, CL 263284, CL 189215 and CL 312622 in alfalfa seed, forage (whole green plant) and hay (whole dried plant) Centre Analytical Laboratories Inc., State College PA, United States of America Unpublished

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ID-244-022	Wickremesinhe, E & Safarpour, H	1998 a	CL 299263 (Imazamox): Independent laboratory validation of HPLC/MSD method M 3178 for the determination of residues of CL 299263, CL 263284, CL 189215 and CL 312622 in alfalfa seed, forage (whole green plant) and hay (whole dried plant) Centre Analytical Laboratories Inc., State College PA, United States of America Unpublished
1994/7003428	Witkonton, S	1994 b	CL 299263: HPLC method for the determination of CL 299263 residues in soya bean seeds American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-244-002	Witkonton, S	1993 a	CL 299263: Validation of method M 2248 for the determination of CL 299263 residues in soya bean seeds American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-244-004	Witkonton, S	1994 a	Independent laboratory validation of method M 2248.01 for the determination of CL 299263 residues in soya bean seed by ABC Laboratories, Incorporated American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-244-003	Witkonton, S <i>et al.</i>	1994 a	CL 299263: Validation of the LC/MS confirmatory method M 2333 for the confirmation of CL 299262 residues in soya bean seed American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-640-008	Wu, SS	1998 b	CL 299263: Metabolism of [Pyridine-6- ¹⁴ C] CL 299263 in alfalfa under field conditions American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-244-026	Xu, B & Nejad, H	1999 a	CL 299263 (Imazamox): Independent laboratory validation of CE method M 3098 for the determination of residues of CL 299263 and CL 263284 in wheat hay, forage, straw and grain Centre Analytical Laboratories Inc., State College PA, United States of America Unpublished
ID-640-006	Zulalian, J	1997 c	CL 299, 263: Metabolism of carbon-14 labelled CL 299, 263 in imidazolinone-resistant corn applied post-emergence under field conditions (Amendment number 1 included) American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-640-007	Zulalian, J	1997 d	CL 299,263: Metabolism of carbon-14 labelled CL 299,263 in imidazolinone-resistant corn applied pre-emergence under field conditions (Amendment number 1 included) American Cyanamid Co., Princeton NJ, United States of America Unpublished

MESOTRIONE (277)

The first draft was prepared by Professor Eloisa Dutra Caldas

University of Brasilia, Brasília/DF, Brazil

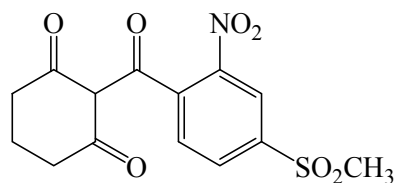
EXPLANATION

Mesotrione is a systemic pre-emergence and post-emergence herbicide for the selective contact and residual control of broadleaf weeds. It is rapidly absorbed by green plant tissue or taken up through the soil during emergence. It acts by inhibiting the enzyme 4-hydroxyphenolpyruvate dioxygenase (HPPD), leading to a reduction of carotenoids causing bleaching symptoms in the plant. Mesotrione was scheduled for the evaluation as a new compound by the 2014 JMPR at the 45th Session of the CCPR (2013). Metabolism studies on animal and plants, environmental fate studies, analytical methods and residue trials on berries, okra, sweet corn, soya bean and tolerant soya bean, asparagus, rhubarb maize, millet, oat, rice, sorghum, sugarcane and linseed were submitted for evaluation.

IDENTITY

ISO Common Name Mesotrione
 Chemical name IUPAC: 2-(4-mesyl-2-nitrobenzoyl)cyclohexane-1,3-dione
 CAS (104206-8): 2-[4-(methylsulfonyl)-2-nitrobenzoyl]-1,3-cyclohexanedione

Structural formula



Molecular formula: C₁₄H₁₃NO₇S
 Molecular mass: 339.3

PHYSICAL AND CHEMICAL PROPERTIES

Mesotrione pure material (> 99%) was used for colour, physical state, odour, melting point, pH and spectroscopic characterisation, and also for density, aqueous solubility, dissociation constant, octanol/water partition coefficient and vapour pressure determinations. Colour, odour, solubility in organic solvents, density, pH and surface tension determinations for mesotrione technical material were conducted with material containing 96.7% mesotrione.

Properties of pure mesotrione

Property	Results	Reference; Report
Melting point	165.3 °C with decomposition	Goodman, 1996; RR95-077B
Density at 20 °C	1.49 g/cm ³	
Vapour pressure at 20 °C	< 5.7 × 10 ⁻⁶ Pa	
Volatility at 20 °C	Henry's law constant: < 5.1 × 10 ⁻⁷ Pa/m ³ /mol.	
Physical state, colour, odour	Odourless pale yellow solid (room temperature)	
Solubility in water at 20 °C	0.16 g/L (unbuffered water) 2.2 g/L at pH 4.8 (buffered water) 15 g/L at pH 6.9 (buffered water) 22 g/L at pH 9 (buffered water)	
Partition coefficient n-octanol/ water at 20 °C	log P _{OW} : 0.11 in unbuffered water 0.90 at pH 5 < -1.0 at pH 7 and 9	
pH at 20 °C	3.38 at 25.4 °C in 1% aqueous solution	

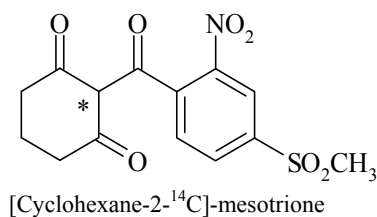
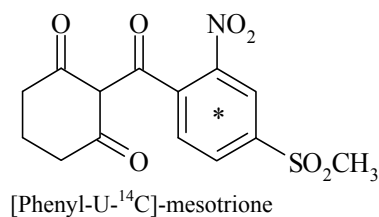
Property	Results	Reference; Report
Hydrolysis in water at 25 °C	Less than 10% degradation of mesotrione (1 µg/ml) occurred during the test period of 30 days in the pH of 4–9 at both 25 and 50 °C.	Miles & Powell, 1995; RJ1776B
Photochemical stability in water	The photolysis half-life and rate constant in sterile aqueous buffer solutions at pH 7 at 25 °C were 83.7 days and 8.40×10^{-3} /day, respectively at 37°56' latitude local sunlight, or 92 days at 50°N. No degradates exceeding 10% of the applied radioactivity were observed during the photolysis of [^{14} C-phenyl] mesotrione. The major degradation product was carbon dioxide (^{14} CO ₂), the others were present at levels < 5% of the applied radioactivity. Recovery ranged from 92.2 to 101.8%. Recovery of 92.2% was only observed prior to trapping volatiles formed during photolysis of [^{14} C-cyclohexane] mesotrione. The average recovery of applied radioactivity after trapping volatiles was 95.4%.	Eya, 1995; RR94-071B
Dissociation constant at 20 °C	pK _a =3.12	Goodman, 1996; RR95-077B

Technical grade material (Goodman, 1996; RR96-004B)

Property	Results
Physical state, colour, odour	Light tan or sand coloured opaque solid with slight odour. sweet (not pungent)
Solubility in organic solvents at 20 °C	Acetone 76.4 g/L Acetonitrile 96.1 g/L 1,2-Dichloroethane 82.7 g/L Ethyl acetate 16.6 g/L Heptane < 0.3 g/L Methanol 3.6 g/L Toluene 2.7 g/L Xylenes 1.4 g/L
Density	The density at 20 °C: 1.46 g/ml; bulk density at 23.3 °C: 0.56 g/mL
pH	The pH of a 1% solution: 3.42 at 24.8 °C
Surface tension at 20 °C	Aqueous solution, 90% saturated: 72.5 mN/m (without indication of surface activity)

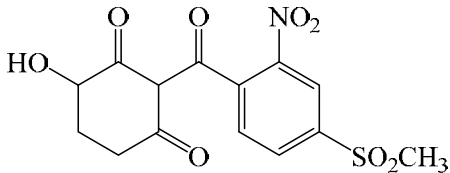
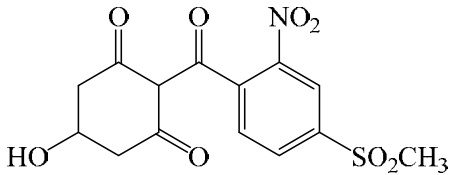
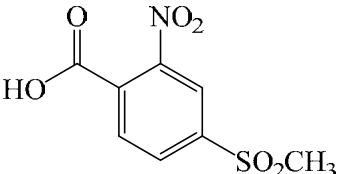
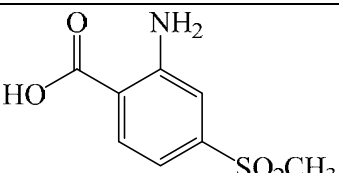
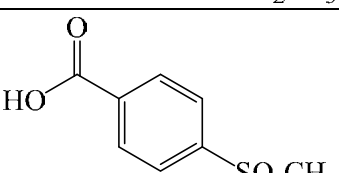
METABOLISM AND ENVIRONMENTAL FATE

The fate and behaviour of mesotrione in animals, plants, and soils were investigated using the following [^{14} C] labelled test materials:



Concentrations of radioactivity are expressed as mg mesotrione equivalents/kg throughout. The chemical structures of the major degradation compounds arising from the metabolism of mesotrione are presented in the following table.

Degradation compounds from metabolism of mesotrione in plants and animals

Compound Name	Structure	Found in:
4-Hydroxy-mesotrione 4-hydroxy-2-[4-(methylsulphonyl)-2-nitrobenzoyl]-1,3-cyclohexanedione		Livestock, plant
5-Hydroxy-mesotrione 5-hydroxy-2-[4-(methylsulphonyl)-2-nitrobenzoyl]-1,3-cyclohexanedione		Livestock, plant
MNBA 4-(methylsulphonyl)-2-nitrobenzoic acid		Livestock, plant
AMBA (2-amino-4-(methylsulphonyl)-benzoic acid)		Livestock, plant
MBA 4-(methylsulphonyl) benzoic acid		Livestock, plant

Animal metabolism

Cow

Two metabolism studies were conducted in lactating cows, one with [phenyl-U-¹⁴C]-mesotrione (Powell & Marples, 1996; RJ 1890B) and one with [cyclohexane-2-¹⁴C]-mesotrione (Hand, 1995; RJ 1830B). The studies used similar dosing regimens, sampling protocol and analysis. In each study, one lactating cow was dosed orally with [¹⁴C]mesotrione for 7 consecutive days at a nominal rate of 10 ppm in the diet (450–650 kg cow, 20 kg feed intake per day). Milk and excreta were collected daily; the cows sacrificed 16 hours after the final dose and tissues taken for analysis. Milk production was around 20 L per day.

Tissue samples were homogenised in sequence in the presence of solvents such as hexane, acetonitrile/hexane, dichloromethane, acetonitrile, acetonitrile/water (1:9) and 2% sodium dodecyl sulphate (SDS). Each of the extracts was radioassayed. Extracts were partitioned in diethyl ether and water, ethyl acetate and water, hexane and water or hexane and acetonitrile. Solubilised protein was denatured with acetone. Hydrolyses of proteins and extracts were conducted to characterise the bound and/or conjugated residues, using a variety of enzymes, including papain (to hydrolyse ester and peptide bonds) and β-glucuronidase (to hydrolyse glucuronides). Subsamples of the milk extract were submitted to chemical hydrolysis using either 1 M HCl or 1 M NaOH. Protein hydrolysate samples

were derivatized with isobutyl chloroformate to facilitate the chromatography. Residues in tissues were determined by combustion analysis. Normal or reserve phase TLC and HPLC radiochromatograms were used to characterise the components.

Table 1 shows the total radioactive residues found in tissues, milk and excreta (Hand, 1995, Powell & Marples, 1996). Over 90% of the administered dose was found in excreta, mostly in faeces. Liver and kidney contained the highest residues (about 0.1 mg/kg eq.). Residues in muscle were below 0.01 mg/kg eq and reached 0.08 mg/kg eq in milk, with a plateau at day 5 in the cyclohexane experiment (Table 2).

Table 1 Radioactive residues in lactating cow tissues, milk and excreta after oral administration of [¹⁴C]mesotrione

Matrix	[phenyl-U- ¹⁴ C]-mesotrione		[Cyclohexane-2- ¹⁴ C]-mesotrione	
	mg/kg eq.	% Total administered	mg/kg eq.	% Total administered
Liver	0.077	—	0.110	—
Kidney	0.067	—	0.110	—
Muscle (fore)	0.002	—	0.007	—
Muscle (hind)	0.002	—	0.007	—
Peritoneal Fat	≤ 0.004	—	0.008	—
Perirenal Fat	0.007	—	0.005	—
Subcutaneous Fat	≤ 0.004	—	0.013	—
Milk	0.01 to 0.04 (day 5)	0.29	0.06 to 0.08 (day 5–8)	—
Urine		9.65d		13.1
Faeces		80.9		79.4
Total in excreta		90.6		92.5

Table 2 Mean daily radioactive residues in milk (mg/kg mesotrione equivalents)

Study	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7	Day 8 ^a
[phenyl-U- ¹⁴ C]	0.006	0.052	0.065	0.067	0.074	0.078	0.079	0.072
[Cyclohexane-2- ¹⁴ C]-	0.01	0.03	0.03	0.03	0.04	0.03	0.03	0.03

^a AM collection only

Table 3 (Powell & Marples, 1996) and Table 4 (Hand, 1995) show the results of the characterization of radioactive residues in liver and kidney. Mesotrione accounted for up to 12.3%TRR in liver and 18%TRR in kidney (< 0.02 mg/kg eq.), while AMBA was only identified in kidney.

Table 3 Summary of the identification and characterisation of radioactive residues of mesotrione in liver and kidney of cows treated with [phenyl-U-¹⁴C]-mesotrione

Commodity		Liver		Kidney	
TRR mg/kg		0.085		0.065	
Origin of component	Component	%TRR	mg/kg	%TRR	mg/kg
Solvent extracts ^a	Mesotrione	10.3	0.009	18.0	0.012
	AMBA	ND	ND	15.0	0.010
	Unknowns	24.1 ^b	0.020	35.8 ^c	0.023
	Remainder ^d	6.9	0.006	5.7	0.004
Protein associated fractions	Acetone precipitate	8.7	0.007	1.7	0.001
	Acetone precipitate/Papain hydrolysis debris	9.9	0.008	2.9	0.002
Uncharacterised organosoluble fractions	Acetone ^e	3.0	0.002	4.5	0.003
	Methanol ^f	8.3	0.007	4.0	0.003
	Acidified methanol ^f	13.2	0.011	—	—

Commodity		Liver		Kidney	
Uncharacterised aqueous soluble fractions	H ₂ O extract	3.7	0.003	2.5	0.002
Unextracted		1.7	0.001	4.1	0.003
Losses/gains		10.2	0.009	5.8	0.004
Total		100	0.083 ^g	100	0.066 ^g

^a Values are based on the mean values obtained from three different TLC systems

^b Containing at least eight individual components

^c Components, none of which were > 7.9% TRR, 0.007 mg/kg

^d Containing at least seven individual components, none of which were > 6.4% TRR, 0.004 mg/kg

^e Area corresponding to quantifiable radioactivity containing no discrete bands

^f Originates from the de-proteination of a subsample of the SDS tissue fraction

^g Originates from the C₁₈ fractionation of the solubilised material from the papain hydrolysis

Table 4 Summary of the identification and characterisation of radioactive residues of mesotrione in liver and kidney of cows treated with [cyclohexane-2-¹⁴C]-mesotrione

Matrix	Liver		Kidney	
TRR ^a	0.101 mg/kg		0.107 mg/kg	
Component/Fraction	%TRR ^a	mg/kg	%TRR ^a	mg/kg
Mesotrione	12.5	0.013	14.4	0.015
Neutral organosoluble unknowns ^b	4.2	0.004	6.0	0.006
Aqueous unknowns ^c	1.8	0.002	—	—
Acidic organosoluble unknowns ^d	—	—	2.5	0.003
Acidic unknowns from debris ^e	—	—	0.7	0.01
Remainder ^f	21.3	0.022	13.6	0.015
Uncharacterised soluble fractions, organosoluble ^g	3.4	0.003	8.2	0.009
Aqueous soluble ^h	12.1	0.012	11.9	0.013
Aqueous soluble (eluted from anion ex. resin) ⁱ	—	—	19.0	0.020
Acid soluble ^j	10.0	0.010	—	—
Base soluble	7.5	0.008	—	—
Proteinaceous solids ^k	29.3	0.030	7.1	0.008
Unextracted	5.4	0.005	—	—
Ion exchange resin-bound	—	—	10.1	0.011
Losses During Fractionation	4.9	0.005	6.5	0.005
Total	100.0	0.101	100	0.106 ^l

^a TRRs was calculated from the summation of fractions obtained from various complex extraction schemes required to determine the nature of this type of residue

^b Consists of three components, the largest represents 2% (0.002 mg/kg) liver and 2.6% (0.003 mg/kg) kidney

^c Consists of two components, largest represents 0.9% (< 0.001 mg/kg)

^d Consists of two components, largest represents 1.4% (0.001 mg/kg)

^e Consists of five components resulting from 6 N HCl and subsequent derivatization (largest 0.2%, < 0.001 mg/kg)

^f Derives from chromatography of multiple extracts. Includes baseline material and non-discrete areas of ¹⁴C

^g Acetonitrile wash of debris (liver), SDS extraction of debris (kidney), which consists of two extracts (max 6.2% TRR, 0.007 mg/kg)

^h Consists of two extracts, largest represents 7.4% (0.007 mg/kg) in liver and 7.1% (0.008 mg/kg) in kidney

ⁱ Consists of three extracts, largest representing 11.0% TRR (0.012 mg/kg) of the which could not be chromatographed due to the high concentration of acid present. These extracts were considered to chemical distinct in terms of their ionic nature

^j Consists of two extracts, largest represents 6.3% TRR (0.006 mg/kg) liver

^k Consists of two precipitates, largest represents 22.7% (0.023 mg/kg) liver |TRR. This was hydrolysed with 6 N HCl and derivatized with isobutyl chloroformate. The resulting acidic organosoluble fraction consisted of five components, the largest representing 4.0% (0.004 mg/kg)

^l The slight discrepancy is due to rounding of the calculated values.

Tables 5 shows the results of the characterization study of residues in milk collected from the phenyl labelled experiment (Powell & Marples, 1996). Most of the radioactivity was found in the skimmed milk (97.3% TRR), which was deproteinated using acetone. The resultant aqueous acetone fraction (78.2% TRR) showed to contain at least three polar components, not hydrolysed with enzymes but degraded to up to seven components under harsh acid and basic conditions.

Table 5 Summary of the identification and characterisation of radioactive residues of mesotrione in milk [phenyl-U-¹⁴C]-mesotrione

Total ^a			0.036 mg/kg	
Component/Fraction			%TRR	mg/kg
Whole milk	Centrifugation	Butterfat	0.6	0.000
		Skim milk ^b	1.8	0.001
Skimmed milk (following centrifugation)	Partition	Dichloromethane soluble ^c	3.6	0.001
	SDS precipitation	Protein	4.6	0.002
		Aqueous acetone fraction ^d	3.9	0.001
	Chromatography of aqueous fraction (following SDS precipitation)	Unknown 1	50.0	0.018
		Unknown 2	2.2	< 0.001
		Unknown 3	21.9	0.008
		Remainder ^e	4.1	0.001
Losses During Fractionation			7.3	0.005
Total			100.0	0.036

Based on the extraction of the Day 6 (initial) milk sample. Further characterisation of additional samples was carried out following derivatization or acid/base hydrolysis but as this did not lead to the identification of discrete components the data is not reported in the table above

^a Total concentration taken from sum of extractions and combustion analyses

^b Consists of one sample, skim milk removed from the butterfat samples

^c Consists of one sample from the partition of skimmed milk with dichloromethane

^d Consists of two samples obtained from the aqueous wash of the protein fraction

^e Area corresponding to quantifiable radioactivity containing no discrete bands

TRR in milk from the cyclohexanedione experiment (Hand, 1995) was 0.07 mg/kg eq., 90% of which was associated with the skimmed milk fraction. Lactose accounted for 35.1% TRR in whole milk (Table 6), with the remainder of the extracted radioactivity comprised several unknown components, the largest of which was aqueous soluble and represented 14.7% TRR (0.010 mg/kg). Low levels of radioactivity were found to be associated with the proteinaceous solids and the butterfat.

The radioactive residues in subcutaneous fat were 0.012 mg/kg, with 37% extracted with acetonitrile, leaving 63.3% associated with the debris, from which 46.7% was not extracted (Hand, 1995).

Table 6 Summary of the identification and characterisation of radioactive residues of mesotrione in milk [cyclohexane-2-¹⁴C]-mesotrione

Total ^a	0.070 mg/kg	
Component/Fraction	%TRR ^a	mg/kg
Lactose	35.1	0.025
Aqueous Fractions ^b	24.4	0.017
Organosoluble Fractions ^c	10.5	0.007
Remainder ^d	10.5	0.007
Proteinaceous material	7.6	0.005
Butterfat	10.2	0.007
Losses During Fractionation	1.7	0.001
Total	100.0	0.069 ^e

^a Total concentration taken from sum of skimmed milk and butterfat

^b Consists of five unknowns, largest of which represents 14.7% TRR (0.01 mg/kg)

^c Consists of nine unknowns, largest of which represents 5.3% TRR (0.004 mg/kg)

^d Remainder derives from the chromatography of four extracts. No discrete peaks

^e Slight difference due to rounding.

Swine

One female Hampshire swine (30 kg) was dosed orally with [phenyl-U-¹⁴C]-mesotrione for 5 consecutive days at 6 ppm (Brown, 2006; T020371-04). Excreta were collected daily, the animal sacrificed 23 hours after the final dose, and tissues taken for analysis. Tissue samples were extracted twice with acetonitrile/water (80:20), and further extraction with water was performed on the solids

obtained from the kidney and liver sub-samples. Liquid-liquid partitions were carried out with ethyl acetate after adjustment to pH 2 with acetic acid. C₁₈ SPE columns were used to purify the isolated metabolites. Protein was precipitated with either ammonium sulphate or acetone. To extract ionic material and membrane-bound proteins from cell walls, liver PES was extracted with 2% SDS solution, and the solids obtained were submitted to enzymatic hydrolysis. Analytes were separated using 1 and 2-D TLC on silica gel plates. The metabolites were characterised and isolated using HPLC coupled to a UV detector, radioisotope flow monitor and a fraction collector. Additional confirmation and identification was undertaken using tandem mass spectrometry and ¹H-NMR.

The radioactivity detected in the excreta accounted for 89.4% of the administered dose. In tissues, highest residues were found in liver (1.75 mg/kg eq) and kidney (0.12 mg/kg eq); residues in muscle reached 0.01 mg/kg eq. (Table 7). Residues in the tissues were readily extracted with solvents, and showed to be mainly mesotrione (up to 90% TRR in liver; Table 8). Residues AMBA accounted for up to 2% TRR in kidney. MNBA was only found in liver (0.3% TRR). The major identified residue in urine collected on day 5 was AMBA which accounted for approximately 36.5% TRR.

Table 7 Radioactive residues in swine tissues and excreta after oral administration of [phenyl-U-¹⁴C]-mesotrione

Matrix	Radioactive residue phenyl-U- ¹⁴ C mesotrione equivalents		
	% of the applied dose	mg/kg eq. ^a	mg/kg eq. ^b
Liver	4.2	1.748	1.630
Kidney	0.05	0.117	0.118
Muscle	0.26	0.0096	0.011
Fat	0.06	0.006	n/d
Urine	36.5	–	–
Faeces	51.1	–	–
Total in excreta	89.4 (includes cage washings)	–	–
Total recovered	94.2	–	–

^a Based on the initial combustion values

^b Based on dpm from extracted sample. These values were used for calculation in the study. Fat and the remaining tissues were not analysed further

Table 8 Summary of the identification and characterisation of radioactive residues of mesotrione in tissues of swine dosed with [phenyl-U-¹⁴C]-mesotrione

Matrix	Liver		Kidney		Muscle	
TRR (mg/kg eq.):	1.630		0.118		0.011	
Extracted (%)	98.7 ^b		90.2		90.7	
Component/Fraction	%TRR ^a	mg/kg eq. ^a	%TRR ^a	mg/kg eq. ^a	%TRR ^a	mg/kg eq. ^a
Mesotrione	89.9	1.465	72.8	0.086	77.9	0.009
AMBA	1.8 ^d	0.029 ^d	2.0	0.002	0.7	< 0.001
MNBA	0.3	0.005	–	–	–	–
Unknown 1	1.0	0.016	–	–	–	–
Unknown 2	–	–	0.3	< 0.001	–	–
Unknown 4	0.2	0.003	0.3	< 0.001	–	–
Unknown 5	< 0.2 ^c	0.003	0.5	0.001	–	–
Unknown 6	1.2	0.020	0.2	< 0.001	–	–
Unknown 7	0.9	0.015	0.3	< 0.001	–	–
Unknown 8	0.7	0.011	1.1	0.001	–	–
Unknown 9	0.2	0.004	0.9	0.001	–	–
Baseline	9.3	0.152	11.5	0.014	6.9	0.001
PES	2.8 ^c	0.046 ^c	9.7	0.011	9.3	0.001
Total ^e	108	1.760 ^f	99.6	0.118 ^f	94.8	0.010 ^f

^a These calculations include values obtained from HPLC fraction collecting. Components in the HPLC fractograms, as % of the chromatogram, were calculated from the total radioactivity in specific fractions divided by the total radioactivity injected. The total radioactivity recovered from the HPLC fractions ranged from 97–112%, compared to the amount injected. These recoveries were not taken into account in the calculation and thus contributed to the slight losses or gains in the total accountability compared to the initial residues. Small discrepancies between the % and mg/kg values are due to rounding

^b Includes 2% SDS and protease extraction values

^c < Value not included in summation

^d Portions not identified (0.2% TRR AMBA)

^e Solids remaining after protease and 2% SDS extractions of original PES (16.5% TRR. 0.269 mg/kg)

^f Calculated by % TRR total × total residue mg/kg

Poultry

Two metabolism studies were conducted in poultry, one with [phenyl-U-¹⁴C]-mesotrione (Young & Skidmore, 1995; RJ 177778B) and one with [cyclohexane-2-¹⁴C]-mesotrione (Grout, 1996; RJ 12071B). The studies used the same dosing regimen, sampling protocol and analysis. In each study, hens were dosed by gelatine capsule with [¹⁴C]- mesotrione for 10 consecutive days at 11 ppm, assuming a daily dietary intake of 150 g and average body weight of 2 kg. Each experiment used 10 hens (*Gallus gallus domesticus*). Excreta and eggs were collected daily.

Eggs were separated into yolks and whites and stored frozen for analysis. The hens were sacrificed approximately 16 hours after the final dose, and tissues stored frozen until analysis. Once thawed, liver, kidney and peritoneal fat samples were chopped, muscle samples were minced whilst partly frozen using a food processor. The skin and subcutaneous fat samples were ground with solid CO₂.

The amount of radioactivity in excreta, edible tissues and eggs was determined in 2–3 hens and samples from the remaining hens were retained for analysis if required. TRR were determined using LSC/combustion or tissue solubilisation/LSC. Tissue samples and egg yolk were homogenised and sequentially extracted with methylene chloride, acetonitrile and acetonitrile/water. Fat samples from the cyclohexane experiment were first extracted with hexane. Between each extraction, the organic phase was separated from the solids (post extraction solids, PES) by centrifugation and analysed for radioactivity. Liquid phases were concentrated and extracted with either ethyl acetate and water or hexane and acetonitrile. SDS was used to extract ionic material and membrane-bound proteins from cell walls; proteins were precipitated out with acetone. Samples of edible tissues and eggs that contained a radioactive residue greater than 0.01 mg/kg were analysed further for residue characterisation. Components were separated using 2-D normal and reverse phase TLC, HPLC-UV or a radioisotope flow monitor and a fraction collector.

The radioactivity in excreta accounted for 90 to 98.7% of the administered dose in both experiments. TRR in edible tissues and egg samples are summarised in Table 9. Highest residues were found in liver and kidney (0.06–1.2 mg/kg eq.) Residues in muscle were below 0.02 mg/kg eq in eggs, residues were higher in the yolk compared to whites, reaching 0.094 mg/kg eq. in yolk in the cyclohexanedione label experiment.

Table 9 Radioactive residues (mg/kg eq.) in eggs and tissues following oral administration of [¹⁴C]mesotrione to hens for 10 consecutive days at 11 ppm

	[Phenyl-U- ¹⁴ C]-mesotrione	[¹⁴ C-cyclohexanedione]-mesotrione
Matrix	Mean TRR (n=3)	Mean TRR (n=2)
Liver	1.121	1.245
Kidney	0.063	0.068
Thigh muscle	< 0.004	0.011
Breast muscle	0.004	0.012
Skin/subcutaneous fat	0.042	0.048
Peritoneal fat	< 0.003	0.010
Egg white	< 0.004 at all days	0.012 (day 1) to 0.025 (day 4); mean=0.019
Egg yolk	< 0.003 (day 1) to 0.024 (day 10); mean=0.015	0.002 (day 1) to 0.094 (day 9/10), mean=0.056

Summaries of the radioactivity extracted sequentially using a range of solvents in each study are present in Tables 10 and 11. In the phenyl label experiment (Table 10), most of radioactivity was extracted from liver and egg yolk with ACN/water, and about 70% TRR extracted from fat with methylene chloride (Young & Skidmore, 1995). Radioactivity in PES ranged from 0.6 to 4.5% TRR in egg yolk.

Table 10 Radioactivity levels extracted from hen tissues and egg samples after oral administration of [phenyl- ^{14}C]-mesotrione using a range of different solvents

Matrix (TRR, mg/kg eq.)	Extraction solvent (% TRR)					PES (%)
	Methylene chloride	ACN:water (9:1)	ACN:water (1:1)	Water	SDS solution	
Liver (1.234)	–	52.8	22.0	10.3	14.3	0.6
Skin/subc. fat (0.037)	69.2	23.1	1.4	2.7	–	3.6
Egg yolk (0.021)	37.2	53.1	5.3	0	–	4.5

PES Post extraction solids

– Not applicable

In the ^{14}C -cyclohexanedione experiment (Table 11), the majority of the radioactive residue in skin and subcutaneous fat and egg white was extracted with acetonitrile and acetonitrile/water, while peritoneal fat residues were mostly extracted with hexane (Grout, 1996). Half of residues present in muscle were extracted with acetonitrile, with the remaining staying in PES. Residues from egg yolk were mostly extracted with methylene chloride.

Table 11 Radioactivity levels extracted from hen tissues and egg samples after oral administration of [^{14}C -cyclohexanedione]-mesotrione using a range of different solvents

Matrix	Extract (% TRR)						PES (%)
	Hexane	Methylene chloride	ACN	ACN/water (1:1)	Water	SDS	
Liver	–	–	19.2	23.5	36.6	18.9	1.8
Skin/subc. fat (n=2)	4.4	–	72.6	11.4	0.0	–	11.6
Peritoneal fat	64.0	–	–	–	–	–	36.0
Thigh muscle	–	–	49.9	–	–	–	50.1
Breast muscle (n=2)	–	–	50	–	–	–	50
Egg white	–	–	32.3	38.5	5.5	–	23.7
Egg yolk	8.1	59.1	12.4	–	7.1	11.0	2.4

DCM: dichloromethane

ACN= acetonitrile

PES: Post extraction solids

– Not applicable

Mesotrione was detected in both experiments, accounting for over 85% TRR (1.1 mg/kg) in liver (Table 12). Mesotrione was not detected in muscle. The lipid fraction present in the combined yolk sample from the [^{14}C -cyclohexanedione] experiment was extracted with acetone which released 51.7% TRR (0.041 mg/kg), and found to contain mesotrione (0.019 mg/kg). The second acetone fraction was saponified, fractionated and showed to contain palmitic/oleic acid (Table 12). Excreta were shown to contain mesotrione (19.7–55%TRR) and AMBA (18% TRR).

Table 12 Mesotrione residues extracted from hen liver, skin, fat and egg yolk after oral administration

	Liver				Subcutaneous Fat and Skin				Egg Yolk			
	[^{14}C -phenyl]		[^{14}C -cyclo]		[^{14}C -phenyl]		[^{14}C -cyclo]		[^{14}C -phenyl]		[^{14}C -cyclo]	
	mg/kg eq.	%TRR	mg/kg eq.	%TRR	mg/kg eq.	%TRR	mg/kg eq.	%TRR	mg/kg eq.	%TRR	mg/kg eq.	%TRR
Mesotrione	1.051	85.2	1.097	90.7	0.032	85.3	0.033	59.0-71.2	0.017	80.9	0.019	19.5
Palmitic/oleic acid											0.015	15.0

Metabolism of AMBA in cow

AMBA is a metabolite found in maize forage and fodder when the plant is treated with mesotrione. A lactating cow was orally administered [phenyl- ^{14}C]-AMBA using gelatine capsule for 7 consecutive days at a rate of 12.2 ppm in the diet, equivalent to 200 mg ai/day (Hand, 1997; RJ

2309B). Milk and excreta were collected daily. The cow was sacrificed approximately 23 hours after the final dose and tissues were taken for analysis.

Samples were sequentially homogenised in the presence of a variety of solvents including hexane, ethyl acetate, acetonitrile/water (1:1) and water. Liquid-liquid partitions were carried out between ethyl acetate and water. Metabolites were separated using normal or reserve phase TLC with visualisation of the radiochromatograms undertaken by phosphor image analysis.

The total recovery was found to be 88.7% of the administered dose, with the majority of the radioactivity recovered in the excreta (Table 13). Highest residues were found in kidney (0.053 mg/kg eq.) and fat (0.018 mg/kg eq.), with no detected residues in muscle or in omental fat. Residues in milk reached a maximum of 0.009 mg/kg eq. (Day 6), decreasing to 0.003 mg/kg eq. on Day 8 after the initial dose.

Table 13 Radioactive residues in lactating cow after oral administration of [^{14}C]AMBA

Matrix	Radioactive residue	
	[^{14}C]AMBA equivalents (mg/kg)	% Total administered
Liver	0.005	
Kidney	0.053	
Fat (perirenal)	0.018	
Fat (omental)	0.000	
Fat (subcutaneous)	0.003	
Milk (peak daily concentration)	0.009 (day 6)	0.06
Urine		32.0
Faeces		56.7
Total in excreta		88.7

Residues in the kidney and perirenal fat were readily extracted with solvents. AMBA represented 79.0 and 61.6% TRR, equivalent to 0.038 and 0.013 mg/kg in the kidney and perirenal fat respectively. Unknown components did not account for more than 0.001 mg/kg (Table 14).

Table 14 Nature of the residues of AMBA in kidney and perirenal fat

	Kidney		Perirenal fat	
TRR, mg/kg (% chromatographed)	0.048 (87.9)		0.021 (81.7)	
Component/Fraction	%TRR	mg/kg	%TRR	mg/kg
AMBA	79.0 ^a	0.038	61.6	0.013
Unknown	0.5	0.000	3.2	0.001
Unidentified	8.4	0.004	16.9	0.004
Aqueous fractions ^b	5.9	0.003	21.3	0.004
Organic fractions	2.5	0.001	—	—
Unextracted	3.1	0.001	9.7	0.002
Gains/Losses	0.6	0.000	-12.7	0.003
Total	100.0	0.047	100.0	0.021

^a Summation of the % AMBA in two major fractions

^b Two fractions, the largest of which represents 12.6% TRR (0.003 mg/kg) in the perirenal fat and 3.0% TRR (0.001 mg/kg) in the kidney;

Based on the metabolism studies conducted with [cyclohexane-2- ^{14}C]-mesotrione, [phenyl- ^{14}C]-mesotrione and [^{14}C]AMBA with poultry, cow and swine, the proposed biotransformation of mesotrione in livestock involves the oxidative cleavage of the parent molecule to yield MNBA and the reduction of the nitro group in MNBA giving AMBA (Figure 1).

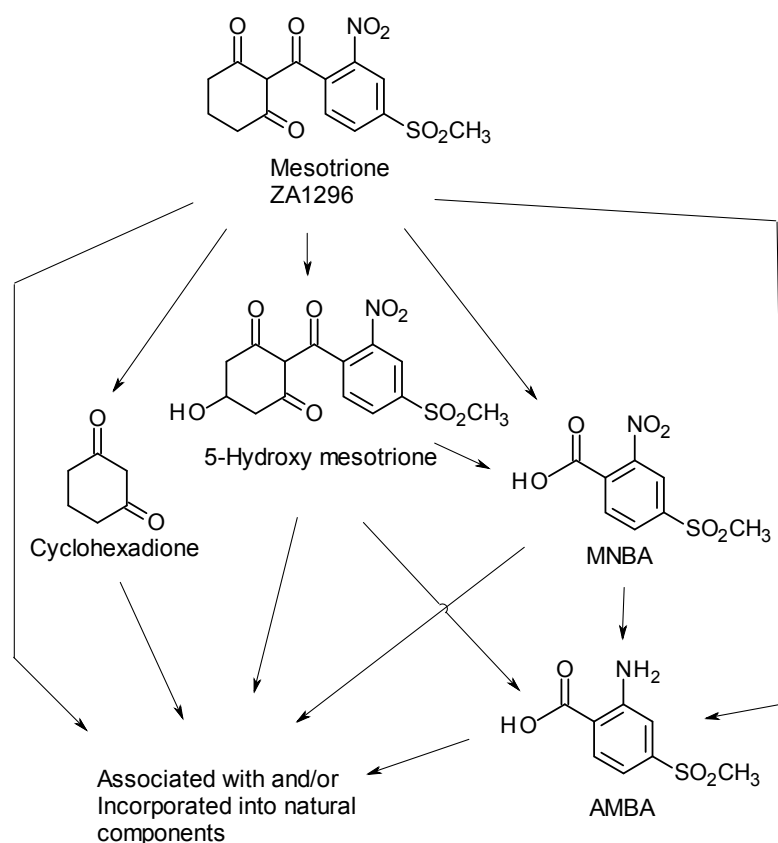


Figure 1 Proposed metabolism of mesotrione in livestock

Plant metabolism

Cranberries

[Phenyl- U - ^{14}C]-mesotrione was applied (diluted with aqueous formulation) twice to pollinated cranberry plants (*Vaccinium spp.* var. Howe), 4 to 5 years in age, in the greenhouse, under two dose regimes (Ray 2005). The first application at 0.331 or 0.919 kg ai/ha, and the second application, 14 days later, at 0.242 or 0.642 kg ai/ha. Total application at 1 $\times$ rate was 0.573 kg ai/ha and at 3 $\times$ rate 1.561 kg ai/ha. The radioactive residues in samples of mature cranberry foliage and fruit, harvested 46 days after the second foliar application, were quantified and characterized. A subsample of each commodity type was homogenised in dry ice and the radioactive residue determined by combustion/LSC. Mature fruit samples that were found to have a radioactive residue greater than 0.01 mg/kg were subject to additional analysis. Samples were extracted with acetonitrile/water and fractionated. Further treatment comprised of hydrolysis with dilute acid under reflux conditions. Extracted radioactivity was analysed by 2D TLC and reverse-phase HPLC. Additional confirmation and identification was undertaken using tandem mass spectrometry and ^1H -NMR.

About 100% of the total residues were extracted from cranberry fruits from both treatments, with about 2.2 to 2.5% corresponding to PES. TRR in the mature foliage were 16.8 mg/kg and 31.8 mg/kg, for the 1 $\times$ and 3 $\times$ treatments, respectively. The extracted TRRs in the mature cranberry fruit were 2.64 mg/kg eq. and 4.94 mg/kg eq., respectively. Only the fruits samples were used for the characterisation and identification of metabolites (Table 15). The extracted residues identified in cranberry fruits were mostly mesotrione and AMBA, accounting for 60.2% TRR and 34.8% at the 1 $\times$ rate, respectively, with MBNA accounting for 3.0% TRR.

Table 15 Summary of radioactive residues in mature cranberry fruit samples following treatment with [phenyl-U-¹⁴C]-mesotrione

Application Rate:	0.573 kg ai/ha		1.561 kg ai/ha	
Total extracted:	2.637 mg/kg eq.		4.936 mg/kg eq.	
Component	mg/kg eq.	%TRR	mg/kg eq.	%TRR
Mesotrione	1.548	60.2	3.257	67.1
AMBA	0.895	34.8	1.178	24.3
MBNA	0.076	3.0	0.078	1.6
Unknown baseline	0.042	1.6	0.057	1.2
Unextracted (PES)	0.064	2.5	0.107	2.2
Losses/gains	0.075	2.9	0.364	7.5
Total recovered	2.701	105.0	5.043	103.9

Mesotrione tolerant (HT) soya beans

HT soya bean seeds (*Glycine max* var. Jack) were grown under greenhouse conditions in containers filled with a sandy loam soil (Dohn & Chu, 2012). [Phenyl-U-¹⁴C]- and [cyclohexane-2-¹⁴C]-mesotrione were applied separately using three separate treatment regimes. The first application was a single pre-emergence treatment at a nominal rate of 0.225 kg ai/ha, the second was a combined pre-emergence treatment at 0.225 kg ai/ha followed by a post-emergence treatment at 0.125 kg ai/ha and the third was a single post-emergence treatment at a nominal 0.225 kg ai/ha. [¹⁴C]Mesotrione was applied to the plots as a suspension concentrate using a plastic hand held sprayer.

The radioactive residues in the soya bean foliage and hay samples were extracted twice with a mixture of acetonitrile and water (1:1). Soya bean seed samples were sequentially extracted with acetone/hexane (1:4), acetonitrile/water (1:1) and acetonitrile. Characterisation of the PES samples with residues $\geq 10\%$ TRR or ≥ 0.05 mg/kg was attempted using with hydrolytic enzymes, 1 N hydrochloric acid at room temperature and/or 60 °C. One and 2D normal phase TLC, reverse-phase HPLC-UV, radioisotope flow monitor and a fraction collector was used for sample analyses. Additional identification was undertaken using LS-MS.

The TRR (extracted plus PES) for soy commodities from each application regime and labels are summarised in the Table 16. In the phenyl label experiment, PES accounted for 41%TRR in pre-emergence forage to 57.7%TRR in post-emergence seed. In the cyclohexane label experiment, the values ranged from 21.9%TRR in pre-/post-emergence hay to 53.3%TRR in post-emergence seed.

Table 16 Total radioactive residues in soya bean RACs

Soya bean RAC	Application Stage	DAA (days)	TRR (mg/kg) Phenyl label	TRR (mg/kg) Cyclohexane label
Forage	Pre-emergence	28	0.212	0.077
	Pre- and post-emergence	28	0.162	0.055
	Post-emergence	22	0.499	0.260
Hay	Pre-emergence	42	0.142	0.076
	Pre- and post-emergence	42 (1 st app.); 9 (2 nd app.)	2.015	1.632
	Post-emergence	40	0.370	0.082
Seed	Pre-emergence	123	0.063	0.039
	Pre- and post-emergence	123 (1 st app.); 90 (2 nd app.)	0.104	0.093
	Post-emergence	110/118 ^a	0.052	0.015

^a [Phenyl-U-¹⁴C]-mesotrione: 110 days. [Cyclohexane-2-¹⁴C]-mesotrione: 118 days. DAA=days after application

A summary of radioactive residues from [phenyl-U-¹⁴C]-mesotrione treated soya bean forage extract is detailed in Table 17. MNBA was the most abundant residue present in the single pre-emergence and combined pre- and post-emergence forage samples and 4 or 5-hydroxy-mesotrione the most abundant residues in the post-emergence sample (0.073 mg/kg). Mesotrione residues were in the range of 0.021–0.03 mg/kg eq.). Extraction and acid hydrolysis released about 30% TRR found in pre-emergence forage PES, and lignin extraction released about 50% TRR in post-emergence forage

PES. Results from the [cyclohexane-2-¹⁴C]-mesotrione experiment showed mesotrione and 4 or 5-hydroxy-mesotrione as the major residues in forage (8.5 to 19.2% TRR), while MNBA and AMBA were not detected. Most of the radioactivity found in PES was released by enzyme, acid extraction and lignin extraction.

Table 17 Summary of radioactive residues in soya bean forage samples following pre- and post-emergence application of [¹⁴C]mesotrione

Treatment regime:	Pre-emergence ^a		Pre-+ post-emergence		Post-emergence	
Experiment	[phenyl- ¹⁴ C]	[Cyclo - ⁴ C]-	[phenyl- ¹⁴ C]	[Cyclo - ⁴ C]-	[phenyl- ¹⁴ C]	[Cyclo - ⁴ C]-
TRR, mg/kg eq.	0.212	0.077	0.162	0.055	0.499	0.260
Component	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)
MNBA	0.052 (24.5)		0.039 (24.1)		0.065 (13.0)	
AMBA	0.003 (1.4)		0.001 (0.6)		0.004 (0.8)	
4/5-Hydroxy-mesotrione	0.017 (8.0)	0.011 (14.3)	0.011 ^b (6.8)	0.007 (12.7)	0.073 (14.6)	0.050 (19.2)
Mesotrione	0.030 (14.2)	0.013 (16.9)	0.021 (13.0)	0.010 (18.2)	0.028 (5.6)	0.022 (8.5)
Polar unknowns ^c	0.005 (2.4)	0.009 (11.7)	0.006 (3.7)	0.005 (9.1)	0.008 (1.6)	0.034 (13.1)
Unassigned (< 10% TRR)	–		–		0.032 (6.4)	
Unassigned (each < 0.01 mg/kg)	0.018 (8.5)	0.010 (13.0)	0.010 (6.2)	0.08 (14.5)	0.044 (8.8)	0.057 (21.9)
Total characterised	0.125 (59.0)	0.043 (55.8)	0.088 (54.3)	0.030 (54.5)	0.254 (50.9)	0.163 (62.7)
PES	0.087 (41.0)	0.034 (44.2)	0.074 (45.7)	0.025 (45.5)	0.245 (49.1)	0.097 (37.3)
Total	0.212 (100)	0.077 (100)	0.162 (100)	0.055 (100)	0.499 (100)	0.260 (100)

^a Forage harvested prior to second application

^b Confirmed by TLC

^c Acetonitrile/water combined extracts

–=Not applicable

The metabolites MNBA and 4/5 hydroxy-mesotrione were the major residues found in hay of soya-treated with [phenyl-U-¹⁴C]-mesotrione in the three application regimes ranging from 9 to 20%TRR (Table 18). Mesotrione accounted for 6.2 to 8.8%TRR (up to 0.178 mg/kg eq.). Treatment of PES with enzymes, 1 N HCl and lignin digestion released 15.5%TRR in plants from the pre-emergence trial and 13.2% TRR in post-emergence. 4/5-hydroxyl-mesotrione was the major identified residue in hay of soya treated with [cyclohexane-2-¹⁴C] mesotrione, accounting for 15 to 25% TRR (0.409 mg/kg eq). Mesotrione accounted for 6.6 to 7.3%TRR, and the metabolites AMBA and MNBA were not detected. PES treatment released a total of 16.8% TRR in the pre-and post-emergence plants.

Table 18 Summary of radioactive residues in soya bean hay samples following pre- and post-emergence application of [phenyl-U-¹⁴C]-mesotrione

Treatment regime:	Pre-emergence		Pre- and post-emergence		Post-emergence	
Experiment	[phenyl- ¹⁴ C]	[Cyclo - ⁴ C]-	[phenyl- ¹⁴ C]	[Cyclo - ⁴ C]-	[phenyl- ¹⁴ C]	[Cyclo - ⁴ C]-
TRR, mg/kg	0.142	0.076	2.015	1.632	0.370	0.082
Component	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)
Mesotrione	0.009 (6.3)	0.005 (6.6)	0.178 (8.8)	0.134 (8.2)	0.023 (6.2)	0.006 (7.3)
MNBA	0.015 (10.6)	ND	0.410 (20.3)	ND	0.042 (11.4)	ND
AMBA	ND	ND	0.055 (2.7)	ND	ND	ND
4/5-Hydroxy-mesotrione	0.013 (9.2)	0.012 (15.8)	0.331 (16.4)	0.407 (24.9)	0.054 (14.6)	0.016 (19.5)
Polar unknowns	0.006 (4.2)	0.012 (15.8)	0.026 (1.3)	0.243 (14.9)	0.006 (1.6)	0.012 (14.6)
Unassigned (each < 6%TRR)	0.012 (8.5)	–	0.335 (16.6)	0.491 (30.1)	–	–
Non-defined (each < 0.01 mg/kg)	0.013 (9.2)	0.010 (13.2)	ND	ND	0.05 (13.5)	0.015 (18.3)
Total characterised	0.068 (47.9)	0.039 (51.3)	1.34 (66.3)	1.275 (78.1)	0.175 (47.3)	0.049 (59.8)
PES	0.074 (52.1)	0.037 (48.7)	0.680 (33.7)	0.357 (21.9)	0.195 (52.7)	0.033 (40.2)
Total	0.142 (100)	0.073 (100)	2.015 (100)	1.632 (100)	0.370 (100)	0.082 (100)

None of the identified residues in soya bean seeds from the experiment with [phenyl-U-¹⁴C]-mesotrione accounted for more than 10% TRR in any treatment or at levels higher than 0.007 mg/kg eq. (Table 19). Mesotrione accounted for up to 9.5% TRR%, or 0.006 mg/kg eq. AMBA was detected only in the pre-and post-emergence regime (1.9%TRR). Enzyme extracts of PES contained multiple components up to ≤ 0.007 mg/kg. Only mesotrione and 4/5-hydroxy-mesotrione were detected in soya bean seeds from the [cyclohexane-2-¹⁴C] mesotrione experiment. The PES Viscozyme extracts from the combined pre- and post-emergence sample mainly contained unknown polar peak components (0.011 mg/kg, 11.8% TRR).

Table 19 Summary of radioactive residues in soya bean seed samples following use of [phenyl-U-¹⁴C]-mesotrione

Treatment regime:	Pre-emergence		Pre- and post-emergence		Post-emergence	
Experiment	[phenyl- ¹⁴ C]	[Cyclo - ¹⁴ C]	[phenyl- ¹⁴ C]	[Cyclo - ¹⁴ C]	[phenyl- ¹⁴ C]	[Cyclo - ¹⁴ C]
TRR, mg/kg	0.063	0.039	0.104	0.093	0.052	0.015
Component	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)
Mesotrione	0.006 (9.5)	0.02 (5.1)	0.003 (2.9)	0.003 (3.2)	0.002 (3.8)	
MNBA	0.001 (1.6)	ND	0.005 (4.8)	ND	ND	
AMBA	ND	ND	0.002 (1.9)	ND	ND	
4/5-Hydroxy-mesotrione	0.003 (4.8)	0.01 (2.6)	0.007 (6.7)	0.003 (3.2)	0.004 (7.7)	
Polar unknowns	0.004 (6.3)	0.07 (17.9)	0.010 (9.6)	0.029 (31.2)	0.008 (15.4)	
Unassigned peaks	–		0.010 (9.6)		–	
Non-defined (each < 0.01 mg/kg)	0.008 (12.8)	0.02 (5.1)	0.007 (6.7)	0.007 (7.5)	0.003 (5.8)	
Total characterised	0.022 (34.9)	0.012 (30.8)	0.044 (42.3)	0.042 (45.2)	0.017 (32.7)	
PES	0.035 (55.6)	0.020 (51.3)	0.052 (50.0)	0.039 (41.9)	0.030 (57.7)	0.008 (53.3)
Acetone/Hexane extractions	0.006 (9.5)	0.007 (17.9)	0.008 (7.7)	0.012 (12.9)	0.005 (9.6)	0.002 (13.3)
Totals	0.063 (100)	0.039 (100)	0.104 (100)	0.093 (100)	0.052 (100)	0.015 (110)

Maize

[Phenyl-U-¹⁴C]-mesotrione was applied to the soil surface after planting the seeds of maize (*Zea mays*) (pre-emergence) at a rate of 0.28 kg ai/ha and post-emergence by application to surfaces of plants and soil 28 days after planting at a rate of 0.164 kg ai/ha (Tarr & van Neste, 1997; RR 96-007B). The radioactive residues in plant samples of forage (55 days after planting) and of grain and straw from mature crops (125 days after post-emergence treatment) were characterised.

Samples were extracted with water and acetonitrile (1:1) and fractionated by partition with ethyl acetate and/or with Amberlite XAD7 resin. Acid hydrolysis (micro-wave or reflux) followed by base hydrolysis or treatment with enzymes (porcine carboxylic acid esterase, papain, pancreatin, pectolyase and driselase) of the extracted residues and PES was attempted for characterisation and further solubilisation of residues. The PES from post-emergence fodder was also treated with a solution of potassium permanganate to solubilise bound residues by oxidation. Residue characterization was performed using normal and reversed-phase TLC and/or reversed-phase HPLC with radio-detector or fraction collecting, and LSC.

Table 20 summarises the TRRs found in maize matrices after pre- or post-emergence application. Total residues were higher in fodder and forage, and similar in both treatments in seed (0.013–0.014 mg/kg eq.). Using acetonitrile/water (1:1 v/v), most of the residues were extracted from forage and seed, but unextracted residues accounted for over 60% TRR in fodder. No additional analysis was conducted in seed due to the low radioactivity found.

Table 20 Total radioactive residues (TRRs) in maize after pre- and post-emergence application of [phenyl-U-¹⁴C]-mesotrione

Sample. DAT	TRR by initial combustion	Extracted residues (acetonitrile/water 1:1 v/v)		Unextracted residues		Sum of extracted and unextracted residues	
	mg/kg eq.	% TRR ^a	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.
Pre-emergence application							
Forage. 27	0.356	84.6	0.301	26.2	0.093	110.8	0.394
Fodder. 153	0.795	43.9	0.349	61.8	0.491	105.7	0.840
Grain. 153	0.013	69.6	0.009	26.8	0.004	96.7	0.013
Post-emergence application							
Forage. 28	0.244	72.7	0.177	23.9	0.058	96.6	0.235
Fodder. 125	1.066	48.1	0.513	67.4	0.719	115.5	1.232
Grain. 125	0.014	61.5	0.008	23.2	0.003	84.7	0.011

DAT: Days after treatment

^a Based on residue values derived from initial combustion

Table 21 shows the characterization of the residues found in forage and fodder from both treatments. Mesotrione was a minor component of the residue, present at a higher level in pre-emergence forage samples (2.2% TRR, 0.008 mg/kg eq.). MNBA was the major identified compound in forage from the pre-emergence treatment (19.7%TRR) and a minor component in post-emergence and in fodder. AMBA was a major compound mostly presented in conjugated form accounting for 12–28%TRR in forage and fodder. The predominant AMBA conjugates isolated were α - and β -anomers of the 2-acylglucosides. Acid or base hydrolysis and enzyme treatment did not release additional AMBA from fodder extracts.

Table 21 Summary of radioactive residues in maize forage and fodder samples following pre- and post-emergence application of ¹⁴C-phenyl labelled mesotrione

Component	Pre-emergence application		Post-emergence application	
	Forage	Fodder	Forage	Fodder
	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)
TRR by combustion	0.356 (100)	0.795 (100)	0.244 (100)	1.066 (100)
Mesotrione	0.008 (2.2)	< 0.003 (< 0.4)	0.001 (0.4)	< 0.003 (< 0.3)
4-OH-mesotrione (total/glucose Conjugate)	0.027/0.013 (7.6/3.8)	0.007/< 0.01 (0.9/< 1.2)	0.016/0.009 (6.6/3.6)	0.007/< 0.01 (0.7/< 0.1)
MNBA	0.070 (19.7)	0.008 (1.0)	0.008 (3.4)	0.019 (1.9)
AMBA (total/conjugates)	0.043/0.031 (12.2/8.9)	0.108/0.104 (13.6/13.0)	0.032/0.029 (13.2/11.5)	0.301/0.295 (28.2/27.6)
Minor components extracted with water/acetonitrile	0.164 (46.0)	0.280 (35.2)	0.128 (52.4) (each < 0.03 mg/kg)	0.320 (30.0) (each < 0.03 mg/kg)
Minor components extracted with 0.1 M HCl /microwave heating	0.049 (13.6)	0.229 (28.8)	0.033 (13.7) (each < 0.03 mg/kg)	0.378 (35.4) (each < 0.03 mg/kg)
Minor components extracted with 1 M NaOH/40 °C		0.030 (3.8)		0.044 (4.1)
Not extracted	0.028 (7.9)	0.033 (4.2)	0.017 (7.0)	0.063 (5.9)

In another study conducted in maize, [phenyl-U-¹⁴C]-mesotrione was applied to the bare sandy loam soil surface one day after planting the seeds (pre-emergence) at a rate of 0.302 kg ai/ha and post-emergence (31 days after planting) by application to surfaces of plants at the true eight leaf stage at a rate of 0.179 kg ai/ha (Vispetto & Smith, 1999; RR 99-006B). Samples of forage (78 days after first pre-emergence treatment) and of stover and grain from mature crops, 121 days after pre-emergence treatment were taken for analysis.

Samples were extracted with a mixture of water and acetonitrile (1:1), extracts fractionated by hydrolysis with HCl or partitioned with ethyl acetate. PES which exceeded 10% TRR or 0.05 mg/kg were hydrolysed by microwave extraction with HCl, and the hydrolysates cleaned up by C₁₈ SPE. HPLC radiochromatograms were obtained by fraction collecting and LSC. TLC was conducted using silica plates.

Highest residues were detected in the stover (0.57 mg/kg), with lower residues in the forage (0.27 mg/kg) and grain (0.03 mg/kg) (Table 22). Over 60% of the radioactivity was extracted and contained a large number of components, all at ≤ 0.01 mg/kg and predominantly water soluble. Mesotrione was not detected in any sample. AMBA, MNBA and their conjugates, and 4-hydroxy-mesotrione were the major compounds identified in forage. 4-hydroxy mesotrione was not detected in grain and stover.

Table 22 Total radioactive residues (TRRs) in maize after pre- and post-emergence application of [phenyl- ^{14}C]-mesotrione

		Forage		Stover		Grain	
		% TRR	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.
Total Residue (by combustion)		100	0.27	100	0.57	100	0.03
Acetonitrile/water		69.6	0.19	70.3	0.40	60.6	0.02
2 M/4 M HCl Hydrolysis of extract	Organosoluble	—	—	9.5	0.05	—	—
	Aqueous soluble	—	—	45.8	0.26	—	—
	Precipitate	—	—	10.7	0.06	—	—
Non-extracted (PES)		29.9	0.08	32.9	0.19	46.9	0.01
2 M/4 M HCl PES Hydrolysis		9.8	0.03	7.0	0.04	—	—
Post-hydrolysed solids (PHS)		22.6	0.06	23.5	0.13		
AMBA/ conjugate		2.4/4.6	0.01/0.01	1.7/2.3	0.01/0.01	—	—
MNBA/ conjugate		3.3/2.2	0.01/< 0.01	2.2/1.0	0.01/0.01	—	—
4-hydroxy-mesotrione		5.4	0.01	—	—	—	—
Unidentified		5.7	0.14	7.7	0.34	6.9	0.02

^a Fraction comprises a number of small components all < 4.6% TRR (≤ 0.02 mg/kg)

In the third study conducted in maize, [cyclohexane-2- ^{14}C]-mesotrione was applied to the soil surface after planting the seeds (pre-emergence) at a rate of 0.307 kg ai/ha and post-emergence 28 days after planting at a rate of 0.161 kg ai/ha (Wei & Dohn, 1997; RR 96-026B). Samples of forage (27 or 28 days after planting) and of grain and straw from mature crops (125 days after post-emergence treatment) were extracted three times with either a mixture of water and acetonitrile (1:1). Acid hydrolysis of the extracted residues and PES from post-emergence fodder samples was attempted for characterization of residues and enzyme digestion was used in pre- and post-emergence forage samples. HPLC radiochromatograms were obtained using a flow through radio-detector or by fraction collecting and LSC. All critical analyses were confirmed by use of at least two TLC and or HPLC methods.

Total radioactive residues in maize were 0.067, 0.001 and 0.015 mg/kg in pre-emergence forage, grain and fodder respectively. Corresponding values for post-emergence treatments were 0.098, 0.011 and 0.330 mg/kg (Table 23). Much of the radioactivity in forage was readily extracted (78–84% TRR). Remaining solids after extraction were not analysed further. About 60% TRR in pre-emergence fodder and post-emergence grain were not extracted with solvents.

Table 23 Total radioactive residues (TRRs) in maize after pre- and post-emergence application of [cyclohexane-2- ^{14}C]-mesotrione

Sample	TRR by initial combustion	Extracted residues		Unextracted residues	
	mg/kg	% TRR ^a	mg/kg eq.	% TRR	mg/kg eq.
Pre-emergence application					
Forage	0.067	78.3	0.053	20.9	0.014
Fodder ^b	0.015 (0.025 in leaves)	38.1	0.006 (0.010 in leaves)	60.8	0.010 (0.015 in leaves)
Grain	0.001	—	—	—	—
Post-emergence application					
Forage ^b	0.098 (0.190 in leaves)	84.0	0.082 (0.159 in leaves)	19.6	0.018 (0.037 in leaves)
Fodder	0.330 ^b (0.649 in leaves)	67.5 ^c	0.223	9.4	0.031
Grain	0.011	35.1	0.004	60.7	0.007

^a % TRR's are based on residue values derived from initial combustion

^b The extractions were performed on the leaf sample, but the mg/kg values are corrected for the total (leaves + stalks) sample mass

^c Includes material released by acid hydrolysis and lignin and cellulose fractions

The major identified residue in forage was 4-dihydroxy-mesotrione, accounting for up to 10.4%TRR in pre-emergence samples (0.007 mg/kg) (Table 24). Mesotrione was found at low levels (up to 0.002 mg/kg). In fodder, mesotrione was detected only in trace amounts (< 0.0005 mg/kg) and no other compound was identified (Table 24). The solubilised material from PES accounted for 13.6% TRR (0.045 mg/kg) and showed to contain glucose. Treatment of remaining solids gave evidence for the incorporation of ¹⁴C into lignin and cellulose.

Table 24 Summary of radioactive residues in maize forage and fodder following pre- and post-emergence application of [cyclohexane-2-¹⁴C]-mesotrione

Component	Forage		Fodder (leaves and stalks)
	Pre-emergence mg/kg (% TRR)	Post-emergence ^a % of TRR ^b	post-emergence mg/kg eq.
TRR combustion value	0.067 (100)	0.098 (100)	0.330 (100)
Mesotrione	0.002 (3.0)	0.001 (1.0)	< 0.0005
4-Hydroxy-mesotrione	0.007 (10.4)	0.006 (6.1)	
Components extractable with water/acetonitrile (including neutral carbohydrates formed by acid hydrolysis of the extract) ^c			0.123 (37.3) (each < 0.012 mg/kg)
Components solubilised by refluxing 6 M HCl. majority were neutral carbohydrates, including glucose ^c			0.045 (13.6) (each < 0.029)
Intractable solids from neutralisation of 6 M HCl reflux			0.016 (4.8)
Lignin ^c			0.044 (13.3)
Cellulose ^c	0.006 (9.0)	0.002 (2.0)	0.011 (3.4)
Minor components including carbohydrates	0.038 (56.7)	0.067 (each ≤ 0.012 mg/kg)	
Post-extracted solids (PES)	0.014 (20.9)	0.018 (18.4)	0.031 (9.4)
Not analysed ^b	–	0.007 (7.1)	0.024 (7.3)

^a Leaves and the woody stalks, the leaves contained over 90% of the residue

^b The characterisation experiments were performed on the leaf sample, but the values are corrected for the total (leaves + stalks) sample

^c The order of the extractions was water/acetonitrile. 6 M HCl, hot dimethylsulphoxide (for lignin), and Schweizer's reagent (for cellulose)

Rice

Rice plants (*Oryza sativa* spp. *japonica* cv Kirana 397) at the 2–3 leaf stage in a flooded paddy greenhouse system were treated with [phenyl-U-¹⁴C]-mesotrione added directly to the paddy water at either 0.090 kg ai/ha or 0.225 kg ai/ha (Humphries & Evans. 2005; RJ 3738B). The day 14 and 27 days after treatment (DAT) samples were whole tops, the 40 DAT samples were separated into ears and stalks, the 109 DAT samples were separated into grain, husk and straw. The paddy was maintained flooded to a depth of 3–5 cm until 7 days before final harvest, when irrigation was stopped allowing the paddy system to dry. Natural daytime sunlight was supplemented with artificial light with a fixed 13 hour day period. Samples were extracted with acetonitrile/water mixture and water and PES were further extracted with microwave assisted extractions and acid hydrolysis. Liquid partitions were carried out between ethyl acetate/water (1:1). Extracted radioactivity was analysed by normal and reversed-phase TLC and reverse-phase HPLC.

Extracted radioactivity in the whole tops up to 27 DAT accounted for at least 60%TRR (Table 25). Total residues in grain were 0.01 and 0.02 mg/kg eq. at 0.09 and 0.225 kg ai/ha, respectively, with extracted residues accounting for up to 0.003 mg/kg eq., which were not further characterized. Residues in straw were higher (0.032 and 0.066 mg/kg eq.), from which 43–45%TRR was extracted.

Table 25 Total radioactive residues (TRRs) in rice after treatment of paddy water with [phenyl-U-¹⁴C]-mesotrione

Treatment. kg ai/ha	DAA		Acetonitrile/water		Non-extracted residues		Total Residue mg/kg eq.
		Commodity	% TRR ^a	mg/kg	% TRR	mg/kg	
0.090	14	Whole tops ^{b)}	70.7	0.0461	29.3	0.0191	0.0652
	27	Whole tops	59.9	0.0198	40.1	0.0133	0.0331
	40	Ears	n/a	n/a	n/a	n/a	0.0057
		Stalks	48.1	0.0092	51.9	0.0099	0.0191
	109	Grain	12.4	0.0011	87.7	0.0087	0.0099
		Husk	22.7	0.0022	77.3	0.0077	0.0099
		Straw	43.1	0.0139	56.9	0.0183	0.0321
0.225	14	Whole tops	76.2	0.1934	23.8	0.0604	0.2538
	27	Whole tops	60.7	0.0416	39.3	0.0270	0.0686
	40	Ears	27.0	0.0032	73.0	0.0085	0.0117
		Stalks	46.4	0.0175	53.6	0.0202	0.0377
	109	Grain	17.2	0.0033	82.8	0.0158	0.0191
		Husk	33.1	0.0108	66.8	0.0217	0.0325
		Straw	45.3	0.0298	54.7	0.0360	0.0659

^a %TRR's are based on residue values derived initial combustion^b Immature plants harvested by cutting the plant 2 cm above the paddy rice water

Tables 26 shows the residues identified in immature whole tops (14 and 27 DAT) and stalks (40 DAT). All commodities analysed contained parent mesotrione, 5-hydroxy-mesotrione, AMBA and MNBA. At early stage, mesotrione comprised the majority of the residues, accounting for 28% TRR for the 0.225 g ai/ha treatment.

Table 26 Summary of radioactive residues in immature rice samples following treatment of paddy water with phenyl-U-¹⁴C labelled mesotrione

Component	0.090 kg ai/ha						0.225 kg ai/ha	
	14 Whole Tops ^a		27 Whole Tops ^a		40 Stalks		14 Whole Tops	
	% TRR	mg/kg	% TRR	mg/kg eq.	% TRR	mg/kg	% TRR	mg/kg
Mesotrione	15.0	0.0098	5.9	0.0020	5.0	0.0010	27.9	0.0708
5-HO-mesotrione ^b	11.4	0.0074	7.5	0.0025	11.1	0.0021	14.1	0.0359
MNBA	4.6	0.0030	4.8	0.0016				
AMBA	2.0	0.0013	3.0	0.0010	1.7	0.0003	2.2	0.0055
Total	33.0	0.0215	21.2	0.0071	17.8	0.0034	44.2	0.1122
Unknowns	4.9	0.0032						
Baseline	16.2	0.0105	30.1	0.0100	16.5	0.0032	16.0	0.0406
Remainder	18.5	0.0120	6.8	0.0021	15.4	0.0030	15.2	0.0386
Unextracted ^c	29.3	0.0191	40.1	0.0133	51.9	0.0099	23.8	0.0604
Loss/gain on fractionation ^c	-1.8	-0.0012	1.8	0.0006	-1.7	-0.0003	0.8	0.0020
Total	100.1	0.0651	100.0	0.0331	99.9	0.0192	100.0	0.2538

Unknowns: unidentified discrete components; Baseline: Polar residue components, which are retained on the baseline after elution

Remainder: diffuse unassigned radioactivity comprising minor indiscrete components and areas of streaking of radioactivity

^a Immature plants harvested by cutting the plant 2 cm above the paddy rice water^b 5-Hydroxy-mesotrione was assigned based on TLC data from later samples where reference standards were available^c Not chromatographed

Residues in straw extracts showed the similar pattern as seen in whole tops and stalk samples (Table 27). Baseline polar extract was acidified to 0.1 M with HCl, heated to 80 °C for two hours to released 5-hydroxy-mesotrione (3.2% TRR, 0.001 mg/kg) and MNBA (2.4% TRR, 0.0008 mg/kg), mesotrione (1.5% TRR, 0.0005 mg/kg) and AMBA (1.1% TRR, 0.0004 mg/kg). The 56.9% TRR

(0.0183 mg/kg) in the PES was further extracted showing mesotrione and AMBA present at < 1% TRR (0.0002 mg/kg), MNBA and 5-hydroxy-mesotrione (0.4% TRR).

Table 27 Summary of radioactive residues in mature rice straw (109 DAT) following treatment of paddy water with phenyl-U-¹⁴C labelled mesotrione at 90 g ai/ha

Component	Solvent extraction		Acid hydrolysis of solvent extract		Acid hydrolysis of debris sample	
	% TRR	Residue	% TRR	Residue	% TRR	Residue
Mesotrione	1.8	0.0006	1.5	0.0005	0.7	0.0002
5-HO-mesotrione ^a	5.0	0.0016	3.2	0.0010	0.4	0.0001
MNBA			2.4	0.0008		
AMBA	1.2	0.0004	1.1	0.0004	0.6	0.0002
Baseline	24.6	0.0079	13.8	0.0044	13.3	0.0042
Remainder	7.6	0.0024	9.0	0.0029	3.5	0.0012
Extracted	n/a	n/a	n/a	n/a	53.7	0.0173
Unextracted ^b	56.9	0.0183	56.9	0.0183	26.1	0.0084
Loss/gain on fractionation ^b	2.8	0.0009	12.0	0.0038	1.6	0.0005
Total	99.9	0.0321	99.9	0.0321	99.9	0.0321

Unknowns: unidentified discrete components; Baseline: Polar residue components which are retained on the baseline after elution; Remainder: diffuse unassigned radioactivity comprising minor indiscrete components and areas of streaking of radioactivity

^a 5-hydroxy-mesotrione was assigned on basis of comparison with TLC data from later samples where reference standards were available

^b Not chromatographed

Peanuts

[Phenyl-U-¹⁴C]-mesotrione was solubilised in acetonitrile, diluted with aqueous blank formulation and applied to peanuts (*Arachis hypogaea* var. NCV 11) planted in silt loam soil in three outdoor subplots (Brown, 2003; Report 1286-01). [¹⁴C]mesotrione was applied to the soil surface after planting the seeds (pre-emergence) at 0.305 and 0.796 kg ai/ha. Peanut foliage was harvested 90 DAT (50% maturity), mature peanuts and peanut hay were harvested 153 DAT.

Samples were extracted four times with water:acetonitrile (8:2) and nutmeat was extracted twice with hexane and twice with acetonitrile:water (8:2). Aliquots of the extracts were partitioned with hexane, dichloromethane or chloroform. Cellulase and amyloglucosidase enzymes were added to sodium acetate buffered subsamples of aqueous and hexane fractions (pH 4.6). Esterase, lipase, and/or pancreatin were added to TRIS-hydrochloric acid buffered subsamples of aqueous and hexane fractions (pH 7.8–8). Samples were incubated for approximately 24 hours at 42 or 47 °C. Aqueous and hexane fractions and PES were hydrolysed with 6 N hydrochloric acid under reflux followed by XAD-7 separation and/or dichloromethane partitioning. Hexane fractions were hydrolysed with 1 N potassium hydroxide in water under reflux for approximately 6 or 24 hours. Aminolysis using diethylamine was attempted. PES was refluxed over night with water to extract various complex sugars. Plant extracts cleaned up with C₁₈ or NH₂ SPE. Components were separated using 2-D normal phase TLC. Reversed-phase HPLC coupled to UV detector, radioisotope flow monitor and a fraction collector was also used. Additional separation was performed by ion exchange chromatography where necessary.

Table 28 shows the residues found in peanut foliage and hay after both treatments. From 30 to 42% TRR was extracted from foliage and hay, with MNBA being the major residue. AMBA residues were released from PES after basic hydrolysis. MBA was found in trace amount only in hay treated at the highest rate.

Table 28 Summary of radioactive residues in peanut foliage and hay samples following pre-emergence treatment with [phenyl-U-¹⁴C]-mesotrione

Application Rate:		0.305 kg ai/ha		0.796 kg ai/ha	
Commodity		Foliage	Hay	Foliage	Hay
Total Radioactive Residue (%TRR):		0.028 (100)	0.012 (100)	0.064 (100)	0.028 (100)
Extracted residues (%TRR) ^a		0.012 (42.2)	0.004 (31.5)	0.026 (40.5)	0.010 (34.2)
Component		mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)
Aqueous following partition (%TRR)		0.007 (23.6)	0.002 (16.0)	0.012 (19.2)	0.007 (24.8)
MNBA		0.003 (10.9)	0.001 (4.0)	0.007 (10.7)	0.002 (5.8)
AMBA		0.001 (2.1)	–	< 0.001 (0.5)	< 0.001 (1.1)
MBA					< 0.001 (1.1)
Unknown 1		0.001 (4.5)	0.001 (4.0)	0.003 (4.4)	0.002 (7.6)
Unknown 2		0.001 (1.9)	< 0.001 (1.0)	–	0.001 (3.4)
Unknown 4			–		0.001 (3.3)
Others		^b	^d	^c	
Organic following partition (%TRR):		0.004 (15.8)	0.002 (13.3)	0.010 (15.6)	0.003 (8.8)
Organic after 6 N HCl hydrolysis		0.003 (12.3)	0.001 (10.3)	0.009 (14.1)	0.002 (6.7)
AMBA		0.001 (4.9)	< 0.001 (2.2)	0.003 (4.3)	< 0.001 (1.5)
Unextracted (PES)		0.014 (48.7)	0.008 (62.8)	0.038 (59.6)	0.019 (67.7)
PES treated with NaOH	MNBA	< 0.001 (1.4)	< 0.001 (1.1)	–	< 0.001 (0.6)
	MBA	–	< 0.001 (3.2)	< 0.001 (0.6)	0.001 (1.7)
	AMBA	0.003 (9.7)	< 0.001 (4.7)	0.002 (2.3)	0.001 (2.0)

^a Extract was separated into aqueous and organic fraction^b At least seven components ranging from 0.2% to 1.4% TRR^c At least eight components ranging from 0.1% to 1.2% TRR^d At least five components ranging from 0.3% to 0.7% TRR

The TRRs in hulls and nutmeat for plots treated with the higher rate were 0.025 and 0.037 mg/kg, respectively (Table 29). Extracted residues were higher from nutmeat samples (35–40%TRR) than hulls (20–23% TRR). Low levels of MNBA residues were detected in hull samples treated at both application rates (≤ 0.002 mg/kg) including residues released from PES. Traces of AMBA, 4-hydroxy-mesotrione and MBA were detected only after hydrolysis of hull PES.

Table 29 Summary of radioactive residues in peanut hulls and nutmeat samples following treatment with [phenyl-U-¹⁴C]-mesotrione

Application Rate:		0.305 kg ai/ha		0.796 kg ai/ha	
Commodity		Hulls	Nutmeat	Hulls	Nutmeat
Total Radioactive Residue, mg/kg eq.		0.011	0.013	0.025	0.037
Extracted residues		0.003 (23.0) ^a	0.005 (40.0)	0.005 (19.6) ^a	0.013 (35.9)
Aqueous following partition (%TRR):		0.002 (15.5)		0.005 (18.2)	
Acetonitrile fraction from nutmeat extract ^b			< 0.001 (3.8)		0.001 (3.9)
Hexane fraction from nutmeat extract ^c			0.005 (36.2)		0.013 (35.9)
Chromatography of aqueous partition for hulls	Component	mg/kg	%TRR	mg/kg	%TRR
	MNBA	< 0.001 (2.4)		< 0.001	1.7
	Unknown 1	< 0.001 (5.4)		0.002	6.7
	Unknown 2	< 0.001 (3.0)			
	Unknown 3	–	–	< 0.001	1.7
	Others	^d		^e	
Organic following partition of hull extract (%TRR):		< 0.001 (5.3)		< 0.001	2.2
Unextracted (PES)		0.007 (60.1)	0.008 (60.7)	0.017 (68.5)	0.023 (61.5)
PES treated using NaOH	MNBA	< 0.001 (1.2)		0.002 (7.9)	< 0.001 (2.4)
	MBA	0.001 (6.7)		–	–

Application Rate:		0.305 kg ai/ha		0.796 kg ai/ha	
	AMBA	< 0.001 (1.6)	0.002 (15.0)	< 0.001 (1.4)	< 0.001 (1.4)
	4-hydroxy-mesotrione	0.001 (6.9)		–	–

^a Extract was separated into aqueous and organic fraction

^b Low residues in the aqueous fraction and its concentrated matrices prevented TLC and HPLC analyses of samples

^c The hexane fraction was further characterised by treatment with base partition treatment with acid and further partition or with a mixture of enzymes which afforded several unknowns none greater than 0.002 mg/kg

^d At least three components ranging from 0.2% to 1.2% TRR

^e At least four components ranging from 0.2% to 1.4% TRR

In another study conducted with peanuts, [cyclohexane-2-¹⁴C]-mesotrione was applied to the soil surface after planting the seeds (pre-emergence) at rates of 0.327 kg ai/ha and 0.836 kg ai/ha (Brumback, 2003; Report 1287-01). Peanut foliage was harvested 90 DAT (50% maturity), and mature peanuts and peanut hay harvested 154 DAT. Auxiliary in vitro experiments with peanut cell culture and excised peanut shoots were performed to generate metabolites for identification. Peanut cells were dosed at 50 mg/L [cyclohexane-2-¹⁴C]-mesotrione, diluted in dimethyl sulfoxide (DMSO) and harvested 7 and 14 days after dosing. Peanut shoots were dosed at 100 mg/L and harvested after 2 days. Plant samples having radioactive residues ≥ 0.010 mg/kg were extracted with water:acetonitrile (8:2). Nutmeat was sequentially extracted with hexane, hexane/ethyl acetate and acetonitrile:water (8:2). Aliquots of the extracts were partitioned between aqueous and organic phases. An aqueous buffer mixture of 0.1 M sodium acetate buffer and 0.1 M acetic acid (1:1) was added to subsample extracts, following addition of cellulase and amyloglucosidase enzymes and incubation for 16 hours at 37 to 47 °C. Nutmeat extracts were acidified to pH 1 after microwave treatment and partitioning between water and hexane. Components in extracts were separated using 2-D normal phase TLC and/or reversed-phase or anion exchange. The components were detected with HPLC-UV detector; radioisotope flow monitor and a fraction collector were also used. Additional confirmation was undertaken using tandem mass spectrometry and 1H-NMR for the metabolites isolated from the in vitro experiments.

Samples of peanuts treated at 0.327 kg ai/ha resulted in TRR < 0.01 mg/kg eq and no further analysis in the samples was performed. Table 30 shows the radioactive residues found in peanut samples from the higher rate plots. TRR was higher in foliage and nutmeat (0.02 mg/kg eq.), with about 40–50%% being extracted. Characterisation of extracts from foliage, hay and hull samples from the 0.836 mg/kg rate showed only one significant metabolite, identified as 4-hydroxy-mesotrione, which was also generated in vitro. The peanut oil fraction was shown to be composed primarily of ¹⁴C-labelled neutral lipids resulting from metabolism of mesotrione to single carbon units that entered the carbon pool.

Table 30 Summary of radioactive residues in peanut samples following treatment with [cyclohexane-2-¹⁴C]-mesotrione at 0.836 kg ai/ha

Crop and Commodity:	Peanut foliage	Peanut hay	Peanut hulls	Peanut nutmeat
TRR, mg/kg eq.:	0.020	0.011	0.015	0.022
Initial extraction (%TRR):	0.008 (38.9%)	0.003 (28.2%)		
Aqueous acetonitrile extract (%TRR):			0.003 (19.9%)	0.001 (4.7%)
Non polar solubles (%TRR)			–	0.011 (51.4%)
Component	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)
Neutral/base components	0.003 (11.7)	0.001 (6.7)	< 0.001 (4.4)	–
Acidic components	0.003 ^a (17.3)	0.002 ^{c, d} (16.0)	0.003 ^{a, c} (12.6)	–
Organic wash	< 0.001 (1.2)	< 0.001 (1.4)	< 0.001 (1.8)	–
Lipids				0.008 (37.8)
Fatty acids				0.001 (5.7)
Phospholipids				0.001 (4.1)
Unextracted (PES)	0.013 (65.9)	0.008 (71.6)	0.013 (84.5)	0.011 (50.8)

^a Three separate regions

^b Two separate regions

^c Contained a component characterised as 4-hydroxy-mesotrione (1.4% TRR)

Based on the metabolism studies conducted in cranberries, soya bean, maize, rice and peanut with labelled mesotrione, a metabolism pathway of mesotrione in plants is proposed in Figure 2. The process involves hydroxylation, hydrolysis to form MNBA, which is reduced to AMBA followed by de-amination to form MBA.

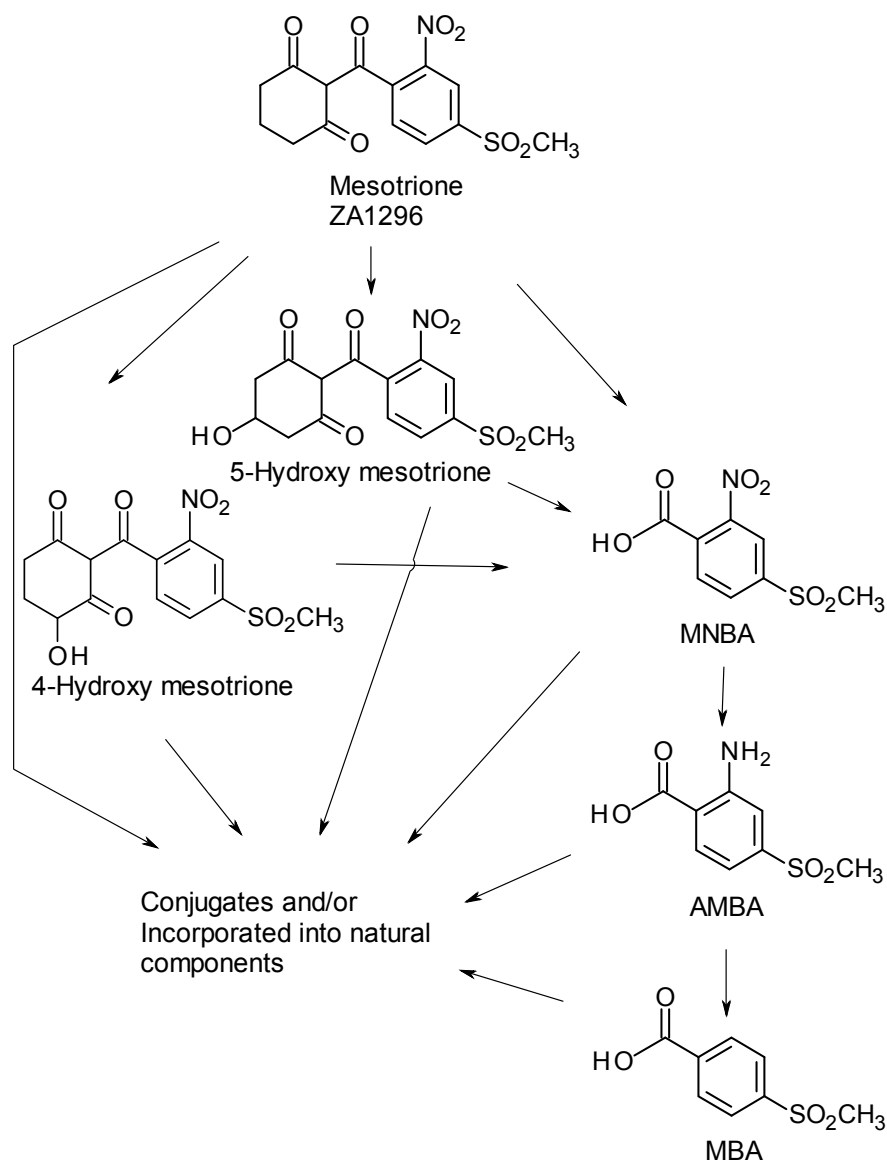


Figure 2 Proposed metabolism of mesotrione in plants

Environmental Fate in Soil

Two soil photolysis studies and five aerobic and anaerobic degradation studies of mesotrione in soil were conducted, using soils collected in 1994 and 1995 in Richmond, Wisconsin (USA). The soil characteristics are shown in Table 31.

Table 31 Silt loam soils used to investigate the metabolism of mesotrione under aerobic conditions

	Studies	Studies
Soil properties	RR95-082B, RR95-047B, RR96-033B, RR96-060B	RR97-033B
% Sand	17.1	16.4

	Studies	Studies
Soil properties	RR95-082B, RR95-047B, RR96-033B, RR96-060B	RR97-033B
% Silt	57.7	58.9
% Clay	25.2	24.8
% Organic Matter	2.72	2.16
pH	6.2	6.1
Cation exchange capacity (meq/100 g)	12.0	10.5
% Moisture holding capacity (saturation)	—	47.2
% Moisture holding capacity (1/3 Bar)	27.4	—
Moisture holding capacity (15 Bar)	7.90	—
Microbial biomass, mg C/kg (zero to end of incubation)	^a	230 to 182–191

^a Biological activity of soil was monitored by measuring the soil respiration for 11 days soil pre-incubation. The soil was active as indicated by the formation of 0.6 mg of CO₂ per gram of soil during 11 days of incubation.

Soil photolysis

The photolysis of [phenyl-¹⁴C] mesotrione and [cyclohexane-¹⁴C] mesotrione was studied silt loam soil (Table 31) (Van Nest *et al.*, 1996; RR 96-060B). Mesotrione was applied at a nominal rate of 0.30 kg ai/ha, the soil incubated for 31 to 36 days in local sunlight (latitude 37° 56') at 20 to 24 °C. Dark control plates were maintained at 20 to 24 °C. Photolysis of mesotrione was studied under experimental conditions specified by EPA and EEC guidelines. In the EPA protocol, the soil disks were air-dried overnight before the treatment, whereas in the EEC conditions, they were further oven-dried at 35 °C for 7 hours prior to treatment. One day of xenon irradiation corresponded to 2.42 days of irradiation by sunlight in the phenyl EPA study, 2.17 days in the phenyl EEC study and 2.20 days in the cyclohexane study.

The percentage of mesotrione recovered at each sampling interval is shown in Tables 32 and 43. Over 90% AR was recovered immediately after application from the photolysed samples and 88.6% from the control samples. In general, CO₂ was the major photodegradation product from the irradiated samples of [phenyl-¹⁴C]- and [cyclohexane-¹⁴C] mesotrione. The total ¹⁴CO₂ produced after 31–36 sunlight equivalent days of photolysis was approximately 12% AR for the [phenyl-¹⁴C] mesotrione and 32% AR for the [cyclohexane-¹⁴C] mesotrione. MNBA and AMBA residues accounted for less than 12% AR each. The calculated half-lives of [phenyl-¹⁴C] mesotrione (EEC study), [cyclohexane-¹⁴C] mesotrione and [phenyl-¹⁴C] mesotrione (EPA study) were 15, 19 and 21 days, respectively, with a mean of 18.5 ± 3.6 days (equivalent to 20.3 days at 50°N; Eya, 1997).

Table 32 Mesotrione soil photolysis treated with [phenyl-¹⁴C] and [cyclohexyl-¹⁴C] mesotrione (EEC guideline; Van Nest *et al.*, 1996; RR 96-060B), in % of applied ¹⁴C in soil extracts

		Equivalent days of summer sunlight at latitude 37° 56' North [phenyl- ¹⁴ C] / [cyclohexyl- ¹⁴ C]						
		0 / 0	4.7 / 4.5	13 / 13	17 / 18	21 / 22	28 / 29	33 / 31
% of applied ¹⁴ C, [phenyl/ [cyclohexyl]	Mesotrione ^a	94.4 / 95.5	71.5 / 68.8	46.5 / 47.6	42.1 / 41.2	39.2 / 72.8	33.3 / 46.8	32.4 / 54.3
	MNBA	0.4	7.6	11.5	10	8.7	9	8.5
	AMBA	0.4	3.4	6.6	7.5	6.7	7.8	7.3
	¹⁴ CO ₂	—	1.78 / 14.9	6.23 / 25.4	8.33 / 28.3	9.74 / 29.9	11.2 / 31.4	11.8 / 31.8

^a Mean of replicate analysis

Table 33 Mesotrione silt loam soil photolysis treated with [phenyl-¹⁴C] mesotrione (EPA protocol, Van Nest *et al.*, 1996; RR 96-060B), in % of applied ¹⁴C

	Equivalent days of summer sunlight at latitude 37° 56' North									
	0.0	4.9	12	19	36	0.0 D	2.0 D	8.0 D	14 D	30 D
Mesotrione ^a	92.4	70.8	52.2	44.3	41.0	88.6	90.1	88.7	87.0	84.5
MNBA	0.1	5.4	6.8	7.6	6.5					

	Equivalent days of summer sunlight at latitude 37° 56' North									
	0.0	4.9	12	19	36	0.0 D	2.0 D	8.0 D	14 D	30 D
AMBA	0.3	2.4	5.2	6.0	5.5					
¹⁴ CO ₂			12.4	14.4	14.4					

^a Mean of replicate analysis

D=dark control. Sunlight equivalent days calculated for dark control assuming 24 h of darkness equivalent to 2 days of photolysis

Aerobic and anaerobic degradation in soil

The metabolism of [phenyl-2-¹⁴C]-mesotrione (Subba-Rao, 1996; RR 95-082B) and [cyclohexane-2-¹⁴C]-mesotrione (Vispetto & Tovshteyn, 1997; RR 95-047B) was investigated in a silt loam soil under aerobic conditions at a rate equivalent to 0.313 and 0.348 kg ai/ha, respectively. The treated soil was incubated in the dark at 25 ± 1 °C and the moisture content was maintained at 20.6% for 58–63 days under conditions which allowed ¹⁴CO₂ and any other volatilised degradates to be trapped. In addition, the metabolism of [phenyl-¹⁴C] mesotrione was further investigated at a rate of 0.233 kg ai/ha in a soil incubated in the dark at 20 ± 2 °C and 23.6% moisture content (Miller, 1997; RR 97-033B). ¹⁴CO₂ was collected in NaOH traps over the course of the study. Polyurethane foam plugs inserted in the sidearm of the flasks were analysed for any other volatile radioactive degradates. Soil was extracted with 0.05 M NH₄OH and acetone or acetonitrile. Unextracted radioactivity was further extracted from soil with 0.5 N NaOH or 0.1 N NaOH and microwave extraction.

Average recoveries ranged from 89.8 to 111% AR in the three studies. The percentages of mesotrione and resultant degradates recovered at each sampling interval are presented in Tables 34–36. Levels of radioactivity bound to the soil (unextracted) in the cyclohexane study ranged from 1.5 to 15.2% AR, with no correlation with incubation time; CO₂ accounted for 75% AR by the end of the study (Table 34). In both phenyl- labelled studies (Table 35 and 36), bound residues in soil (unextracted with 0.5 M NaOH) increased throughout the incubation period, when accounted for 34.1 to 37.0% AR. DT₅₀ and DT₉₀ values for the three studies calculated using first order kinetic ranged from 12 to 14 days and from 45 to 54 days, respectively.

Table 34 Mesotrione and degradates under aerobic conditions in silt loam soil treated with [cyclohexane-¹⁴C] mesotrione at 0.313 kg/ai/ha, in % AR

Distribution of residues	Incubation period, days									
	0	1	3	6	9	13	15	21	30	58
Mesotrione	91.0	86.7	73.6	61.5	55.7	47.7	39.5	29.8	19.6	5.67
Reminder—NH ₄ OH/acetone ^a	2.40	4.30	4.10	4.40	1.10	0.00	6.40	0.70	1.6	1.17
Unidentified ^b	— ^e	— ^e	9.07	— ^e	— ^e	9.84	— ^e	— ^e	11.4	8.59
Reminder—0.1 N NaOH ^c	— ^e	— ^e	0.83	— ^e	— ^e	1.63	— ^e	— ^e	— ^e	0.71
¹⁴ CO ₂	0.04	2.08	7.83	17.1	25.4	34.6	38.9	50.1	60.3	75.2
Trapped volatiles ^d	0.00	0.00	0.27	0.05	0.45	0.00	0.00	0.00	0.00	0.02
Unextracted	2.70 ^d	3.00 ^d	1.50	6.80 ^d	11.2 ^d	4.43	12.5 ^d	15.2 ^d	3.63	3.10
Total Recovered	96.1	96.1	96.4	89.8	93.9	96.6	97.3	95.8	96.5	93.8

^a Radioactivity remaining in the NH₄OH/acetone extracts after accounting for parent from the total via reverse-phase HPLC quantification of the residue

^b Unidentified; represents two metabolites extracted from 0.1 N NaOH microwave extractions of soil which had been previously extracted with NH₄OH, neither metabolite exceeded 10% of the applied radioactivity

^c Radioactivity remaining in the 0.1 N NaOH extract after accounting for the unidentified metabolites

^d Average of two flasks at each interval

^e NaOH extractions were not completed on these sampling points

MNBA reached a maximum level of 7.6% of the [phenyl-¹⁴C] applied radioactivity after six days and declined rapidly, with less than 1% remaining after 63 days (Table 35). The calculated DT₅₀ for MNBA in the soil was 1.1 days (Subba-Rao, 1996, RR 95-082B).

Table 35 Mesotrione degradates under aerobic conditions silt loam soil treated with [phenyl- ^{14}C]-mesotrione at 0.348 kg ai/ha, in % AR

Distribution of residues	Incubation period. Days									
	0	1	3	6	9	13	16	23	30	63
Mesotrione	97.9	96.4	89.1	74.4	57.4	46.3	35.5	27.2	19.1	6.5
MNBA	0.0	4.1	6.8	7.6	5.7	5.5	4.1	1.8	1.8	0.7
AMBA	0.0	2.2	3.7	5.5	7.4	8.7	9.4	9.7	7.3	4.5
$^{14}\text{CO}_2$	0.0	0.1	0.8	0.2	5.0	7.4	10.9	15.7	17.8	27.5
NaOH extract ^a	6.1	5.8	6.5	8.9	14.6	13.7	15.2	16.2	23.7	21.1
Unextracted	1.6	2.3	4.4	9.8	8.5	16.4	22.8	24.8	26.9	34.1
Total Recovered	106	111	111	106	98	98	98	95	97	94

^a The radioactivity not extracted by ammonium hydroxide and acetone was further extracted from soils with 0.5 N NaOH. The alkali extracts humic and fulvic acid components of soil organic matter. This is the overall total for the NaOH fraction before additional clean-up and analysis steps

In the study conducted by Miller (1997; RR 97-033B).MNBA reached a maximum level of 5.8% of the [phenyl- ^{14}C] applied radioactivity after 10 days and declined rapidly to non-detectable levels at 56 days (Table 36). In this study the DT₅₀ of MNBA, calculated using a first order metabolite model was 0.7 day.

Table 36 Mesotrione degradates under aerobic conditions silt loam soil treated with [phenyl- ^{14}C]-mesotrione at 0.233 kg ai/ha, in % AR

Distribution of residues	Incubation period. Days							
	0	3	7	10	14	21	28	56
Mesotrione	94.6	82.4	71.2	53.1	48.5	33.4	24.2	11.9
MNBA	1.4	4.5	5.0	5.8	5.1	1.0	0.9	nd
AMBA	nd	2.3	1.8	3.2	3.0	2.3	4.9	7.9
$^{14}\text{CO}_2$	na	1.1	3.1	4.9	6.9	10.9	14.7	24.5
Unidentified	nd	nd	nd	7.0	3.2	7.5	2.5	8.1
Water soluble radioactivity	na	1.7	2.9	3.9	4.0	3.5	4.0	6.1
Reminder ^a	3.0	0.7	1.0	1.1	1.3	1.7	1.8	2.0
Unextracted	0.8	5.9	11.7	16.0	22.7	33.0	37.6	37.0
Total Recovered	99.8	98.6	96.8	95	94.6	93.1	90.6	97.5

nd=not detected

na=not applicable

^a Radioactivity associated with the flocculate which precipitated after acidification of the 0.05 M NH_4OH extract

In two studies to investigate the degradation of mesotrione under anaerobic conditions, [cyclohexane-2- ^{14}C] (Vispetto & Tovshteyn, 1996; RR 95-048B) and [phenyl- ^{14}C]-mesotrione (Carley, 1996; RR 96-033B) were applied to a silt loam soil at 0.280 and 0.340 kg ai/ha, respectively. The soil was incubated in the dark at $25 \pm 2^\circ\text{C}$ and anaerobic conditions were produced by flooding the soil under nitrogen for 30 days prior to treatment.

Average total recoveries ranged from 86.2 to 105% AR throughout the incubation period, with the exception of day 59 of the phenyl-labelled experiment (Tables 36 and 37). Using the [cyclohexane-2- ^{14}C]-mesotrione, the amount of unextracted radioactivity increased to a maximum of 23.4% AR after 30 days of incubation (Table 36), while it reached 17% AR after 59 days of incubation using [phenyl- ^{14}C]-mesotrione (Table 37). AMBA was found to be the only degradation product (Table 37).

Table 36 Mesotrione and degradates under anaerobic conditions in silt loam soil treated with [cyclohexyl-2- ^{14}C]-mesotrione, in % AR

Distribution of residues	Incubation period. Days					
	0	1	3	7	14	30
Mesotrione (s + w)	102	77.6	74.5	39.4	9.32	< 0.01
$^{14}\text{CO}_2$	0.05	0.09	0.48	1.96	4.83	16.1
Trapped volatiles	0.01	0.00	0.00	0.04	0.93	0.00
Unidentified ^a (s + w)	—	—	4.48	8.19	9.18	10.4

Distribution of residues	Incubation period. Days					
	0	1	3	7	14	30
Remaining ^{14}C from microwave hydrolysis	1.13	3.21	3.97	3.91	9.12	8.0
0.1 N NaOH precipitate (fulvic and humic acids)	0.47	1.57	3.95	7.90	11.4	12.2
Reminder (s + w)	0.21	11.4	4.95	14.4	28.5	16.2
Unextracted (soil after base and microwave)	1.13	4.43	8.70	17.7	14.3	23.4
Total Recovered	105	98.3	101	93.5	87.6	86.3

(s)=Detected in soil extracts

(w)=Detected in flood water

^a Up to two compounds detected at any sampling interval, with no individual compound exceeding 9.7%Table 37 Mesotrione and degradates under anaerobic conditions in silt loam soil treated with [phenyl- ^{14}C]-mesotrione, in % AR

Distribution of residues	Incubation period. Days						
	0	1	3	7	14	30	59
Mesotrione (w)	90	80	47	18	2.7	0	0
Mesotrione (s)	0	3.2	16	18	3.5	0	0
AMBA (w)	0	0	1.5	1.8	9.2	2.7	7.9
AMBA (s)	0	0	1.8	15	28	38	14
$^{14}\text{CO}_2$	< 0.1	< 0.5	< 0.1	< 0.1	< 0.1	< 0.3	< 0.5
Reminder ^a (w)	2.5	6.4	5.1	7.9	8.1	5.9	4.9
Reminder ^a (s)	0	2.6	3.9	18	32.9	24.8	12.4
Unextracted	1.5	5.1	11.7	7.4	8.4	14.5	17
Total Recovered	93.7	97.1	87.6	86.3	92.8	86.2	56

(s)=Detected in soil extracts

(w)=Detected in flood water

^a Unresolved and background radioactivity in chromatograms

Table 38 summarizes the degradation rates of mesotrione in silt loam soil estimated under aerobic and anaerobic conditions.

Table 38 Mesotrione DT₅₀ and DT₉₀ in silt loam soil under aerobic and anaerobic conditions

Labels (ref)	Conditions	DT ₅₀ (days)	DT ₉₀ (days)
[Cyclohexane- ^{14}C] mesotrione (Vispetto & Tovshteyn, 1995)	Aerobic	14	45
[Phenyl- ^{14}C] mesotrione (Subba-Rao, 1996)	Aerobic	12	54
[Phenyl- ^{14}C] mesotrione (Miller, 1997)	Aerobic	14	47
[Cyclohexane- ^{14}C] mesotrione (Vispetto & Tovshteyn, 1996)	Anaerobic	4.1	14
[Phenyl- ^{14}C] mesotrione (Carley, 1996)	Anaerobic	3.6	12

The rate of aerobic degradation of mesotrione was studied in sandy loam, loam and clay loam soils with the characteristics summarised in Table 39 (Miller & Wilson, 1997; RR 96-090B). with [phenyl- ^{14}C]-mesotrione applied at a nominal rate of 0.165 kg ai/ha and the soils incubated for up to 56 days in the dark at 20 ± 2 °C. Moisture content was 50% of the maximum holding capacity and under conditions which allowed $^{14}\text{CO}_2$ to be trapped.

Table 39 Soils used to investigate mesotrione rate of degradation under aerobic laboratory conditions

Soil Property	ERTC ^a	GARRONNE ^b	Pickett Piece ^c
% Sand	73.2	43.5	41.3
% Silt	19.2	34.9	25.5
% Clay	7.6	21.6	33.2
% Organic Matter	0.98	1.46	5.70
pH	6.4	7.7	7.1
Cation exchange capacity (meq/100 g)	2.44	8.60	22.90
% Moisture holding capacity (zero saturation)	23.2	44.9	60.5
% Moisture holding capacity (1/3 Bar)	13.1	23.6	32.6
Moisture holding capacity (15 Bar)	—	—	—

Soil Property	ERTC ^a	GARRONNE ^b	Pickett Piece ^c
Microbial biomass. mg C/kg (zero time)	160	180	604
Microbial biomass. mg C/kg (end of incubation)	109	171	667
Soil classification	silt loam	Loam	clay loam

^a Zeneca Eastern Research Technical Centre (ERTC), North Carolina, USA

^b Zeneca Toulouse Field Station, Garomie field, Grisolles, France

^c Pickett Piece, Oxfordshire, England

Degradation of mesotrione in sandy soils was slower, with a DT₅₀ of 12 days. In loam and clay soils the DT₅₀ were 5.9 and 4.5 days, respectively Table 40.

Table 40 Mesotrione degradation rate (Miller & Wilson, 1997) in % AR

Sampling Interval (days)	Residue of mesotrione detected (% of applied ¹⁴ C)		
	Sandy loam	Loam	Clay loam
0	99.9	96.8	102
3	80.0	63.3	73.7
7	42.1	22.3	35.5
10	50.1	16.6	31.8
14	28.4	16.1	18.0
21	39.8	17.2	3.7
28	29.9	1.8	na
56	2.5	na	na
DT ₅₀ (days) mesotrione	12	5.9	4.5
DT ₉₀ (days) mesotrione	39	20	15
DT ₅₀ (days) MNBA	1.3	1.7	-

Na: not analysed as DT₉₀ already achieved

[Phenyl-U-¹⁴C]-mesotrione was applied at rates ranging from 0.60 to 0.85 mg/kg soil, equivalent to approximately 0.600 to 0.850 kg ai/ha, to 13 soils (characteristics summarised in Table 41) (Tarr, 1997, RR 93-092B). The soils were incubated in the dark, under aerobic conditions at 25 ± 2 °C, maintaining a moisture content of 100% of 1/3 bar moisture capacity and evolved ¹⁴CO₂ was trapped. The soils were incubated for up to 28 days, and samples were extracted with 0.05 M NH₄OH and acetone, acidified and portioned with ethyl acetate.

Table 41 Soils used to investigate mesotrione rate of aerobic degradation

	Clay loam	Silt loam			Loam	Loamy sand	loam sandy	Clay loam		Silty clay loam				
Soil Property	721	729	723	725	724	728	727	730	722	731	732	741	742	
% Sand	26.2	18.9	7.2	35.0	86.6	52.2	29.0	19.3	5.8	10.8	8.2	13.8	12.0	
% Silt	36.8	58.7	69.5	37.7	9.2	29.5	42.8	48.5	63.0	57.0	57.4	52.6	48.8	
% Clay	36.9	22.4	23.3	27.3	4.2	18.3	28.2	32.2	31.2	32.3	34.3	33.7	39.2	
% Organic Matter	5.4	1.9	2.1	2.3	1.0	1.6	2.5	1.7	3.2	2.0	5.2	3.2	4.5	
pH	5.8	5.6	5.8	6.2	5.0	6.4	5.6	6.1	6.0	7.2	5.7	5.5	§	
Cation exchange capacity (meq/100 g)	26.2	9.4	8.3	11.9	4.0	6.2	13.9	10.9	16.4	18.2	20.5	16.1	25.5	
% Moisture holding capacity (1/3 Bar/15 Bar)	35.0/16.9	28.0/7.3	31.2/6.4	24.2/8.8	5.2/2.4	19.5/5.4	27.0/10.2	27.4/10.4	34.8/12.6	31.9/13.2	36.8/13.9	28.6/13.2	34.4/13.9	

Average total recoveries immediately after treatment ranged from 86.4 to 92.7% of the applied radioactivity and decreased below 90% for six of the soils after 14 days of incubation (Tarr, 1997). Mesotrione degraded rapidly with DT₅₀ and DT₉₀ values ranging from 8–32 days and 27–105 days, respectively (Table 42). In most soils (10 out of 13), levels of MNBA represented less than 4% AR at any sampling day (DT₅₀ < 2 days).

Table 42 Mesotrione degradation rate (aerobic), in % AR

	721	729	723	725	724	728	727	730	722	731	732	741	742
DT ₅₀ (days)	22	13	17	8	26	8.5	24	19	11	14	16	32	8.2
DT ₉₀ (days)	73	43	55	27	86	28	80	63	35	48	53	105	27

The aerobic degradation of [phenyl-U-¹⁴C]-mesotrione was studied in subsamples of two soils from Michigan (Table 43) treated at a rate of about 1/10 the rate of application to surface soils, or 0.03 mg/kg (Tarr & Tovshteyn, 1997; RR 97-053B). The soils were incubated for up to 42 days in the dark at 20 ± 1 °C, maintaining moisture contents of 100% to 600% of 1/3 bar moisture capacity (about 5 to 10% of the dry soil weight). The higher percentage moisture content was necessary because the soil was very sandy and appeared too dry at < 100% of 1/3 bar. Mesotrione was degraded in soils collected from all depths, at a slower rate in subsoil collected from 1.8–4.8 m below the surface (Table 43).

Table 43 Properties of soils from two locations in Michigan, the USA

	Three Rivers				White Pigeon			
Soil	Loamy sand	Sand	Sand	Sand	Sand	Sand	Sand	Sand
Depth (meters)	0–0.15	1.2–1.8	1.8–2.4	4.2–4.8	0–0.15	1.2–1.8	1.8–2.4	4.2–4.8
Acidity (pH)	6.8	6.6	7.5	8.6	6.4	6.6	7.4	8.4
WHC at Saturation (%)	12.8	12.2	22.2	14.0	14.2	28.8	22.1	18.3
WHC at 1/3 bar (%)	8.1	4.2	3.3	1.5	5.0	4.0	2.6	1.7
WHC at 15 bars (%)	3.6	2.5	1.8	0.8	3.4	2.2	1.3	0.7
Organic Matter (%)	1.4	0.4	0.2	0.1	1.3	0.5	0.2	0.2
CEC (meq/100 g)	6.9	3.0	2.2	1.3	3.6	2.5	2.1	1.2
Sand (%)	79.1	91.9	93.7	96.3	89.5	93.3	96.3	96.8
Silt (%)	12.1	3.4	3.6	1.0	3.8	2.0	0.9	1.3
Clay (%)	8.8	4.8	2.8	2.8	6.8	4.8	2.8	2.0
Dry Bulk Density (g/cm ³)	1.35	1.34	1.43	1.66	1.45	1.46	1.52	1.65
DT ₅₀ (aerobic)	6	24	105	63	13	12	68	98

Aerobic soil degradation of MNBA

One study was conducted with the purpose to determine the rate of degradation of MNBA in four soils collected from agricultural fields in England and the United States (Lay & Peyton, 2000; RR 99-098B). MNBA was applied to the incubated soil at 0.22 µg/g soil (equivalent to 0.220 kg ai/ha). Immediately after treatment of the soil, eight soil samples were taken for analysis and the remaining soil samples were incubated in the dark under aerobic conditions at 20 °C±2 °C. Samples were collected 0.3, 1, 2, 3, 4, 7, 14, 21, 45 and 60 days after treatment, and extracted with 0.05 N NH₄OH. The MNBA in the extract was chemically reduced to AMBA, which was analysed by a reversed-phase HPLC system using fluorescence detection. DT₅₀s ranged from 0.6 days to 10.6 days (Table 44).

Table 44 DT₅₀ and DT₉₀ values for MNBA in soils under laboratory aerobic conditions

Soil Origin	Spinks Soil (Three Rivers, Michigan, USA)	Delavan Soil (Wisconsin, USA)	Wisborough Green Soil (West Sussex, England)	East Anglia Soil (Suffolk, England)
	Loamy Sand	Silt Loam	Loam	Sand
pH	6.7	6	5.5	7.9
% Organic Matter	1.4	2.4	5.1	2.2
DT ₅₀ . days	8.6	10.1	0.6	10.6
DT ₉₀ . days	28.5	33.6	2.0	35.1

Aerobic soil degradation of AMBA

One study was conducted with the purpose to determine the rate of degradation of MNBA in four soils collected from agricultural fields in England and the United States (Lay & Peyton, 2000; RR 99-

098B). MNBA was applied to the incubated soil at 0.22 µg/g soil (equivalent to 0.220 kg ai/ha). Immediately after treatment of the soil, 8 soil samples were taken for analysis and the remaining soil samples were incubated in the dark under aerobic conditions at 20 °C±2°C. Samples were collected 0.3, 1, 2, 3, 4, 7, 14, 21, 45 and 60 days after treatment, and extracted with 0.05 N NH₄OH. The MNBA in the extract was chemically reduced to AMBA, which was analysed by a reversed-phase HPLC system using fluorescence detection. DT₅₀'s ranged from 0.6 days to 10.6 days (Table 45).

Table 45 DT₅₀ and DT₉₀ values for MNBA in soils under laboratory aerobic conditions

Soil Origin	Spinks Soil (Three Rivers, Michigan, USA)	Delavan Soil (Wisconsin, USA)	Wisborough Green Soil (West Sussex, England)	East Anglia Soil (Suffolk, England)
	Loamy Sand	Silt Loam	Loam	Sand
pH	6.7	6	5.5	7.9
% Organic Matter	1.4	2.4	5.1	2.2
DT ₅₀ . days	8.6	10.1	0.6	10.6
DT ₉₀ . days	28.5	33.6	2.0	35.1

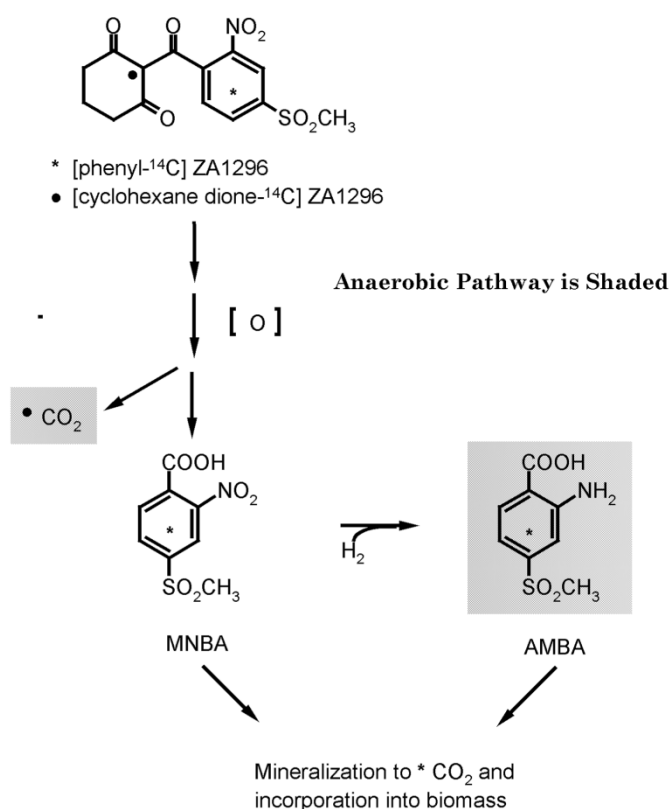


Figure 3 Proposed metabolism of mesotrione in soil

Water sediment study

Mesotrione labelled in either the cyclohexane or phenyl rings was applied to two water sediment systems at a rate equivalent to 0.200 kg ai/ha, evenly distributed throughout a 30 cm deep water body (Cary, 1999; RR 96 033B). The physico-chemical characteristics of the systems are shown in Table 48. The systems were incubated up to 101 days in the dark at 20 °C.

Radioactivity recovered from the water/sediment systems immediately after application ranged from 93% and 102% of the applied dose. The mean recovery from all the samples taken throughout the remainder of the 101 days incubation period was 100% AR. DT₅₀ were 3–6 days (Table 46). The level of mesotrione in the sediment did not exceed 4% AR at any time point, and DT₅₀ and DT₉₀ values in the water-sediment were equivalent to the ones in the water phase.

Table 46 Physico-chemical characteristics of the sediments and water prior to the study start

Sediment			Water		
Property	Old Basing	Virginia Water	Properties	Old Basing	Virginia Water
Soil classification	Sandy loam	Sand	Dissolved oxygen	82	77
Sand (%)	57	96	pH (at collection)	7.2	6.8
Silt (%)	26	2	Suspended solids mg/L	72.0	122
Clay (%)	18	3	Organic carbon, mg/L	64.9	37.1
Organic Matter (%)	7.5	0.5	Total oxidised nitrogen mg/L	5.5	2.0
pH	7.8	7.5	Nitrite mg N/L	< 0.1	< 0.1
Cation exchange capacity, eq/100 g	16.3	1.3	Nitrate mg N/L	5.5	2.0
Phosphorus, mg/kg	19.8	16.9	Ammonia mg N/L	< 0.1	< 0.1
Total nitrogen (%)	0.708	0.033	Orthophosphate mg P/L	< 0.50	< 0.50
			Calcium mg/kg Ca/L	115	37.2
			Magnesium mg/L	1.8	11.4
			Total hardness mg CaCO ₃ /L	294	140
			Conductivity µS/cm	503	379
			Alkalinity	310	52.7
Old Basing	DT ₅₀ : 3 days; DT ₉₀ : 10 days				
Virginia Water	DT ₅₀ : 6 days; DT ₉₀ : 30 days				

[Phenyl-¹⁴C] mesotrione was degraded to MNBA and AMBA (Tables 49 and 50). MNBA was only detected in the Virginia Water system. It reached a maximum of 8% AR on Day 3 and was almost entirely present in the water phase. AMBA was distributed in both the water and the sediment and reached up to 18% at Day 28 in the ‘Old Basing’ system and up to 19% in ‘Virginia Water’ system at Day 14.

Table 47 Distribution of radioactivity in ‘Old Basing’ sediment treated with [Phenyl-¹⁴C] mesotrione, in % of applied radioactivity

		Days after treatment									
		0	3	6	10	14	28	42	56	69	101
	¹⁴ CO ₂	nd	0.0	0.0	0.0	0.0	0.4	1.0	1.8	2.9	5.5
Surface Water	Mesotrione	73.2	39.2	16.6	9.9	1.4	0.0	0.0	0.0	0.0	0.0
	MNBA	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	AMBA	0.0	2.6	5.0	7.2	7.1	9.6	9.2	6.6	8.1	7.1
	Baseline	8.3	7.9	5.9	7.4	6.5	3.6	4.5	3.7	3.6	3.9
	Others	6.0	6.2	11.3	12.8	7.1	3.2	4.4	3.3	2.1	2.3
Sediment	Mesotrione	nd	0.5	0.9	0.9	0.3	0.0	0.0	0.0	0.0	0.0
	MNBA	nd	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	AMBA	nd	2.0	4.7	5.2	5.8	7.9	6.1	7.9	7.0	6.6
	Baseline	nd	1.9	3.2	2.8	4.0	3.2	3.4	2.9	3.6	4.0
	Others	2.9	6.6	6.9	7.6	8.0	7.4	5.2	6.4	6.8	6.0
	Unextracted	2.3	21.3	48.1	55.5	64.7	70.8	67.3	73.8	75.0	73.7
Surface Water + Sediment	Mesotrione	73.2	39.6	17.6	10.8	1.7	0.0	0.0	0.0	0.0	0.0
	MNBA	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	AMBA	0.0	4.5	9.7	12.4	12.9	17.5	15.3	14.5	15.1	13.8
	Unidentified ^a	17.3	22.6	27.3	30.6	25.7	17.4	17.5	16.3	16.1	16.1
	Unextracted	2.3	21.3	48.1	55.5	64.7	70.8	67.3	73.8	75.0	73.7
Total		92.7	88.1	102.7	109.3	105.0	106.1	101.1	106.4	109.1	109.1

^a Unidentified is Baseline+ Others (from TLC of extracts). 0= < 0.05%

nd=Not determined

Table 48 Distribution of radioactivity in 'Virginia Water' sediment treated with [Phenyl-¹⁴C] mesotrione, in % of applied radioactivity

		Days after treatment									
		0	3	6	10	14	28	42	56	69	101
¹⁴ CO ₂		nd	0.0	0.0	0.0	0.1	2.6	6.6	9.9	12.1	15.6
Surface Water	Mesotrione	71.2	53.5	41.0	34.8	10.7	3.0	0.5	0.0	Nd	nd
	MNBA	0.0	7.4	4.9	0.8	1.3	2.5	0.9	0.0	Nd	nd
	AMBA	0.0	2.5	4.8	3.0	11.5	9.0	2.9	0.8	Nd	nd
	Baseline	11.4	12.3	7.7	8.5	8.8	4.9	2.9	2.4	Nd	nd
	Others	7.8	6.2	11.6	8.1	8.3	6.4	3.8	2.2	4.3	2.2
Sediment	Mesotrione	nd	3.8	1.3	1.9	0.3	0.0	0.0	0.0	0.0	0.0
	MNBA	nd	0.6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	AMBA	nd	1.2	2.7	6.4	7.7	6.1	6.2	3.0	4.3	3.1
	Baseline	nd	2.5	4.6	9.2	11.5	10.4	11.3	12.5	12.0	12.1
	Others	5.3	4.4	7.7	9.0	12.6	12.6	11.5	14.7	13.0	9.9
	Unextracted	0.2	3.7	12.4	20.0	27.5	42.1	47.7	56.5	54.8	64.5
Surface Water.+ Sediment	Mesotrione	71.2	57.3	42.2	36.6	10.9	3.0	0.5	0.0	0.0	0.0
	MNBA	0.0	7.9	4.9	0.8	1.3	2.5	0.9	0.0	0.0	0.0
	AMBA	0.0	3.7	7.5	9.4	19.2	15.1	9.1	3.8	4.3	3.1
	Unidentified ^a	24.4	25.4	31.6	34.8	41.2	34.3	29.6	31.8	29.4	24.2
	Unextracted	0.2	3.7	12.4	20.0	27.5	42.1	47.7	56.5	54.8	64.5
Total		95.8	98.0	98.6	101.7	100.3	99.6	94.3	102.0	100.6	107.4

^a Unidentified is Baseline+ Others (from TLC of extracts)

0.0= < 0.05%

nd=Not determined

[Cyclohexane-¹⁴C] mesotrione degraded into CO₂, which accounted for approximately 27% of the applied radioactivity at Day 101 in both the 'Virginia Water' and 'Old Basing' systems respectively (Tables 49–50).

Table 49 Distribution of radioactivity in 'Old Basing' sediment treated with [Cyclohexane-¹⁴C] mesotrione, in % of applied radioactivity

		Days after treatment									
		0	3	6	10	14	28	42	56	69	101
¹⁴ CO ₂		nd	0.1	0.2	0.9	3.0	12.5	18.5	22.1	24.7	27.8
Surface Water	Mesotrione	86.0	74.7	31.7	16.3	12.7	0.0	0.4	nd	Nd	nd
	Baseline	8.7	10.4	8.1	8.7	8.2	4.6	2.6	nd	Nd	nd
	Others	5.6	8.2	17.6	14.0	12.4	5.1	1.6	3.8	3.0	2.5
Sediment	Mesotrione	nd	nd	0.0	0.0	0.0	0.0	0.0	nd	Nd	nd
	Baseline	nd	nd	2.5	2.9	3.6	2.8	2.7	2.4	Nd	nd
	Others	0.8	1.8	8.2	9.5	11.2	8.8	3.4	7.0	8.8	7.6
	Unextracted	0.9	3.2	28.8	39.2	43.8	57.6	58.4	60.6	58.0	63.8
Surface water + Sediment	Mesotrione	86.0	74.7	31.7	16.3	12.7	0.0	0.4	nd	Nd	nd
	Unidentified ^b	15.1	20.4	36.4	35.1	35.5	21.3	10.3	13.2	11.8	10.1
	Unextracted	0.9	3.2	28.8	39.2	43.8	57.6	58.4	60.6	58.0	63.8
Total		102.0	98.4	97.1	91.5	95.0	91.4	87.6	95.9	94.5	101.7

^a Any summation differences in values within the table result from rounding of numbers within individual calculations^b Unidentified is Baseline + Others (from TLC of extracts)

0.0= < 0.05%

nd=Not determined

Table 50 Distribution of radioactivity in 'Virginia Water' sediment treated with [Cyclohexane-¹⁴C] mesotrione, in % of applied radioactivity

		Days after treatment									
		0	3	6	10	14	28	42	56	69	101
¹⁴ CO ₂		nd	0.3	0.7	2.1	3.6	8.6	16.5	21.0	23.6	26.8
Surface	Mesotrione	80.0	na	59.7	26.4	33.0	4.8	0.8	0.0	nd	nd

		Days after treatment									
		0	3	6	10	14	28	42	56	69	101
Water	Baseline	9.5	na	9.1	6.0	7.0	2.1	2.4	0.0	nd	nd
	Others	6.8	na	18.4	10.5	7.6	4.1	3.6	4.4	4.2	4.8
Sediment	Mesotrione	nd	nd	1.4	0.7	1.9	0.0	0.0	0.0	0.0	0.0
	Baseline	nd	nd	3.3	11.5	9.0	9.6	14.9	12.0	8.7	8.2
	Others	3.1	1.1	5.4	17.1	18.3	20.3	20.4	17.2	16.9	14.7
	Unextracted	0.1	0.4	5.7	24.6	19.8	48.4	42.9	48.0	43.9	44.7
Surface water + Sediment	Mesotrione	80.0	na	61.2	27.1	34.9	4.8	0.8	0.0	0.0	0.0
	Unidentified ^a	19.5	na	36.2	45.1	41.8	36.1	41.3	33.6	29.8	27.7
	Unextracted	0.1	na	5.7	24.6	19.8	48.4	42.4	48.0	43.9	44.7
Total		99.6	103.0	103.8	98.9	100.1	97.4	101.0	102.6	97.3	99.2

^a Unidentified is Baseline+ Others (from TLC of extracts)

0.0= < 0.05%

nd=Not determined

na=Not available

Degradation in soil-field studies

Six field studies were conducted to evaluate the rate of degradation of mesotrione in soil. A single application of a 0.100 kg ai/L suspension concentrate of mesotrione was applied to bare soil at rates of 0.150 and 0.200 g ai/ha in 1995/96 and 1996/97 seasons, respectively (Table 51). Soil samples were collected up to 30 cm depth, prepared in 10 cm horizons and analysed for mesotrione, MNBA and AMBA. Calculated DT₅₀ and DT₉₀ for mesotrione are also shown in Table 51.

In the 1996-1997 Italian trial, MNBA was detected at up to 0.031 mg/kg in 0–10 cm horizon, six days after application and declined to < 0.005 mg/kg by 89 days after treatment. AMBA was detected only in the 0-10 cm horizon six days after application (0.006 mg/kg). In one 1996–1997 German trial, MNBA reached a maximum of 0.016 mg/kg in the 0–10 cm horizon eight days after application and decreased to <0.005 mg/kg 89 days after treatment. No measurable residues of mesotrione or metabolites were detected in the soil below 10 cm.

Table 51 Field dissipation of mesotrione in Europe

Year	Location	Application Rate (kg ai/ha)	Soil Type	Soil Property		DT ₅₀	DT ₉₀	Reference
				Organic Carbon (%)	pH			
1995-1996	France (south)	0.150	0 - 10 cm (clay loam)	2.1	6.0	7	73	Graham <i>et al.</i> , 1997a
			10 - 20 cm (clay loam)	1.7	6.3			
			20 - 30 cm (clay)	0.4	7.1			
	Italy	0.150	0 - 10 cm (clay loam)	2.7	6.1	7	36	Graham <i>et al.</i> , 1997b
			10 - 20 cm (silt loam)	1.9	6.6			
			20 - 30 cm (silt clay)	0.4	7.1			
1996-1997	Germany	0.150	0 - 10 cm (sandy clay loam)	2.6	6.2	3	59	Graham <i>et al.</i> , 1997c
			10 - 20 cm (sandy clay)	0.7	6.5			
			20 - 30 cm (sandy loam)	0.4	7.2			
	Italy	0.200	0 - 10 cm (sandy loam)	0.8	8.0	8	26	Graham <i>et al.</i> , 1998a
			10 - 20 cm (sandy loam)	0.6	8.3			
			20 - 30 cm (loamy sand)	0.2	8.5			
	Germany 1	0.200	0 - 10 cm (loam)	2.5	7.0	8	26	Graham <i>et al.</i> , 1997b
			10 - 20 cm (silt loam)	1.6	7.4			
			20 - 30 cm (silt loam)	1.0	7.6			
	Germany 2	0.200	0 - 10 cm (sandy clay loam)	2.8	6.9	2	21	Wiebe, 1999
			10 - 20 cm (sandy loam)	1.6	7.1			
			20 - 30 cm (sandy loam)	0.5	7.4			

Aerobic soil degradation of AMBA

In another study, [phenyl-U-14C]-AMBA was applied at 0.213 kg ai/ha (Marth, 1997; RR 97-032B) or 0.225 kg ai/ha (Lay, 2000; RR 99-096B). Soil characteristics are shown in Table 52. The soils were incubated for up to 60 days in the dark under aerobic conditions at a temperature of 20 ± 2°C,

maintaining a moisture content of 40% of water holding capacity. Average total recoveries ranged from 92.7–98.6% AR at Day 0. AMBA DT₅₀ ranged from 2 to 6 days and no AMBA degradates exceeded 10% AR in any of the three soils (Table 52).

Table 52 AMBA rate of degradation in various soils under aerobic conditions

Soil Property	Marth, 1997			Lay, 2000	
	Wisborough Green, England	Delevan, USA	East Anglia, England	Spinks, USA	
	Clay	Silt loam	Sandy loam	Loamy sand	
% organic matter	3.1	2.4	2.46	1.4	
pH	4.9	6.4	7.9	6.7	
Cation exchange capacity, meq/100 g	10.52	12.11	8.47	6.1	
Sampling days	% of AR			Sampling days	% of AR
0	98.6	98.6	98.1	0	92.7
0.1	72.2	82.8	90.8	0.3	84.1
3	40.1	55.2	45.6	1	71.4
7	30.3	45.7	28.4	2	54.1
10	25.4	41.5	19.3	3	43.6
14	19.2	37.4	18.1	4	39.1
21	13.4	26.6	17.1	7	24.5
28	7.8	23.7	15.1	14	13.2
56	7.8	20.2	11.1	21	11.4
				45	14.1
				60	11.8
DT ₅₀ (days)	3	6	2	1.8	
DT ₉₀ (days)	39	>56	63	56	

Confined rotational crops

This study was designed to provide information on the uptake and metabolism of [phenyl-U-¹⁴C]-mesotrione in rotational crops following a single application onto the bare surface of a sandy loam soil at a rate of 0.165 kg ai/ha (Gorder *et al.*, 1997; RR 96-084B). At intervals of 120 and 300 days after treatment (DAT), three representative rotational crops (endive, radish and wheat) were sown into the treated soil. Crops planted in the 120 DAT soils were harvested: endive at 78 days after planting (DAP), radish roots and leaves at 56 DAP, wheat forage at 22 DAP, wheat hay at 57 DAP and wheat grain and straw at 134 DAP. The 300 DAT crops were not harvested due to low residues found in 120 DAT crops. Crops with residues ≥ 0.01 mg/kg were extracted with acetonitrile/water and the extracted residues (> 0.01 mg/kg) were analysed by HPLC-radiodetector.

Analysis of soil cores showed that the residue had declined to 34% of the applied radioactivity (AR) after 120 days and to 15% AR after 300 days. In soil, mesotrione accounted for 0.1% AR after 120, MNBA for 8% and AMBA for 2% AR.

Total radioactive residues in crop commodities from the 120 DAT are shown in Table 53. Residue levels were ≤ 0.035 mg/kg for feed commodities and ≤ 0.014 mg/kg eq. for food commodities. Untreated soil and pots contained low levels of radioactivity (≤ 0.004 mg/kg eq.), resulting from mineralisation of [¹⁴C]mesotrione in the soil. Unextracted residues represented ≤ 0.015 mg/kg eq. and were not further analysed.

Commodities with residues > 0.01 mg/kg eq. were extracted with acetonitrile/water. From 46 to 59% TRRs were extracted. The major metabolite in wheat commodities was MNBA accounting for 36%TRR in forage (Table 53). AMBA was present mostly in conjugated form.

Table 53 Summary of total radioactive residues in rotational crop samples grown in soil treated at 0.165 kg/ai/ha with [phenyl-2-¹⁴C]-mesotrione–120 DAT

Crop	TRR, mg/kg eq.	Extracted, mg/kg (%TRR)	PES, mg/kg (%TRR)	Losses in sample work-up, mg/kg (%TRR)
Wheat forage, 22 DAP	0.031	0.018 (58)	0.006 (21)	0.007 (21)
Wheat hay, 57 DAP	0.019	0.011 (59)	0.007 (36)	0.001 (5)
Wheat straw, 134 DAP	0.035	0.016 (46)	0.015 (43)	0.004 (11)
Wheat grain, 134 DAP	0.006	–	–	–
Endive, 78 DAP	0.014	0.007 (51)	0.007 (51)	(+2)
Radish root, 56 DAP	0.004	–	–	–
Radish leaves, 56 DAP	0.004	–	–	–

Table 54 Summary of total radioactive residues in 120 DAT wheat feed commodities

Metabolite ¹⁾	Forage		Hay		Straw	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
MNBA	0.011	36	0.003	16	0.003	9
AMBA Sulphate conjugate	0.002	6	0.002	11	0.004	10
AMBA Conjugate	0.002	5	–	–	–	–
AMBA	–	–	0.001	5	0.001	3
Others (each < 0.001) ^a	0.004	11	0.005	26	0.009	24

^a All radioactivity that did not form discrete HPLC peaks

In another study, [phenyl-U-¹⁴C]-mesotrione was applied at 0.308 kg ai/ha and 0.462 kg ai/ha, the lower rate representative of the pre-emergence use and the higher rate being representative of a pre-emergent followed by post-emergent use (Gorder *et al.*, 1996; RR 96-035B). Each application was sprayed onto the bare surface of a sandy loam soil contained in 51 cm diameter pots. The pots were sown with combinations of wheat, soy, endive and radish at intervals of 30, 120 and 300 DAT. Leaf and root crops could not be planted at 30 DAT due to crop sensitivity

Samples having radioactive residues greater than or equal to 0.01 mg/kg were extracted 2–3 times with acetonitrile/water. The 30-DAT wheat grain sample was extracted by refluxing with 2 M HCl. Solids with residues > 0.05 mg/kg (> 10% TRR) were subjected to further extraction with 1 M/10M ammonium hydroxide and 6 M HCl at ambient temperature and/or under reflux. HPLC and TLC were used for the characterisation of residues.

Radioactive residues in soil declined to 27% AR at 300 DAT. Mesotrione levels were < 1% AR between 28 and 300 DAT, forming MNBA and to a lesser extent AMBA present at the 120 and 300 day sowing intervals.

Radioactive residues in wheat feed commodities declined with increasing sowing intervals, being highest in wheat straw (2.58 mg/kg eq. at 30 DAT). In wheat grain, it was 0.038 mg/kg at 30 DAT and < 0.02 mg/kg at all other planting intervals. Soya bean residues were 0.145 mg/kg at DAT with feed commodities containing 0.462 and 0.645 mg/kg for hay and forage respectively. Residues in endive and radish root declined to 0.019 and 0.005 mg/kg respectively after 300 DAT (Table 55). Residues in control did not exceed 0.007 mg/kg eq.

Table 55 Total Radioactive Residues (mg/kg eq.) in rotational crop samples grown in soil treated with [phenyl-U-¹⁴C]-mesotrione

Crop	30 DAT (0.308 kg ai/ha)		120 DAT (0.462 kg ai/ha)		300 DAT (0.462 kg ai/ha)	
	Treated	Control	Treated	Control	Treated	Control
Wheat straw	2.58	0.005	0.233	0.003	0.197	0.001
Wheat forage	1.011	0.001	0.303	0.001	0.100	0.000
Wheat hay	0.756	0.003	0.127	0.001	0.044	0.000
Wheat grain	0.038	0.004	0.014	0.004	0.015	0.001

Crop	30 DAT (0.308 kg ai/ha)		120 DAT (0.462 kg ai/ha)		300 DAT (0.462 kg ai/ha)	
	Treated	Control	Treated	Control	Treated	Control
Soy forage	0.645	0.001	Not planted	Not planted	Not planted	Not planted
Soy hay	0.462	0.003	Not planted	Not planted	Not planted	Not planted
Soya bean	0.145	0.007	Not planted	Not planted	Not planted	Not planted
Endive	Not planted	Not planted	0.053	0.000	0.019	0.000
Radish root	Not planted	Not planted	0.037	0.000	0.005	0.005
Radish leaves	Not planted	Not planted	0.048	0.000	0.009	0.000

The major identified metabolite in all wheat samples was MNBA, reaching 0.63 mg/kg in 30 DAT wheat forage (Tables 56 and 57). AMBA was mostly present as sulphate conjugate. Wheat grain samples from 30 DAT contained MNBA at 0.003 mg/kg and an unidentified glucose conjugate (0.013 mg/kg). About 70% TRR of grain samples from 120 and 300 DAT were retained in PES, but no further work was conducted in the samples.

Table 56 Radioactive residues in wheat RACs following treatment of soil with [phenyl- ^{14}C]-mesotrione

Commodity	0.308 kg ai/ha /30 DAT				0.462 kg ai/ha /120 DAT			
	Forage	Hay	Straw	Grain*	Forage	Hay	Straw	Grain
Component	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)
MNBA	0.625 (62)	0.172 (23)	0.458 (18)	0.003 (8)	0.165 (54)	0.023 (18)	0.018 (8)	
AMBA	0.086 (9)	0.083 (11)	0.330 (13)	–	0.019 (6)	0.020 (16)	0.012 (5)	
AMBA sulphate	0.088 (9)	0.131 (17)	0.317 (12)	–	0.028 (9)	0.015 (12)	0.018 (8)	
AMBA conj.	0.036 (4)	0.021 (3)	0.021 (1)	–	0.021 (7)	0.001 (1)	0.002 (1)	
4-OH mesotrione	0.014 (1)	–	–	–	–	–	–	
Mesotrione	0.012 (1)	–	–	–	–	–	–	
Unknown M2	–	0.012 (2)	0.022 (1)	–	–	–	–	
Unknown M12	–	0.011 (1)	–	–	–	0.001 (1)	–	
Unknown M13	–	0.009 (1)	0.017 (1)	–	–	–	–	
Unknown M18	–	0.018 (2)	–	–	–	–	–	
Unknown M19	–	0.017 (2)	–	–	–	–	–	
Unknown M22	–	–	0.015 (1)	–	–	–	–	
Glucose conj.	–	–	–	0.013 (34)	–	–	–	
Others (each < 0.01)	0.118 (12)	0.184 (24)	1.01 (39)	0.009 (24)	0.035 (12)	0.032 (25)	0.119 (51)	
PES	0.051 (5)	0.073 (10)	0.35 (14)	0.011 (29)	0.014 (5)	0.012 (9)	0.034 (15)	0.001 (72)
Losses on workup	0.02 (–2)	0.03 (3)	0.04 (2)	< 0.01 (5)	0.02 (7)	0.02 (18)	0.03 (13)	< 0.01 (12)
Total	1.011 (100)	0.756 (100)	2.58 (100)	0.038 (100)	0.303 (100)	0.127 (100)	0.233 (100)	0.014 (100)

Table 57 Radioactive residues in wheat RACs following treatment of soil with [phenyl- ^{14}C]-mesotrione

Commodity	0.462 kg ai/ha / 300 DAT			
	Forage	Hay	Straw	Grain ^a
Component	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)
Mesotrione	–	0.001 (2)	–	–
MNBA	0.043 (43)	0.007 (17)	0.015 (8)	–
AMBA	0.007 (7)	0.003 (7)	0.014 (7)	–
AMBA conj.	0.005 (5)	0.002 (4)	0.003 (1)	–
AMBA sulphate conj.	0.011 (11)	0.009 (21)	0.026 (13)	–
Unknown	–	0.002 (4)	0.005 (3)	–
Others (each < 0.01 mg/kg)	0.016 (16)	0.011 (24)	0.072 (37)	–
PES	0.015 (15)	0.013 (30)	0.029 (15)	0.001 (71)
Loss on Partitioning	0 (0)	< 0.01 (–7)	0.03 (15)	0 (0)
Total	0.100 (100)	0.044 (100)	0.197 (100)	0.015 (100)

^a The aqueous fraction for the wheat grain sample was a hydrolysate

Radioactive residues in soy forage represented 0.645 mg/kg, of which 61% TRR was extracted (Table 58). The PES containing 38% TRR was hydrolysed releasing a further 20% TRR (0.129 mg/kg). MNBA was the major metabolite in soy forage and hay (up to 48% TRR). Over 40%TRR in the soya beans was extracted. The PES containing 54% TRR (0.078 mg/kg) was further extracted with base, acid with hexane, which solubilised a further 24% TRR (0.036 mg/kg). AMBA was the major compound in soya bean (0.024 mg/kg), in addition to a unknown glucose metabolite (0.003 mg/kg). A number of unknown metabolites were observed in all commodities, no one representing > 0.011 mg/kg.

Table 58 Radioactive residues in soya bean RACs following treatment of soil with [phenyl-U-¹⁴C]-mesotrione 0.308 kg ai/ha / 30 DAT

Commodity	Forage		Hay		Bean	
Component	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
MNBA	0.312	48	0.166	35	0.014	10
AMBA	0.069	11	0.034	7	0.024	17
Unknown M1	0.004	1	0.003	1	0.01	7
Unknown M2	0.011	2	0.013	3	—	—
Unknown M18	0.009	1	0.009	2	0.005	4
Unknown M20	0.005	1	—	—	—	—
Unknown M14	—	—	0.004	1	—	—
Unknown M15	—	—	0.005	2	—	—
Glucose	—	—	—	—	0.004	3
Others (each < 0.01 mg/kg)	0.112	17	0.096	21	0.033	23
PES	0.104	16	0.124	27	0.026	18
Loss on Partitioning	0.02	3	< 0.01	2	0.03	19
Total	0.645	100	0.462	100	0.145	100

Not applicable

The major residue in endive was MNBA, accounting for up to 38% TRR (0.02 mg/kg) at 120 DAT (Table 59). Traces of AMBA were detected at 300 DAT. A number of minor unknowns were seen, each at ≤ 0.002 mg/kg. Residue levels in radish were low (≤ 0.05 mg/kg), and distributed with slightly more radioactivity in tops than roots, with MNBA accounting for to up to 53%TRR (Table 59).

Table 59 Summary of radioactive residues in endive and radish RACs following treatment of soil with [phenyl-U-¹⁴C]-mesotrione at 0.462 kg ai/ha

Rotation Period	Endive		Radish Top	Radish Root
	120 DAT	300 DAT	120 DAT	
Component	mg/kg (% TRR)	mg/kg (%TRR)	mg/kg (% TRR)	mg/kg (%TRR)
MNBA	0.02 (38)	0.007 (35)	0.025 (53)	0.013 (36)
AMBA	—	0.001 (6)		
AMBA conj.	—	—		0.003 (8)
Unknown M1	0.001 (3)	0.002 (13)		
Unknown M5	0.001 (3)	—		
Others (each < 0.001 mg/kg)	0.005 (9)	0.005 (25)	0.010 (20)	0.004 (10)
PES	0.023 (43)	0.007 (37)	0.012 (25)	0.017 (45)
Total	0.053 (100)	0.019 (100)	0.048 (100)	0.037 (100)

[Cyclohexane-2-¹⁴C]-mesotrione at a nominal rate of 0.165 kg ai/ha was sprayed once onto the bare surface of a sandy loam soil contained in pots in greenhouse (Spillner *et al.*, 1997; RR 95-042B). Endive, radish and wheat were sown into the treated soil (120 DAT). Soil cores were taken immediately after sowing and at 273 and 300 DAT. Endive was harvested 63 DAP, radish roots and leaves at 56 DAP, forage wheat at 22 DAP, forage hay at 57 DAP and wheat grain and straw at 131 DAP. Total radioactive residues in all samples ranged from < 0.001 mg/kg eq. in endive and radish to 0.004 mg/kg eq. in wheat straw. Residues in control reached 0.002 mg/kg in wheat grain and straw.

Radioactive residues in soil declined to 4% of the applied radioactivity by 120 DAT. No further analysis was conducted since the radioactive residues were < 0.01 mg/kg.

In another rotational study, [cyclohexane-2-¹⁴C]-mesotrione was sprayed once at 0.308 kg ai/ha or 0.462 kg ai/ha to sandy loam soil pots (Spillner *et al.*, 1996; RR 96-005B). Wheat was planted at 30, 120 and 300 DAT, soy at 30 DAT and endive and radish at 120 DAT. Samples with residues ≥ 0.01 mg/kg were extracted with acetonitrile:water. Soya beans were first extracted with hexane to remove the oil. The 30-DAT wheat grain sample was hydrolysed using 2 N hydrochloric acid; the hydrolysate partitioned with ethyl acetate to remove the organosoluble residues, and the aqueous phase was analysed for ¹⁴C-glucose. Reverse-phase HPLC was used for the characterisation mesotrione and its metabolites.

Radioactive residues in the soil declined to 10, 5 and 4% AR after 30, 120 and 300 DAT, respectively. Radioactive residues in wheat commodities declined over time, with the highest residues found in feed (Table 60). Residues in the soy ranged from 0.017 in beans to 0.026 mg/kg in forage at 30 DAT. In root crops at 120 DAT they reached 0.004 mg/kg in radish leaves and were not further investigated.

Table 60 Summary of total radioactive residues in rotational crop samples grown in soil treated with [cyclohexane-2-¹⁴C]-mesotrione

Crop	30 DAT (0.308 kg/ha)		120 DAT (0.462 kg/ha)		300 DAT (0.462 kg/ha)	
	Treated (mg/kg)	Control (mg/kg)	Treated (mg/kg)	Control (mg/kg)	Treated (mg/kg)	Control (mg/kg)
Wheat straw	0.059	0.002	0.043	0.002	0.006	< 0.001
Wheat forage	0.054	0.001	0.017	< 0.001	0.002	< 0.001
Wheat hay	0.048	0.001	0.013	< 0.001	0.002	< 0.001
Wheat grain	0.010	0.002	0.008	0.002	0.001	< 0.001
Soy forage	0.026	0.001	–	–	–	–
Soy hay	0.021	0.001	–	–	–	–
Soya bean	0.017	0.003	–	–	–	–
Endive	–	–	0.003	< 0.001	–	–
Radish root	–	–	0.002	< 0.001	–	–
Radish leaves	–	–	0.004	< 0.001	–	–

Mesotrione was a major residue in feed wheat commodities, but was not detected in grain, in which the only identified residue was a glucose conjugate (Table 61). Residues in wheat grain was 0.01 mg/kg at 30 DAT. 4-hydroxy mesotrione was the major metabolite identified, accounting for up to 15% TRR in forage (0.008 mg/kg). Over 70% of the residues were extracted from all matrices, and PES was not analysed further.

Table 61 Radioactive residues in wheat RACs following treatment with [cyclohexane-2-¹⁴C]-mesotrione

		0.308 kg ai/ha/ 30 DAT				0.462 kg ai/ha/120 DAT		
Commodity		Forage	Hay	Straw	Grain ^a	Forage	Hay	Straw
Component		mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)
mesotrione		0.009 (17)	0.003 (7)	0.0034 (5.7)	–	0.001 (5)	–	0.001 (3)
4-OH		0.008 (15)	0.004 (8)	0.0021 (3.6)	–	0.002 (12)	–	0.003 (6)
Unknown 1		–	–	0.0021 (3.6)	–	–	–	< 0.001 (1)
Unknown 2		0.002 (4)	0.003 (6)	–	–	0.001 (8)	–	0.002 (5)
XanH4		0.001 (2)	0.001 (2)	0.0021 (3.6)	–	< 0.001 (3)	–	0.002 (4)
Unknown 3		0.002 (3)	0.002 (4)	0.0025 (4.3)	–	–	–	0.001 (3)
4-OGlu		0.002 (4)	0.003 (6)	0.0017 (2.8)	–	0.001 (6)	–	0.002 (4)
5-OH		0.001 (1)	0.002 (4)	0.0025 (4.3)	–	< 0.001 (2)	–	0.001 (2)
Unknown 4		0.001 (1)	0.001 (3)	0.0029 (5.0)	–	< 0.001 (2)	–	0.001 (2)
Unretained		0.007 (12)	0.009 (20)	0.0147 (24.9)	–	0.002 (10)	–	0.010 (23)
Remainder		0.006 (10)	0.005 (10)	0.0080 (13.5)	–	0.001 (8)	–	0.006 (13)
Glucose					0.007 (69)			

	0.308 kg ai/ha/ 30 DAT				0.462 kg ai/ha/120 DAT		
Commodity	Forage	Hay	Straw	Grain ^a	Forage	Hay	Straw
acetone rinse + acetonitrile	0.009 (16)	0.004 (8)	< 0.001 (1)	< 0.001 (5)	0.003 (20)	0.0026 (20)	0.001 (2)
PES	0.008 (15)	0.011 (22)	0.017 (28%)	0.003 (26)	0.004 (24)	0.0027 (21)	0.014 (32)
Total	0.056 (100)	0.048 (100)	0.059 (100)	0.010 (100)	0.017 (100)	0.013 (100)	0.043 (100)

^a The aqueous fraction for the wheat grain sample was a hydrolysate, not an extract; 4-OH: 4-hydroxy-mesotrione; 4-OGlu: 4-hydroxy-mesotrione glucoside conjugate; 5-OH: 5-hydroxy-mesotrione; XanH4: 6-(methylsulfonyl)-1-oxo-1.2.3.4-tetrahydro-9H-xanthen-9-one

About 70% of radioactivity was extracted from soy forage and hay, and PES was not analysed further. 13% TRR was extracted from soya beans with hexane and 20% TRR with acetonitrile/water mixtures (< 0.01 mg/kg); PES accounted for 64% TRR (0.01 mg/kg). Neither the soya bean extracts nor PES were analysed further. Mesotrione was the major residue in soy forage and hay (up to 11% TRR, 0.003 mg/kg), while 5-hydroxy-mesotrione and 4-hydroxy-mesotrione represented 4 to 6% TRR (Table 62).

Table 62 Radioactive residues in soy samples following treatment with [cyclohexane-2-¹⁴C]-mesotrione (0.308 kg ai/ha/30DAT)

Commodity:		Forage		Hay		Bean	
	Component	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
	Mesotrione	0.003	11	0.002	10	—	—
	5- hydroxy-mesotrione	0.001	6	0.001	5	—	—
	4-hydroxy-mesotrione	0.001	4	—	—	—	—
	4-OH—glucose conjugate	—	—	0.001	5	—	—
	XanH4	0.001	3	0.001	4	—	—
	Unretained	0.005	20	0.005	22	—	—
	Unknown 1	—	—	< 0.001	3	—	—
	Unknown 3	—	—	0.001	5	—	—
	Remainder	0.005	19	0.003	13	—	—
	Acetone rinse + acetonitrile	0.002	7	0.001	6	0.004 ^a	23 ^a
	PES	0.08	29	0.006	27	0.011	64
	Total	0.026	100	0.021	100	0.017	100

^a Organic extract also included value obtained from the hexane extraction step; XanH4: 6-(methylsulfonyl)-1-oxo-1.2.3.4-tetrahydro-9H-xanthen-9-one

The proposed pathways of mesotrione in rotated crops obtained from studies using the mesotrione labelled in phenyl and the cyclohexane are shown in Figure 4.

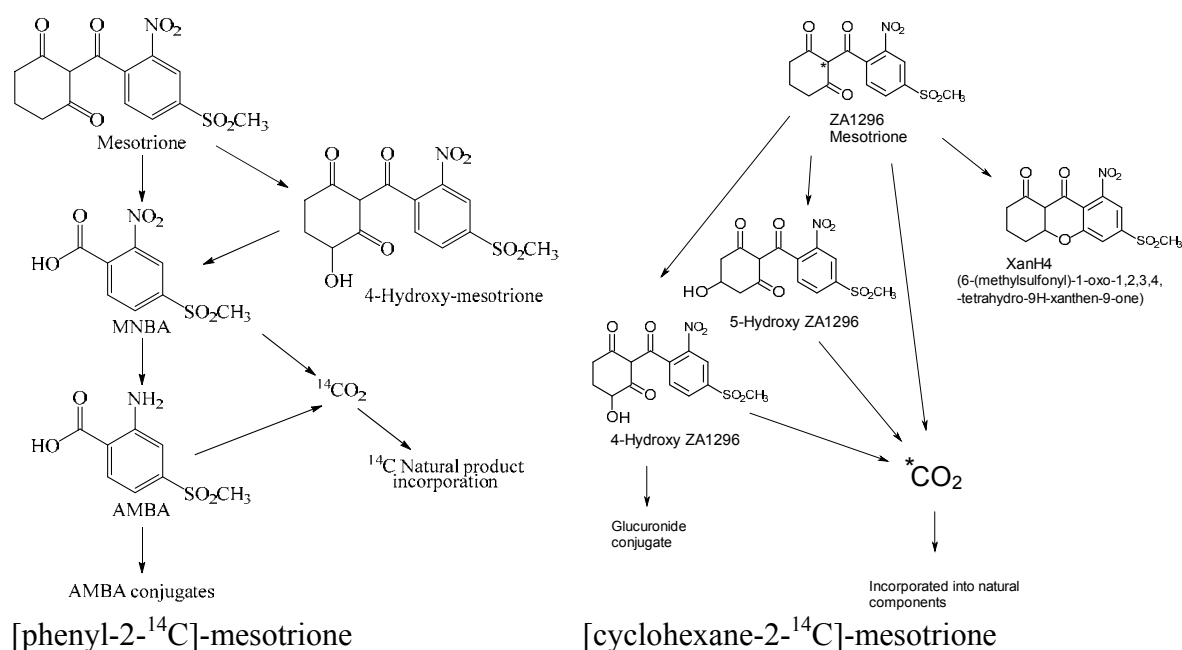


Figure 4 Proposed metabolic pathway of mesotrione in rotational crops

Analytical Methods

Vegetable crops

In Method RAM 366/01, residues of mesotrione and MNBA were extracted from crop samples with acetonitrile/water containing 10 g/L sodium chloride (50:50), an aliquot diluted with water and formic acid and cleaned up by Oasis[®] HLB SPE. The extract is partitioned into methylene chloride, evaporated to dryness, taken up with acetonitrile/water and analysed by HPLC-MS/MS, in negative ion mode, 290.9 m/z ion for quantification and no confirmation ion included. LOQ was 0.01 mg/kg in all crop matrices. Recovery data for mesotrione in corn (Hill, 2001; Report 2704 and Crook, 2001; RJ3253B), and cranberry (Salzman, 2005; 08903) are summarised in Table 63. The method was also successfully validated for MNBA.

Table 63 Recovery data for mesotrione by LC-MS/MS (negative ion mode; m/z=338 → 291)

Matrix	Fortification Level (mg/kg)	N	Mean (%)	RSD (%)	Report
Corn/maize (whole plant)	0.01	5	95	5	2704 and RJ3253B
	0.10	5	100	3	
	1.0	5	91	9	
	10	5	105	4	
Corn/maize (grain)	0.01	5	86	12	
	0.10	5	84	7	
	1.0	5	96	5	
Cranberries	0.01	6	100	21	08903
	0.10	3	99	7	
	1.0	5	114	11	

Results of the recovery experiments conducted as part of the residue studies in which method RAM 366/01 was used are summarised in Table 64.

Table 64 Mesotrione procedural recovery data obtained during residue analysis

Matrix	Fortification Level (mg/kg)	n	Mean (%)	RSD (%)	Report
Cranberries	0.01	3	113	5	08903
	0.10	5	93	9	
Blueberries	0.01	9	84	7	T010288-04
	1.0	5	93	16	
Raspberries	0.01	6	90	11	
	1.0	3	94	10	
Blackberries	0.01	1	87	–	T021571-04
Okra	0.01	13	80	11	
	0.10	7	91	6	
	1.0	6	100	9	
Sweet corn (kernel)	0.01	1	80	–	04-7012
	0.02	1	81	–	
Sweet corn (cob)	0.01	1	103	–	
	0.02	1	90	–	
Field corn (kernel)	0.01	1	96	–	T000921-09-REG
	0.10	1	83	–	
Field corn (cob)	0.01	1	100	–	
	0.10	1	85	–	
Field corn (whole cob)	0.01	1	84	–	
	0.10	1	62	–	
Field corn (rest plant)	0.01	2	98	–	
	0.10	2	89	–	
Field corn (kernel)	0.01	1	97	–	T000920-09-REG
	0.10	1	98	–	
Field corn (cob)	0.01	1	115	–	
	0.10	1	78	–	
Field corn (whole cob)	0.01	1	91	–	
	0.10	1	85	–	
Field corn (rest plant)	0.01	2	104	–	
	0.10	2	88	–	
Sweet corn (forage w/o ears)	0.01	2	97	–	T001589-08
	0.50	2	90	–	
Sweet corn (ears)	0.01	1	99	–	
	0.50	1	96	–	
Sweet corn (stover)	0.01	1	105	–	
	0.50	1	81	–	
Soya bean (seed)	0.01	6	114	10	T005595-06
	0.10	6	108	13	
Soya bean (meal)	0.01	1	114	–	
	0.10	1	113	–	
Soya bean (hulls)	0.01	1	107	–	
	0.10	1	122	–	
Soya bean (oil)	0.01	1	134	–	T000908-07
	0.10	1	100	–	
Soya bean (seed)	0.01	21	83	15	
	0.10	21	87	11	
Soya bean (meal)	0.01	1	76	–	
	1.0	1	118	–	
Soya bean (hulls)	0.01	1	82	–	
	1.0	1	90	–	
Soya bean (crude oil)	0.01	1	86	–	
	1.0	1	104	–	
Soya bean (refined oil)	0.01	1	96	–	
	1.0	1	96	–	
Soya bean (AGF)	0.01	1	71	–	
	1.0	1	85	–	
Soya bean (flour)	0.01	1	87	–	
	1.0	1	95	–	
Soya bean	0.01	1	77	–	

Matrix	Fortification Level (mg/kg)	n	Mean (%)	RSD (%)	Report
(milk)	1.0	1	101	–	
Soya bean (tofu)	0.01	1	70	–	
	1.0	1	97	–	
Soya bean (soy sauce)	0.01	1	70	–	
	1.0	1	84	–	
Soya bean (miso)	0.01	1	86	–	
	1.0	1	90	–	T021572-04
Asparagus	0.01	14	84	15	
	0.05	1	82	–	
	0.10	2	88	–	
	0.50	1	97	–	
	1.0	2	94	–	
	10	1	93	–	
Rhubarb (petioles)	0.01	7	88	10	T014372-05
	0.10	5	86	8	
	1.0	2	95	–	
Millet (forage)	0.01	7	81	10	T010289-04
	1.0	7	91	7	
Millet (hay)	0.01	7	78	13	
	1.0	7	86	9	
Millet (straw)	0.01	6	95	19	
	1.0	5	93	13	
Millet (grain)	0.01	7	88	18	
	1.0	6	90	10	
Oat (forage)	0.01	14	77	8	T004407-05
	0.02	1	74	–	
	0.05	6	85	6	
	0.10	1	86	–	
	0.50	1	70	–	
	1.0	1	94	–	
Oat (hay)	0.01	12	73	7	
	0.02	3	71	6	
	0.05	4	76	4	
	0.10	3	84	2	
	0.20	1	83	–	
	0.50	1	74	–	
Oat (straw)	0.01	11	77	11	
	0.02	2	83	–	
	0.10	1	78	–	
	0.50	3	85	4	
Oat (grain)	0.01	10	75	11	
	0.02	1	70	–	
	0.05	5	80	3	
	0.20	1	84	–	
Sorghum (forage)	0.01	12	86	12	T020419-04
	0.05	3	87	11	
	0.10	2	91	–	
	0.20	2	94	–	
	0.50	1	80	–	
	1.0	1	94	–	
	2.0	1	119	–	
	20	2	99	–	
Sorghum (stover)	0.01	13	84	10	
	0.02	1	75	–	
	0.05	1	83	–	
	0.10	1	112	–	
	0.20	1	95	–	
	0.50	1	91	–	
	1.0	2	92	–	
	2.0	1	96	–	

Matrix	Fortification Level (mg/kg)	n	Mean (%)	RSD (%)	Report
Sorghum (grain)	0.01	12	90	11	
	0.05	4	78	6	
	0.10	3	92	1	
	0.20	3	101	14	
	0.50	3	98	12	
	1.0	1	94	–	
	2.0	1	105	–	
Sorghum (AGF)	0.01	2	82	–	
	0.10	2	88	–	
Sugarcane	0.01	16	71	6	T020420-04
	0.05	2	70	–	
	0.10	9	77	4	
	0.20	2	83	–	
	0.50	1	87	–	
	1.0	1	82	–	
	10	1	90	–	
Flax / Linseed (seed)	0.01	6	97	17	T010290-04
	1.0	6	98	10	
Flax / Linseed (meal)	0.01	1	85	–	
	1.0	1	102	–	

In Methods TMR0643B and TMR0882B, mesotrione and MNBA residues are extracted with acetonitrile/water (1:1). An aliquot is diluted with water, acidified, partitioned with ethyl acetate and subjected to silica SPE clean-up. Further clean-up is done by reversed phase HPLC. The MNBA, isolated in the first fraction, is chemically reduced to AMBA with SnCl_2 in HCl. The mesotrione residue, isolated in the second fraction, is oxidised to MNBA using hydrogen peroxide, which is reduced to AMBA using acidic SnCl_2 . After clean-up by C18 SPE, mesotrione and MNBA fractions are each analysed for the AMBA conversion product by a reversed-phase HPLC-fluorescence detection (ex 227 nm; em 424 nm). Mesotrione and MNBA standards were converted to AMBA concurrent with the samples. The method was validated for corn with a LOQ of 0.01 mg/kg for both mesotrione and MNBA. Independent validation was also provided. The recovery data for mesotrione are shown in Table 65.

Table 65 Mesotrione recovery data of methods TMR0643B and TMR0882B (HPLC-FL)

Matrix	Fortification Level (mg/kg)	N	Mean (%)	RSD (%)	Report
Corn (forage)	0.01	13	82	16	Method TMR0643B ADD Alfness, 1996, 1997
	0.10	11	85	15	
Corn (fodder)	0.01	9	78	10	
	0.10	5	67	15	
Corn (grain)	0.01	2	109	–	
	0.10	2	86	–	
Corn (forage)	0.01	2	85	–	Bolygo, 1996 RJ2149B Independent laboratory validation
	0.05	2	94	–	
Corn (grain)	0.01	2	76	–	
	0.05	2	81	–	
Corn (fodder)	0.01	2	108	–	
	0.05	2	104	–	
Field corn (forage)	0.01	11	85	12	RR 96-018B Residue trials
	0.10	7	86	17	
Field corn (fodder)	0.01	11	78	10	
	0.10	3	82	11	
Field corn (grain)	0.01	13	95	13	
	0.10	3	100	12	
Corn (forage)	0.01	4	77	6	Method TMR0882B Alfness, 1999
	0.10	4	79	7	
Corn (fodder)	0.01	8	78	4	
	0.10	5	81	4	

Matrix	Fortification Level (mg/kg)	N	Mean (%)	RSD (%)	Report
Corn (grain)	0.01	4	85	14	
	0.10	4	81	2	
Sugar cane	0.01	4	75	5	
	0.10	4	73	2	
Corn (fodder)	0.01	2	99	—	James, 1999; RR 99-062B Independent validation
	0.02	2	79	—	
Sweet corn (ears)	0.01	5	67	10	487-01 residue trial
	0.10	5	74	8	
	1.0	1	74	—	
Sweet corn (forage w/o ears)	0.01	6	88	20	
	0.10	6	84	11	
	1.0	3	90	15	
Sweet corn (forage with ears)	0.01	6	76	19	
	0.10	6	76	6	
	1.0	1	93	—	
Sweet corn (stover)	0.01	4	73	15	
	0.10	5	75	7	
	1.0	1	83	—	
	2.0	1	83	—	
Field corn (forage)	0.01	3	89	13	1847-01 residue trial
	0.10	5	78	5	
Field corn (stover)	0.01	4	87	22	
	0.10	4	80	5	
Field corn (grain)	0.01	4	80	10	
	0.10	4	84	9	
Sugarcane	0.05	2	80	—	RJ3076B; Residue trial

Method TMR0643B was radio-validated using incurred radioactive residues in a forage sample treated pre-emergence (Tarr & van Neste, 1997). An aliquot of the forage sample was extracted once with an acetonitrile:water mixture (1:1) using a high speed homogeniser, an aliquot of the supernatant partitioned three times into ethyl acetate, the combined fractions evaporated and reconstituted in acetonitrile. Residues of mesotrione and MNBA were quantified by TLC with storage-phosphor autoradiography. Levels of mesotrione and MNBA in forage were 0.008 and 0.073 mg/kg, respectively. Residues of mesotrione and MNBA obtained by exhaustive extraction within this metabolism study were 0.008 mg/kg and 0.070 mg/kg, respectively.

In Method TMR0689B (Meyers, 1996), mesotrione and MNBA residues are extracted from corn commodities with acetonitrile/water (1:1), an aliquot diluted with a sodium sulphate solution, acidified and partitioned with methylene chloride. The methylene chloride extract is evaporated and the residue heated with Jones Reagent (chromium^{VI} oxide acid solution) to convert mesotrione to MNBA. The total MNBA is extracted with ethyl acetate, evaporated to dryness, the residue dissolved in methyl ethyl ketone and the isopropyl ester of MNBA is formed by heating the mixture with 2-iodopropane and potassium carbonate. The methyl ethyl ketone is evaporated, the residue extracted with acetone, evaporated to dryness and the isopropyl-MNBA residue dissolved in toluene containing 0.05% (w/v) 5-nitrovanillin for analyse by GC-MS (monitoring ion: m/z 246). The method determines the sum of mesotrione and MNBA at a combined LOQ of 0.01 mg/kg (Table 66).

Table 66 Mesotrione procedural recovery data obtained during residue analysis by GC-MS

Matrix	Fortification Level (mg/kg)	N	Mean (%)	RSD (%)	Report
Field corn (grain)	0.1	1	85	—	RR 97-043B
	0.3	2	98	—	
Field corn (silage)	0.01	1	94	—	
	0.03	3	93	8	
Field corn (grain)	0.01	3	80	5	RR 98-035B
Field corn (silage)	0.01	2	83	—	

Samples were analysed based on the QuEChERS multi-residue method (Watson, 2013). The method involves extraction with acetonitrile, addition of magnesium sulphate, sodium chloride and buffering citrate salts, centrifugation, an aliquot of the acetonitrile phase transferred to the freezer overnight (oilseed rape seed only) and cleaned up with magnesium sulphate prior to analysis by LC-MS/MS. Primary secondary amine (PSA) used in the original QuEChERS was not included due to low procedural recoveries for mesotrione. The LOQ was 0.01 mg/kg in all matrices. Recovery data in orange, maize and oilseed rape are summarised in Table 67.

Table 67 Mesotrione recovery data for method based on QuEChERS by LC-MS/MS (negative mode)

Matrix	Fortification Level (mg/kg)	n	Transition m/z=338 → 291		Transition m/z=338 → 212		Report
			Mean (%)	RSD (%)	Mean (%)	RSD (%)	
Orange (whole fruit)	0.01	5	77	14	81	15	Watson, 2013; Report S12-03251
	0.10	5	74	5.0	74	4.9	
Maize (grain)	0.01	5	89	2.0	87	5.2	
	0.10	5	89	2.7	90	3.8	
Maize (forage)	0.01	5	97	6.2	99	5.3	
	0.10	5	84	6.9	84	8.8	
Oilseed Rape (seeds)	0.01	5	80	2.5	88	14	
	0.10	5	81	4.2	82	4.2	
Maize (forage)	0.01	5	92	9	101	10	Independent validation (report S12-04607)
	0.10	4	72	5	71	8	
Maize (grain)	0.01	5	85	3	83	5	
	0.10	5	77	4	76	5	

Food of animal origin

In method TMR0914B, mesotrione and MNBA residues are extracted from milk and eggs with acetone and from animal tissues with an acetone/water (60:40). An aliquot is diluted with acidified water, partitioned twice into methylene chloride, and residues of mesotrione are converted to MNBA using hydrogen peroxide. After elimination of excess peroxide with catalase enzyme, MNBA is reduced to AMBA by heating with SnCl₂ and HCl and AMBA determined by reversed phase HPLC-fluorescence detection. The LOQ was 0.01 mg/kg in all matrices. Recovery data for mesotrione is summarised in Table 68.

Table 68 Mesotrione recovery data for Method TMR0914B

Matrix	Fortification Level (mg/kg)	n	Mean (%)	RSD (%)	Report
Meat (bovine)	0.01	6	88	4	Meyers, 1999 TMR0914B
	0.10	6	84	2	
Liver (bovine)	0.01	6	79	6	
	0.10	6	74	6	
Milk (cow)	0.01	6	90	4	
	0.10	6	93	3	
Egg (hen)	0.01	6	88	5	
	0.10	6	88	3	
Meat (bovine)	0.01	2	78	—	Brookey, 2000 RR 96-005B independent laboratory validation
	0.02	2	79	—	
Milk (cow)	0.01	2	88	—	
	0.02	2	86	—	

Mesotrione was determined in animal matrices using the modified QuEChERS, excluding PSA (Watson, 2013; Report no. S12-03250). LOQ was 0.01 mg/kg. The same procedure was independently validated (Bernal, 2013; Report no. S12-04608). The results are shown in Table 69.

Table 69 Mesotrione validation data (n=5 at each level)

Matrix	Fortification level (mg/kg)	Report no. S12-03250				Report no. S12-04608			
		m/z=338 → 291		m/z=338 → 212		m/z=338 → 291		m/z=338 → 212	
		Mean (%)	RSD (%)	Mean (%)	RSD (%)	Mean (%)	RSD (%)	Mean (%)	RSD (%)
Milk	0.01	92	1.8	93	3.9	84	3	82	4
	0.10	87	1.1	88	104	83	4	80	2
Eggs	0.01	77	3.1	77	3.0	90	6	97	5
	0.10	71	2.2	71	2.3	92	4	93	3
Muscle	0.01	92	2.6	93	3.5				
	0.10	86	2.7	88	2.9				
Fat	0.01	102	1.4	103	2.0				
	0.10	97	1.8	99	2.7				
Liver	0.01	86	2.7	88	3.9	79	5	71	9
	0.10	84	0.7	83	1.6	75	3	75	2
Kidney	0.01	99	2.0	92	3.0				
	0.10	91	0.9	87	2.2				

Storage stability under frozen conditions

Samples of maize grain, maize forage, maize fodder, radish root, and soya bean seed were homogenized and fortified at 0.1 mg/kg with either mesotrione or MNBA (Wiebe & Peyton, 1999). Duplicate samples were stored under frozen conditions ($-18^{\circ}\text{C} \pm 5^{\circ}\text{C}$) and analysed at intervals up to 44 months. Mesotrione and MNBA were quantified as AMBA by HPLC fluorescence detection (Method TMR0643B). The limit of quantification for mesotrione and MNBA in all matrices was 0.01 mg/kg. The results are shown in Table 70.

Table 70 Stability of residues in crop commodities during frozen storage, in % remaining (not corrected for analytical recovery). Mean of duplicate samples fortified at 0.1 mg/kg

Month of storage	Maize grain		Maize forage		Maize fodder		Radish root		Soya bean seed	
	mesotrione	MNBA	mesotrione	MNBA	mesotrione	MNBA	mesotrione	MNBA	mesotrione	MNBA
0	72	79	79	78	53 ^a	76	86	95	89	92
0.5					58	87				
1	89	88	70	89	61	85	79	83		
2									80	81
3			75	86	77	92			78	84
4	86	86								
5							94	89		
6									79	101
7					86	100	80	90		
8	81	80	75	84						
13							87	91 ^a		
14									102	114
15							77	80		
17	86	103			78	107				
18			74	105						
29		89				87			129	94
30					110	81				
31	109		81	85					97	
32							104	81		
40									91 ^a	92
42	86	100	65 ^a	85 ^a	73 ^a	91				
44							85	93		

^a Single value reported; duplicate sample lost during sample clean-up

Samples of blueberry, asparagus, sugarcane and okra were grounded in dry ice and fortified with mesotrione at 1.0 mg/kg (Link, 2007). Duplicate samples were prepared and stored under frozen conditions (-20°C) and analysed at intervals over 13 months for sugarcane and okra and over 22

months for blueberry and asparagus. Mesotrione was quantified by HPLC-MS/MS (LOQ of 0.01 mg/kg). The results shown in Table 71 are not corrected for procedural recovery.

Table 71 Stability of residues, in crop commodities, during frozen storage. Samples were fortified at 1.0 mg/kg of mesotrione

Commodity	Month of storage	% remaining individuals	% remaining, mean	Normalised to day 0 (%)
Blueberry	0	92; 93	93	100
	1	83; 81	82	89
	3	90; 89	90	97
	6	82; 90	86	93
	13	98; 101	100	108
	22	78; 79	79	85
Asparagus	0	92; 104	98	100
	1	97; 92	95	96
	3	92; 96	94	96
	6	94; 90	92	94
	13	98; 99	99	101
	22	93; 93	93	95
Sugarcane	0	77; 74	76	100
	1	89; 87	88	117
	3	81; 83	82	109
	6	83; 84	84	111
	13	80; 86	83	110
	22	80; 86	83	110
Okra	0	102; 101	102	100
	1	98; 96	97	96
	3	87; 91	89	88
	6	94; 99	97	95
	13	98; 97	98	96
	22	98; 97	98	96

USE PATTERNS

Mesotrione is an herbicide used pre-emergence and post-emergence for selective control of annual broad-leaved weeds, which cease growth soon after application (high or low volume sprayers). Mesotrione is registered in many countries, but only the relevant GAP information for this evaluation is given in Table 72. DAT means days after treatment.

Table 72 Registered uses of mesotrione relevant for the evaluation

Crop	Country	Formulation		Application		No	PHI (days)
		kg ai/L kg	Type	Method	Rate kg ai/ha		
Asparagus	USA	0.480	SC	Pre-emergence prior to spear emergence	0.270	1	–
Asparagus	USA	0.480	SC	Post-emergence after completion of harvesting	0.105	1	–
Asparagus	USA	0.480	SC	Pre-+ post-emergence	0.165 + 0.105	2	–
Bush and Cane berries	USA	0.480	SC	Pre-emergence	0.210	1	GS
		0.480	SC	Direct spray to the base of the plant, before bloom	0.105	2	
Cranberries	USA	0.480	SC	Broadcast foliar	0.280	2	45
Flax/Linseed	USA	0.480	SC	Post-emergence	0.210	1	NS
Maize	Canada	0.480	SC	Pre-or early post-emergence	0.14	1	GS
Maize	Canada	0.480	SC	Late post-emergence	0.10	1	GS
Maize	Germany	0.100	SC	Broadcast	0.150	1	GS
Maize	USA	0.480	SC	Pre-emergence	0.270	1	GS
Maize	USA	0.480	SC	Pre-+ post-emergence (up to 8 leaf stage)	0.165 + 0.105	2	
Millet	USA	0.480	SC	Pre-emergence	0.210	1	–
Oat	USA	0.480	SC	Pre-emergence	0.210	1	50
Oat	USA	0.480	SC	Post-emergence	0.105	1	

Crop	Country	Formulation		Application		No	PHI (days)
		kg ai/L kg	Type	Method	Rate kg ai/ha		
Okra	USA	0.480	SC	Pre-emergence	0.210	1	28
		0.480	SC	Post-emergence, directed to the weed	0.105	1	
Rhubarb	USA	0.480	SC	Pre-emergence	0.210	1	21
Rice	Republic of Korea	0.006	GR	Post planting into the water, 5–7 days after transplanting	0.09	1	Ns
Soy	USA	0.480	SC	Pre-emergence	0.210	1	Ns
Soy HT	Canada	0.480	SC	Pre-emergence	0.144	1	45
Soy HT	Canada	0.480	SC	Early Post-emergence	0.144	1	45
Soy HT	Canada	0.480	SC	Post-emergence	0.100	1	45
Soy HT	USA	0.480	SC	Pre-emergence	0.225	1	Ns
Soy HT	USA	0.480	SC	Early post-emergence, up to BBCH 13	0.225	1	Ns
Soy HT	USA	0.480	SC	Pre-+ post-emergence (BBCH 14 to 60)	0.225 + 0.125	2	GS
Sorghum	USA	0.480	SC	Pre-emergence	0.224	1	–
Sugar Cane	South Africa	0.480	SC	Broadcast	0.150	1	181
Sugar Cane	USA	0.480	SC	Pre-emergence	0.270	1	–
Sugar Cane	USA	0.480	SC	Post-emergence	0.105	2	114
Sugar Cane	USA	0.480	SC	Pre-+ post-emergence	0.36	1	114
Sweet corn	Germany	0.100	SC	Post-emergence (BBCH 12–18)	0.150	1	GS
Sweet corn	USA	0.480	SC	Pre-emergence	0.270	1	45
Sweet corn	USA	0.480	SC	Pre-+ post-emergence (up to 8 leaf stage)	0.165 + 0.105	2	45

NS= not specified

GS= growth stage

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Supervised residue trials conducted with mesotrione on a variety of crops in Europe, Canada and USA were submitted to the Meeting. Studies were conducted according to GLP, and specified concurrent determination of residues in untreated crops gave residues < LOQ, unless specified. Residues of mesotrione arising from use patterns where rate or days after treatment (DAT) $\pm$ 25% of GAP are underlined and considered for estimation of maximum residue levels and STMRs. When residues in samples harvested at a later stage were higher than those found at the critical PHI, they were used for the estimations. In trials conducted in the USA, duplicate or multiple field samples from replicate plots were taken for analysis at each sampling time and the mean was selected for the estimations. In total, 373 supervised trials were submitted and food commodities analysed for residues; in 193 cereal trials, feed commodities were also analysed. The data are summarized in Table 73.

Table 73 Summary of the supervised trials conducted with mesotrione

Codex group	Commodity	Region	No. of trials	Table No.
Berries and other small fruits	Blueberries, Raspberries, blackberries and cranberries	USA	25	74
Fruiting vegetables, other than Cucurbits	Okra	USA	20	75
Pulses	Sweet corn	France/USA	8/15	76
	Soy	USA	23	77
	HT Soy	USA	49	78
Stalk and stem vegetables	Asparagus	USA	24	79
	Rhubarb	USA	8	80
Cereal grains	Maize	EU/CAN/USA	3/24/24	81
	Millet	USA	15	82
	Oats	USA	34	83
	Rice	Japan/Korea	8/2	84
	Sorghum	USA	28	85
Grasses, for sugar or syrup production	Sugarcane	USA/South Africa	26/4	86

Codex group	Commodity	Region	No. of trials	Table No.
Oilseeds	Flax/Linseed	USA	17	87
Feed commodities	Corn/maize	EU/CAN/USA	101	88
	Millet	USA	15	89
	Oat	USA	34	90
	Rice	Japan/Korea	16	91
	Sorghum	USA	28	92

Berries and other small fruits

Twenty five supervised residue trials were conducted on berries in the USA using broadcast application during 2004 and 2005. Five trials were conducted on cranberries and ten trials were conducted on bush and cane berries. The results are shown in Table 74. Samples were stored deep-frozen for a maximum of 16.4 months and analysed for residues of mesotrione using method RAM 366/01 (LC-MS/MS).

Table 74 Summary of residue trials conducted with mesotrione (SC formulation) in USA on berries in 2004/2005

State	Crop (Variety)	Application rate, kg ai/ha	Growth Stage at Application	DAT (days)	Residue (mg/kg)	Report, trial
Massachusetts	Cranberry (Early Black)	0.341 + 0.224 (+ NIS)	Fruit development Fruit sizing—Early colour	43	< 0.01 (2)	08903.05-CAR20, MA01
New Jersey	Cranberry (Early Black)	0.346 + 0.226 (+ NIS)	fruiting fruiting	44	< 0.01 (2)	08903.05-CAR20, NJ26
Oregon	Cranberry (McFarlin)	0.388 + 0.240	small green berry White fruit—Pink fruit	48	< 0.01 (2)	08903.05-CAR20, OR18
Wisconsin ^a	Cranberry (McFarlin)	0.343 + 0.220 (+ NIS)	fruiting	43	< 0.01 (2)	08903.05-CAR20, WI18
Wisconsin ^a	Cranberry (Stevens)	0.354 + 0.220 (+ NIS)	fruiting	43	< 0.01 (2)	08903.05-CAR20, WI19
New York	Blueberry (Blue Ray)	0.102	BBCH 59	77	< 0.01 (2)	T010288-04, 5A-HR04-5630
		0.211	BBCH 59	77	< 0.01 (2)	
North Caroline ^b	Blueberry (Reveille)	0.107	Early Fruit set	32	< 0.01 (2)	T010288-04, SJ-HR04-5631
				35	< 0.01 (2)	
				39	< 0.01 (2)	
				43	< 0.01 (2)	
	Blueberry (Reveille)	0.216	Early Fruit set	32	< 0.01 (2)	
				35	< 0.01 (2)	
				39	< 0.01 (2)	
				43	< 0.01 (2)	
North Caroline ^b	Blueberry (Reveille)	0.109	Early Fruit set	34	< 0.01 (2)	T010288-04, SJ-HR04-5632
		0.214	Early Fruit set	34	< 0.01 (2)	
Michigan (Freemont)	Blueberry (Blue Crop)	0.106	Early pink bud	72	< 0.01 (2)	T010288-04, NL-HR04-5633
		0.209	Early pink bud	72	< 0.01 (2)	
Michigan (Conklin)	Blueberry (Blue Ray)	0.105	Pink bud	64	< 0.01 (2)	T010288-04, NL-HR04-5634
		0.212	Pink bud	64	< 0.01 (2)	
Washington	Blueberry (Nelson and Iliot)	0.104	Fruit present—Blooming	88	< 0.01 (2)	T010288-04, WF- HR04-5635
		0.210		88	< 0.01 (2)	
Michigan (Belding)	Raspberry (K81-6)	0.106	Pre-bloom—Leaves present	64	< 0.01 (2)	T010288-04, NL-HR04-5636
				70	< 0.01 (2)	
				74	< 0.01 (2)	
				78	< 0.01 (2)	
	Raspberry (K81-6)	0.208	Pre-bloom—Leaves present	64	< 0.01 (2)	
				70	< 0.01 (2)	
				74	< 0.01 (2)	
				78	< 0.01 (2)	

State	Crop (Variety)	Application rate, kg ai/ha	Growth Stage at Application	DAT (days)	Residue (mg/kg)	Report, trial
Oregon (Corvallis) ^c	Raspberry (Caroline)	0.105	BBCH 51	52	< 0.01 (2)	T010288-04, WG-WG-HR04-5637
	Raspberry (Caroline)	0.209	BBCH 51	52	<u>< 0.01</u> (2)	
Oregon (Hullsboro)	Blackberry (Kotata)	0.107	Pre-bloom	62	< 0.01 (2)	T010288-04, WG-HR04-5638
	Blackberry (Kotata)	0.218	Pre-bloom	62	<u>< 0.01</u> (2)	
Oregon (Corvallis) ^c	Raspberry (Caroline)	0.109	BBCH 51	83	< 0.01 (2)	T010288-04, WG-HR05-6370
	Raspberry (Caroline)	0.208	BBCH 51	83	<u>< 0.01</u> (2)	

NIS=Non-ionic surfactant

^a Trials conducted at the same location and dates, different variety

^b Trials conducted at the same location and dates, one value was considered

^c Trials conducted at the same location, but in different periods.

Okra

Twenty supervised residue trials were conducted on *okra* in the USA (Report T021571-04) during 2005 using either one pre-emergence application or one post-emergence application directed to the weed. A suspension concentrate (SC) formulation containing mesotrione was applied using soil surface spray pre-emergence (SS), post-emergence broadcast application over-the-top of the weed (POT) and or post-emergence directed application (PD). Samples were analysed for mesotrione residues using the method RAM 366. The results are summarized in Table 75.

Table 75 Summary of residue trials of mesotrione on okra (pods) using SC formulation in USA

State, trial	Okra variety	Application rate, kg	Growth stage	DAT (days)	Residues (mg/kg)
North Carolina, SJ-HR05-6260	Clemson Spineless	0.225 (SS)	00	73	< 0.01 (2)
		0.106 (POT)	2-3 leaf	45	< 0.01 (2)
		0.105 (PD)	(38) 10 leaf stage	28	< 0.01 (2)
		0.229 (SS) + 0.105 (POT)	00 + 2-3 leaf	45	< 0.01 (2)
		0.228 (SS) + 0.105 (PD)	00 + (36) 9 leaf stage	28	<u>< 0.01</u> (2)
Florida, VQ-HR05-6261	Clemson Spineless	0.105 (POT)	12 to 13	45	< 0.01 (2)
Mississippi, 3A-HR2005-6262	Perkins Long	0.109 (POT)	13	45	< 0.01 (2)
		0.106 (PD)	pre-bloom	28	<u>< 0.01</u> (2)
		0.232 (SS) + 0.105 (POT)	00 + 13	45	< 0.01 (2)
		0.233 (SS) + 0.103 (PD)	00 + pre-bloom	28	< 0.01 (2)
Texas, 3A-HR2005-6262	Louisiana Green Velvet	0.228 (SS)	00	84	< 0.01 (2)
				98	< 0.01 (2)
				104	< 0.01 (2)
				112	< 0.01 (2)
				119	< 0.01 (2)
		0.108 (POT)	89	0	0.19, 0.16
				15	< 0.01 (2)
				30	<u>< 0.01</u> (2)
				45	< 0.01 (2)
				52	< 0.01 (2)
		0.107 (PD)	89	0	< 0.01 (2)
				14	< 0.01 (2)
				21	< 0.01 (2)
				28	<u>< 0.01</u> (2)
				35	< 0.01 (2)
		0.228 (SS) + 0.105 (POT)	00 + 89	0	0.09, 0.2
				15	< 0.01 (2)
				30	< 0.01 (2)
				45	< 0.01 (2)
				52	< 0.01 (2)

State, trial	Okra variety	Application rate, kg)	Growth stage	DAT (days)	Residues (mg/kg)
		0.223 (SS) + 0.106 (PD)	00 + 89	0 14 21 28 35	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)
Oklahoma, SC-HR2005-6264	Clemson Spineless	0.220 (SS)	00	NA	< 0.01 (2)
		0.105 (POT)	13	45	< 0.01 (2)
		0.107 (PD)	53	28	< 0.01 (2)
		0.214 (SS) + 0.105 (POT)	00 + 13	45	< 0.01 (2)
		0.224 (SS) + 0.104 (PD)	00 + 59	28	< 0.01 (2)

SS=soil surface spray

POT=post over-the-top spray

PD=post directed application

Sweet corn

Eight post-emergence trials were conducted on sweet corn in France in 2003/2004, and 18 trials in the USA during 2001 and 2008, with either one pre-emergence and one post-emergence application or with two early post-emergence applications. Samples were analysed for mesotrione and MNBA using method RAM 366/1 (LC-MS/MS) or TM0643B (HPLC-FL). The data for mesotrione residue are summarised in Table 76. Residue levels for MNBA were < 0.01 mg/kg in all cases.

Table 76 Residue of mesotrione after the use of mesotrione on sweet corn

Country (Region) year	Sweet corn variety	Application rate (kg ai/ha) formulation	Growth Stage	DAT (days)	Crop Part	Residue (mg/kg)	Report; trial year
FRANCE 2003	620 Spirit	0.102 SC	BBCH 19	38	kernels	< 0.01	03-7049
				38	cob	< 0.01	
	620 Spirit	0.153 SC	BBCH 12	38	kernels	< 0.01	
				38	cob	< 0.01	
				61	kernels	< 0.01	
				61	cob	< 0.01	
FRANCE 2003	620 Spirit	0.98 SC	BBCH 19	38	kernels	< 0.01	03-7050
				38	cob	< 0.01	
	620 Spirit	0.143 SC	BBCH 12	62	kernels	< 0.01	
				62	cob	< 0.01	
				62	kernels	< 0.01	
				62	cob	< 0.01	
FRANCE 2004	620 Spirit	0.153 SC	BBCH 12	61	kernels	< 0.01	04-7012
				61	cob	< 0.01	
				74	kernels	< 0.01	
				74	cob	< 0.01	
FRANCE 2004	620 Spirit)	0.154 SC	BBCH 12	61	kernels	< 0.01	04-7012
				61	cob	< 0.01	
				74	kernels	< 0.01	
				74	cob	< 0.01	
	620 Spirit	0.103 SC	BBCH 59	39	kernels	< 0.01	
				39	cob	< 0.01	
USA (Wisconsin) 2001	NK 199	0.302 + 0.177 SC	pre-emergence + Vt (tassel fully emerged)	30	ears	< 0.01 (2)	487-01; NI-HR001-01
USA (Washington) 2001	Jubilee	0.303 + 0.179 SC		28	ears	< 0.01 (2)	487-01; WF-HR003-01
USA (California) 2001	Silver Queen	0.300 + 0.182 SC	BBCH 01 + BBCH 59	23	ears	< 0.01 (2)	487-01; W2-HR102-01
				30	ears	< 0.01 (2)	
				37	ears	< 0.01 (2)	

Country (Region) year	Sweet corn variety	Application rate (kg ai/ha) formulation	Growth Stage	DAT (days)	Crop Part	Residue (mg/kg)	Report; trial year
USA (N. Carolina) 2001	G90 F1	0.308 + 0.182 SC	BBCH 55	26	ears	< 0.01 (2)	487-01; SJ-HR003-01
USA (Ohio) 2001	Bodacious	0.314 + 0.190 SC		30	ears	< 0.01 (2)	487-01; NK-HR001-01
USA (New York) 2001	GH-2783	0.305 + 0.182 SC	at planting + 8–9 leaves	30	ears	< 0.01 (2)	487-01; EE-HR003-01
USA (Idaho) 2001	Sugar Buns	0.303 + 0.179 SC	post planting + pollen shed	30	ears	< 0.01 (2)	487-01; WG-HR005-01
USA (Illinois) 2001	Kandy King	0.301 + 0.186 SC	BBCH 00 + BBCH 34	31	ears	< 0.01 (2)	487-01; N4-HR003-01
USA (Pennsylvania) 2001	Argent	0.320 + 0.186 SC	BBCH 00 + early tassel	30	ears	< 0.01 (2)	487-01; EC-HR002-01
USA (Florida) 2001	Silver Queen	0.313 + 0.177 SC	Just planted + visible tassel	28	ears	< 0.01 (2)	487-01; VB-HR101-01
USA (Michigan) 2001	Excellency	0.306 + 0.180 SC	pre-emergence + Vt	23	ears	< 0.01 (2)	487-01; ED-HR006-01
				30	ears	< 0.01 (2)	
				36	ears	< 0.01 (2)	
USA (Maine) 2001	Maple Sweet	0.304 + 0.189 SC	0 + tassel initiation	30	ears	< 0.01 (2)	487-01; NF-HR002-01
USA (N. Carolina) 2008	Rogers WH0809	0.107 + 0.106 SC	BBCH 36 + BBCH 37	32	ears	< 0.01 (2)	T001589-08; E10NC081871
		0.104 + 0.104 WG	BBCH 36 + BBCH 37	32	ears	< 0.01 (2)	
USA (N. Dakota) 2008	Peaches & Cream	0.107 + 0.105 SC	BBCH 16 + BBCH 55	28	ears	< 0.01 (2)	T001589-08; C13ND081872
		0.107 + 0.106 WG	BBCH 16 + BBCH 55	28	ears	< 0.01 (2)	
USA (California) 2008	Sweetie 82	0.103 + 0.107 SC	BBCH 15 + BBCH 17	45	ears	≤ 0.01 (2)	T001589-08; W30CA081873
		0.105 + 0.105 WG	BBCH 15 + BBCH 17	45	ears	< 0.01 (2)	

Soya bean, dry

Twenty three supervised residue trials were conducted on soya bean in the USA during 2007 (Report T005595-06), with mesotrione applied pre-emergence direct to the soil. The DAT is driven by the normal vegetation period of the crop and is in the range of 113 to 160 days. Samples were analysed for mesotrione and MNBA using RAM 366/1. The results for mesotrione are shown in Table 77. MNBA residues were < 0.01 mg/kg in all samples.

Table 77 Summary of residue data for mesotrione (SC formulation) on soya bean seed from trials conducted in the USA in 2007

State (Location)	Soya bean variety	Application rate, kg ai/ha	Growth Stage	DAT (days)	Residues (mg/kg)	Trial
North Carolina	S56-D7	0.212	BBCH 00	158	≤ 0.01 (2)	E10NC078240
South Carolina	S76-L9	0.215	BBCH 00	160	≤ 0.01 (2)	E11SC078241
Arkansas (New Port)	S56-D7	0.213	BBCH 00	137	≤ 0.01 (2)	C23AR078242
Louisiana	S56-D7	0.214	BBCH 00	140	≤ 0.01 (2)	E17LA078243
Arkansas (Proctor)	Garst 3512RR/	0.213	BBCH 00	113	≤ 0.01 (3)	C24AR078244
		1.07	BBCH 00	113	< 0.01 (3)	
Iowa (Richland)	S29-C)	0.212	BBCH 00	127	< 0.01 (2)	C18IA078245
				134	< 0.01 (2)	
				141	< 0.01 (2)	

State (Location)	Soya bean variety	Application rate, kg ai/ha	Growth Stage	DAT (days)	Residues (mg/kg)	Trial
Iowa (Ollie)	Garst 3512 R/N	0.217	BBCH 00	152	≤ 0.01 (2)	C18IA078246
Illinois (Carlyle)	3512RR/N	0.213	BBCH 00	134	≤ 0.01 (2)	C06IL078247
		0.668	BBCH 00	134	< 0.01 (2)	
		1.111	BBCH 00	134	< 0.01 (2)	
Illinois (Wyoming)	S29-C9	0.214	BBCH 00	147	≤ 0.01 (2)	C06IL078248
Minnesota	CL081215	0.215	BBCH 00	126	≤ 0.01 (2)	C09MN078249
				133	< 0.01 (2)	
				140	< 0.01 (2)	
Indiana (Sheridan)	S29-C9	0.216	BBCH 00	141	≤ 0.01 (2)	C05IN078250
Indiana (Frankfort)	S29-C9	0.215	BBCH 00	141	≤ 0.01 (2)	C05IN078251
Missouri	S29-C9 RR	0.210	BBCH 00	122	≤ 0.01 (2)	C19MO078252
Nebraska	NK S19-R5 RR	0.218	BBCH 00	117	≤ 0.01 (2)	C17NE078253
Ohio	3512 RR/N	0.220	BBCH 00	126	≤ 0.01 (2)	C02OH078254
				134	< 0.01 (2)	
				139	< 0.01 (2)	
South Dakota	S19-R5	0.215	BBCH 01	119	≤ 0.01 (2)	C16SD078255
Kansas	04RM820605	0.212	BBCH 00	174	≤ 0.01 (2)	C22KS078256
Michigan	S19-R5	0.214	BBCH 00	156	≤ 0.01 (2)	C03MI078257
North Dakota	S10-T1	0.215	BBCH 00	140	≤ 0.01 (2)	C13ND078258
Wisconsin	CL081215	0.213	BBCH 00	127	≤ 0.01 (2)	C08WI078259

Mesotrione-tolerant soya bean, dry

Forty-nine supervised residue trials were conducted on herbicide-tolerant soya bean in the USA during 2009 and 2012 with either one pre-emergence and one post-emergence application at growth stage R1 (BBCH 60) or with one early post-emergence application at growth stage V2 (BBCH 13). Samples were analysed for mesotrione and MNBA using RAM 366/1. The results for mesotrione are shown in Table 78. MNBA residues were < 0.01 mg/kg in all samples.

Table 78 Summary of residue data for mesotrione on herbicide-tolerant soya bean (SC formulation)

State (location) year	HT Soya bean variety	Application rate (kg ai/ha)	Growth Stage at Application	DAT (days)	Portion	Residues (mg/kg)	Report; Trial
South Carolina 2009	Jack/ SYHT04R	0.226 + 0.125	BBCH 00 + 60	89	Seed (m)	≤ 0.01 (2)	T000908-07; E11-9652
		0.225	BBCH 14	98	Seed (m)	≤ 0.01 (2)	
Arkansas 2009	Jack/ SYHT04R	0.226 + 0.125	BBCH 00 + 60	50 76	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07; C24-9653
Louisiana 2009		0.226	BBCH 13	59 79 85 92 99	Seed Seed (– 7d) Seed (m) Seed (+7d) Seed (+14d)	< 0.01 (2) < 0.01 ≤ 0.01 (2) < 0.01 < 0.01	
Missouri (Fisk) 2009	Jack/ SYHT04R	0.234 + 0.125	BBCH 00 + 61	45	Seed (m)	≤ 0.01 (2)	T000908-07; E18-9654
		0.234	BBCH 12	66	Seed (m)	≤ 0.01 (2)	
Iowa (Richland) 2009	Jack/SYHT04R	0.225 + 0.125	BBCH 00 + 63	49 58	Seed Seed (m)	< 0.01 (2) 0.02 (2)	T000908-07 C23-9655
		0.226 + 0.124	BBCH 00 + 63	49 58	Seed Seed (m)	0.01, 0.02 0.02 (2)	
				0.225	BBCH 12	67 76	
		Missouri (La Plata) 2009	Jack/SYHT04R	0.226 + 0.126	BBCH 00 + 16	48 102	
0.222	BBCH 12.5			97 151	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
				1.130 + 0.623	BBCH 00 + 16	102	Seed (m)

State (location) year	HT Soya bean variety	Application rate (kg ai/ha)	Growth Stage at Application	DAT (days)	Portion	Residues (mg/kg)	Report; Trial
Iowa (Miles) 2009	Jack/SYHT04R	0.228 + 0.123	BBCH 0 + 18	45 85	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 C18-0657
		0.224	BBCH 12	66 106	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Iowa (Bagley) 2009	Jack/SYHT04R	0.227 + 0.122	BBCH 0 + 59	47 90	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 C18-0658
		0.223 +	BBCH 12	73 116	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Iowa (Berkley) 2009	Jack/SYHT04R	0.226 + 0.117	BBCH 00 + 63	45 76	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 C30-9659
		0.222	BBCH 12	77 108	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
		1.147 + 0.640	BBCH 00 + 63	76	Seed (m)	0.04 (3)	
Missouri (Oregon) 2009	Jack/ SYHT04R	0.233 + 0.118	BBCH 00 + 63	43 75	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 C30-9660
		0.224	BBCH 12	70 102	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Kansas 2009	Jack/ SYHT04R	0.226 + 0.121	BBCH 0 + 60- 65	47 69	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 2 C19-9661
		0.225	BBCH 13	82 93 104 110 117	Seed Seed Seed (m) Seed Seed	< 0.01 (2) < 0.01 ≤ 0.01 (2) < 0.01 < 0.01	
Michigan 2009	Jack/ SYHT04R	0.230 + 0.121	BBCH 00 + 60–65	47 70	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 C19-9662
		0.229	BBCH 13	76 99	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Missouri (Dudley) 2009	Jack/ SYHT04R	0.224 + 0.124	BBCH 00 + 60	45 76	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 C03-9663
		0.225	BBCH 13	81 112	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Nebraska (Stromsburg) 2009	HT Soya bean/ Jack/ SYHT04R	228 128	BBCH 00 + 61	47 55	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 C23-9664
		0.223	BBCH 12	64 72	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Nebraska (Hampton) 2009	HT Soya bean/ Jack/ SYHT04R	0.228 + 0.123	BBCH 00 + 61	46 85	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 E13-9665
		0.227	BBCH 12	76 115	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
North Carolina (York) 2009	Jack/ SYHT04R	0.225 + 0.125	BBCH 00 + 61	48 94	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 E13-9666
		0.231	BBCH 12	83 129	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Nebraska (York) 2009	Jack/ SYHT04R	0.219 + 0.123	BBCH 00 + 61	49 86	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 E13-9667
		0.230	BBCH 12	76 113	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Ohio (Richwood) 2009	Jack/ SYHT04R	0.227 + 0.125	BBCH 00 + 61	47 87	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 C01-9668
		0.226	BBCH 12	66 106	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Ohio (Marysville) 2009	Jack/ SYHT04R	0.227 + 0.124	BBCH 00 + 61	47 87	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 C01-9669
		0.230	BBCH 12	67 107	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Iowa (Lime Springs) 2009	Jack/ SYHT04R	0.222 + 0.126	BBCH 00 + 61	46 123	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 E19-9670
		0.231	BBCH 12	68 145	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	

State (location) year	HT Soya bean variety	Application rate (kg ai/ha)	Growth Stage at Application	DAT (days)	Portion	Residues (mg/kg)	Report; Trial
North Carolina (Seven springs) 2009	Jack/SYHT04R	0.226 + 0.125	BBCH 00 + 62	50 79	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	T000908-07 E10-9671
		0.223	BBCH 13	67 96	Seed Seed (m)	< 0.01 (2) ≤ 0.01 (2)	
Iowa (Richland) 2012	Jack/SYHT0H2	0.225 + 0.126	BBCH 00 + 60	94	Seed (m)	≤ 0.01 (2)	TK0112226 -1
Iowa (Bagley) 2012	Jack/SYHT0H2	0.237 + 0.125	BBCH 00 + 60	90	Seed (m)	0.02 (2)	TK0112226 -2
Iowa (Lime Spring) 2012	Jack/SYHT0H2	0.228 + 0.122	BBCH 00 + 60	97	Seed (m)	≤ 0.01 (2)	TK0112226 -3
Minneapolis 2012	Jack/SYHT0H2	0.225 + 0.127	BBCH 00 + 60	106	Seed (m)	≤ 0.01 (2)	TK0112226 -4
Nebraska 2012	Jack/SYHT0H2	0.228 + 0.126	BBCH 00 + 60	79	Seed (m)	≤ 0.01 (2)	TK0112226 -5
Iowa (Atlanta) 2012	Jack/SYHT0H2	0.235 + 0.127	BBCH 00 + 61-62	77	Seed (m)	≤ 0.01 (2)	TK0112226 -6

(m)—Mature

Asparagus

Twenty four supervised residue trials were conducted on asparagus in the USA (Report T021572-04 2005) during 2005 using either one pre-emergence soil surface spray (PSS), post-emergence over-the-top of the weeds (POT) or a combination of the two applications. Samples were analysed for mesotrione using RAM 366/1. The results are shown in Table 79.

Table 79 Summary of residue data for mesotrione on asparagus (spears) using SC formulation in the USA during 2005

State (location)	Asparagus variety	Application (kg ai/ha)	Growth Stage at Application	DAT (days)	Residue (mg/kg)	Trial
North Caroline	Jersey Knight	0.276 (PSS)	BBCH 00	9	≤ 0.01 (2)	SJ-HR-05-6270
		0.106 (POT)	emerged	2	0.09 (2)	
		0.278 (PSS) + 0.105 (POT)	BBCH 00 + emerged	2	0.05, 0.18	
Michigan (Conklin)	Centennial	0.266 (PSS)	BBCH 00	13	≤ 0.01 (2)	NL-HR-05-6271
				14	< 0.01 (2)	
				15	< 0.01 (2)	
				16	< 0.01 (2)	
		0.106 (POT)	mature spears	0	0.61, 0.87	
				1	0.17, 0.21	
Michigan (Comstock)	Jersey Giant	0.271 (PSS) + 0.105 (POT)	BBCH 00 + mature spears	2	0.08, 0.06	NL-HR-05-6272
				3	0.05, 0.04	
				0	0.59, 0.66	
				1	0.14, 0.15	
		0.270 (PSS)	BBCH 00	2	0.09, 0.06	
				3	0.05, 0.07	
California (Gonzales)	UC 157	0.270 (PSS) + 0.105 (POT)	BBCH 00 + mature	2	0.25, 0.21	WC-HR-05-6273
		0.265 (PSS)	BBCH 00	25	≤ 0.01 (2)	
		0.104 (POT)	market size spears	2	0.05, 0.03	
California (Walnut Grove)	UC157	0.270 (PSS) + 0.105 (POT)	BBCH 00 + mature	2	0.04, 0.03	WD-HR-05-6274
		0.267 (PSS)	BBCH 08	11	≤ 0.01 (2)	
		0.106 (POT)	BBCH 45	3	≤ 0.01 (2)	
California (Fire)	UC 157	0.268 (PSS) + 0.105 (POT)	BBCH 08 + BBCH 45	3	< 0.01 (2)	WC-HR-05-6274
		0.275 (PSS)	BBCH 00	12	≤ 0.01 (2)	
		0.108 (POT)	emerging spears	2	0.13, 0.14	

State (location)	Asparagus variety	Application (kg ai/ha)	Growth Stage at Application	DAT (days)	Residue (mg/kg)	Trial
Baugh)		0.280 (PSS) + 0.109 (POT)	BBCH 00 + emerging	2	0.16, 0.13	6275
Oregon	Martha Washington	0.274 (PSS)	BBCH 00	8	< 0.01 (2)	WF-HR-05-6277
		103 (POT)	BBCH 09	2	0.03, 0.05	
		0.274 (PSS) + 0.103 (POT)	BBCH 00 + BBCH 09	2	0.05, 0.08	
Washington	902-62	0.272 (PSS)	BBCH 00	10	< 0.01 (2)	WF-HR-05-6278
		0.107 (POT)	spears 2-12" tall	2	0.03 (2)	
		0.266 (PSS) + 0.106 (POT)	BBCH 00 spears 2-12" tall	2	0.04, 0.04	

PSS: Pre-emergence soil surface spray

POT: post-emergence over-the-top of the weeds

Rhubarb

Eight supervised residue trials were conducted on rhubarb in the USA (Report T014372-05) during 2006 with one application performed pre-emergence. The results are shown in Table 80.

Table 80 Summary of residue data for the use of mesotrione on rhubarb from trials conducted in USA in 2006 using SC formulation

Country (Region)	Rhubarb variety	Application rate (kg ai/ha)	Growth Stage at Application	DAT (days)	Residues (mg/kg)
Illinois		0.202	BBCH 00	28	< 0.01 (2)
				35	< 0.01 (2)
				42	< 0.01 (2)
				47	< 0.01 (2)
		0.350	BBCH 00	28	0.01, < 0.01
				35	< 0.01 (2)
				42	< 0.01 (2)
				47	< 0.01 (2)
Michigan	McDonald	0.212	pre-emergence	42	< 0.01 (2)
		0.340	pre-emergence	42	< 0.01 (2)
Oregon	Crimson	0.210	dormant	42	< 0.01 (2)
		0.343	dormant	42	< 0.01 (2)
Washington	RubyRed	0.217	BBCH 08	42	< 0.01 (2)
		0.337	BBCH 08	42	< 0.01 (2)

Field corn (maize)

Three trials with field corn were conducted in Europe in 2009, 24 in Canada in 1996/1997 and 42 in the USA in 1995 or 2001 with either one pre-emergence and one post-emergence application or with two early post-emergence applications. The results on maize grain are shown in Table 81.

Table 81 Summary of residue data for the use of mesotrione on maize (grain)

Country (Region) year	Maize variety	Application			DAT (days)	Residues (mg/kg)	Report trial
		Formulation	Rate (kg ai/ha)	Growth Stage			
Canada (Ontario) 1996	Pioneer 3902	SC	0.300	Pre-emergence	123	< 0.01	RR 97-043B 94-CN-96-201
		SC	0.600	Pre-emergence	123	< 0.01	
		SC	0.200	5-6 Leaf	108	< 0.01	
		SC	0.400	5-6 Leaf	108	< 0.01	
		SC	0.300 0.200	Pre-emergence 5-6 leaf	108	< 0.01	
		SC	0.600 0.400	Pre-emergence 5-6 leaf	108	< 0.01	
Canada (Ontario) 1996	Ciba seeds G-4064	SC	0.300	Pre-emergence	122	< 0.01	RR 97-043B 94-CN-96-202
		SC	0.600	Pre-emergence	122	< 0.01	
		SC	0.200	4-5 Leaf stage	111	< 0.01	

Country (Region) year	Maize variety	Application			DAT (days)	Residues (mg/kg)	Report trial
		Formulation	Rate (kg ai/ha)	Growth Stage			
		SC	0.400	4–5 Leaf stage (	111	< 0.01	
		SC	0.300 0.200	Pre-emergence 4–5 leaf	111	< 0.01	
		SC	0.600 0.400	Pre-emergence 4–5 leaf	111	< 0.01	
Canada (Ontario) 1997	Funks-BT Maximizer	SC	0.300	Pre-emergence	155	< 0.01	RR 98-035B 94-CN-97-901
		SC	0.600	Pre-emergence	155	< 0.01	
		SC	0.200	24	126	< 0.01	
		SC	0.400	24	126	< 0.01	
		SC	0.300 0.200	Pre-emergence 24	126	< 0.01	
		SC	0.600 0.400	Pre-emergence 24	126	< 0.01	
Canada (Ontario) 1997	Funks-BT Maximizer	SC	0.300	Pre-emergence	145	< 0.01	RR 98-035B 94-CN-97-902
		SC	0.600	Pre-emergence	145	< 0.01	
		SC	0.200	23	126	< 0.01	
		SC	0.400	23	126	< 0.01	
		SC	0.300 0.200	Pre-emergence 23	126	< 0.01	
			0.600 0.400	Pre-emergence 23	126	< 0.01	
Germany 2009	Nescio	WG	0.138	BBCH 16–18	82 91 143	≤ 0.01 < 0.01 < 0.01	T000920-09- REG
Spain 2009	Castellano	WG	0.154	BBCH 15	42 71 82	< 0.01 < 0.01 < 0.01	T000921-09- REG
United Kingdom 2009	Ohio	WG	0.142	BBCH 16–17	98 112	≤ 0.01 < 0.01	T000920-09- REG
USA (Iowa, Sheffield), 1995	ICI 8543	SC	0.336 0.224	Pre-plant vegetative	109	< 0.01 (2)	RR 96-018B 63-IA-95-805
USA (Illinois, Brimfield), 1995	Hoblit 428	SC	0.336 0.224	pre-emergence	95	< 0.01 (2)	RR 96-018B 60-IL-95-806
USA (Indiana, Lafayette), 1995	Pioneer 3394	SC	0.336 0.224	pre-emergence 7–9 leaves	96	< 0.01 (2)	RR 96-018B 67-IN-95-807
USA (Michigan) 1995	NK 4640	SC	0.336 0.224	pre-bloom	114	< 0.01 (2)	RR 96-018B 24-MI-95-808
USA (Nebraska) 1995	Ottile RO 2455	SC	0.336 0.224	Pre-plant V6	88	< 0.01 (2)	RR 96-018B 68-NE-95-809
USA (Iowa, Albia), 1995	ICI 8532	SC	0.336 0.224	Pre-plant vegetative	102	< 0.01 (2)	RR 96-018B 63-IA-95-810
USA (Illinois, Towanda), 1995	Ainsworth 640	SC	0.336 0.224	Pre-plant post emergence	87	< 0.01 (2)	RR 96-018B 60-IL-95-811
USA (Indiana, Rochester), 1995	Pioneer 3394	SC	0.336 0.224	Early pre-plant 5–7 leaves	96	< 0.01 (2)	RR 96-018B 67-IN-95-812
USA (Nebraska, Waverly), 1995	Producers PH785	SC	0.336 0.224	Pre-plant V5–V6	96	< 0.01 (2)	RR 96-018B 68-NE-95-814
USA (Kansas, La Cygnet), 1995	CIBA 4575	SC	0.336 0.224	early pre-plant post	68	< 0.01 (2)	RR 96-018B 37-KS-95-825
USA (Pennsylvania, Ephrata), 1995	CI 8541	SC	0.336 0.224	Pre-emergence vegetative	98	< 0.01 (2)	RR 96-018B 70-PA-95-815
		SC	0.336 0.224	pre-emergence vegetative	98	< 0.01 (2)	
USA (Minnesota, St. Peter)	Cenex 424	SC	0.336 0.224	Pre-emergence vegetative	92	< 0.01 (2)	RR 96-018B 36-MN-95-816

Country (Region) year	Maize variety	Application			DAT (days)	Residues (mg/kg)	Report trial
		Formulation	Rate (kg ai/ha)	Growth Stage			
1995		SC	0.336 0.224	Pre-emergence vegetative	92	< 0.01 (2)	
USA (Ohio, Urbana) 1995	Vigoro V1122	SC	0.336 0.224	Pre-emergence	113	< 0.01 (2)	RR 96-018B 24-OH-95-817
		SC	0.336 0.224	Pre-emergence	113	< 0.01 (2)	
USA (Wisconsin, Baraboo) 1995	Cenex LOL 357	SC	0.336 0.224	Pre-emergence	104	< 0.01 (2)	RR 96-018B 79-WI-95-818
		SC	0.336 0.224	Pre-emergence	104	< 0.01 (2)	
USA (Texas) 1995	G4673B (D&PL Co.)	SC	0.336 0.224	Pre-emergence pre-tassel	92	< 0.01 (2)	RR 96-018B 25-TX-95-819
		SC	0.336 0.224	Pre-emergence pre-tassel	92	< 0.01 (2)	
USA (North Carolina) 1995	Field corn (Pioneer 3165)	SC	0.336 0.224	Pre-plant rapidly growing	95	< 0.01 (2)	RR 96-018B 01-NC-95-820
		SC	0.336 0.224	Pre-plant rapidly growing	88 95 101 108	< 0.01 < 0.01 (2) < 0.01 < 0.01	
USA (Iowa, Boone) 1995	ICI 8543	SC	0.336 0.224	Early pre-plant vegetative	112	< 0.01 (2)	RR 96-018B 63-IA-95-821
		SC	0.336 0.224	Early pre-plant vegetative	112	< 0.01 (2)	
USA (Illinois, Champagne) 1995	Pioneer 3394	SC	0.336 0.224	Early pre-plant post emergence	107	< 0.01 (2)	RR 96-018B 04-IL-95-822
		SC	0.336 0.224	Pre-plant post-emergence	100 107 113 121	< 0.01 < 0.01 (2) < 0.01 < 0.01	
USA (Indiana, New Richmond) 1995	Pioneer 3394	SC	0.336 0.224	Early pre-plant 6-8 leaf	86	< 0.01 (2)	RR 96-018B 67-IN-95-823
		SC	0.336 0.224	Early pre-plant 6-8 leaf	86	< 0.01 (2)	
USA (Nebraska, Crete) 1995	ICI 8541	SC	0.336 0.224	Early pre-plant V6-V7	89	< 0.01 (2)	RR 96-018B 68-NE-95-824
		SC	0.336 0.224	Pre-plant V6-V7	89	< 0.01 (2)	
USA Iowa) 2001	Pioneer 34B23)	SC	0.268 0.225	BBCH 00 BBCH 18-19	99	< 0.01 (2)	1847-01 NE-HR-003-01
		SE SC	0.267 0.233	BBCH 00 BBCH 18/19	99	< 0.01 (2)	
		SC	0.274 0.234	BBCH 13 BBCH 18/19	99	< 0.01 (2)	
		SE SC	0.267 0.223	BBCH 13 BBCH 18/19	99	< 0.01 (2)	
USA (North Carolina)	ICI 8543)	SC	0.269 0.226	BBCH 00 75 cm high	98	< 0.01 (2)	1847-01

Country (Region) year	Maize variety	Application			DAT (days)	Residues (mg/kg)	Report trial
		Formulation	Rate (kg ai/ha)	Growth Stage			
2001		SE	0.269	BBCH 00	98	< 0.01	SJ-HR-013-01
		SC	0.227	75 cm high		(2)	
		SC	0.271	BBCH 12/13	98	< 0.01	
			0.227	75 cm high		(2)	
		SE	0.270	BBCH 12/13	98	< 0.01	
USA (Illinois) 2001	Pioneer 34B24	SC	0.285	BBCH 00	112	< 0.01	1847-01 N4-HR-005-01
			0.253	BBCH 36		(2)	
		SE	0.278	BBCH 00	112	< 0.01	
		SC	0.236	BBCH 36		(2)	
		SC	0.286	BBCH 12	112	< 0.01	
USA (Nebraska) 2001	ICI 8543		0.244	BBCH 36		(2)	
		SE	0.268	BBCH 12	112	< 0.01	1847-01 NB-HR-004-01
		SC	0.237	BBCH 36		(2)	
		SC	0.270	BBCH 00	103	< 0.01	
			0.227	BBCH 19		(2)	
		SE	0.273	BBCH 00	103	< 0.01	
		SC	0.227	BBCH 19		(2)	
		SC	0.266	BBCH 12/13	103	< 0.01	NB-HR-004-01
			0.226	BBCH 19		(2)	
		SE	0.268	BBCH 12/13	103	< 0.01	
		SC	0.226	BBCH 19		(2)	

Millet

Fifteen supervised residue trials were conducted on millet in the USA during 2004 (Report T010289-04) with one application performed pre-emergence. The results on millet grain are shown in Table 82.

Table 82 Summary of residue data for the use of mesotrione on millet (grain) using SC formulation in the USA in 2004

State	Millet variety	Application Rate (g ai/ha)	Growth Stage	PHI (days)	Residue (mg/kg)	Trial number
Illinois	Max Perl	0.109	at planting	130	< 0.01 (2)	4A-HR-04- 5640
		0.214	at planting	130	< 0.01 (2)	
		0.108	BBCH 32	113	< 0.01 (2)	
Nebraska	Huntsman	0.103	at planting	84	< 0.01 (2)	4A-HR-04- 5641
		0.210	at planting	84	< 0.01 (2)	
		0.103	BBCH 12	61	< 0.01 (2)	
South Dakota	Rise	0.104	at planting	95	< 0.01 (2)	4A-HR-04- 5642
		0.208	at planting	95	< 0.01 (2)	
		0.105	BBCH 21	69	< 0.01 (2)	
Colorado (Ault) ^a	Early Bird	0.107	at planting	132	< 0.01 (2)	4A-HR-04- 5643
		0.212	at planting	132	< 0.01 (2)	
		0.107	vegetative	92	< 0.01 (2)	
Colorado (Ault) ^a	Early Bird	0.110	at planting	104	< 0.01 (2)	4A-HR-04- 5644
				111	< 0.01 (2)	
				120	< 0.01 (2)	
		0.218	at planting	104	< 0.01 (2)	
				111	< 0.01 (2)	
				120	< 0.01 (2)	
		0.108	vegetative	64	< 0.01 (2)	
				71	< 0.01 (2)	
				80	< 0.01 (2)	

^a Trials conducted at the same site during the same period. Only one was considered

Oat

Thirty four supervised residue trials were conducted in oat in the USA (Report T004407-05) during 2005 with either one pre-emergence or one post-emergence application. The results on oat grain are shown in Table 83.

Table 83 Summary of residue data for the use of mesotrione on oat (grain) using SC formulation in 2005 in the USA

State (location)	Oat variety	Application rate (kg ai/ha)	Growth Stage	DAT (days)	Residues (mg/kg)	Trial
New York	Armor	0.214	BBCH 00	90	< 0.01 (2)	5A-HR-05-6380
		0.104	BBCH 59–61	49	< 0.01 (2)	
Virginia	Coker	0.216	BBCH 00	246	< 0.01 (2)	SJ-HR-05-6381
		0.108	BBCH 75	51	< 0.01 (2)	
North Dakota (Northwood)	Morton	0.211	bare soil	90	< 0.01 (2)	NN-HR-05-6382
		0.104	BBCH 23	50	< 0.01 (2)	
South Dakota (Lesterville)	Jerry	0.209	BBCH 01	84	< 0.01 (2)	NF-HR-05-6383
		0.106	BBCH 32	54	< 0.01 (2)	
Wisconsin	Esker	0.216	pre-emergence	91	< 0.01 (2)	NI-HR-05-6384
		0.106	late boot -early head	49	< 0.01 (2)	
Minnesota	Drumlin	0.209	BBCH 00	80	< 0.01 (2)	NF-HR-05-6385
		0.107	BBCH 21	52	< 0.01 (2)	
Nebraska (York)	Jerry	0.213	BBCH 00	87	< 0.01 (2)	NB-HR-05-6386
		0.105	BBCH 31	49	< 0.01 (2)	
Iowa	Jerry	0.213	BBCH 00	96	< 0.01 (2)	NE-HR-05-6387
		0.106	BBCH 80	49	< 0.01 (2)	
Kansas (Sabetha)	Loyal	0.210	pre-emergence	83	< 0.01 (2)	ND-HR-05-6388
		0.107	1–3 tillers	50	< 0.01 (2)	
Michigan	Prairie	0.213	pre-emergence	88	< 0.01 (2)	NL-HR-05-6389
		0.104	5–6 leaves	50	< 0.01 (2)	
Illinois	VHS	0.209	BBCH 00	81	< 0.01 (2)	4A-HR-05-6390
		0.104	BBCH 26	52	< 0.01 (2)	
		0.317	BBCH 26	52	< 0.01 (2)	
		0.531	BBCH 26	52	< 0.01 (2)	
Texas	La-604	0.209	BBCH 00	180	< 0.01 (2)	SA-HR-05-6391
		0.106	BBCH 43	54	< 0.01 (2)	
North Dakota (New Rockford)	La-604	0.211	soil surface spray	92	< 0.01 (2)	NM-HR-05-6392
		0.104	BBCH 43	49	< 0.01 (2)	
South Dakota (Frederick)	Morton	0.211	BBCH 01	81	< 0.01 (2)	NF-HR-05-6393
		0.106	BBCH 21–31	51	< 0.01 (2)	
Nebraska (Grand Island)	Reeves	0.215	BBCH 00	90	< 0.01 (2)	NB-HR-05-6394
		0.105	BBCH 31	49	< 0.01 (2)	
Kansas (Larned)	Jerry	0.210	pre-emergence	83	< 0.01 (2)	NM-HR-05-6395
		0.107	1–2 tillers	50	< 0.01 (2)	

Rice

Two residue trials were conducted in rice in Korea in 2005 and eight in Japan in 2004 and 2006 using one or two applications into the water, post-transplanting. None of the trials were under GLP. The results in brown rice (husked rice) are shown in Table 84.

Table 84 Summary of residue data for mesotrione on rice (brown rice) after application using a granular formulation

Country, year	Rice variety	Application rate (g ai/ha)	Growth Stage	DAT (days)	Residue (mg/kg)	Trial
Korea 2005		0.180	5–15 days after transplanting	140	< 0.05 (3)	A14928B
		0.360	5–15 days after transplanting	140	< 0.05 (3)	

Country, year	Rice variety	Application rate (g ai/ha)	Growth Stage	DAT (days)	Residue (mg/kg)	Trial
Japan, 2004	Matsuribare	0.100	21 days after transplanting	91	< 0.002 (2)	A137723B
Japan, 2004	Hi-no-hikari	0.100	21 days after transplanting	89	< 0.002 (2)	A137723B
Japan 2006	Koshihikari	0.100	4.5 leaf period	76	< 0.002 (2)	A137723B
		0.100	growing period	61	< 0.002 (2)	
		0.100	growing period	45	< 0.002 (2)	
Japan 2006	Hi-no-hikari	0.100	4.5 leaf period before harvest	45	< 0.002 (2)	A137723B
		0.100	10 days after plant	75	< 0.002 (2)	
		0.100	75 days before harvest	60	< 0.002 (2)	
		0.100	10 days after plant	45	< 0.002 (2)	
		0.100	60 days before harvest			
		0.100	10 days after plant			
		0.100	45 days before harvest			

Sorghum

Twenty eight supervised residue trials were conducted on sorghum in the USA (T020419-04) during 2005, with one application made pre-emergence of the crop (SC formulation). The results on sorghum grain are shown in Table 85.

Table 85 Summary of residue data for mesotrione (SC formulation) in sorghum in the USA in 2005

State (location)	Sorghum variety	Application rate (kg ai/ha)	Growth Stage	DAT (days)	Residues (mg/kg)	Trial
South Caroline	NK8416	0.225	Soil surface at planting	113	< 0.01 (2)	SJ-HR-05-6225
		0.227	Pre-planting incorporated	113	< 0.01 (2)	
		0.224 ^a	BBCH 17	86	< 0.01 (2)	
Louisiana	Pioneer 83G66	0.231	Soil surface at planting	120	< 0.01 (2)	SD-HR-05-6226
		0.228	Pre-planting incorporated	120	< 0.01 (2)	
		0.222 ^a	6–8 leaf	87	< 0.01 (2)	
Kansas	Dekalb DKS5400	0.228	Soil surface at planting	134	< 0.01 (2)	ND-HR-05-6227
		0.224 ^a	4–6 collar	111	< 0.01 (2)	
Nebraska (York)	NC + 6B50	0.225	Soil surface at planting	127	< 0.01 (2)	NB-HR-05-6228
		0.224 ^a	BBCH 16	94	< 0.01 (2)	
Missouri	Pioneer 8500	0.223	Soil surface at planting	127	< 0.01 (2)	ND-HR-05-6229
		0.221 ^a	BBCH 16	98	< 0.01 (2)	
South Dakota	Partner 251	0.224	Soil surface at planting	128	< 0.01 (2)	NF-HR-05-6230
		0.226 ^a	BBCH 18	101	< 0.01 (2)	
Texas (Wharton)	DK52	0.219	Soil surface at planting	102 108 116	< 0.01 (2) < 0.01 (2) < 0.01 (2)	SA-HR-05-6231
		0.225 ^a	BBCH 23–32	63 69 77	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
Oklahoma	SG95207	0.229	Pre-planting incorporated	128	< 0.01 (2)	SC-HR-05-6232
		0.226 ^a	4–5 leaf	90	< 0.01 (2)	
Nebraska (Grand Island)	NC + 6B50	0.222	Soil surface at planting	132	< 0.01 (2)	NB-HR-05-6233
		0.225	Pre-planting incorporated	132	< 0.01 (2)	
		0.228 ^a	BBCH 16	99	< 0.01 (2)	
Texas (Levelland)	F222E	0.228	Pre-planting incorporated	128	< 0.01 (2)	SC-HR-05-6234
		0.223 ^a	BBCH 16	103	< 0.01 (2)	
Colorado	DG-720B	0.228	Pre-planting incorporated	145	< 0.01 (2)	NM-HR-05-6235
		0.231 ^a	vegetative	85	< 0.01 (2)	
New Mexico	7117	0.229	Soil surface at planting	113	< 0.01 (2)	NB-HR-05-6236
		0.226	Pre-planting incorporated	113	< 0.01 (2)	
		0.226 ^a	8 leaf	78	< 0.01 (2)	

^a Formulation with non-ionic surfactant

Sugarcane

Twenty six supervised residue trials were conducted on sugarcane in the USA during 2005 and four trials were conducted in South Africa during the growing period 1998/99. The trial data are summarised in Table 86.

Table 86 Summary of residue data for mesotrione in sugarcane (SC) formulation

Country (Region)	Sugarcane variety	Application Rate (g ai/ha)	Growth Stage	DAT (days)	Residues (mg/kg)	Report; trial
USA (Florida, South Bay)	CP-2143	0.27 (SS) + 0.11 (POT)	pre-emergence + 114 days to harvest	0	0.03, 0.02	T020420-04/VN-HR-05- 6240
				30	< 0.01 (2)	
				62	< 0.01 (2)	
				118	≤ 0.01 (2)	
				125	< 0.01 (2)	
		0.27 (SS) + 0.11 (PD)	pre-emergence + 100 days to harvest	0	0.02, < 0.01	
				30	< 0.01 (2)	
				61	< 0.01 (2)	
		0.10 (POT) + 0.11 (PD)	114 days + 100 days to harvest	104	< 0.01 (2)	
				111	≤ 0.01 (2)	
USA (Florida, South Bay)	CP-2086	0.27 (SS) + 0.10 (POT)	pre-emergence+ 114 days to harvest	118	≤ 0.01 (2)	T020420-04/VN-HR-05- 6241
		0.27 (SS) + 0.10 (PD)	pre-emergence + 100 days to harvest	104	≤ 0.01 (2)	
		0.10 (POT) + 0.106 (PD)	114 days + 100 days to harvest	104	≤ 0.01 (2)	
		0.52 (POT) + 0.52 (PD) ¹	114 days + 100 days to harvest	104	< 0.01 (2)	
USA (Florida, South Bay)	CP-2086	0.28 (SS) + 0.10 (POT)	pre-emergence + 114 days to harvest	118	≤ 0.01 (2)	T020420-04/VN-HR-05- 6242
		0.27 (SS) + 0.11 (PD)	pre-emergence + 100 days to harvest	104	≤ 0.01 (2)	
		0.11 (POT) + 0.10 (PD)	114 days + 100 days to harvest	104	≤ 0.01 (2)	
USA (Louisiana, Bunkie)	LCP 85384	0.25 (SS) + 0.10 (POT)	BBCH 00 + pre-internode	0	3.72, 5.75	T020420-04/ SD-HR- 2005
				30	< 0.01 (2)	
				60	< 0.01 (2)	
				114	≤ 0.01 (2)	
				121	< 0.01 (2)	
		0.26 (SS) + 0.10 (PD)	BBCH 00 + pre-internode	0	0.70, 0.31	
				30	< 0.01 (2)	
				60	< 0.01 (2)	
		0.11 (POT) + 0.10 (PD)	pre-internode + pre-internode	100	< 0.01 (2)	
				107	≤ 0.01 (2)	
USA (Louisiana, Cheney Ville)	LCP85384 Kleentek	0.29 (SS) + 0.11 (POT)	BBCH 00 + pre-internode	113	≤ 0.01 (2)	T020420-04/ SD-HR-05- 6244
		0.28(SS) + 0.10 (PD)	BBCH 00 + pre-internode	99	≤ 0.01 (2)	
		0.11 (POT) + 0.12 (PD)	pre-internode + pre-internode	99	≤ 0.01 (2)	

Country (Region)	Sugarcane variety	Application Rate (g ai/ha)	Growth Stage	DAT (days)	Residues (mg/kg)	Report; trial
		0.56 (POT) + 0.54 (PD)	pre-internode + pre-internode	99	< 0.01 (2)	
USA (Louisiana, Washington)	384	0.27(SS) + 0.11 (POT)	BBCH 00 + 75	114	≤ 0.01 (2)	T020420-04/SD-HR-05- 6245
		0.27 (SS) + 0.11 (PD)	BBCH 00 + 43	100	≤ 0.01 (2)	
		0.11 (POT) + 0.11 (PD)	BBCH 00 + 43	100	≤ 0.01 (2)	
USA (Texas)	3388	0.28 (SS) + 0.11 (POT)	BBCH 08+ 37	114	≤ 0.01 (2)	T020420-04/SA-HR-05- 6246
		0.28 (SS) + 0.11 (PD)	BBCH 08 + 37	100	≤ 0.01 (2)	
		0.11 (POT) + 0.11 (PD)	BBCH 37 + 38	100	≤ 0.01 (2)	
USA (Hawaii)	65-7052	0.27 (SS) + 0.11 (POT)	prior emergence + pre-crop closure	114	≤ 0.01 (2)	T020420-04/WD-HR-05- 6247
		0.27(SS) + 0.10 (PD)	prior emergence + prior to layby	100	≤ 0.01 (2)	
		0.11 (POT) + 0.10 (PD)	pre-canopy close + prior to layby	100	< 0.01 (2)	
South Africa	N27	0.25	growth high 50–70 cm	182	< 0.01	RJ3076B/ZA18-99-H310
South Africa	N14	0.25	growth high 60–75 cm	182 273	< 0.01 < 0.01	RJ3076B/ZA18-99-H311
South Africa	N19	0.25	high 50–60 cm	181	< 0.01	RJ3076B/ZA22-99-H410
		0.50	growth high 50–60 cm	181	< 0.01	

SS= soil surface spray

POT= post-emergence over-the-top

PD= post-emergence application directed to the base of the sugarcane

Linseed

Seventeen supervised residue trials were conducted in linseed in the USA during 2004 with one pre-emergence application of a suspension concentrate (SC) formulation (Report T010290-04 2004). The results are shown in Table 87.

Table 87 Summary of residue data for the use of mesotrione on linseed in the USA in 2004 using SC formulation

State (location)	Linseed variety	Application Rate (g ai/ha)	Growth Stage	PHI (days)	Crop Part	Residues (mg/kg)	Trial number
North Dakota (Northwood)	Rehab	0.11	at planting	144	seed	< 0.01 (2)	NN-HR-04-5650
		0.21	at planting	144	seed	< 0.01 (2)	
		0.11	multiple stems and no buds	103	seed meal	< 0.01 (2) < 0.01	
		0.32	multiple stems and no buds	103	seed	< 0.01 (2)	
		0.53	multiple stems and no buds	103	seed meal	< 0.01 (2) < 0.01	
Minnesota	York	0.10	pre-emergence	170	Seed	< 0.01 (2)	NF-HR-04-5651
		0.21	pre-emergence	170	Seed	< 0.01 (2)	
		0.10	25 cm high	130	seed	< 0.01 (2)	
North Dakota (New Rockford)	Rehab	0.11	at planting	136	seed	< 0.01 (2)	NN-HR-04-5652
		0.21	at planting	136	seed	< 0.01 (2)	
		0.10	ca. 25 cm high	104	seed	< 0.01 (2)	
Montana	Neché	0.10	at planting	89	seed	< 0.01 (2)	NN-HR-04-5653
		0.21	at planting	89	seed	< 0.01 (2)	
		0.11	stem elongation	46	seed	< 0.01 (2)	

State (location)	Linseed variety	Application Rate (g ai/ha)	Growth Stage	PHI (days)	Crop Part	Residues (mg/kg)	Trial number
South Dakota	Webster	0.10	BBCH 01 (at planting)	133 140 147	seed	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NF-HR-04-5654
		0.21	BBCH 01 (at planting)	133 140 147	seed	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
		0.10	BBCH 39	95 102 109	seed	< 0.01 (2) < 0.01 (2) < 0.01 (2)	

Animal Feed

Cereal trials from the studies reported previously have included the analysis of feed samples. The results are shown in Tables 88 to 92.

Table 88 Summary of residue data for the use of mesotrione on feed from corn (sweet and field)

Country (Region)	Corn variety	Application			DAT	Crop Part	Residue (mg/kg)	Report; trial
		Form.	kg ai/ha	Growth Stage				
Canada (Ontario) 1996	Pioneer 3902	SC	0.300	Pre-emergence	102	silage	< 0.01	RR 97-043B 94-CN-96-201
		SC	0.60	Pre-emergence	102	silage	< 0.01	
		SC	0.200	5-6 Leaf	87	silage	< 0.01	
		SC	0.400	5-6 Leaf	87	silage	< 0.01	
		SC	0.300 0.200	Pre-emergence 5-6 leaf	87	silage	< 0.01	
		SC	0.600 0.400	Pre-emergence 5-6 leaf	87	silage	< 0.01	
Canada (Ontario) 1996	Ciba seeds G-4064	SC	0.300	Pre-emergence	101	silage	< 0.01	RR 97-043B 94-CN-96-202
		SC	0.600	Pre-emergence	101	silage	< 0.01	
		SC	0.200	4-5 Leaf	90	silage	< 0.01	
		SC	0.400	4-5 Leaf	90	silage	< 0.01	
		SC	0.300 0.200	Pre-emergence 4-5 leaf	90	silage	< 0.01	
		SC	0.600 0.400	Pre-emergence 4-5 leaf	90	silage	< 0.01	
Canada (Ontario) 1997	Funks-BT Maximizer	SC	0.300	Pre-emergence	120 155	silage grain	< 0.01 < 0.01	RR 98-035B 94-CN-97-901
		SC	0.600	Pre-emergence	120	silage	< 0.01	
		SC	0.200	24	91	silage	< 0.01	
		SC	0.400	24	91	silage	< 0.01	
		SC	0.300 0.200	Pre-emergence 24	91	silage	< 0.01	
		SC	0.600 0.400	Pre-emergence 24	91	silage	< 0.01	
Canada (Ontario) 1997	Funks-BT Maximizer	SC	0.300	Pre-emergence	112	silage	< 0.01	RR 98-035B 94-CN-97-902
		SC	0.600	Pre-emergence	112	silage	< 0.01	

Country (Region)	Corn variety	Application			DAT	Crop Part	Residue (mg/kg)	Report; trial
		Form.	kg ai/ha	Growth Stage				
		SC	0.200	23	93	silage	< 0.01	
		SC	0.400	23	93	silage	< 0.01	
		SC	0.300 0.200	Pre- emergence 23	93	silage	< 0.01	
		SC	0.600 0.400	Pre- emergence 23	93	silage	< 0.01	
France (South)	620 Spirit	SC	0.102	BBCH 19	38	Cob	< 0.01	03-7049 2003
	620 Spirit		0.153	BBCH 12	38 61 62	cob cob cob	< 0.01 < 0.01 < 0.01	
France (South)	620 Spirit		0.98	BBCH 19	38	Cob	< 0.01	03-7050 2003
	620 Spirit		0.143	BBCH 12	62	cob	< 0.01	
France (South)	620 Spirit		0.153	BBCH 12	61 74	cob cob	< 0.01 < 0.01	04-7012 2004
	620 Spirit		0.104	BBCH 59	39	Cob	< 0.01	
	620 Spirit)	SC	0.154	BBCH 12	61 74	cob cob	< 0.01 < 0.01	
	620 Spirit		0.103	BBCH 59	39	Cob	< 0.01	04-7012 2004
France (South)	LG35.05	WG	0.148	BBCH 15– 16	27	whole plant	< 0.01	
					35	whole plant	< 0.01	
					43	whole cob	< 0.01	
					43	remaining plant	< 0.01	
					61	whole plant	< 0.01	
					68	whole cob	< 0.01	
					68	remaining plant	< 0.01	
					99	whole plant	< 0.01	
					99	whole cob	< 0.01	
					99	remaining plant	< 0.01	
					127	whole cob	< 0.01	
					127	remaining plant	< 0.01	
Germany	Nescio	WG	0.138	BBCH 16– 18	34	whole plant	< 0.01	T000920-09- REG 2009
					47	whole plant	< 0.01	
					47	whole cobs	< 0.01	
					47	remaining plant	< 0.01	
					82	cobs	< 0.01	
					82	remaining plant	< 0.01	
					66	whole plant	< 0.01	
					91	cobs	< 0.01	
					91	remaining plant	< 0.01	
					110	whole plant	< 0.01	
					143	cobs	< 0.01	
					143	remaining plant	< 0.01	
Spain	Castellano	WG	0.154	BBCH 15	7	whole plant	< 0.01	T000921-09- REG 2009
					14	whole plant	< 0.01	
					42	cobs	< 0.01	
					42	remaining plant	< 0.01	
					63	whole plant	< 0.01	
					71	cobs	< 0.01	
					71	remaining plant	< 0.01	
					82	cobs	< 0.01	
					82	remaining plant	< 0.01	

Country (Region)	Corn variety	Application			DAT	Crop Part	Residue (mg/kg)	Report; trial
		Form.	kg ai/ha	Growth Stage				
United Kingdom	Ohio	WG	0.142	BBCH 16– 17	20	whole plant	< 0.01	T000920-09- REG 2009
					35	whole plant	< 0.01	
					41	whole cobs	< 0.01	
					41	remaining plant	< 0.01	
					53	whole cobs	< 0.01	
					53	remaining plant	< 0.01	
					60	whole plant	< 0.01	
					87	whole plant	< 0.01	
					98	cobs	< 0.01	
					98	remaining plant	< 0.01	
					112	cobs	< 0.01	
					112	remaining plant	< 0.01	
USA (Wisconsin) 2001	NK 199	SC	0.302 0.177	pre- emergence Vt (tasseling)	14	forage with ears	< 0.01 (2)	487-01; NI- HR001-01
					30	forage without stover	< 0.01 (2)	
					56		< 0.01 (2)	
USA (Washington) 2001	Jubilee	SC	0.303 0.179		14	forage w/ ears	0.02, 0.07	487-01; WF- HR003-01
					28	forage w/o ears	< 0.01 (2)	
					71	stover	< 0.01 (2)	
USA (California) 2001	Silver Queen	SC	0.300 0.182	BBCH 01 BBCH 59	0	forage w/ ears	4.29, 4.96	487-01; W2-HR102-01
					7	forage w/ ears	0.82, 1.28	
					14	forage w/ ears	0.73, 0.88	
					23	forage w/o ears	0.45, 0.34	
					30	forage w/o ears	0.40, 0.38	
					37	forage w/o ears	0.23, 0.33	
					58	stover	1.2, 0.90 (1.1)	
USA (N. Carolina) 2001	G90 F1	SC	0.308 0.182	BBCH 55	14	forage w/ ears	< 0.01, 0.02	487-01; SJ- HR003-01
					26	forage w/o ears	< 0.01 (2)	
					54	stover	< 0.01 (2)	
USA (Ohio) 2001	Bodacious	SC	0.314 0.190		14	forage w/ ears	< 0.01 (2)	487-01; NK- HR001-01
					30	forage w/o ears	< 0.01 (2)	
					52	stover	< 0.01 (2)	
USA (New York) 2001	GH-2783	SC	0.305 0.182	at planting + 8–9 leaves	14	forage w/ ears	< 0.01 (2)	487-01; EE- HR003-01
					30	forage w/o ears	< 0.01 (2)	
					67	stover	< 0.01 (2)	
USA (Idaho) 2001	Sugar Buns		0.303 0.179	post planting + pollen shed	14	forage w/ ears	0.02, 0.01	487-01; WG- HR005-01
					30	forage w/o ears	0.05, < 0.01	
					61	stover	< 0.01 (2)	
USA (Illinois) 2001	Kandy King	SC	0.301 0.186	BBCH 00 + BBCH 34	14	forage w/ ears	< 0.01 (2)	487-01; N4- HR003-01
					31	forage w/o ears	< 0.01 (2)	
					60	stover	< 0.01 (2)	
USA (Pennsylvania) 2001	Argent	SC	0.320 0.186	BBCH 00 + early tassel	14	forage w/ ears	< 0.01 (2)	487-01; EC- HR002-01
					30	forage w/o ears	< 0.01 (2)	
					70	stover	< 0.01 (2)	
USA (Florida) 2001	Silver Queen	SC	0.313 0.177	Just planted + visible tassel	14	forage w/ ears	< 0.01 (2)	487-01; VB- HR101-01
					28	forage w/o ears	< 0.01 (2)	
					62	stover	< 0.01 (2)	
USA (Michigan) 2001	Excellency	SC	0.306 0.180	pre- emergence Vt	0	forage w/ ears	3.09, 3.96	487-01; ED- HR006-01
					7	forage w/ ears	0.02, 0.01	
					14	forage w/ ears	< 0.01 (2)	
					23	forage w/o ears	< 0.01 (2)	
					30	forage w/o ears	< 0.01 (2)	
					36	forage w/o ears	< 0.01 (2)	
					70	stover	< 0.01 (2)	
USA (Maine) 2001	Maple Sweet	SC	0.304 0.189	0 + tassel initiation	14	forage w/ ears	< 0.01 (2)	487-01; NF- HR002-01
					30	forage w/o ears	< 0.01 (2)	
					55	stover	< 0.01 (2)	
USA N. Carolina)	Rogers WH0809	SC	0.107 0.106	BBCH 36 + BBCH 37	32	forage w/o ears	< 0.01 (2)	T001589-08; E10NC081871
					53	stover	< 0.01 (2)	

Country (Region) 2008	Corn variety	Application			DAT	Crop Part	Residue (mg/kg)	Report; trial
		Form.	kg ai/ha	Growth Stage				
		WG	0.104 0.104	BBCH 36 + BBCH 37	32 53	forage w/o ears stover	< 0.01 (2) < 0.01 (2)	
USA (N. Dakota 2008)	Peaches & Cream	SC	0.107 0.105	BBCH 16 + BBCH 55	28 66	forage w/o ears stover	< 0.01 (2) < 0.01 (2)	T001589-08; C13ND081872
		WG	0.107 0.106	BBCH 16 + BBCH 55	28 66	forage w/o ears stover	< 0.01 (2) < 0.01 (2)	
USA (California) 2008	Sweetie 82	SC	0.103 0.107	BBCH 15 + BBCH 17	45 69	forage w/o ears stover	0.12 (0.07, 0.17) 0.06, 0.09	T001589-08; W30CA081873
		WG	0.105 0.105	BBCH 15 + BBCH 17	45 69	forage stover	(0.06) 0.07, 0.06 0.09, 0.07	
USA (Iowa, Sheffield) 1995	ICI 8543	SC	0.336 0.224	Pre-plant vegetative	82 109	forage stover	< 0.01 (3) < 0.01 (2)	RR 96-018B 63-IA-95-805
USA (Illinois, Brimfield) 1995	Hoblit 428	SC-	0.336 0.224	pre- emergence	85 95	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 60-IL-95-806
USA (Indiana, Lafayette) 1995	Pioneer 3394	SC	0.336 0.224	pre- emergence 7–9 leaves	76 96 96	forage grain stover	< 0.01 (2) < 0.01 (2) < 0.01 (2)	RR 96-018B 67-IN-95-807
USA (Michigan) 1995	NK 4640	SC	0.336 0.224	pre-bloom	85 114	forage stover	< 0.01 (2) < 0.01	RR 96-018B 24-MI-95-808
USA (Nebraska) 1995	Ottillie RO 2455	SC	0.336 0.224	Pre-plant V6	62 88	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 68-NE-95-809
USA (Iowa, Albia) 1995	ICI 8532	SC	0.336 0.224	Pre-plant vegetative	74 102	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 63-IA-95-810
USA (Illinois, Towanda) 1995	Ainsworth 640	SC	0.336 0.224	Pre-plant post emergence	76 87	forage stover	< 0.01 (3) < 0.01 (2)	RR 96-018B 60-IL-95-811
USA (Indiana, Rochester) 1995	Pioneer 3394	SC	0.336 0.224	Early pre- plant 5–7 leaves	92 96	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 67-IN-95-812
USA (Nebraska, Waverly) 1995	Producers PH785	SC	0.336 0.224	Pre-plant V5–V6	71 96	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 68-NE-95-814
USA (Kansas, La Cygne) 1995	CIBA 4575	SC	0.336 224	early pre- plant post	46 68	forage stover	< 0.01 (3) < 0.01 (2)	RR 96-018B 37-KS-95-825
USA (Pennsylvania, Ephrata) 1995	CI 8541	SC	0.336 224	pre- emergence vegetative	76 98	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 70-PA-95-815
		SC	0.336 0.224	pre- emergence vegetative	76 98	forage stover	< 0.01 (2) < 0.01 (2)	
USA (Minnesota, St. Peter) 1995	Cenex 424	SC	0.336 0.224	pre- emergence vegetative	76 92	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 36-MN-95-816
		SC	0.336 0.224	pre- emergence vegetative	76 92	forage stover	< 0.01 (2) < 0.01 (2)	
USA (Ohio, Urbana) 1995	Vigoro V1122	SC	0.336 0.224	pre- emergence	85 113	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 24-OH-95-817
		SC	0.336 0.224	pre- emergence	85 113	forage stover	< 0.01 (2) < 0.01 (2)	
USA (Wisconsin, 1995)	Cenex LOL 357	SC	0.336 0.224	pre- emergence	72 104	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 79-WI-95-818

Country (Region)	Corn variety	Application			DAT	Crop Part	Residue (mg/kg)	Report; trial
		Form.	kg ai/ha	Growth Stage				
Baraboo) 1995		SC	0.336 0.224	pre- emergence	72 104	forage stover	< 0.01 (2) < 0.01 (2)	
USA (Texas) 1995	G4673B (D&PL Co.)	SC	0.336 0.224	pre- emergence pre-tassel	56 92	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 25-TX-95-819
		SC	0.336 0.224	pre- emergence pre-tassel	56 92	forage stover	< 0.01 (2) < 0.01 (2)	
USA (north Carolina) 1995	Field corn (Pioneer 3165)	SC	0.336 0.224	pre-plant rapidly growing	68 95	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 01-NC-95-820
		SC	0.336 0.224	pre-plant rapidly growing	62 68 75 82 88 95 101 108	forage forage forage stover stover stover stover	< 0.01 < 0.01 (2) < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	
USA (Iowa, Boone) 1995	ICI 8543	SC	0.336 0.224	Early pre- plant vegetative	86 112	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 63-IA-95-821
		SC	0.336 0.224	Early pre- plant vegetative	86 112	forage stover	< 0.01 (2) < 0.01 (2)	
USA (Illinois, Champagne) 1995	Pioneer 3394	SC	0.336 0.224	pre-plant post emergence	74 107	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 04-IL-95-822
		SC	0.336 0.224	pre-plant post emergence	60 74 81 88 100 107 113 121	forage forage forage stover stover stover stover	< 0.01 < 0.01 (2) < 0.01 < 0.01 < 0.01 < 0.01 (2) < 0.01 < 0.01	
USA (Indiana, N. Richmond) 1995	Pioneer 3394	SC	0.336 20.24	pre-plant 6–8 leaf	64 86	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 67-IN-95-823
		SC	0.336 0.224	pre-plant 6–8 leaf	64 86	forage stover	< 0.01 (2) < 0.01 (2)	
USA (Nebraska, Crete) 1995	ICI 8541	SC	0.336 0.224	pre-plant V6-V7	62 89	forage stover	< 0.01 (2) < 0.01 (2)	RR 96-018B 68-NE-95-824
		SC	0.336 0.224	pre-plant V6-V7	62 89	forage stover	< 0.01 (2) < 0.01 (2)	
USA Iowa) 2001	Pioneer 34B23)	SC	0.268 0.225	BBCH 00 BBCH 18– 19	46 99	forage stover	< 0.01 (2) < 0.01 (2)	1847-01 NE-HR-003-01
		SE SC	0.267 0.233	BBCH 00 BBCH 18/19	46 99	forage stover	< 0.01 (2) < 0.01 (2)	
		SC	0.274 0.234	BBCH 13 BBCH 18 19	46 99	forage stover	< 0.01 (2) < 0.01 (2)	
		SE SC	0.267 0.223	BBCH 13 BBCH 18/19	46 99	forage stover	< 0.01 (2) < 0.01 (2)	
USA (North Carolina) 2001	ICI 8543)	SC	0.269 0.226	BBCH 00 75 cm high	45 98	forage stover	< 0.01 (2) < 0.01 (2)	1847-01 SJ-HR-013-01
		SE SC	0.269 0.227	BBCH 00 75 cm high	45 98	forage stover	< 0.01 (2) < 0.01 (2)	

Country (Region)	Corn variety	Application			DAT	Crop Part	Residue (mg/kg)	Report; trial
		Form.	kg ai/ha	Growth Stage				
		SC	0.271 0.227	BBCH 12/13 75 cm high	45 98	forage stover	< 0.01 (2) < 0.01 (2)	
		SE	0.270	BBCH	45	forage	< 0.01 (2)	
		SC	0.227	12/13 75 cm high	98	stover	< 0.01 (2)	
USA (Illinois) 2001	Pioneer 34B24	SC	0.285 0.253	BBCH 00 BBCH 36	45 112	forage stover	< 0.01 (2) < 0.01 (3)	1847-01 N4-HR-005-01
		SE	0.278	BBCH 00	45	forage	< 0.01 (2)	
		SC	0.236	BBCH 36	112	stover	< 0.01 (2)	
		SC	0.286 0.244	BBCH 12 BBCH 36	45 112	forage stover	< 0.01 (2) < 0.01 (2)	
		SE	0.268	BBCH 12	45	forage	< 0.01 (2)	
		SC	0.237	BBCH 36	112	stover	< 0.01 (2)	
USA (Nebraska) 2001	ICI 8543	SC	0.270 0.227	BBCH 00 BBCH 19	41 103	forage stover	< 0.01 (2) < 0.01 (2)	1847-01 NB-HR-004-01
		SE	0.273	BBCH 00	41	forage	< 0.01 (2)	
		SC	0.227	BBCH 19	103	stover	< 0.01 (2)	
		SC	0.266 0.226	BBCH 12/13 BBCH 19	41 103	forage stover	< 0.01 (2) < 0.01 (2)	
		SE	0.268	BBCH	41	forage	< 0.01 (2)	
		SC	0.226	12/13 BBCH 19	103	stover	< 0.01 (2)	

Table 89 Summary of residue data for mesotrione on millet (feed) using SC formulation in the USA in 2004 (Report T010289-04)

State	Millet variety	Application Rate (g ai/ha)	Growth Stage	DAT	Crop Part	Residue (mg/kg)	Trial number
Illinois	Max Perl	0.109	at planting	31 31	forage hay	< 0.01 (2) < 0.01 (2)	4A-HR-04- 5640
		0.214	at planting	31 31	forage hay	< 0.01 (2) < 0.01 (2)	
		0.108	BBCH 32	31 31	forage hay	< 0.01 (2) < 0.01 (2)	
Nebraska	Huntsman	0.103	at planting	51 51 84	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	4A-HR-04- 5641
		0.210	at planting	51 51 84	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
		0.103	BBCH 12	28 28 61	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
South Dakota	Rise	0.104	at planting	34 34 95	forage hay straw	< 0.01 (2) 0.01, < 0.01 < 0.01 (2)	4A-HR-04- 5642
		0.208	at planting	34 34 95	forage hay straw	0.01, < 0.01 0.01 (2) < 0.01 (2)	
		0.105	BBCH 21	30 30 69	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
Colorado (Ault)	Early Bird	0.107	at planting	70 70 132	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	4A-HR-04- 5643
		0.212	at planting	70 70 132	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	

State	Millet variety	Application Rate (g ai/ha)	Growth Stage	DAT	Crop Part	Residue (mg/kg)	Trial number
		0.107	vegetative	30 30 92	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
Colorado (Ault)	Early Bird	0.110	at planting	63	forage	< 0.01 (2)	4A-HR-04-5644
				63	hay	< 0.01 (2)	
				70	forage	< 0.01 (2)	
				70	hay	< 0.01 (2)	
				77	forage	< 0.01 (2)	
				77	hay	< 0.01 (2)	
				104	straw	< 0.01 (2)	
				111	straw	< 0.01 (2)	
				120	straw	< 0.01 (2)	
		0.218	at planting	63	forage	< 0.01 (2)	
				63	hay	< 0.01 (2)	
				70	forage	< 0.01 (2)	
				70	hay	< 0.01 (2)	
				77	forage	< 0.01 (2)	
				77	hay	< 0.01 (2)	
				104	straw	< 0.01 (2)	
				111	straw	< 0.01 (2)	
				120	straw	< 0.01 (2)	
		0.108	vegetative	23	forage	< 0.01 (2)	
				23	hay	< 0.01 (2)	
				30	forage	< 0.01 (2)	
				30	hay	< 0.01 (2)	
				37	forage	< 0.01 (2)	
				37	hay	< 0.01 (2)	
				64	straw	< 0.01 (2)	
				71	straw	< 0.01 (2)	
				80	straw	< 0.01 (2)	

Table 90 Summary of residue data for the use of mesotrione on oats (feed) (SC formulation) in 2005 in the USA (Report T004407-05)

State (location)	Oat variety	Application rate (kg ai/ha)	Growth Stage at Application	DAT	Crop Part	Residues (mg/kg)	Trial number
New York	Armor	0.214	BBCH 00	70 70 90	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	5A-HR-05-6380
		0.104	BBCH 59–61	29 29 49	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
Virginia	Coker	0.216	BBCH 00	225 225 246	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	SJ-HR-05-6381
		0.108	BBCH 75	30 30 51	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
North Dakota (Northwood)	Morton	0.211	bare soil application	73 73 90	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NN-HR-05-6382
		0.104	BBCH 23	33 33 50	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
South Dakota (Lesterville)	Jerry	0.209	BBCH 01	60 60 84	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NF-HR-05-6383
		0.106	BBCH 32	30 30 54	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2))	

State (location)	Oat variety	Application rate (kg ai/ha)	Growth Stage at Application	DAT	Crop Part	Residues (mg/kg)	Trial number
Wisconsin	Oat (Esker)	0.216	pre-emergence	70 70 91	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NI-HR-05-6384
		0.106	late boot to early head	28 28 49	forage hay straw	<u>< 0.01</u> (2) <u>< 0.01</u> (2) <u>< 0.01</u> (2)	
Minnesota	Oat (Drumlin)	0.209	BBCH 00	58 58 80	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NF-HR-05-6385
		0.107	BBCH 21	30 30 52	forage hay straw	<u>< 0.01</u> (2) <u>< 0.01</u> (2) <u>< 0.01</u> (2)	
Nebraska (York)	Oat (Jerry)	0.213	BBCH 00	54 54 63 63 68 68 75 75 87	forage hay forage hay forage hay forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	NB-HR-05-6386
		0.105	BBCH 31	16 16 25 25 30 30 37 37 49	forage hay forage hay forage hay forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	
Iowa	Oat (Jerry)	0.213	BBCH 00	75 75 96	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NE-HR-05-6387
		0.106	BBCH 80	28 28 49	forage hay straw	<u>< 0.01</u> (2) <u>< 0.01</u> (2) <u>< 0.01</u> (2)	
Kansas (Sabetha)	Oat (Loyal)	0.210	pre-emergence	63 63 83	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	ND-HR-05-6388
		0.107	tillering (1–3 tillers)	30 30 50	forage hay straw	<u>< 0.01</u> (2) <u>< 0.01</u> (2) <u>< 0.01</u> (2)	
Michigan	Oat (Prairie)	0.213	pre-emergence	55 55 61 61 68 68 75 75 88	forage hay forage hay forage hay forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	NL-HR-05-6389
		0.104	5–6 leaves	17 17 23 23 30 30 37 37 50	forage hay forage hay forage hay forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	
Illinois	Oat (VHS)	0.209	BBCH 00	62 62 81	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	4A-HR-05-6390

State (location)	Oat variety	Application rate (kg ai/ha)	Growth Stage at Application	DAT	Crop Part	Residues (mg/kg)	Trial number
		0.104	BBCH 26	33 33 52	forage hay straw	≤ 0.01 (2) ≤ 0.01 (2) ≤ 0.01 (2)	
		0.317	BBCH 26	33 33 52	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
		0.531	BBCH 26	33 33 52	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
Texas	Oat (La-604)	0.209	BBCH 00	156 156 180	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	SA-HR-05-6391
		0.106	BBCH 43	30 30 54	forage hay straw	≤ 0.01 (2) ≤ 0.01 (2) ≤ 0.01 (2)	
North Dakota (New Rockford)	Oat (La-604)	0.211	soil surface spray	73 73 92	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NM-HR-05-6392
		0.104	BBCH 43	30 30 49	forage hay straw	≤ 0.01 (2) ≤ 0.01 (2) ≤ 0.01 (2)	
South Dakota (Frederick)	Oat (Morton)	0.211	BBCH 01	60 60 81	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NF-HR-05-6393
		0.106	BBCH 21–31	30 30 51	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	
Nebraska (Grand Island)	Oat (Reeves)	0.215	BBCH 00	70 70 90	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NB-HR-05-6394
		0.105	BBCH 31	29 29 49	forage hay straw	≤ 0.01 (2) ≤ 0.01 (2) ≤ 0.01 (2)	
Kansas (Larned)	Oat (Jerry)	0.210	pre-emergence	63 63 83	forage hay straw	< 0.01 (2) < 0.01 (2) < 0.01 (2)	NM-HR-05-6395
		0.107	tillering (1–2 tillers)	30 30 50	forage hay straw	≤ 0.01 (2) ≤ 0.01 (2) ≤ 0.01 (2)	

Table 91 Summary of residue data for mesotrione on rice (feed) after application using a granular formulation

Country year	Rice variety	Application rate (kg ai/ha)	Growth Stage	DAT	Crop Part	Residue (mg/kg)	Trial number
Korea 2005		0.180	5–15 days after transplanting	140	Straw	< 0.1 (3)	A14928B
		0.360	5–15 days after transplanting	140	Straw	< 0.1 (3)	
Japan 2004	Matsuribare	0.100	21 days after transplanting	63 91	Forage Straw	< 0.002 (2) < 0.01 (2)	A13723B
Japan 2004	Hi-no-hikari	0.100	21 days after transplanting	77 89	Forage Straw	< 0.002 (2) < 0.01 (2)	A13723B
Japan 2006	Koshihikari	0.100	4.5 leaf period growing period	76	Straw	< 0.01 (2)	A13723B
		0.100	4.5 leaf period growing period	61	Straw	< 0.01 (2)	
		0.100	4.5 leaf period growing period				
		0.100	4.5 leaf period before harvest	45	Straw	< 0.01 (2)	

Country year	Rice variety	Application rate (kg ai/ha)	Growth Stage	DAT	Crop Part	Residue (mg/kg)	Trial number
Japan 2006	Hi-no-hikari	0.100	10 days after plant	75	Straw	< 0.01 (2)	A13723B
		0.100	75 days before harvest				
		0.100	10 days after plant	60	Straw	< 0.01 (2)	
		0.100	60 days before harvest				
Japan 2007	Koshihikari	0.100	10 days after plant	45	Straw	< 0.01 (2)	A13723B
		0.100	45 days before harvest				
		0.100	growing period	75	Forage	< 0.002 (2)	
		0.100	21 cm high				
Japan	Hi-no-hikari	0.100	growing period	60	Forage	< 0.002 (2)	A13723B
		0.100	growing period				
		0.100	growing period	45	Forage	< 0.002 (2)	
		0.100	before harvest				
Japan	Hi-no-hikari	0.100	24 days after plant	53	Forage	< 0.002 (2)	A13723B
		0.100	24 days after plant	46	Forage	< 0.002 (2)	
		0.100	35 days before harvest				
		0.100	24 days after plant	34	Forage	< 0.002 (2)	
		0.100	formation of young panicle				

Table 92 Summary of residue data for mesotrione in sorghum feed in USA in 2005 (SC formulation) (Report T020419-04)

State (location)	Sorghum variety	Application rate (kg ai/ha)	Growth Stage	DAT (days)	Crop Part	Residues (mg/kg)	Trial
South Caroline	NK8416	0.225	pre-emergence	57 113	forage stover	<u>< 0.01</u> (2) <u>< 0.01</u> (2)	SJ-HR-05-6225
		0.227	before planting	57 113	forage stover	<u>< 0.01</u> (2) <u>< 0.01</u> (2)	
		0.224	BBCH 17	30 86	forage stover	< 0.01 (2) < 0.01 (2)	
Louisiana	Pioneer 83G66	0.231	pre-emergence	65 120	forage stover	< 0.01 (2) <u>< 0.01</u> (2)	SD-HR-05-6226
		0.228	pre-plant	65 120	forage stover	<u>< 0.01</u> (2) <u>< 0.01</u> (2)	
		0.222	6-8 leaf	32 87	forage stover	< 0.01 (2) < 0.01 (2)	
Kansas	Dekalb DKS5400	0.228	pre-emergence	54 134	forage stover	<u>< 0.01</u> (2) <u>< 0.01</u> (2)	ND-HR-05-6227
		0.224	4-6 collar	31 111 111	forage stover AGF	< 0.01 (2) < 0.01 (2) < 0.01	
Nebraska (York)	NC + 6B50	0.225	pre-emergence	33 43 49 63 127	forage forage forage forage stover	< 0.01 (2) < 0.01 (2) < 0.01 (2) <u>< 0.01</u> (2) <u>< 0.01</u> (2)	NB-HR-05-6228
		0.224	BBCH 16	0 10 16 30 94	forage forage forage forage stover	14.4, 11.8 < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	
Missouri	Pioneer 8500	0.223	pre-emergence	57 127	forage stover	< 0.01 (2) <u>< 0.01</u> (2)	ND-HR-05-6229
		0.221	BBCH 16	28 98	forage stover	< 0.01 (2) < 0.01 (2)	
South Dakota	Partner 251	0.224	BBCH 00	57 128	forage stover	<u>< 0.01</u> (2) <u>< 0.01</u> (2)	NF-HR-05-6230
		0.226	BBCH 18	30 101	forage stover	< 0.01 (2) < 0.01 (2)	

State (location)	Sorghum variety	Application rate (kg ai/ha)	Growth Stage	DAT (days)	Crop Part	Residues (mg/kg)	Trial
Texas (Wharton)	DK52	0.219	BBCH 00	39 49 55 70 102 108 116	forage forage forage forage stover stover stover	< 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	SA-HR-05-6231
		0.225	BBCH 23–32	0 10 16 31 63 69 77	forage forage forage forage stover stover stover	15.0, 15.1 < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	
Oklahoma	SG95207	0.229	pre-planting	71 128	forage stover	< 0.01 (2) < 0.01 (2)	SC-HR-05-6232
		0.226	4–5 leaf	33 90	forage stover	< 0.01 (2) < 0.01 (2)	
Nebraska (Grand Island)	NC + 6B50	0.222	pre-emergence	63 132	forage stover	< 0.01 (2) < 0.01 (2)	NB-HR-05-6233
		0.225	BBCH 00	63 132	forage stover	< 0.01 (2) < 0.01 (2)	
		0.228	BBCH 16	30 99	forage stover	< 0.01 (2) < 0.01 (2)	
Texas (Levelland)	F222E	0.228	pre-planting	55 128	forage stover	< 0.01 (2) < 0.01 (2)	SC-HR-05-6234
		0.223	BBCH 16	30 103 103	forage stover AGF	< 0.01 (2) < 0.01 (2) < 0.01	
Colorado	DG-720B	0.228	at planting	90 145	forage stover	< 0.01 (2) < 0.01 (2)	NM-HR-05-6235
		0.231	vegetative	30 85	forage stover	< 0.01 (2) < 0.01 (2)	
New Mexico	7117	0.229	pre-emergence	64 113	forage stover	< 0.01 (2) < 0.01 (2)	NB-HR-05-6236
		0.226	pre-plant	64 113	forage stover	< 0.01 (2) < 0.01 (2)	
		0.226	8 leaf	29 78	forage stover	0.02, 0.01 < 0.01 (2)	

Processing studies

A residue trial on soya beans conducted in the USA with one pre-emergence application at a 1.1 kg ai/ha (Table 77; report T005595-06). Soya beans harvested at 113 DAT were processed into meal, hulls and refined oil simulating commercial practices, and samples analysed using method RAM 366/01. Residues of mesotrione were < 0.01 mg/kg in the soy sample, meal, hulls and refined oil, and no processing factors were derived

Two residue trials on mesotrione-tolerant HT soya beans were conducted in the USA using pre- plus post-emergence applications at 1.1 and 0.6 kg ai/ha, respectively (Table 93, Report T000908-07). Soya bean samples were harvested at 76 or 102 DAT, processed according to commercial practices, and samples analysed using method RAM 366/01. The results are show in in Table 93.

Table 93 Processing factors for mesotrione residues in mesotrione-tolerant soya beans

Mesotrione	Soya bean	Meal	Hulls	Crude oil	Refined oil	AGF	Flour	Soya milk	Tofu	Soya sauce	Miso
Residue (mg/kg) ^a	0.04	0.01	0.02	< 0.01	< 0.01	0.02	0.07	< 0.01	< 0.01	< 0.01	< 0.01

Processing factor		0.25	0.5	< 0.25	< 0.25	0.5	1.8	< 0.25	< 0.25	< 0.25	< 0.25
Residue (mg/kg) ^b	< 0.01	< 0.01	< 0.01	< 0.01	0.01	< 0.01	0.02	< 0.01	< 0.01	< 0.01	< 0.01
Processing factor		–	–	–	< 0.01	–	> 2	–	–	–	–

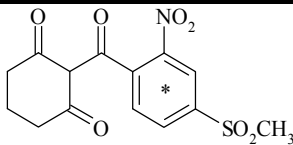
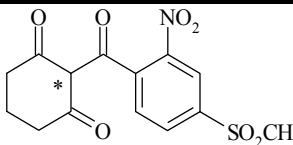
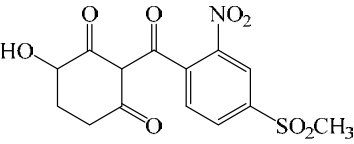
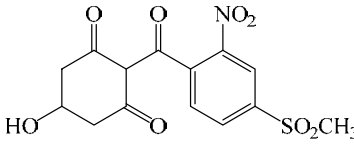
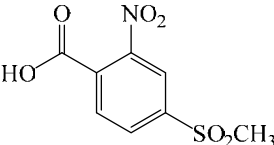
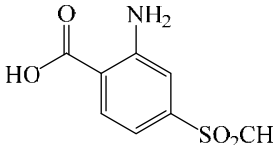
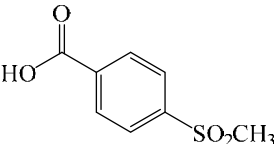
^a Trial C30-9659, 79 DAT^b Trial C18-9656, 102 DAT

RESIDUES IN ANIMAL COMMODITIES

No studies were provided

APPRAISAL

Mesotrione is a systemic pre-emergence and post-emergence herbicide for the selective contact and residual control of broadleaf weeds. The compound was scheduled for evaluation by 2014 JMPR as a new compound at the Forty-fifth Session of the CCPR (2013). Metabolism studies on animal and plants, confined rotational crops and environmental fate studies, analytical methods and residue trials on berries, okra, sweet corn, soybean, asparagus, rhubarb maize, millet, oat, rice, sorghum, sugarcane and linseed were submitted for evaluation. The structure of mesotrione and the main metabolites found in livestock, plant tissues and soil are shown below

 [Phenyl-U-¹⁴C] Mesotrione	 [Cyclohexane-2-¹⁴C] Mesotrione
 4-Hydroxy-mesotrione	 5-Hydroxy-mesotrione
 MNBA [4-(methylsulphonyl)-2-nitrobenzoic acid]	 AMBA [(2-amino-4-(methylsulphonyl)-benzoic acid)]
 MBA [4-(methylsulphonyl) benzoic acid]	

Animal metabolism

Rats

The metabolism of mesotrione was evaluated at the present Meeting by the JMPR WHO Panel. The compound is rapidly and extensively absorbed, minimally metabolized and excreted primarily in urine after a single or repeated dose. The majority of the radioactivity was excreted as the parent compound within 12 hours post-dose, accounting for 43–64% of the dose in urine. The metabolites found in the excreta includes 4 and 5-hydroxy mesotrione, MNBA and AMBA.

Livestock animals

Metabolism studies with mesotrione were conducted in lactating cows, swine and poultry. Additionally, the metabolism of AMBA, was investigated in the cow.

In two metabolism studies conducted in lactating cows, the animals were dosed with [phenyl- $U-^{14}C$]-mesotrione or [cyclohexane-2- ^{14}C]-mesotrione for 7 consecutive days at a nominal rate of 10 ppm in the diet, and sacrificed 16 hours after the final dose. Over 90% of the administered dose was found in excreta, mostly in faeces. TRR was higher in liver and kidney (0.07 to 0.11 mg eq./kg), reached 0.007 mg eq./kg in muscle, 0.013 mg/kg eq in fat and 0.08 mg eq./kg in milk (at least 90% TRR in skimmed milk). Mesotrione accounted for 10–18% TRR in liver and kidney (0.01–0.02 mg eq./kg). AMBA was identified in kidney of the phenyl label experiment (0.01 mg eq./kg).

One female swine was dosed orally with [phenyl- $U-^{14}C$]-mesotrione for 5 consecutive days at 6 ppm, and sacrificed 23 hours after the final dose. About 90% of the administered dose was recovered in the excreta, mostly in the faeces. Highest TRRs were found in liver (1.75 mg eq./kg) and kidney (0.12 mg eq./kg), with 0.01 mg eq./kg in muscle and 0.006 mg eq./kg in fat. Mesotrione was the main identified residues (90% TRR in liver, 73% TRR in kidney and 78% TRR in muscle). AMBA accounted for up to 2% TRR in tissues (up to 0.029 mg eq./kg in liver). MNBA was only detected in liver (0.005 mg eq./kg).

Two metabolism studies were conducted in poultry, in which hens were dosed for 10 consecutive days at 11 ppm either with [phenyl- $U-^{14}C$]-mesotrione or [cyclohexane-2- ^{14}C]-mesotrione; the hens were sacrificed 16 hours after the final dose. The radioactivity in excreta accounted for over 90% of the administered dose, and contained mesotrione (up to 55% TRR) and AMBA (18% TRR). TRRs were similar in both experiments for liver (1.1–1.2 mg eq./kg) and kidney (0.06–0.07 mg eq./kg), but were higher in the cyclohexane experiment in muscle (up to 0.012 mg/kg eq), fat (up to 0.048 mg eq./kg), reaching 0.094 mg eq./kg in egg yolk, and 0.025 mg eq./kg in the white. Mesotrione was not detected in muscle in any experiment, and was the only compound identified in tissues and eggs in both experiments, corresponding to at least 70% TRR in the liver and fat. In egg yolk, mesotrione accounted for 81% TRR in the phenyl experiment, and 19.5% TRR in the cyclohexane experiment, in which about 15% TRR was shown to be incorporated into palmitic/oleic acid.

A lactating cow received [phenyl- $U-^{14}C$]-AMBA for 7 days at 12.2 ppm in the diet and was sacrificed 23 hours after the final dose. About 90% of the dose was recovered in the excreta, mostly in the faeces. Highest residues were found in kidney (0.053 mg eq./kg), with AMBA accounting for 79% TRR. Perineal fat contained 0.018 mg eq./kg, 62% identified as AMBA. TRR in liver were 0.005 mg eq./kg, and reached a maximum of 0.009 mg eq./kg in milk (day 6), but were not characterized. No radioactive residues were detected in muscle.

In summary, the biotransformation of mesotrione in livestock involves the oxidative cleavage of the parent molecule to yield MNBA, which is reduced in the nitro group to give AMBA. Highest residues were found in liver and kidney, and the levels in muscle were low, reaching a maximum of 0.012 mg eq./kg Mesotrione accounted for up to 18% TRR in cow liver and kidney, at least 70% TRR in tissues of swine and poultry, and up to 80% TRR in egg yolk. No single compound was detected in muscle. The metabolism of Mesotrione in rats was found to be similar to that described for livestock.

Plant metabolism

Metabolism studies were conducted in cranberries, tolerant soya bean, maize, rice and peanuts. [Phenyl- U - ^{14}C]-mesotrione was applied twice to cranberry plants at $0.331 + 0.242$ kg ai/ha ($1\times$) or $0.919 + 0.642$ kg ai/ha ($3\times$ rate), and samples harvested 46 days after the last treatment (DAT). TRRs in mature foliage were 16.8 mg eq./kg and 31.8 mg eq./kg for $1\times$ and $3\times$, respectively. TRRs in the mature cranberry fruit were 2.6 mg eq./kg and 4.9 mg eq./kg, respectively, mostly as mesotrione (60–67% TRR) and AMBA (24–35% TRR), MNBA accounted for up to 3% TRR.

Mesotrione tolerant soya bean seeds grown in sandy loam soil were treated with either [phenyl- U - ^{14}C]- or [cyclohexane-2- ^{14}C]-mesotrione using three GAP application regimes: one pre-emergence at 0.225 kg ai/ha (T1), a combined pre-emergence at 0.225 kg ai/ha followed by a post-emergence at 0.125 kg ai/ha (T2), or one post-emergence at 0.225 kg ai/ha (T3). Forage was sampled at 22–28 DAT, hay at 40–42 DAT and seeds at 90–123 DAT.

Higher radioactivity was recovered from the phenyl label experiment. In forage, TRR were 0.16 to 0.5 mg eq./kg, mostly as MNBA (13 to 24% TRR; 0.04 to 0.06 mg eq./kg); mesotrione and its 4 and 5-hydroxy metabolite accounted for up to 14.6% TRR each (0.01 to 0.08 mg eq./kg). In hay, TRR ranged from 0.14 mg/kg eq (T1) to 2 mg eq./kg (T2), mostly MNBA (up to 20% TRR) and 4/5-hydroxy-mesotrione (up to 16% TRR); mesotrione accounted for up to 9% TRR. AMBA was only detected in T2 hay (0.055 mg eq./kg; 2.7% TRR). Residues in soya bean seed ranged from 0.052 to 0.104 mg eq./kg, with mesotrione and 4/5-hydroxy-mesotrione the main compounds identified (< 10% TRR). Low levels of MNBA and AMBA were found in the T1 and T2 samples (< 5% TRR, 0.005 mg eq./kg).

Results from the cyclohexane experiment showed mesotrione accounting for up to 18% TRR in forage, 8.2% TRR in hay and 5.1% TRR in seed (0.02 mg eq./kg). 4 and 5-hydroxy-mesotrione accounted for up to 19% TRR in forage and hay, and 7% TRR in seeds.

Three studies were conducted with maize, two with [phenyl- U - ^{14}C]-mesotrione and/or one with [cyclohexane-2- ^{14}C]-mesotrione. In all cases, the compound was applied to the soil surface after planting the seeds at a rate of 0.3 kg ai/ha (pre-emergence; T1) or post-emergence at 0.16–0.18 kg ai/ha, 28 days after planting (T2).

Results from the phenyl label experiments showed higher total residues in fodder/stover (0.8 to 1.1 mg eq./kg) and forage (0.244 to 0.356 mg eq./kg). Over 60% of the residues in fodder were not extracted with ACN/water. In T1 forage, MNBA and AMBA were the major residues (up to 19.7 and 12.2% TRR, respectively). In fodder, AMBA was the major residue (up to 28% TRR in T2). 4-hydroxy-mesotrione was mainly present in forage (up to 8% TRR, about 50% conjugated). Mesotrione was a minor component of the residues in all cases, present at a higher level in T1 forage samples (2.2% TRR, 0.008 mg eq./kg). TRR in grain was 0.01 mg/kg eq, and was not further characterized.

In the cyclohexane experiment, TRR reached 0.1 mg eq./kg in forage and 0.33 mg eq./kg in fodder. In forage, the identified residues were mesotrione (up to 3% TRR) and 4-hydroxy-mesotrione (up to 10% TRR). About 18% TRR was incorporated into lignin and cellulose. Residues in grain were low (up to 0.011 mg eq./kg) and were not be further characterized.

Rice plants were treated at the 2–3 leaf stage with [phenyl- U - ^{14}C]-mesotrione added directly to the paddy water at either 0.09 kg ai/ha ($1\times$) or 0.225 kg ai/ha ($2.5\times$). TRRs were higher in whole tops and straw (0.03 to 0.06 mg eq./kg at $1\times$), with 60–71% extracted by ACN/water. Residues in grain and husk (109 DAT) reached 0.01 mg eq./kg, about 33% being extracted (acid released up to 75.1% TRR in grain). Immature whole tops from $1\times$ rate contained mesotrione and 5-hydroxy-mesotrione at up to 0.01 mg/kg eq. each (11 to 15% TRR), and traces of MNBA and AMBA (< 5% TRR from $1\times$). In $1\times$ stalk and straw, mesotrione and its metabolites represented < 10% TRR each. No characterization was performed in grain. Residues from $2.5\times$ samples were 2–5 times higher (0.02 mg eq./kg in grain).

[Phenyl- ^{14}C] or [cyclohexane-2- ^{14}C]-mesotrione were applied to the soil surface after planting peanut seed (pre-emergence) at 0.3 (T1) or 0.8 kg ai/ha (T2). Peanut foliage was harvested 90 DAT (50% maturity), mature peanuts and peanut hay at 153 DAT. Residues from [phenyl- ^{14}C] treatment were higher in foliage (0.028 and 0.064 mg eq./kg, in T1 and T2, respectively) and approximately 0.01 mg eq./kg in hay, hull and nutmeat. Traces of MNBA, MBA, AMBA and 4-hydroxy-mesotrione were found in hay (< 6% TRR, ≤ 0.002 mg eq./kg), but only AMBA was found in nutmeat (up to 15% TRR, 0.002 mg eq./kg, in T1). TRR from [cyclohexane-2- ^{14}C] treatment were ≤ 0.01 mg eq./kg in T1 samples ranged from 0.01 to 0.02 mg eq./kg in T2. 4-hydroxy-mesotrione was only identified in hulls (7% TRR). The peanut oil fraction was shown to be composed primarily of ^{14}C -labelled neutral lipids.

In summary, the metabolic pathway of mesotrione following pre- and/or post-emergence foliar applications in cranberries, maize, rice, peanut and tolerant soya bean are similar. It proceeds via cleavage of the parent molecule to yield MNBA and reduction to AMBA, which either conjugated or degraded to MBA. Mesotrione is also hydroxylated in the cyclohexane-dione ring to give 4 or 5-hydroxy-mesotrione. Incorporation of radioactive residues into natural products (lignin cellulose sugar or lipid) was seen in all crops, except cranberry fruit. Residues in cranberry fruits were mostly mesotrione and AMBA (over 20% TRR each). Maize, soya and rice feed commodities contained mostly MNBA and AMBA (> 10% TRR in most cases). Residues in grains were low and mesotrione only represented higher than 10% TRR in tolerant soya bean seed.

Environment fate

The photolysis of [phenyl- ^{14}C] mesotrione and [cyclohexane- ^{14}C]-mesotrione was studied in silt loam soil treated at 0.3 kg ai/ha and incubated in local sunlight (latitude 37° 56') at 20 to 24°C. About half of the radioactivity was present as mesotrione at 12–13 DAT. MNBA and AMBA accounted for 2–8% TRR at 5 DAT, increasing up to 8% TRR at 30 DAT.

The metabolism of [phenyl-2- ^{14}C] or [cyclohexane- ^{14}C]-mesotrione applied to various soils at rates ranging from 0.165 to 0.85 kg ai/ha and kept under aerobic conditions in the dark at 25±1°C for 28 to 60 days was investigated. Mesotrione degrades relatively fast, with DT₅₀ values ranging from 4.5 to 32 days. DT₅₀ for MNBA was < 2 days in these studies.

In two water sediment systems experiments conducted with either [phenyl-2- ^{14}C] or [cyclohexane- ^{14}C]-mesotrione at 0.20 kg ai/ha and incubated in the dark for 101 days, showed DT₅₀ were from 3 to 6 days, with mesotrione in the sediment never exceeding 4% AR. MNBA and AMBA were found in both water and sediment, starting at day 3.

The aerobic degradation of [phenyl-2- ^{14}C]-AMBA was studied in soils incubated up to 60 days in the dark, showing DT₅₀ ranging from 2 to 6 days.

Field studies

In six studies conducted with soils collected from different regions of Europe, mesotrione was applied at 0.15–0.2 kg ai/ha. MNBA and AMBA were detected at 6 DAT in 0–10 cm horizon (0.031 and 0.006 mg eq./kg, respectively). No residues of mesotrione or metabolites were detected in the soil below 10 cm. DT₅₀ ranged from 2 to 8 days.

In one study conducted with four soils from England and USA treated with MNBA at 0.22 kg ai/ha, DT₅₀ ranged from 0.6 to 10.6 days.

Confined rotational crops

Endive, radish and wheat were sown 120 days after a sandy loam soil being treated with [phenyl- ^{14}C] or [cyclohexane-2- ^{14}C]-mesotrione at 0.165 kg ai/ha. Endive was harvested at 78–63 days after planting (DAP), radish roots and leaves at 56 DAP, wheat forage at 22 DAP, wheat hay at 57 DAP and wheat grain and straw at 134–131 DAP. In the [phenyl- ^{14}C] experiment, residues in soil declined to 34% of the applied radioactivity (AR) at 120 DAT, with the most abundant metabolites being MNBA (8% AR) and AMBA (2% AR); mesotrione accounted for only 0.1% AR. TRR were

0.02 to 0.04 mg eq./kg in wheat forage, hay and straw, mostly MNBA (0.011 mg eq./kg in forage). Residues were 0.006 mg eq./kg in wheat grain, 0.014 mg eq./kg in endive and 0.004 mg eq./kg in radish root and leaves. TRR in all cyclohexane-2-¹⁴C samples were < 0.005 mg eq./kg in endive, and wheat straw, and were not further characterized.

[Phenyl-U-¹⁴C] or [cyclohexane-2-¹⁴C]-mesotrione was applied at 0.308 kg ai/ha (T1, characteristic of pre-emergence) and 0.462 kg ai/ha (T2, characteristic of pre + post-emergence) onto a sandy loam soil, and wheat, soya, endive or radish planted at 30, 120 and/or 300 DAT. Residues in soil declined to 27% AR at 300 DAT. Residues in wheat commodities from the [phenyl-U-¹⁴C] experiment were higher in straw (2.58 mg eq./kg at 30 DAT, T1). In wheat grain, residues were 0.038 mg eq./kg at 30 DAT (T1) and 0.014–0.015 mg eq./kg at 120 and 300 DAT (T2). At 30 DAT (T1), the major identified metabolite was MNBA, with residues ranging from 0.17 to 0.63 mg eq./kg in wheat forage, hay and straw and 0.003 mg eq./kg in grain. AMBA was mostly present as sulphate conjugate (total of 0.67 mg eq./kg in straw), and mesotrione and its 4-OH metabolite were only detected in forage (0.01 mg eq./kg).

At 30 DAT (T1), residues were 0.145 mg eq./kg in soya bean, and 0.46–0.64 mg eq./kg in soya feed. MNBA was 0.17–0.31 mg eq./kg in forage and hay and 0.014 mg eq./kg in soya bean. AMBA levels were 0.02–0.07 mg eq./kg. Residues in endive and radish ranged from 0.037 to 0.053 mg eq./kg at 120 DAT, declining to 0.005 to 0.019 mg eq./kg at 300 DAT (T2). The major residue was MNBA (0.02 mg eq./kg at 120 DAT, T2, in endive and radish tops).

Highest residues from [cyclohexane-2-¹⁴C] experiment were found at 30 DAT, T1: 0.05 - 0.06 mg eq./kg in wheat feed, 0.01 mg eq./kg in wheat grain, and 0.02–0.03 mg eq./kg in soya bean samples. Residues in endive and radish were < 0.01 mg eq./kg. Mesotrione and 4-hydroxy mesotrione were identified in wheat and soya bean feed (< 0.01 mg eq./kg at 30 DAT, T1).

In summary, mesotrione degrades quickly in soil under aerobic conditions. Although mesotrione is relatively stable to hydrolysis at pH 5–9 (less than 10% degradation after 30 days at 25 °C), it degrades rapidly in flooded systems with a half-life of approximately 4 days. Mesotrione metabolites, mainly MNBA, are expected in wheat and soya bean feed, endive and radish root when the crops are planted up to 120 days after the soil is treated with mesotrione at 0.3 kg ai/ha rate or higher. As currently the compound is used at rates lower than 0.3 kg ai/ha, no residues arriving from the use of mesotrione are expected in rotational crops.

Methods of analysis

Mesotrione residues in vegetable crops may be analysed by LC-MS/MS (negative mode, $m/z=338 \rightarrow 291$) after extraction with acetonitrile/water and cleaned up by SPE. Recovery data for mesotrione in maize commodities and cranberries showed good performance (84–114% recovery, 3–21% RSD, $n=3-6$) at the 0.01 mg/kg (LOQ) to 10 mg/kg range. The method was used in various supervised trials, with recovery data for mesotrione and MNBA within the acceptable levels at the LOQ or higher.

A modified QuEChERS LC-MS/MS multi-residue method (no clean up with primary-secondary amine (PSA) is used) was validated for mesotrione in oranges, maize and oilseed rape, with a LOQ of 0.01 mg/kg.

In a reversed-phase HPLC-fluorescence method, mesotrione and MNBA residues are extracted with acetonitrile:water (1:1) and cleaned up on silica SPE. The extract is submitted to reversed phase HPLC, the mesotrione fraction converted to MNBA with H_2O_2 and reduced to AMBA using acidic $SnCl_2$ and the MNBA fraction reduced to AMBA. Each fraction is cleaned-up by C18 SPE, and the AMBA conversion product quantified by HPLC-fluorescence. The method was validated for corn commodities with a LOQ of 0.01 mg/kg.

In a GC-MS method, mesotrione and MNBA residues are extracted from corn commodities with acetonitrile:water (1:1), acidified, partitioned with methylene chloride, which is evaporated and the residue heated with Jones Reagent (Cr^{VI} oxide acid solution) to oxidize mesotrione to MNBA. The total MNBA is extracted with ethyl acetate, evaporated to dryness, and the residue reacted with 2-

iodopropane and potassium carbonate to form isopropyl ester of MNBA for analyse by GC-MS. The method determines both mesotrione and MNBA at a combined LOQ of 0.01 mg/kg.

The acetonitrile:water (1:1) extraction efficiency was radio-validated using incurred radioactive residues in forage. After extraction using a high speed homogeniser, an aliquot was partitioned three times into ethyl acetate, and residues of mesotrione and MNBA quantified by TLC with storage-phosphor autoradiography. Levels of mesotrione and MNBA in forage were similar to the results obtained after exhaustive extraction within the metabolism study.

Mesotrione and MNBA residues are extracted from milk and eggs with acetone and from animal tissues with an acetone:water, the extract acidified, partitioned into methylene chloride, and residues of mesotrione oxidised to MNBA using H₂O₂. MNBA is reduced with acidic SnCl₂ and AMBA determined by reversed phase HPLC-fluorescence detection. The LOQ was 0.01 mg/kg in all matrices. Mesotrione may also be determined in animal matrices using the modified QuEChERS, excluding PSA, at an LOQ of 0.01 mg/kg.

The analytical methods were considered fit for purpose to determine mesotrione alone or in combination with MNBA in plant and animal commodities at a LOQ of 0.01 mg/kg.

Stability under frozen conditions

Residues of mesotrione and/or MNBA in fortified samples of maize commodities, radish root, and soya bean seed at 0.1 mg/kg were stable under frozen conditions for at least 32 months (at least 80% of the residues remained, quantified as AMBA by HPLC-FL). Samples of blueberry, asparagus, sugarcane and okra fortified with mesotrione at 1.0 mg/kg were shown to be stable for at least 13 months when stored frozen (quantified by HPLC-MS/MS). The residue trials reports also include additional information on storage stability, and the samples were stored within the period that guaranteed the integrity of the residues at the time of analysis.

Definition of the residue

Metabolism studies conducted in cow, swine and poultry fed with ¹⁴C mesotrione at 6 to 11 ppm showed higher residues in liver and kidney, and ranged from 0.01 to 0.08 mg eq./kg in muscle, milk and in eggs. When detected, mesotrione was the main residue found in animal commodities, accounting for up to 18% TRR in cow liver and kidney, at least 70% TRR in tissues of swine and poultry, and up to 80% TRR in egg white. When cow was fed with ¹⁴C AMBA, residues reached a maximum of 0.05 mg eq./kg in kidney and fat, with over 60% as AMBA. Residues in other tissues and in milk were < 0.01 mg eq./kg

The Meeting agreed that the residue definition of mesotrione in animal commodities for enforcement and dietary exposure assessment is mesotrione.

The residues do not concentrate in fat and mesotrione has a log P_{ow} of 0.1, confirming that mesotrione is not fat soluble.

Mesotrione is a herbicide that can be applied to the soil pre and/or post emergence of the plant, with exception of cranberry, for which the use is foliar. The compound is rapidly degraded in soil. Metabolism study showed residues in cranberry fruits mostly as mesotrione (over 60% TRR) and AMBA (over 20% TRR). Metabolism studies conducted in tolerant soya bean, maize, rice and peanut showed higher residues in feed commodities, mostly as mesotrione (up to 28% TRR in rice tops), MNBA (up to 24% TRR in soya bean forage) and AMBA (up to 29% TRR in maize fodder). Total residues in edible commodities were low (≤ 0.03 mg eq./kg) and when characterized, showed mesotrione as the main residue.

The Meeting concluded that MNBA and AMBA appear to be of low toxicological concern. When the information was available, MNBA was not detected in any sample from the residue trials.

The Meeting agreed that mesotrione is an adequate marker for the uses of mesotrione in plants and is suitable for dietary intake assessment

The Meeting agreed in the following residue definition for both plant and animal commodities for enforcement and dietary risk assessment: *Mesotrione*

The residues are not fat soluble.

Residues of supervised residue trials on crops

Cranberry

GAP in USA for cranberries is 2 broadcast foliar applications at 0.28 kg ai/ha, PHI 45 days. In five trials using 2 applications, the first being at 0.388 kg ai/ha, residues were: < 0.01 mg/kg (5), indicating that no residues are expected when the product is applied at the GAP rate.

The Meeting agreed to recommend a maximum residue level of 0.01* mg/kg, and a STMR of 0 mg/kg for mesotrione in cranberries

Bush berries and cane berries

GAP in USA for bush and cane berries is 1 post-direct spray at 0.21 kg ai/ha before bloom, with no PHI specified.

In one trial conducted in blueberry in USA according to GAP (application at BBCH 59), residues were < 0.01 mg/kg (77 DAT). In five other trials where the application was done after bloom residues at 32 to 88 DAT were < 0.01 mg/kg.

In four trials conducted with raspberry at GAP, residues at 32 to 88 DAT were < 0.01 mg/kg (4) at 52 to 83 DAT.

As no residues above the LOQ were found even in the late application trials, the Meeting agreed to estimate a maximum residue level of 0.01* mg/kg and a STMR of 0 mg/kg for bush berries and cane berries

Okra

In USA, mesotrione can be applied either at pre-emergence (0.21 kg ai/ha) or post emergence (0.105 kg ai/ha). PHI in both cases is 28 days. Five post-emergence trials conducted according to GAP gave residues < 0.01 mg/kg (5).

The Meeting agreed to recommend a maximum residue level of 0.01* mg/kg, and a STMR of 0.01 mg/kg for mesotrione in okra.

Sweet corn

Mesotrione is registered in Germany for post-emergence use on sweet-corn (BBCH 12–18) at 0.15 kg ai/ha. In four trials conducted in France at this GAP rate gave residues were: < 0.01 mg/kg (4) in the kernels and in the cob at 38 to 61 DAT.

In USA, mesotrione can be used in sweet corn via three application regimes: 1) one pre-emergence application at 0.27 kg ai/ha, 2) two post emergence applications, with a maximum of 0.21 kg ai/ha; or 3) 1× pre + 1× post emergence, with a maximum of 0.27 kg ai/ha. In all cases, the PHI is 45 days. The second application should be done up to the 8 leaf stage. In one trial, conducted according to regime 2, residues in the ears were: < 0.01 mg/kg (2); other two trials conducted at the same rate residue were the same 28 to 32 DAT. In 12 trials conducted at a higher rate (0.48 to 0.50 kg ai/ha; regimes 1 or 2 residues were: < 0.01 mg/kg from 23 to 36 DAT.

Although only one trial was conducted in USA according to GAP, 14 trials conducted at higher rates and/or lower PHI showed that no residues are expected in the ears of sweet corn after treatment according to GAP.

The Meeting agreed to estimate a maximum residue level of 0.01* mg/kg and a STMR of 0 mg/kg for mesotrione in sweet corn (kernels plus cob without husk).

Soya bean, dry

In USA, GAP for mesotrione in conventional soya is one pre-emergence application at 0.21 kg ai/ha, with no PHI specified. In 20 trials conducted according to GAP, residues at 117 to 174 DAT were < 0.01 mg/kg (20). Three trials conducted at higher rates (0.6–1 kg ai/ha) gave the same results.

GAPs for mesotrione tolerant soya are i) one pre-emergence or ii) early post-emergence (up to BBCH 13) application at 0.225 kg ai/ha, or iii) one pre + one post emergence application (BBCH 14–60) at 0.225 kg ai/ha and 0.125 kg ai/ha, respectively. Forty seven trials were conducted with tolerant soya using application using regimes 2 or 3, residues in the mature seeds were: < 0.01 (44) and 0.02 (3) mg/kg.

Using the data from trials conducted in tolerant crops, the Meeting agreed to estimate a maximum residue level of 0.03 mg/kg and a STMR of 0.01 mg/kg for mesotrione in soya bean, dry.

Asparagus

In the USA, mesotrione can be use in asparagus either as a pre-emergence application on the soil surface at 0.27 kg ai/ha in the spring prior to spear emergence, one application after completion of harvesting directed to the weed at 0.105 kg ai/ha, or both at a maximum of 0.27 kg ai/ha. In eight trials conducted in USA using the pre-emergence GAP, residues at 8 to 18 DAT were < 0.01 mg/kg (8). In 16 other trials the application was done after emergence of the plant.

The Meeting agreed to estimate a maximum residue level of 0.01*mg/kg and a STMR of 0.01 mg/kg for mesotrione in asparagus.

Rhubarb

In USA, mesotrione can be use in rhubarb as pre-emergence application on the soil surface prior to any spring green-up at 0.21 kg ai/ha and 21 days PHI. In four trials conducted at GAP rate, residues at 28 to 42 DAT were < 0.01 mg/kg. Four trials were conducted at higher rates and gave the same result.

As the PHI is not relevant to a pre-emergence application, the Meeting agreed to estimate maximum residue level of 0.01* mg/kg and a STMR of 0.01 mg/kg for mesotrione in rhubarb.

Maize

Mesotrione is registered in Germany for post-emergence use on maize (BBCH 12–18) at 0.15 kg ai/ha. In two trials conducted in Germany and UK at this GAP gave results at 112 to 143 DAT of < 0.01 mg/kg (2).

In the USA, mesotrione can be used in maize in three application regimes: 1) one pre-emergence at 0.27 kg ai/ha, 2) two post emergence, with a maximum of 0.21 kg ai/ha; or 3) 1× pre + 1× post emergence, with a maximum of 0.27 kg ai/ha. The second application should be done up to the 8 leaf stage. In all cases, the PHI was 45 days. Eight trials were conducted in Canada and the USA using regime 1 and 32 trials using regime 3 at rates higher than USA GAP. Grain harvested at 68 to 145 DAT gave residues < 0.01 mg/kg.

The results from North American trials conducted at higher rate show that no residues are expected in maize grain after treatment according to GAP.

The Meeting agreed to estimate a maximum residue level of 0.01*mg/kg and a STMR of 0 mg/kg for mesotrione in maize grain.

Millet

Mesotrione is registered in the USA as one pre-emergence use at 0.21 kg ai/ha, and no PHI specified. In five trials conducted according to GAP residues were: < 0.01 mg/kg (5) in millet grain (84 to 132 DAT).

With the support from the data from other cereals, the Meeting agreed to estimate a maximum residue level of 0.01*mg/kg and a STMR of 0 mg/kg for mesotrione in millet grain.

Oat

Mesotrione is registered in USA either as one pre-emergence use at 0.21 kg ai/ha or as a post-emergence application at 0.105 kg ai/ha. PHI is 50 days. In sixteen post-emergence trials conducted at GAP, residues in oat grain were < 0.01 mg/kg (16). Two trials conducted at up to 5 times the rate gave the same results.

The Meeting agreed to estimate a maximum residue level of 0.01*mg/kg and a STMR of 0 mg/kg for mesotrione in oat grain.

Rice

Mesotrione is registered in paddy rice in Republic of Korea as post-planting into the water (5–7 days after transplanting) at 1×0.09 kg ai/ha and no PHI specified. Ten trials were conducted in Republic of Korea and Japan using either a single application at higher rate, two applications at the GAP rate and/or applying latter in the season. In all cases, residues at 45 to 140 DAT were < 0.01 mg/kg. Although the trials were not according to GAP, these results indicate that no mesotrione residues are expected when the product is used according to GAP.

The Meeting agreed to estimate a maximum residue level of 0.01*mg/kg and a STMR of 0 mg/kg for mesotrione in rice grain, husked.

Sorghum

The registered use for mesotrione in sorghum in the USA is one pre-emergence application at 0.224 kg ai/ha up to 21 days before planting. In nine trials conducted according to GAP, residues at 78 to 134 DAT were < 0.01 mg/kg (9). Twelve post-emergence trials gave the same results.

The Meeting agreed to estimate a maximum residue level of 0.01*mg/kg and a STMR of 0 mg/kg for mesotrione in sorghum.

Sugar cane

The GAP for mesotrione in sugarcane in the USA is either two post-emergence applications at 0.10 kg ai/ha to the base of the sugar cane or a combination of one pre- and one post-emergence application not exceeding a total rate of 0.36 kg ai/ha. PHI is 114 days. In twenty four trials conducted according to either of the GAPs in USA gave residues of < 0.01 mg/kg (24) from 30 to 118 DAT. Two trials conducted at 3 or 5× the rate gave the same results within 114 days PHI.

In South Africa, GAP is a single early post-emergence at 0.15 kg ai/ha, and 181 days PHI. In four trials conducted at higher rate no residues were detected.

The Meeting agreed to estimate a maximum residue level of 0.01*mg/kg and a STMR of 0 mg/kg for mesotrione in sugar cane.

Linseed

Mesotrione is registered in the USA for linseed at one pre or one post-emergence application at 0.21 kg ai/ha, and no PHI specified. Five pre-emergence trials conducted according to GAP gave residues of < 0.01 mg/kg (5). Two post-emergent trials conducted at higher rates gave the same result.

The Meeting agreed to estimate a maximum residue level of 0.01* mg/kg and a STMR of 0.01 mg/kg for mesotrione in linseed.

*Animal feed**Forage*

Mesotrione is registered in Germany for post-emergence use on maize (BBCH 12–18) at 0.15 kg ai/ha, and no PHI specified. In three trials conducted in maize in France, Germany and the UK matching German GAP, residues in stover (remaining plant) at 41–47 DAT were 0.01 mg/kg (3).

In the USA, mesotrione can be used in maize under three application regimes: 1) one pre-emergence at 0.27 kg ai/ha; 2) two post emergence, with a maximum of 0.21 kg ai/ha; or 3) 1× pre + 1× post emergence, with a maximum of 0.27 kg ai/ha. In all cases, PHI was 45 days for forage and stover. The second application should be made up to the 8 leaf stage (or BBCH 19).

In one trial conducted in USA according to GAP, residues in maize forage were 0.12 mg/kg.

Mesotrione is registered in USA in millet as one pre-emergence use at 0.21 kg ai/ha. In five trials conducted at GAP, residues in forage were < 0.01 mg/kg (5).

The Meeting estimated a median residue and a highest residue of 0.01 mg/kg for mesotrione in millet forage

Mesotrione is registered in oats in USA either as one pre-emergence use at 0.21 kg ai/ha or as post-emergence application at 0.105 kg ai/ha. In 16 pre-emergence trials and 16 post-emergence US trials matching GAP, residues in oat forage were < 0.01 mg/kg (32).

The Meeting estimated a median residue and a highest residue of 0.01 mg/kg for mesotrione in oat forage.

The registered use for mesotrione in sorghum in USA is one pre-emergence application at 0.224 kg ai/ha up to 21 days before planting. In 13 trials conducted according to GAP, residues in sorghum forage were < 0.01 mg/kg (13).

The Meeting estimated a median residue and a highest residue of 0.01 mg/kg for mesotrione in sorghum forage.

Hay

In 16 pre-emergence trials and 16 post-emergence trials conducted in oats in USA, matching GAP, residues in oat hay were < 0.01 mg/kg (32). Post-emergence application trials gave the same results.

In five trials conducted in millet at GAP, residues in hay were < 0.01 mg/kg.

The Meeting estimated a median residue of 0.01 mg/kg and a highest residue of 0.01 mg/kg for mesotrione in oat hay and millet hay.

Straw

Mesotrione is registered in paddy rice in Korea as post-planting into the water (5–7 days after transplanting) at 1×0.09 kg ai/ha and no PHI specified. In eight trials conducted in Japan at this GAP gave residues in straw of < 0.002 mg/kg.

The Meeting agreed to estimate a maximum residue level of 0.01* mg/kg for mesotrione in rice straw and fodder, dry

The Meeting estimates a median residue and a highest residue of 0.01 mg/kg for mesotrione in rice straw

Fate of residues during processing

A processing study conducted with soya bean containing 0.04 mg/kg mesotrione showed residues of 0.01 mg/kg in the meal and 0.07 mg/kg in flour, with calculated processing factors of 0.25 and 1.8 mg/kg, respectively. Residues in soya oil, milk, tofu, sauce and miso were < 0.01 mg/kg, with an estimated processing factor of < 0.25. Based on a STMR of 0.01 mg/kg for soya bean, dry, the Meeting estimated a STMR-P of 0.018 mg/kg in soya bean flour, and of 0.002 mg/kg for soya oil, milk, tofu, sauce and miso.

Residue in animal commodities

Farm dietary burden

The Meeting estimated the dietary burden of mesotrione in farm animals on the basis of the OECD Animal Feed data published in the 2009 FAO Manual, and the median and highest residue levels estimated at the present Meeting for oat and sorghum forage, oat hay and rice straw.

The maximum and the mean dietary burden was 0.03 ppm for cattle, 0.01 and 0.001 ppm, for swine, respectively, and 0 ppm for poultry.

Animal commodity maximum residue level

No feeding study on mesotrione was provided to the Meeting. The metabolism study conducted with cattle at 10 ppm, gave residues of mesotrione up to 0.02 mg/kg in tissues and milk. Swine fed with radiolabeled mesotrione at 6 ppm gave residues of 1.5 mg/kg in liver, 0.09 mg/kg in kidney and 0.01 mg/kg in muscle. Interpolation of the residues found in the metabolism studies to what would be expected at the calculated dietary burden indicates that no residue will exceed 0.0025 mg/kg (in swine liver).

The Meeting agreed to estimate a maximum residue level of 0.01* mg/kg for mesotrione in milks, edible offal (mammalian) and meat (from mammals other than marine mammals).

The Meeting also estimated a STMR of 0 for mesotrione in milk and meat (from mammals other than marine mammals), and edible offal (mammalian).

Metabolism study conducted with poultry at 11 ppm showed mesotrione residues of 1.1 mg/kg in liver, 0.03 mg/kg in fat, < 0.01 mg/kg in meat and 0.02 mg/kg in eggs. As the dietary burden for poultry is 0, the Meeting agreed to estimate a maximum residue level of 0.01* mg/kg, and a STMR and a HR of 0 mg/kg for mesotrione poultry meat, poultry offal and eggs.

RECOMMENDATIONS

Definition of the residue (for compliance with the MRL and for estimation of dietary intake for plant and animal commodities): mesotrione.

The residue is not fat soluble.

CCN	Commodity name	Maximum residue level (mg/kg)	STMR (P) (mg/kg)
VS 0621	Asparagus	0.01*	0.01
FB 2006	Bush berries	0.01*	0
FB 2005	Cane berries	0.01*	0
FB 0265	Cranberry	0.01*	0
MO 0105	Edible offal (mammalian)	0.01*	0
PE 0112	Eggs	0.01*	0
SO 0693	Linseed	0.01*	0.01
GC 0645	Maize	0.01*	0
MM 0095	Meat (from mammals other than marine mammals)	0.01*	0
GC 0646	Millet	0.01*	0
MI 0106	Milks	0.01*	0
GC 0647	Oat	0.01*	0
VO 0442	Okra	0.01*	0.01
PO 0111	Poultry, Edible offal of	0.01*	0
PM 0110	Poultry meat	0.01*	0
CM 0649	Rice, husked	0.01*	0
VS 0627	Rhubarb	0.01*	0.01
GC 0651	Sorghum	0.01*	0

VD 0541	Soya bean, dry	0.03	0.01
	Miso		0.002
	Soya flour		0.018
	Soya milk		0.002
	Soya oil		0.002
	Soya sauce		0.002
	Tofu		0.002
GS 0659	Sugarcane	0.01*	0
VO 0447	Sweet corn (kernels plus cob without husk)	0.01*	0

Animal Feed items

	Median residue, mg/kg	Highest residue, mg/kg
Millet forage	0.01	0.01
Oat forage	0.01	0.01
Sorghum forage	0.01	0.01
Millet hay	0.01	0.01
Oat hay	0.01	0.01
Rice straw	0.01	0.01

DIETARY RISK ASSESSMENT

Long-term intake

The IEDI of mesotrione based on the STMRs estimated by this Meeting for the 17 GEMS/Food regional diets were 0% of the maximum ADI of 0–0.3 mg/kg bw (see Annex 3 of the 2014 Report). The Meeting concluded that the long-term dietary intake of residues of mesotrione is unlikely to present a public health concern.

Short-term intake

The 2014 JMPR decided that an ARfD is unnecessary for mesotrione. The Meeting therefore concluded that the short-term intake of residues of mesotrione is unlikely to present a public health concern.

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METRAFENONE (278)

The first draft was prepared by Mr David Lunn, Food Safety Authority, Wellington, New Zealand

EXPLANATION

Metrafenone is a benzophenone fungicide, active mainly against powdery mildews and eyespot, inhibiting mycelium growth, leaf penetration, haustoria formation and sporulation.

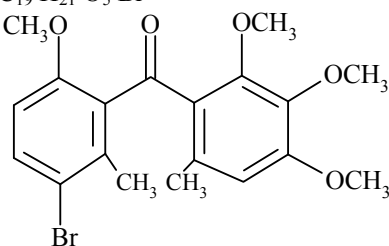
Authorisations exist for the use of metrafenone on cereals, grapes, strawberries and fruiting vegetables in over 50 countries in Europe, the Americas, Asia and the Pacific.

Metrafenone was scheduled by the Forty-fifth Session of the CCPR as a new compound for consideration by the 2014 JMPR. Residue and analytical aspects of metrafenone were considered for the first time by the present meeting. The manufacturer submitted studies on metabolism, analytical methods, supervised field trials, processing, freezer storage stability, environmental fate in soil and rotational crop residues.

In this evaluation, the values presented in the tables are as reported in the various studies, but in the accompanying text, they have generally been rounded to two significant digits.

IDENTITY

ISO common name:	Metrafenone
Code number	BASF 560 F
IUPAC name:	3'-bromo-2,3,4,6'-tetramethoxy-2',6-dimethylbenzophenone
Chemical Abstracts name:	(3-bromo-6-methoxy-2-methylphenyl) (2,3,4-trimethoxy-6-methylphenyl)-methanone
CAS number	220899-03-6
CIPAC number	752
Molecular mass:	409.3
Molecular formula	C ₁₉ H ₂₁ O ₅ Br
Structural formula:	

**PHYSICAL AND CHEMICAL PROPERTIES***Pure active ingredient*

A detailed chemical and physical characterisation of the active ingredient, technical grade and selected metabolites are given in Tables 1 and 2.

Table 1 Physical and chemical data of metrafenone (active ingredient) and metabolites CL 377160, CL 4084564 and CL 375816

Test or Study & Annex point	Test material purity and specification	Findings and comments	Reference
Melting point	Pure ai (99.5%)	99.2–100.8 °C	1998/7000370
Boiling point	Pure ai (99.5%)	No boiling point measured decomposition at ca. 310 °C (to a black tar)	1998/7000371
Relative density	Pure ai (99.7%)	1.45	1999/7000299
Vapour pressure	Pure ai (99.7%)	1.53·10 ⁻⁴ Pa at 20 °C 2.56·10 ⁻⁴ Pa at 25 °C	2001/5002313

Test or Study & Annex point	Test material purity and specification	Findings and comments	Reference
Henry's law constant	calculated	$K_H = 0.132 \text{ Pa m}^3 \text{ mol}^{-1}$ (20 °C)	2002/7004412
Appearance	Pure ai (99.5%)	White to chalky-white crystalline solid, light musty smell	1998/7000367 1998/7000369
Solubility in water	Pure ai (99.5%)	0.474 mg/L in deionized water 0.552 mg/L in pH 5 buffer 0.492 mg/L in pH 7 buffer 0.457 mg/L in pH 9 buffer	1998/7000347
	CL 377160 metabolite (97%)	1.1 mg/L in deionized water (pH ca. 4.0, 20 °C) 1.0 mg/L in acidified water (pH 2.8, 20 °C) 175 mg/L in basified water (pH 11.6, 20 °C)	2001/1019497
n-octanol/water partition coefficient	Pure ai (99.5%)	$\text{Log } K_{ow} 4.3$ (pH 4.0, 25 °C)	1999/7000293
	CL 4084564 Metabolite	$\text{Log } K_{ow} 3.52$ (neutral form)—pH ca 8 $\text{Log } K_{ow} 0.37$ (ionized form)—pH ca 8	2002/7005227
	CL 375816 Metabolite	$\text{Log } K_{ow} 3.01$ (neutral form) $\text{Log } K_{ow} 1.09$ (ionized form) pH dependent—dissociation of the carboxylic acid function.	2002/7005228
Hydrolysis (sterile buffer in the dark)	^{14}C labelled pure ai (> 99%) radiolabel purity 98.3%	Stable to hydrolysis in the dark after incubation for 5 days at 50 °C in pH 4, pH 7, pH 9 buffers	1999/7000284
Photolysis in sterile water	^{14}C labelled pure ai (> 98%)	Extensive degradation in sterile water after irradiation by simulated sunlight (15 days, pH 7, 22 °C) First order kinetics, rate constant: 0.225 day^{-1} , DT_{50} : 3.1 days, DT_{90} : 10.2 days) Multiple photoproducts observed, all < 10% AR After 15 days irradiation ca 97% conversion to very polar anionic materials and CO_2 .	2002/7005112
Photolysis in natural water	^{14}C labelled pure ai, radiochemical purity of > 99%	Rapid degradation in natural water under light at 22 °C (DT_{50} : 2.6 days, DT_{90} : 8.5 days) Many degradation products were formed, all < 10% AR. Main degradates included CL 377160, CL 377095, CL 377096, CL 4084564, and CL 375816. Maximum $^{14}\text{CO}_2$ formation ca. 5%.	2002/7004458
Dissociation in water	Pure ai (99.5%)	No evidence of dissociation—no hydrogens or basic groups present in the molecule and no appreciable differences in the UV spectra of the ai over a pH range of 1.0–13.0	1998/7000353
	CL 4084564 Metabolite	Estimated pK_a : 9.63 ± 0.3	2002/7005227
	CL 375816 Metabolite	Estimated pK_a : 3.35 ± 0.2 .	2002/7005228

Table 2 Physical and chemical data of metrafenone (technical grade material) and metabolites CL 377160, CL 4084564 and CL 375816

Test or Study & Annex point	Test material purity and specification	Findings and comments	Reference
Relative density	Technical (95.86%)	1.45	1999/7000296
Appearance	Technical (95.86%)	Yellow-white powdery, fine-crystalline solid, light musty smell	1998/7000368 1999/7000312 1999/7000311 1999/7000313
Solubility in organic solvents (g/L, 20 °C)	Technical (97.1%)	Dichloromethane: 1950 Acetone: 403 Toluene: 363 Ethyl acetate: 261 Acetonitrile: 165 Methanol: 26.1 n-Hexane: 4.8	1998/7000349

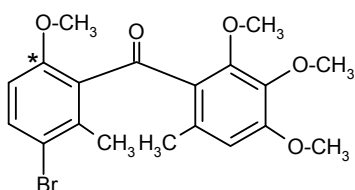
Formulations

Formulations of metrafenone are available for use as foliar applications, both as solo products or co-formulated with other actives.

Formulation type	Active substance/s and content	Other active ingredients
SC (Soluble Concentrate)	Metrafenone 300 g/L Metrafenone 500 g/L Metrafenone 100 g/L	— — Epoxiconazole 83 g/L
SE (Suspo-emulsion)	Metrafenone 75 g/L	Fenpropimorph 200 g/L + Epoxiconazole 62.5 g/L

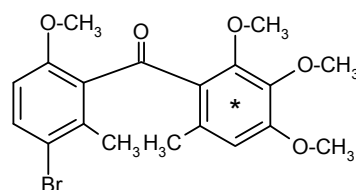
METABOLISM

The Meeting received metrafenone metabolism studies on animals (rats, lactating goats and laying hens), plants (grape, cucumber and wheat), soil and rotational crops. Metrafenone radiolabelled on the bromophenyl or the trimethoxyphenyl groups were used in these studies. The label positions are given below:



[Bromophenyl-6-¹⁴C]-metrafenone
(Bromophenyl-label)

* = location of the radiolabel



[Trimethoxyphenyl-U-¹⁴C]-metrafenone
(Trimethoxyphenyl-label)

* = location of the radiolabel

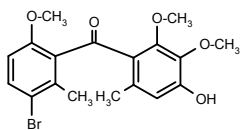
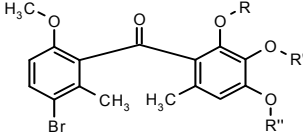
Major metabolites identified in these studies and discussed in this evaluation are listed below.

Table 3 Major metrafenone metabolites identified in plant, animal and soil matrices

CODE	STRUCTURE	CHEMICAL NAME	OCCURRENCE
CL 1023361		3-[3-bromo-2-(hydroxymethyl)-6-methoxybenzoyl]-2,6-dimethoxy-4-methylphenyl β-D-glucopyranosiduronic acid 3-[3-bromo-2-(hydroxymethyl)-6-methoxybenzoyl]-6-hydroxy-2-methoxy-4-methylphenyl β-D-glucopyranosiduronic acid 4-[3-bromo-2-(hydroxymethyl)-6-methoxybenzoyl]-2-hydroxy-3-methoxy-5-methylphenyl β-D-glucopyranosiduronic acid 4-[3-bromo-2-(hydroxymethyl)-6-methoxybenzoyl]-2,3-dimethoxy-5-methylphenyl β-D-glucopyranosiduronic acid	goat
CL 1023362		4-(3-bromo-6-hydroxy-2-methylbenzoyl)-2-hydroxy-3-methoxy-5-methylphenyl β-D-glucopyranosiduronic acid 3-(3-bromo-6-hydroxy-2-methylbenzoyl)-6-hydroxy-2-methoxy-4-methylphenyl β-D-glucopyranosiduronic acid	goat

CODE	STRUCTURE	CHEMICAL NAME	OCCURRENCE
CL 1023363		3-(3-bromo-6-methoxy-2-methylbenzoyl)-6-hydroxy-2-methoxy-4-methylphenyl β-D-glucopyranosiduronic acid 4-(3-bromo-6-methoxy-2-methylbenzoyl)-2-hydroxy-3-methoxy-5-methylphenyl β-D-glucopyranosiduronic acid	goat
CL 1500698		3-(3-bromo-6-methoxy-2-methylbenzoyl)-2,6-dimethoxy-4-methylphenyl β-D-glucopyranosiduronic acid	rat goat
CL 1500699		Methanone, (3-bromo-6-methoxy-2-methylphenyl)[4-(β-D-glucopyranurosyloxy)-2,3-dimethoxy-6-methylphenyl]-	goat
CL 1500701		Methanone, [3-bromo-6-(β-D-glucopyranurosyloxy)-2-methylphenyl](2,3,4-trimethoxy-6-methylphenyl)-	goat
CL 1500702		3-(3-bromo-6-hydroxy-2-methylbenzoyl)-2,6-dimethoxy-4-methylphenyl β-D-glucopyranosiduronic acid	goat
CL 1500831		1(3H)-isobenzofuranone, 4,5,6-trimethoxy-3-(3-bromo-6-methoxy-2-methylphenyl)-	wheat
CL 1500832		Methanone, [3-bromo-6-methoxy-(4 or 5)-hydroxy-2-methylphenyl](2,3,4-trimethoxy-6-methylphenyl)-	wheat
CL 1500833		Methanone, [3-bromo-6-methoxy-2-methylphenyl](2,3 or 4)-hydroxy-[2,3 or 4-dimethoxy-6-formylphenyl]-	wheat
CL 1500834		Benzaldehyde, 6-bromo-3-hydroxy-2-(2,3,4-trimethoxy-6-methylbenzoyl)-	wheat

CODE	STRUCTURE	CHEMICAL NAME	OCCURRENCE
CL 1500835	$R_1, R_2, R_3 = 1H, 2CH_3$	Methanone, [3-bromo-6-hydroxy-2-methylphenyl](2, 3 or 4)-hydroxy-[2, 3 or 4-dimethoxy-6-methylphenyl]-	wheat
CL 1500836		3-methoxy-2-(2,3,4-trimethoxy-6-methylbenzoyl) benzaldehyde	wheat grape
CL 1500837	$R_1, R_2, R_3 = 1H, 2CH_3$	Benzaldehyde, 6-bromo-3-methoxy-2-(2,3 or 4-dimethoxy-6-methylbenzoyl)-	wheat
CL 1500838		Methanone, (3-bromo-6-methoxy-2-methylphenyl)[4-(β-D-glucopyranosyloxy)-2-methylphenyl](2,3,4-trimethoxy-6-methylphenyl)-	wheat (glucose conjugate)
CL 1500839		Methanone, (3-bromo-6-(β-D-glucopyranosyloxy)-2-methylphenyl)(2,3,4-trimethoxy-6-methylphenyl)-	wheat
CL 197675		Methanone, (3-bromo-6-methoxy-2-carboxyl)(2,3,4-trimethoxy-6-methylphenyl)-	grape
CL 3000402		7-bromo-4-methoxy-3-(2,3,4-trimethoxy-6-methylphenyl)-2-benzofuran-1(3H)-one	wheat grape
CL 376991		Methanone, (3-bromo-6-methoxy-2-methylphenyl)(2,3,4-trimethoxy-6-methylphenyl)-	rat wheat
CL 377160		Methanone, (3-bromo-6-methoxy-2-methylphenyl)(3-hydroxy-2,4-dimethoxy-6-methylphenyl)-	wheat
CL 379395		2-(3-bromo-6-methoxy-2-methylbenzoyl)-3,4,5-trimethoxybenzaldehyde	grape

CODE	STRUCTURE	CHEMICAL NAME	OCCURRENCE
CL 434223		Methanone, (3-bromo-6-methoxy-2-methylphenyl)(4-hydroxy-2,3-dimethoxy-6-methylphenyl)-	wheat
M560F06	 R = H, R' and R'' = CH₃ or R' = H, R and R'' = CH₃ or R'' = H, R and R' = CH₃	Methanone, (3-bromo-6-methoxy-2-methylphenyl)(2-hydroxy-3,4-dimethoxy-6-methylphenyl)- or Methanone, (3-bromo-6-methoxy-2-methylphenyl)(3-hydroxy-2,4-dimethoxy-6-methylphenyl)- or Methanone, (3-bromo-6-methoxy-2-methylphenyl)(4-hydroxy-2,3-dimethoxy-6-methylphenyl)-	hen

Animal metabolism

The Meeting received animal metabolism studies on rats, lactating goats and laying hens, following oral dosing with [bromophenyl-6 (¹⁴C)] metrafenone or [trimethoxyphenyl-(U-¹⁴C)] metrafenone. As no cleavage of the molecule was observed in these metabolism studies, the results for both radiolabels are reported together.

Rats

The metabolism of metrafenone in rats was evaluated by the WHO Core Assessment Group of the 2014 JMPR. Absorption of metrafenone is rapid and complete (> 88%) at the low dose of 10 mg/kg bw, limited to 15–20% at the high dose of 1000 mg/kg bw suggesting saturation of the absorption processes. Metrafenone is widely distributed in the body, with highest residue levels mainly found in the gastro-intestinal (GI) tract, liver and fat. There is no evidence of accumulation. The labelled material is relatively rapidly excreted into the GI tract via the bile (85–90%) resulting in extensive excretion via faeces. Excretion via urine is relatively low (5–6% depending on radiolabel position), and even lower at the high dose level (*ca.* 1%). Metrafenone is extensively metabolised, with most of the radioactivity (*ca.* 80%) not identified, consisting of many (11–26) different components and totalling < 0.1 ppm at the low dose and < 1 ppm at the high dose. The identified metabolites, mostly < 1.0 mg eq/kg, included metrafenone and glucuronic acid conjugates in fat, liver and kidney.

Lactating goats

In a study reported by Fung, 2002 [Ref: 2002/7005114], the metabolism and distribution of [¹⁴C]metrafenone were investigated in lactating goats following repeated oral administration of radiolabelled metrafenone in gelatin capsules over five consecutive days at nominal dose levels equivalent to 10 ppm and 70 ppm in the diet (based on an average feed consumption of 2 kg per goat per day). The goat body weights ranged from 46–55 kg prior to treatment and 45–54 kg at sacrifice (21–23 hours after the last dose), and mean feed consumptions ranged from 1.5–2.3 kg.

[Bromophenyl-6-¹⁴C]-metrafenone, enriched with C¹³ (49%) as a mass marker to assist in mass spectrometric analysis of metabolites was administered to two goats at daily doses equivalent to 13 ppm or 87 ppm feed and [trimethoxyphenyl-(U-C¹⁴)] metrafenone enriched with C¹³ (49%) was administered to two goats at doses equivalent to 8 ppm or 87 ppm in the diet.

The majority of the radioactivity (76–86% AR) was excreted, mainly through the faeces. The highest residue levels were found in liver (0.21–0.23 mg eq/kg at the lower dose and 0.72–1.3 mg eq/kg at the higher dose) and kidney (0.05–0.06 mg eq/kg at the low dose and 0.16–0.33 mg eq/kg at the higher dose). Residues were significantly lower in fat (0.015–0.022 mg eq/kg)

and were < 0.01 mg eq/kg in muscle and up to 0.01 mg eq/kg in milk regardless of the dose rate. In milk, a plateau level not higher than 0.01 mg eq/kg was reached within about 3 days.

Table 4 Total radioactive residues in dissected tissues, excreta and milk of lactating goats following five daily oral administrations of [¹⁴C]metrafenone

	Treatment Groups			
Group ID	B	D	C	E
Radiolabel	Trimethoxyphenyl-label	Bromophenyl-label	Trimethoxyphenyl-label	Bromophenyl-label
Dose Group	Low	Low	High	High
Dose Rate (ppm)	8	13	60	87
Sample type	Total Radioactive Residues (TRR mg eq/kg)			
Liver	0.208	0.231	0.718	1.278
Kidneys	0.047	0.06	0.157	0.329
Muscle	< 0.005	< 0.005	0.006	0.008
Adipose tissue	< 0.005	< 0.005	0.022	0.015
Milk (Day 1)	< 0.005	< 0.005	0.005	0.009
Milk (Day 2)	< 0.005	< 0.005	0.006	0.009
Milk (Day 3)	< 0.005	< 0.005	0.006	0.009
Milk (Day 4)	< 0.005	< 0.005	0.006	0.008
Milk (Day 5)	< 0.005	< 0.005	0.006	0.01
Sample type	Excretion (% total dose)			
Urine + Faeces	85.7%	84.0%	82.2%	75.7%

Liver and kidney samples from the higher dose groups were sequentially extracted with acetonitrile, acetonitrile:water (80:20), methanol:acetone:water:trimethylamine (24:24:50:2) and methanol:water:trifluoroacetic acid (99:30:1). The post extraction solids were subject to pepsin hydrolysis followed by extraction with hydrochloric acid in methanol. Fat samples were triple-extracted with methanol. Milk samples were extracted with acetone, concentrated, redissolved in acetonitrile and partitioned with hexane, with the aqueous layer retained and the hexane extract partitioned with methanol. The two aqueous layers were then combined for analysis. The sample extracts were analysed by HPLC with a variable wavelength detector and radio detector within 15 days of sampling and the fractions were isolated for LC-MS analysis. A storage stability study indicated that residues of the glucuronide metabolites were stable in frozen storage for up to 3 months in liver, 6 months in kidney and that metrafenone was stable for up to 10 months in fat.

In liver, 95–97% TRR was able to be extracted (0.7–1.2 mg eq/kg) and 98–101% TRR (0.15–0.33 mg eq/kg) was extractable from kidney. The two sequential extractions of milk with acetone accounted for 99.9% TRR (ca 0.01 mg eq/kg).

Metrafenone was the predominant residue in fat (0.01–0.02 mg eq/kg), making up 60–85% TRR but was only found at 2.7–4.4% TRR in liver and kidney (0.025–0.035 mg eq/kg in liver and 0.005–0.014 mg eq/kg in kidney).

In liver and kidney, the predominant residues were two metabolites (CL 1500698 and CL 1023363) which together represented 15–21% of the TRR in liver (max 0.27 mg eq/kg) and 26–28% of the TRR in kidney (max 0.09 mg eq/kg). An additional group of three metabolites (CL 1023361, CL1023362 and CL 1500702) were found at up to 13% of TRR (0.17 mg eq/kg) in liver. While a number of other metabolites were present, these generally made up < 10% TRR.

In milk, residues of parent (24% TRR) and metabolites (up to 10% TRR) were all < 0.005 mg eq/kg and no metabolites were found in fat above 0.005 mg eq/kg (9% TRR).

Table 5 Characterisation and identification of radioactive residues in goat liver and kidney following five daily oral administrations of [^{14}C]metrafenone

Sample Type	Liver				Kidney			
Dose Group (Rate in Diet)	C (60 mg/kg)		E (87 mg/kg)		C (60 mg/kg)		E (87 mg/kg)	
Radiolabel	Trimethoxyphenyl		Bromophenyl		Trimethoxyphenyl		Bromophenyl	
Fractions	%TRR	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg
TRR	100	0.718	100	1.278	100	0.157	100	0.329
Combined extracts	96.8	0.695	94.6	1.21	97.7	0.153	100.6	0.331
Distribution of Extractable Radioactive Residues								
ROI-1 (Unknown)	5.99	0.043	3.60	0.046	5.54	0.009	6.00	0.020
ROI-2 (Unknown)	3.76	0.027	2.97	0.038	0.61	0.001	1.23	0.004
ROI-3 (Unknown)	11.7	0.084	5.94	0.076	1.21	0.002	1.82	0.006
ROI-4 (Unknown)	4.18	0.030	4.77	0.061	3.12	0.005	7.39	0.024
ROI-5 (Unknown)	5.01	0.036	3.60	0.046	3.82	0.006	5.23	0.017
ROI-6 (Unknown)	2.64	0.019	3.68	0.047	4.97	0.008	4.16	0.014
ROI-7 (Unknown)	4.73	0.034	5.09	0.065	1.53	0.002	1.25	0.004
ROI-8 (Unknown)	2.92	0.021	2.97	0.038	4.33	0.007	3.80	0.013
ROI-9A (CL 1023361) + ROI-9B1 (CL 1023362) + ROI-9B2 (CL 1500702)	9.89	0.071	13.23	0.169	13.57	0.021	9.51	0.031
ROI-10 (Unknown)	2.65	0.019	2.35	0.030	2.80	0.004	4.80	0.016
ROI-11 (Unknown)	2.93	0.021	12.11	0.027	1.66	0.003	2.13	0.007
ROI-12 (Unknown)	2.23	0.016	1.96	0.025	5.67	0.009	3.10	0.010
ROI-13A1 (CL 1500698) + ROI-13A2 (CL 1023363) + Unknown	14.77	0.106	21.05	0.269	28.15	0.044	26.29	0.087
ROI-14 (Unknown)	3.35	0.024	2.90	0.037	4.14	0.007	4.32	0.014
ROI-15 (Unknown)	2.09	0.015	1.88	0.024	4.08	0.006	1.43	0.005
ROI-16A (CL 1500701) + ROI-16B (CL 1500699) + Unknown	6.41	0.046	7.12	0.091	1.46	0.002	4.19	0.014
ROI-17 (Unknown)	1.53	0.011	1.72	0.022	0.64	0.001	1.22	0.004
ROI-18 (Unknown)	1.53	0.011	1.25	0.016	0.63	0.001	1.49	0.005
ROI-19 (Unknown)	0.98	0.007	0.70	0.009	0.49	0.001	1.28	0.004
ROI-20 (Unknown)	1.81	0.013	1.80	0.023	1.21	0.002	2.16	0.007
ROI-21 (Unknown)	1.67	0.012	1.33	0.017	2.36	0.004	2.28	0.008
ROI-22 (Metrafenone)	3.49	0.025	2.74	0.035	3.25	0.005	4.35	0.014

Table 6 Characterisation and identification of radioactive residues in goat milk and fat following five daily oral administrations of [^{14}C]metrafenone

Sample Type	Milk				Fat			
Dose Group (Rate in Diet)	C (60 mg/kg)		E (87 mg/kg)		C (60 mg/kg)		E (87 mg/kg)	
Radiolabel	Trimethoxyphenyl		Bromophenyl		Trimethoxyphenyl		Bromophenyl	
Fractions	%TRR	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg
TRR	100	0.006	100	0.010	100	0.022	100	0.015
Combined extracts ^a	NP	NP	98.9	~0.010	116	~0.026	112.6	0.020
Distribution of Extractable Radioactive Residues								
ROI-1 (Unknown)	NP	NP	5.3	< 0.005	8.0	< 0.005	9.1	< 0.005
ROI-2 (Unknown)	NP	NP	1.8	< 0.005	ND	ND	ND	ND
ROI-3 (Unknown)	NP	NP	2.2	< 0.005	ND	ND	ND	ND

Sample Type	Milk				Fat			
Dose Group (Rate in Diet)	C (60 mg/kg)		E (87 mg/kg)		C (60 mg/kg)		E (87 mg/kg)	
Radiolabel	Trimethoxyphenyl		Bromophenyl		Trimethoxyphenyl		Bromophenyl	
Fractions	%TRR	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg
TRR	100	0.006	100	0.010	100	0.022	100	0.015
Combined extracts ^a	NP	NP	98.9	~0.010	116	~0.026	112.6	0.020
Distribution of Extractable Radioactive Residues								
ROI-4 (Unknown)	NP	NP	2.9	< 0.005	ND	ND	ND	ND
ROI-5 (Unknown)	NP	NP	1.4	< 0.005	ND	ND	ND	ND
ROI-6 (Unknown)	NP	NP	1.6	< 0.005	ND	ND	ND	ND
ROI-7 (Unknown)	NP	NP	1.1	< 0.005	ND	ND	ND	ND
ROI-8 (Unknown)	NP	NP	2.0	< 0.005	ND	ND	ND	ND
ROI-9A (CL 1023361) + ROI-9B1 (CL 1023362) + ROI-9B2 (CL 1500702)	NP	NP	2.8	< 0.005	ND	ND	ND	ND
ROI-10 (Unknown)	NP	NP	1.0	< 0.005	ND	ND	ND	ND
ROI-11 (Unknown)	NP	NP	1.2	< 0.005	ND	ND	ND	ND
ROI-12 (Unknown)	NP	NP	1.3	< 0.005	ND	ND	ND	ND
ROI-13A1 (CL 1500698) + ROI-13A2 (CL 1023363) + Unknown	NP	NP	10.7	< 0.005	ND	ND	ND	ND
ROI-14 (Unknown)	NP	NP	2.8	< 0.005	ND	ND	ND	ND
ROI-15 (Unknown)	NP	NP	7.2	< 0.005	ND	ND	ND	ND
ROI-16A (CL 1500701) + ROI-16B (CL 1500699) + Unknown	NP	NP	3.8	< 0.005	ND	ND	ND	ND
ROI-17 (Unknown)	NP	NP	2.6	< 0.005	ND	ND	ND	ND
ROI-18 (Unknown)	NP	NP	5.0	< 0.005	ND	ND	ND	ND
ROI-19 (Unknown)	NP	NP	4.3	< 0.005	ND	ND	ND	ND
ROI-20 (Unknown)	NP	NP	6.7	< 0.005	ND	ND	ND	ND
ROI-21 (Unknown)	NP	NP	5.5	< 0.005	5.4	< 0.005	5.3	< 0.005
ROI-22 (Metrafenone)	NP	NP	24.1	< 0.005	85.4	0.019	60.0	0.009

NP—not performed

^a For milk, combined acetone extracts analysed by HPLC, for adipose tissues combined methanol extracts analysed by HPLC

In summary, when goats were treated orally for five consecutive days with metrafenone, the majority (76–86%) of the dose was excreted, with TRR being highest in liver and 0.01 mg eq/kg or less in milk and muscle. Metrafenone made up about 3–4% of the TRR in liver and kidney and was the main component in fat. Most of the residues in liver and kidney were the glucuronide conjugates of metrafenone (CL 1500698, CL 1023363) which together made up 15–30% TRR, with several other conjugates also found at lower levels.

The proposed metabolic pathway includes hydroxylation and demethylation of the methyl groups and the phase II glucuronidation of the hydroxylated metabolites to various mono-O-glucuronides, qualitatively similar to the metabolic pathway in the rat.

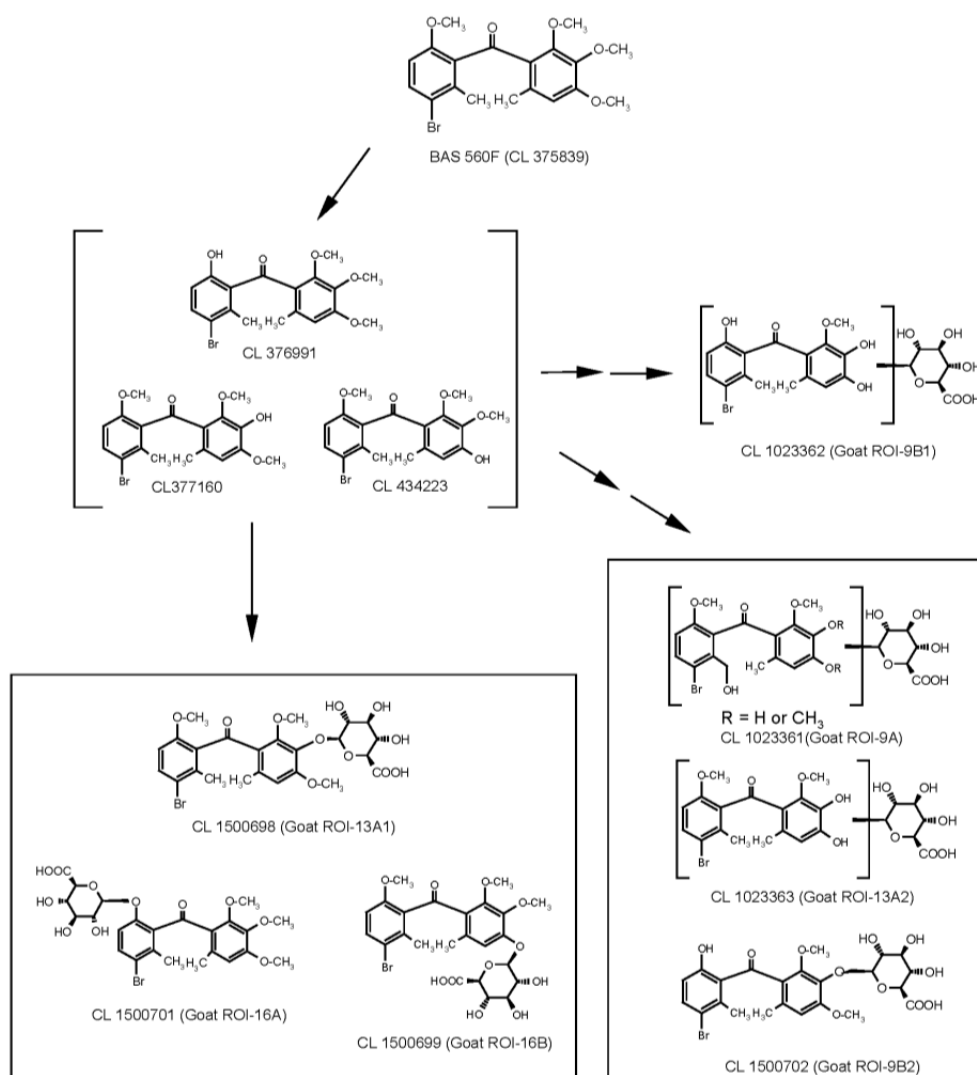


Figure 1 Metabolic pathway in lactating goats

Laying hens

In a study reported by Hoefs, 2008 [Ref: 2005/1026047], the metabolism and distribution of [¹⁴C]metrafenone were investigated in laying hens following repeated oral administration of radiolabelled metrafenone in gelatin capsules over twelve consecutive days at nominal dose levels equivalent to 12 ppm in the diet (based on an average feed consumption of 120 g per hen per day). The actual dose rates for the two groups of eight laying hens were 14.2 mg/kg (trimethoxyphenyl label) and 13.9 mg/kg (bromophenyl label). Average body weights for the two dose groups were 1.55 kg and 1.64 kg prior to treatment and 1.58 kg and 1.66 kg at sacrifice (22 hours after the last dose), and mean feed consumptions ranged from 102–185 g/day.

Overall recoveries of radioactivity were approximately 96 and 86% of the total applied dose for the trimethoxyphenyl and bromophenyl labels, respectively. Radioactivity was rapidly eliminated within 24 hours with 95% (trimethoxyphenyl label) and 86% (bromophenyl label) of the administered dose recovered in the excreta. About 0.25% AR was found in eggs and 0.06–0.09% AR was measured in liver. Radioactivity in skin + fat was < 0.01% AR and muscle contained 0.003% AR. TRRs in eggs reached a plateau about day 9–10.

Table 7 Total radioactive residues in dissected tissues, excreta and eggs of laying hens following 12 daily oral administrations of [^{14}C]metrafenone

Matrix	Dose Group A trimethoxyphenyl label		Dose Group B bromophenyl label	
	mg eq./kg	% AR	mg eq./kg	% AR
Eggs (Day 1)	< 0.001		< 0.001	
Eggs (Day 2)	0.016	0.003	0.018	0.003
Eggs (Day 3)	0.026	0.004	0.039	0.009
Eggs (Day 4)	0.054	0.013	0.055	0.012
Eggs (Day 5)	0.072	0.019	0.064	0.014
Eggs (Day 6)	0.086	0.024	0.102	0.025
Eggs (Day 7)	0.095	0.02	0.094	0.024
Eggs (Day 8)	0.097	0.027	0.106	0.031
Eggs (Day 9)	0.107	0.034	0.115	0.03
Eggs (Day 10)	0.109	0.028	0.117	0.035
Eggs (Day 11)	0.106	0.032	0.118	0.031
Eggs (Day 12)	0.110	0.032	0.118	0.036
Eggs (Days 9–12 pooled)	0.099	0.25	0.118	0.236
Muscle	0.010	0.003	0.013	0.003
Skin with fat	0.060	0.006	0.084	0.009
Liver	0.489	0.088	0.326	0.063
Bile	10.214	0.027	10.641	0.028
Excreta (Days 6–12 pooled)	14.758	85.9	13.289	95.1

Tissue and egg samples were extracted sequentially with three aliquots of methanol and two aliquots of water, with the combined methanol extracts and the combined water extracts being analysed by LSC. Residues in the post extraction solids were determined by LSC after combustion. Eggs were homogenized with an equal weight of water before extraction.

Extraction of pooled eggs (days 9–12) yielded 63% TRR (0.06–0.07 mg eq/kg) in the methanol extract and a further 17% (0.017 to 0.02 mg eq/kg) in water, giving a total of 80% TRR (0.079 to 0.094 mg eq/kg) as extractable residues. Unextractable residues accounted for about 20% TRR.

Low levels of radioactivity (0.01–0.013 mg eq/kg) were found in muscle with about 27–31% TRR extracted with methanol and a further 1–1.8% TRR extracted in water. Unextractable residues accounted for about 68–72% TRR (0.007 to 0.009 mg eq/kg).

About 58–61% TRR in skin + fat was able to be extracted and overall extractability in liver was about 30% TRR.

HPLC analysis of acetone-extracted egg samples and bile extracts stored for more than 19 months showed comparable metabolite patterns to those from samples analysed within 8 days of extraction.

Table 8 Extractability of [^{14}C]metrafenone residues in hen matrices following 12 daily oral administrations of [^{14}C]metrafenone.

Matrix	TRR ^a	Methanol	Water	ERR ^b	PES ^c	TRR ^d Recovery
	mg eq./kg	mg eq./kg (% TRR)	mg eq./kg (% TRR)	mg eq./kg (% TRR)	mg eq./kg (% TRR)	mg eq./kg [%]
Trimethoxyphenyl Label						
Eggs pooled (days 9–12)	0.099	0.062 (62.6)	0.017 (17.2)	0.079 (79.8)	0.020 (20.2)	0.099 (100)
Muscle	0.010	0.003 (26.8)	0.0001 (1.0)	0.003 (27.8)	0.007 (72.2)	0.010 (100)
Skin with fat	0.060	0.033 (53.9)	0.002 (4.1)	0.035 (58.0)	0.025 (42.0)	0.060 (100)
Liver	0.489	0.058 (11.8)	0.099 (20.2)	0.157 (32.0)	0.333 (68.0)	0.490 (100)
Excreta pool (days 6–12)	14.758	11.656 (79.0)	1.567 (10.6)	13.233 (89.6)	1.535 (10.4)	14.758 (100)
Bromophenyl Label						
Eggs pooled (days 9–12)	0.118	0.074 (62.7)	0.020 (16.9)	0.094 (79.7)	0.024 (20.3)	0.118 (100)
Muscle	0.013	0.004 (30.7)	0.0002 (1.8)	0.004 (32.5)	0.009 (67.5)	0.013 (100)
Skin with fat	0.084	0.048 (57.7)	0.002 (2.9)	0.050 (60.6)	0.033 (39.4)	0.083 (100)
Liver	0.326	0.036 (11.0)	0.060 (18.6)	0.096 (29.6)	0.230 (70.4)	0.326 (100)
Excreta pool (days 6–12)	13.289	9.965 (75.0)	1.548 (11.6)	11.513 (86.6)	1.776 (13.4)	13.289 (100)

^a TRR values reported as sum of radioactivity on methanol and water extract measured directly by LSC and in post extraction solids after combustion

^b ERR—extractable radioactive residues (sum of methanol and water extracts)

^c PES—Post extraction solids remaining after extraction with methanol and water

^d Sum of all extracts and post extraction solids

Characterisation of metabolites was performed by LC-MS and LC-MS/MS analysis. Metrafenone was found only in eggs (1.8–2.2% TRR) and in skin + fat (1.9% TRR). A comparison with the retention times and metabolite patterns for the components in the methanol extract of excreta (bromophenol-label), allowed the assignment of the metabolite M560F06 in the skin + fat extracts (at about 6–11% TRR) and tentatively in eggs (unquantifiable). With the exception of one unknown component in eggs (found at about 14% TRR and 0.015 mg eq/kg) all other components were below 10% TRR (< 0.01 mg eq/kg) in all tissues and eggs.

Table 9 Residues of metrafenone and other characterised components in hen matrices following following 12 daily oral administrations of [^{14}C]metrafenone.

Matrix	Metrafenone		Characterised (Methanol extract)		Characterised (Aqueous extract)	
	mg/kg	%TRR	mg eq./kg	%TRR	mg eq./kg	%TRR
Trimethoxyphenyl Label						
Eggs pooled (days 9–12)	0.002	2.2	0.04 ^a	37.5	0.009 ^b	8.4
Muscle	NA	NA	0.003	26.8	0.0001	1.0
Skin with fat	0.001	1.9	0.034 ^c	48.6		
Liver			0.097 ^d	17.3	0.07 ^e	12.5

Matrix	Metrafenone		Characterised (Methanol extract)		Characterised (Aqueous extract)	
	mg/kg	%TRR	mg eq./kg	%TRR	mg eq./kg	%TRR
Bromophenyl Label						
Eggs pooled (days 9–12)	0.002	1.8	0.041 ^f	35.1	0.01 ^g	8.0
Muscle	NA	NA	0.004	30.7	0.0002	1.8
Skin with fat	< 0.001	< 0.001	0.042 ^h	42.9		
Liver	< 0.001	< 0.001	0.096 ⁱ	27.8	0.058 ^j	16.6

^a Includes nine peaks, each < 0.01 mg eq/kg or < 1.5% TRR, including 1 peak tentatively identified as M560F06

^b Includes eight peaks, each < 0.01 mg eq/kg or < 3% TRR

^c Includes 12 peaks, each < 7% TRR, and one peak identified as M560F06 (0.008 mg eq/kg, 11.4% TRR)

^d Includes 10 peaks, each < 3.7% TRR

^e Includes 12 peaks, each < 1.6% TRR

^f Includes six peaks, each < 0.01 mg eq/kg or < 3% TRR, including one peak identified as M560F06

^g Includes eight peaks, each < 0.01 mg eq/kg or < 2.7% TRR

^h Includes 12 peaks, each < 9% TRR, and one peak identified as M560F06 (0.006 mg eq/kg (5.8% TRR)

ⁱ Includes six peaks, each < 4.5% TRR

^j Includes five unknown peaks, each < 4.1% TRR

In summary, after 12 consecutive daily oral administrations of [¹⁴C]metrafenone to laying hens, there was rapid absorption and almost complete excretion within 24 hours. The total radioactive residues in edible tissues and organs were about 0.11 mg eq/kg in eggs, up to 0.57 mg eq/kg in liver, about 0.012 mg eq/kg in muscle, and up to 0.1 mg eq/kg in skin with adhering fat.

Metrafenone made up about 2% of the TRR in eggs and skin + fat (0.002 mg/kg) and the M560F06 metabolite was tentatively identified in skin + fat and eggs. The proposed metabolic pathway for metrafenone involves:

Hydroxylation at the bromophenyl ring or the attached methyl group to form M560F05, which can be demethylated and conjugated with glucuronic acid in bile to form M560F07

Demethylation at the trimethoxyphenyl ring to form M560F06, with glucuronic acid conjugation in bile to form the metabolites M560F08 and M560F09

Oxidation of the methyl group on the bromomethoxytoluene ring to the carboxylic acid (via the respective isomer of M560F05) to form M560F03.

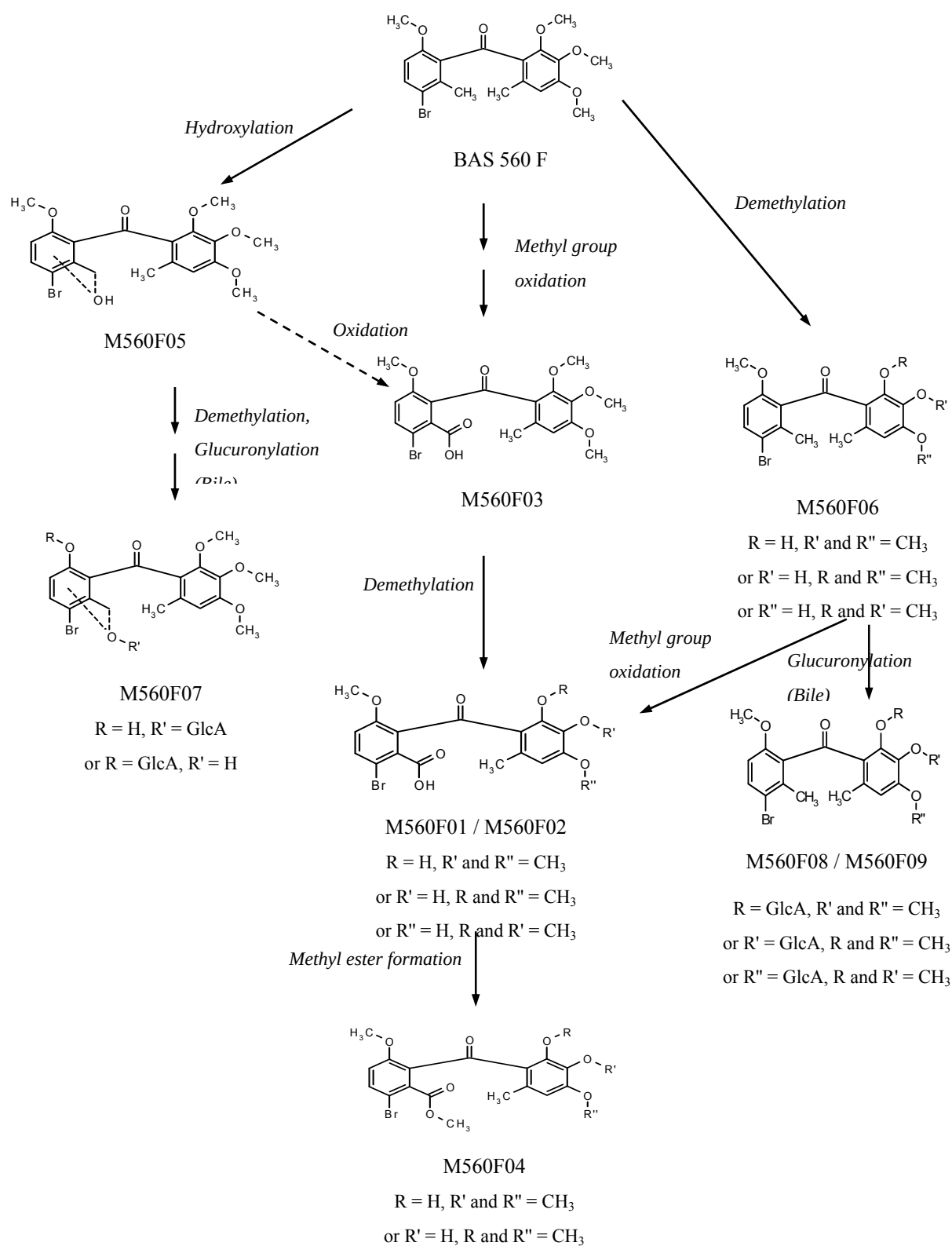


Figure 2 Metabolic pathway of metrafenone in laying hens

Plant metabolism

The Meeting received plant metabolism studies on grapes, cucumbers and wheat following foliar treatments with with [bromophenyl-6 (^{14}C)] metrafenone or [trimethoxyphenyl-(U- ^{14}C)] metrafenone.

Grape

In a study reported by Class & Schluter, 2001 [Ref: 2001/7000342], the metabolism of metrafenone in outdoor grapevines was investigated following five foliar applications at 10–11 day intervals of about 0.2 kg ai/ha of [^{14}C]metrafenone (trimethoxyphenyl-label or bromophenyl-label, both being diluted with ^{13}C as a mass marker). Grape and leaf samples were taken immediately after each application; nineteen days after the last treatment (61 days after the first application, BBCH 85) and at maturity, 35 days after the last application (77 days after the first application, BBCH 89).

Grapes from the second and third samples were separated into juice and marc, and the marc was sequentially extracted with acetone, methanol:water (4:1) and then water. Leaves were surface washed with acetone and then sequentially extracted with acetone, methanol:water (4:1) and water. The post-extraction solids from the mature leaf and grape samples were further extracted with HCl:methanol and then sequentially treated with pepsin, cellulose, surfactant and refluxed with 6 M HCl. Radioactivity was analysed by LSC or combustion LSC and characterisation of the radioactive residues was conducted by TLC and HPLC-UV and HPLC-MS.

TRRs (calculated from extracted and non-extracted radioactivity) immediately after the last treatment were 0.6–0.77 mg eq/kg in grapes immediately after the last treatment and about 40 mg eq/kg in leaves. At maturity, 35 days after the last treatment, TRRs were 0.28–0.44 mg eq/kg in grapes and 25–38 mg eq/kg in leaves (with about 39–45% TRR present in the leaf surface wash).

Table 10 Radioactive residues (mg eq./kg) in grapes and grape leaves following five foliar applications of [^{14}C]metrafenone

Sample	Bromophenyl-label					Trimethoxyphenyl-label				
	Grapes		Leaves			Grapes		Leaves		
	TRR	TER ^a	TRR	Surface ^b	TER ^a	TRR	TER ^a	TRR	Surface ^b	TER ^a
0 DAT1	0.43	0.361 (83.8)	30.67	26.87 (87.6)	30.64 (99.9)	0.552	0.55 (99.6)	21.92	18.93 (86.4)	21.96 (100.0)
0 DAT2	1.07	0.998 (93.7)	25.9	14.79 (57.1)	23.88 (92.2)	0.471	0.421 (89.4)	20.41	13.27 (65.0)	19.31 (94.6)
0 DAT3	0.386	0.341 (88.3)	42.58	23.77 (55.8)	37.94 (89.1)	0.326	0.283 (86.8)	37.43	22.79 (60.9)	33.94 (90.7)
0 DAT4	0.241	0.2 (83.0)	59.22	32.27 (54.5)	54.31 (91.7)	2.1	1.61 (76.5)	52.41	33.62 (64.1)	49.14 (93.8)
0 DAT5	0.768	0.745 (97.0)	39.82	18.88 (47.4)	31.34 (78.7)	0.604	0.487 (80.6)	42.37	19.98 (47.2)	35.66 (84.2)
19 DAT5	0.314	0.26 (82.8)	59.11	22.23 (37.6)	45.68 (77.3)	0.15	0.135 (90.0)	55.48	23.78 (42.9)	43.59 (78.6)
35 DAT5	0.442	0.389 (88.0) ^c	38.13	17.14 (44.9)	35.88 (94.1) ^c	0.275	0.256 (93.1) ^c	24.74	9.57 (38.7)	22.75 (91.9) ^c

^a TER = Total extracted radioactivity in mg eq./kg (%TRR)

^b Extracted radioactivity in acetone surface wash in mg eq./kg (%TRR)

^c Includes enzyme/acid extraction

Table 11 Radioactive residues in grape juice and marc following five foliar applications of [^{14}C]metrafenone

Sample	Juice		Grape solids (marc)		Non-extractable residue	
	mg eq./kg	%TRR	mg eq./kg	%TRR	mg eq./kg	%TRR
Bromophenyl-label						
0 DAT1	NA	NA	0.361	83.8	0.070	16.2

Sample	Juice		Grape solids (marc)		Non-extractable residue	
	mg eq./kg	%TRR	mg eq./kg	%TRR	mg eq./kg	%TRR
0 DAT2	0.031	2.9	0.967	90.7	0.088	6.4
0 DAT3	0.024	6.3	0.317	82.1	0.045	11.6
0 DAT4	0.021	8.7	0.178	73.8	0.042	17.4
0 DAT5	0.054	7.1	0.690	89.9	0.023	3.0
19 DAT5	0.060	19.2	0.201	63.9	0.053	16.9
35 DAT5	0.080	18.0	0.277	62.6	0.086	19.4
Trimethoxyphenyl-label						
0 DAT1	NA	NA	0.550	99.7	0.002	0.3
0 DAT2	0.012	2.6	0.408	86.7	0.050	10.7
0 DAT3	0.019	5.8	0.264	81.1	0.043	13.1
0 DAT4	0.224	10.7	1.384	65.9	0.493	23.5
0 DAT5	0.075	12.5	0.413	68.3	0.116	19.3
19 DAT5	0.049	32.6	0.086	57.5	0.015	9.8
35 DAT5	0.070	25.3	0.174	63.1	0.032	11.6

NA = not applicable

Characterisation of the residues in juice indicated the presence of several metabolites more polar than parent. One of these, CL197675, made up about 9% TRR (0.006 mg eq/kg), with the others present at lower concentrations. Metrafenone was not found. Low levels of radioactivity precluded further investigation.

In pomace extracts, metrafenone was the major residue, making up about 23–25% TRR (0.06–0.11 mg eq/kg), with other, more polar fractions not exceeding 0.05 mg eq/kg (12–17% TRR). These fractions were not investigated further.

Analysis of leaf samples showed rapid metabolism to several polar compounds. At maturity (35 days after the last treatment), unchanged parent had decreased to about 15% TRR (5.8 mg/kg) and 11% (2.7 mg/kg) for the bromophenyl-label and trimethoxyphenyl-label, respectively. None of the degradation products could be positively identified against the reference standards. However analysis of fragmentation patterns allowed for the proposal of several metabolite structures, identified as CL3000402, CL379395 and CL1500836. As with grapes, no other extracts could be investigated due to low levels of radioactivity.

Table12 Residues of unchanged metrafenone in grape leaf samples following five foliar applications of [^{14}C]metrafenone

Sample	Bromomethyl-label				Trimethoxyphenyl-label			
	Metrafenone		Other metabolites		Metrafenone		Other metabolites	
	mg/kg	%TRR	mg eq./kg	%TRR	mg/kg	%TRR	mg eq./kg	%TRR
0 DAT1	26.6	86.9	4.07	13.3	20.3	92.4	1.62	7.4
0 DAT2	9.6	36.9	16.3	62.9	10.6	52.2	9.81	48.1
0 DAT3	15.6	36.6	26.98	63.4	13.9	37.2	23.53	62.9
0 DAT4	20.2	33.7	39.22	66.2	17.1	32.7	35.31	67.4
0 DAT5	14.2	35.6	25.62	64.3	14.2	33.5	28.17	66.5
19 DAT5	8.4	14.2	50.71	85.8	9.8	17.6	45.68	82.3
35 DAT5	5.8	15.2	32.33	84.8	2.7	11.0	22.04	89.1

In summary, after five foliar applications of about 0.2 kg ai/ha of [^{14}C]metrafenone, TRRs in mature grapes, 35 days after the last application were about 0.3–0.4 mg eq/kg, with parent being the major component (0.06 to 0.11 mg/kg) in the marc, but not found in juice. Unidentified polar

metabolites did not exceed 0.05 mg eq/kg (12–17% TRR) in mature grape pomace and in juice, CL197675 made up about 9% TRR (0.006 mg eq/kg).

The proposed metabolic pathway involves oxidation of the methyl groups on the bromophenyl and trimethoxyphenyl rings to yield the corresponding aldehydes. In the case of the bromophenyl ring, the aldehyde can undergo further oxidation to the carboxylic acid, cyclization to form the lactone, and/or dehalogenation to form the des-bromo aldehyde.

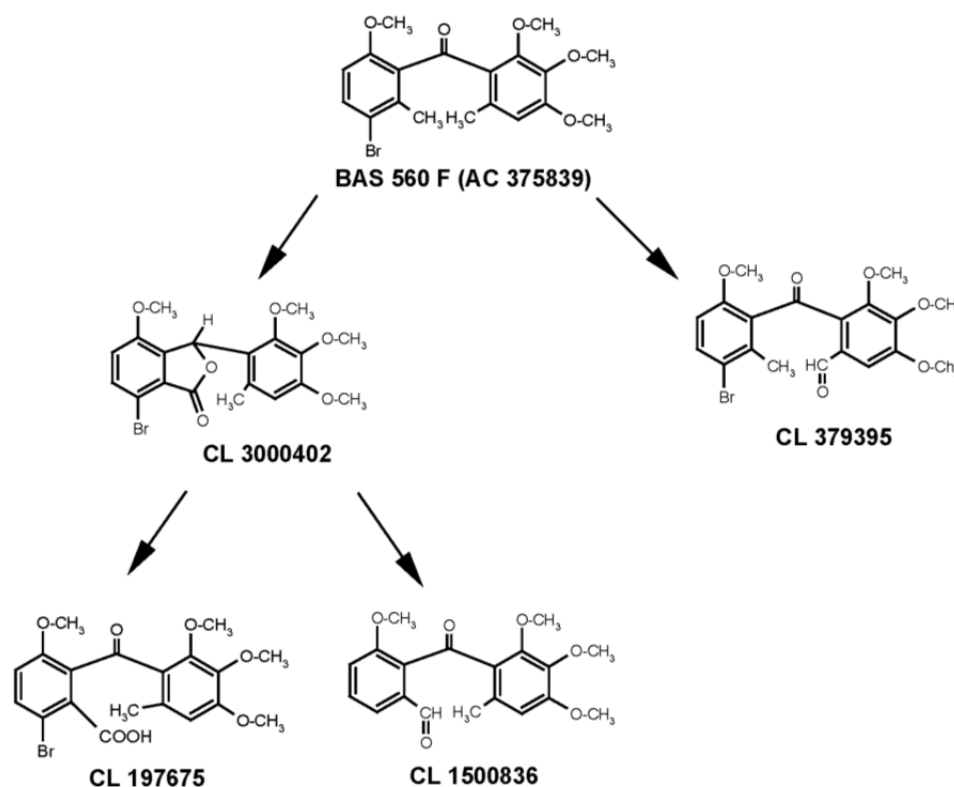


Figure 3 Metabolic pathway for metrafenone in grapevines

Cucumber

A metrafenone metabolism study in cucumber was reported by Grosshans & Ockert, 2010 [Ref: 2010/1054630]. Cucumber plants were treated with two foliar applications equivalent to 0.2 kg ai/ha (48 g ai/100L) [¹⁴C]metrafenone (trimethoxyphenyl-label), 17 and 3 days before harvest. Leaf samples were taken immediately after the first application and cucumbers were sampled just before the second application (14 DAT1). At maturity, 3 days after the second application, whole plants (without roots) were sampled to investigate residues in cucumber peel, pulp and the vines.

Samples were sequentially extracted three times with methanol and twice with water and radioactivity was measured by LSC or combustion LSC and characterisation of the radioactive residues was by HPLC. Samples were stored frozen for up to 50 days before extraction and a further 36 days before analysis.

TRRs (calculated from extracted and non-extracted radioactivity) in leaves sampled directly after the first application were 6.4 mg eq/kg. In cucumber fruit sampled at maturity, 3 days after the second application, the TRR in whole fruit was 0.05 mg eq/kg with 0.013 mg eq/kg in pulp and 0.26 mg eq/kg in the peel. The TRR in the vines (rest of the plant) was 8.8 mg eq/kg.

Methanol extraction efficiency ranged from 87.5% to 93% in fruit, 91% in peel and pulp, 98.8% in leaves (0DAT1) and 92.6% in vines (rest of plant) at harvest. Up to an additional 1.6% TRR in cucumbers (whole fruit, peel and pulp) was able to be extracted with water.

Table13 Extractability of radioactive residues in cucumber samples after two foliar applications of [¹⁴C]metrafenone

Matrix	DAT ^a	TRR ^b	Methanol extract		Aqueous extract		ERR ^c		RRR ^d	
		mg eq./kg	mg eq./kg	%TRR	mg eq./kg	%TRR	mg eq./kg	%TRR	mg eq./kg	%TRR
Leaf	0DAT1	6.397	6.319	98.78	0.020	0.31	6.339	99.09	0.058	0.91
Fruit	14DAT1	0.016	0.014	87.46	0.000	1.55	0.015	89.01	0.002	10.99
Fruit	3DAT2	0.051	0.047	92.86	0.001	1.42	0.048	94.27	0.003	5.73
Pulp	3DAT2	0.013	0.012	91.09	0.000	1.12	0.012	92.21	0.001	7.79
Peel	3DAT2	0.263	0.240	91.16	0.003	1.07	0.243	92.23	0.020	7.77
Vines ^e	3DAT2	8.807	8.151	92.56	0.282	3.20	8.433	95.76	0.374	4.24

^a DAT = Days after treatment

^b TRR calculated as the sum of ERR and RRR

^c ERR = Extractable Radioactive Residue

^d RRR = Residual Radioactive Residue

^e Vines = Whole plant without roots and fruit

Metabolite identification and characterisation in leaves (0DAT1) and fruit (14DAT1) was achieved by HPLC co-chromatography with metrafenone and peak assignment in the other samples was done by comparison of the retention times and the HPLC elution profiles with those of the extracts investigated by co-chromatography.

Metrafenone was the main residue component in leaves immediately after the first application (95% TRR) and represented 80% TRR in vines. In fruit at harvest, metrafenone was also the predominant residue, making up 42% TRR (0.022 mg/kg) in whole fruit, 61% TRR (0.16 mg/kg) in peel. In pulp, metrafenone was found at 0.0009 mg/kg or 6.5% TRR, with the majority of the residue being polar or medium polar components (23 peaks), each below 0.002 mg eq/kg.

Table 14 Residues of metrafenone and other characterised components in cucumber matrices following foliar applications of [¹⁴C]metrafenone

Matrix	DAT ^a	Metrafenone		Characterised (total) (Methanol extract)		Characterised (total) (Aqueous extract)	
		mg/kg	%TRR	mg eq./kg	%TRR	mg eq./kg	%TRR
Leaf	0DAT1	6.076	95.0	0.243 ^b	3.8	0.0197	0.3
Fruit	14DAT1	0.002	12.6	0.011 ^c	66.1	0.0003	1.6
Fruit	3DAT2	0.022	42.4	0.019 ^d	36.5	0.0007	1.4
Pulp	3DAT2	0.0009	6.5	0.011 ^e	83.7	0.0001	1.1
Peel	3DAT2	0.161	61.0	0.048 ^f	18.3	0.0028	1.1
Vines ^g	3DAT2	7.084	80.4	1.301 ^g	14.8		

^a DAT = Days after treatment

^b 4 peaks, each ≤ 2.1% TRR

^c 17 peaks, each ≤ 8.9% TRR

^d 22 peaks, each ≤ 7.3% TRR

^e 23 peaks, each ≤ 12.1% TRR, 0.0016 mg eq/kg

^f 20 peaks, each ≤ 2.9% TRR

^g 26 peaks, each ≤ 3.1% TRR

^h Vines = Whole plant without roots and fruit

In summary, after two foliar applications 0.2 kg ai/ha [¹⁴C]metrafenone to cucumber plants, TRR in mature fruit, sampled 3 days after the second application were about 0.05 mg/kg (TRR), with 0.013 mg eq/kg present in pulp and 0.26 mg eq/kg in peel. More than 89% TRR was able to be extracted with methanol. Metrafenone was the only identified residue component, making up 42% of

the TRR in mature fruit (0.02 mg/kg), mostly in the peel (61% TRR, 0.16 mg/kg). Numerous polar and medium polar metabolites at low concentrations (each less than 9% TRR) were characterized by their retention time behaviour in HPLC or by extractability with water.

Wheat

The metabolism and distribution of metrafenone in wheat was investigated in a study reported by Zulalian, 2002 [Ref: 2002/7005253]. Spring wheat plants were treated with three foliar applications of [¹⁴C]metrafenone (trimethoxyphenyl-label or bromophenyl-label, both being diluted with ¹³C as a mass marker), at nominal rates of 0.3, 0.3 and 0.2 kg ai/ha, applied at 13–14 day intervals with the last application being 35 days before harvest.

Wheat plants (without roots) were sampled immediately after each application and forage samples were also taken 3 days after the first application. Wheat plants (hay) were cut 14 days after the second application and dried for about 33 hours before being collected as samples. Straw (including chaff) and seed were collected at mature harvest, 35 days after the last application. Analysis for total radioactive residues (TRR) was conducted by combustion and liquid scintillation counting (LSC) and residues were characterized and identified by HPLC and where possible, by mass spectrometry.

Highest radioactive residues were found in the straw (ca 8 to 9 mg eq/kg), with similar levels (5–8 mg eq/kg) in forage and straw, and lower levels of 0.2 to 0.4 mg eq/kg measured in grain. Methanol:water extraction was able to release about 95% TRR in forage, 78% TRR in hay, 61% TRR in straw and 35% TRR in grain. Additional extraction with hexane and acidified methanol was able to release a further 12–14% TRR in grain.

Table 15 Extractability of radioactive residues in wheat matrices after three foliar applications of [¹⁴C]metrafenone

Radiolabel		Bromophenyl				Trimethoxyphenyl			
Matrix		Forage	Hay	Straw	Grain	Forage	Hay	Straw	Grain
DAT ^a		3DAT1	14DAT2	35DAT3	35DAT3	3DAT1	14DAT2	35DAT3	35DAT3
TRR ^b	mg eq./kg	8.17	7.78	8.91	0.21	5.27	8.5	8.25	0.4
Main Extracts									
Hexane	% mg eq./kg	NC	NC	NC	6.4 0.013	NC	NC	NC	3.5 0.014
Methanol:Water	% mg eq./kg	96.6 7.89	79.2 6.17	60.3 5.39	35.8 0.075	92.6 4.88	77.0 6.55	62.0 5.11	34.7 0.138
Methanol:2% HCl	% mg eq./kg	NC	NC	NC	8.0 0.017	NC	NC	NC	8.0 0.032
ERR ^c	% mg eq./kg	96.6 7.89	79.2 6.17	60.3 5.39	50.2 0.11	92.6 1(4.88)	77.0 6.55	62.0 5.11	46.2 0.184
RRR ^d	% mg eq./kg	3.4 0.278	20.8 1.62	39.6 3.53	49.8 0.1	7.4 0.388	23.0 1.95	38.1 3.14	53.8 0.215

^a DAT = Days after treatment; 3DAT1=3 days after the 1st treatment

^b TRR was calculated as the sum of ERR and RRR

^c ERR = Extractable Radioactive Residue

^d RRR = Residual Radioactive Residue

NC=Extraction not conducted

Metrafenone was the main component of the TRR in all matrices, making up 3–7.7% TRR in grain (0.013–0.016 mg/kg), 59–64% TRR in forage (3.1–5.3 mg/kg), 13–26% TRR in hay (1.1–2.0 mg/kg) and 7.7–14% TRR in straw (0.64–1.2 mg/kg). While TRRs in several regions of interest were each found at 10–20% TRR, these were described as metabolites more polar than the parent compound and consisted of up to five unidentified components. All other components in all matrices were present at less than 10% TRR. Significant single metabolites in forage, hay and straw were identified as the CL 3000402, CL 434223 and CL 1500831, each present at less than 7% TRR

(0.6 mg eq/kg) in these matrices. In grain, although no identified metabolites were found above 0.004 mg eq/kg, up to half the TRR was not solvent-extracted and while additional residues were released by alpha-amylase treatment of the post-extraction solids, these were not quantified. However HPLC analysis of the alpha-amylase extract showed that the radioactive residues were made up of multiple minor components.

Table 16 Characterisation and identification of radioactive residues in wheat matrices after three foliar applications of [^{14}C]metrafenone (bromophenol-label)

Matrix	Forage		Hay		Straw		Grain	
Sampling Time	3DAT1		14DAT2		35DAT3		35DAT3	
	%TRR	mg eq./kg	%TRR	mg eq./kg	%TRR	mg eq./kg	%TRR	mg eq./kg
TRR	100	8.17	100	7.78	100	8.9	100	0.21
TRR in extract (ERR)	96.6	7.889	79.2	6.168	60.3	5.387	35.8	0.075
ROI-12 (Metrafenone)	64.4	5.261	26.0	2.021	13.6	1.215	7.7	0.016
ROI-1 (Unknowns) ^a	4.3	0.355	15.0	1.165	8.3	0.740	15.2	0.045
ROI-2 (Unknowns) ^a	3.9	0.320	11.5	0.896	10.5	0.934	< 2.0	< 0.004
ROI-3A (CL 1500837) + ROI-3B (CL 1500836)	4.2	0.342	3.7	0.291	4.6	0.412	< 2.0	< 0.004
ROI-4 (CL 3000402)	4.7	0.381	4.7	0.367	2.3	0.203	< 2.0	< 0.004
ROI-5 (CL 1500838), Conjugate of ROI 8	1.1	0.091	4.2	0.328	3.2	0.289	< 2.0	< 0.004
ROI-6A (CL 1500839) + ROI-6B (1500832)	1.6	0.132	3.7	0.289	1.5	0.132	< 2.0	< 0.004
ROI-7A1 (CL 1500833) + ROI-7A2 (CL 1500834) + ROI-7A3 (CL 1500835) + ROI-7B (CL 377160) + ROI-7C (Unknown) +	2.0	0.159	3.1	0.245	4.9	0.434	< 2.0	< 0.004
ROI-8 (CL 434223)	3.0	0.243	0.8	0.065	2.5	0.219	< 2.0	< 0.004
ROI-9 (CL 376991)	0.3	0.024	0.4	0.033	0.7	0.062	< 2.0	< 0.004
ROI-10 (CL 1500831)	1.4	0.114	1.5	0.119	0.7	0.066	< 2.0	< 0.004
ROI-11 (Unknowns)	0.8	0.067	1.4	0.107	0.2	0.020	< 2.0	< 0.004
Total Identified	91.7	7.489	76	5.926	53.0	4.726	42.9	0.093
PES	3.4	0.278	20.8	1.615	39.6	3.527	49.8	0.104

^a Consisting of at least five minor unknown components

Table 17 Characterisation and identification of radioactive residues in wheat matrices after three foliar applications of [^{14}C]metrafenone (trimethoxyphenyl-label)

Matrix	Forage		Hay		Straw		Grain	
Sampling Time	3DAT1		14DAT2		35DAT3		35DAT3	
	%TRR	mg eq./kg	%TRR	mg eq./kg	%TRR	mg eq./kg	%TRR	mg eq./kg
TRR	100	5.27	100	8.5	100	8.25	100	0.4
TRR in extract (ERR)	92.6	4.877	77.0	6.545	62.0	5.109	38.2	0.152
ROI-12 (metrafenone)	58.78	3.101	12.7	1.078	7.7	0.635	3.1	0.013
ROI-1 (Unknowns) ^a	3.9	0.203	17.4	1.478	9.1	0.751	20.3	0.081
ROI-1 (Unknowns) ^a	3.1	0.163	12.5	10.61	10.6	0.876	< 2.0	< 0.004
ROI-3A (CL 1500837) + ROI-3B (CL 1500836)	4.5	0.238	4.7	0.403	5.0	0.410	< 2.0	< 0.004
ROI-4 (CL 3000402)	4.2	0.219	6.6	0.564	3.9	0.319	< 2.0	< 0.004
ROI-5 (CL 1500838), Conjugate of ROI 8	2.0	0.103	5.0	0.429	4.1	0.338	< 2.0	< 0.004

Matrix	Forage		Hay		Straw		Grain	
Sampling Time	3DAT1		14DAT2		35DAT3		35DAT3	
	%TRR	mg eq./kg	%TRR	mg eq./kg	%TRR	mg eq./kg	%TRR	mg eq./kg
ROI-6A (CL 1500839) + ROI-6B (1500832)	2.4	0.127	5.3	0.446	2.3	0.194	< 2.0	< 0.004
ROI-7A1 (CL 1500833) + ROI-7A2 (CL 1500834) + ROI-7A3 (CL 1500835) + ROI-7B (CL 377160) + ROI-7C (Unknown) +	2.6	0.136	3.2	0.275	6.5	0.540	< 2.0	< 0.004
ROI-8 (CL 434223)	3.4	0.177	1.2	0.105	2.3	0.189	< 2.0	< 0.004
ROI-9 (CL 376991)	0.2	0.011	0.4	0.033	0.2	0.016	< 2.0	< 0.004
ROI-10 (CL 1500831)	0.4	0.021	1.8	0.153	0.6	0.046	< 2.0	< 0.004
ROI-11 (unknowns)	0.9	0.046	1.7	0.147	0.3	0.023	< 2.0	< 0.004
Total Identified	86.3	4.536	72.5	6.171	52.6	4.337	43.0	0.133
PES	7.4	0.388	23.0	1.952	38.1	3.139	53.6	0.214

^a Consisting of at least five minor unknown components

In summary, after three foliar applications of [¹⁴C]metrafenone to wheat (totalling 0.8 kg ai/ha), highest radioactive residues (up to 9 mg eq/kg) were found in hay and straw, with the lowest residues found in the grain (0.2 to 0.4 mg eq/kg). The TRR in forage (3DAT1) amounted to ca. 5 to 8 mg eq/kg. Metrafenone was the major component in all matrices, comprising up to 64% TRR in forage, up to 26% TRR in hay, up to 14% in straw and up to about 8% TRR in grain. In forage, hay and straw, other characterized or identified metabolites represented less than 10% TRR. In grain, no identified metabolites were found above 0.004 mg eq/kg and although only about 50% of the radioactivity was extracted, further investigation showed that the unextracted residue was made up of multiple minor components.

The proposed metabolic pathway involves oxidative demethylation of the parent to form CL 434223, CL 376991, and CL 377160 and subsequently CL 1500835. The lactones CL 3000402 and CL 1500831 are formed by oxidation at each benzylic carbon followed by cyclization. Oxidation of the methyl groups on either of the two phenyl rings gives rise to the aldehyde derivatives, CL 1500833, CL 1500834 and CL 1500837. Aromatic oxidation and oxidative de-methylations can produce CL 1500832. Additionally, CL 1500836 is formed by oxidation at the benzylic carbon of the bromophenyl ring followed by reductive de-bromination. The oxidations are proposed to be either enzymatic and/or chemical (photolysis) in nature. Subsequent glucoside conjugation reactions yield the conjugates CL 1500838 and CL 1500839 from their corresponding aglycones, CL 434223 and CL 376991.

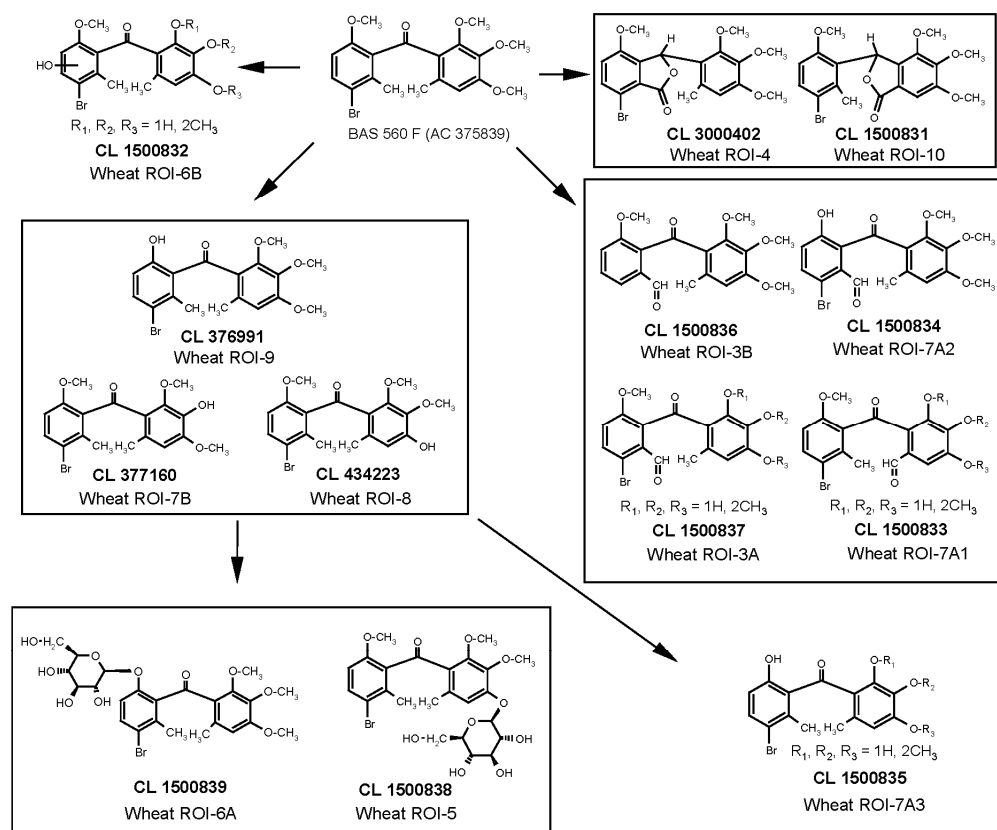


Figure 4 Metabolic pathway for metrafenone in wheat

Environmental fate

The Meeting received information the environmental fate and behaviour of metrafenone, including hydrolytic stability, photolysis in aqueous solutions, aerobic metabolism and rotational crop metabolism studies.

Hydrolysis

The hydrolysis of metrafenone was evaluated in a study reported by An, 1999 [Ref: 1999/7000284]. Sterile buffered solutions of 0.2 mg/L [bromophenyl-6-¹⁴C]-metrafenone at pH 4, 7, and 9 were incubated in the dark under sterile conditions at 50 °C, sampled after 5 days and analysed by reversed phase radio-HPLC.

There was no loss of radioactivity during the course of the incubation at any pH values with metrafenone making up 97–106% of the applied radioactivity after 5 days of incubation.

Table 18 Hydrolytic stability of [bromophenyl-6-¹⁴C]-metrafenone in sterile buffer solutions at 50 °C

pH	DAT	metrafenone	others	Total Recovery
4	0	99.7	0.1	99.8
	5	104.4	1.2	105.6
7	0	90.8	1.9	92.7
	5	97.8	2.7	100.5
9	0	96.8	0.1	96.9
	5	100.1	0.6	100.7

DAT = Days after treatment

Aerobic soil metabolism

In a study reported by Steinfuehrer, 2000 [Ref: 2000/7000152], a silty loam soil (pH 7.5, 2.03% organic C, CEC 18 meq/100 g soil and MWHC 47.3 g water/100 g dry soil) was treated with the 0.9 mg/kg dry soil [trimethoxy-label]-metrafenone or 0.98 mg/kg dry soil [bromophenyl-label]-metrafenone and incubated at 20 ± 2 °C in the dark for up to 210 days. The soil moisture was maintained at about 50% of maximum water holding capacity (MWHC). The application rates corresponded to 0.675 kg ai/ha and 0.735 kg as/ha respectively, assuming a soil mixing depth of 5 cm and a soil density of 1.5 g/cm³.

Duplicate soil samples were taken at treatment and after incubation for 4, 7, 14, 28, 60, 120, and 210 days and exhaustively extracted using acetone, methanol:water (4:1), water, and/or acetonitrile:0.5 N HCl (1:1). The various soil extracts were concentrated by rotary evaporation and analysed separately by radio-TLC (except most water and acetonitrile/HCl extracts, which were not analysed because of low radioactivity content. HPLC and LC/MS were used for metabolite identification. In addition, the non-extracted (bound) radioactivity in the day 210 soil samples was further characterized by fractionation of the extracted soil between fulvic acids, humic acids and humin. The fulvic acid fraction was partitioned into methylene chloride and analysed by TLC.

Between 92–106% of the applied radioactivity (% AR) was able to be recovered and extractable residues accounted for approximately 100% AR up to 14 days, but thereafter decreased steadily to 61% AR after 210 days for both labels. Accordingly, non-extractable (bound) residues increased to 28 and 29% AR at the end of the study. ¹⁴CO₂ reached 3.3% and 2.9% AR after 210 days, showing that both labels were eventually mineralized.

The levels of metrafenone in the extractable residues decreased to 55–57% AR at the end of the 201-day study and the metabolite fraction (< 0.2% AR) detected with TLC in the 210-day methanol/water and water extracts and was shown by HPLC analysis to consist of the substituted benzoic acids CL 197675 and CL 197676, formed by oxidation of the respective methyl group.

The characterization of bound residues in the day 210 samples showed that approximately two-thirds of the radioactivity (18.5–20% AR) was in the humin fraction, while 2–3% AR was in the humic acid fraction and 4–5% AR was in the fulvic acid fraction. Approximately half of the radioactivity in the fulvic fraction was partitioned into methylene chloride and analysed by TLC, which showed a small amount of metrafenone and a polar fraction.

Table 19 Recovery and distribution of radioactivity in soil treated with [¹⁴C]metrafenone and incubated under aerobic conditions in the dark at 20 °C

DAT	¹⁴ CO ₂	Extractable Residues (mg/eq./kg)				Bound residues	Total Recovery
		Metrafenone	CL 197675 + CL 197676	Unidentified ^a	Total		
Trimethoxyphenyl-U- ¹⁴ C							
0	n.a.	97.8	n.d.	2.6	100.4	4.7	105.1
4	< 0.1	96.8	n.d.	5.2	102.0	1.7	103.7
7	< 0.1	97.1	n.d.	4.3	101.4	2.5	103.9
14	< 0.1	93.0	n.d.	7.0	100.0	4.1	104.1
28	0.3	86.3	n.d.	8.3	94.6	7.3	102.2
60	0.7	77.5	n.d.	8.7	86.2	13.1	100.0
120	1.8	66.2	n.d.	7.3	73.5	19.4	94.7
210	3.3	54.7	0.15	5.9	60.8	27.7	91.8
Bromophenyl-6- ¹⁴ C							
0	n.a.	99.4	n.d.	2.3	101.7	4.7	106.4
4	< 0.1	99.9	n.d.	3.7	103.6	1.7	105.3
7	< 0.1	97.4	n.d.	5.2	102.6	2.6	105.2

14	< 0.1	94.6	n.d.	5.8	100.4	4.3	104.7
28	0.2	90.5	n.d.	6.6	97.1	7.4	104.7
60	0.6	81.1	n.d.	6.6	88.4	14.3	102.6
120	1.5	69.0	n.d.	4.9	73.9	22.8	98.2
210	2.9	57.4	0.15	3.7	61.3	29.2	93.4

DAT = Days after treatment

^a Unidentified radioactivity consists of radioactivity in non-analysed water and acetonitrile/HCl extracts, radioactivity remaining at TLC origin and radioactivity lost during sample work-out.

n.a. = not analysed

n.d. = not detected

In a similar study reported by Steinfuehrer, 2000 [Ref: 2000/7000151], three soils (loamy sand, sandy loam, and clay loam) were treated with [bromophenyl-6-¹⁴C]-metrafenone at a concentration of 1.48 mg/kg dry soil (corresponding to field application rates of 1.11 kg ai/ha) and incubated in the dark at 20 ± 2 °C and 40–50% MWHC. Duplicate soil samples taken at treatment time and after incubation for 3, 7, 14, 28, 58, 90, and 120 days were exhaustively extracted with acetone, methanol:water (4:1), and water using ultrasonication. The acetone and methanol:water extracts were concentrated by rotary evaporation and analysed by radio-TLC.

Total recoveries for the three soils were 94–100% of the applied radioactivity and the levels of extractable radioactivity continuously decreased over the incubation period to 67–78% AR after 120 days. Accordingly, increasing amounts of bound residues were formed in the three soils, accounting for up to 17.4–24.8% AR at the end of the 120-day study period. Mineralization was also observed with evolved ¹⁴CO₂ accounting for 2.7 to 5.3% AR after 120 days of incubation for the three soils.

Table 20 Recovery and distribution of radioactivity in three soils treated with [¹⁴C]metrafenone and incubated under aerobic conditions in the dark at 20 °C

Soil	DAT	Extractable Residues (mg eq./kg)				CO ₂	Bound Residues	Total Recovery
		Acetone	MeOH/Water	Water	Total			
Sporkenheim loamy sand pH: 6.2 Organic C: 0.63% CEC: 8 meq/100 g MWHC: 32.8%	0	93.7	5.4	0.4	99.5	n.a.	0.3	99.8
	3	82.3	14.3	0.9	97.5	< 0.1	1.2	98.7
	7	75.3	17.8	1.4	94.5	0.1	2.6	97.1
	14	85.3	5.0	1.0	91.3	0.3	4.3	95.9
	28	65.0	18.5	2.3	85.8	0.8	9.1	95.8
	58	71.1	5.0	2.3	78.4	2.2	13.9	94.5
	90	56.2	12.1	3.6	71.9	3.5	19.7	95.1
	120	50.6	12.3	3.7	66.6	5.3	24.8	96.8
Binger Pfad sandy loam pH: 7.1 Organic C: 1.02% CEC: 13 meq/100 g MWHC: 33.6%	0	92.0	6.4	0.6	99.1	n.a.	0.7	99.8
	3	82.7	12.6	1.2	96.5	< 0.1	1.9	98.4
	7	77.5	16.7	1.4	95.6	0.1	2.8	98.5
	14	84.9	4.7	0.9	90.5	0.2	3.7	94.4
	28	73.1	13.8	2.5	89.4	0.5	7.4	97.3
	58	74.3	9.2	2.3	85.8	1.2	11.0	97.9
	90	65.7	12.6	3.1	81.4	1.8	14.0	97.2
	120	62.8	11.9	2.9	77.6	3.0	17.4	98.1
Gensingen Pfad clay loam pH: 7.3 Organic C: 0.96% CEC: 18 meq/100 g	0	89.2	8.1	1.1	98.4	n.a.	1.4	99.8
	3	87.9	8.1	1.1	97.1	< 0.1	1.8	98.9
	7	85.7	9.3	1.2	96.2	< 0.1	2.8	99.0
	14	81.3	6.6	1.3	89.2	0.1	4.5	93.8
	28	80.9	9.1	1.9	91.9	0.3	8.3	100.4

Soil	DAT	Extractable Residues (mg eq./kg)				CO ₂	Bound Residues	Total Recovery
		Acetone	MeOH/Water	Water	Total			
MWHC: 40.5%	58	71.9	7.0	2.5	81.4	1.0	14.3	96.7
	90	69.6	7.6	2.7	79.9	1.5	17.5	98.9
	120	64.5	7.6	3.2	75.3	2.7	21.8	99.8

DAT = Days after treatment

n.a. = Not analysed

Metrafenone accounted for approximately 90% AR in the extracts at day 0 and slowly decreased to 56.1–68.7 % AR after 120 days. Only one minor metabolite fraction was observed at various sampling times in the three soils, this accounting for less than 0.9% AR and was not identified.

Table 21 Metrafenone residues (%AR) in soils treated with [bromophenyl-6-¹⁴C]-metrafenone and incubated under aerobic conditions in the dark at 20 °C

Time (days)	Sporkenheim	Binger Pfad	Gensingen
0	89.4	90.4	89.5
3	90.0	85.2	87.7
7	86.4	85.0	84.7
14	80.7	79.8	79.7
28	69.1	77.7	81.9
58	67.9	75.1	70.8
90	62.2	72.5	71.3
120	56.1	68.7	65.7
1 st order DT50 (r ²)	182 days (0.8824)	365 days (0.8637)	289 days (0.8705)

In a further study reported by Steinfuehrer, 2000 [Ref: 2000/7000150], a loamy sand soil (pH: 6.3, Organic C: 0.72%, CEC: 8 meq/100 g and MWHC: 34.2 g water/100 g dry soil) was treated with [bromophenyl-6-¹⁴C]-metrafenone at a concentration of 1.52 mg/kg dry soil (corresponding to field application rates of 1.14 kg ai/ha) and incubated in the dark at a lower temperature of 10 ± 2 °C and moisture content was maintained at 40–50% MWHC. Duplicate soil samples taken at treatment time and after incubation for 3, 7, 14, 28, 48, 90, and 120 days were exhaustively extracted with acetone, methanol:water (4:1), and water using ultrasonication. The acetone and methanol:water extracts were concentrated by rotary evaporation and analysed by radio-TLC.

Total recoveries were in the range of 100–102% AR and the levels of extractable radioactivity continuously decreased over the incubation period to about 91% AR after 120 days with bound residues increasing to up to 8.2% AR at the end of the 120-day study. Mineralization was also observed, with evolved ¹⁴CO₂ accounting for 1.4% AR after 120 days.

Metrafenone accounted for 93.4% AR at day 0 and slowly decreased to 82.0% AR after 120 days, with no other defined peaks or metabolite fractions observed in the TLC chromatograms at any sampling time.

Table 22 Recovery and distribution of radioactivity in soil treated with [¹⁴C]metrafenone and incubated under aerobic conditions in the dark at 10 °C

Soil	Time (days)	Extractable Residues (mg eq./kg)			CO ₂	Bound Residues	Total Recovery
		Metrafenone	Others ^a	Total			
Sporkenheim	0	93.4	6.3	99.7	n.a.	1.5	102.2
	3	91.8	7.3	99.1	< 0.1	1.3	100.4

	7	89.9	9.0	98.9	< 0.1	1.5	100.5
	14	91.4	7.6	99.0	0.1	1.5	100.5
	28	89.1	8.2	97.3	0.2	2.5	100.0
	58	87.5	8.1	95.6	0.5	4.5	100.6
	90	82.6	11.5	94.1	0.9	6.5	101.6
	120	82.0	8.6	90.6	1.4	8.2	100.2

^a Others include radioactivity lost during sample work-up, unresolved background in TLC traces and radioactivity in extracts that were not analysed by TLC. It does not consist of defined peaks.

n.a. = Not analysed

In summary, metrafenone degraded slowly in the loamy sand, sandy loam and clay loam soils incubated for up to 210 days under aerobic laboratory conditions at 10 °C and 20 °C. About 66–69% AR was still present as the parent compound at the end of the 20 °C study and about 82% AR remaining as metrafenone at the end of the 10 °C study. Calculated half-lives (1st order kinetics) ranged from 182–365 days.

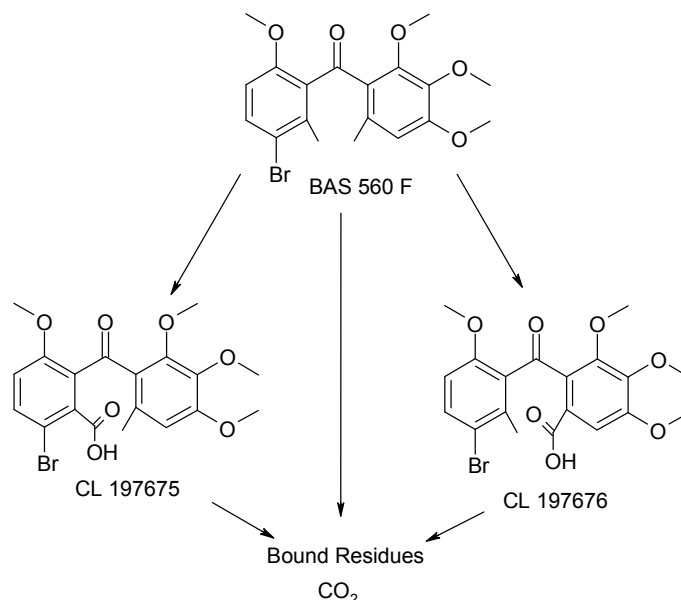


Figure 5 Proposed pathway of degradation in soil under aerobic laboratory conditions for metrafenone

Residues in rotational crops

Rotational crops metabolism

The Meeting received information on the fate of residues in lettuce, radish and canola grown as rotational crops in metrafenone-treated soil.

In an outdoor confined rotational crop study with ¹⁴C labelled metrafenone reported by Zulalian, 2002 [Ref: 2002/7005187], representative leafy vegetable crop (lettuce), root crop (radish) and oil crop (canola) were planted back at various time intervals (30, 60, 90 and 365 days) after a single application of [trimethoxy-label]-metrafenone or [bromophenyl-label]-metrafenone to bare soil at a rate equivalent to 0.625 kg ai/400 L/ha.

Harvested samples were homogenised with dry ice and the TRR determined by combustion of aliquots to yield ¹⁴CO₂, followed by quantification by liquid scintillation counting (LSC). For characterization and identification, samples were extracted with mixtures of methanol:water or hexane and methanol:HCl. The extracts were then subjected to HPLC and resolved into fractions which were

quantified by LSC. Identification was performed by co-chromatography with known reference standards. The TRR of the post extraction solid (PES) was determined either by LSC or combustion.

In general, the highest total radioactive residues (TRR) were found in plants sampled from the 30-day plant back interval, while the lowest residues (< 0.01 mg eq/kg) were detected in plants from the 365 DAT plant back interval. The canola straw/pod contained the highest residues, while very low residues were detected in canola seed at all sampling intervals. In soil, TRR declined by about 50% after 90 days, mostly found in the top 10 cm of soil samples.

Highest TRRs in the 30-day plant back samples residues were 0.048 mg eq/kg in canola straw/pods, 0.008 mg eq/kg in canola seed, 0.023–0.025 mg eq/kg in radish root and top and 0.006 mg eq/kg in lettuce. In the 365 day plant back samples, highest TRRs were < 0.004 mg eq/kg in canola straw/pods, 0.008 mg eq/kg in canola seed, 0.005–0.007 mg eq/kg in radish root and top and < 0.004 mg eq/kg in lettuce.

Table 23 Total Radioactive Residues in rotational crops planted in soil treated with [¹⁴C]metrafenone at a rate equivalent to 0.625 kg ai/ha

Crop	RAC	Bromophenyl-label TRR (mg eq./kg)				Trimethoxyphenyl-label TRR (mg eq./kg)			
		Days after treatment (DAT)				Days after treatment (DAT)			
		30	60	90	365	30	60	90	365
Lettuce		0.006	< 0.004	0.034	< 0.004	0.005	< 0.004	0.030	< 0.004
Radish	Top	0.025	0.007	0.023	0.007	0.015	0.018	0.024	0.005
	Root	0.023	0.020	0.010	0.005	0.012	0.015	0.009	0.004
Canola	Straw/Pod	0.048	0.027	0.033	0.023	0.037	0.023	0.029	0.029
	Seed	0.007	0.004	0.010	0.005	0.008	0.005	0.009	0.008
	Plant	NA	0.005	NA	NA	NA	0.006	NA	NA

NA = Not analysed

Characterisation of the radioactive residue in the rotational crop samples containing more than 0.01 mg eq/kg TRR was conducted using reversed phase HPLC/¹⁴C analysis of the solvent extracts. With the exception of canola seed, extraction with methanol:water and methanolic HCl was able to recover 64–88% TRR. Hexane and methanol:water extraction recovered 42–86% TRR in canola seed.

The HPLC results showed that the residue was comprised of unchanged metrafenone and a group of polar compounds (designated ROI 1 (Region of Interest 1)). These were not investigated further because of the low concentrations of the individual peaks. Metrafenone accounted for < 0.005 mg/kg of the TRR in lettuce and radish roots. The major portion of the extractable residues in most of the crops was shown to contain multiple components, all present at < 0.02 mg eq/kg. All other components of the extractable residues were < 0.01 mg eq/kg. The non-extractable residues were less than 0.01 mg eq/kg for all crops.

Table 24 Extraction and identification of radioactive residues in lettuce planted as a rotational crop after soil treatment with [¹⁴C]metrafenone at a rate equivalent to 0.625 kg ai/ha

Plant Back Interval	30DAT		60DAT		90DAT		365DAT	
	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg
	Bromophenol-label							
TRR	100	0.006	100	< 0.004	100	0.034	100	< 0.004
MeOH: H ₂ O	82.3	0.005	NA	NA	65.0	0.022	NA	NA
Metrafenone		< 0.001			11.08	0.004		
ROI -1(Unknown)	17.2	0.001			8.1	0.003		
ROI -2(Unknown)	ND	ND			ND	ND		
MeOH: 2% HCl	NA	NA	NA	NA	16.3	0.006	NA	NA

Plant Back Interval	30DAT		60DAT		90DAT		365DAT	
	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg
Total ERR	82.3	0.005	NA	NA	81.3	0.028	NA	NA
Total RRR	17.7	0.001	NA	NA	18.7	0.006	NA	NA
	Trimethoxyphenyl-label							
TRR	100	0.005	100	< 0.004	100	0.030	100	< 0.004
MeOH: H ₂ O	82.3	0.004	NA	NA	70.2	0.021	NA	NA
Metrafenone		< 0.001			ND	ND		
ROI -1(Unknown)	17.3	0.001			10.73	0.003		
ROI -2(Unknown)	ND	ND			11.91	0.004		
MeOH: 2% HCl	17.7	NA	NA	NA	9.5	0.003	NA	NA
Total ERR	82.3	0.004	NA	NA	79.7	0.024	NA	NA
Total RRR	17.7	0.001	NA	NA	19.6	0.006	NA	NA

ERR–Extractable Radioactive Residues

RRR–Residual Radioactive Residues

NA–Not analysed

ND–Not detected

Table 25 Extraction of radioactive residues in radish planted as a rotational crop after soil treatment with [¹⁴C]metrafenone at a rate equivalent to 0.625 kg ai/ha

Plant Back Interval	30DAT		60DAT		90DAT		365DAT	
Radish tops	Bromophenyl-label							
TRR	%	mg eq./kg	%	mg eq./kg	%	mg eq./kg	%	mg eq./kg
	100	0.025	100	0.007	100	0.023	100	0.007
MeOH: H ₂ O	68.9	0.017	70.7	0.005	85.2	0.020	NA	NA
Metrafenone			ND	ND	ND	ND		
ROI -1(Unknown)			15.3	0.001	43.7	0.01		
ROI -2(Unknown)			8.8	ND	20.0	0.005		
MeOH: 2% HCl	10.3	0.003	NA	NA	NA	NA	NA	NA
Total ERR	79.2	0.020	70.7	0.005	85.2	0.020	NA	NA
Total RRR	20.8	0.005	29.3	0.002	14.8	0.003	NA	NA
Radish tops	Trimethoxyphenyl-label							
TRR	%	mg eq./kg	%	mg eq./kg	%	mg eq./kg	%	mg eq./kg
	100	0.015	100	0.018	100	0.024	100	0.005
MeOH: H ₂ O	67.7	0.010	64.0	0.012	81.5	0.20	NA	NA
Metrafenone	ND	ND	ND	ND	ND	ND		
ROI -1(Unknown)	17.9	0.003	10.3	0.002	14.29	0.003		
ROI -2(Unknown)	ND	ND	ND	ND	ND	ND		
MeOH: 2% HCl	14.7	0.002	NA	NA	NA	NA	NA	NA
Total ERR	82.4	0.012	64.0	0.012	81.5	0.020	NA	NA
Total RRR	17.6	0.003	36.0	0.006	18.5	0.004	NA	NA
Radish roots	Bromophenyl-label							
TRR	%	mg eq./kg	%	mg eq./kg	%	mg eq./kg	%	mg eq./kg
	100	0.023	100	0.020	100	0.010	100	0.005
MeOH: H ₂ O	80.2	0.018	65.9	0.013	70.9	0.007	NA	NA
Metrafenone	16.1	0.004	9.2	0.002		< 0.001		
ROI -1(Unknown)	19.4	0.004	19.1	0.004	16.6	0.002		
ROI -2(Unknown)	ND	ND	ND	ND	ND	ND		
MeOH: 2% HCl	NA	NA	9.3	0.002	NA	NA	NA	NA
Total ERR	80.2	0.018	75.2	0.015	70.9	0.007	NA	NA

Plant Back Interval	30DAT		60DAT		90DAT		365DAT	
Total RRR	19.8	0.005	25.0	0.005	29.1	0.003	NA	NA
Radish roots	Trimethoxyphenyl-label							
TRR	%	mg eq./kg	%	mg eq./kg	%	mg eq./kg	%	mg eq./kg
	100	0.012	100	0.015	100	0.009	100	0.004
MeOH: H ₂ O	75.0	0.009	62.1	0.009	68.8	0.006	NA	NA
Metrafenone	27.7	0.003	3.9	0.001		< 0.001		
ROI -1(Unknown)	16.8	0.002	25.7	0.004	26.5	0.002		
ROI -2(Unknown)	ND	ND	12.8	0.002	ND	ND		
MeOH: 2% HCl	NA	NA	6.4	0.001	NA	NA	NA	NA
Total ERR	75.0	0.009	68.5	0.01	68.8	0.006	NA	NA
Total RRR	25.0	0.003	31.8	0.005	31.2	0.003	NA	NA

ERR–Extractable Radioactive Residues

RRR–Residual Radioactive Residues

NA–Not analysed

ND–Not detected

Table 26 Extraction of radioactive residues in canola planted as a rotational crop after soil treatment with [¹⁴C]metrafenone at a rate equivalent to 0.625 kg ai/ha

Plant Back Interval	30DAT		60DAT		90DAT		365DAT	
	%	mg eq./kg	%	mg eq./kg	%	mg eq./kg	%	mg eq./kg
Canola straw/pod	Bromophenyl-label							
TRR	100	0.048	100	0.027	100	0.033	100	0.023
MeOH: H ₂ O	76.9	0.037	74.3	0.020	64.0	0.021	66.2	0.015
Metrafenone	ND	ND	ND	ND	ND	ND	ND	ND
ROI -1(Unknown)	27.0	0.013	32.7	0.009	39.1	0.013	31.2	0.007
ROI -2(Unknown)	9.6	0.005	ND	ND	10.3	0.003	ND	ND
MeOH: 2% HCl	11.0	0.005	16.1	0.004	18.1	0.006	18.5	0.004
Total ERR	87.9	0.042	81.8	0.022	82.1	0.027	84.7	0.019
Total RRR	11.8	0.006	18.2	0.005	17.9	0.006	14.3	0.004
Canola straw/pod	Trimethoxyphenyl-label							
TRR	100	0.037	100	0.023	100	0.029	100	0.029
MeOH: H ₂ O	76.1	0.028	67.8	0.016	64.6	0.019	64.7	0.019
Metrafenone	ND	ND	ND	ND	ND	ND	ND	ND
ROI -1(Unknown)	40.0	0.015	42.2	0.01	25.1	0.007	26.5	0.008
ROI -2(Unknown)	12.4	0.005	ND	ND	ND	ND	ND	ND
MeOH: 2% HCl	11.5	0.004	12.0	0.003	19.9	0.006	5.9	0.002
Total ERR	87.6	0.035	79.8	0.019	84.5	0.025	70.6	0.021
Total RRR	12.5	0.005	20.1	0.004	15.5	0.004	29.4	0.008
Canola seed	Bromophenyl-label							
TRR	100	0.007	100	0.004	100	0.010	100	0.005
Hexane	61.5	0.004	41.9	0.002	51.6	0.005	NA	NA
MeOH: H ₂ O	<1.0	< 0.001	NA	NA	NA	NA	NA	NA
Total ERR	61.5	0.004	41.9	0.002	51.6	0.005	NA	NA
Total RRR	38.5	0.003	58.1	0.002	48.4	0.005	NA	NA
Canola seed	Trimethoxyphenyl-label							
TRR	100	0.008	100	0.005	100	0.009	100	0.008
Hexane	45.6	0.004	86.2	0.004	47.9	0.004	NA	NA
MeOH: H ₂ O	24.5	0.002	NA	NA	NA	NA	NA	NA
Total ERR	70.1	0.006	86.2	0.004	47.9	0.004	NA	NA

Total RRR	29.9	0.002	13.8	0.001	52.1	0.005	NA	NA
Canola plant	Bromophenyl-label							
TRR			100	0.005				
MeOH: H ₂ O			76.1	0.004				
MeOH: 2% HCl			NA	NA				
Total ERR			76.1	0.004				
Total RRR			23.9	0.001				
Canola plant	Trimethoxyphenyl-label							
TRR			100	0.006				
MeOH: H ₂ O			< 0.1	< 0.001				
MeOH: 2% HCl			NA	NA				
Total ERR			< 0.1	< 0.001				
Total RRR			100	0.006				

ERR–Extractable Radioactive Residues

RRR–Residual Radioactive Residues

NA–Not analysed

ND–Not detected

In summary, translocation of radiolabelled metrafenone from soils to representative rotational crops (lettuce, radish, canola) was low, with TRRs ranging from < 0.004 to 0.048 mg eq/kg (in canola pods), generally highest in the samples from the 30-day plant back interval. In soil, radioactive residues declined by about 50% after 90 days, and were mostly found in the top 10 cm of soil samples.

Total extractable residues ranged from 64.0 to 88% TRR in the majority of the samples (42–86% TRR in canola seed) and comprised mostly of multiple unidentified polar components, all present at < 0.02 mg eq/kg. Metrafenone accounted for 0.004 mg/kg of the TRR in lettuce (90DAT) and radish roots (30DAT) and was not found in canola.

METHODS OF RESIDUE ANALYSIS

Analytical methods

The meeting received analytical method descriptions and validation data for metrafenone in crop and animal commodities and in soil and water. A summary of the analytical methods for plant and animal commodities is provided below.

Table 27 Summary of metrafenone analytical methods developed for plant and animal matrices

Matrix	Analyte	Method	Principle	LOQ (mg/kg)	Reference
Wheat forage Wheat straw Wheat grain Barley forage Barley straw Barley grain Barley products	Metrafenone CL 3000402 CL 434223 CL 376991	RLA 12619.02 RLA 12619.03V (993/0)	Methanol/water extraction Dichloromethane partition SPE clean-up LC-MS/MS analysis Metrafenone m/z 409 → m/z 209 / m/z 411 → m/z 209 CL 3000402 m/z 423 → m/z 241 / m/z 425 → m/z 243 CL 434223 m/z 395 → m/z 195 / m/z 397 → m/z 195 CL 376991 m/z 395 → m/z 209 / m/z 397 → m/z 209	0.01	2001/7001048, 2001/7001770, 2002/1004080

Grape Wine Barley grain	Metrafenone	DFG S19	Aqueous acetone extraction Acetone/ethyl acetate/cyclohexane partition GPC and silica gel column clean-up GC-ECD analysis	0.01	2000/7000136
Grapes	Metrafenone	RLA 12612V (99105V)	n-heptane/acetone extraction SPE clean-up GC-ECD or GC-MS analysis	0.05	2000/7000111
Wheat forage Wheat straw Wheat grain Cucumber Lemon Beans Oilseed rape (seed) Hops (dry cones)	Metrafenone	QuEChERS 1	Acetonitrile extraction (pH 5-5.5 buffer) SPE clean-up LC-MS/MS analysis m/z 409 → m/z 209 / m/z 409 → m/z 227 m/z 409 → m/z 209 / m/z 411 → m/z 209 for dry hop cones	0.01	2011/7007816
Hops (green cones) Hops (dry cones) Beer	Metrafenone	535/3 (L0076/03)	Methanol/water/HCl extraction cyclohexane partition (alkaline) HPLC-MS/MS analysis m/z 411 → m/z 209 / m/z 411 → m/z 229	0.01	2010/1089964
Eggs Meat Milk	Metrafenone	DFG S19	Aqueous acetone extraction Ethyl acetate/cyclohexane partition GPC clean-up GC-MS analysis m/z 377 → 395 / m/z 377 → m/z 408	0.05 0.05 0.01	2001/7000486

Data collection methods

RLA 12619.02

This method for measuring residues of metrafenone and major metabolites (CL 3000402, CL 434223 and CL 376911) in cereal matrices and was described and validated by Smalley, 2001 [Ref: 2001/7001048], by Kang, 2001 [Ref: 2001/7001770] and for measuring residues of metrafenone in barley 'processing products' by Pollmann, 2002 [Ref: 2002/1004080]. Residues were extracted with methanol:water (80:20), filtered and reduced by rotary evaporation before partitioning into dichloromethane and final clean up through a strong anion exchange cartridge. Residues were measured using LC/APCI mass spectrometry (LC-MS/MS) calculating results using bracketing standards.

Average recovery rates in samples spiked with 0.01–0.1 mg/kg metrafenone ranged from 88–98% (RSD ≤ 13.3%) and the LOQ was 0.01 mg/kg for grain and barley 'processing products' and 0.1 mg/kg for forage and straw.

Table 28 Metrafenone analytical recovery rates for analytical method RLA 12619.02 and RLA 12619.03

Sample Matrix	Method	Analyte	Fortification (mg/kg)	Average recovery (%)	RSD (%)	No	Reference
Wheat whole plant	LC-MS/MS	Metrafenone	0.1 1.0	88 88	11.7 2.8	5 5	2000/7001048
		CL 3000402	0.1 1.0	78 95	12.7 2.6	5 5	
		CL 434223	0.1 1.0	75 76	18.1 4.3	5 5	
		CL 376911	0.1 1.0	82 91	10.6 4.8	5 5	
Wheat grain	LC-MS/MS	Metrafenone	0.01 0.1	95 98	13.3 3.4	5 5	2000/7001770

Sample Matrix	Method	Analyte	Fortification (mg/kg)	Average recovery (%)	RSD (%)	No	Reference
Wheat straw	LC-MS/MS	Metrafenone	0.1	86	9.1	5	2000/7001048
			1.0	91	8.2	5	
		CL 3000402	0.1	90	0.8	5	
			1.0	90	4.2	5	
		CL 434223	0.1	94	1.7	5	
			1.0	87	5.2	5	
		CL 376911	0.1	92	6.7	5	
			1.0	86	3.1	5	
Barley grain	LC-MS/MS	Metrafenone	0.01	100	-	2	2002/1004080
			0.1	100	-	2	
Malt	LC-MS/MS	Metrafenone	0.01	81	-	2	
			0.1	83	-	2	
Brewers grain	LC-MS/MS	Metrafenone	0.01	91	-	2	
			1.0	98	-	2	
Spent hops	LC-MS/MS	Metrafenone	0.01	59	-	2	
			0.1	62	-	2	
Brewer's yeast	LC-MS/MS	Metrafenone	0.01	85	-	2	
			0.1	77	-	2	
Beer	LC-MS/MS	Metrafenone	0.01	90	-	2	
			0.1	93	-	2	
Pearl barley	LC-MS/MS	Metrafenone	0.01	91	-	2	
			0.1	106	-	2	
Pearl barley abrasion	LC-MS/MS	Metrafenone	0.01	106	-	2	
			0.1	116	-	2	

MRM DFG S19 (plant matrices)

The German multi-residue method DFG S19 with modified extraction was described and reported by Hausmann & Class, 2000 [Ref: 2000/7000136] as suitable for as a data-collection method to measure residues of metrafenone in wheat and barley grain, grapes and wine, with an LOQ of 0.01 mg/kg.

Residues in barley grain, grapes, and wine were extracted with water:acetone (1:2) and partitioned into acetone/ethyl acetate/cyclohexane. Gel permeation chromatography was used to eliminate fat and macromolecules (as described in DFG clean-up method 6) with further silica gel column clean-up before GC/ECD analysis using a 5% phenyl methyl silicone column (non-polar stationary phase). Confirmatory analysis used a methyl siloxane column (non-polar stationary phase) and the 393 m/z ion was the target ion for quantitation.

Average recovery rates in samples spiked with 0.01–0.2 mg/kg metrafenone ranged from 88–128% (RSD ≤ 13%) and the LOQ was 0.01 mg/kg.

This method was independently validated in a study reported by Steinhauer & Pelz, 2001 [Ref: 2001/7001286] with average recovery rates in samples spiked with 0.02–0.2 mg/kg metrafenone (barley grain, grapes) or 0.01–0.1 mg/kg (wine) ranged from 88–114% (RSD ≤ 17%).

Table 29 Metrafenone analytical recovery rates for analytical method DFG S19 (modified extraction)

Sample Matrix	Method	Fortification (mg/kg)	Average recovery (%)	RSD (%)	No	Reference
Wheat grain	GC/ECD	0.01	95	13.3	5	2000/7000136
		0.1	98	3.4	5	
Barley Grain	GC/ECD	0.01	91	4	5	2000/7000136
		0.02	91	3	5	
		0.20	88	3	5	
		0.02	92	4.9	5	2001/7001286
		0.2	88	2.5	5	

Sample Matrix	Method	Fortification (mg/kg)	Average recovery (%)	RSD (%)	No	Reference
	GC/MS	0.01	121	NA	1	2000/7000136
		0.02	128	NA	1	
		0.20	104	NA	1	
		0.02	114	14	3	2001/7001286
Grapes	GC/ECD	0.01	93	4	5	2000/7000136
		0.02	102	4	5	
		0.20	101	7	5	
		0.02	100	3.3	5	2001/7001286
		0.2	97	10	5	
	GC/MS	0.01	92	NA	1	2000/7000136
		0.02	98	NA	1	
		0.20	91	NA	1	
		0.02	102	17	3	2001/7001286
Wine	GC/ECD	0.01	96	4	5	2000/7000136
		0.10	97	2	5	
		0.01	94	3.2	5	2001/7001286
		0.1	88	4.7	5	
	GC/MS	0.01	112	NA	1	2000/7000136
		0.10	105	NA	1	
		0.1	103	4.4	3	2001/7001286

NA = Not Applicable

RLA 12612V (grapes)

This GC-MS method was described and validated by Smalley, 2000 [Ref: 2000/7000111] as suitable for the analysis of metrafenone residues in grapes with an LOQ of 0.05 mg/kg.

Residues of metrafenone were extracted from grapes with n-heptane and acetone. Metrafenone was cleaned up further using solid phase extraction using silica cartridges. Measurement of metrafenone was carried out by gas chromatography using an electron capture detector. The specificity of the method was determined using GC-MS.

Average recovery rates in samples spiked with 0.05–1.0 mg/kg metrafenone ranged from 93–96% (RSD ≤ 2.0%) and the LOQ was 0.05 mg/kg and this method was independently validated with average recovery rates in samples spiked with 0.05–1.0 mg/kg metrafenone ranged from 79–91% (RSD ≤ 5%).

Table 30 Metrafenone analytical recovery rates for RLA 12612V analytical method

Matrix	Fortification (mg/kg)	Average recovery (%)	RSD (%)	No of tests	Reference
Grapes	0.05	96	1.7	5	2000/7000111
	1.0	93	2.0	5	
Grapes (ILV)	0.05	91	5.0		
	1.0	79	4.2		

QuEChERS (plant matrices)

The QuEChERS method (including Amendment 1) was described and reported by Meyer, 2011 [Ref: 2011/7007816] as suitable for as a data-collection method to measure residues of metrafenone in wheat forage, straw and grain, cucumber, lemon, dry bean seed, oilseed rape seed and dried hop cones, with an LOQ of 0.01 mg/kg.

Homogenised samples were extracted with acetonitrile in frozen conditions. After addition of magnesium sulphate, sodium chloride and citrate salts for buffering to pH 5–5.5, the samples were centrifuged for phase separation and an aliquot of the acetonitrile phase was cleaned-up by a

dispersive SPE on PSA (primary secondary amine sorbent). Analysis was by LC-MS/MS, monitoring two parent daughter ion transitions (MRM). The LOQ of the method was 0.01 mg/kg for each matrix.

A matrix effect was noted in the undiluted dry hop cone extracts and matrix-matched standard calibration solutions were therefore used for the evaluation of the recovery rates of these extracts fortified at 0.01 mg/kg and 0.1 mg/kg. The extracts fortified at 20 mg/kg were diluted by a factor of 100 and therefore determined against solvent standard calibration solutions.

Average recovery rates in samples spiked with 0.01–5.0 mg/kg metrafenone (0.01–20 mg/kg for hop cones) ranged from 86–111% (RSD $\leq$ 5.1%) and the LOQ was 0.01 mg/kg.

This method was independently validated in a study reported by Weber, 2011 [Ref: 2001/7007817] with average recovery rates in samples spiked with 0.01–5.0 mg/kg metrafenone (wheat forage, straw and grain, cucumber, lemon, dry bean seed and oilseed rape seed) or 0.01–20 mg/kg (dried hop cones) ranged from 82–94% (RSD $\leq$ 11%) except in oilseed rape seed where the average recovery rate was 70% (RSD $\leq$ 5.7%). In this study, extracts were shown to be stable for up to 3 days when stored in the dark at about 8 °C and up to 7 days at 3 °C.

In a number of the supervised field trials, method validation was also conducted prior to analysis the field samples, and the recovery rates in these studies are also summarized in the following table.

Table 31 Metrafenone analytical recovery rates for QuEChERS analytical method

Matrix	Fortification (mg/kg)	Average recovery (%)	RSD (%)	No of tests	Reference
Wheat forage	0.01	100	2.5	5	2011/7007816
	0.10	104	2.5	5	
	5.0	108	6.1	5	
	0.01	89	5.4	5	2001/7007817
	0.10	94	9.8	5	
	5.0	76	4.5	4	
Wheat straw	0.01	100	3.4	5	2011/7007816
	0.10	101	2.6	5	
	5.0	96	3.8	5	
	0.01	94	7.6	5	2001/7007817
	0.10	89	8.9	5	
	5.0	85	11	5	
Wheat grain	0.01	104	1.1	5	2011/7007816
	0.10	103	1.7	5	
	5.0	103	0.9	5	
	0.01	85	6.6	5	2001/7007817
	0.10	86	8.3	5	
	5.0	85	5.7	5	
Grape	0.01	89	7.2	2	2013/7001430
	0.1	80	15.9	2	
Cucumber	0.01	104	1.1	5	2011/7007816
	0.10	103	2.3	5	
	5.0	109	5.0	5	
	0.01	89	9.4	5	2001/7007817
	0.10	90	5.4	5	
	5.0	84	8.9	5	
	0.01	106	17.3	2	2012/7003736
	0.1	97	3.7	2	
Summer squash	0.01	89	17.1	3	2013/7001798
	0.1	96	4.2	3	
	1.0	99	1.5	3	
Melon (cantaloupe)	0.01	90	4.5	3	2013/7001797
	0.1	87	3.7	3	
	1.0	96	6.0	3	

Matrix	Fortification (mg/kg)	Average recovery (%)	RSD (%)	No of tests	Reference
Tomato	0.01	104	2.0	3	2013/7001658
	0.1	113	1.8	3	
	1.0	107	2.0	3	
Pepper	0.01	87	12.3	2	2013/7000577
	0.1	112	1.9	2	
	3.0	97	4.9	3	
Lemon	0.01	104	4.4	5	2011/7007816
	0.10	111	1.5	5	
	5.0	110	3.7	5	
	0.01	94	3.0	5	2001/7007817
	0.10	94	9.0	5	
	5.0	91	9.4	5	
Beans (dried seed)	0.01	86	5.7	5	2011/7007816
	0.10	91	2.8	5	
	0.01	94	3.8	5	2001/7007817
	0.10	90	5	5	
Oilseed rape (seed)	0.01	97	2.0	5	2011/7007816
	0.10	94	1.8	5	
	0.01	70	8.6	5	2001/7007817
	0.10	71	11	4	
Hops (green cones)	0.01	90	8.0	3	2013/7001795
	0.1	106	4.7	3	
	1.0	96	4.7	3	
Hops (dried cones)	0.01	99	1.8	5	2011/7007816
	0.10	97	0.9	5	
		100	1.1	5	
	0.01	85	13	5	2001/7007817
	0.10	82	13	5	
	20	84	5.5	5	
	0.01	91	7.2	3	2013/7001795
	0.1	92	6.0	3	
	1.0	112	7.7	3	
	200	100	6.7	3	

BASF Method 535/3 (L0076/03)—hops and beer

The BASF Method 535/3 was described and reported by Lehmann, 2010 [Ref: 2010/1089964] as suitable for as a data-collection method to measure residues of metrafenone in hop cones (green and dried) and in beer, with an LOQ of 0.01 mg/kg.

Metrafenone residues are extracted with a mixture of methanol water and hydrochloric acid. An aliquot of the extract is centrifuged and partitioned at alkaline conditions against cyclohexane. The final determination was performed by HPLC-MS/MS and the LOQ of the method was 0.01 mg/kg for each matrix.

Average recovery rates in samples spiked with 0.01 mg/kg and 0.1 mg/kg metrafenone ranged from 88–100% (RSD ≤ 5.2%) and the LOQ was 0.01 mg/kg.

Table 32 Metrafenone analytical recovery rates for BASF method 535/3 (L0076/3)

Matrix	Fortification (mg/kg)	Average recovery (%)	RSD (%)	No of tests
Hops (green cones)	0.01	99.6	3.9	5
	0.10	96.0	2.4	5
Hops (dried cones)	0.01	87.7	4.6	5
	0.10	92.1	4.9	5
Beer	0.01	95.9	3.6	5
	0.10	93.5	2.6	5

MRM DFG S19 (animal matrices)

The German multi-residue method DFG S19 (extended version) was described and reported by Pelz & Steinhauer, 2001 [Ref: 2001/7000486] as suitable for as a data-collection method to measure residues of metrafenone in milk, meat (muscle) and eggs, with an LOQ of 0.01 mg/kg for milk and 0.05 mg/kg for meat and eggs.

Residues were extracted from the various animal matrices with water:acetone (2:1) as described in module E.1 and partitioned into ethyl acetate:cyclohexane (1:1) and the organic extract was cleaned up using gel permeation chromatography (GPC module). Extracts were analysed for metrafenone using capillary gas chromatography with mass selective detection (module D.4.). The limit of quantitation (LOQ) was 0.01 mg/kg (ppm) for milk and 0.05 mg/kg (ppm) for meat and eggs.

Average recovery rates in samples spiked with 0.01 mg/kg and 0.1 mg/kg metrafenone ranged from 69–89% (RSD ≤ 15%) and the LOQs were 0.01 mg/kg (milk) and 0.05 mg/kg (meat and eggs).

This method was independently validated in a study reported by Class, 2001 [Ref: 2001/7001287] that used matrix-matched calibration standards. Analysis was by GC-MS (module D.4.). Average recovery rates in samples spiked with 0.01 mg/kg and 0.1 mg/kg (milk) or 0.05 mg/kg 0.5 mg/kg (muscle and eggs) ranging from 76–101% (RSD ≤ 14%).

Table 33 Metrafenone analytical recovery rates for analytical method DFG S19

Matrix	Fortification (mg/kg)	Average recovery (%)	RSD (%)	No of tests	Reference
Milk	0.01	71	2.7	5	2001/7000486
	0.1	69	2.8	5	
	0.01	101	9	5	2001/7001287
	0.1	95	14	5	
Meat (bovine muscle)	0.05	97	5.1	5	2001/7000486
	0.5	93	15	5	
	0.05	101	8	5	2001/7001287
	0.5	89	9	5	
Eggs	0.05	89	3.8	5	2001/7000486
	0.5	94	7.7	5	
	0.05	76	6	5	2001/7001287
	0.5	97	14	5	

Enforcement methods*Multi-residue method DFG S19*

The German method DFG S19 with GC-ECD or GC-MS analysis is suitable for enforcement of the MRL for metrafenone in plant commodities with a high starch content and high acid content (based on the method validation for wheat and barley grain and in grapes), with an LOQ of 0.01 mg/kg and in muscle, milk and eggs with an LOQ of 0.01 mg/kg for milk and 0.05 mg/kg for meat and eggs.

QuEChERS (plant matrices)

The QuEChERS method (including Amendment 1) is suitable for enforcement of the MRL for metrafenone in plant commodities, based on the method validation for cucumber and wheat forage (representing high water content), lemon (high acid content), wheat grain (high starch content), dry beans (high protein content) and oilseed rape (high oil content).

Analytical (concurrent) recoveries in supervised crop trials

Analytical recovery rates were measured in all the supervised crop field trials, with control samples being fortified with metrafenone at 0.01 mg/kg and at higher levels that generally reflected the range

of expected residues. In the European trials the common analytical method was Method 535/3 and the QuEChERS method was predominantly used in the North American trials. Some of the earlier European cereal trials used method RLA 12619 to measure residues of metrafenone and several metabolites.

For each study, average recoveries per fortification level generally fell within the 70–120% range, with a relative standard deviation of 20% or less. Information on the concurrent recovery rates for individual commodities are summarised in the relevant supervised crop field trial sections.

Stability of residues in stored analytical samples

Plant matrices

The Meeting received information on the stability of residues of metrafenone in various substrates with a high water content (lettuce, tomato, wheat forage and straw), a high starch content (carrot, wheat grain), a high protein content (dry peas), a high oil content (soya beans) and a high acid content (grape) stored at freezer temperatures for 24 months.

In a study by Class, 2001, [Ref: BN-326-010], samples of carrots and lettuce were fortified with 0.5 mg/kg metrafenone and stored in the dark at -18 °C. Samples were taken for extraction (n-heptane:acetone (8:2), partitioning into ethyl acetate and analysis after 0, 3, 8, 12, 18 and 24 months, with the stored control samples being freshly fortified and analysed concurrently to determine the procedural recovery efficiency. The 1-month and 3-month samples were analysed by GC-MS and the remaining samples were cleaned up using gel permeation chromatography before GC-ECD analysis. Mean procedural recovery rates over the study period were $92 \pm 13\%$ in carrots and $92 \pm 4\%$ in lettuce.

After 24 months storage the measured residues of metrafenone in stored lettuce and carrot samples were greater than 80% of the spiked levels.

Table 34 Stability of metrafenone residues in carrot and lettuce samples spiked at 0.5 mg/kg and stored at -18 °C.

Commodity	Storage interval (months)	Residues remaining ^a (mg/kg)	Residues remaining (%)	Procedural recovery ^b (%)
Carrot	0	0.48	96	99
	3	0.51	102	104
	8	0.4	80	96
	12	0.43	86	71
	18	0.44	88	93
	24	0.47	94	92
Lettuce	0	0.44	88	92
	3	0.47	94	94
	8	0.38	76	90
	12	0.41	82	89
	18	0.45	90	94
	24	0.43	86	95

^a Mean of three analyses

^b Mean of two analyses

In a study by Class, 2000, [Ref: 2000/7000144], spiked samples of grapes (fortified with 0.5 mg/kg metrafenone and wine (fortified with 0.1 mg/kg metrafenone) were stored in the dark at -18 °C. Samples were taken for extraction (n-heptane:acetone (8:2), partitioning into ethyl acetate and analysis after 0, 3, 8, 12, 18 and 24 months, with the stored control samples being freshly fortified and analysed concurrently to determine the procedural recovery efficiency. The 1-month, 3-month and 6-month samples were cleaned up and analysed using on line coupled HPLC/GC-MS and the remaining samples were cleaned up using gel permeation chromatography before GC-ECD analysis. Mean procedural recovery rates over the study period were 95% in grapes and 103% in wine.

After 18 months storage the measured residues of metrafenone in stored grape and wine samples were greater than 80% of the spiked levels.

Table 35 Stability of metrafenone residues in grape and wine samples spiked at 0.5 mg/kg and 0.1 mg/kg respectively and stored at –18 °C

Commodity (fortification)	Storage interval (months)	Residues remaining ^a (mg/kg)	Residues remaining ^a (%)	Procedural recovery ^b (%)
Grape (0.5 mg/kg)	0	0.56	112	108
	3	0.43	86	95
	6	0.46	92	101
	12	0.43	86	90
	18	0.42	83	81
Wine (0.1 mg/kg)	0	0.1	100	108
	3	0.101	101	109
	6	0.106	106	108
	12	0.09	90	85
	18	0.083	83	104

^a % fortified level, mean of three analyses

^b Recovery in freshly fortified samples, mean of two analyses

In a study by Class, 2002, [Ref: 2002/7004653], spiked samples of wheat grain and wheat straw fortified with 0.5 mg/kg metrafenone were stored in the dark at –18 °C. Samples were taken for extraction (n-heptane:acetone (8:2), partitioning into ethyl acetate and analysis after 0, 11-12, 18, 24 and 29 months (grain only), with the stored control samples being freshly fortified and analysed concurrently to determine the procedural recovery efficiency. The initial (0-month) samples were cleaned up and analysed using on line coupled HPLC/GC-MS and the remaining samples were cleaned up using gel permeation chromatography before GC-ECD analysis. Mean procedural recovery rates over the study period were 101% in wheat grain and 93% in wheat straw.

After 24 months (grain) and 29 months (straw) storage, the measured residues of metrafenone in frozen samples were greater than 97% of the spike level and in wheat straw were greater than 82% of the spike level.

Table 36 Stability of metrafenone residues in wheat grain and straw samples spiked at 0.5 mg/kg and stored at -18 °C

Commodity (fortification)	Storage interval (months)	Residues remaining ^a (mg/kg)	Residues remaining ^a (%)	Procedural recovery ^b (%)
Wheat grain (0.5 mg/kg)	0	0.5	100	99
	12	0.5	100	97
	18	0.46	92	92
	29	0.5	100	116
Wheat straw (0.5 mg/kg)	0	0.41	82	84
	11	0.38	76	93
	18	0.4	80	102
	24	0.35	70	94

^a % fortified level, mean of three analyses

^b Recovery in freshly fortified samples, mean of two analyses

In a study by Smalley, 2003, [Ref: 2003/1013928], spiked samples of wheat plants, grain and straw fortified with a combined solution of metrafenone and three metabolites (CL 3000402, CL434223 and CL 376991), each at a concentration of 1.0 mg/kg. Spiked samples were stored in the dark at –20 °C. Samples were taken for extraction and analysis using Method RLA 12619V after 1–3, 7, 15, 19, 24 and 31 months, with the stored control samples being freshly fortified and analysed concurrently to determine the procedural recovery efficiency. Metrafenone procedural recovery rates over the study period ranged from 91–107% in wheat plants, 81–97% in wheat straw and 71–90% in wheat grain.

After 31 months the measured residues of metrafenone in samples of wheat plants and straw were greater than 77% of the spike level and in wheat grain were greater than 74% of the spike level.

Table 37 Stability of residues in wheat matrices spiked at 1.0 mg/kg metrafenone and metabolites CL 3000402, CL434223 and CL 376991 and stored at -18 °C

Commodity (fortification)	Storage interval (months)	Residues remaining ^a (%)	Procedural recovery ^b (%)
Metrafenone			
Wheat plant (1.0 mg/kg)	1	102	107
	7	100	103
	15	106	107
	19	99	99
	24	96	97
	31	83	92
Wheat straw (1.0 mg/kg)	3	100	81
	7	94	97
	15	87	75
	19	92	90
	24	91	93
	31	87	90
Wheat grain (1.0 mg/kg)	2	74	80
	7	79	80
	15	90	90
	19	73	80
	24	87	82
	31	74	71
CL 3000402			
Wheat plant (1.0 mg/kg)	1	95	100
	7	105	103
	15	102	99
	19	96	97
	24	96	96
	31	84	94
Wheat straw (1.0 mg/kg)	3	97	96
	7	96	92
	15	93	83
	19	91	90
	24	94	95
	31	83	82
Wheat grain (1.0 mg/kg)	2	74	77
	7	97	83
	15	88	84
	19	80	88
	24	99	95
	31	83	81
CL434223			
Wheat plant (1.0 mg/kg)	1	93	98
	7	103	104
	15	100	101
	19	93	96
	24	93	95
	31	86	92
Wheat straw (1.0 mg/kg)	3	92	95
	7	97	92
	15	93	89
	19	90	90
	24	92	92
	31	96	89

Commodity (fortification)	Storage interval (months)	Residues remaining ^a (%)	Procedural recovery ^b (%)
Wheat grain (1.0 mg/kg)	2	75	90
	7	94	88
	15	84	80
	19	82	95
	24	99	84
	31	83	79
CL 376991			
Wheat plant (1.0 mg/kg)	1	99	99
	7	103	103
	15	101	105
	19	93	97
	24	91	98
	31	85	92
Wheat straw (1.0 mg/kg)	3	98	91
	7	99	92
	15	89	86
	19	90	91
	24	94	90
	31	93	87
Wheat grain (1.0 mg/kg)	2	77	80
	7	96	87
	15	87	86
	19	82	93
	24	92	89
	31	77	78

^a % fortified level, mean of three analyses

^b Recovery in freshly fortified samples, mean of two analyses

In a study by Lehmann & Mackenroth, 2011 [Ref: 2011/1043493], spiked samples of wheat plants, grain and straw, grapes, tomatoes, and dry pea and soya bean seed, fortified with 0.1 mg/kg metrafenone were stored in the dark at –20 °C. Samples were taken for extraction and analysis using BAF Method 535/3 after about 1, 6, 12, 15 and 24 months, with additional soya bean seed samples being analysed after about 2 and 3 month storage. Stored control samples were also freshly fortified and analysed concurrently to determine the procedural recovery efficiency. Mean procedural recovery rates over the study period were greater than 85% in all matrices.

After 24 months the measured residues of metrafenone in samples of wheat plants, grain and straw, grapes, tomato, dried peas and soya bean seeds were greater than 80% of the spike level after correction for procedural recovery.

Table 38 Stability of metrafenone residues in a range of plant matrices spiked at 0.1 mg/kg and stored at -20 °C

Commodity (fortification)	Storage interval (months)	Residues remaining ^a (mg/kg)	Residues remaining ^a (%)	Procedural recovery ^b (%)
Wheat plant (0.1 mg/kg)	0	0.09	86	83
	1	0.07	73	78
	6	0.08	83	86
	12	0.08	80	80
	24	0.085	85	99
Wheat straw (0.1 mg/kg)	0	0.09	91	89
	1	0.09	90	90
	6	0.085	84	89
	12	0.09	91	95
	24	0.08	78	78

Commodity (fortification)	Storage interval (months)	Residues remaining ^a (mg/kg)	Residues remaining ^a (%)	Procedural recovery ^b (%)
Wheat grain (0.1 mg/kg)	0	0.11	107	107
	1	0.08	78	91
	6	0.08	83	97
	12	0.085	85	102
	24	0.08	77	90
Grape (0.1 mg/kg)	0	0.09	89	88
	1	0.075	77	81
	6	0.1	97	99
	12	0.11	106	96
	24	0.085	83	84
Tomato (0.1 mg/kg)	0	0.09	89	85
	1	0.09	88	90
	6	0.1	101	101
	12	0.075	75	97
	15	0.08	76	77
	24	0.095	96	91
Pea (dried seeds)	0	0.1	104	99
	1	0.09	90	96
	6	0.095	96	104
	12	0.09	90	100
	24	0.085	83	96
Soya bean (seeds)	0	0.1	98	100
	1	0.07	72	96
	2	0.08	78	93
	3 (2)	0.09, 0.08	89, 82	106, 102
	6	0.06	60	99
	12	0.07	72	100
	24	0.1	98	103

^a % fortified level, mean of two analyses

^b Recovery in freshly fortified samples, mean of three analyses

In summary, metrafenone residues were stable in analytical samples stored frozen, i.e., -18 to -20 °C) for up to 24 months in representative substrates with a high water content (lettuce, tomato), a high starch content (carrot), a high protein content (dry peas), a high oil content (soya bean) and a high acid content (grape, wine) and in wheat grain (high starch), wheat forage and straw (high water content) residues were stable for up to 31 months. In general, residues in the stored samples were greater than 80% of the spiked levels.

USE PATTERNS

Information on GAP in 50 countries in Europe, the Americas, Asia and the Pacific was provided to the Meeting on the use of metrafenone, available as SC formulations, often co-formulated with either epoxiconazole and/or fenpropimorph. Proposed uses were also provided but are not reported.

The following table summarises the representative critical national or regional GAPs for the crops for which supporting residue trials have been provided.

Table 39 Registered uses of metrafenone (300 g ai/L or 500 g ai/L SC formulations)

Crop	Country	Application				Max/season		PHI (days)	Comments
		kg ai/ha	kg ai/hL	water L/ha	RTI (days)	no	kg ai/ha		
Berries and other small fruit (004)									
Grapes									
	Australia		0.01		7–10	4		35	
	Austria	0.04–0.16		100–800	10–14	3		28	Higher rate/ha from BBCH 75
	Germany								
	Bulgaria	0.01	0.01	200–1000	12–14	2		28	
	Canada	0.225			14–21	6	1.35	14	

Crop	Country	Application				Max/season		PHI (days)	Comments
		kg ai/ha	kg ai/hL	water L/ha	RTI (days)	no	kg ai/ha		
	Chile	0.1–0.15	0.075–0.01		15	3		7	
	Czech Republic	0.08–0.16		300–1000	10–14	2			Higher rate/ha from BBCH 61
	France	0.1			10–12	2		28	
	Greece	0.1	0.01	1000	10–14	3		28	
	Hungary	0.05–0.125		600–1000	10–14	4		28	Wine grapes
	Italy	0.1–0.125	0.01–0.0125		8–12	3		28	
	Luxembourg	0.1			10–14	2		28	
	Macedonia	0.1	0.01	600–1000	10–14	3		28	
	Mexico	0.1			7	3		7	
	Portugal	0.1	0.01	600–1000	10–14	3		28	
	Peru	0.125–0.15						28	
	Serbia		0.01	600–1000		3		28	
	Slovakia	0.1		1000	10–14	3		28	
	Slovenia	0.08–0.1			10–12			28	Higher rate/ha from BBCH 79
	South Africa		0.0125		10–14	3		28 (table) 56 (wine)	To BBCH 75
	Spain	0.1	0.01	600–1000	14	3		28	
	Switzerland	0.08–0.16	0.01		10–14	3			To veraison
	Turkey		0.01		10–14			28	
	Ukraine	0.1		500–1000	7–14	3		50	
Strawberries									
	Netherlands	0.15			7	2		3	Indoor crops
	Korea		0.015		7	2		NS	
Fruiting vegetables, Cucurbits (011)									
Cucurbits									
	Australia	0.075–0.15		250–500	7–10	4		7	
Cucumber									
	Bulgaria	0.1	0.01	200–1000	12–14	2		3	Field crops
	France	0.1			7–10	2		3	
	Greece	0.1	0.01	1000	7–10	2		3	
	Korea		0.015		10	2		NS	
Cucumber (indoor)									
	Belarus	0.09		1000	7–10	3		3	
	Bulgaria	0.1	0.01	200–1000	7–10	2		3	
	France	0.1				2		3	
	Turkey		0.01		7–10	2		1	
Melon									
	France	0.1			7–10	2		3	
	Greece	0.1	0.01	1000	7–10	2		3	
	Korea		0.015		10			NS	Also watermelon
Summer squash									
	France	0.1			7–10	2		3	
	Greece	0.1	0.01	1000	7–10	2		3	
Pumpkin									
	Korea		0.015		10			NS	
	New Zealand	0.15			14–21	2		14	
Winter squash									
	New Zealand	0.15			14–21	2		14	
Fruiting Vegetables, other than Cucurbits (012)									
Tomato									
	Bulgaria	0.15	0.015	200–1000	12–14 (F) 7–10 (P)	2		3	F=field crops P=protected crops
	Ecuador		0.01					7	
	France	0.15 (F) 0.225 (P)	0.015		7–10	2		3	F=field crops P=protected crops
	Greece	0.15	0.015		7–10	2		3	
	Spain		0.015			2		3	

Crop	Country	Application				Max/season		PHI (days)	Comments
		kg ai/ha	kg ai/hL	water L/ha	RTI (days)	no	kg ai/ha		
Eggplant									
	France	0.15			7–10	2	3		
Eggplant (indoor)									
	Bulgaria	0.15	0.015	200–1000	7–10	2		3	
	Greece	0.15	0.015		7–10	2		3	
Peppers									
	Korea		0.015		10	2		NS	
Peppers (indoor)									
	Bulgaria	0.15	0.015	200–1000	7–10	2		3	
	France	0.15			7–10	2		3	
	Greece	0.15	0.015		7–10	2		3	
Mushrooms									
	France	0.05 kg ai/100m ²		15 L/100m ²		1		10	
Cereal grains (020)									
Barley, oats, rye, triticale, wheat									
	Denmark	0.075–0.15				2			To BBCH 59
	Estonia	0.15			21	2		35	To BBCH 69
	France	0.15				1		35	
	Hungary	0.12			21	2		35	
	Ireland	0.15				2			To BBCH 61
	Latvia	0.15		300–400		2		35	To BBCH 69
	Sweden	0.075–0.15		200–400		2			To BBCH 61
Barley, rye, triticale, wheat									
	Belgium	0.15		200–400	21	2			To BBCH 59
	Czech Republic	0.15				2		35	To BBCH 61
	Germany	0.15		200–400		2			To BBCH 61
	Belarus	0.09				1		71	
Barley, triticale, wheat									
	Netherlands	0.15				2		35	To BBCH 69
	Poland	0.15		300	21	2		35	
Rye, triticale, wheat									
	Switzerland	0.15		300–400		1			To BBCH 61
	UK	0.15		100–400		2			To BBCH 61
Barley, oats									
	Switzerland	0.15		300–400		1			To BBCH 51
	UK	0.15		100–400		2			To BBCH 59

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials involving foliar treatments of metrafenone to the following crops.

Group	Crop	Countries	Table no
Berries and other small fruits	Grape	USA	40–41
	Strawberry	Europe	42
Fruiting vegetables, Cucurbits	Cucumber	Europe, Nth America	43–45
	Summer squash	Europe, Nth America	46–47
	Melon	Europe, Nth America	48–49
Fruiting vegetables, other than Cucurbits	Mushrooms	Europe	50
	Peppers	Europe, Nth America	51–52
	Tomato	Europe, Nth America	53–55
Cereals	Wheat	Europe	56
	Barley	Europe	57

Group	Crop	Countries	Table no
Cereal forage and fodders	Wheat	Europe	58 and 60
	Barley	Europe	59 and 61

The supervised trials were well documented with laboratory and field reports. Laboratory reports included method validation including procedural recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables unless residues in control samples exceeded the LOQ. In such cases, the residues found are noted as “c=nn mg/kg” in the Reference and Comments columns. Residue data are recorded unadjusted for recovery.

Results from replicated field plots are presented as individual values. When residues were not detected they are shown as ND. Residues and application rates have been reported as provided in the study reports, although the results from trials used for the estimation of maximum residue levels (underlined> have been rounded to two significant digits (or if close to the LOQ, rounded to one significant digit) in the Appraisal.

In some trials, samples were taken just before the final application and then, again on the same day after the spray had dried. In the data tables the notation for these two sampling times is '-0' and '0' respectively.

When multiple applications were made to a crop, the application rate, spray concentration and spray volume were not always identical from one application to the next. In most trials, the actual treatment rates were within 10% of the listed ‘target’ application rates, but if not, the actual treatment rates are listed.

Berries and other small fruits

Grape

Results from supervised trials from the USA on grapes conducted in 2005 and in 2011 were provided to the Meeting. In the 2011 trials, three foliar airblast applications of metrafenone (0.33 kg ai/ha, SC formulation) with added non-ionic surfactant were applied to 12–24 vine plots, 14–15 days apart, using about 1000–1500 L water/ha. Grape samples (min 1 kg and at least 12 bunches or part bunches) were frozen within 3 hours of sampling and stored frozen for up to 20 months before analysis of berries for metrafenone using the QuEChERS method. Procedural recovery rates in grapes fortified at 0.01 to 1.5 mg/kg ranged from 74 to 104% (mean $91 \pm 10\%$, n=10) and the LOQ was 0.01 mg/kg.

Table 40 Residues in grapes from supervised trials in the USA involving three foliar applications of metrafenone (SC formulation).

GRAPE Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	mean	
USA, 2011 Alton, NY (Cayuga White)	3	0.336		935	berries	0DAA1	0.79, 0.84	0.81	2013/7001430 R110152
		0.337		935		0DAA2	0.004 ^a , 0.007 ^b	< 0.01	
		0.337		935		0	0.92, 0.99	0.95	
						14	0.46, 0.36	0.41	
						15	0.5, 0.41	0.46	
						17	0.29, 0.43	0.36	
						19	0.3, 0.45	0.38	
						21	0.27, 0.29	0.28	
USA, 2011 Dundee, NY (Vidal)	3	0.337		945	berries	14	1.1 ^c , 0.94 ^d	1.0	2013/7001430 R110153
		0.341		945					
		0.337		945					

GRAPE Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	mean	
USA, 2011 Templeton, CA (Marsanne)	3	0.336 0.337 0.335		1496 1422 1468	berries	14	0.28, 0.42 (c=0.015)	0.35	2013/7001430 R110154
USA, 2011 Kingsburg, CA (Crimson)	3	0.337 0.337 0.339		1366 1347 1422	berries	13	0.51, 0.45	0.48	2013/7001430 R110155
USA, 2011 Lindsay, CA (Red Globe)	3	0.336 0.337 0.337		1366 1328 1347	berries	0 DAA1	0.47, 0.39	0.43	2013/7001430 R110156
						0 DAA2	0.77, 0.81	0.79	
						0	0.54, 0.63	0.59	
						14	0.38, 0.42	0.4	
						15	0.34, 0.41	0.38	
						17	0.58, 0.35	0.47	
						19	0.42, 0.44	0.43	
						21	0.35, 0.39	0.37	
USA, 2011 Dinuba, CA (Ruby Red)	3	0.337 0.337 0.339		1347 1366 1375	berries	13	0.27, 0.4	0.34	2013/7001430 R110157
USA, 2011 Porterville, CA (Thompson Seedless)	3	0.337 0.339 0.34		1394 1375 1337	berries	14	0.25, 0.2	0.22	2013/7001430 R110158
USA, 2011 Ephrata, WA (White Riesling)	3	0.331 0.333 0.333		954 954 954	berries	14	0.4, 0.49	0.45	2013/7001430 R110159

^a Mean of values 0.005, 0.004, 0.003

^b Mean of values 0.006, 0.008, 0.006

^c Mean of 1.2, 0.91, 1.1

^d Mean of 1.1, 0.84, 0.88

In the 2005 trials, six foliar applications of metrafenone (0.33 kg ai/ha, SC formulation) with no added surfactant were applied 12–15 days apart, with dilute (100–1500 L water/ha) and concentrate (500–700 L water/ha) treatments being applied to separate 40–220 square metre plots.

Grape samples (min 1 kg and at least 12 bunches or part bunches) were frozen within 3 hours of sampling, and stored frozen for up to 6 months before analysis of berries for metrafenone using Method 535/3. Average procedural recoveries of metrafenone from grapes fortified with 0.01–20 mg/kg ranged from 82–127% (mean $99 \pm 13\%$, n=26) and the LOQ was 0.01 mg/kg.

Table 41 Residues in grapes from supervised trials in the USA involving six foliar applications of metrafenone (SC formulation).

GRAPE Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	mean	
GAP:Canada	6	0.225				PHI: 14	RTI: 14-21d		1.35 kg ai/ha/season
USA, 2005 Seneca, NY (Catawba)	6	0.33	0.047	700	berries	0 14 28		1.0 <u>3.2</u> 0.64	2006/70070 RCN R05009
	6	0.33	0.024	1440	berries	0 14 28		1.8 1.9 1.1	2006/70070 RCN R05009
USA, 2005 Yates, NY (Vidal Blanc)	6	0.33	0.048	690	berries	0 14 28		1.8 <u>1.5</u> 1.4	2006/70070 RCN R05010

GRAPE Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	mean	
	6	0.33	0.024	1440	berries	0 14 28		1.5 1.3 1.0	2006/70070 RCN R05010
USA, 2005 Kern, CA (Ruby Seedless)	6	0.33	0.064	510	berries	0 14 28		0.75 <u>0.62</u> 0.52	2006/70070 RCN R05011
	6	0.34	0.026	1300	berries	0 14 28		0.67 0.55 0.52	2006/70070 RCN R05011
USA, 2005 Tulare, CA (Thompson)	6	0.34	0.065	520	berries	0 7 14 21 28		0.69 0.4 0.2 0.15 0.17	2006/70070 RCN R05012
	6	0.33	0.026	1290	berries	0 7 14 21 28		0.64 0.51 <u>0.32</u> 0.26 0.24	2006/70070 RCN R05012
USA, 2005 Tulare, CA (Thompson)	6	0.35	0.067	530	berries	0 14 28		0.4 <u>0.27</u> 0.16	2006/70070 RCN R05013 (Processing study)
	6	0.34	0.027	1270	berries	0 14 28	0.27, 0.31	0.28 0.17 0.16	2006/70070 RCN R05013 (Processing study)
USA, 2005 Glenn, CA (Centurion)	6	0.34	0.048	700	berries	0 14 28		0.22 <u>0.17</u> 0.07	2006/70070 RCN R05014
	4+	0.34 0.34	0.024 0.032	1450 1060	berries	0 14 28		0.13 0.04 0.02	2006/70070 RCN R05014
USA, 2005 Colusa, CA (Zinfandel)	6	0.33	0.047	700	berries	0 14 28		0.15 0.05 0.08	2006/70070 RCN R05015
	6	0.33	0.031	1050	berries	0 14 28		0.11 <u>0.18</u> 0.13	2006/70070 RCN R05015
USA, 2005 Sacramento, CA (Merlot)	6	0.33	0.047	700	berries	0 14 28		0.04 0.02 0.02	2006/70070 RCN R05016
	6	0.33	0.031	1050	berries	0 14 28		0.11 <u>0.11</u> 0.08	2006/70070 RCN R05016
USA, 2005 Madera, CA (Merlot)	6	0.34	0.048	710	berries	0 14 28		2.6 <u>2.1</u> 1.8	2006/70070 RCN R05017
	6	0.34	0.024	1400	berries	0 14 28		3.3 1.9 2.1	2006/70070 RCN R05017
USA, 2005 Fresno, CA (Not specified)	6	0.34	0.048	710	berries	0 14 28		2.5 <u>2.4</u> 2.0	2006/70070 RCN R05018
	6	0.34	0.024	1400	berries	0 14 28		2.8 2.2 2.3	2006/70070 RCN R05018

GRAPE Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	mean	
USA, 2005 Grant, WA (White Riesling)	6	0.34	0.048	710	berries	0 14		4.0 2.3	2006/70070 RCN R05019
	6	0.34	0.024	1400	berries	0 14		5.6 <u>3.0</u>	2006/70070 RCN R05019
USA, 2005 Benton, OR (Pinot Noir)	6	0.35	0.026	1320	berries	0 14 28		4.1 <u>2.3</u> 1.9	2006/70070 RCN R05020
	6	0.33	0.05	690	berries	0 14 28		1.4 1.3 0.6	2006/70070 RCN R05020 Dilute Spray

Strawberry

Results from supervised trials from Europe on protected strawberries conducted in 2009 were provided to the Meeting. In these trials, two foliar applications of 0.15 kg ai/ha metrafenone (SC formulation) in about 200 L water/ha were applied 7 days apart, using motorized knapsacks or 6-nozzle mini-boom sprayers.

Fruit samples (min 1 kg) were frozen within 12 hours of sampling and stored frozen (−18 °C) for up 15 months before analysis for metrafenone using Method 535/3. Average procedural recoveries of metrafenone from strawberries fortified with 0.01 or 1.0 mg/kg were 101% and 98% respectively and the LOQ was 0.01 mg/kg.

Table 42 Residues in protected strawberries from supervised trials in Europe involving two foliar applications of metrafenone (SC formulation).

STRAWBERRY Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	
GAP: Netherlands	2	0.15				3	RTI: 7–10days	
France, 2009 Loiret (Charlotte)	2	0.15	0.08	200	fruit	0 1 3 7	0.16 0.36 <u>0.34</u> 0.18	2010/1201582 S09 00766-01
Germany, 2009 Lower Saxony (Elsanta)	2	0.15	0.08	200	fruit	0 1 4 6	0.23 0.3 <u>0.28</u> 0.25	2010/1201582 S09 00766-02
United Kingdom, 2009 Nottingham (Elsanta)	2	0.15	0.08	200	fruit	0 1 3 7	0.07 0.11 <u>0.08</u> 0.07	2010/1201582 S09 00766-03
Belgium, 2009 Maasmechelen (Florin)	2	0.15	0.08	200	fruit	0 1 2 7	0.15 0.1 <u>0.1</u> 0.1	2010/1201582 S09 00766-04
France, 2009 Tarn-et-Garonne (Guarioutte)	2	0.15	0.08	200	fruit	0 1 3 7	0.06 0.07 <u>0.06</u> 0.04	2010/1201582 S09 00766-05

STRAWBERRY Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	
GAP: Netherlands	2	0.15				3	RTI: 7–10days	
Italy, 2009 Bologna (Honey)	2	0.15	0.08	200	fruit	0 1 4 7	0.1 0.13 <u>0.05</u> 0.05	2010/1201582 S09 00766-06
Greece, 2009 Pieria (Karmoroza)	2	0.15	0.08	200	fruit	0 1 2 7	0.13 0.19 <u>0.16</u> 0.11	2010/1201582 S09 00766-07
Spain, 2009 Lucena Del Puerto (Candongra)	2	0.15	0.08	200	fruit	0 1 3 6	0.34 0.32 <u>0.23</u> 0.12	2010/1201582 S09 00766-08

Fruiting vegetables, Cucurbits

Results from supervised trials from Europe and the USA on cucumbers, zucchini (summer squash) and melons (cantaloupes) were provided to the Meeting.

Cucumber

In the European outdoor trials, two foliar applications of 0.1 kg ai/ha metrafenone (SC formulation) in about 300–1000 L water/ha were applied 7 days apart, using motorized knapsacks or 4–8 nozzle mini-boom sprayers. Plot sizes were larger than 30 square metres. In the European indoor cucumber trials, two foliar applications of 0.15 kg ai/ha were applied at 7-day intervals using motorized knapsacks or vertical boom sprayers to apply about 1500 L spray mix/ha to plots of at least 18 square metres.

Fruit samples (min 1 kg) fruit (without stems) were frozen within 12 hours of sampling and stored frozen (-18 °C) for up to 16 months before analysis for metrafenone using Method 535/3. Average procedural recoveries of metrafenone from samples fortified with 0.01 mg/kg and 1.0 or 2.0 mg/kg ranged from 90% to 101% and the LOQ was 0.01 mg/kg.

In the North American trials, three foliar applications of 0.34 kg ai/ha metrafenone (SC formulation) with added adjuvant were applied at 6–8 day intervals, using motorized knapsacks or tractor-mounted 4–9 nozzle sprayers to apply about 300–700 L/ha. Plot sizes were larger than 33 square metres.

Duplicate fruit samples (min 2 kg, 12 units) were taken, with the larger cucumber fruit being sub-sampled in the field, frozen within 12 hours of sampling and stored frozen for up to 24 months before analysis for metrafenone using the QuEChERS LC-MS/MS method. The average procedural recovery of metrafenone from samples fortified with 0.01 mg/kg or 1.0 mg/kg was 104% and the LOQ was 0.01 mg/kg.

Table 43 Residues in outdoor cucumbers from supervised trials in Europe involving two foliar applications of metrafenone (SC formulation)

CUCUMBER Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	
GAP:France	2	0.1				3	RTI 7-10d	
France (S), 2009 Montauban (Ginial)	2	0.1	0.01	1000	fruit	0 1 3 7	0.04 0.04 <u>0.02</u> 0.01	2010/1033967 BSF 0729-05

CUCUMBER Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone		
France (S), 2010 Montauban (Raider)	2	0.1	0.033	300	fruit	0 1 3 7	0.08 0.05 <u>0.03</u> < 0.01		2011/1041880 S10-00479-05
Germany, 2009 Nierswalde (Rhinsk Drue)	2	0.1	0.033	300	fruit	0 1 3 7	0.03 0.03 <u>0.02</u> 0.01		2010/1033967 BSF 0729-01
Germany, 2010 Baden- Wurttemberg (Travilo)	2	0.1	0.033	300	fruit	0 1 3 8	0.02 0.01 <u>0.01</u> < 0.01		2011/1041880 S10-00479-02 Knapsack, mid Aug
Germany, 2010 Baden- Wurttemberg (Travilo)	2	0.11	0.033	333	fruit	0 1 2 6	0.03 0.02 <u>0.04</u> 0.01		2011/1041880 S10-00479-09 Boom sprayer, late Aug-Sep
Italy, 2009 Verona (Caman)	2	0.1	0.01	1000	fruit	0 1 3 7	0.07 0.04 <u>0.02</u> < 0.01		2010/1033967 BSF 0729-06
Italy, 2010 Fondi (Caman)	2	0.1	0.033	300	fruit	0 1 3 7	< 0.01 0.03 <u>0.02</u> 0.02		2011/1041880 S10-00479-06
Netherlands, 2009 Ven-Zelderheide (Rhinsk Drue)	2	0.1	0.033	300	fruit	0 1 3 7	0.03 0.02 <u>0.02</u> < 0.01		2010/1033967 BSF 0729-02

Table 44 Residues in outdoor cucumbers from supervised trials in North America involving three foliar applications of metrafenone (SC formulation)

CUCUMBER Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	mean	
USA, 2010 Jeffersonville, GA (Speedway)	3	0.33	0.11	280	fruit	0 1 3 7 10	0.11, 0.09 0.12, 0.05 0.06, 0.04 0.02, 0.02 0.016, < 0.01	0.1 0.09 0.05 0.02 0.01	2012/7003736 R100008
USA, 2010 Chula, GA (Thunder)	3	0.34	0.12	280	fruit	0	0.13, 0.15	0.14	2012/7003736 R100009
USA, 2010 Zellwood, FL (Expedition)	3	0.35	0.12	290	fruit	0	0.09, 0.07	0.08	2012/7003736 R100010
USA, 2010 Richland, IA (Pickle)	3	0.34	0.12	290	fruit	0	0.15, 0.17	0.16	2012/7003736 R100011
USA, 2010 Campbell, MN (Speedway)	3	0.34	0.12	280	fruit	0	0.05, 0.06	0.05	2012/7003736 R100012
USA, 2010 Hinton, OK (Thunder)	3	0.34	0.12	280	fruit	0	0.11, 0.08	0.1	2012/7003736 R100013

Table 45 Residues in indoor cucumbers from supervised trials in Europe involving two foliar applications of metrafenone (SC formulation)

CUCUMBER Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	
GAP: Turkey	2		0.01			1	RTI: 7–10d	
Belgium, 2009 Villers-Perwin (Pepinova)	2	0.15	0.01	1500	fruit	0	0.02	2010/1033969 BSF 0728-03
						1	<u>0.02</u>	
						3	0.02	
						7	0.01	
France (N), 2009 St Genough (Aramon)	2	0.15	0.01	1500	fruit	0	0.05	2010/1033969 BSF 0728-09 Early August treatments
						1	<u>0.06</u>	
						3	0.03	
						7	0.02	
France (N), 2009 St Genough (Aramon)	2	0.15	0.01	1500	fruit	0	0.06	2010/1033969 BSF 0728-04 Late August treatments
						1	<u>0.06</u>	
						3	0.03	
						7	0.03	
France (S), 2009 Noves (Columbia)	2	0.15	0.01	1500	fruit	0	0.06	2010/1033969 BSF 0728-05
						1	<u>0.04</u>	
						3	0.03	
						7	0.02	
Germany, 2009 Straelen (Proloog)	2	0.15	0.01	1500	fruit	0	0.07	2010/1033969 BSF 0728-01
						1	<u>0.07</u>	
						3	0.06	
						7	0.05	
Greece, 2009 Nea Magnisia (Galileo)	2	0.15	0.01	1500	fruit	0	0.1	2010/1033969 BSF 0728-08
						1	<u>0.09</u>	
						3	0.06	
						7	0.022	
Italy, 2009 Verona (Caman)	2	0.15	0.01	1500	fruit	0	0.08	2010/1033969 BSF 0728-06
						1	<u>0.05</u>	
						3	0.04	
						7	0.02	
Netherlands, 2009 AN Oirlo (Anastasia)	2	0.15	0.01	1500	fruit	0	0.07	2010/1033969 BSF 0728-02
						1	<u>0.05</u>	
						3	0.03	
						7	0.01	
Spain, 2009 Valencia (Dasher)	2	0.15	0.01	1500	fruit	0	0.03	2010/1033969 BSF 0728-07
						1	<u>0.04</u>	
						3	0.03	
						7	0.03	

Summer squash

In the European outdoor trials, two foliar applications of 0.1 kg ai/ha metrafenone (SC formulation) in about 300–1000 L water/ha were applied 7 days apart, using motorized knapsacks or 4–8 nozzle mini-boom sprayers. Plot sizes were larger than 30 square metres.

Fruit samples (min 1 kg) fruit (without stems) were frozen within 12 hours of sampling and stored frozen (-18 °C) for up 14 months before analysis for metrafenone using Method 535/3. The average procedural recovery of metrafenone from samples fortified with 0.01 or 1.0 mg/kg was 96% and the LOQ was 0.01 mg/kg.

In the North American trials, three foliar applications of 0.34 kg ai/ha metrafenone (SC formulation) with added adjuvant were applied at 6–8 day intervals, using motorized knapsacks or

tractor-mounted 4–9 nozzle sprayers to apply about 300–700 L/ha. Plot sizes were larger than 33 square metres.

Duplicate fruit samples (min 2 kg, 12 units) were frozen within 12 hours of sampling and stored frozen for up to 28 months before analysis for metrafenone using the QuEChERS LC-MS/MS method. The average procedural recovery of metrafenone from samples fortified with 0.01 mg/kg or 1.0 mg/kg as 100% and the LOQ was 0.01 mg/kg.

Table 46 Residues in outdoor summer squash (zucchini) from supervised trials in Europe involving two foliar applications of metrafenone (SC formulation)

SUMMER SQUASH Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	
GAP: France	2	0.1				3	RTI 7–10d	
Belgium, 2009 Villers-Perwin (Black Beauty)	2	0.1	0.033	300	fruit	0 1 3 7	0.02 0.01 <u>0.01</u> < 0.01	2010/1033967 BSF 0729-03
France (N), 2009 La Poitevine (Quirinal)	2	0.1	0.033	300	fruit	0 1 3 7	0.09 0.05 <u>0.02</u> < 0.01	2010/1033967 BSF 0729-04
Greece, 2009 Nea Magnisia (Ezra)	2	0.1	0.01	1000	fruit	0 1 3 7	0.06 0.05 <u>0.01</u> 0.01	2010/1033967 BSF 0729-07
Spain, 2009 San Antonio de Benageber (Consul)	2	0.1	0.01	1000	fruit	0 1 3 7	0.03 0.04 <u>0.02</u> < 0.01	2010/1033967 BSF 0729-08
France (N), 2010 Essone (Tosca)	2	0.1	0.033	300	fruit	0 1 3 7	0.05 0.04 <u>0.02</u> < 0.01	2011/1041880 S10-00479-03
France (N), 2010 Loiret (Ambassador)	2	0.1	0.033	300	fruit	0 1 3 7	0.04 0.02 <u>0.01</u> < 0.01	2011/1041880 S10-00479-04
Greece, 2010 Thessalonika (Demeter F1)	2	0.1	0.033	300	fruit	0 1 3 6	0.17 0.12 <u>0.04</u> < 0.01	2011/1041880 S10-00479-07
Spain, 2010 Xativa (Nieves)	2	0.11	0.033	330	fruit	0 1 4 7	0.04 0.11 <u>0.01</u> < 0.01	2011/1041880 S10-00479-08

Table 47 Residues in outdoor summer squash from supervised trials in North America involving three foliar applications of metrafenone (SC formulation)

SUMMER SQUASH Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	mean	
USA, 2010 Maricopa, AZ (Sunray Hybrid)	1+ 2	0.35 0.35	0.12 0.08	290 440	Fruit	0	0.25,0.36	0.31	2013/7001798 10478.10-AZ06

SUMMER SQUASH Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	mean	
Canada, 2010 Agassiz, BC (Golden Dawn III)	3	0.34	0.09	380	Fruit	0	0.24, 0.35	0.29	2013/7001798 10478.10-BC09
USA, 2010 Holtville, CA (Golden Dawn III)	3	0.34	0.07	480-500	Fruit	0	0.12, 0.13	0.13	2013/7001798 10478.10- CA136
USA, 2010 Citra, FL (Gentry)	3	0.33	0.05	640-650	Fruit	0	0.1, 0.16	0.13	2013/7001798 10478.10-FL40
USA, 2010 Salisbury, MD (Conqueror III)	3	0.33	0.08	390-400	Fruit	0	0.11, 0.09	0.1	2013/7001798 10478.10- MD18
USA, 2010 (Clinton, NC (Multipik)	3	0.34	0.085	400	Fruit	0	0.17, 0.18	0.17	2013/7001798 10478.10-NC29
USA, 2010 Freeville, NY (Multipik)	1+ 2	0.33 0.33	0.12 0.06	270 560	Fruit	0	0.08, 0.07	0.07	2013/7001798 10478.10-NY30
Canada, 2010 Delhi, ON (Leopard)	3	0.33	0.05	690	Fruit	0	0.13, 0.14	0.14	2013/7001798 10478.10-ON21
Canada, 2010 Harrow, ON (Select)	1+ 3	0.14+ 0.34	0.04 0.08	380-410	Fruit	0 1 4 6 9 13	0.25, 0.18 0.07, 0.06 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01	0.22 0.07 < 0.01 < 0.01 < 0.01 < 0.01	2013/7001798 10478.10-ON22 3-d interval between 1 st and 2 nd applications
Canada, 2010 L'Acadie, QB (Golden Dawn III)	3	0.35	0.06	600-620	Fruit	0	0.1, 0.1	0.11	2013/7001798 10478.10-QC11
USA, 2010 Charleston, SC (Superpik F1)	3	0.34	0.07	500-520	Fruit	0	0.08, 0.12	0.1	2013/7001798 10478.10-SC13
USA, 2010 Weslaco, TX (Anton)	3	0.34	0.09	390-400	Fruit	0	0.14, 0.08	0.11	2013/7001798 10478.10-TX21
USA, 2010 Weslaco, TX (Multipik)	3	0.34	0.07	510	Fruit	0	0.24, 0.31	0.28	2013/7001798 10478.10-TX22
USA, 2010 Moxee, WA (Noche F1)	3	0.34	0.07	500	Fruit	0	0.12, 0.13	0.12	2013/7001798 10478.10- WA36

Melons, except Watermelon

In the European outdoor trials, two foliar applications of 0.1 kg ai/ha metrafenone (SC formulation) in about 300–1000 L water/ha were applied 7 days apart, using motorized knapsacks or 4–8 nozzle mini-boom sprayers. Plot sizes were larger than 30 square metres.

Fruit samples (min 2 kg or 12 units) were subsampled in the field (two opposite quarters/fruit) and frozen within 12 hours and stored at or below -18 °C for up 14 months before analysis for metrafenone using Method 535/3. Average procedural recoveries of metrafenone from samples fortified with 0.01 mg/kg and 1.0 mg/kg ranged from 93% to 98% and the LOQ was 0.01 mg/kg.

In the North American trials, three foliar applications of 0.34 kg ai/ha metrafenone (SC formulation) with added adjuvant were applied at 6–8 day intervals, using motorized knapsacks or

tractor-mounted 4–9 nozzle sprayers to apply about 300–700 L/ha. Plot sizes were larger than 33 square metres.

Duplicate fruit samples (min 2 kg, 12 units) were sub-sampled in the field (two opposite quarters, eighths or sixteenths/fruit), frozen within 12 hours and stored frozen for up to 27 months before analysis for metrafenone using the QuEChERS LC-MS/MS method. The average procedural recovery of metrafenone from samples fortified with 0.01 mg/kg or 1.0 mg/kg was 104% and the LOQ was 0.01 mg/kg.

Table 48 Residues in outdoor melons from supervised trials in Europe involving two foliar applications of metrafenone (SC formulation)

MELON Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	
GAP: France	2	0.1				3	RTI: 7–10d	
France (N), 2009 La Roche Clermont (Hugo)	2	0.1	0.033	300	fruit	0 1 3 7	0.04 0.02 <u>0.02</u> 0.02	2010/1033968 BSF 0727-02 Late May planting into sandy clay
France (S), 2009 Cheval Blanc (Anasta)	2	0.1	0.01	1000	fruit	0 1 3 7	0.02 0.01 <u>< 0.01</u> < 0.01	2010/1033968 BSF 0727-03
Italy, 2009 Lupatoto (Macigno)	2	0.1	0.01	1000	fruit	0 1 3 7	0.13 0.07 <u>0.07</u> 0.02	2010/1033968 BSF 0727-04
Spain, 2009 La Llosa (Medellin)	2	0.1	0.01	1000	fruit	0 1 3 8	0.02 0.01 <u>0.01</u> < 0.01	2010/1033968 BSF 0727-05
Greece, 2009 Svoronos, Piera (Lavigal)	2	0.1	0.01	1000	fruit	0 1 3 7	0.06 0.04 <u>0.03</u> 0.02	2010/1033968 BSF 0727-06
France (N), 2009 La Roche Clermont (Hugo)	2	0.1	0.033	300	fruit	0 1 3 7	0.05 0.04 <u>0.02</u> 0.02	2010/1033968 BSF 0727-07 Late June planting into clay loam
Germany, 2010 Goch (Charentais)	2	0.1	0.033	300	fruit	0 1 4 6	0.03 0.04 <u>0.02</u> < 0.01	2011/1041395 S10-00481 -01
Germany, 2010 Baden-Wurttemberg (Charentaise)	1+ 1	0.097 0.094	0.032 0.032	300 290	fruit	0 1 3 6	0.13 0.06 <u>0.06</u> 0.03	2011/1041881 S10-00481 -01
France (N), 2010 Yvelines (Delta)	2	0.1	0.033	300	fruit	0 1 3 7	0.07 0.04 <u>0.05</u> 0.03	2011/1041881 S10-00481 -02
France (S), 2010 Tarn-et-Garonne (Hugo)	2	0.1	0.033	300	fruit	0 1 3 7	0.04 0.02 <u>0.01</u> 0.01	2011/1041881 S10-00481 -03

MELON Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	
Italy, 2010 Lombardia (Bacir)	2	0.1	0.033	300	fruit	0 1 3 7	0.01 0.01 <u>0.02</u> 0.01	2011/1041881 S10-00481 -04
Spain, 2010 Los Palacios y Villafranca (Nicolas)	2	0.1	0.033	300	fruit	0 1 2 7	0.03 0.03 <u>0.02</u> 0.01	2011/1041881 S10-00481 -05
Greece, 2010 Thessaloniki (Lavigal)	2	0.1	0.033	300	fruit	0 1 3 7	0.05 0.01 <u>0.02</u> < 0.01	2011/1041881 S10-00481 -06

Table 49 Residues in outdoor melons (cantaloupes) from supervised trials in North America involving three foliar applications of metrafenone (SC formulation)

MELON Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	mean	
USA, 2010 Maricopa, AZ (Primo)	3	0.32	0.07	386–434	fruit	0	0.17, 0.14	0.15	2013/7001797 10477.10-AZ05
USA, 2010 Riverside, CA (Caravelle)	3	0.33	0.07	466–471	fruit	0	0.21, 0.24	0.23	2013/7001797 10477.10-CA133
USA, 2010 Holtville, CA (Navigator)	3	0.33	0.07	488–498	fruit	0	0.12, 0.14	0.13	2013/7001797 10477.10-CA134
USA, 2010 Parlier, CA (Durango)	3	0.35	0.07	466–471	fruit	0	0.07, 0.1	0.09	2013/7001797 10477.10-CA135
USA, 2010 Tifton, GA (Edisto 47)	3	0.33	0.08	392–405	fruit	0	0.23, 0.33	0.28	2013/7001797 10477.10-GA14
USA, 2010 Salisbury, MD (Athena)	3	0.33	0.08	399–405	fruit	0 1 3 7 10 13	0.21, 0.14 0.12, 0.09 0.04, 0.05 0.04, 0.04 0.02, 0.03 0.03, 0.03	0.18 0.11 0.04 0.04 0.03 0.03	2013/7001797 10477.10-MD17
USA, 2010 Las Cruces, NM (PMR 45)	3	0.34	0.08	409–435	fruit	0	0.15, 0.12	0.13	2013/7001797 10477.10-NM13
USA, 2010 Freemont, OH (Odyssey)	3	0.34	0.08	426–442	fruit	0	0.04 ^a , 0.04 ^a	0.04	2013/7001797 10477.10-OH-18
Canada, 2010 Delhi, ON (Jaipur)	3	0.34	0.085	402–404	fruit	0 1 3 7 11 15	0.13, 0.14 0.11, 0.09 0.08, 0.08 0.06, 0.05 0.03, 0.07 0.04, 0.03	0.13 0.1 0.08 0.05 0.05 0.03	2013/7001797 10477.10-ON20
Canada, 2010 L'Acadie, QC (Athena)	3	0.35	0.08	407–423	fruit	0	0.17, 0.24	0.21	2013/7001797 10477.10-QC10

MELON Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	mean	
USA, 2010 Weslaco, TX (Mission)	3	0.34	0.09	391–396	fruit	0	0.19, 0.17	0.18	2013/7001797 10477.10-TX20
USA, 2010 Weslaco, TX (Sarah's Choice)	3	0.34	0.09	393–396	fruit	0	0.08, 0.08	0.08	2013/7001797 10477.10-TX19

^a Average results from three replicate analyses

Fruiting vegetables, other than Cucurbits

Results from supervised trials from Europe and the USA on peppers and tomatoes and on mushrooms from Europe were provided to the Meeting.

Mushrooms

In a trial conducted in France with mushroom compost sourced from four different suppliers, one drench application equivalent to 0.05 kg ai metrafenone/100 square metres was applied to the mushroom compost in 15–17 litres water, 20 days after inoculation and mushroom samples (min 0.8 kg) were harvested 10 days after treatment and stored frozen for up to 17 months before analysis using the MRM DFG S19 method to measure metrafenone. The LOQ for this method was 0.1 mg/kg and average procedural recovery rates were 86% and 88% in samples spiked with 0.1 mg/kg and 1.0 mg/kg respectively.

Duplicate samples were also analysed at a separate laboratory after a further 9 months frozen storage using Method 535/1 with a lower LOQ of 0.01 mg/kg and with average procedural recovery rates of 101% and 91% in samples spiked with 0.01 mg/kg and 0.2 mg/kg respectively.

One (5 kg) sample was also taken for processing, with the mushrooms being washed 3–4 times in cold running water, stored for 24 hours in sulphured water and blanched for 15 minutes at 98 °C before cooling and placed in cans with added salt, sugar and citric acid. After sealing, the cans were sterilised at 136 °C for 6 minutes.

Table 50 Residues in indoor mushrooms from supervised trials in Europe involving one application of metrafenone (SC formulation) to mushroom compost

MUSHROOMS Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ 100 m ²	kg ai/hL	water (L/100 m ²)			metrafenone	mean	
GAP: France	1	0.05		15		10			
France, 2009 Chace (Amycel Delta)	1	0.056		16.7	fruit	10	0.17, 0.2	<u>0.19</u>	2011/1151102 2011/1144320 RE09001
France, 2009 St Laurent du Lin (Amycel Delta)	1	0.052		15.3	fruit	10	< 0.1, 0.09	<u>0.1</u>	2011/1151102 2011/1144320 RE09002
France, 2009 Roiffe (Amycel Delta)	1	0.053		15.7	fruit	10	< 0.1, 0.11	<u>0.11</u>	2011/1151102 2011/1144320 RE09003
France, 2009 Longue Jumelles (Amycel Delta)	1	0.051		15.3	fruit canned	10 10	0.1, 0.1 < 0.1, 0.02	<u>0.1</u> 0.02	2011/1151102 2011/1144320 RE09004

Results are duplicate analyses by separate laboratories (different analytical methods and LOQs)

Processing factor (residues in processed commodity/residues in RAC) = 0.16

Peppers

In the European trials on indoor sweet peppers, two foliar applications of 0.15 kg ai/ha metrafenone (SC formulation) in about 1000 L water/ha were applied 7 days apart, using motorized knapsack sprayers. Whole fruit samples (min 1 kg) were frozen within 24 hours of sampling and stored frozen (–18 °C) for up to 12 months before analysis for metrafenone using Method 535/3. Average procedural recoveries of metrafenone from samples fortified with 0.01–2.0 mg/kg ranged from 73% to 132% with an overall mean of 96% and the LOQ was 0.01 mg/kg.

In the North American trials on outdoor sweet (bell) peppers and chili (non-bell) peppers, three foliar applications of 0.34 kg ai/ha metrafenone (SC formulation) with added adjuvant were applied at 7 day intervals, using pressurised knapsack sprayers (2–6 nozzles) to apply about 200–300 L/ha. Plot sizes were larger than 28 square metres.

Duplicate whole fruit samples (min 2 kg, 12 large or 24 small fruit) were frozen within 2 hours of sampling and stored frozen (–15 °C) for up to 25 months before analysis for metrafenone using the QuEChERS LC-MS/MS method. Average procedural recoveries of metrafenone from samples fortified with 0.01–0.1 mg/kg ranged from 109% to 114% with an overall mean of 112% and the LOQ was 0.01 mg/kg.

Table 51 Residues in indoor sweet peppers from supervised trials in Europe involving two foliar applications of metrafenone (SC formulation)

PEPPER, SWEET Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	
GAP: France	2	0.15				3	RTI 7–10 d	
Germany, 2009 Baden-Wuerttemberg (Golden Calwander)	2	0.15	0.015	1000	fruit	0 1 3 7	0.74 0.7 <u>1.3</u> ^a 1.0 ^a	2010/1199010 S09-00770-01
Netherlands 2009 Bemmel (Fantasy)	2	0.15	0.015	1000	fruit	0 1 3 7	0.08 0.06 <u>0.07</u> 0.07	2010/1199010 S09-00770-02
Belgium, 2009 Sint-Katelijne-waver (Morraine)	2	0.15	0.015	1000	fruit	0 1 3 7	0.18 0.17 <u>0.12</u> 0.1	2010/1199010 S09-00770-03
France (N), 2009 Loriet (Spartakus)	2	0.15	0.015	1000	fruit	0 1 3 7	0.33 0.31 <u>0.2</u> 0.18	2010/1199010 S09-00770-04
Italy, 2009 Fondi (San Marco)	2	0.15	0.015	1000	fruit	0 1 4 8	0.09 0.11 <u>0.11</u> 0.1	2010/1199010 S09-00770-05
France (N), 2009 Bioule (Mariner)	2	0.15	0.015	1000	fruit	0 1 3 7	0.1 ^a 0.06 ^a 0.08 <u>0.08</u>	2010/1199010 S09-00770-06
Spain, 2009 Valencia (Filon)	2	0.15	0.015	1000	fruit	0 1 3 7	0.17 0.17 <u>0.21</u> 0.2	2010/1199010 S09-00770-07
Greece, 2009 Thessaloniki (Raikon)	2	0.15	0.015	1000	fruit	0 1 3 7	0.08 0.15 <u>0.1</u> 0.06	2010/1199010 S09-00770-08

^a Mean of two analyses

Table 52 Residues in outdoor peppers (bell and non-bell) from supervised trials in North America involving three foliar applications of metrafenone (SC formulation)

PEPPER Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	mean	
USA, 2010 Madera, CA (Jupiter) Bell pepper	3	0.34	0.12	280	fruit	0 3 7 10 14	0.39, 0.43 0.16, 0.18 0.11, 0.08 0.11, 0.08 0.04, 0.04	0.41 0.17 0.1 0.09 0.04	2013/7000577 R100014
USA, 2010 Chula, GA (Bell) Bell pepper	3	0.34	0.15	230	fruit	0 7	0.47, 0.33 0.17, 0.27	0.4 0.22	2013/7000577 R100015
USA, 2010 Shady Grove, FL (Aristotle) Bell pepper	3	0.33	0.15	220	fruit	0 7	0.18, 0.11 0.13, 0.11	0.15 0.12	2013/7000577 R100016
USA, 2010 Marengo, IL (Lady Bell) Bell pepper	3	0.33	0.15	230	fruit	0 7	0.33, 0.17 0.2, 0.19	0.25 0.19	2013/7000577 R100017
USA, 2010 Hinton, OK (California Wonder) Bell pepper	3	0.34	0.14	250	fruit	0 7	0.33, 0.21 0.1, 0.1	0.27 0.1	2013/7000577 R100018
USA, 2010 Madera, CA (Jupiter) Bell pepper	3	0.36	0.12	280	fruit	0 7	0.51, 0.34 0.2, 0.27	0.43 0.23	2013/7000577 R100019
USA, 2010 Chula, GA (Antillano) Non-bell pepper	3	0.34	0.15	230	fruit	0 7	0.37, 0.34 0.17, 0.12	0.35 0.14	2013/7000577 R100020
USA, 2010 Larned, KS (Jalapeno M) Non-bell pepper	3	0.34	0.16	210	fruit	0 7	0.07, 0.1 0.06, 0.07	0.08 0.07	2013/7000577 R100021
USA, 2010 Madera, CA (Jalapeno RPP7072) Non-bell pepper	3	0.34	0.12	280	fruit	0 7	0.33, 0.67 0.3, 0.29	0.5 0.3	2013/7000577 R100022

Tomato

In the European trials on outdoor and indoor tomatoes, two foliar applications of 0.225 kg ai/ha metrafenone (SC formulation) in about 1500 L water/ha (0.015 kg ai/hL) were applied 7 days apart, using motorized knapsack (1–2 nozzle) or mini-boom (6-nozzle) sprayers. Whole fruit samples (min 1 kg) were frozen within 24 hours of sampling and stored frozen (–18 °C) for up to 12 months before analysis for metrafenone using Method 535/3. Average procedural recoveries of metrafenone from samples fortified with 0.01–1.0 mg/kg ranged from 78% to 111% with an overall mean of 102% in the outdoor trials and 99% in the indoor trials. The LOQ was 0.01 mg/kg.

In the North American trials on outdoor tomatoes (large and small fruited varieties), three foliar applications of 0.34 kg ai/ha metrafenone (SC formulation) with added adjuvant were applied at 6–8 day intervals, using knapsack or tractor-mounted boom sprayers (3–11 nozzles) to apply about 300–800 L/ha. Plot sizes were larger than 30 square metres.

Duplicate fruit samples (min 2 kg, 12 large or 24 small fruit) were frozen within 3 hours of sampling and stored frozen (–15 °C) for up to 24 months before analysis for metrafenone using the QuEChERS LC-MS/MS method. Average procedural recoveries of metrafenone from samples fortified with 0.01–0.1 mg/kg ranged from 91% to 118% with an overall mean of 105% and the LOQ was 0.01 mg/kg.

Table 53 Residues in outdoor tomatoes from supervised trials in Europe involving two foliar applications of metrafenone (SC formulation)

TOMATO Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)	Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	
GAP: Spain	2		0.015			3		
France, 2009 Lot et-Garonne (Perfect Peel)	1+	0.23	0.015	1540	fruit	0	0.09	2010/1193371 S09-00772-01
	1	0.22	0.015	1480		1	0.11	
						3	0.08	
						7	0.05	
Greece, 2009 Thessaloniki (Meteor)	2	0.23	0.015	1500	fruit	0	< 0.01	2010/1193371 S09-00772-02
						1	< 0.01	
						4	0.02	
						7	0.02	
Italy, 2009 Granarola (Guadalete)	1+	0.23	0.015	1560	fruit	0	0.14	2010/1193371 S09-00772-03
	1	0.21	0.015	1430		1	0.15	
						4	0.05	
						7	0.05	
Spain, 2009 Remolinos (Guadivia)	2	0.23	0.015	1500	fruit	0	0.06	2010/1193371 S09-00772-04
						1	0.06	
						3	0.05	
						7	0.05	
France (S), 2010 Tarn-et-Garonne (Perfect Peel)	2	0.23	0.015	1500	fruit	0	0.31	2011/1041882 S10-00480-01
						1	0.25	
						3	0.15	
						7	0.07	
Greece, 2010 Thessaloniki (Meteor)	2	0.23	0.015	1500	fruit	0	< 0.01	2011/1041882 S10-00480-02
						1	0.06	
						4	0.06	
						7	0.06	
Italy, 2010 Bologna (Gigantico)	2	0.23	0.015	1500	fruit	0	0.14	2011/1041882 S10-00480-03
						1	0.1	
						3	0.07	
						7	0.05	
Spain, 2010 Remolinos (H-9036)	2	0.23	0.015	1500	fruit	0	0.12	2011/1041882 S10-00480-04
						1	0.12	
						3	0.06	
						7	0.04	

Table 54 Residues in indoor tomatoes from supervised trials in Europe involving two foliar applications of metrafenone (SC formulation)

TOMATO Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone		
GAP: France	2	0.225	0.015			3	RTI: 7–10d		
GAP: Spain	2		0.015			3			
France (N), 2009 Loiret (Recento)	2	0.22	0.015	1480	fruit	0 1 3 7	0.22 0.21 <u>0.17</u> 0.15		2010/1199009 S09-00769-01
Netherlands, 2009 Huissen (Tourance)	2	0.225	0.015	1500	fruit	0 1 3 7	0.09 0.14 <u>0.1</u> 0.09		2010/1199009 S09-00769-02
Germany, 2009 Heidelberg (Sakura)	2	0.22	0.015	1500	fruit	0 1 3 7	0.14 0.12 <u>0.1</u> 0.11		2010/1199009 S09-00769-03
Belgium, 2009 St-Katelijne-waver (Tricia)	1+ 1	0.22 0.21	0.015	1450 1400	fruit	0 1 3 7	0.19 0.15 0.15 <u>0.16</u>		2010/1199009 S09-00769-04
France (N), 2009 Bouloc (Gourdena)	2	0.225	0.015	1500	fruit	0 1 3 7	0.1 0.13 <u>0.1</u> 0.1		2010/1199009 S09-00769-05
Italy, 2009 Fondi (Caramba)	2	0.225	0.015	1500	fruit	0 1 3 7	0.1 0.17 <u>0.09</u> 0.09		2010/1199009 S09-00769-06
Spain, 2009 Valencia (Rambo)	1+ 1	0.23 0.2	0.015 0.015	1500 1320	fruit	0 1 3 7	0.07 0.07 <u>0.09</u> 0.07		2010/1199009 S09-00769-07
Greece, 2009 Thessaloniki (Optima)	2	0.225	0.015	1500	fruit	0 1 3 7	0.08 0.03 <u>0.06</u> 0.06		2010/1199009 S09-00769-08

Table 55 Residues in outdoor tomatoes from supervised trials in North America involving three foliar applications of metrafenone (SC formulation)

TOMATO Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	mean	
USA, 2010 Tifton, GA (Amelia) large fruited	3	0.34	0.11	300	fruit	0 8	0.09, 0.11 0.01, 0.01	0.1 0.01	2013/7001658 10467.10- GA13
USA, 2010 Davis, CA (Sun 6366) processing	3	0.34	0.11	300	fruit	0 6	0.22, 0.18 0.11, 0.12	0.2 0.12	2013/7001658 10467.10- CA127

TOMATO Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	mean	
USA, 2010 Clinton, NC (Amelia) large, fruited	3	0.34	0.09	380	fruit	0 6	0.08, 0.08 0.05, 0.03	0.08 0.04	2013/7001658 10467.10- NC28
USA, 2010 Las Cruces, NM (Celebrity) large-fruited	3	0.34	0.04	840	fruit	0 6	0.11, 0.11 0.11, 0.08	0.11 0.09	2013/7001658 10467.10- NM12
USA, 2010 Holt, MI (Mountain Spring) large-fruited	3	0.34	0.11	300	fruit	0 6	0.15, 0.19 0.07, 0.11	0.17 0.09	2013/7001658 10467.10- MI42
USA, 2010 Maricopa, AZ (Phoenix) large-fruited	4	0.34	0.09	360	fruit	0 7	0.1, 0.11 0.03, 0.03	0.11 0.03	2013/7001658 10467.10- AZ04
USA, 2010 Davis, CA (Shady Lady) large-fruited	3	0.34	0.11	310	fruit	0 7	0.17, 0.18 0.09, 0.08	0.18 0.09	2013/7001658 10467.10- CA125
USA, 2010 Holtville, CA (Shady Lady) large-fruited	3	0.34	0.11	300	fruit	0 7	0.2, 0.29 0.1, 0.07	0.25 0.09	2013/7001658 10467.10- CA119
USA, 2010 Freemont, OH (Heinz 3402) processing	3	0.34	0.08	430	fruit	0 6	0.09, 0.09 0.04, 0.04	0.09 0.04	2013/7001658 10467.10- OH17 rain over picking
USA, 2010 Freeville, NY (Scarlet Red) processing	3	0.34	0.06	580	fruit	0 8	0.29, 0.28 0.14, 0.19	0.29 0.16	2013/7001658 10467.10- NY27
USA, 2010 Parlier, CA (AB-2) processing	3	0.34	0.09	390	fruit	0 7	0.09, 0.12 0.05, 0.05	0.11 0.05	2013/7001658 10467.10- CA120
USA, 2010 Riverside, CA (Celebrity) large-fruited	3	0.34	0.07	470	fruit	0 6	0.11, 0.08 0.05, 0.06	0.1 0.05	2013/7001658 10467.10- CA122
USA, 2010 Parlier, CA (H 3155) processing	3	0.34	0.07	500	fruit	0 7	0.13, 0.08 0.03, 0.03	0.1 0.03	2013/7001658 10467.10- CA121
USA, 2010 Riverside, CA (Sun 6788) processing	3	0.34	0.07	470	fruit	0 7	0.24, 0.23 0.09, 0.1	0.23 0.09	2013/7001658 10467.10- CA126
USA, 2010 Davis, CA (Sun 6788) processing	3	0.34	0.11	310	fruit	0 7	0.25, 0.26 0.14, 0.12	0.26 0.13	2013/7001658 10467.10- CA123
USA, 2010 Holtville, CA (Naomi) small fruited	3	0.34	0.11	310	fruit	0 7	0.4, 0.45 0.11, 0.18	0.43 0.15	2013/7001658 10467.10- CA128

TOMATO Country, year Location (Variety)	Application				Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)			metrafenone	mean	
USA, 2010 Holtville, CA (Hypeel 45) processing	3	0.34	0.1	340	fruit	0	0.15 ^a , 0.05 ^a	0.1	2013/7001658 10467.10- CA124
						1	0.04, 0.07	0.05	
						3	0.06, 0.05	0.06	
						6	0.03, 0.03	0.03	
						10	0.02, 0.01	0.02	
						12	0.02, 0.01	0.02	
USA, 2010 Citra, FL (BHN602) large-fruited	3	0.34	0.05	620	fruit	0	0.1, 0.04	0.07	2013/7001658 10467.10- FL38
						1	0.09, 0.06	0.07	
						3	0.07, 0.08	0.08	
						7	0.07, 0.11	0.09	
						9	0.07, 0.04	0.05	
USA, 2010 Citra, FL (Jolly Elf) small-fruited	3	0.34	0.05	620	fruit	0	0.18, 0.26	0.22	2013/7001658 10467.10- FL39
						7	0.1, 0.17	0.11	

^a Sample re-injected in duplicate to confirm initial result.

Cereal grains

Results from supervised trials from Europe on wheat and barley were provided to the Meeting.

Wheat

In the European trials on winter and spring wheat, two foliar applications of 0.15–0.2 kg ai/ha metrafenone were applied by knapsack or plot sprayers with 4–12 nozzle booms to apply 20–400 Litres spray mix/ha to plots larger than 30 square metres. Generally, applications were made during the stem elongation period (BBCH 30–39) and again 4–8 weeks later (towards the end of flowering or later), up to about 5–6 weeks before harvest. In some trials, different formulations (SC, EC, SE) of metrafenone, alone or in combination with other fungicides were applied to separate plots.

Samples of grain (min 1 kg) were taken at maturity, frozen within 24 hours and stored at or below –18 °C for up to 29 months before analysis. In the trials conducted before 2004, samples were analysed for metrafenone and the metabolites CL 3000402, CL 434223 and CL 376991 using Method RLA 12619 and in the later trials, Method 535/3 was used to measure only the parent compound. Average procedural recoveries of metrafenone from samples fortified with 0.01 and 0.1 mg/kg ranged from 71% to 104% and the LOQ was 0.01 mg/kg.

Table 56 Residues in wheat grain from supervised trials in Europe involving 1–3 foliar applications of metrafenone

WHEAT Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
GAP: Poland		2	0.15				35		
Netherlands, 2000 Biddinghuizen (Tremie)	300SC (09957)	2	0.16	310	BBCH 76	grain	34	<u>0.04</u>	2001/7000487 00-770-01 RLA 12619.02V
	300SC (10358)	2	0.15	310	BBCH 76	grain	33	0.04	

WHEAT Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
France (N), 2000 Fains (Charger)	300SC (09957)	2	0.14	330	BBCH 77	grain	33	<u>0.03</u>	2001/7001657 00-831-346 RLA 12619.02V
	300SC (10358)	2	0.14	330	BBCH 77	grain	33	0.01	
UK, 2000 Burton-upon-Trent (Aardvark)	300SC (09957)	2	0.15	200	BBCH 79–83	grain	41	<u>0.03</u>	2001/7001658 00-832-01 RLA 12619.02V
	300SC (10358)	2	0.15	200	BBCH 79–83	grain	41	<u>0.04</u>	
Germany, 2000 Schwabenheim (Monopol)	300SC (09957)	2	0.15	300	BBCH 75	grain	35	< 0.01	2002/7004672 00-922-01 RLA 12619.02V
	300SC (10358)	2	0.15	300	BBCH 75	grain	35	<u>< 0.01</u>	
Germany, 1999 Zulpich-Mulheim (Bandit)	300SC (09957)	3	0.2	400	BBCH 65	grain	35 41	< 0.01 < 0.01	2001/7001675 99-106-01 RLA 12619.02V
Germany, 2000 Haimhausen (Tambor)	300SC (09957)	2	0.2	390	BBCH 69	grain	35 41	< 0.01 < 0.01	2001/7001675 99-106-02 RLA 12619.02V
UK, 1999 Newton (Consort)	300SC (09957)	2	0.2	300	BBCH 59–61	grain	41 49	< 0.01 < 0.01	2002/7004680 99-107-01 RLA 12619.02V
Netherlands, 1999 Biddinghuizen (Vivant)	300SC (09957)	2	0.2	270	BBCH 75	grain	35 41	0.01 < 0.01	2002/7004745 99-108-01 RLA 12619.02V
France (N), 2000 Le Plessis Hebert (Isengrain)	300SC (10358)	2	0.15	370	BBCH 73	grain	35	<u>< 0.01</u>	2001/7001660 00-834-347 RLA 12619.02V
Denmark, 2005 Middelfart (Kris)	75SE	2	0.15	200	BBCH 69	grain	35 42	< 0.01 <u>0.01</u>	2005/7004267 ALB/11/05 Method 535/0
	100SC	2	0.15	200	BBCH 69	grain	35 42	< 0.01 < 0.01	
	300SE	2	0.15	200	BBCH 69	grain	35 42	< 0.01 < 0.01	
Germany, 2005 Wurtenberg (Isengrain)	75SE	2	0.15	200	BBCH 69	grain	27 34 41	< 0.01 0.01 <u>0.02</u>	2005/7004267 DU2/11/05 Method 535/0
	100SC	2	0.15	200	BBCH 69	grain	27 34 41	< 0.01 < 0.01 0.01	

WHEAT Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
	300SE	2	0.15	200	BBCH 69	grain	27 34 41	0.01 < 0.01 0.01	
France (N), 2005 Villeveque (Royssac)	75SE	2	0.15	200	BBCH 69	grain	35 42	< 0.01 < 0.01	2005/7004267 FBM/11/05 Method 535/0
	100SC	2	0.15	200	BBCH 69	grain	35 42	< 0.01 < 0.01	
	300SE	2	0.15	200	BBCH 69	grain	35 42	<u>< 0.01</u> < 0.01	
UK, 2003 Bicester (Malacca)	100EC	2	0.15	300	BBCH 83	grain	34 41	<u>0.01</u> < 0.01	2004/1010542 OAT/02/03 Method 535/0
	300SC	2	0.15	300	BBCH 83	grain	34 41	0.01 < 0.01	
Denmark, 2002 Fuenen (Vinjett)	300SC	2	0.15	300	BBCH 51	grain	59	< 0.01	2003/1001354 ALB/01/02 Method 993/0 RLA 12619.03V
		1	0.15	300	BBCH 51	grain	59	< 0.01	
Germany, 2002 Baden-Wurttemberg (Transit)	300SC	2	0.15	300	BBCH 49	grain	58	< 0.01	2003/1001354 DU2/03/02 Method 993/0 RLA 12619.03V
	300SC	1	0.15	300	BBCH 49	grain	58	< 0.01	
Denmark, 2003 Fuenen (Triso) spring wheat	100EC	2	0.15	300	BBCH 77	grain	35 41	< 0.01 < 0.01	2004/1010542 ALB/01/03 Method 535/0
	300SC	2	0.15	300	BBCH 77	grain	35 41	<u>< 0.01</u> 0.01	
France S), 2000 Marguet-Meymes (Aztec)	300SC (09957)	2	0.16 0.145	370 350	BBCH 77-83	grain	29	<u>< 0.01</u>	2001/7001656 00-833-290 RLA 12619.02V
	300SC (10358)	2	0.15 0.14	350 340	BBCH 77-83	grain	29	< 0.01	
France (S), 2000 Mormes (Sideral)	300SC (09957)	2	0.15 0.14	360 330	BBCH 77-83	grain	23	< 0.01	2001/7001656 00-833-291 RLA 12619.02V
	300SC (10358)		0.15 0.14	345 338	BBCH 77-83	grain	23	0.01	
France (S), 2000 Pernes les Fontaines (Florence Aurore)	300SC (09957)	2	0.15	400	BBCH 83	grain	34	<u>0.01</u>	2001/7001656 00-833-643 RLA 12619.02V
	300SC (10358)		0.15	400	BBCH 83	grain	34	< 0.01	

WHEAT Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
France (S), 2000 Le Thor (Manital)	300SC (09957)	2	0.15	400	BBCH 83	grain	33	< 0.01	2001/7001656 00-833-644 RLA 12619.02V
	300SC (10358)		0.15 0.16	400 420	BBCH 83	grain	33	<u>0.01</u> (0.01)	
France (S), 1999 Averon Bergelle (Soissons Real)	300SC (09957)	2	0.2	400	BBCH 65–69	grain	35 42	0.01 < 0.01	2002/7004740 99-109-295 RLA 2619.02V
France (S), 1999 Espas (Soissons)	300SC (09957)	2	0.2	400	BBCH 65–69	grain	35 42	0.01 < 0.01	2002/7004740 99-109-296 RLA 2619.02V
France (S), 1999 Le Thor (Manital)	300SC (09957)	2	0.2	400	BBCH 75–77	grain	35 42	< 0.01 < 0.01	2002/7004740 99-109-631 RLA 2619.02V
France (S), 1999 Orange (Grenat)	300SC (09957)	2	0.2	400	BBCH 75–77	grain	35 42	< 0.01 < 0.01	2002/7004740 99-109-632 RLA 2619.02V
00-923-292 France (S), 2000 Averon-Bergelle (Soissons)	300SC (10358)	2	0.16	350	BBCH 75	grain	33	<u>≤ 0.01</u>	2001/7001676 00-923-292 RLA 2619.02V
France (S), 2000 Ausonne (Courtaud)	300SC	2	0.14 0.15	280 290	BBCH 75	grain	35	<u>0.03</u>	2002/7004890 FTL/32/01 RLA 12619.02V
France (S), 2005 Rhone-Alpes (Caphorn)	75SE	2	0.15	200	BBCH 69	grain	28 35 42	< 0.01 < 0.01 < 0.01	2005/7004267 FBD/33/05 Method 535/0
	100SC	2	0.15	200	BBCH 69	grain	28 35 42	< 0.01 < 0.01 < 0.01	
	300SE	2	0.15	200	BBCH 69	grain	28 35 42	< 0.01 <u>≤ 0.01</u> < 0.01	
Spain, 2003 Salteras (Vitromax) spring wheat	100EC	2	0.15	300	BBCH 69	grain	35 42	<u>≤ 0.01</u> < 0.01	2004/1010542 ALO/03/03 Method 535/0
	300SC	2	0.15	300	BBCH 69	grain	35 42	< 0.01 < 0.01	
France (S), 2003 Ausonne (Nefer)	100EC	2	0.15	300	BBCH 73	grain	35 42	<u>≤ 0.01</u> < 0.01	2004/1010542 FTL/03/03 Method 535/0
	300SC	2	0.15	300	BBCH 73	grain	35 42	< 0.01 < 0.01	

Values in (brackets) are residues of the CL 3000402 metabolite found above LOQ

Barley

In the European trials on winter and spring barley, two foliar applications of 0.15–0.2 kg ai/ha metrafenone were applied by knapsack or plot sprayers with 5–12 nozzle booms to apply 200–400 Litres spray mix/ha to plots larger than 30 square metres. Generally, applications were made 4–8 weeks apart over the flowering and grain development stages (BBCH 61–83). In some trials, different formulations (SC, EC, SE) of metrafenone, alone or in combination with other fungicides were applied to separate plots.

Samples of grain (min 1 kg) were taken at maturity, frozen within 24 hours and stored at or below –18 °C for up to 25 months before analysis. In the trials conducted before 2004, samples were analysed for metrafenone and the metabolites CL 3000402, CL 434223 and CL 376991 using Method RLA 12619 and in the later trials, Method 535/3 was used to measure only the parent compound. Average procedural recoveries of metrafenone from samples fortified with 0.01 and 0.1 mg/kg ranged from 71% to 108% and the LOQ was 0.01 mg/kg.

Table 57 Residues in barley grain from supervised trials in Europe involving 1–3 foliar applications of metrafenone.

BARLEY Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
GAP: Poland		2	0.15				35		
Germany, 2005 Gerolsheim (Scarlett)	75SE	2	0.15	200	BBCH 69	grain	29 36 42	< 0.01 < 0.01 < 0.01	2005/7004267 DU4/11/05 Method 535/0
	100SC	2	0.15	200	BBCH 69	grain	29 36 42	< 0.01 0.02 < 0.01	
	300SE	2	0.15	200	BBCH 69	grain	29 36 42	0.01 <u>0.02</u> < 0.01	
Sweden, 2005 Bjarred (Prestige)	75SE	2	0.15	200 200	BBCH 69	grain	36 43	<u>0.09</u> 0.06	2005/7004267 HUS/07/05 Method 535/0
	100SC	2	0.15	200 200	BBCH 69	grain	29 36 43	0.94 0.05 0.04	
	300SE	2	0.15	200 200	BBCH 69	grain	36 43	0.04 0.02	
UK, 2005 Bicester (Pearl)	75SE	2	0.15	200 200	BBCH 73	grain	29 36 42	0.05 0.05 0.02	2005/7004267 OAT/16/05 Method 535/0
	100SC	2	0.15	200 200	BBCH 73	grain	28 35 42	0.06 0.04 0.02	
	300SE	2	0.15	200 200	BBCH 73	grain	28 35 42	0.09 <u>0.05</u> 0.04	
France (N), 2002 Alsace (Astoria) spring barley	300SC	2	0.15	300	BBCH 56	grain	34	<u>< 0.01</u>	2003/1001354 FAN/02/02 Method 993/0 RLA 12619.03V
	300SC	1	0.15	300	BBCH 56	grain	39	< 0.01	

BARLEY Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
UK, 2002 Oxfordshire (Siberia)	300SC	2	0.15	300	BBCH 59	grain	39	<u>0.02</u>	2003/1001354 OAT/05/02 Method 993/0 RLA 12619.03V
	300SC	1	0.15	300	BBCH 59	grain	39	0.02	
Germany, 2003 Lentzke (Cadesse)	100EC	2	0.15	300	BBCH 69	grain	35 42	0.01 0.02	2004/1010542 ACK/04/03 Method 535/0
	300SC	2	0.15	300	BBCH 69	grain	35 42	<u>0.06</u> 0.05	
France (N), 2003 Seebach (Astoria) spring barley	100EC	2	0.15	300	BBCH 73	grain	36 42	0.02 0.05	2004/1010542 FAN/05/03 Method 535/0
	300SC	2	0.15	300	BBCH 73	grain	36 42	0.02 <u>0.05</u>	
Germany, 1999 Euskirchen- Oberwichterich (Uschi)	300SC	3	0.2	400	BBCH 65	grain	35 44	0.04 0.04	2001/7001659 99-111-01 RLA 12619.02V
Germany, 1999 Gemarkung (Duet)	300SC	2	0.2	390	BBCH 61	grain	35 42	0.02 0.03	2001/7001659 99-111-02 RLA 12619.02V
Germany, 1999 Ramsen (Angora)	300SC	2	0.2	300	BBCH 69	grain	36 44	0.09 0.07	2001/7001659 99-111-03 RLA 12619.02V
France (N), 2000 Bonnieres Sur Seine (Esterel)	300SC (09957)	1+ 1	0.13 0.14	310 350	BBCH 77	grain	36	<u>0.15</u> (0.01)	2002/7004445 00/835/355 RLA 12619.02V
	300SC (10358)	2	0.14	340	BBCH 77	grain	36	0.15 (0.01)	
Germany, 2000 Obersteinach (Hanna)	300SC (09957)	1+ 1	0.15 0.14	300 290	BBCH 77	grain	42	0.01 (0.02)	2002/7004463 00/837/01 RLA 12619.02V
	300SC (10358)	2	0.15	300	BBCH 77	grain	42	<u>0.11</u> (0.01)	
UK 2000 Lichfield (Jewel)	300SC (09957)	2	0.15	200	BBCH 77– 79	grain	35	0.14 (0.02)	2002/7004529 00/836/01 RLA 12619.02V
	300SC (10358)	2	0.15	200	BBCH 77– 79	grain	35	<u>0.16</u> (0.01)	
UK, 2000 Bradwall (Jewel)	300SC (09957)	2	0.15	200	BBCH 75– 77	grain	35	<u>0.07</u>	2002/7004529 00/836/02 RLA 12619.02V
	300SC (10358)	2	0.15	200	BBCH 75– 77	grain	35	0.06	

BARLEY Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
UK, 1999 Newton (Intro)	300SC	2	0.2	300	BBCH 61	grain	43 50	0.02 (0.01) 0.02	2002/7004681 99-110-01 RLA 12619.02V
France (N) 2000 Mousseaux (Majestic)	300SC	2	0.15	370	BBCH 77	grain	36	<u>0.4</u> (0.02)	2002/7004922 00/840/356 RLA 12619.02V
France (S), 2000 LaGardelle Sur Leze (Nevada)	300 SC	2	0.15 0.14	300 280	BBCH 73	grain	35	<u>0.05</u>	2002/7004890 FTL/31/01 RLA 12619.02V
France (S), 2005 Genissieux (Orelie)	75SE	2	0.15	200 200	BBCH 69	grain	35 41	0.02 0.03	2005/7004267 FBD/34/05 Method 535/0
	100SC	2	0.15	200 200	BBCH 69	grain	29 35 41	0.4 0.02 0.01	
	300SE	2	0.15	200 200	BBCH 69	grain	35 41	<u>0.03</u> 0.03	
France (S), 2003 Rhone-Alpes (Orelie)	100EC	2	0.15	300	BBCH 83	grain	35 42	0.03 0.02	2004/1010542 FBD/02/03 Method 535/0
	300SC	2	0.15	300	BBCH 83	grain	35 42	<u>0.08</u> 0.07	
Italy, 2003 Pozzolo (Prosa) spring barley	100EC	2	0.15	300	BBCH 55	grain	35 42	< 0.01 < 0.01	2004/1010542 ITA/03/03 Method 535/0
	300SC	2	0.15	300	BBCH 55	grain	35 42	<u>0.02</u> < 0.01	
France (S), 1999 Margouet-Meynes (Sunrise)	300SC	2	0.2	400	BBCH 60– 61	grain	34 40	0.08 0.12	2001/7000488 99-112-297 RLA 12619.02V
France (S), 1999 Espas (Systel)	300SC	2	0.2	400	BBCH 65– 69	grain	34 40	0.05 0.12	2001/7000488 99-112-298 RLA 12619.02V
France (S), 1999 Courthezon (Baraka)	300SC	2	0.2	400	BBCH 75– 77	grain	35 42	0.08 0.06	2001/7000488 99-112-633 RLA 12619.02V
France (S), 1999 Althen des Paluds (Baraka)	300SC	2	0.2	400	BBCH 75– 77	grain	35 42	0.04 0.04	2001/7000488 99-112-634 RLA 12619.02V
France (S), 2000 Averon-Bergelle (Plantine)	300SC (09957)	2	0.16	240	BBCH 77– 83	grain	34	<u>0.13</u> (0.01)	2002/7004525 00/839/294 RLA 12619.02V
	300SC (10358)	2	0.16	240	BBCH 77– 83	grain	34	0.1 (0.01)	

BARLEY Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
France (S), 2000 Saumane (Baraka)	300SC (09957)	2	0.15	410	BBCH 77– 83	grain	36 36	<u>0.06</u> (0.01)	2002/7004525 00/839/646 RLA 12619.02V
	300SC (10358)	1+ 1	0.15 0.16	410 420	BBCH 77– 83	grain	36	0.05 (0.01)	
France (S), 2000 Althen Des Paluds (Baraka)	300SC (09957)	2	0.15	400	BBCH 83	grain	34	<u>0.04</u>	2002/7004525 00/839/645 RLA 12619.02V
	300SC (10358)	1+ 1	0.16 0.15	420 410	BBCH 83	grain	34	0.04	
France (S), 2000 Bedarrides (Baraka)	300SC	1+ 1	0.14 0.15	390 400	BBCH 77– 83	grain	35	<u>0.23</u>	2002/7004744 00-841-647 RLA 12619.02V

Values in (brackets) are residues of the CL 3000402 metabolite found above LOQ

Animal feeds

Wheat forage, hay and straw

In some of the above European trials on winter and spring wheat, whole plants (without roots) were sampled 0–14 days after the last application and again 28–35 DALT, with these later samples being separated into ears and the rest-of- the-plant ('foliage'). Samples were frozen within 24 hours and stored at or below -18 °C for up 29 months before analysis. In the trials conducted before 2004, samples were analysed for metrafenone and the metabolites CL 3000402, CL 434223 and CL 376991 using Method RLA 12619 and in the later trials, Method 535/3 was used to measure only the parent compound. Average procedural recoveries of metrafenone from samples fortified with 0.01–20 mg/kg ranged from 71% to 104% and the LOQs were 0.1 mg/kg.

Table 58 Residues in wheat forage from supervised trials in Europe involving 1–3 foliar applications of metrafenone

WHEAT FORAGE Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
GAP: Poland		2	0.15				35		
Germany, 1999 Zulpich-Mulheim (Bandit)	300SC (09957)	3	0.2	400	BBCH 65	plant	–0 0 14	< 0.1 < 0.1 < 0.1	2001/7001675 99-106-01 RLA 12619.02V
						ears foliage	28 28	< 0.1 0.15	
Germany, 2000 Haimhausen (Tambor)	300SC (09957)	2	0.2	390	BBCH 69	plant	–0 0 14	< 0.1 < 0.1 < 0.1	2001/7001675 99-106-02 RLA 12619.02V
						ears foliage	28 28	< 0.1 0.13	

WHEAT FORAGE Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
UK, 1999 Newton (Consort)	300SC (09957)	2	0.2	300	BBCH 59–61	plant	–0 0 14	0.17 2.6 0.76	2002/7004680 99-107-01 RLA 12619.02V
						ears	28 35	< 0.01 1.4 ^a	
						foliage	28 35	1.1 0.04 ^a	
Netherlands, 1999 Biddinghuizen (Vivant)	300SC (09957)	2	0.2	270	BBCH 75	plant	–0 0 13	0.11 3.3 0.58	2002/7004745 99-108-01 RLA 12619.02V
						ears foliage	26 26	0.17 0.94	
France (N), 2000 Le Plessis Hebert (Isengrain)	300SC (10358)	2	0.15	370	BBCH 73	plant	–0 0 14	< 0.1 <u>2.6</u> 1.62	2001/7001660 00-834-347 RLA 12619.02V
						ears foliage	28 28	0.25 1.5	
Denmark, 2005 Middelfart (Kris)	75SE	2	0.15	200	BBCH 69	foliage	0 28	<u>2.6</u> 1.6	2005/7004267 ALB/11/05 Method 535/0
						ear	0 28	1.1 0.04	
	100SC	2	0.15	200	BBCH 69	foliage	0 28	2.2 0.86	
						ear	0 28	1.4 0.07	
	300SE	2	0.15	200	BBCH 69	foliage	0 28	2.2 2.0	
						ear	0 28	1.4 0.15	
Germany, 2005 Wurtemberg (Isengrain)	75SE	2	0.15	200	BBCH 69	foliage ear	0 0	1.8 1.3	2005/7004267 DU2/11/05 Method 535/0
	100SC	2	0.15	200	BBCH 69	foliage ear	0 0	2.1 1.4	
	300SE	2	0.15	200	BBCH 69	foliage ear	0 0	<u>2.6</u> 2.1	
France (N), 2005 Villeveque (Royssac)	75SE	2	0.15	200	BBCH 69	foliage	0 28	2.1 0.93	2005/7004267 FBM/11/05 Method 535/0
						ear	0 28	1.6 0.11	
	100SC	2	0.15	200	BBCH 69	foliage	0 28	<u>2.8</u> 0.33	
						ear	0 28	1.7 0.09	

WHEAT FORAGE Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
	300SE	2	0.15	200	BBCH 69	foliage	0 28	2.5 1.8	
						ear	0 28	3.6 0.09	
UK, 2003 Bicester (Malacca)	100EC	2	0.15	300	BBCH 83	plant	0	<u>2.0</u>	2004/1010542 OAT/02/03
						ear	27	0.16	Method 535/0
						foliage	27	2.5	
	300SC	2	0.15	300	BBCH 83	plant	0	1.2	
						ear	27	0.17	
						foliage	27	2.8	
Denmark, 2002 Fuenen (Vinjett)	300SC	2	0.15	300	BBCH 51	plant	0	<u>1.8</u>	2003/1001354 ALB/01/02
						foliage	36	0.2	Method 993/0 RLA 12619.03V
						ear	36	< 0.01	
		1	0.15	300	BBCH 51	plant	0	2.7	
						foliage	36	0.09	
						ear	36	< 0.01	
Germany, 2002 Baden-Wuerttemberg (Transit)	300SC	2	0.15	300	BBCH 49	plant	0	<u>2.0</u>	2003/1001354 DU2/03/02
						foliage	35	0.62	Method 993/0 RLA 12619.03V
						ear	35	< 0.01	
	300SC	1	0.15	300	BBCH 49	plant	0	1.6	
						foliage	35	0.46	
						ear	35	< 0.01	
Denmark, 2003 Fuenen (Triso)	100EC	2	0.15	300	BBCH 77	plant	0	2.7	2004/1010542 ALB/01/03
spring wheat						ear	28	0.09	Method 535/0
						foliage	28	0.9	
	300SC	2	0.15	300	BBCH 77	plant	0	<u>3.7</u>	
						ear	28	0.24	
						foliage	28	4.9	
France (S), 1999 Averon Bergelle (Soissons Real)	300SC (09957)	2	0.2	400	BBCH 65–69	plant	–0 0 13	0.11 4.1 1.2	2002/7004740 99-109-295
						ears	28	0.23	RLA 2619.02V
						foliage	28	0.23	
France (S), 1999 Espas (Soissons)	300SC (09957)	2	0.2	400	BBCH 65–69	plant	–0 0 13	0.13 2.9 1.1	2002/7004740 99-109-296
						ears	28	0.13	RLA 2619.02V
						foliage	28	1.1	
France (S), 1999 Le Thor (Manital)	300SC (09957)	2	0.2	400	BBCH 75–77	plant	–0 0 13	< 0.1 3.7 0.96	2002/7004740 99-109-631
						ears	28	0.19	RLA 2619.02V
						foliage	28	1.11	

WHEAT FORAGE Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
France (S), 1999 Orange (Grenat)	300SC (09957)	2	0.2	400	BBCH 75–77	plant	–0 0 13	0.1 3.4 (c=0.1) 1.3 (c=0.14)	2002/7004740 99-109-632 RLA 2619.02V
						ears foliage	28 28	0.18 1.9	
France (S), 2000 Averon-Bergelle (Soissons)	300SC (10358)	2	0.16	350	BBCH 75	plant	–0 +0 14	< 0.1 <u>3.3</u> 0.56	2001/7001676 00-923-292 RLA 2619.02V
						ears foliage	28 28	0.18 0.7	
France (S), 2005 Rhone-Alpes (Caphorn)	75SE	2	0.15	200	BBCH 69	foliage ear	0 0	3.6 1.7	2005/7004267 FBD/33/05 Method 535/0
	100SC	2	0.15	200	BBCH 69	foliage ear	0 0	<u>4.8</u> 2.0	
	300SE	2	0.15	200	BBCH 69	foliage ear	0 0	3.9 2.0	
Spain, 2003 Salteras (Vitromax) spring wheat	100EC	2	0.15	300	BBCH 69	plant	0	<u>3.8</u>	2004/1010542 ALO/03/03 Method 535/0
						ear foliage	29 29	0.45 2.0	
	300SC	2	0.15	300	BBCH 69	plant ear foliage	0 29 29	3.6 0.19 3.5	
France (S), 2003 Ausonne (Nefer)	100EC	2	0.15	300	BBCH 73	plant	0	2.3	2004/1010542 FTL/03/03 Method 535/0
						ear foliage	29 29	0.06 1.8	
	300SC	2	0.15	300	BBCH 73	plant ear foliage	0 29 29	<u>4.3</u> 0.28 5.6	

Values in (brackets) are residues of the CL 3000402 metabolite found above LOQ

Foliage = plants without ears. Also described as 'culm' in some studies

^a Samples may have been transposed

Barley forage

In some of the above European trials on winter and spring barley, whole plants (without roots) were sampled just after the last application and again 28–35 DALT, with these later samples being separated into ears and the rest-of- the-plant (‘foliage’). In the trials conducted before 2004, samples were analysed for metrafenone and the metabolites CL 3000402, CL 434223 and CL 376991 using Method RLA 12619 and in the later trials, Method 535/3 was used to measure only the parent compound. All samples were frozen within 24 hours and stored at or below –18 °C for up to 25 months before analysis. Average procedural recoveries of metrafenone from samples fortified with 0.01–20 mg/kg ranged from 71% to 108% and the LOQs were 0.1 mg/kg.

Table 59 Residues in barley forage from supervised trials in Europe involving 1–3 foliar applications of metrafenone

BARLEY FORAGE Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
GAP: Poland		2	0.15				35		
Germany, 2005 Gerolsheim (Scarlett)	75SE	2	0.15	200	BBCH 69	plant	0	4.4	2005/7004267 DU4/11/05 Method 535/0
	100SC	2	0.15	200	BBCH 69	plant	0	4.3	
	300SE	2	0.15	200	BBCH 69	plant	0	<u>5.0</u>	
Sweden, 2005 Bjarred (Prestige)	75SE	2	0.15	200 200	BBCH 69	foliage	0 29	3.8 0.79	2005/7004267 HUS/07/05 Method 535/0
						ear	0 29	2.5 0.22	
	100SC	2	0.15	200 200	BBCH 69	foliage ear	0 0	<u>5.8</u> 2.7	
	300SE	2	0.15	200 200	BBCH 69	foliage	0 29	3.5 0.76	
						ear	0 29	0.73 0.12	
UK, 2005 Bicester (Pearl)	75SE	2	0.15	200 200	BBCH 73	foliage ear	0 0	2.3 2.0	2005/7004267 OAT/16/05 Method 535/0
	100SC	2	0.15	200 200	BBCH 73	foliage ear	0 0	<u>2.5</u> 2.0	
	300SE	2	0.15	200 200	BBCH 73	foliage ear	0 0	2.0 1.3	
France (N), 2002 Alsace (Astoria) spring barley	300SC	2	0.15	300	BBCH 56	plant	0	<u>3.4</u>	2003/1001354 FAN/02/02
						foliage ear	34 34	< 0.01 < 0.01	Method 993/0 RLA 12619.03V
	300SC	1	0.15	300	BBCH 56	plant	0	2.3	
						foliage ear	34 34	< 0.01 < 0.01	
UK, 2002 Oxfordshire (Siberia)	300SC	2	0.15	300	BBCH 59	plant	0	<u>1.8</u>	2003/1001354 OAT/05/02
						foliage ear	35 35	0.24 0.03	Method 993/0 RLA 12619.03V

BARLEY FORAGE Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
	300SC	1	0.15	300	BBCH 59	plant	0	2.5	
						foliage	35	0.28	
						ear	35	0.05	
Germany, 2003 Lentzke (Cadesse)	100EC	2	0.15	300	BBCH 69	plant	0	<u>3.1</u>	2004/1010542 ACK/04/03
						ear	28	0.03	
						foliage	28	0.08	Method 535/0
	300SC	2	0.15	300	BBCH 69	plant	0	2.0	
						ear	28	0.11	
						foliage	28	0.84	
France (N), 2003 Seebach (Astoria)	100EC	2	0.15	300	BBCH 73	plant	0	2.8	2004/1010542 FAN/05/03
						ear	28	0.36	
spring barley						foliage	28	1.5	Method 535/0
	300SC	2	0.15	300	BBCH 73	plant	0	<u>3.8</u>	
						ear	28	0.35	
						foliage	28	3.3	
Germany, 1999 Euskirchen- Oberwichterich (Uschi)	300SC	3	0.2	400	BBCH 65	plant	-0 0 14	1.5 5.1 1.7	2001/7001659 99-111-01
						foliage	28	2.3	RLA 12619.02V
						ear	28	0.51	
Germany, 1999 Gemarkung (Duet)	300SC	2	0.2	390	BBCH 61	plant	-0 0 14	< 0.1 <u>4.6</u> 0.83	2001/7001659 99-111-02
						ear	28	0.29	RLA 12619.02V
						foliage	28	0.49	
Germany, 1999 Ramsen (Angora)	300SC	2	0.2	300	BBCH 69	plant	-0 0 14	0.62 6.8 2.0	2001/7001659 99-111-03
						ear	28	1.4	RLA 12619.02V
						foliage	28	1.9	
UK, 1999 Newton (Intro)	300SC	2	0.2	300	BBCH 61	plant	-0 0 15	0.12 4.0 0.37	2002/7004681 99-110-01
						ear	27 35	0.26 0.49	RLA 12619.02V
						foliage	43 50	0.8 0.1	
France (N) 2000 Mousseaux (Majestic)	300SC	2	0.15	370	BBCH 77	plant	-0 0 14	0.14 <u>2.3</u> 1.5	2002/7004922 00/840/356
						ear	28	0.87	RLA 12619.02V
						foliage	28	3.1 (0.25)	

BARLEY FORAGE Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
France (S), 2005 Genissieux (Orelie)	75SE	2	0.15	200 200	BBCH 69	foliage	0	4.2	2005/7004267 FBD/34/05 Method 535/0
						ear	0	4.4	
						foliage ear	29 29	0.39 0.02	
	100SC	2	0.15	200 200	BBCH 69	foliage ear	0 0	<u>4.6</u> 4.8	
	300SE	2	0.15	200 200	BBCH 69	foliage ear	0 0	3.0 3.8	
						foliage ear	29 29	1.4 0.03	
France (S), 2003 Rhône-Alpes (Orelie)	100EC	2	0.15	300	BBCH 83	plant	0	2.7	2004/1010542 FBD/02/03 Method 535/0
						ear	29	0.09	
						foliage	29	0.78	
	300SC	2	0.15	300	BBCH 83	plant	0	<u>3.7</u>	
						ear	29	0.37	
						foliage	29	3.4	
Italy, 2003 Pozzolo (Prosa) spring barley	100EC	2	0.15	300	BBCH 55	plant	0	<u>5.9</u>	2004/1010542 ITA/03/03 Method 535/0
						ear	28	0.05	
						foliage	28	0.94	
	300SC	2	0.15	300	BBCH 55	plant	0	3.6	
						ear	28	0.25	
						foliage	28	2.7	
France (S), 1999 Margouet-Meynes (Sunrise)	300SC	2	0.2	400	BBCH 60– 61	plant	–0 0 13	0.1 4.1 0.86	2001/7000488 99-112-297 RLA 12619.02V
						foliage	26	< 0.1	
						ear	26	0.22	
France (S), 1999 Espas (Systel)	300SC	2	0.2	400	BBCH 65– 69	plant	–0 0 13	< 0.1 <u>4.6</u> 0.53	2001/7000488 99-112-298 RLA 12619.02V
						foliage	26	0.63	
						ear	26	0.13	
France (S), 1999 Courthezon (Baraka)	300SC	2	0.2	400	BBCH 75– 77	plant	–0 0 14	0.29 (0.16) 2.7 0.9	2001/7000488 99-112-633 RLA 12619.02V
						foliage	28	3.7	
						ear	28	0.37	
France (S), 1999 Althen des Paluds (Baraka)	300SC	2	0.2	400	BBCH 75– 77	plant	–0 0 14	< 0.1 1.6 0.88	2001/7000488 99-112-634 RLA 12619.02V
						foliage	28	1.6 (0.13)	
						ear	28	0.35	

BARLEY FORAGE Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
France (S), 2000 Bedarrides (Baraka)	300SC	1+ 1	0.14 0.15	390 400	BBCH 77– 83	plant	–0	< 0.1	2002/7004744 00-841-647 RLA 12619.02V
							0	<u>2.5</u>	
							14	0.82	
						foliage ear	28 28	0.61 0.41	

Values in (brackets) are residues of the CL 3000402 metabolite found above LOQ

Foliage = plants without ears. Also described as 'culm' in some studies

Wheat hay and straw

In the above European trials on winter and spring wheat, samples of straw (min 0.5 kg) were taken at maturity, and in many cases, whole plants (without roots) were sampled 0–14 days after the last application and again 28–35 DALY, with these later samples being separated into ears and the rest-of-the-plant ('foliage').

Frozen within 24 hours and stored at or below –18 °C for up to 29 months before analysis. In the trials conducted before 2004, samples were analysed for metrafenone and the metabolites CL 3000402, CL 434223 and CL 376991 using Method RLA 12619 and in the later trials, Method 535/3 was used to measure only the parent compound.

Average procedural recoveries of metrafenone from samples fortified with 0.01–20 mg/kg ranged from 71% to 104% and the LOQs were 0.1 mg/kg.

Table 60 Residues in wheat fodder from supervised trials in Europe involving 1–3 foliar applications of metrafenone

WHEAT FODDER Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
GAP: Poland		2	0.15				35		
Netherlands, 2000 Biddinghuizen (Tremie)	300SC (09957)	2	0.16	310	BBCH 76	straw	34	<u>0.98</u>	2001/7000487 00-770-01 RLA 12619.02V
	300SC (10358)	2	0.15	310	BBCH 76	straw	33	0.61	
France (N), 2000 Fains (Charger)	300SC (09957)	2	0.14	330	BBCH 77	straw	33	<u>2.3</u> (0.12)	2001/7001657 00-831-346 RLA 12619.02V
	300SC (10358)	2	0.14	330	BBCH 77	straw	33	1.8 (0.11)	
UK, 2000 Burton-upon-Trent (Aardvark)	300SC (09957)	2	0.15	200	BBCH 79–83	straw	41	<u>2.0</u> (0.16)	2001/7001658 00-832-01 RLA 12619.02V
	300SC (10358)	2	0.15	200	BBCH 79–83	straw	41	1.7 (0.18)	
Germany, 2000 Schwabenheim (Monopol)	300SC (09957)	2	0.15	300	BBCH 75	straw	35	0.4	2002/7004672 00-922-01 RLA 12619.02V

WHEAT FODDER Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
	300SC (10358)	2	0.15	300	BBCH 75	straw	35	<u>0.67</u>	
Germany, 1999 Zulpich-Mulheim (Bandit)	300SC (09957)	3	0.2	400	BBCH 65	straw	35 41	0.11 0.11	2001/7001675 99-106-01 RLA 12619.02V
Germany, 2000 Haimhausen (Tambor)	300SC (09957)	2	0.2	390	BBCH 69	straw	35 41	< 0.1 < 0.1	2001/7001675 99-106-02 RLA 12619.02V
UK, 1999 Newton (Consort)	300SC (09957)	2	0.2	300	BBCH 59–61	straw	41 49	0.59 0.58	2002/7004680 99-107-01 RLA 12619.02V
Netherlands, 1999 Biddinghuizen (Vivant)	300SC (09957)	2	0.2	270	BBCH 75	straw	35 41	1.1 1.9	2002/7004745 99-108-01 RLA 12619.02V
France (N), 2000 Le Plessis Hebert (Isengrain)	300SC (10358)	2	0.15	370	BBCH 73	straw	35	<u>1.4</u>	2001/7001660 00-834-347 RLA 12619.02V
Denmark, 2005 Middelfart (Kris)	75SE	2	0.15	200	BBCH 69	straw	35 42	1.0 1.2	2005/7004267 ALB/11/05 Method 535/0
	100SC	2	0.15	200	BBCH 69	straw	35 42	1.6 0.86	
	300SE	2	0.15	200	BBCH 69	straw	35 42	<u>3.1</u> 2.8	
Germany, 2005 Wurttemberg (Isengrain)	75SE	2	0.15	200	BBCH 69	straw	27 34 41	0.88 0.68 0.7	2005/7004267 DU2/11/05 Method 535/0
	100SC	2	0.15	200	BBCH 69	straw	27 34 41	0.76 0.71 0.76	
	300SE	2	0.15	200	BBCH 69	straw	27 34 41	2.3 <u>1.8</u> 1.4	
France (N), 2005 Villeveque (Royssac)	75SE	2	0.15	200	BBCH 69	straw	35 42	0.9 1.3	2005/7004267 FBM/11/05 Method 535/0
	100SC	2	0.15	200	BBCH 69	straw	35 42	0.81 1.2	
	300SE	2	0.15	200	BBCH 69	straw	35 42	3.0 <u>3.5</u>	
UK, 2003 Bicester (Malacca)	100EC	2	0.15	300	BBCH 83	straw	34 41	2.6 3.4	2004/1010542 OAT/02/03
	300SC	2	0.15	300	BBCH 83	straw	34 41	<u>3.6</u> 2.9	Method 535/0

WHEAT FODDER Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
Denmark, 2002 Fuenen (Vinjett)	300SC	2	0.15	300	BBCH 51	straw	59	0.11	2003/1001354 ALB/01/02 Method 993/0 RLA 12619.03V
		1	0.15	300	BBCH 51	straw	59	0.1	
Germany, 2002 Baden-Wuerttemberg (Transit)	300SC	2	0.15	300	BBCH 49	straw	58	0.23	2003/1001354 DU2/03/02 Method 993/0 RLA 12619.03V
	300SC	1	0.15	300	BBCH 49	straw	58	0.14	
Denmark, 2003 Fuenen (Triso) spring wheat	100EC	2	0.15	300	BBCH 77	straw	35 41	0.49 0.47	2004/1010542 ALB/01/03 Method 535/0
	300SC	2	0.15	300	BBCH 77	straw	35 41	<u>3.6</u> 2.4	
France S), 2000 Marguet-Meymes (Aztec)	300SC (09957)	2	0.16 0.145	370 350	BBCH 77–83	straw	29	<u>1.3</u> [0.11]	2001/7001656 00-833-290 RLA 12619.02V
	300SC (10358)	2	0.15 0.14	350 340	BBCH 77–83	straw	29	1.1	
France (S), 2000 Mormes (Sideral)	300SC (09957)	2	0.15 0.14	360 330	BBCH 77–83	straw	23	1.6	2001/7001656 00-833-291 RLA 12619.02V
	300SC (10358)		0.15 0.14	345 338	BBCH 77–83	straw	23	1.1	
France (S), 2000 Pernes les Fontaines (Florence Aurore)	300SC (09957)	2	0.15	400	BBCH 83	straw	34	<u>1.1</u>	2001/7001656 00-833-643 RLA 12619.02V
	300SC (10358)		0.15	400	BBCH 83	straw	34	1.1	
France (S), 2000 Le Thor (Manital)	300SC (09957)	2	0.15	400	BBCH 83	straw	33	<u>2.1</u>	2001/7001656 00-833-644 RLA 12619.02V
	300SC (10358)		0.15 0.16	400 420	BBCH 83	straw	33	1.7	
France (S), 1999 Averon Bergelle (Soissons Real)	300SC (09957)	2	0.2	400	BBCH 65–69	straw	35 42	0.89 0.88	2002/7004740 99-109-295 RLA 2619.02V
France (S), 1999 Espas (Soissons)	300SC (09957)	2	0.2	400	BBCH 65–69	straw	35 42	1.1 1.0	2002/7004740 99-109-296 RLA 2619.02V
France (S), 1999 Le Thor (Manital)	300SC (09957)	2	0.2	400	BBCH 75–77	straw	35 42	0.8 1.1	2002/7004740 99-109-631 RLA 2619.02V

WHEAT FODDER Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
France (S), 1999 Orange (Grenat)	300SC (09957)	2	0.2	400	BBCH 75–77	straw	35 42	1.7 1.6	2002/7004740 99-109-632 RLA 2619.02V
France (S), 2000 Averon-Bergelle (Soissons)	300SC (10358)	2	0.16	350	BBCH 75	straw	33	<u>0.67</u>	2001/7001676 00-923-292 RLA 2619.02V
France (S), 2000 Ausonne (Courtaud)	300SC	2	0.14 0.15	280 290	BBCH 75	straw	35	<u>1.6</u> (0.17) [0.11] {0.07}	2002/7004890 FTL/32/01 RLA 12619.02V
France (S), 2005 Rhone-Alpes (Caphorn)	75SE	2	0.15	200	BBCH 69	straw	28 35 42	3.2 2.1 1.6	2005/7004267 FBD/33/05 Method 535/0
	100SC	2	0.15	200	BBCH 69	straw	28 35 42	4.6 2.6 1.5	
	300SE	2	0.15	200	BBCH 69	straw	28 35 42	4.8 <u>6.7</u> 3.8	
Spain, 2003 Salteras (Vitromax) spring wheat	100EC	2	0.15	300	BBCH 69	straw	35 42	<u>1.7</u> 1.3	2004/1010542 ALO/03/03 Method 535/0
	300SC	2	0.15	300	BBCH 69	straw	35 42	0.96 0.99	
France (S), 2003 Ausonne (Nefer)	100EC	2	0.15	300	BBCH 73	straw	35 42	0.95 1.1	2004/1010542 FTL/03/03 Method 535/0
	300SC	2	0.15	300	BBCH 73	straw	35 42	2.2 <u>3.1</u>	

Values in (brackets) are residues of the CL 3000402 metabolite found above LOQ

Values in [brackets] are residues of the CL 434223 metabolite found above LOQ

Values in {brackets} are residues of the CL376991 metabolite found above LOQ

Foliage = plants without ears. Also described as 'culm' in some studies

Barley hay and straw

In the above European trials on winter and spring barley, samples of straw (min 0.5 kg) were taken at maturity, frozen within 24 hours and stored at or below –18 °C for up to 25 months before analysis. In the trials conducted before 2004, samples were analysed for metrafenone and the metabolites CL 3000402, CL 434223 and CL 376991 using Method RLA 12619 and in the later trials, Method 535/3 was used to measure only the parent compound. Average procedural recoveries of metrafenone from samples fortified with 0.01–20 mg/kg ranged from 71% to 108% and the LOQs were 0.1 mg/kg.

Table 61 Residues in barley fodder from supervised trials in Europe involving 1–3 foliar applications of metrafenone

BARLEY FODDER Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
GAP: Poland		2	0.15				35		
Germany, 2005 Gerolsheim (Scarlett)	75SE	2	0.15	200	BBCH 69	straw	29 36 42	0.44 <u>0.77</u> 0.67	2005/7004267 DU4/11/05 Method 535/0
	100SC	2	0.15	200	BBCH 69	straw	29 36 42	0.73 <u>0.86</u> 0.54	
	300SE	2	0.15	200	BBCH 69	straw	29 36 42	3.3 <u>3.6</u> 2.9	
Sweden, 2005 Bjarred (Prestige)	75SE	2	0.15	200 200	BBCH 69	straw	36 43	<u>1.2</u> 1.4	2005/7004267 HUS/07/05 Method 535/0
	100SC	2	0.15	200 200	BBCH 69	straw	29 36 43	<u>0.3</u> 1.8 1.2	
	300SE	2	0.15	200 200	BBCH 69	straw	36 43	0.71 0.85	
UK, 2005 Bicester (Pearl)	75SE	2	0.15	200 200	BBCH 73	straw	29 36 42	0.38 1.1 1.2	2005/7004267 OAT/16/05 Method 535/0
	100SC	2	0.15	200 200	BBCH 73	straw	28 35 42	0.69 1.0 1.0	
	300SE	2	0.15	200 200	BBCH 73	straw	28 35 42	1.2 <u>1.3</u> 1.2	
France (N), 2002 Alsace (Astoria) spring barley	300SC	2	0.15	300	BBCH 56	straw	39	<u>≤ 0.01</u>	2003/1001354 FAN/02/02 Method 993/0 RLA 12619.03V
	300SC	1	0.15	300	BBCH 56	straw	39	< 0.01	
UK, 2002 Oxfordshire (Siberia)	300SC	2	0.15	300	BBCH 59	straw	39	0.24	2003/1001354 OAT/05/02 Method 993/0 RLA 12619.03V
	300SC	1	0.15	300	BBCH 59	straw	39	0.26	

BARLEY FODDER Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
Germany, 2003 Lentzke (Cadesse)	100EC	2	0.15	300	BBCH 69	straw	35 42	0.11 0.07	2004/1010542 ACK/04/03 Method 535/0
	300SC	2	0.15	300	BBCH 69	straw	35 42	0.95 0.83	
France (N), 2003 Seebach (Astoria) spring barley	100EC	2	0.15	300	BBCH 73	straw	36 42	1.1 1.2	2004/1010542 FAN/05/03 Method 535/0
	300SC	2	0.15	300	BBCH 73	straw	36 42	3.9 3.1	
Germany, 1999 Euskirchen- Oberwichterich (Uschi)	300SC	3	0.2	400	BBCH 65	straw	35 44	0.76 1.6	2001/7001659 99-111-01 RLA 12619.02V
Germany, 1999 Gemarkung (Duet)	300SC	2	0.2	390	BBCH 61	straw	35 42	0.39 0.64	2001/7001659 99-111-02 RLA 12619.02V
Germany, 1999 Ramsen (Angora)	300SC	2	0.2	300	BBCH 69	straw	36 44	01.7 (0.1) 0.92	2001/7001659 99-111-03 RLA 12619.02V
France (N), 2000 Bonnieres Sur Seine (Esterel)	300SC (09957)	1+ 1	0.13 0.14	310 350	BBCH 77	straw	36	1.1	2002/7004445 00/835/355 RLA 12619.02V
	300SC (10358)	2	0.14	340	BBCH 77	straw	36	1.2	
Germany, 2000 Obersteinach (Hanna)	300SC (09957)	1+ 1	0.15 0.14	300 290	BBCH 77	straw	42	1.1	2002/7004463 00/837/01 RLA 12619.02V
	300SC (10358)	2	0.15	300	BBCH 77	straw	42	0.78	
UK 2000 Lichfield (Jewel)	300SC (09957)	2	0.15	200	BBCH 77– 79	straw	35	1.1 (0.11)	2002/7004529 00/836/01 RLA 12619.02V
	300SC (10358)	2	0.15	200	BBCH 77– 79	straw	35	1.3 (0.13)	
UK, 2000 Bradwall (Jewel)	300SC (09957)	2	0.15	200	BBCH 75– 77	straw	35	1.1	2002/7004529 00/836/02 RLA 12619.02V
	300SC (10358)	2	0.15	200	BBCH 75– 77	straw	35	0.83	

BARLEY FODDER Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
UK, 1999 Newton (Intro)	300SC	2	0.2	300	BBCH 61	plant straw	43 50	0.41 0.54 (0.4)	2002/7004681 99-110-01 RLA 12619.02V
France (N) 2000 Mousseaux (Majestic)	300SC	2	0.15	370	BBCH 77	straw	-36	2.0 (0.14)	2002/7004922 00/840/356 RLA 12619.02V
France (S), 2000 LaGardelle Sur Leze (Nevada)	300 SC	2	0.15 0.14	300 280	BBCH 73	straw	35	2.1 (0.13) [0.04] {0.07}	2002/7004890 FTL/31/01 RLA 12619.02V
France (S), 2005 Genissieux (Orelie)	75SE	2	0.15	200 200	BBCH 69	straw	35 41	0.26 0.21	2005/7004267 FBD/34/05 Method 535/0
	100SC	2	0.15	200 200	BBCH 69	straw	29 35 41	0.02 0.51 0.32	
	300SE	2	0.15	200 200	BBCH 69	straw	35 41	1.1 0.31	
France (S), 2003 Rhone-Alpes (Orelie)	100EC	2	0.15	300	BBCH 83	plant straw	35 42	0.44 0.46	2004/1010542 FBD/02/03 Method 535/0
	300SC	2	0.15	300	BBCH 83	straw	35 42	1.3 1.5	
Italy, 2003 Pozzolo (Prosa) spring barley	100EC	2	0.15	300	BBCH 55	straw	35 42	0.6 0.75	2004/1010542 ITA/03/03 Method 535/0
	300SC	2	0.15	300	BBCH 55	straw	35 42	1.9 1.6	
France (S), 1999 Margouet-Meynes (Sunrise)	300SC	2	0.2	400	BBCH 60- 61	straw	34 40	2.8 (0.12) 4.0	2001/7000488 99-112-297 RLA 12619.02V
France (S), 1999 Espas (Systel)	300SC	2	0.2	400	BBCH 65- 69	straw	34 40	0.68 0.9	2001/7000488 99-112-298 RLA 12619.02V
France (S), 1999 Courthezon (Baraka)	300SC	2	0.2	400	BBCH 75- 77	straw	35 42	2.5 (0.12) 4.3 (0.3)	2001/7000488 99-112-633 RLA 12619.02V
France (S), 1999 Althen des Paluds (Baraka)	300SC	2	0.2	400	BBCH 75- 77	straw	35 42	1.2 (0.13) 0.97 (0.12)	2001/7000488 99-112-634 RLA 12619.02V

BARLEY FODDER Country, year Location (Variety)	Application					Matrix	DAT	Residues (mg/kg)	Reference & Comments
	form	no	kg ai/ha	water (L/ha)	GS last application			metrafenone	
France (S), 2000 Averon-Bergelle (Plantine)	300SC (09957)	2	0.16	240	BBCH 77– 83	straw	34	1.7 (0.12)	2002/7004525 00/839/294 RLA 12619.02V
	300SC (10358)	2	0.16	240	BBCH 77– 83	straw	34	1.6 (0.13)	
France (S), 2000 Saumane (Baraka)	300SC (09957)	2	0.15	410	BBCH 77– 83	straw	36	1.9	2002/7004525 00/839/646 RLA 12619.02V
	300SC (10358)	1+ 1	0.15 0.16	410 420	BBCH 77– 83	straw	36	1.3	
France (S), 2000 Althen Des Paluds (Baraka)	300SC (09957)	2	0.15	400	BBCH 83	straw	34	1.0	2002/7004525 00/839/645 RLA 12619.02V
	300SC (10358)	1+ 1	0.16 0.15	420 410	BBCH 83	straw	34	0.96	
France (S), 2000 Bedarrides (Baraka)	300SC	1+ 1	0.14 0.15	390 400	BBCH 77– 83	straw	35	0.41	2002/7004744 00-841-647 RLA 12619.02V

Values in (brackets) are residues of the CL 3000402 metabolite found above LOQ

Values in [brackets] are residues of the CL 434223 metabolite found above LOQ

Values in {brackets} are residues of the CL376991 metabolite found above LOQ

Foliage = plants without ears. Also described as 'culm' in some studies

FATE OF RESIDUES IN STORAGE AND PROCESSING

High temperature hydrolysis

In a study reported by AN, 2000 [Ref: 2000/7000137], the high-temperature hydrolysis of [bromophenyl-6-¹⁴C]-metrafenone in buffered solutions of pH 4, 5, and 6 was investigated to simulate representative processing conditions: pasteurization at 90 °C for 20 minutes in pH 4 solution; baking, brewing, or boiling at 100 °C for 60 minutes in pH 5 solution; and sterilization at 120 °C for 20 minutes in pH 6 solution.

Solutions of buffered [bromophenyl-6-¹⁴C]-metrafenone were incubated in the dark at 90, 100, and 120 °C at pH 4, 5, and 6, respectively, and samples were analysed after 20 min. (pH 4 and 6) and 1 h (pH 5) by LSC and HPLC. Examination of the radio-chromatograms showed only one major peak in all samples, identified as the parent compound.

Recoveries of radioactivity at 0-time were 91–92% of the applied radioactivity and after incubation were 96–102% of applied dose. Metrafenone made up about 91% of the radioactivity at 0-time and 95–101% of the applied dose after incubation. The relatively low pre-incubation recovery was attributed to retention on the glass tubes. The results indicate that metrafenone is hydrolytically stable under the simulated processing conditions of pasteurization, baking, brewing, boiling and sterilization.

Table 62 Distribution of radioactivity for [bromophenyl-6-¹⁴C]-metrafenone in buffer solutions—high temperature hydrolysis (% radioactivity)

Component	pH 4		pH 5		pH 6	
	Control	90°C	Control	100°C	Control	120°C
Metrafenone (parent)	90.75	100.5	91.24	101.37	90.9	94.8
Others	0.69	1.01	0.81	0.95	1.25	1.42
Total % recovery	91.44	101.15	92.05	102.32	92.15	96.22

Processing

Apples

An apple processing study was reported by Carringer, 2013 [Ref 2012/7004393]. In two field trials in USA, involving three foliar applications of 1.68 kg ai/ha metrafenone (SC formulation), applied at 6–7 day intervals in 750–1000 L water/ha, with added surfactant, bulk samples (36 kg) were taken 6–7 days after the last application and cool-stored for up to 4 days before processing using simulated commercial practices into juice, wet pomace, apple sauce, canned apples, and dried apples).

The bulk apples for processing were quartered, sliced and a portion of these apple slices were set aside for raw juice generation. The remaining portion was ground in a food strainer/sauce maker, producing puree and wet pomace. Apple sauce was prepared by evaporating the puree at 82–93 °C in a kettle. Apple slices for juice were processed through a juice processor to produce juice and the resulting wet pomace was combined with the wet pomace generated during puree production. Canned apples were prepared from whole apples by peeling, slicing and coring, with the sliced fruit heated with water to about 70 °F for approximately 45 minutes and stirred to produce slices in syrup. For dried fruit production, whole apples were peeled, cut into 0.3 cm slices, cored and placed one layer deep on dehydrator trays and dried at 65–70 °C for about 24 hours to achieve a moisture content of approximately 10%.

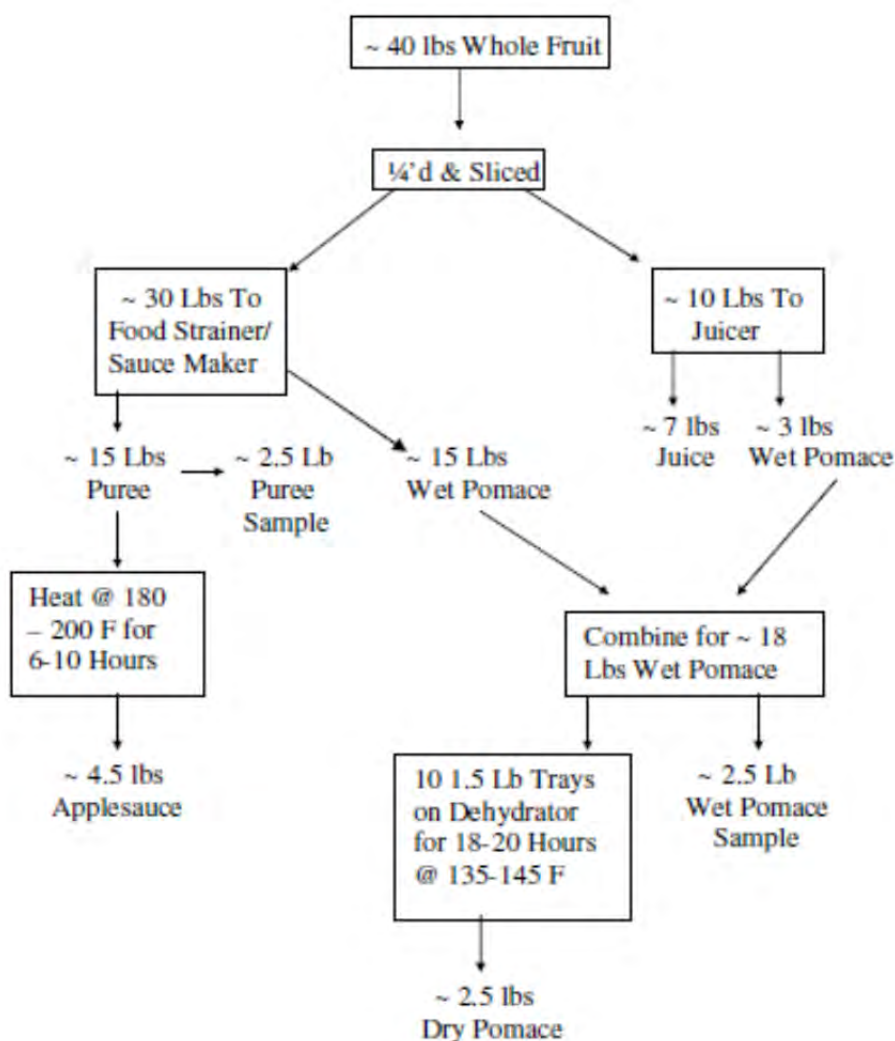


Figure 6 Processing flowchart for apple sauce, juice, and wet pomace

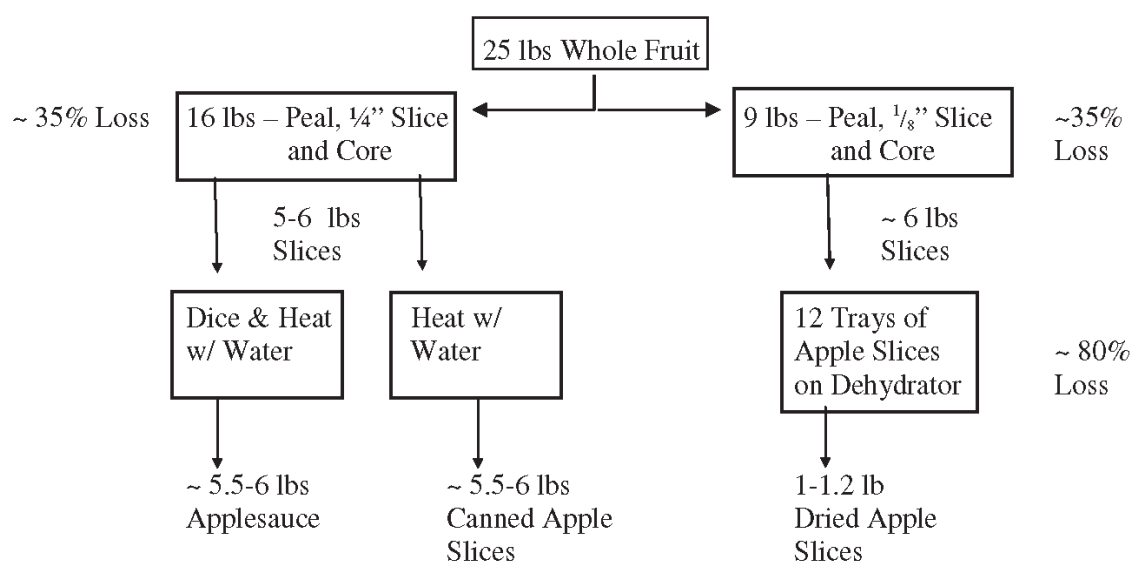


Figure 7 Processing flowchart for canned and dried apple

Representative samples of whole apples (≥ 2 kg), dried apples (approximately 0.5 kg), juice (2 litres) and other processed fractions (≥ 1 kg) were stored at ≤ -16 °C for up to 22 months before analysis for metrafenone using the LC-MS/MS QuEChERS method. The reported LOQ in all matrices was 0.01 mg/kg and average concurrent recoveries were 92–106% in samples spiked with 0.01 to 10.0 mg/kg.

Table 63 Residues in fresh and processed apples from supervised trials in North America involving three foliar applications of metrafenone (SC formulation)

APPLE Study ID	Commodity	Metrafenone (mg/kg)	Processing Factor ^a
R080753 Alton, NY, 2010 3 × 1.7 kg ai/ha PHI: 7 days	Whole Fresh Fruit	1.46	—
	Juice (raw)	0.28	0.189
	Wet Pomace	1.88	1.29
	Apple Sauce	7.01	4.80
	Canned Apples	0.2	0.140
	Dried Apples	1.1	0.719
R080754 Weiser, ID, 2010 3 × 1.7 kg ai/ha PHI: 6 days	Whole Fresh Fruit	2.17	—
	Juice (raw)	0.489	0.225
	Wet Pomace	2.37	1.09
	Apple Sauce	8.94	4.12
	Canned Apples	0.226	0.104
	Dried Apples	0.851	0.392

^a The processing factor was calculated by dividing the residue in the apple processed commodity by the residue in the apple RAC sample.

Grapes

One grape processing study conducted in USA was reported by Jordan & Kasiri, 2006 [Ref: 2006/7007012]. In one field trial in the USA, involving six foliar applications of 0.34 kg ai/ha metrafenone (SC formulation) in 1280 Litres water/ha, applied at 12–15 day intervals from BBCH 75 (berries pea-sized), bulk grape samples (170 kg) were taken immediately after the last application and either cool-stored for up to 3 days before processing into juice, red wine, white wine or sun-dried in the field for 28 days before washing.

Grapes were processed into grape juice by crushing and after removing the stems, the pulp was heated to 52–57 °C for 10 minutes and then to 60–66 °C for a further 10 minutes before pressing to separate the juice and pulp. The collected juice was filtered through a U.S. #40 screen to remove course solids and samples were then frozen for analysis.

For wine, the grapes were de-stemmed and crushed, the juice/pulp was heated to approximately 60 °C then cooled to approximately 21 °C (red wine only), and allowed to stand for about an hour after the addition of potassium metabisulfite and pectic enzyme before pressing. The must was allowed to rest overnight and after addition of yeast, primary fermentation was continued at room temperature until the specific gravity reached approximately 1.03. The wine was then racked and stored at approximately 13 °C for 1–2 weeks before the sediment was removed. This ‘young wine’ was then treated with gelatine as a fining agent and stored for about 3 months before the lees were removed and the finished wine was vacuum filtered over diatomaceous earth.

Representative samples of grapes and processed fractions were stored at ≤ -17 °C for up to 4 months (6 months for grapes) before analysis for metrafenone using the HPLC-MS/MS Method 535/3. The reported LOQ in all matrices was 0.01 mg/kg and average concurrent recoveries were 80–127% in samples spiked with 0.01 to 20.0 mg/kg.

Table 64 Residues in fresh and processed grapes from supervised trials in North America involving three foliar applications of metrafenone (SC formulation)

GRAPE Study ID	Commodity	Metrafenone (mg/kg)	Processing factor
RCN R05013 Tulare, CA 6 × 0.35 kg ai/ha PHI: 0 days Batch 1	Unwashed grapes	0.27	–
	Juice	0.01	0.04
	Must (white wine)	0.04	0.15
	Wet pomace	0.75	2.78
	Yeast	0.5	1.85
	Young wine (white)	0.02	0.07
	Wine (white)	0.02	0.07
	Unwashed raisins	1.11	4.11
	Washed raisins	0.98	3.63
RCN R05013 Tulare, CA 6 × 0.35 kg ai/ha PHI: 0 days Batch 2	Unwashed grapes	0.31	–
	Juice	0.02	0.06
	Must (red wine)	0.01	0.03
	Wet pomace	1.11	3.58
	Yeast	0.18	0.58
	Young wine (red)	0.01	0.03
	Wine (red)	0.01	0.03
	Unwashed raisins	0.86	2.77
	Washed raisins	1.22	3.94

In a series of studies conducted in Europe and reported by Smalley, 2002 [Refs: 2002/7004451, 2002/7004455, 2002/7004459, 2002/7004460], grapes from field trials involving eight applications of 0.01–0.033 kg ai/hL metrafenone (0.09–0.13 kg ai/ha), applied at either 7 or 14 day intervals, were taken 27–29 days after the last application and processed into red or white wine or sun-dried to produce raisins.

The wine processing steps were generally as described above, with potassium metabisulphite added as a fermentation inhibitor and gelatin used as a fining agent. For the sparkling wine, sugar and yeasts were added to wine prior to bottling and the bottles stored at 5–10 °C for about 9 months (turned regularly during the last month) before uncapping, disgorging and recapping and frozen for subsequent analysis.

Representative samples of grapes and processed fractions were stored at about –20 °C for up to 6 months before analysis for metrafenone using Method RLA 12612V. The reported LOQ in all matrices was 0.05 mg/kg and average concurrent recoveries were 77–102% in samples spiked with 0.05–0.5 mg/kg.

Table 65 Residues in fresh and processed grapes from supervised trials in Europe involving eight foliar applications of metrafenone (SC formulation)

GRAPE Trial reference	Application details	Commodity	Metrafenone (mg/kg)	Processing factor
Ref: 2002/7004451 Trial:00-845-01 VIN-A Italy, 2000 Castelfranco	8 × 0.01 kg ai/hL (0.13 kg ai/ha) 7 day spray interval DAT: 29 days	Grapes	0.3	–
		Must	0.17	0.57
		Young wine (red)	< 0.05	< 0.17
		Wine (red)	< 0.05	< 0.17
Ref: 2002/7004451 Trial:00-845-01 VIN-B Italy, 2000 Castelfranco	8 × 0.01 kg ai/hL (0.13 kg ai/ha) 7 day spray interval DAT: 29 days	Grapes	0.24	–
		Must	0.28	1.17
		Young wine (red)	< 0.05	< 0.2
		Wine (red)	< 0.05	< 0.2
Ref: 2002/7004451 Trial: 00-845-02 VIN-A Italy, 2000 Toscanello	8 × 0.01 kg ai/hL (0.12 kg ai/ha) 7 day spray interval DAT: 29 days	Grapes	0.13	–
		Must	0.10	0.77
		Young wine (red)	< 0.05	< 0.38
		Wine (red)	< 0.05	< 0.38

GRAPE Trial reference	Application details	Commodity	Metrafenone (mg/kg)	Processing factor
Ref: 2002/7004451 Trial: 00-845-02 VIN-B Italy, 2000 Toscanello	8 × 0.01 kg ai/hL (0.12 kg ai/ha) 7 day spray interval DAT: 29 days	Grapes Must Young wine (red) Wine (red)	0.07 0.09 < 0.05 < 0.05	– 1.29 < 0.71 < 0.71
Ref: 2002/7004455 Trial: 00-843-441-A France (N), 2000 Oger	8 × 0.02 kg ai/hL (0.1 kg ai/ha) 14 day spray interval DAT: 27 days	Grapes Must Still wine (white) Sparkling wine (white)	0.28 < 0.05 < 0.05 < 0.05	– < 0.18 < 0.18 < 0.18
Ref: 2002/7004455 Trial: 00-843-441-B France (N), 2000 Oger	8 × 0.02 kg ai/hL (0.1 kg ai/ha) 14 day spray interval DAT: 27 days	Grapes Must Still wine (white) Sparkling wine (white)	0.19 0.05 < 0.05 < 0.05	– 0.26 < 0.26 < 0.26
Ref: 2002/7004460 Trial: 00-844-648-A France (S), 2000 Jonquerettes	8 × 0.033 kg ai/hL (0.1 kg ai/ha) 7 day spray interval DAT: 29 days	Grapes Must Young wine (red) Wine (red)	0.26 0.21 < 0.05 < 0.05	– 0.81 < 0.19 < 0.19
Ref: 2002/7004460 Trial: 00-844-648-B France (S), 2000 Jonquerettes	8 × 0.033 kg ai/hL (0.1 kg ai/ha) 7 day spray interval DAT: 29 days	Grapes Must Young wine (red) Wine (red)	0.27 0.21 0.08 < 0.05	– 0.78 0.3 < 0.19
Ref: 2002/7004459 Trial: 00-846-11-A Spain, 2000 Malaga	8 × 0.01 kg ai/hL (0.09 kg ai/ha) 7 day spray interval DAT: 28 days	Grapes Raisins	0.07 < 0.05	– < 0.71
Ref: 2002/7004459 Trial: 00-846-11-B Spain, 2000 Malaga	8 × 0.01 kg ai/hL (0.09 kg ai/ha) 7 day spray interval DAT: 28 days	Grapes Raisins	0.08 < 0.05	– < 0.63

Strawberries

In a study reported by Plier, 2011 [Ref: 2011/1041883], bulk samples (min 10 kg) of strawberries were taken from four field trials conducted in Germany (2 × 0.45 kg ai/200 Litres/ha metrafenone, 7 days apart) 3–4 days after the last application and chilled to about 5–8 °C for up to 3 days before processing into jam, preserved fruit and syrup.

Fruit (without stems and crowns) were washed and processed into jam by cooking with sugar and glucose syrup until a dry matter content of about 64% was achieved and after the addition of citric acid and pectin, heated further until a dry matter content reached about 62%. The jam (pH 2.7–3.3) was then cooled and samples frozen for up to 8 months before analysis.

Washed fruit were also preserved by heating with ascorbic acid, citric acid, sugar and water until boiling and immediately pouring the mixture into jars which were topped up with boiled syrup, sealed and pasteurized for one minute at 90–95 °C. Cooled samples (pH 3–4) were then frozen for up to 8 months before analysis.

The analytical method 535/1 was used to measure residues of metrafenone, with an LOQ of 0.01 mg/kg and concurrent recoveries from samples spiked with 0.01, 0.1 or 1.0 mg/kg ranged from 82–94%, with an average recovery of 89%.

Table 66 Residues in fresh and processed strawberries from supervised trials in Europe involving two foliar applications of metrafenone (SC formulation)

STRAWBERRY Trial reference	Application details	Commodity	Metrafenone (mg/kg)	Processing factor
Ref: 2011/1041883 Trial:L100269 Germany, 2010 Leisnig	2 × 0.45 kg ai/ha (200 L water/ha) 7 day spray interval DAT: 3 days	Strawberries Washed fruit wash water Preserved fruit Jam after cooking Jam before cooking Syrup	0.41 0.166 0.543 0.344 0.087 0.077 0.064	– 0.4 1.3 0.84 0.2 0.19 0.16
Ref: 2011/1041883 Trial:L100270 Germany, 2010 Coswig	2 × 0.45 kg ai/ha (200 L water/ha) 7 day spray interval DAT: 3 days	Strawberries Washed fruit wash water Preserved fruit Jam after cooking Jam before cooking Syrup	0.157 0.078 0.221 0.155 0.038 0.045 0.028	– 0.5 1.4 0.99 0.24 0.29 0.18
Ref: 2011/1041883 Trial:L100271 Germany, 2010 Pillnitz	2 × 0.47 kg ai/ha (210 L water/ha) 7 day spray interval DAT: 4 days	Strawberries Washed fruit wash water Preserved fruit Jam after cooking Jam before cooking Syrup	0.284 0.148 0.28 0.225 0.061 0.066 0.053	– 0.52 0.99 0.79 0.21 0.23 0.19
Ref: 2011/1041883 Trial:L100272 Germany, 2010 Alitzheim	2 × 0.45 kg ai/ha (200 L water/ha) 7 day spray interval DAT: 3 days	Strawberries Washed fruit wash water Preserved fruit Jam after cooking Jam before cooking Syrup	0.195 0.088 0.261 0.222 0.055 0.055 0.03	– 0.45 1.34 1.14 0.28 0.28 0.15

Tomatoes

In a study reported by Plier, 2011 [Ref: 2011/1041884], tomatoes were taken for processing from four field trials conducted in Germany. In these field trials, two applications of 0.6–0.7 kg ai/ha were applied 6–8 days apart using 450–550 Litres spray mix/ha. Bulk samples of 13–21 kg ripe tomatoes were taken 3–4 days after the last application and chilled to about 5–8 °C for 1–12 days before processing into preserve, juice, paste, ketchup and puree.

Tomatoes (without stems) were washed, blanched (1 minute at 75–85 °C), cooled and peeled manually. The peeled tomatoes were placed in jars, topped up with water, sealed and autoclave sterilised (5–12 minutes at 118–125 °C) before cooling and sampling for subsequent analysis.

For juice, puree and ketchup, washed tomatoes were crushed and heated for 30 minutes at 80–87 °C, then pressed and sieved to separate the juice and pomace. Samples of the raw juice were concentrated to achieve a dry matter content of 7–14% (for paste) and 18–24% (for puree). Ketchup was prepared by adding vinegar (0.4%), sugar (42%) and salt (15%) to the paste. The paste, ketchup and puree were pasteurised at 90–95 °C for 1–20 minutes, cooled and samples were stored frozen for up to 6 months before analysis for metrafenone using Method 535/1, with a limit of quantification of 0.01 mg/kg. Concurrent recoveries from samples spiked with 0.01–5.0 mg/kg ranged from 80–109%, with an average recovery of 93%.

Table 67 Residues in fresh and processed tomatoes from supervised trials in Europe involving two foliar applications of metrafenone (SC formulation)

TOMATO Trial reference	Application details	Commodity	Metrafenone (mg/kg)	Processing factor
Ref: 2011/1041884 Trial: L100261 Germany, 2010 Motterwitz	2 × 0.71 kg ai/ha (530 L water/ha) 7 day spray interval DAT: 3 days	Tomatoes Blanched fruit Blanching water Preserved fruit Peeled fruit Wash water Tomato peel Wet pomace Washed fruit Raw juice Ketchup (pasteurised) Tomato paste Tomato puree Preserving stock	0.423 0.535 < 0.01 < 0.01 < 0.01 0.066 3.184 2.034 0.382 0.168 0.177 0.225 0.335 < 0.01	– 1.26 < 0.02 < 0.02 < 0.02 0.16 7.5 4.8 0.9 0.4 0.42 0.53 0.79 < 0.02
Ref: 2011/1041884 Trial: L100262 Germany, 2010 Gerichshain	2 × 0.73 kg ai/ha (540 L water/ha) 7 day spray interval DAT: 3 days	Tomatoes Blanched fruit Blanching water Preserved fruit Peeled fruit Wash water Tomato peel Wet pomace Washed fruit Raw juice Ketchup (pasteurised) Tomato paste Tomato puree Preserving stock	0.775 0.352 0.013 0.015 < 0.01 0.507 4.807 2.567 0.477 0.198 0.327 0.207 0.501 < 0.01	– 0.45 0.02 0.02 < 0.01 0.65 6.2 3.31 0.62 0.26 0.42 0.27 0.65 < 0.01
Ref: 2011/1041884 Trial: L100263 Germany, 2010 Dupow	2 × 0.71–0.61 kg ai/ha (530–450 L water/ha) 6 day spray interval DAT: 4 days	Tomatoes Blanched fruit Blanching water Preserved fruit Peeled fruit Wash water Tomato peel Wet pomace Washed fruit Raw juice Ketchup (pasteurised) Tomato paste Tomato puree Preserving stock	0.583 0.528 0.017 < 0.01 0.031 0.218 4.585 3.592 0.488 0.194 0.219 0.174 0.485 < 0.01	– 0.91 0.03 < 0.02 0.05 0.37 7.9 6.2 0.84 0.33 0.38 0.3 0.84 < 0.02
Ref: 2011/1041884 Trial: L100264 Germany, 2010 Oderberg	2 × 0.69–0.63 kg ai/ha (510–460 L water/ha) 8 day spray interval DAT: 3 days	Tomatoes Blanched fruit Blanching water Preserved fruit Peeled fruit Wash water Tomato peel Wet pomace Washed fruit Raw juice Ketchup (pasteurised) Tomato paste Tomato puree Preserving stock	0.425 0.398 0.012 < 0.01 0.01 0.152 1.588 2.679 0.242 0.149 0.211 0.201 0.478 < 0.01	– 0.94 0.03 < 0.02 0.02 0.36 3.7 6.3 0.57 0.35 0.5 0.47 1.12 < 0.02

Barley

In a study reported by Pollmann, 2002 [Ref: 2002/1004080], samples of summer barley grain were taken for processing from four field trials conducted in Germany. In these field trials, two applications of about 0.5 kg ai/ha were applied 10–15 days apart using about 300 Litres spray mix/ha. Bulk samples of at least 25 kg grain were taken 35 days after the last application and stored at ambient temperature for 2–3 months before processing into pearl barley and beer.

Pearl barley was prepared by mechanical de-awning and cleaning (< 2.5 mm slotted screen) to separate husks and other impurities, conditioning to 14% moisture content and decortications to achieve an abrasion rate of 20–30%. Samples were frozen within 4 hours for subsequent analysis

The malting and brewing process involved steeping the barley (2 × 3 hours at 14 °C), sprouting over 7 days at about 12 °C and kilning (7 hours at 55 °C and 1.5 hours at 85 °C) before the sprouts were separated over a wire mesh and the malt was conditioned for 8–21 days at 12–15 °C before being ground for brewing. Mash was produced using the infusion method and wort was removed during the lautering process. Hops were added to the wort during the 1 hour boiling period and after cooling, yeast was added to initiate fermentation. Primary fermentation proceeded for 7–8 days at 8–14 °C and after bottling, secondary fermentation continued for 21–32 days (8–14 °C) when samples were frozen for subsequent analysis.

Samples were stored frozen (about -18 °C) for up to 7 months before analysis for metrafenone using Method RLA 12619.03V (993/0), with a limit of quantification of 0.01 mg/kg. Concurrent recoveries from samples spiked with 0.01–0.1 mg/kg ranged from 73–93% (except malt sprouts, where the recovery was 47%), with an average recovery of 85%.

Table 68 Residues in barley and processed barley fractions from supervised trials in Europe involving two foliar applications of metrafenone (SC formulation)

BARLEY Trial reference	Application details	Commodity	Metrafenone (mg/kg)	Processing factor
Ref: 2002/1004080 Trial: G01N055R Germany, 2001 Mulsum	2 × 0.47 kg ai/ha (310 L water/ha) 14 day spray interval DAT: 35 days	Stored barley	0.06	–
		Beer	< 0.01	< 0.17
Ref: 2002/1004080 Trial: G01N056R Germany, 2001 Mulsum	2 × 0.45–0.46 kg ai/ha (310 L water/ha) 13 day spray interval DAT: 35 days	Stored barley	0.08	–
		Pearl barley	< 0.01	< 0.13
Ref: 2002/1004080 Trial: G01N056R Germany, 2001 Mulsum	2 × 0.45–0.46 kg ai/ha (310 L water/ha) 13 day spray interval DAT: 35 days	Stored barley	0.03	–
		Beer	< 0.01	< 0.33
Ref: 2002/1004080 Trial: G01N057R Germany, 2001 Kleinsachsenheim	2 × 0.44–0.46 kg ai/ha (300 L water/ha) 10 day spray interval DAT: 35 days	Stored barley	0.05	–
		Pearl barley	< 0.01	< 0.2
Ref: 2002/1004080 Trial: G01N057R Germany, 2001 Kleinsachsenheim	2 × 0.44–0.46 kg ai/ha (300 L water/ha) 10 day spray interval DAT: 35 days	Stored barley	0.1	–
		Malt	0.04	0.4
		Malt sprouts	0.08	0.8
		Brewer's grain	0.03	0.3
		Spent hops	< 0.01	< 0.1
		Brewer's yeast	< 0.01	< 0.1
		Beer	< 0.01	< 0.1
		Stored barley	0.08	–
		Pearl barley	0.01	0.13
		Pearl barley abrasion	0.2	2.5
Ref: 2002/1004080 Trial: G01N058R Germany, 2001 Gemmrigheim	2 × 0.44–0.45 kg ai/ha (310 L water/ha) 10 day spray interval DAT: 35 days	Stored barley	0.08	–
		Beer	< 0.01	< 0.13
		Stored barley	0.09	–
		Pearl barley	0.02	0.22

Wheat

In a study reported by Pollmann, 2002 [Ref: 2002/1006302], samples of summer wheat grain were taken for processing from four field trials conducted in Germany. In these field trials, two applications of about 1.5 kg ai/ha were applied 27 or 38 days apart using about 300 Litres spray mix/ha. Bulk samples of at least 7 kg grain were taken 35 days after the last application and stored frozen for up to 2 months before processing into flour, bran and bread.

After cleaning to remove dust and plant fragments, 6–10 kg samples of wheat grain were conditioned to a moisture content of about 17% and milled by passing through a series of corrugated and smooth rollers (0.5 mm down to 0.01 mm apertures) to obtain flour and bran + adhesive flour, the latter being centrifuged to separate the coarse bran, fine bran and the bran flour. Samples were frozen within 9 hours for subsequent analysis.

A sample of the whole meal flour was kneaded with water, ascorbic acid, yeast, salt, sugar and peanut fat to produce dough, which was allowed to ferment at about 27 °C for 30 minutes before being formed into a loaf and given a further 60 minute fermentation period (32 °C) before baking at 210 °C for 50 minutes. The loaves were then cooled and samples were immediately frozen for subsequent analysis.

Samples were stored frozen (about –18 °C) for up to 7 months before analysis for metrafenone using Method RLA 12619.03V (993/0), with a limit of quantification of 0.01 mg/kg. Concurrent recoveries from samples spiked with 0.01–0.1 mg/kg ranged from 76–104% with an average recovery of 89%.

Table 69 Residues in wheat and processed wheat fractions from supervised trials in Europe involving two foliar applications of metrafenone (SC formulation)

WHEAT Trial reference	Application details	Commodity	Metrafenone (mg/kg)	Processing factor
Ref: 2002/1006302 Trial: G01N051R Germany, 2001 Ladekop	2 × 1.5 kg ai/ha (300 L water/ha) 38 day spray interval DAT: 36 days	Grain ^a	0.28	–
		Wholemeal flour	0.31	1.1
		Flour type 550	0.06	0.21
		Bran flour	0.45	1.6
		Coarse bran	0.08	0.29
		Fine bran	0.97	3.5
		Whole grain bread	0.18	0.64
Ref: 2002/1006302 Trial: G01N052R Germany, 2001 Ladekop	2 × 1.5 kg ai/ha (300 L water/ha) 38 day spray interval DAT: 36 days	Grain ^a	0.14	–
		Wholemeal flour	0.27	1.9
		Flour type 550	0.04	0.29
		Bran flour	0.37	2.6
		Coarse bran	0.06	0.43
		Fine bran	0.69	4.9
		Whole grain bread	0.14	1.0
Ref: 2002/1006302 Trial: G01N053R Germany, 2001 Ludwigsburg-Ossweil	2 × 1.5–1.6 kg ai/ha (300 L water/ha) 27 day spray interval DAT: 35 days	Grain ^b	0.18	–
		Wholemeal flour	0.17	0.94
		Flour type 550	0.03	0.17
		Bran flour	0.24	1.3
		Coarse bran	0.06	0.33
		Fine bran	0.47	2.6
		Whole grain bread	0.11	0.61
Ref: 2002/1006302 Trial: G01N054R Germany, 2001 Affalterbach	2 × 1.4–1.5 kg ai/ha (280–310 L water/ha) 27 day spray interval DAT: 35 days	Grain ^b	0.07	–
		Wholemeal flour	0.12	1.7
		Flour type 550	0.01	0.14
		Bran flour	0.18	2.6
		Coarse bran	0.04	0.57
		Fine bran	0.37	5.3
		Whole grain bread	0.05	0.71

^a Conditioned to approximately 17% by drying

^b Conditioned to about 17% by addition of water

Hops

In two studies reported by Braun, 2011 [Ref: 2011/1041879] and Plier, 2011 [Ref: 2011/1041886], hops from field trials conducted in Germany were taken for drying or processing into beer. In these field trials, two applications of about 0.33–0.35 kg ai/ha were applied 6–8 days apart using about 3300 Litres spray mix/ha.

Samples of green cones were taken 3–4 days after the last application and kiln-dried for about 7–8 hours at approximately 58 °C to produce dried cones. The green and dried cone samples were frozen within 12 hours and stored below –18 °C for subsequent analysis (up to 8 months after sampling). In addition, bulk dried cone samples (min 1.8 kg) were stored at ambient temperatures for about 1 month prior to processing.

Dried cones were milled and added to the separated wort and after boiling for about 90 minutes, the flocs (hops draff) were separated in a whirlpool and after cooling, yeast was added to initiate fermentation. Primary fermentation proceeded for 9–11 days at about 9 °C and secondary (cask) fermentation continued for a further 2 days at 20 °C and under pressure for a further 24 days at 2 °C before being filtered and sampled for subsequent analysis.

Dried cones were also processed into Extracted Hops by dissolving with ethanol using a soxhlet extractor for 3–4 hours, with the miscella being filtered and concentrated twice using a vacuum evaporator (above 50 °C and 0.5–1.0 bar) before cooling and sampling for subsequent analysis.

Samples were stored frozen (about -18 °C) for up to 4 months before analysis for metrafenone using Method 535/3, with a limit of quantification of 0.01 mg/kg. Concurrent recoveries from samples spiked with 0.01–1.0 mg/kg (and 10 mg/kg in extracted hops and 50–100 mg/kg in dried cones) ranged from 73–111% (except in extracted hops, where recovery rates were 65–68%) with an average recovery of 87%.

Table 70 Residues in hops and processed hop fractions from supervised trials in Europe involving two foliar applications of metrafenone (SC formulation)

HOPS Trial reference	Application details	Commodity	Metrafenone (mg/kg)	Processing factor
Ref: 2011/1041879 Trial: L090302 Germany, 2009 Golzen	2 × 0.31–0.34 kg ai/ha (3200–3400 L water/ha) 8 day spray interval DAT: 2 days	Green cones	3.74	–
		Dried cones	21.3	5.7
		Dried cones	17.7	–
		Extracted hops	31.1	1.76
		Beer	< 0.01	< 0.0006
		Brewer's yeast	0.2	0.01
		Hops Draff	4.17	0.24
Ref: 2011/1041879 Trial: L090303 Germany, 2009 Hohenebra	2 × 0.33–0.35 kg ai/ha (3300–3600 L water/ha) 7 day spray interval DAT: 3 days	Green cones	3.4	–
		Dried cones	22.8	6.7
		Dried cones	21.1	–
		Extracted hops	37.9	1.8
		Beer	< 0.01	< 0.0005
		Brewer's yeast	0.24	0.01
		Hops Draff	5.17	0.25
Ref: 2011/1041879 Trial: L090304 Germany, 2009 Kleinbadegast	2 × 0.31–0.34 kg ai/ha (3100–3300 L water/ha) 6 day spray interval DAT: 3 days	Green cones	1.78	–
		Dried cones	13.3	7.5
		Dried cones	18.3	–
		Extracted hops	32.7	1.79
		Beer	< 0.01	< 0.0005
		Brewer's yeast	0.15	0.008
		Hops Draff	4.39	0.24

HOPS Trial reference	Application details	Commodity	Metrafenone (mg/kg)	Processing factor
Ref: 2011/1041879 Trial: L090305 Germany, 2009 Simonshofen	2 × 0.35–0.34 kg ai/ha (3500–3400 L water/ha) 7 day spray interval DAT: 4 days	Green cones Dried cones	3.05 19.7	— 6.5
Ref: 2011/1041886 Trial: L100073 Germany, 2010 Weddegast	2 × 0.33 kg ai/ha (3300 L water/ha) 8 day spray interval DAT: 3 days	Green cones Dried cones	4.5 34	7.6
Ref: 2011/1041886 Trial: L10074 Germany, 2010 Golzern	2 × 0.33 kg ai/ha (3300 L water/ha) 7 day spray interval DAT: 3 days	Green cones Dried cones	8.5 33	3.9

Flowchart brewing (hop)

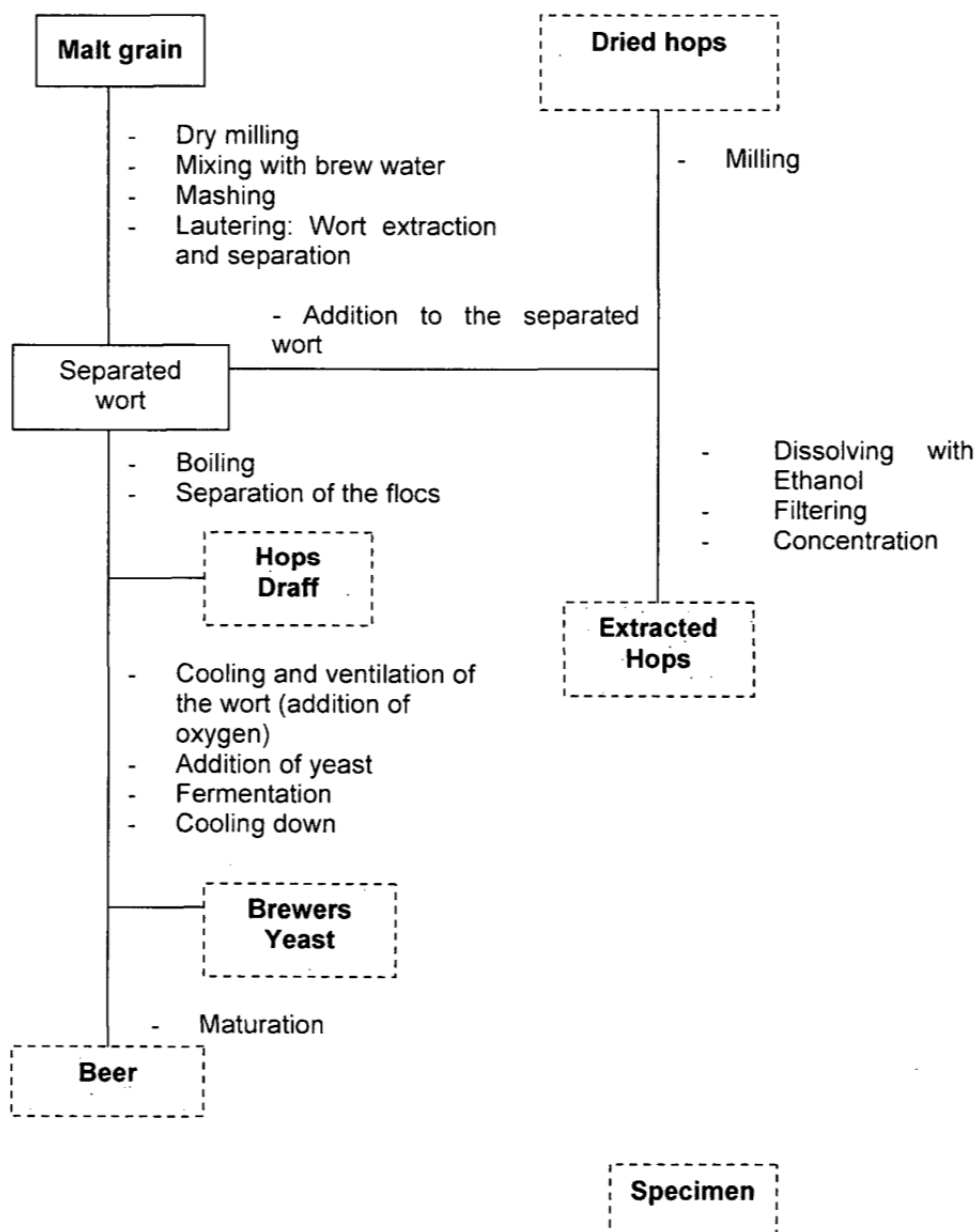


Figure 8 Processing Flowchart for Hops

Table 71 Summary of processing factors for metrafenone

RAC	Matrix	Metrafenone ^a	
		Calculated processing factors	PF median or best estimate
Apple	fruit		
	canned	0.1, 0.14	0.12
	juice	0.19, 0.23	0.21
	wet pomace	1.1, 1.3	1.2
	dried slices	0.39, 0.72	0.56
	sauce	4.1, 4.8	4.45
Grape	grapes		

RAC	Matrix	Metrafenone ^a	
		Calculated processing factors	PF median or best estimate
	must (red wine)	0.03, 0.57, 0.77, 0.78, 0.81, 1.2, 1.3	0.78
	must (white wine)	0.15, < 0.18, 0.26	0.18
	wet pomace	2.8, 3.6	3.2
	young wine (white)	0.07	< 0.2
	young wine (red)	0.03, < 0.17, < 0.19, < 0.21, 0.3, < 0.38, < 0.71	
	wine (white)	0.07, < 0.18, < 0.26	< 0.19
	wine (red)	0.03, < 0.17, < 0.19, < 0.19, < 0.21, < 0.38, < 0.71	
	juice	0.04, 0.06	0.05
	raisins	0.63, < 0.71, 3.6, 3.9	3.75
Strawberry	fruit		
	washed fruit	0.4, 0.45, 0.5, 0.52	0.475
	preserved fruit	0.79, 0.84, 0.99, 1.1	0.915
	jam	0.21, 0.21, 0.24, 0.28	0.225
	syrup	0.15, 0.16, 0.18, 0.19	0.17
Tomato	fresh		
	washed	0.57, 0.62, 0.84, 0.9	0.73
	blanched	0.45, 0.91, 1.3, 0.94	1.1
	peeled	< 0.01, < 0.02, 0.02, 0.05	0.02
	preserved	< 0.02, < 0.02, < 0.02, 0.02	< 0.02
	juice (raw)	0.26, 0.33, 0.35, 0.4	0.34
	wet pomace	3.3, 4.8, 6.2, 6.3	5.5
	peel	3.7, 6.2, 7.5, 7.9	6.85
	paste	0.27, 0.3, 0.47, 0.53	0.385
	ketchup	0.38, 0.42, 0.42, 0.5	0.42
	puree	0.65, 0.79, 0.83, 1.1	0.81
Mushroom	fresh		
	canned ^b	0.16	0.16
Barley	grain		
	pearl barley	< 0.13, 0.13, < 0.2, 0.22	0.165
	pearl barley abrasion	2.5	2.5
	malt	0.4	0.4
	Brewer's grain	0.3	0.3
	spent hops	< 0.1	< 0.1
	Brewer's yeast	< 0.1	< 0.1
	beer	< 0.1, < 0.13, < 0.17, < 0.33,	< 0.15
Wheat	grain		
	wholemeal flour	0.94, 1.1, 1.7, 1.9	1.4
	flour type 550	0.14, 0.17, 0.21, 0.29	0.19
	bran flour	1.3, 1.6, 2.6, 2.6	2.1
	coarse bran	0.29, 0.33, 0.43, 0.57	0.38
	fine bran	2.6, 3.5, 4.9, 5.3	4.2
	whole grain bread	0.6, 0.64, 0.71, 1.0	0.675
Hops	dried cones		
	extracted hops	1.8, 1.8, 1.8	1.8
	Brewer's yeast	0.008, 0.01, 0.01	0.01
	hops draff	0.24, 0.24, 0.25	0.24
	beer	< 0.0005, < 0.0005, < 0.0006	< 0.0005

^a Each value represents a separate study where residues were above the LOQ in the RAC. The factor is the ratio of metrafenone residues in the processed item divided by the residue of metrafenone in the RAC.

^b See Table 50

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

No livestock feeding studies were provided.

NATIONAL RESIDUE DEFINITIONS

Table 72 Metrafenone: National residue definitions for MRL-compliance and dietary intake estimation

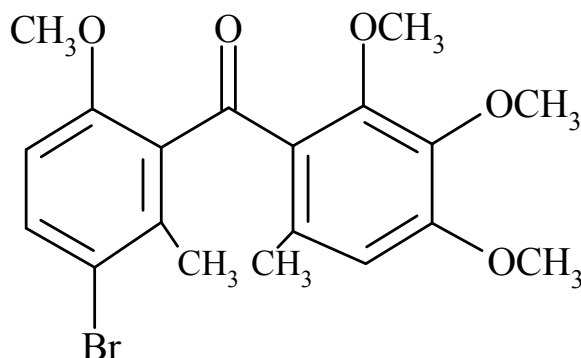
Country	MRL-compliance	Dietary intake estimation
Australia	metrafenone	metrafenone
Canada	metrafenone	metrafenone
Europe	metrafenone	metrafenone
Korea	metrafenone	metrafenone
New Zealand	metrafenone	metrafenone
USA	metrafenone	metrafenone

APPRAISAL

Metrafenone is a benzophenone fungicide, active mainly against powdery mildews and eyespot, inhibiting mycelium growth, leaf penetration, haustoria formation and sporulation.

It was scheduled by the Forty-fifth Session of the CCPR as a new compound for consideration by the 2014 JMPR. The manufacturer submitted studies on metabolism, analytical methods, supervised field trials, processing, freezer storage stability, environmental fate in soil and rotational crop residues.

Authorisations exist for the use of metrafenone on cereals, grapes, strawberries and fruiting vegetables in over 50 countries in Europe, the Americas, Asia and the Pacific.



Metrafenone
(MW 409.3)

The following abbreviations are used for the major metabolites discussed below:

Major metrafenone metabolites identified in plant, animal and soil matrices

Code	Structure	Chemical Name	Occurrence
CL 1023363		3-(3-bromo-6-methoxy-2-methylbenzoyl)-6-hydroxy-2-methoxy-4-methylphenyl β-D-glucopyranosiduronic acid Mono-O-glucuronide of Methanone, (3-bromo-6-hydroxy-2-methylphenyl)(3,4-dihydroxy-2-methoxy-6-methylphenyl-	goat

Code	Structure	Chemical Name	Occurrence
CL 1500698		3-(3-bromo-6-methoxy-2-methylbenzoyl)-2,6-dimethoxy-4-methylphenyl β-D-glucopyranosiduronic acid Methanone, (3-bromo-6-methoxy-2-methylphenyl)[3-beta-D-glucopyranuronosyloxy]-2,4-dimethoxy-6-methylphenyl-	rat, goat
CL 1500836		3-methoxy-2-(2,3,4-trimethoxy-6-methylbenzoyl)benzaldehyde	wheat grape
CL 197675		Methanone, (3-bromo-6-methoxy-2-carboxyl)(2,3,4-trimethoxy-6-methylphenyl)-	grape
CL 3000402		7-bromo-4-methoxy-3-(2,3,4-trimethoxy-6-methylphenyl)-2-benzofuran-1(3H)-one	rat wheat grape
CL 376991		Methanone, (3-bromo-6-methoxy-2-methylphenyl)(2,3,4-trimethoxy-6-methylphenyl)-	rat wheat
CL 379395		2-(3-bromo-6-methoxy-2-methylbenzoyl)-3,4,5-trimethoxybenzaldehyde	grape
CL 434223		Methanone, (3-bromo-6-methoxy-2-methylphenyl)(4-hydroxy-2,3-dimethoxy-6-methylphenyl)-	rat wheat
M560F06	R = H, R' and R'' = CH₃ or R' = H, R and R'' = CH₃ or R'' = H, R and R' = CH₃	Methanone, (3-bromo-6-methoxy-2-methylphenyl)(2-hydroxy-3,4-dimethoxy-6-methylphenyl)- or Methanone, (3-bromo-6-methoxy-2-methylphenyl)(3-hydroxy-2,4-dimethoxy-6-methylphenyl)- or Methanone, (3-bromo-6-methoxy-2-methylphenyl)(4-hydroxy-2,3-dimethoxy-6-methylphenyl)-	hen

Animal metabolism

The Meeting received information on the metabolism of ¹⁴C-metrafenone, separately labelled at the bromophenyl and the trimethoxyphenyl groups, in rats, lactating goats and laying hens. As no cleavage of the molecule was observed in these metabolism studies, the results for both radiolabels are reported together.

The metabolism of metrafenone in rats was evaluated by the WHO Core Assessment Group of the 2014 JMPR. Absorption of metrafenone is rapid and complete (> 88%) at the low dose of 10 mg/kg bw, limited to 15–20% at the high dose of 1000 mg/kg bw suggesting saturation of the

absorption processes. Metrafenone is widely distributed in the body, with highest residue levels mainly found in the gastro-intestinal (GI) tract, liver and fat. There is no evidence of accumulation. The labelled material is relatively rapidly excreted into the GI tract via the bile (85–90%) resulting in extensive excretion via faeces. Excretion via urine is relatively low (5–6% depending on radiolabel position), and even lower at the high dose level (*ca.* 1%). Metrafenone is extensively metabolised, with most of the radioactivity (approximately 80%) not identified, consisting of many (11–26) different components and totalling < 0.1 ppm at the low dose and < 1 ppm at the high dose. The identified metabolites, mostly < 1.0 mg eq./kg, included metrafenone and glucuronic acid conjugates in fat, liver and kidney.

Lactating goats

Lactating goats were orally dosed with ^{14}C -metrafenone at doses equivalent to about 10 ppm (8–13 ppm) and 70 ppm (60–87 ppm) in the feed for 5 consecutive days and sacrificed 23 hours after the last dose.

The majority of the radioactivity (76–86% AR) was excreted, mainly through the faeces. The highest residue levels were found in liver (0.21–0.23 mg eq./kg at the lower dose and 0.72–1.3 mg eq./kg at the higher dose) and kidney (0.05–0.06 mg eq./kg at the low dose and 0.16–0.33 mg eq./kg at the higher dose). Residues were significantly lower in fat (0.015–0.022 mg eq./kg at the high dose rate) and were ≤ 0.01 mg eq./kg in muscle and milk regardless of the dose rate. Residues reached a plateau in milk (0.01 mg eq./kg) within 3 days.

Residue characterization and identification was conducted on samples from the high dose groups with more than 95% TRR could be extracted from liver, kidney, milk and fat. Muscle samples were not investigated further because of the low TRR levels (< 0.01 mg eq./kg).

In fat, the predominant residue was metrafenone (0.01–0.02 mg/kg), making up 60–85% TRR and no other residues above 0.005 mg eq./kg (9% TRR) were found.

In liver and kidney, metrafenone made up about 3–4% TRR. The predominant residues were the glucuronide metabolites CL 1500698 and CL 1023363, not measured individually but together represented up to 15–21% TRR (0.27 mg eq./kg) in liver and up to 26–28% TRR (max 0.09 mg eq./kg) in kidney. An additional radiolabel fraction that included the glucuronide metabolites CL 1023361, CL1023362 and CL 1500702 totalled about 9–14% of TRR (max 0.17 mg eq./kg in liver and 0.03 mg/kg in kidney). About half the TRR was made up of a number of other unidentified metabolites, each present at < 5% TRR.

In milk, residues of metrafenone (24% TRR) and numerous metabolites, including one radiolabel fraction containing CL 1500698 and CL 1023363 (11% TRR), were all < 0.005 mg eq./kg.

Laying hens

In a poultry study laying hens were orally dosed with ^{14}C -metrafenone at doses equivalent to about 14 ppm in the feed for 12 consecutive days and sacrificed 22 hours after the last dose.

The majority (86–95%) of the administered dose was excreted, with about 0.25% AR (0.1 mg eq./kg) remaining in eggs, up to 0.09% AR (0.3–0.5 mg eq./kg) found in liver, < 0.01 % AR (0.06–0.08 mg eq./kg) present skin+fat and 0.003% AR (0.01 mg eq./kg) in muscle. Residues reached a plateau in eggs after 9 days.

Extraction was able to retrieve about 80% TRR from eggs, 60% TRR from skin+fat and about 30% TRR in liver and muscle. Characterisation and identification of residues in solvent-extracted samples indicated the presence of numerous polar and non-polar components. The one identified metabolite M560F06 was found in poultry skin+fat (6–11% TRR, < 0.01 mg eq./kg) and was identified but not quantified in eggs.

Metrafenone was found only in eggs and skin+fat, making up about 2% TRR (0.001–0.002 mg/kg) and the metabolite M560F06 was also measured in skin+fat (6–11% TRR) and

identified in eggs. With the exception of one unknown component in eggs (about 14% TRR, 0.015 mg eq./kg) all other metabolites were < 10% TRR (< 0.01 mg eq./kg) in all tissues and eggs.

In summary, residues were rapidly eliminated in the excreta (76–95% of the dose) with up to 0.5% of the total administered dose remaining in liver, 0.25% remaining in eggs, and TRRs were up to 0.02 mg eq./kg in fat and ≤ 0.01 mg eq./kg in muscle, poultry skin+fat and in milk.

The proposed metabolic pathways include hydroxylation and demethylation of the methyl groups and the phase II glucuronidation of the hydroxylated metabolites to various mono-O-glucuronides, qualitatively similar to the metabolic pathway in the rat.

Metrafenone made up about 2–4% of the TRR in liver, kidney, eggs and poultry skin+fat, was the main component in fat and was about 24% TRR in milk, but at very low levels (< 0.005 mg eq./kg). Most of the residues in liver and kidney were the glucuronide conjugates of metrafenone (CL 1500698, CL 1023363) which together made up 15–30% TRR, and numerous unidentified components, each present at < 10% TRR.

Plant metabolism

The Meeting received information on the metabolism of ^{14}C -metrafenone, separately labelled at the bromophenyl and the trimethoxyphenyl groups, in grapes, cucumber and wheat.

Grape

In outdoor grapevines treated with five foliar applications of ^{14}C -metrafenone at a rate equivalent to 0.2 kg ai/ha, 10–11 days apart, TRRs in grapes immediately after the last application were 0.6–0.77 mg eq./kg, reducing to 0.28–0.44 mg eq./kg at maturity, 35 days later. In leaves sampled immediately after the last application, TRRs were 40–42 mg eq./kg, reducing to 25–38 mg eq./kg after 35 days.

In grape juice, pomace and in leaves, 77–100% TRR was able to be sequentially extracted with acetone, methanol:water and water, with about 39–45% of the TRR in leaves being present in the acetone surface wash. Whole grapes were not analysed.

Metrafenone was not found in juice, but was the major residue in pomace (23–25% TRR, 0.06–0.11 mg/kg), and made up about 11–15% TRR in mature leave (35 days after the last application).

Characterisation of the residues in grape juice and pomace indicated the presence of several chromatographic fractions more polar than the parent, not exceeding 0.05 mg eq./kg and not more than 17% TRR. These were not identified further except for CL197675, found in juice at about 9% TRR (0.006 mg eq./kg).

Cucumber

In cucumber plants (confined) treated with two foliar applications of ^{14}C -metrafenone, applied 17 and 3 days before harvest, at a rate equivalent to 0.2 kg ai/ha, TRR in mature fruit, sampled 3 days after the second application were about 0.05 mg eq./kg (TRR), with 0.013 mg eq./kg present in pulp and 0.26 mg eq./kg in peel. More than 89% TRR was able to be extracted with methanol.

Metrafenone was the only identified residue component, making up 42% of the TRR in the mature fruit (0.02 mg/kg), and mostly in the peel (61% TRR, 0.16 mg/kg). Metrafenone also made up about 80% TRR in vines (without roots and fruit) at harvest.

Numerous polar and medium polar metabolites, characterized by their HPLC retention times and elution profiles, were present in fruit and vines at low concentrations (each less than 9% TRR).

Wheat

In outdoor wheat plants treated with 3 foliar applications of ^{14}C -metrafenone at rates equivalent to 0.3, 0.3 and 0.2 kg ai/ha, applied at 13–14 day intervals and with the last application being 35 days before

harvest, highest radioactive residues (up to 9 mg eq./kg) were found in hay and straw, with the lowest residues found in the grain (0.2–0.4 mg eq./kg). The TRR in foliage, 3 days after the first application were 5–8 mg eq./kg. Methanol:water extraction was able to release about 95% TRR in foliage, 78% TRR in hay, 61% TRR in straw and 35% TRR in grain. Additional extraction with hexane and acidified methanol was able to release a further 12–14% TRR in grain.

Metrafenone was the major component in all matrices, about 59–64% TRR in foliage, 13–26% TRR in hay, 8–14% in straw and 3–8% TRR in grain.

Other characterized or identified metabolites in foliage, hay and straw each represented less than 10% TRR. In grain, no identified metabolites were found above 0.004 mg eq./kg and although only about 50% of the radioactivity was extracted, further investigation showed that residues in the PES were made up of multiple minor components.

The proposed metabolic pathway involves oxidation of the methyl groups on the bromophenyl and trimethoxyphenyl rings to yield the corresponding aldehydes. In the case of the bromophenyl ring, the aldehyde can undergo further oxidation to the carboxylic acid, cyclization to form the lactone, and/or dehalogenation to form the des-bromo aldehyde.

In summary, metrafenone is the predominant residue in crops, with numerous minor metabolites or fractions present at low concentrations and generally more polar than the parent. While these were not all identified or quantified, individual peaks were < 10% TRR or < 0.01 mg eq./kg.

Environmental fate

The Meeting received information the environmental fate and behaviour of metrafenone, including hydrolytic stability, photolysis in aqueous solutions, aerobic metabolism and rotational crop metabolism studies.

Metrafenone was stable in sterile buffered solutions at pH 4, 7, and 9 but rapidly degraded in aqueous pH 7 solutions by photolysis (DT₅₀ values of 2.6–3.1 days) with the formation of multiple degradation products, all found at < 10% of the applied radioactivity. DT₉₀ values were 8.5 days (natural water) and 10.2 days (sterile water).

Aerobic soil metabolism

Metrafenone degraded slowly in loamy sand, sandy loam and clay loam soils treated with the equivalent of about 0.1 kg ai/ha [bromophenyl-label]-metrafenone or [trimethoxy-label]-metrafenone and incubated for up to 210 days under aerobic laboratory conditions. Metrafenone made up about 82% AR after 120-days and 66–69% AR after 210 days. Calculated half-lives (1st order kinetics) ranged from 182–365 days.

Residues in succeeding crops

In one outdoor rotational crop metabolism study, a leafy vegetable crop (lettuce), root crop (radish) and oil crop (canola) were planted back at various time intervals (30, 60, 90 and 365 days) after a single application of [trimethoxy-label]-metrafenone or [bromophenyl-label]-metrafenone to bare soil at a rate equivalent to 0.625 kg ai/ha.

The uptake of residues in these representative rotational crops (lettuce, radish, canola) was low, with TRRs at all plant-back intervals ranging from < 0.004 to 0.048 mg eq./kg (in canola pods), generally highest in the samples from the 30-day plant back interval. In soil, radioactive residues declined by about 50% after 90 days, and were mostly found in the top 10 cm of soil samples.

Total extractable residues ranged from 64% to 88% TRR in the majority of the samples (42–86% TRR in canola seed) and comprised mostly of multiple unidentified polar components, all present at < 0.02 mg eq./kg. Metrafenone accounted for 0.004 mg/kg of the TRR in lettuce (90 DAT) and radish roots (30 DAT) and was not found in canola or radish tops.

In summary, metrafenone is stable to hydrolysis, rapidly degraded by photolysis, slowly degraded in soil under aerobic conditions (remaining mostly in the top 10 cm) and not found at

significant levels in rotational crops. The Meeting concluded that residues are not expected in rotational crops following treatments according to the GAPs under consideration.

Analytical methods

Several analytical methods have been reported and validated for the analysis of metrafenone in plant and animal commodities. One method has also been validated for measuring residues of the CL 300402, CL 434223 and CL 376991 metabolites. The basic approach employs extraction with methanol/water, aqueous acetone or n-heptane/acetone, SPE or GPC clean-up and analysis by GC-ECD, GC-MS, or LC-MS/MS. In some methods, an additional partition step is included, using dichloromethane, cyclohexane, acetone, ethyl acetate, singly or sequentially.

For plant and processed plant commodities, the DFG S19 (GC-ECD or GC-MS) or the QuEChERS (LC-MS/MS) methods were used in most of the supervised residue field trials, with the RLA 12619 (LC-MS/MS) method used to measure parent and metabolites in some cereal trials. These methods were validated in a range of matrices (wheat, barley, grapes, cucumber, summer squash, melon, tomato, pepper, lemon, dry bean, oilseed rape and hops). The LOQ is 0.1 mg/kg for mushrooms and cereal forage and straws and 0.01 mg/kg for all other matrices.

For animal commodities, the DFG S19 (GC-MS) method was validated for the analysis of metrafenone in muscle, milk and eggs. After extraction with aqueous acetone, extracts are partitioned into ethyl acetate/cyclohexane before SPE clean-up and analysis. The LOQ is 0.01 mg/kg for milk and 0.05 mg/kg for muscle and eggs.

Stability of pesticide residues in stored analytical samples

Metrafenone residues were stable in analytical samples stored frozen (-18 to -20 °C) for up to 24 months in representative substrates with a high water content (lettuce, tomato), a high starch content (carrot, wheat grain), a high protein content (dry peas), a high oil content (soya bean) (grape, wine) and in , wheat forage and straw residues were stable for at least 31 months. In general, residues in the stored samples were greater than 80% of the spiked levels.

Definition of the residue

In animal commodities, metrafenone was the main identified component in goat fat (60–85% TRR) and poultry eggs and skin+fat (2% TRR) but made up about only 3–4% of the TRR in goat liver and kidney. Most of the identified residues in goat liver and kidney were in the radiolabel fraction that included CL 1500698 and CL 1023363 (totalling 15-30% TRR), with numerous unidentified minor fractions found at lower levels, each generally < 5% TRR. In muscle, TRRs were not found above 0.01 mg eq./kg in the highest dose groups of 65–87 ppm (goat) and 14 ppm (hen) and the TRR in milk was also < 0.01 mg eq./kg.

Based on the metabolism studies, a residue definition for animal commodities that includes metrafenone and the CL 1500698 and the CL 1023363 glucuronides could be considered. However the Meeting noted that these metabolites were not found in hens and only present in goats at low levels (totalling up to 0.27 mg eq./kg in liver and 0.09 mg eq./kg in kidney) following dosing at levels more than 7 times higher than the anticipated maximum livestock dietary burdens. CL 1023363 is structurally similar to rat metabolites and CL 1500698 was found in the rat metabolism study. Both metabolites are accommodated in the ADI. The Meeting therefore concluded that they need not be included in the residue definitions.

Based on the anticipated dietary exposure, the Meeting concluded that significant residues of the CL 1500698 and CL 1023363 metabolites are not expected in animal commodities and as a multi-residue method existed to measure the parent compound in animal commodities, a suitable residue definition for MRL-compliance and dietary intake estimation was metrafenone. Based on the Log P_{ow} of 4.3 and since residues of metrafenone were only found in fat and milk, the Meeting concluded that metrafenone is fat-soluble.

In plant commodities from treated crops, the metabolism studies indicated that metrafenone was the major residue in grape (up to 25% TRR in grape pomace), cucumber (up to 42% TRR) and wheat matrices (up to 8% TRR in grain and 14-64% TRR in foliage and straw), with numerous minor metabolites or radiolabel fractions present at low concentrations and generally more polar than the parent. While these were not all identified or quantified, individual peaks were <10% TRR or <0.01 mg eq./kg.

Metabolite CL 3000402 was occasionally found in grain at levels up to about 10% of the parent concentration but were <0.02 mg/kg. Metabolites CL 3000402, CL 434223 and CL 376991, found in the wheat metabolism study at up to 7% TRR in foliage and straw, were also measured in the foliage, straw and hay from a number of wheat and barley field trials, generally at levels less than 10% of the parent residue. These three metabolites were also found in the rat metabolism study and are accommodated in the ADI.

The Meeting noted that multiresidue methods exist to measure parent residues and agreed that for MRL-compliance and dietary intake estimation, the residue definition for plant commodities should be metrafenone.

Proposed definition of the residue (for compliance with the MRL and estimation of dietary intake for plant commodities): *metrafenone*.

Proposed definition of the residue (for compliance with the MRL and estimation of dietary intake for animal commodities): *metrafenone*.

Metrafenone is fat-soluble.

Results of supervised residue trials on crops

The Meeting received supervised trial data for foliar applications of metrafenone on a range of berries and other small fruits, fruiting vegetables, cereals and hops. These trials were conducted mainly in Europe and/or North America.

Where residues have been reported as ND (<LOD) the values have been considered as <LOQ (<0.01 mg/kg) for the purposes of MRL setting. If a higher residue level was observed at a longer PHI than the GAP, the higher value has been used in MRL setting.

The Meeting noted that GAP has been authorised for the use of metrafenone in more than 50 countries in Europe, the Americas, Asia and the Pacific and that product labels were available from many of these countries. Supervised trial data were also provided for pome fruit, stone fruit and hops, but no GAP information was available to support maximum residue level estimations for these commodities.

Berries and small fruit

Results from supervised trials on grapes conducted in USA and strawberries conducted in Europe were provided to the Meeting.

Grape

The critical GAP for metrafenone on grapes is in Canada, up to 6 foliar applications of 0.225 kg ai/ha applied at least 14–21 days apart with a PHI of 14 days and with a total of 1.35 kg ai/ha/season. In trials from USA conducted at about 1.5 times higher rate than the Canadian GAP, metrafenone residues in grapes were: 0.11, 0.17, 0.18, 0.27, 0.32, 0.62, 2.1, 2.3, 2.4, 3.0, and 3.2 mg/kg. When proportionally adjusted to the Canadian GAP (scaling factors (S_f) of 0.64–0.68), metrafenone residues in these trials are: 0.08, 0.11, 0.12, 0.17, 0.22, 0.42, 1.1, 1.4, 1.5, 1.6, 2.0 and 2.2 mg/kg (n=12).

The Meeting estimated an STMR of 0.74 mg/kg and a maximum residue level of 5 mg/kg for metrafenone on grapes.

Strawberry

The critical GAP for metrafenone on strawberries is on protected crops in the Netherlands, up to 2 foliar applications of 0.15 kg ai/ha, applied at least 7 days apart with a PHI of 3 days. In trials on protected strawberries matching this GAP in Netherlands, metrafenone residues in fruit were: 0.05, 0.06, 0.08, 0.1, 0.16, 0.23, 0.28 and 0.34 mg/kg (n=8).

The Meeting estimated an STMR of 0.13 mg/kg and a maximum residue level of 0.6 mg/kg for metrafenone on strawberries.

Fruiting vegetables, Cucurbits

Results from supervised trials on cucumbers, summer squash (zucchini) and melons (cantaloupes) conducted in Europe and North America were provided to the Meeting. However no GAP information was available from North America

Cucumber

The critical GAP for metrafenone on cucumbers is in France, up to 2 foliar applications of 0.1 kg ai/ha, applied at least 7–10 days apart with a PHI of 3 days. This GAP applies to both outdoor and protected crops.

In trials on outdoor cucumbers in Europe matching this GAP in France, metrafenone residues in cucumbers were: 0.01, 0.02, 0.02, 0.02, 0.02, 0.02, 0.03 and 0.04 mg/kg (n=8).

In trials on protected cucumbers matching this GAP in France, metrafenone residues in cucumbers were: 0.02, 0.04, 0.04, 0.05, 0.05, 0.06, 0.06, 0.07 and 0.09 mg/kg (n=9).

Based on the data set for protected cucumbers, the Meeting estimated an STMR of 0.05 mg/kg and a maximum residue level of 0.2 mg/kg for metrafenone on cucumber.

The Meeting also agreed to extrapolate these estimations to gherkins.

Summer squash

The critical GAP for metrafenone on summer squash is in France, up to 2 foliar applications of 0.1 kg ai/ha, applied at least 7–10 days apart with a PHI of 3 days. In trials on summer squash in Europe matching this GAP in France, metrafenone residues in summer squash were: 0.01, 0.01, 0.01, 0.01, 0.02, 0.02, 0.02 and 0.04 mg/kg (n=8).

The Meeting estimated an STMR of 0.015 mg/kg and a maximum residue level of 0.06 mg/kg for metrafenone on summer squash.

Melons (except watermelon)

The critical GAP for metrafenone on melons is in France, up to 2 foliar applications of 0.1 kg ai/ha, applied at least 7–10 days apart with a PHI of 3 days. In trials on melons in Europe matching this GAP in France, metrafenone residues in melons were: < 0.01, 0.01, 0.01, 0.02, 0.02, 0.02, 0.02, 0.02, 0.02, 0.03, 0.05, 0.06 and 0.07 mg/kg (n=13).

However the Meeting noted that in these trials, the melons had been quartered in the field and although the subsamples had been frozen within 12 hours after sampling, no information was available on residue stability in chopped or sliced samples.

The Meeting was unable to estimate a maximum residue level for metrafenone on melons.

Fruiting vegetables, other than Cucurbits

Results from supervised trials on tomatoes and peppers (sweet, bell and non-bell) conducted in Europe and North America and from trials on mushrooms in Europe were provided to the Meeting.

Mushrooms

The critical GAP for metrafenone on mushrooms is in France, one broadcast treatment of 0.05 kg ai/15 litres water/100 square metres of compost, applied up to 10 days before harvest. In trials on mushrooms in Europe matching this GAP in France, metrafenone residues in mushrooms were: 0.1, 0.1, 0.11 and 0.19 mg/kg (n=4)

The Meeting estimated an STMR of 0.105 mg/kg and a maximum residue level of 0.5 mg/kg for metrafenone on mushrooms.

The Meeting noted that the OECD MRL-calculator proposed a maximum residue level of 0.4 mg/kg, but agreed that a higher value on 0.5 mg/kg was more appropriate due to the small data set and because the relatively close spread of results may not reflect the residue variability arising from different composts used in mushroom production.

Pepper, Sweet

The critical GAP for metrafenone on peppers is in France for protected crops, up to 2 foliar applications of 0.15 kg ai/ha, applied at least 7–10 days apart with a PHI of 3 days. In trials on protected sweet peppers matching this GAP in France, metrafenone residues in peppers were: 0.07, 0.08, 0.1, 0.11, 0.12, 0.2, 0.21 and 1.3 mg/kg (n=8).

The Meeting estimated an STMR of 0.115 mg/kg and a maximum residue level of 2.0 mg/kg for metrafenone on peppers, sweet and agreed to extrapolate these estimations to chili pepper.

For dried chili peppers, applying the default processing factor of 10 to the STMR and the maximum residue level estimated for peppers, the Meeting estimated an STMR-P of 1.15 mg/kg and a maximum residue level of 20 mg/kg for metrafenone on dried chili peppers.

Tomato

The critical GAP for metrafenone on tomatoes is in Spain, up to 2 foliar applications of 0.015 kg ai/hL with a PHI of 3 days. This GAP applies to both outdoor and protected crops.

In trials on outdoor tomatoes in Europe matching this GAP in Spain, metrafenone residues in tomatoes were: 0.02, 0.05, 0.05, 0.06, 0.06, 0.07, 0.08 and 0.15 mg/kg (n=8).

In trials on protected tomatoes matching this GAP in Spain, metrafenone residues in tomatoes were: 0.06, 0.09, 0.09, 0.1, 0.1, 0.1, 0.16 and 0.17 mg/kg (n=8).

Based on the data set for protected tomatoes, the Meeting estimated an STMR of 0.1 mg/kg and a maximum residue level of 0.4 mg/kg for metrafenone on tomato.

Cereal grains

Results from supervised trials on wheat and barley conducted in Europe were provided to the Meeting.

Wheat

The critical GAP for metrafenone on wheat is in Poland, up to 2 foliar applications of 0.15 kg ai/ha with a PHI of 35 days. In trials in Europe matching this GAP in Poland, metrafenone residues in wheat grain were: < 0.01 (9), 0.01 (4), 0.02, 0.03, 0.03, 0.04 and 0.04 mg/kg (n=18).

The Meeting estimated an STMR of 0.01 mg/kg and a maximum residue level of 0.06 mg/kg for metrafenone on wheat.

The Meeting also agreed to extrapolate these estimations to rye and triticale.

Barley

The critical GAP for metrafenone on barley is in Poland, up to 2 foliar applications of 0.15 kg ai/ha with a PHI of 35 days. In trials in Europe matching this GAP in Poland, metrafenone residues in

barley grain were: < 0.01, 0.02 (3), 0.03, 0.04, 0.05 (3), 0.06, 0.06, 0.07, 0.08, 0.09, 0.11, 0.13, 0.15, 0.16, 0.23 and 0.4 mg/kg (n=20).

The Meeting estimated an STMR of 0.06 mg/kg and a maximum residue level of 0.5 mg/kg for metrafenone on barley.

The Meeting also agreed to extrapolate these estimations to oats.

Animal feeds

Cereal forages

Wheat and barley plant or foliage samples were collected in many of the European trials matching the GAP in Hungary/Poland (up to 2 foliar applications of 0.15 kg ai/ha).

Wheat forage

In wheat trials matching the GAP in Poland, metrafenone residues in plant (forage) samples taken 0-days after the last application were: 1.8, 2.0, 2.0, 2.6, 2.6, 2.6, 2.8, 3.3, 3.7, 3.8, 4.3 and 4.8 mg/kg (fresh weight).

The Meeting estimated a median residue of 2.7 mg/kg (fresh weight) and a highest residue of 4.8 mg/kg (fresh weight) for wheat forage and agreed to extrapolate these estimations to rye and triticale.

Barley forage

In barley trials matching the GAP in Poland, metrafenone residues in plant (forage) samples taken 0-days after the last application were: 1.8, 2.3, 2.5, 2.5, 3.1, 3.4, 3.7, 3.8, 4.6, 5.0, 5.8 and 5.9 mg/kg (fresh weight).

The Meeting estimated a median residue of 3.75 mg/kg (fresh weight) and a highest residue of 5.9 mg/kg (fresh weight) for barley forage and agreed to extrapolate these estimations to oats.

Cereal and grass straws and hays

Wheat and barley straw samples were collected in many of the European trials matching the GAP in Poland (up to 2 foliar applications of 0.15 kg ai/ha).

Wheat straw

In trials in Europe matching this GAP in Poland, metrafenone residues in wheat straw (fresh weight) were: 0.67, 0.67, 0.98, 1.1, 1.3, 1.4, 1.6, 1.7, 1.8, 2.0, 2.1, 2.3, 3.1, 3.1, 3.5, 3.6, 3.6 and 6.7 mg/kg (n=18). After correction for an average 88% dry matter content, residues (dry weight) were: 0.76, 0.76, 1.1, 1.3, 1.5, 1.6, 1.8, 1.9, 2.1, 2.3, 2.4, 2.6, 3.5, 3.5, 4.0, 4.1, 4.1 and 7.6 mg/kg.

The Meeting estimated a median residue of 1.9 mg/kg (fresh weight), a highest residue of 6.7 mg/kg (fresh weight) and a maximum residue level of 10 mg/kg (dry weight) for metrafenone in wheat straw.

The Meeting also agreed to extrapolate these estimations to rye and triticale.

Barley straw

In trials in Europe matching the GAP in Poland, metrafenone residues in barley straw (fresh weight) were: < 0.01, 0.24, 0.41, 0.95, 1.0, 1.1, 1.1, 1.1, 1.2, 1.3, 1.3, 1.5, 1.7, 1.8, 1.9, 1.9, 2.0, 2.1, 3.6 and 3.9 mg/kg (n=20). After correction for an average 89% dry matter content, residues (dry weight) were: < 0.01, 0.29, 0.46, 1.1, 1.1, 1.2, 1.2, 1.24, 1.4, 1.5, 1.5, 1.5, 1.9, 2.0, 2.1, 2.1, 2.3, 2.4, 4.0 and 4.4 mg/kg.

The Meeting estimated a median residue of 1.3 mg/kg (fresh weight), a highest residue of 3.9 mg/kg (fresh weight) and a maximum residue level of 6 mg/kg (dry weight) for metrafenone in barley straw.

The Meeting also agreed to extrapolate these estimations to oats.

Fate of residues during processing

The effect of processing on the nature of residues was investigated using radiolabelled metrafenone in buffer solutions incubated under conditions simulating pasteurisation (in pH 4 buffer at 90 °C for 20 minutes); baking, brewing, or boiling (in pH 5 buffer at 100 °C for 60 minutes); and sterilization (in pH 6 buffer at 120 °C for 20 minutes). Metrafenone was stable under these processing conditions with no significant changes in the radio-chromatograms.

The fate of metrafenone residues has been examined in a number of studies simulating household and commercial processing of apples, grapes, strawberries, tomatoes, barley, wheat and hops. Estimated processing factors and STMR-Ps for the commodities considered at this Meeting are summarised below.

Summary of selected processing factors and STMR-P values for metrafenone

RAC	Matrix	Metrafenone ^a	PF best estimate	STMR (mg/kg)	STMR-P (mg/kg)
		Calculated processing factors			
Grape	grapes			0.76	
	must (red wine)	0.03, 0.15, < 0.18, 0.26, 0.57, 0.77, 0.78, 0.81, 1.17, 1.29	0.67		0.51
	wet pomace	2.8, 3.6	3.2		2.4
	wine	0.03, 0.07, < 0.17, < 0.18, < 0.19, < 0.19, < 0.21, < 0.26, < 0.38, < 0.71	0.19		0.14
	juice	0.04, 0.06	0.05		0.038
	raisins	< 0.63, < 0.71, 3.63, 3.94	3.75		2.85
Tomato	fresh			0.1	
	preserved	< 0.02, < 0.02, < 0.02, 0.02	< 0.02		< 0.002
	juice (raw)	0.26, 0.33, 0.35, 0.4	0.34		0.034
	wet pomace	3.3, 4.8, 6.2, 6.3	5.5		0.55
	paste	0.27, 0.3, 0.47, 0.53	0.385		0.039
	puree	0.65, 0.79, 0.83, 1.1	0.81		0.081
Mushroom	fresh			0.105	
	canned	0.16	0.16		0.017
Barley	grain			0.06	
	pearl barley	< 0.13, 0.13, < 0.2, 0.22	0.165		0.01
	abraded fraction	2.5	2.5		0.15
	malt	0.4	0.4		0.024
	brewers grain	0.3	0.3		0.018
	beer	< 0.1, < 0.13, < 0.17, < 0.33,	< 0.15		< 0.009
Wheat	grain			0.01	
	wholemeal flour	0.94, 1.1, 1.7, 1.9	1.4		0.014
	flour type 550	0.14, 0.17, 0.21, 0.29	0.19		0.002
	fine bran	2.6, 3.5, 4.9, 5.3	4.2		0.042
	whole grain bread	0.6, 0.64, 0.71, 1.0	0.675		0.007

^a Each PF value represents a separate study where residues were above the LOQ in the RAC and is the ratio of the metrafenone residues in the processed item divided by the residues in the RAC.

The Meeting noted that in the studies available, metrafenone residues did not concentrate in food commodities during processing, except in dehydrated commodities such as raisins, bran and flour. Residues also increased in wet pomace (grape and tomato), tomato peel and barley abrasion fractions.

The Meeting estimated a maximum residue level for dried grapes of 20 mg/kg based on the maximum residue level estimated for grapes (5.0 mg/kg) and the median processing factor (3.75) from the USA processing studies.

The Meeting estimated a maximum residue level for wheat bran (processed) of 0.25 mg/kg based on the maximum residue level estimated for wheat (0.06 mg/kg) and a median processing factor of 4.2.

The Meeting estimated a maximum residue level for wheat wholemeal of 0.08 mg/kg based on the maximum residue level estimated for wheat (0.06 mg/kg) and a median processing factor of 1.4.

Residues in animal commodities

Farm animal dietary burden

The Meeting estimated the dietary burden of metrafenone in farm animals on the basis of the diets listed in Appendix IX of the 2009 edition of the JMPR Manual. Noting that fresh forage commodities are not significant in international trade, the Meeting only included the burden contributions from the cereal forages in the European dietary burden calculation, as metrafenone is not authorised for use on cereals in US-Canada, Australia or Japan.

Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are presented in Annex 6 of the 2014 Report and are summarized below:

Estimated maximum and mean dietary burdens of farm animals

	Animal dietary burden, metrafenone, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	0.8	0.26	5.9	3.8	9.3 ^a	4.9 ^c	0.07	0.07
Dairy cattle	0.8	0.25	5.9	3.8	9.2 ^b	4.9 ^d	0.42	0.14
Poultry – broiler	0.05	0.05	0.05	0.05	0.02	0.02	0.007	0.007
Poultry – layer	0.05	0.05	2.0 ^{e g}	1.3 ^{f h}	0.015	0.015	0.008	0.008

^a Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian tissues

^b Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk

^c Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

^d Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

^e Highest maximum poultry dietary burden suitable for MRL estimates for poultry tissues.

^f Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues.

^g Highest maximum poultry dietary burden suitable for MRL estimates for poultry eggs.

^h Highest mean poultry dietary burden suitable for STMR estimates for poultry eggs.

For beef and dairy cattle, the calculated maximum dietary burden is 9.3 ppm dry weight of feed and for poultry, noting that in some countries, laying hens may also be consumed, the calculated maximum dietary burden suitable is 2.0 ppm dry weight of feed.

Farm animal feeding studies

No livestock feeding studies were provided.

Animal commodity maximum residue levels

The Meeting noted that in the goat metabolism study, up to 0.014 mg/kg metrafenone was found in the kidney from the high (87 ppm) dose group animals and by extrapolation, this would equate to a maximum level of 0.0015 mg/kg in kidney from animals exposed to the calculated maximum dietary burden of 9.3 ppm.

In liver, metrafenone residues were up to 0.025 mg/kg in the high (60 ppm) dose group animals and by extrapolation, this would equate to a maximum level of 0.004 mg/kg in liver from animals exposed to the calculated maximum dietary burden of 9.3 ppm.

In animals dosed with 10 ppm in the diet (approximating the maximum calculated dietary burden for beef and dairy cattle, radiolabel residues were < 0.005 mg eq/kg in muscle, milk and fat.

The Meeting estimated maximum residue levels of 0.01* mg/kg for metrafenone in meat (from mammals other than marine mammals), 0.01 mg/kg for edible offal (mammalian), 0.01* mg/kg for mammalian fat and 0.01* mg/kg for milks. Estimated STMRS for dietary intake estimation are 0 mg/kg for meat, 0.01 mg/kg for edible offal, 0 mg/kg for fat and 0 mg/kg for milk.

In the hen metabolism study, residues of metrafenone were up to 0.002 mg/kg in eggs and up to 0.001 mg/kg in skin+fat in hens dosed with 14 ppm in the diet (about 7-fold higher than the maximum calculated dietary burden for poultry). In muscle and liver, metrafenone residues were not detected.

The Meeting estimated maximum residue levels of 0.01* mg/kg for metrafenone in poultry meat, 0.01* mg/kg for poultry offal, 0.01* mg/kg for poultry fat and 0.01* mg/kg for eggs. Estimated STMRS for dietary intake estimation are 0 mg/kg for poultry fat, 0 mg/kg for poultry meat, 0 mg/kg for poultry offal and 0 mg/kg for eggs.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI assessment.

Definition of the residue (for MRL-compliance and estimation of dietary intake, plant commodities): *metrafenone*.

Definition of the residue (for MRL-compliance and estimation of dietary intake, animal commodities): *metrafenone*.

The residue is fat soluble.

	Commodity	MRL	STMR or
CCN	Name	New	STMR-P
FB 0269	Grapes	5.0	0.76
DF 0269	Dried grapes	20	2.85
FB 0275	Strawberry	0.6	0.13
VC 0424	Cucumber	0.2	0.05
VC 0245	Gherkin	0.2	0.05
VC 0431	Squash, Summer	0.06	0.015
VO 0488	Tomato	0.4	0.1
VO 0445	Pepper, Sweet	2.0	0.115
VO 0444	Peppers, Chili	2.0	0.115
HS 0444	Peppers, Chili, dried	20	1.15
VO 0450	Mushroom	0.5	0.105
GC 0640	Barley	0.5	0.06
GC 0647	Oats	0.5	0.06
GC 0654	Wheat	0.06	0.01
GC 0650	Rye	0.06	0.01
GC 0653	Triticale	0.06	0.01
AS 0654	Wheat straw and fodder, Dry	10 (dw)	1.9 (fw) (hi-res 6.7)

	Commodity	MRL	STMR or
CCN	Name	New	STMR-P
AS 0650	Rye straw and fodder, Dry	10 (dw)	1.9 (fw) (hi-res 6.7)
AS 0653	Triticale straw and fodder, Dry	10 (dw)	1.9 (fw) (hi-res 6.7 fw)
AS 0640	Barley hay and straw	6 (dw)	1.3 (fw) (hi-res 3.9 fw)
AS 0647	Oat straw and fodder, Dry	6 (dw)	1.3 (fw) (hi-res 3.9 fw)
MM 0095	Meat (from mammals other than marine mammals)	0.01 (*)	0
MM 0100	Mammalian fats (except milk fats)	0.01 (*)	0
MO 0105	Edible offal (Mammalian)	0.01	0.01
ML 0106	Milks	0.01 (*)	0
PM 0110	Poultry meat	0.01 (*)	0
PF 0111	Poultry fat	0.01 (*)	0
PO 0111	Poultry, Edible offal of	0.01 (*)	0
PE 0112	Eggs	0.01 (*)	0
	Grape must		0.51
	Wine		0.14
JF 0269	Grape juice		0.04
JF 00488	Tomato juice		0.03
VW 0488	Tomato paste		0.04
	Tomato puree		0.08
	Tomato (canned)		0.002
	Mushrooms (canned)		0.017
CF 0654	Wheat bran, Processed	0.25	0.042
CF 1212	Wheat wholemeal	0.08	0.014
	Pearl barley		0.01
	Malt		0.024
	Beer		0.009
	Flour		0.002
	Bread (wholegrain)		0.007
	Tomato pomace (wet)		0.55
	Grape pomace (wet)		2.4
	Barley bran fractions		0.15
	Brewers grain		0.018
	Wheat forage		2.7 (fw) hi res 4.8 fw)
AF 0650	Rye forage (green)		2.7 (fw) hi res 4.8 fw)
	Triticale forage		2.7 (fw) hi res 4.8 fw)
	Barley forage		3.75 (fw) hi res 5.9 fw)
AF 0647	Oat forage (green)		3.75 (fw) hi res 5.9 fw)

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intake (IEDI) for metrafenone was calculated for the food commodities for which STMRs or HRs were estimated and for which consumption data were available. The results are shown in Annex 3.

The International Estimated Daily Intakes of metrafenone for the 17 GEMS/Food cluster diets, based on estimated STMRs were 0% of the maximum ADI of 0.3 mg/kg bw (Annex 3). The Meeting concluded that the long-term intake of residues of metrafenone from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The 2014 JMPR decided that an ARfD was unnecessary. The Meeting therefore concluded that the short-term intake of metrafenone residues is unlikely to present a public health concern.

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7003736	Riley, M	2013	Magnitude and decline of the residue following application of BAS 560 03 F cucumbers. Eurofins Agrosience Services Inc., Forsyth GA, United States of America. 2012/7003736. GLP/GEP: Yes. Unpublished
1033967	Schaeufele, M	2010	Residue study (decline) with BAS 560 02 F applied to cucumber (field) and zucchini (field) in Germany, the Netherlands, Belgium, Northern France, Southern France, Italy, Greece and Spain in 2009. Huntingdon Life Sciences Ltd., Eye Suffolk IP23 7PX, United Kingdom. 2010/1033967. GLP/GEP: Yes. Unpublished
1033968	Schaeufele, M	2010	Residue study (decline) with BAS 560 02 F applied to melons (field) in Northern France, Southern France, Italy, Greece and Spain in 2009. Huntingdon Life Sciences Ltd., Eye Suffolk IP23 7PX, United Kingdom. 2010/1033968. GLP/GEP: Yes. Unpublished
1033969	Schaeufele, M	2010	Residue study (decline) with BAS 560 02 F applied to cucumber (greenhouse) in Germany, the Netherlands, Belgium, Northern France, Southern France, Italy, Greece and Spain in 2009. Huntingdon Life Sciences Ltd., Eye Suffolk IP23 7PX, United Kingdom. 2010/1033969. GLP/GEP: Yes. Unpublished
1041395	Schaeufele, M	2011	Residue study (decline) with BAS 560 02 F applied to melons (field) in Germany in 2010. Huntingdon Life Sciences Ltd., Eye Suffolk IP23 7PX, United Kingdom. 2011/1041395. GLP/GEP: Yes. Unpublished
7001657	Smalley, R	2001	BAS 560 F (AC 375839) 300 g as/L SC (SF10358) and BAS 560 F (AC 375839) 300 g as/L SC (SF09957): At harvest residue study on BAS 560 F in winter wheat—North France 2000. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2001/7001657. GLP/GEP: Yes. Unpublished
7001658	Smalley, R	2001	BAS 560 F (AC 375839) 300 g as/L SC (SF10358) and BAS 560 F (AC 375839) 300 g as/L SC (SF09957): At harvest residue study on BAS 560 F in winter wheat—United Kingdom 2000. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2001/7001658. GLP/GEP: Yes. Unpublished
7001675	Smalley, R	2001	AC 375839 300 g as/L SC (SF 09957): Decline curve residue study on AC 375839 in winter wheat—Germany 1999. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2001/7001675. GLP/GEP: Yes. Unpublished
7001660	Smalley, R	2001	BAS 560 F (AC 375839) 300 g as/L SC (SF 10358): Decline curve residue study on BAS 560 F in winter wheat—North France 2000. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2001/7001660. GLP/GEP: Yes. Unpublished
7001656	Smalley, R	2001	BAS 560 F (AC 375839) 300 g as/L SC (SF10358) and BAS 560 F (AC 375839) 300 g as/L SC (SF09957): At harvest residue study on BAS 560 F in winter wheat—South France 2000. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2001/7001656. GLP/GEP: Yes. Unpublished
7001676	Smalley, R	2001	BAS 560 F (AC 375839) 300 g as/L SC (SF 10358): Decline curve residue study on BAS 560 F in winter wheat—South France 2000. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2001/7001676. GLP/GEP: Yes. Unpublished
7001659	Smalley, R	2001	AC 375839 300 g as/L SC (SF 09957): Decline curve residue study on AC 375839 in winter barley—Germany 1999. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2001/7001659. GLP/GEP: Yes. Unpublished
7004680	Smalley, R	2002	AC 375839 300 g as/L SC (SF 09957): Decline curve residue study on AC 375839 in winter wheat—United Kingdom 1999. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2002/7004680. GLP/GEP: Yes. Unpublished
7004745	Smalley, R	2002	AC 375839 300 g as/L SC (SF 09957): Decline curve residue study on AC 375839 in winter wheat—Netherlands 1999. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2002/7004745. GLP/GEP: Yes. Unpublished

Code	Author	Year	Title, Institute, Report reference
7004740	Smalley, R	2002	AC 375839 300 g as/L SC (SF 09957): Decline curve residue study on AC 375839 in winter wheat—France South 1999. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2002/7004740. GLP/GEP: Yes. Unpublished
7004445	Smalley, R	2002	BAS 560 F (AC 375839) 300 g as/L SC (SF10358) and BAS 560 F (AC 375839) 300 g as/L SC (SF09957): At harvest residue study on BAS 560 F in winter barley—North France 2000. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2002/7004445. GLP/GEP: Yes. Unpublished
7004463	Smalley, R	2002	BAS 560 F (AC 375839) 300 g as/L SC (SF10358) and BAS 560 F (AC 375839) 300 g as/L SC (SF09957): At harvest residue study on AC 375839 in winter barley—Germany 2000. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2002/7004463. GLP/GEP: Yes. Unpublished
7004525	Smalley, R	2002	BAS 560 F (AC 375839) 300 g as/L SC (SF10358) and BAS 560 F (AC 375839) 300 g as/L SC (SF09957): At harvest residue study on BAS 560 F in winter barley—South France 2000. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2002/7004525. GLP/GEP: Yes. Unpublished
7004529	Smalley, R	2002	BAS 560 F (AC 375839) 300 g as/L SC (SF10358) and BAS 560 F (AC 375839) 300 g as/L SC (SF09957): At harvest residue study on BAS 560 F in winter barley—United Kingdom 2000. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2002/7004529. GLP/GEP: Yes. Unpublished
7004744	Smalley, R	2002	BAS 560 F (AC 375839) 300 g as/L SC (SF 10358): Decline curve residue study on BAS 560 F in winter barley—South France 2000. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2002/7004744. GLP/GEP: Yes. Unpublished
7004922	Smalley, R	2002	BAS 560 F (AC 375839) 300 g as/L SC (SF 10358): Decline curve residue study on BAS 560 F in winter barley—North France 2000. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2002/7004922. GLP/GEP: Yes. Unpublished
7000487	Trewhitt, JA	2001	AC 375839 300 g as/L SC (SF10358 (BAS 560 00 F) and SF09957): At harvest residues study on AC 375839 (BAS 560 F) in winter wheat—The Netherlands, 2000. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2001/7000487. GLP/GEP: Yes. Unpublished
7000488	Trewhitt, JA	2001	AC 375839 300 g as/L SC (SF09957): Decline curve residue study on AC 375839 (BAS 560 F) in winter barley, South France, 1999. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2001/7000488. GLP/GEP: Yes. Unpublished
7004681	Trewhitt, JA	2002	AC 375839 300 g as/L SC (SF09957): Decline curve residue study on AC 375839 (BAS 560 F) in winter barley—UK, 1999. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2002/7004681. GLP/GEP: Yes. Unpublished
7004267	White, MT and Stewart, J	2006	Residue of Metrafenone (BAS 560 F) Fenpropimorph, (BAS 421 F) Epoxiconazol (BAS 480 F) in formulation bridging on wheat, barley after BAS 565 00 F BAS 562 00 F BAS 560 00 F BAS 421 12 F BAS 480 31 F field, in DE, DK, N-FR, S-FR, SE, UK 2005. BASF Agro Research RTP, Research Triangle Park NC, United States of America. 2005/7004267. GLP/GEP: Yes. Unpublished
7001430	Wyatt, DR	2013	Magnitude and decline of the residues of Metrafenone in or on grape raw agricultural commodities following three foliar applications of BAS 560 03 F fungicide. The Carringers Inc., Apex NC, United States of America. 2013/7001430. GLP/GEP: Yes. Unpublished
7000144	Class, T	2000	AC 375839 (CL 375839): Storage stability of AC 375839 residues at <−18 °C in grapes and wine. PTRL Europe GmbH, Ulm, Germany Fed.Rep. 2000/7000144. GLP/GEP: Yes. Unpublished
BN-326-010	Class, T	2001	BAS 560 F (AC 375839): Storage stability of BAS 560 F residues at less than or equal to −18 °C in carrots and lettuce. PTRL Europe GmbH. BN-326-010. GLP/GEP: Yes. Unpublished
7004653	Class, T	2002	BAS 560 F (AC 375839): Storage stability of BAS 560 F residues at less than or equal to −18 °C in cereal grain and straw. PTRL Europe GmbH, Ulm, Germany Fed.Rep. 2002/7004653. GLP/GEP: Yes. Unpublished

Code	Author	Year	Title, Institute, Report reference
1043493	Lehmann, A and Mackenroth, C	2011	Investigation of the storage stability of BAS 560 F in plant matrices. BASF SE, Limburgerhof, Germany Fed.Rep. 2011/1043493. GLP/GEP: Yes. Unpublished
1166088	Lehmann, A and Mackenroth, C	2012	Investigation of the storage stability of BAS 560 F in plant matrices. BASF SE, Limburgerhof, Germany Fed.Rep. 2012/1166088. GLP/GEP: Yes. Unpublished
1187284	Mackenroth, C and Radzom, M	2011	Metrafenone (BAS 560 F)—Statement to request of Chemical Regulation Directorate on Metrafenone storage stability. BASF SE, Limburgerhof, Germany Fed.Rep. 2011/1187284. GLP/GEP: No. Unpublished
1013928	Smalley, R	2003	AC 375839 and metabolites—Freezer storage stability in wheat (whole plant, straw and grain). BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2003/1013928. GLP/GEP: Yes. Unpublished
7000137	An, D	2000	AC 375839: Effects of processing on the nature of the residues due to hydrolysis. BASF Corp., Ewing NJ, United States of America. 2000/7000137. GLP/GEP: Yes. Unpublished
1041879	Braun, D	2011	Determination of residues of BAS 560 F in hops and its processed products after two applications of BAS 560 02 F in Germany. BioChem agrar Labor fuer biologische und chemische Analytik GmbH, Gerichshain, Germany Fed.Rep. 2011/1041879. GLP/GEP: Yes. Unpublished
7004393	Carringer, SJ	2013	Magnitude of the residue of Metrafenone in or on apple processed commodities following three foliar applications of BAS 560 03 F fungicide. The Carringers Inc., Apex NC, United States of America. 2012/7004393. GLP/GEP: Yes. Unpublished
7007012	Jordan, JM and Kasiri, A	2006	Magnitude of BAS 560 F residues in grapes and grape processed fractions following applications of BAS 560 00 F (amended final report). BASF Agro Research RTP, Research Triangle Park NC, United States of America. 2006/7007012. GLP/GEP: Yes. Unpublished
1041884	Plier, S	2011	Determination of residues of BAS 560 F (Metrafenone) in tomatoes and their processed products after two applications of BAS 560 02 F in Germany. BioChem agrar Labor fuer biologische und chemische Analytik GmbH, Gerichshain, Germany Fed.Rep. 2011/1041884. GLP/GEP: Yes. Unpublished
1041883	Plier, S	2011	Determination of residues of BAS 560 F (Metrafenone) in strawberries and their processed products after two applications of BAS 560 02 F in Germany. BioChem agrar Labor fuer biologische und chemische Analytik GmbH, Gerichshain, Germany Fed.Rep. 2011/1041883. GLP/GEP: Yes. Unpublished
1004080	Pollmann, B	2002	Determination of residues of BAS 560 F in field samples and in processed goods after application of BAS 560 00 F in summer barley at 4 sites in Germany in 2001. GAB Biotechnologie GmbH & IFU Umweltanalytik GmbH, Niefern-Oeschelbronn, Germany Fed.Rep. 2002/1004080. GLP/GEP: Yes. Unpublished
1006302	Pollmann, B	2002	Determination of residues of BAS 560 F in field samples and processed goods after application of BAS 560 00 F in summer wheat at 4 sites in Germany in 2001. GAB Biotechnologie GmbH & IFU Umweltanalytik GmbH, Niefern-Oeschelbronn, Germany Fed.Rep.. 2002/1006302. GLP/GEP: Yes. Unpublished
7004451	Smalley, R	2002	BAS 560 01 F (AC 375839) 500 g as/L SC (SF 09955): At harvest residue and processing study on BAS 560 F in vines—Italy, 2000. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2002/7004451. GLP/GEP: Yes. Unpublished
7004455	Smalley, R	2002	BAS 560 01 F (AC 375839) 500 g as/L SC (SF 09955): At harvest residue and processing study on BAS 560 F in vines—North France, 2000. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2002/7004455. GLP/GEP: Yes. Unpublished
7004459	Smalley, R	2002	BAS 560 01 F (AC 375839) 500 g as/L SC (SF 09955): At harvest residue and processing study on BAS 560 F in vines—Spain, 2000. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2002/7004459. GLP/GEP: Yes. Unpublished

Code	Author	Year	Title, Institute, Report reference
7004460	Smalley, R	2002	BAS 560 01 F (AC 375839) 500 g as/L SC (SF 09955): At harvest residue and processing study on BAS 560 F in vines—South France, 2000. BASF plc, Gosport Hampshire PO13 0AU, United Kingdom. 2002/7004460. GLP/GEP: Yes. Unpublished
7001048	Smalley, R	2001	Method Validation of RLA 12619.00 "Determination of CL 375839, CL 3000402, CL434223 and CL 376991 Residues in Cereals (whole plant and straw) using LC-MS". BASF Agro Research, BASF plc. Gosport Hampshire PO13 0AU, United Kingdom. 2001/70001048. GLP/GEP: Yes. Unpublished
7001770	Kang, J	2001	Validation of RLA 12619.02V for CL 375839 in Wheat Grain to a Limit of Quantification of 0.01 mg/kg. BASF Agro Research, BASF plc. Gosport Hampshire PO13 0AU, United Kingdom. 2001/70001770. GLP/GEP: Yes. Unpublished
1004080	Pollmann, B	2002	Determination of residues of BAS 560 F in field samples and in processed goods after application of BAS 560 00 F in summer barley at four sites in Germany in 2001. GAB Biotechnologie GmbH & IFU Umweltanalytik GmbH, Niefern-Oeschelbronn, Germany Fed.Rep. 2002/1004080. GLP/GEP: Yes. Unpublished
7000111	Smalley, R	2000	Validation of a gas chromatography method for the determination of CL 375839 residues in grapes. BASF Agro Research, BASF plc. Gosport Hampshire PO13 0AU, United Kingdom. 2000/7000111. GLP/GEP: Yes. Unpublished

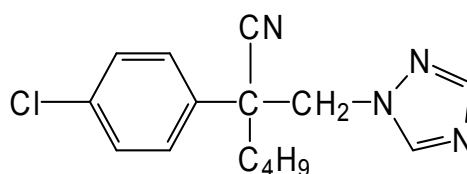
MYCLOBUTANIL (181)*First draft prepared by Dr. Yibing He,**Department of Science and Education, Ministry of Agriculture, China***EXPLANATION**

Myclobutanil was originally evaluated by the JMPR in 1992 and re-evaluated for residues several times up to 1998. It was reviewed as part of the periodic re-evaluation programme of CCPR on residue and toxicity in 2014 JMPR. Myclobutanil is a systemic protectant fungicide used to control brown rot, powdery mildew and leaf spot in stone fruit crops, and belongs to the sterol demethylation inhibitor (DMI) class of fungicides.

At the Forty-fifth Session of the CCPR (REP13/PR, Appendix XIV), myclobutanil was scheduled for periodic residue review by the 2014 JMPR. The Meeting received information on physical and chemical properties, metabolism, environmental fate, analytical methods and freezer storage stability, national registered use patterns, as well as supervised trials, processing studies and livestock feeding studies.

IDENTITY

ISO Common name	myclobutanil
Chemical name	
IUPAC name	(R,S)-2-(4-chlorophenyl)-2-(1H-1,2,4-triazole-1-ylmethyl)hexanenitrile
CAS name	α -n-butyl- α -(4-chlorophenyl)-1H-1,2,4-triazole-1-propanenitrile
CAS Registry Number	88671-89-0
CIPAC Number	442
Synonyms and trade names	
Manufacturer's codes	RH-53,866
Structural formula	



Molecular formula	C ₁₅ H ₁₇ Cl N ₄
Molecular weight	288.8 g/mol
Minimum content of ai	

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient		Ref
Appearance (purity 98.75%) (purity 95.4%)	White crystal Colourless to white crystal	134760 134761
Odour (purity 98.75%) (purity 95.4%)	Odourless Faint aldehyde	134760 134761
Vapour pressure (purity 99.9%):	1.98×10^{-4} Pa at 20 °C	94926
Henry's law constant	4.33×10^{-4} kPa m ³ /mol	94865
Boiling point (purity 98.75%)	390.8 °C at 97.6 kPa	94922
Melting point (purity 98.75%)	70.9 °C	111087
Octanol-water partition coefficient at 22 °C: (purity 99.9%)	log P _{OW} = 2.556 There is no pH effect as the molecule is neither acidic nor basic. It does not dissociate.	138549
Solubility in water at 20 °C (purity 99.9%):	pH 3-5: 124 g/L pH 7: 132 g/L pH 9-11: 115 g/L	94924
Relative density (purity 98/75%)	1.243 g/cm ³ 20.4 °C	95055
Dissociation constant in water (purity 99.6%)	The pure ingredient does not have acidic hydrogen and is expected to be a very weak base. Attempts to measure pK _a by titration failed to detect any inflection curve indicating no dissociation	
Hydrolysis rate at pH 4, 7 and 9 under sterile and dark conditions	At 25 °C myclobutanil is hydrolytically stable more than 1 year at pH 4, 7 and 9.	94798
Direct phototransformation in sterile water using artificial light		
Surface tension, 24 °C (purity 99.8%)	46.8 mN/m for 90% saturated aqueous solution at 24 °C.	95055
Quantum yield efficiency (purity 97.3%)		
Solubility in organic solvents at 20 °C:	Acetone > 250 g/L Dichloromethane > 250 g/L Ethyl acetate > 250 g/L n-Heptane 1.02 g/L Methanol > 250 g/L Xylene 270 g/L	134458

Formulations

Myclobutanil is available in various formulations such as the following formulations:

Formulation	Products
Suspension concentrate	45 g/L SC, 60 g/L SC
Wettable Powder	2.25% WP, 40% WP, 35% WP, 10% WP, 10.8% WP, 1% WP, 2.25% WP, 8 g/kg WP, 2.0% WP, 3.8% WP

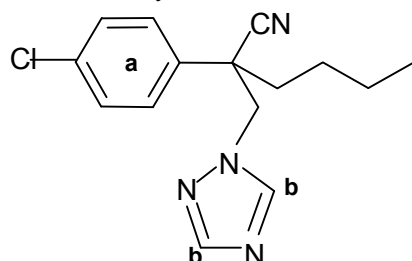
Emulsion Concentrate	240 g/L EC, 125 g/L EC, 32.4 g/L EC, 75 g/L EC, 25.8% EC, 45 g/L EC, 23.4%EC, 30% EC
Emulsion Oil in Water	200 g/L EW, 45 g/L EW, 25 g/L EW

METABOLISM AND ENVIRONMENTAL FATE

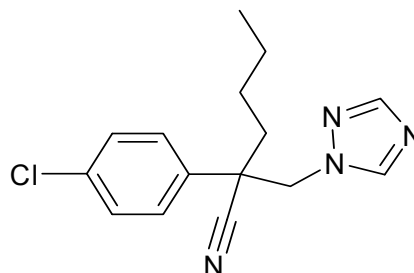
The Meeting received information on animal metabolism, plant metabolism and environmental fate studies using [^{14}C]-myclobutanil (phenyl and triazole ring labelled) and unlabelled myclobutanil.

a ^{14}C -Phenyl myclobutanil

b ^{14}C -Triazole myclobutanil

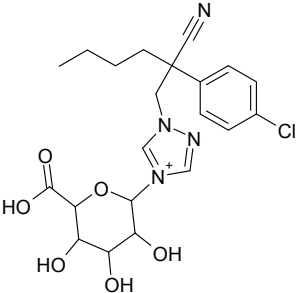
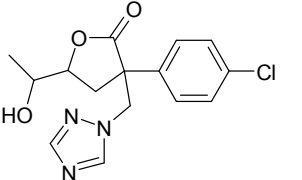
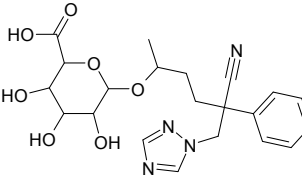
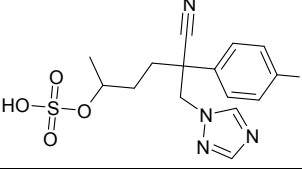
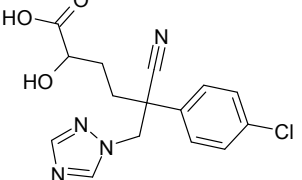
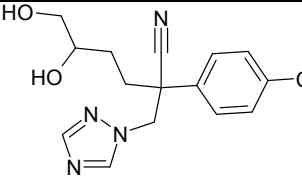
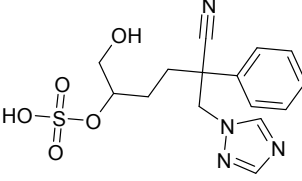
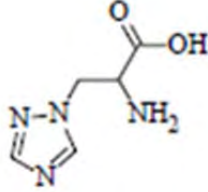


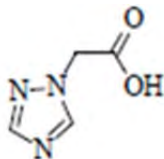
Unlabelled myclobutanil



Structures, names and codes for myclobutanil and its metabolites in metabolism and environmental fate studies are summarized below.

Code Name	Chemical Name	Metabolite Identity	Matrix where found
Myclobutanil Parent	α -butyl- α -(4-chlorophenyl)-1H-1,2,4-triazole-1-propanenitrile		Grapes Apples Wheat Sugar beet Laying hens Ruminants
(RH-9089)	(2RS)-2-(4-chlorophenyl)-5-oxo-2-(1H-1,2,4-triazol-1-ylmethyl)hexanenitrile		Grapes Apples Sugar beet Ruminants
(RH-9090)	α -(4-chlorophenyl)- α -(3-hydroxybutyl)-1H-1,2,4-triazole-1-propanenitrile		Grapes Apples Wheat Sugar beet Ruminants
MW 318 Acid (butyl carboxylic acid of myclobutanil)	5-(4-chlorophenyl)-5-cyano-6-(1H-1,2,4-triazol-1-yl)hexanoic acid		Groundwater Soil Ruminants

Code Name	Chemical Name	Metabolite Identity	Matrix where found
N-Glucuronic Acid Conjugate of Myclobutanil	1-[2-(4-chlorophenyl)-2-cyanoethyl]-4-hexopyranuronosyl-1H-1,2,4-triazol-4-ium		Ruminants
Hydroxy-lactone	3-(4-chlorophenyl)-5-(1-hydroxyethyl)-3-(1H-1,2,4-triazol-1-ylmethyl)dihydrofuran-2(3H)-one		Ruminants
RH-9090 Glucuronic Acid Conjugate	5-(4-chlorophenyl)-5-cyano-6-(1H-1,2,4-triazol-1-yl)hexan-2-yl hexopyranosiduronic acid		Grapes Apples Wheat Sugar beet Ruminants
RH-9090 Sulfate Conjugate	5-(4-chlorophenyl)-5-cyano-6-(1H-1,2,4-triazol-1-yl)hexan-2-yl hydrogen sulfate		Laying hens Ruminants
MW 334 Acid	5-(4-chlorophenyl)-5-cyano-2-hydroxy-6-(1H-1,2,4-triazol-1-yl)hexanoic acid		Ruminants
RH-294 (Diol)	α -(4-chlorophenyl)- α -(3,4-hydroxy-butyl)-1H-1,2,4-triazole-1-propane-nitrile		Laying hens Ruminants
RH-294 Sulfate Conjugate	5-(4-chlorophenyl)-5-cyano-1-hydroxy-6-(1H-1,2,4-triazol-1-yl)hexan-2-yl hydrogen sulfate		Ruminants
TA (Triazolyl Alanine)	(2 <i>RS</i>)-2-amino-3-(1H-1,2,4-triazol-1-yl)propanoic acid		Rotational wheat

Code Name	Chemical Name	Metabolite Identity	Matrix where found
TAA (Triazolyl Acetic Acid)	1H-1,2,4-triazol-1-ylacetic acid		Rotational wheat

Animal Metabolism

The Meeting received animal metabolism studies with myclobutanil in lactating goats and laying hens.

Lactating goats

Myclobutanil was orally given to lactating goats (Alpine dairy goats) for five days. Individual goats were dosed separately with myclobutanil radiolabelled in either the triazole (TZ) portion or the phenyl ring (PH) at the rate of 24 ppm and 14 ppm (Rotondaro, S. L. *et al.*, 2010, 2006417) in dry feed of the diet per day, respectively. Milk, urine and faeces were collected daily. The animals were sacrificed 6–7 hours after the final dose and the following tissues were collected: liver, muscle (flank and loin), fat (subcutaneous, omental, and renal), and kidney. Samples were stored at -10 °C or below until analysis. Levels of total radioactive residues (TRR) in urine and milk samples were determined by direct liquid scintillation counting (LSC). TRR levels in the liver, kidney, muscle and faecal samples were determined by oxidative combustion followed by LSC. TRR levels in fat were determined by the heated solubilisation followed by direct LSC of the liquefied samples. The amount of the milk, fat, excreta and edible tissues, and after extraction with acetone, hexane and acetonitrile/water (80/20) and liquid-liquid partition they were analysed by HPLC. Myclobutanil and metabolites in urine, liver and kidney were identified by MS. The results were summarized in Table 1 and Table 4.

Table 1 TRRs in milk, edible tissues and excreta from goats orally dosed a for 5 consecutive days with either ¹⁴C-[PH] myclobutanil or ¹⁴C-[TZ] myclobutanil

Matrix	Collection	¹⁴ C-TZ-label (24.2 mg/kg)			¹⁴ C-PH-label (14.2 mg/kg)		
		(mg/kg)	(% dose)	(% dose)	(mg/kg)	(% dose)	(% dose)
Milk	Day 1 pm	0.044	0.02	–	0.021	0.02	–
	Day 1 am	0.031	0.02	–	0.010	0.01	–
	Day 2 pm	0.070	0.02	–	0.028	0.02	–
	Day 2 am	0.042	0.03	–	0.014	0.02	–
	Day 3 pm	0.075	0.02	–	0.027	0.02	–
	Day 3 am	0.043	0.03	–	0.016	0.02	–
	Day 4 pm	0.079	0.03	–	0.033	0.02	–
	Day 4 am	0.043	0.03	–	0.016	0.02	–
	Day 5 pm	0.076	0.02	0.23	0.033	0.02	0.18
	Day 5 am	0.076	0.02	0.23	0.033	0.02	0.18
Urine	Day 1	19.105	10.59	–	4.863	11.41	–
	Day 2	30.863	11.29	–	7.229	12.81	–
	Day 3	28.153	15.32	–	6.471	14.42	–
	Day 4	23.492	9.29	–	11.039	13.73	–
	Day 5	37.663	2.87	49.36	19.260	5.18	57.55
Faeces	Day 1	3.934	2.34	–	1.593	2.17	–
	Day 2	7.589	4.54	–	3.347	4.49	–
	Day 3	8.377	6.34	–	4.565	4.83	–
	Day 4	9.158	5.79	–	4.595	6.30	–
	Day 5	8.594	2.89	21.90	4.334	3.79	21.58
Cage Rinse	Sacrifice	27.690	7.86	7.86	1.863	1.26	1.26
Muscle—Flank	Sacrifice	0.063	0.03	–	0.023	0.03	–
Muscle—Loin	Sacrifice	0.061	0.04	0.07	0.024	0.02	0.05
Liver	Sacrifice	0.918	0.46	0.46	0.487	0.48	0.48

Matrix	Collection	¹⁴ C-TZ-label (24.2 mg/kg)			¹⁴ C-PH-label (14.2 mg/kg)		
Kidney	Sacrifice	0.518	0.04	0.04	0.206	0.04	0.04
Fat—Subcutaneous	Sacrifice	0.040	0.004	—	0.017	0.004	—
Fat—Omental	Sacrifice	0.027	0.014	—	0.016	0.013	—
Fat—Renal	Sacrifice	0.035	0.006	0.02	0.020	0.006	0.02
Total		NA	79.95	79.95	NA	81.15	81.15

Table 2 Summary of characterization and identification of radioactive residues in Day 1 and Day 5 milk from goats dosed a with either ¹⁴C-[TZ] myclobutanil or ¹⁴C-[PH] myclobutanil

Fraction ID	TZ Label				PH Label			
	Day 1 (pm)		Day 5 (pm)		Day 1 (pm)		Day 5 (pm)	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Organic Extract ^a	95.0	0.042	95.0	0.072	95.0	0.020	88.0	0.029
Myclobutanil	ND	ND	ND	ND	ND	ND	ND	ND
RH-9089	0.6	< 0.001	0.9	< 0.001	ND	ND	0.6	< 0.001
RH-9090	45.0	0.020	28.3	0.022	57.8	0.012	49.3	0.016
MW 318 Acid	10.4	0.005	7.4	0.006	11.9	0.002	5.7	0.002
N-glucuronide of Myclobutanil	ND	ND	ND	ND	ND	ND	0.3	< 0.001
Hydroxy-lactone	3.4	0.001	5.4	0.004	6.5	0.001	6.4	0.002
RH-9090 Conjs	3.7	0.002	0.7	< 0.001	ND	ND	0.5	< 0.001
MW 334 Acid	0.5	< 0.001	ND	ND	ND	ND	0.2	< 0.001
RH-294	13.6	0.006	8.2	0.006	5.5	0.001	4.4	0.001
RH-294 Conj.	ND	ND	ND	ND	ND	ND	ND	ND
Polar Unks. (2–4 Minutes)	ND	ND	7.5	0.006	ND	ND	ND	ND
Unidentified	18.6	0.008	36.5	0.028	13.4	0.003	20.3	0.007
Total Identified	77.2	0.034	50.9	0.039	81.7	0.017	67.4	0.022
Total Characterized	95.0	0.042	95.0	0.072	95.0	0.020	88.0	0.029
Total Extractable	95.0	0.042	95.0	0.072	95.0	0.020	88.0	0.029
Post Extn Solids	5.0	0.002	6.0	0.005	.0	0.002	11.0	0.004
Accountability	100	—	100	—	100	—	100	—

^a Extracted with acetone.

ND = Not Detectable

Table 3 Summary of characterization and identification of radioactive residues in the edible tissues of a goat dosed with ¹⁴C-[TZ] myclobutanil

Fraction ID	Liver		Kidney		Muscle		Fat	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Hexane Extract	NA	NA	NA	NA	NA	NA	7.0	0.003
Organic Extract ^a	98.0	0.900	101.0	0.523	94.0	0.059	75.0	0.030
Myclobutanil	2.1	0.019	ND	ND	ND	ND	ND	ND
RH-9089 ^b	14.8	0.136	12.6	0.065	ND	ND	2.3	< 0.001
RH-9090	16.0	0.147	13.6	0.070	44.0	0.028	35.0	0.014
MW 318 Acid	0.3	0.003	1.5	0.008	ND	ND	ND	ND
N-glucuronide of Myclobutanil	ND	ND	0.4	0.002	ND	ND	ND	ND
Hydroxy-lactone	12.1	0.111	4.7	0.024	0.8	< 0.001	ND	ND
RH-9090 Conjs	43.0	0.395	44.1	0.228	3.3	0.002	10.9	0.004
MW 334 Acid	4.0	0.037	9.6	0.050	0.4	< 0.001	1.8	< 0.001
RH-294	1.9	0.017	10.6	0.055	11.6	0.007	5.0	0.002
RH-294 Conj.	0.6	0.006	ND	ND	ND	ND	ND	ND
Polar Unks. (2–4 Minutes)	ND	ND	ND	ND	10.9	0.007	ND	ND

Fraction ID	Liver		Kidney		Muscle		Fat	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Unidentified	3.2	0.029	3.8	0.020	22.9	0.014	19.0	0.008
Total Identified	94.8	0.870	97.1	0.503	60.1	0.038	56.0	0.022
Total Characterized	98.0	0.900	100.9	0.523	94.0	0.059	75.0	0.030
Total Extractable	98.0	0.900	101.0	0.523	94.0	0.059	82.0	0.033
Post Extn Solids	8.0	0.073	1.0	0.005	4.0	0.003	2.0	< 0.001
Accountability	100	—	100	—	100	—	84	—

^a Extracted with acetonitrile/water (80/20)

^b Reported residues for RH-9089 in liver and kidney may be artificially high as the radioactivity eluting in the RH-9089 zone in the HPLC system used was often very broad in nature. Reanalysis of these extracts in another system or by LC-MS often showed only very low levels of RH-9089. Much of the radioactivity during reanalysis appeared to be additional RH-9090 or conjugates of RH-9090.

ND = Not Detectable

NA= Not Applicable

Table 4 Summary of characterization and identification of radioactive residues in the edible tissues of a goat dosed with [¹⁴C][PH] myclobutanil

Fraction ID	Liver		Kidney		Muscle		Fat	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Hexane Extract	NA	NA	NA	NA	NA	NA	10.0	0.002
Organic Extract ^a	96.0	0.467	100.0	0.206	96.0	0.022	68.0	0.012
Myclobutanil	6.0	0.029	ND	ND	ND	ND	ND	ND
RH-9089 ^b	0.2	0.001	24.3	0.050	ND	ND	ND	ND
RH-9090	25.0	0.122	17.2	0.035	80.0	0.018	39.1	0.007
MW 318 Acid	4.9	0.024	2.2	0.005	ND	ND	ND	ND
N-glucuronide of Myclobutanil	ND	ND	0.1	< 0.001	ND	ND	ND	ND
Hydroxy-lactone	15.6	0.076	2.5	0.005	3.6	0.001	ND	ND
RH-9090 Conjs	34.8	0.169	43.7	0.099	ND	ND	4.8	0.001
MW 334 Acid	3.0	0.015	4.4	0.009	ND	ND	ND	ND
RH-294	1.2	0.006	4.2	0.009	6.1	0.001	0.9	< 0.001
RH-294 Conj.	0.2	0.001	ND	ND	ND	ND	ND	ND
Polar Unks. (2–4 Minutes)	ND	ND	ND	ND	ND	ND	ND	ND
Unidentified	5.1	0.025	1.3	0.003	6.3	0.001	23.1	0.004
Total Identified	90.9	0.443	98.6	0.203	89.7	0.021	45.1	0.008
Total Characterized	96.0	0.467	99.9	0.206	96.0	0.022	68.2	0.012
Total Extractable	96.0	0.467	100.0	0.206	96.0	0.022	8.0	0.013
Post Extn Solids	9.0	0.044	1.0	0.002	3.0	0.001	4.0	< 0.001
Accountability	100	—	100	—	100	—	82	—

^a Extracted with acetonitrile/water (80/20)

^b Reported residues for RH-9089 in liver and kidney may be artificially high as the radioactivity eluting in the RH-9089 zone in the HPLC system used was often very broad in nature. Reanalysis of these extracts in another system or by LC-MS often showed only very low levels of RH-9089. Much of the radioactivity during reanalysis appeared to be additional RH-9090 or conjugates of RH-9090.

ND = Not Detectable

NA= Not Applicable

The unchanged parent compound was only observed in liver. The metabolite RH-9090 and its sulphate and glucuronic acid conjugates were the primary residue in liver, in kidney, in muscle, in fat and in milk. The hydroxyl-lactone was the only other metabolite present in liver more than 10% of the TRR. The only other two metabolites representing 10% or more of the TRR were RH-294 and the MW 318 carboxylic acid in milk and no parent compound was detected in milk.

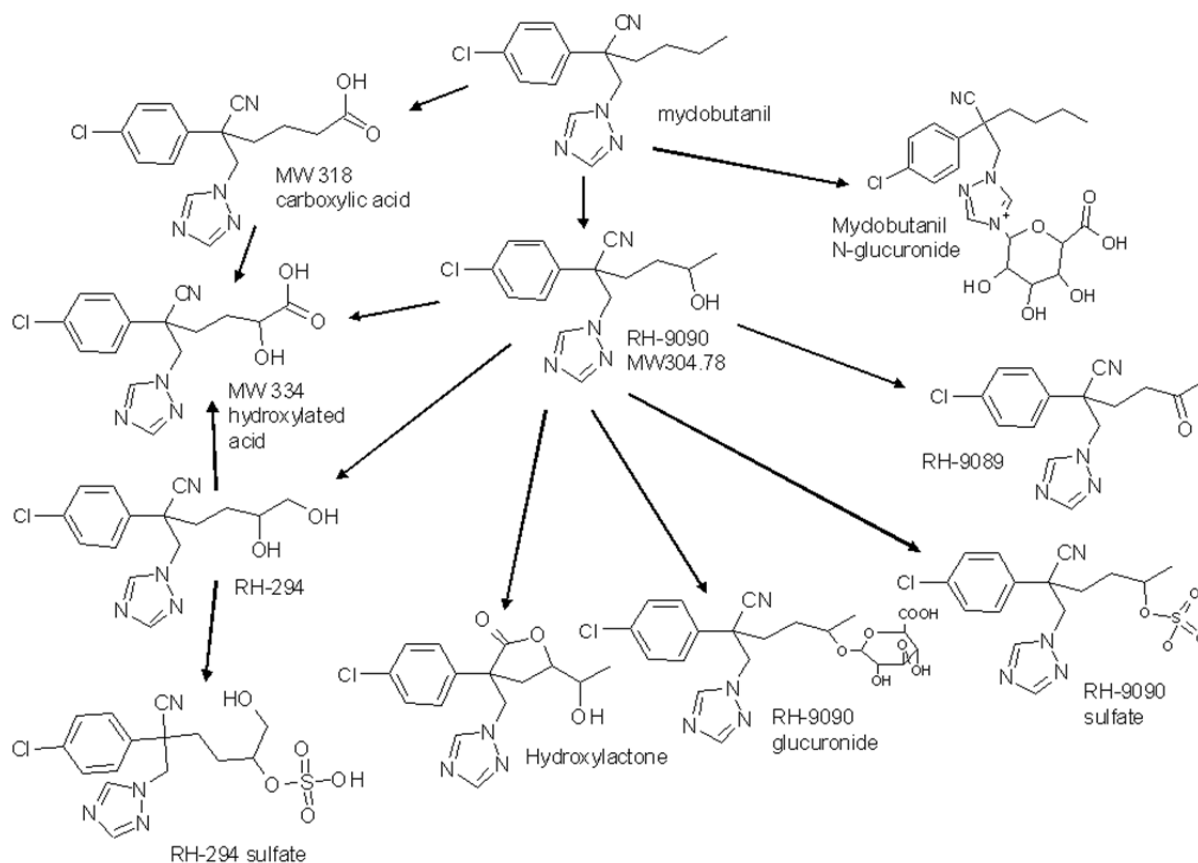


Figure 1 Proposed metabolic profile for myclobutanil in ruminants

Laying hens

[¹⁴C]-Myclobutanil or a mixture of RH9090/9089 (82:18) were each administered orally by gelatine capsule to separate groups of three laying hens (Leghorn) once daily for 7 consecutive days at the nominal equivalent of 110 ppm in the diet (Jacobson, 1986, 94610; Martin, 1986, 94169). The test substances were uniformly labelled in the phenyl ring (myclobutanil: 110 ppm) and in the triazole ring (82 ppm RH9090: 18 ppm RH9089). Ten hens were used as a control group. All chickens in these groups were sacrificed 24 hours after final dose. All eggs were separated into yolks and whites on study days 1 and 6. All samples were frozen pending analysis. Radioactivity was measured by liquid scintillation counting (LSC). Whole eggs and muscle were extracted with ethyl acetate, and then extracted with methanol. Fat and edible offal were extracted with n-hexane and methanol. The nature of radioactivity in all tissues was investigated further. The nature of radioactivity in eggs and tissues was investigated further. The results were summarized in Tables 5 to 7.

Table 5 Myclobutanil residue equivalents (expressed as mg myclobutanil equivalents/kg) in tissues from hens dosed orally at rates equivalent to 110 ppm in the diet

Sample Type	Residue Level (mg/kg)	
	Myclobutanil group	RH-9090/RH-9089 group
Liver	0.52	0.31
Kidney	0.32	0.16
Heart	ns	ns
Gizzard	ns	ns
Breast	0.060	0.077
Thigh	0.056	0.065
Fat	0.017	0.010

ns = Not sampled.

Table 6 Summary of characterization and identification of radioactive residues in eggs and the edible tissues of treated with [¹⁴C]-myclobutanil

Fraction ID	Eggs	Fat	Breast muscle	Thigh muscle	kidney	liver
	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR
Total extracted	69%	79%	97%	83%	156%	72%
myclobutanil		67.2	4	2	11.5	4.8
RH-294	4.4			8	2.7	
RH-9090-sulphate	4.7		4	5		2.2
RH-9090	35.6		4		14.9	3.9
RH-9089	10.4		72	61		
Lactone	12.3					
Unknown 1	2.3					
Unknown 2						
Less polar than RH-3866						
Hydroxy lactone		9.5				
Organic unknown				6	112.5	58.4
Polar unknown		2.4	0		5.5	3.0
Total characterised	70	79	84	82	147	72
Solids	31%	21%	21%	32%		28%
Total	100%	100%	118%	115%	156%	100%

Table 7 Summary of characterization and identification of radioactive residues in eggs and the edible tissues of treated with a mixture of [¹⁴C]-RH9090/9089

Fraction ID	Eggs	Fat	Breast muscle	Thigh muscle	Kidney	Liver
	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR
Total extracted	77%	79%	86%	72%	125%	104%
myclobutanil	0.0					
RH-294	6.9		3	3	2.5	1.8
RH-9090-sulphate	1.0		6	5		
RH-9090	47.2	18.2			20.8	11.2
RH-9089	4.9		57	49		
Lactone	11.8					
Unknown 1	2.2					
Unknown 2	3.1					
Less polar than parent		27.7				
Hydroxy lactone		21.3				
Organic unknown			16	13	100.8	87.1
Polar unknown		12.6				3.9
Total characterised	77.0	79.8	81.6	71.5	124.0	104.0
Solids	23%	21%	20%	18%		
Total	100%	100%	106%	90%	125%	104%

Residues in tissues were extremely low at the exaggerated dose level of 110 ppm. Highest levels were observed in the liver and kidney. Residues in fat were extremely low and there was no evidence of accumulation of residues in fatty tissues. The major components of the residue were attributed to the RH-9090, RH-9089, RH-294 (diol), RH-9090-sulphate and the 4-hydroxy-lactone, which were all detected at very low levels, and would be anticipated to be not-detectable or barely detectable at a normal dose rate.

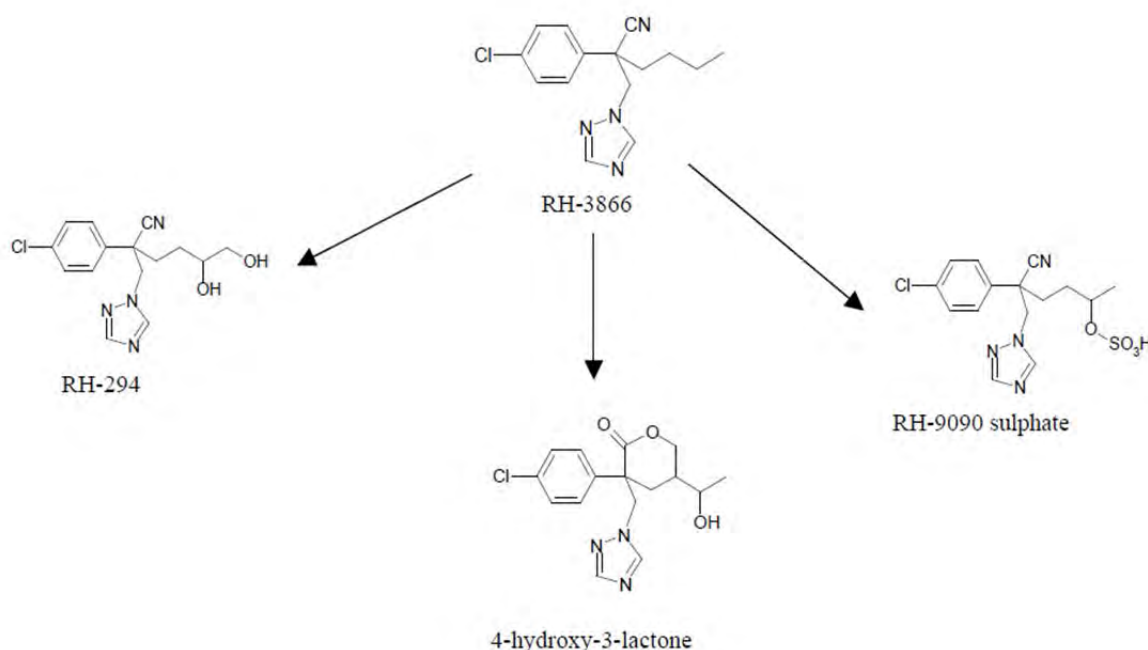


Figure 2 Proposed metabolic pathway of myclobutanil in laying hens

Plant metabolism

The Meeting received plant metabolism studies with myclobutanil in grapes, apples, wheat, and sugar beets.

Grapes

Experiments on grape seedlings (*Vitis*, variety De Chaunac) were conducted in nutrient solution with either 68.5 μg (4.6 mg/L) of ^{14}C -phenyl myclobutanil (specific activity 10.28 mCi/g, radiochemical purity 99%) or 52.4 μg (3.5 mg/L) of ^{14}C -triazole myclobutanil (specific activity 10.98 mCi/g, radiochemical purity 99%). The plants were placed in the greenhouse. Additional nutrient solution was added as needed. The study was terminated after either 7 or 16 days uptake (Nelson, SS, 1984, 94279). The roots were washed in 20 mL of water to remove any radiolabelled material on the root surface. Plants were stored frozen prior to analysis. Radioactivity was measured by liquid scintillation counting (LSC). The plants were extracted with methanol followed by successive partitioning with chloroform and 1-butanol for the 7 day uptake plants and with hexane and chloroform for the 16 day uptake plants. The effect of enzymatic hydrolysis using α - and β -glucosidase enzymes on the chromatographic behaviour of conjugates was investigated. The natures of radioactivity in the selected samples were investigated further with TLC. The results were summarized in Tables 8 and Table 9.

Table 8 Distribution in different extracts (% TRR) of grape seedlings treated with [^{14}C]myclobutanil

	7 Day Uptake		16 Day Uptake	
Solvent	Phenyl	Triazole	Phenyl	Triazole
Hexane	–	–	2.1	1.5
Chloroform	72.6	75.8	64.6	64.7
1-butanol	15.3	11.0	18.0	17.4
Aqueous	0.5	1.8	0.4	2.2
Unextracted	11.6	11.3	15.0	14.1
Total	100.1	99.6	100.1	99.9

– = Not extracted with hexane.

Table 9 Distribution of radioactive residues (%TRR) in grape seedlings among myclobutanil and its metabolites

Metabolite	7 Day Uptake		16 Day Uptake	
	Phenyl	Triazole	Phenyl	Triazole
myclobutanil	36	38	55	51
RH-9090	8	4	7	8
RH-9090 glucoside	11	11	11	14
Polar metabolite	13	15	1	1
unextracted	12	11	15	14
Total	80	79	89	88

Grape vines (variety not stated) received five weekly sprays of myclobutanil, labelled with ^{14}C in the phenyl ring or the triazole ring each at a rate equivalent to 0.05 kg ai/ha (Nelson, SS, 1998, 98373) during August 1984. Samples of grapes and foliage were taken after each treatment. Final harvest was 7 days after the final treatment. Two bunches of grapes (from upper and lower branches) were taken at each harvest except for the final harvest when all remaining fruit were removed from the vines. Grapes and foliage taken after each of the treatments were ground with dry ice. The radioactivity in all samples was determined by combustion of samples and analysed by liquid scintillation counting (LSC). The residues in juice were partitioned successively against chloroform and 1-butanol. Wet pomace and foliage were soxhlet extracted with methanol and subsequently partitioned against hexane. The nature of radioactivity in the selected samples were investigated further with TLC and confirmed by GC-ECD. The results were summarized in Tables 10 to Table 12.

Table 10 Total radioactive residues in grapes receiving up to five weekly applications of [^{14}C]myclobutanil

Sampling Date	Residue Levels (mg myclobutanil equivalents/kg tissue)	
	Phenyl	Triazole
03 Aug 1984	0.047	0.090
09 Aug 1984	0.23	0.014
16 Aug 1984	0.051	0.18
24 Aug 1984	0.036	0.15
30 Aug 1984	0.38	0.31
06 Sept 1984	0.32	0.24

Table 11 Magnitude of radioactive residues (mg myclobutanil equivalents/kg tissue) in grape samples from the final harvest

Fraction	Harvest Residue Levels (mg myclobutanil equivalents/kg tissue)	
	Phenyl	Triazole
Whole Grape	0.32	0.24
Juice	0.042	0.034
Pomace (dried)	2.81	2.43
Pomace (wet)	0.97	0.91

Table 12 Nature of radioactive residues (%TRR) in whole grapes at harvest

Metabolites	Harvest Residue Levels (mg myclobutanil equivalents/kg tissue)			
	Phenyl		Triazole	
	% TRR	mg/kg	% TRR	mg/kg
Myclobutanil	66	0.21	66	0.16
RH-9089	1	0.003	1	0.002
RH-9090	9	0.028	7	0.017
RH-9090 glucoside	6	0.019	5	0.012
Total	82	0.26	79	0.191

Apples

Apple trees (semi-dwarf MacIntosh variety) received ten approximately weekly sprays of myclobutanil, labelled with ^{14}C in the phenyl ring or the Triazole ring in 1984 (Nelson, SS and Streelman, DR, 1984, 94618). One apple tree received phenyl labelled myclobutanil whilst the other received triazole labelled material. The apples from both trees were separately harvested fourteen days after the final application. Applications were begun in mid-June until end August. The application rate was equivalent to 240 g ai/ha. Harvested apples were quartered and separated into juice and pomace. Wet pomace obtained in this way was frozen and ground to a fine consistency with dry ice. Both juice and pomace were stored frozen until analysis. The radioactivity in pomace samples was determined by combustion of samples and analysed by liquid scintillation counting (LSC). Juice samples were assayed directly by LSC. The residues in juice were extracted successively with chloroform and 1-butanol. Pomace was soxhlet extracted with methanol and subsequently partitioned against chloroform. The natures of radioactivity in the selected samples were investigated further with TLC. The results were summarized in Tables 13 and Table 14.

Table 13 Magnitude of radioactive residues in apple fractions from fruit receiving ten sprays of [^{14}C] myclobutanil

Fraction	Residue Levels (mg myclobutanil equivalents/kg tissue)	
	Phenyl	Triazole
Juice	0.15	0.12
Pomace	1.00	0.66
Whole Fruit ^a	0.48	0.32

^a Calculated from residues in juice and pomace.

Table 14 Nature of radioactive residues (%TRR) in apples

Fraction	Harvest Residue Levels (% TRR)	
	Phenyl	Triazole
Juice		
myclobutanil	21.7	23.8
RH-9089	1.3	1.2
RH-9090	26.5	24.7
Conjugated RH-9090	40.7	30.0
Total	90.2	79.7
Pomace		
myclobutanil	54.9	56.0
RH-9089	1.9	3.4
RH-9090	7.9	7.6
Conjugated RH-9090	19.7	18.3
Total	84.4	85.3
Whole Fruit ^a		
myclobutanil	48.5	48.7
RH-9089	1.8	2.9
RH-9090	11.5	11.5
Conjugated RH-9090	23.7	20.9
Total	85.5	84.0

^a Calculated from residues in juice and pomace.

Wheat

Approximately 30 wheat (*Triticum aestivum*, variety Wanser) seedlings were placed in a foil covered jars containing 65 mL of 1/3 strength Hoaglands nutrient solution and either 2700 μg (42 ppm) of ^{14}C -phenyl myclobutanil (specific activity 10.28 mCi/g, radiochemical purity 99%) or 4170 μg (64 ppm) of ^{14}C -triazole myclobutanil (specific activity 10.98 mCi/g, radiochemical purity 99%). The plants were placed in the greenhouse (Nelson, SS, 1984, 94218). After 11 days, the plants' roots were placed in 90 mL of water to wash off any myclobutanil remaining on the root surface. Wheat seedlings,

excised wheat shoots, and excised wheat heads were stored frozen prior to analysis. Radioactivity was measured by liquid scintillation counting (LSC). The different plant parts were homogenized with methanol followed by liquid/liquid partitioning against hexane. The nature of radioactivity in samples was investigated further with TLC or GLC. The results were summarized in Table 15.

Table 15 Distribution (% of total samples radioactivity) and nature of residues in wheat following application of [^{14}C]myclobutanil (Nelson, SS, 1984, 94218)

	Intact Seedlings 11 day Uptake		Excised Shoots 5 day Uptake		Excised Heads 13 day Uptake	
	Ph	Tz	Ph	Tz	Ph	Tz
myclobutanil	62	71	73	72	73	75
RH-9090	2	2	6	6	5	4
RH-9090 glucoside	15	11	5	7	16	18
RH-9090 malonyl glucoside	15	10	5	5	-	-
Non-extractable Residue	2	1	0.5	0.4	1	1
Total	96	95	89.5	90.4	95	96

The metabolism of myclobutanil, radiolabelled in either the phenyl (Ph) or triazole (Tz) ring, was studied in wheat under field conditions at a rate equivalent to 0.28 kg ai/ha (Streelman, DR, 1984, 94246). The Tz labelled wheat received 2× 0.28 kg ai/ha treatments and the Ph treated wheat 1× 0.28 kg ai/ha treatment. The first application was in April 1983 (BBCH 30) and the second in early May (BBCH 32) with Tz labelled myclobutanil. A single application of phenyl labelled myclobutanil was BBCH 45. In a second experiment, wheat grown in the greenhouse was treated with 2× 0.28 kg ai/ha of Ph-labelled myclobutanil. The first application was made to plants as BBCH 31 and the second at BBCH 45. Plants were harvested on the day of application and at 41 days for PH label under field, 68 days for TZ label under field and 43 days for PH label under greenhouse after last treatment (DALT) then separated into straw, chaff and grain by threshing. Samples of all plant fractions were combusted to $^{14}\text{CO}_2$ which was determined by liquid scintillation counting (LSC). Straw and grain samples were extracted with methanol and followed by liquid/liquid partitioning between water and chloroform. The nature of radioactivity in the selected samples were investigated further with TLC and/or further analysed by GC-MS or MS. The results are summarized in Tables 16 to 20.

Table 16 Total radioactive residues (expressed as mg eq/kg tissue) in wheat grain and straw from crops treated with [^{14}C]myclobutanil

	Phenyl Field	Triazole Field	Phenyl Greenhouse
Application Regime / Growth stage at application	1× 0.28 kg ai/ha BBCH 45	2× 0.28 kg ai/ha BBCH 30 and 32	2× 0.28 kg ai/ha BBCH 31 and 45
Grain	0.09	3.57	0.07
Straw	3.20	2.76	68.6

Table 17 Distribution and nature of residues in wheat following application of radiolabelled myclobutanil

	Ph Grain	Tz Grain	Ph Straw	Tz Straw	Ph Straw
	Field	Field	Field	Field	Greenhouse ^b
Total Residue (mg/kg)	0.09	3.57	3.20	2.76	68.6
% Total Radioactive Residues (% TRR)					
myclobutanil	10.5	0.4	29.5	28.7	46.9
RH-9089	3.7	2.4	6.2	4.9	1.0
RH-9090	24.7	7.1	33.3	16.3	2.7
Glucoside	6.3	1.3	5.9	5.8	22.1
Malonate	3.8	0.5	1.9	1.3	10.1
RH-4098 (TAA)	—	25.4	—	15.5	—
RH-3968 (TA)	—	51.3	—	1.2	—
Other—organic ^a	41.9	9.0	21.0	24.7	15.0

	Ph Grain	Tz Grain	Ph Straw	Tz Straw	Ph Straw
	Field	Field	Field	Field	Greenhouse ^b
Other—aqueous	9.2	2.6	2.2	1.7	2.2

^a Radioactivity could not be assigned to a specific zone on TLC.

^b Parent and metabolites were quantitated in the straw of the greenhouse experiment.

Sugar beet

The metabolism of myclobutanil, radiolabelled in either the phenyl (specific activity 4.00 mCi/mmol, radiochemical purity 98%) or triazole ring (specific activity 6.10 mCi/mmol, radiochemical purity 98%), was studied in sugar beets (*Beta vulgaris* cv. Wildcat) at approximately twice the maximum commercial use rate (150 g ai/kg) and approximately 20 times the maximum use rate (1500 g ai/ha) 30 days prior to maturity (MacDonald, AMG, Gray, J, Coyle, D, 2004, 207048). Plants were harvested from each plot on the day of application (when the spray had dried), and at 15 and 30 days after treatment (DAT). All samples were stored frozen (−20 °C). Radioactivity was measured by liquid scintillation counting (LSC). Samples were extracted with acetonitrile, aqueous acetonitrile and where necessary by acid and base hydrolysis. The natures of radioactivity in the selected samples were investigated further with HPLC or by LC-MS. The results are summarized in Tables 18 to 20.

Table 18 TRR from testing with [¹⁴C]-phenyl-myclobutanil

Days After Treatment	150 g ai/ha		1500 g ai/ha	
	Raw Agricultural Commodity (mg eq/kg)			
	Roots	Leaves	Roots	Leaves
0	0.043	2.98	0.88	26.1
15	0.21	1.2	0.37	7.1
30	0.081	0.53	0.87	5.32

Table 19 TRR from testing with [¹⁴C]-triazole-myclobutanil

Days After Treatment	150 g ai/ha		1500 g ai/ha	
	Raw Agricultural Commodity (mg eq/kg)			
	Roots	Leaves	Roots	Leaves
0	0.13	2.70	1.50	16.0
15	0.072	0.92	2.49	10.1
30	0.044	0.69	1.19	4.57

Table 20 The distribution of TRR between the various components for shoot and roots at maturity (MacDonald, AMG, Gray, J, Coyle, D, 2004, 207048)

		PH	label			TZ	label	
Identified		leaves		roots		leaves		roots
components	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
myclobutanil	34.3	0.18	32.8	0.027	16.1	0.11	26.7	0.011
RH-9089	ND	ND	ND	ND	ND	ND	ND	ND
RH-9090	7.2	0.038	ND	ND	ND	ND	4.7	0.002
Glucose conjugate	42.4	0.223	8.4	0.007	61.6	0.427	8.9	0.004
Of RH-9090 uncharacterized components								
Unknown 3	ND	ND	10.5	0.008	ND	ND	ND	ND
Unknown 4	ND	ND	10.5	0.008	ND	ND	10.2	0.004
Unknown 5	ND	ND	ND	ND	ND	ND	5.5	0.002
Unknown 6	7.6	0.040	13.2	0.011	ND	ND	15.6	0.007

		PH	label			TZ	label	
Identified		leaves		roots		leaves		roots
components	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Unknown 7	ND	ND	ND	ND	ND	ND	ND	ND
Uncharacterized fractions								
Extractable	4.9	0.025	17.7	0.014	11.7	0.081	29.8	0.009
Non extractable	1.1	0.006	10.3	0.008	1.17	0.008	12.7	0.006
Recovered	97.5	0.51	103	0.083	90.5	0.63	105	0.045
Original	100	0.53	100	0.081	100	0.69	100	0.044

In summary, the metabolism of myclobutanil in crops is qualitatively consistent and considered comparable except in wheat treatment with TZ label. The conversion of myclobutanil to RH-9090 followed by conjugation with glucose is the major metabolic pathway. Minor amounts of RH-9089 are probably a result of oxidation of RH-9090. The presence of RH-3968 and RH-4098 in wheat treated with TZ label indicates that the phenethyl triazole linkage in parent was metabolically cleaved.

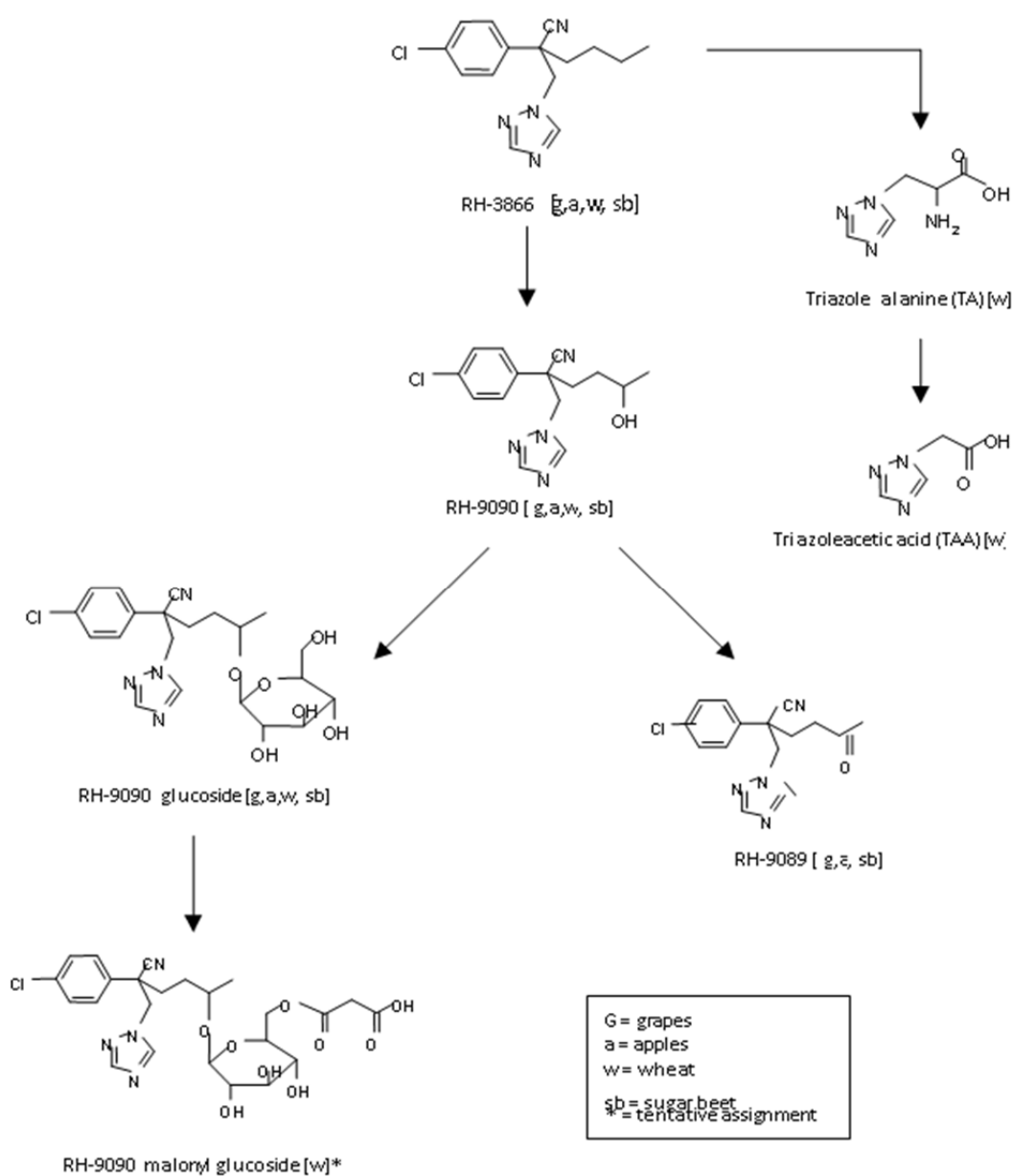


Figure 3 Proposed metabolic pathway of myclobutanil in crops

Environmental fate in soil

The Meeting received information on the environmental fate of myclobutanil in soil, including studies on soil metabolism, and crop rotational studies.

Soil metabolism (aerobic degradation)

The aerobic soil metabolism of myclobutanil was investigated in a silt loam (pH 5.3, 0.7% OC) freshly collected from the farm field and sieved through a 3.45 mm screen before use (Ackermann, IB, 1984, 94786; Ackermann, IB, 1986, 94822). The soil was treated with either ^{14}C -phenyl-myclobutanil (uniformly labelled in the chlorophenyl ring, 22616 DPM/ug, radiochemical purity 99%) or ^{14}C -triazole-myclobutanil (uniformly labelled in the triazole ring, 24380 DPM/ug, radiochemical purity 97%) at a nominal rate of 1 mg per kg dry soil. Soil aliquots of 200 g (dry weight basis) in the flasks were maintained aerobically for one year. The total CO_2 evolved by myclobutanil was measured with the trap solutions. On selected dates, soil samples were removed from the flasks to radioassay the ^{14}C content.

Soil samples were added acetonitrile/1 N acetic acid and placed on a mixer for thirty minutes. The suspension was centrifuged and transferred to a graduate cylinder. This procedure was performed twice. The suspension was vacuum filtered and two 1 mL aliquots were radioassayed. The filtrate was quantitatively transferred to a separatory funnel and water was added. Then it was partitioned three times with methylene chloride. The methylene chloride extract was evaporated for TLC analysis. Triplicate 0.5 gram samples of the air dried soil were radioassayed.

Thirty-five mL of 0.5 N NaOH was added to samples of aged aerobic soil and mixed for seven hours. The suspension was centrifuged and transferred to a graduate cylinder. Duplicate 0.5 mL aliquots were taken for radioassay. Thirty-five mL of 1.0 N NaOH were added to the remaining soil. Then it was mixed overnight (fifteen hours) with a mixer. The suspension was centrifuged and the supernatant was transferred to a graduate cylinder. The extracted soil was washed with 1.0 N NaOH and distilled water, respectively. The supernatant was added to the graduate. Duplicate 1 mL aliquots were taken for radioassay. The soil was dried and then pulverized. Triplicate 0.5 g samples were taken for radioassay. Results are presented in Tables 21 to 23.

Table 21 Distribution of radioactivity and material balance after application of ^{14}C -triazole-myclobutanil and ^{14}C -phenyl-myclobutanil

Day	^{14}C -triazole				^{14}C -phenyl			
	CH_2Cl_2	H_2O	CO_2	Remain in soil	CH_2Cl_2	H_2O	CO_2	Remain in soil
0	100	nd	nd	nd	97	nd	nd	nd
3	98	nd	nd	nd	97	nd	nd	nd
7	96	nd	nd	nd	95	nd	nd	nd
14	94	nd	nd	nd	93	nd	nd	nd
21	89	2	nd	9	88	nd	2	10
30	82	4	nd	14	83	nd	4	13
51	69	10	1	20	69	1	9	21
62	63	13	1	23	63	1	14	22
90	51	16	1	32	56	1	18	25
120	44	17	2	37	49	1	22	28
150	43	18	3	36	52	1	21	26
181	42	18	2	38	46	2	25	27
240	38	15	3	44	42	1	28	29
367	35	13	4	48	40	2	30	28

nd = Not detected

Table 22 Summary of distribution and characterization of radioactivity following extraction and mineralization after application of [¹⁴C]-triazole-myclobutanil

	1,2,4-triazole		Metabolites				
Day	(H ₂ O)	myclobutanil	Polar metabolite	Origin	Other	CO ₂	Unextracted
0	0	98	nd	2	nd	0	0
3	0	98	nd	2	nd	0	2
7	0	93	1	2	nd	0	4
14	0	87	5	2	nd	0	6
21	2	78	8	2	nd	0	9
30	4	71	9	2	nd	0	14
51	10	63	5	1	nd	1	20
62	13	54	4	5	nd	1	23
90	16	41	4	nd	5	1	31
120	17	42	nd	nd	3	2	38
150	18	41	nd	nd	nd	3	36
180	18	40	nd	nd	2	2	38
240	15	34	1	nd	3	3	44
367	13	29	nd	2	4	4	48

nd = Not detected.

Table 23 Summary of distribution and characterization of radioactivity following extraction and mineralization after application of [¹⁴C]-phenyl-myclobutanil

	1,2,4-triazole		Metabolites				
Day	(H ₂ O)	myclobutanil	Polar metabolite	Origin	Other	CO ₂	Unextracted
0	0	94	nd	3	nd	0	3
3	0	94	1	2	nd	0	3
7	0	91	2	1	1	0	5
14	0	85	6	1	1	1	6
21	0	82	5	1	nd	2	10
30	0	72	9	1	1	4	13
51	1	64	6	1	nd	10	21
62	1	54	7	2	nd	14	22
90	1	50	3	1	2	18	25
120	1	43	3	2	nd	22	28
150	1.5	47	3	1	nd	21	26
180	2	43	nd	nd	nd	25	27
240	1	37	2	nd	3	28	29
367	2	33	nd	3	4	30	28

nd = Not detected.

The majority of radioactivity in the extracts was always the unchanged compound. None of metabolites exceeded 10% TAR. The half-lives were calculated to be 61 to 71 days for degradation of parent compound. Mineralization to ¹⁴CO₂ reached a total of 4.0% to 30% TAR. No other volatile compounds were detected.

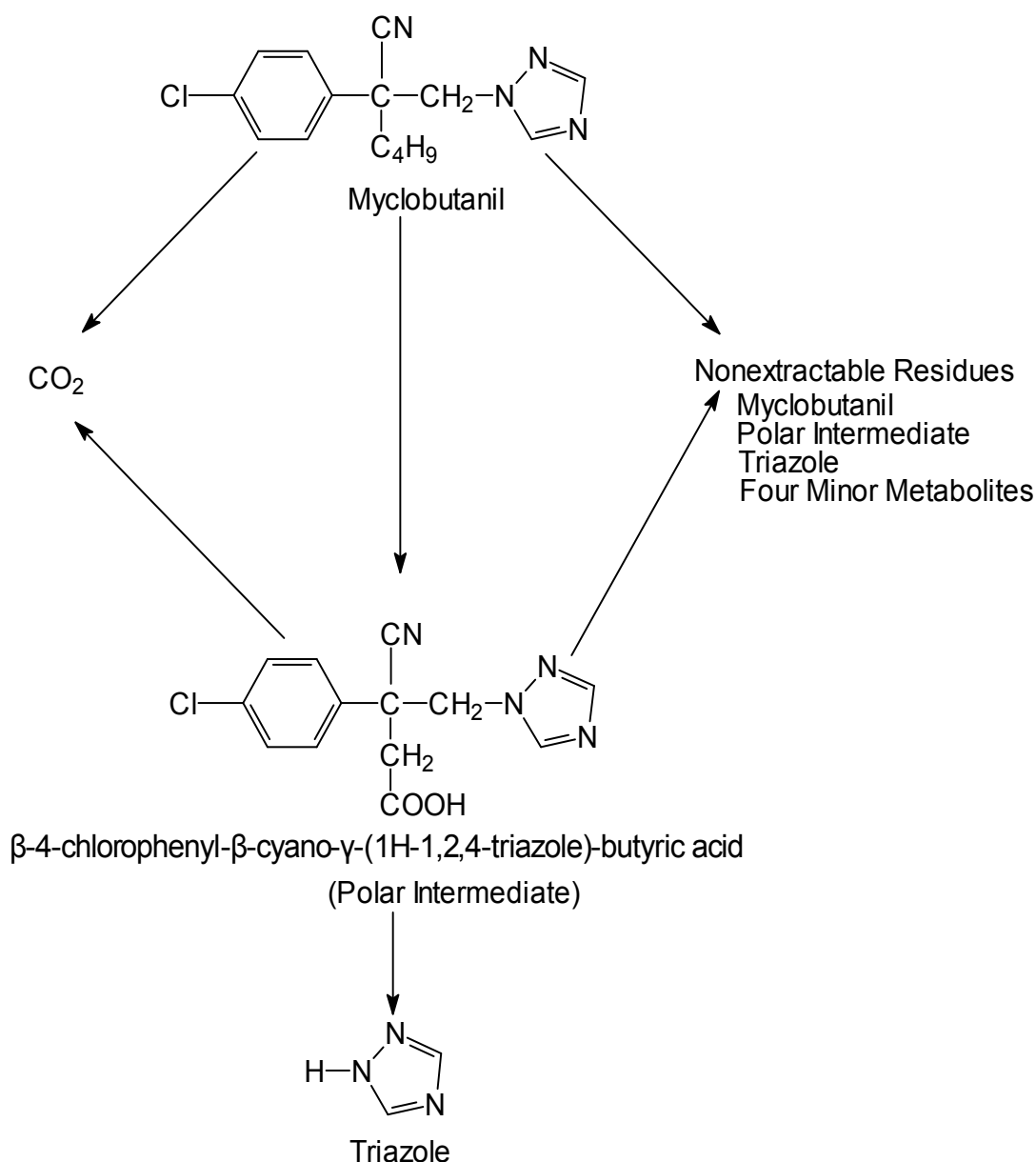


Figure 4 Proposed metabolic pathway of myclobutanil in aerobic soil

Soil Photolysis

The photodegradation of [¹⁴C]-myclobutanil (labelled uniformly in the phenyl ring, 10.28 mCi/g, radiochemical purity 98.0%; labelled in the 3,5 position of the triazole ring, 10.98 mCi/g, radiochemical purity 100%) were studied on a silt loam soil (1.20% OM, pH 5.3) under black lamps and fluorescent sun lamps (Nelson, SS, 1985, 94821; Nelson, SS, 1985, 222060). Either [¹⁴C]-phenyl-myclobutanil or [¹⁴C]-triazole-myclobutanil was fortified on the surface of the dry soil in flasks at the nominal concentration of 5 mg/kg dry soil. The incubation conditions were irradiation from an array of three fluorescent blacklights and three fluorescent sunlamps at 34 °C for 30 days. Samples were taken 0, 3, 7, 16 and 30 days after treatment. Dark control samples were analysed at the same sampling days. Volatiles were measured in appropriate trapping solutions. Soil samples were extracted with 70:30 acetonitrile/1 M acetic acid and the extracts analysed by liquid scintillation

counting (LSC) and GC-ECD or NPD. Bound residues were quantified by combustion and subsequent LSC measuring.

The light source in this study had a range of about 290–480 nm. The intensities of the wavelength emitted from the light source in the 290–480 nm range were greater than that found in natural sunlight. Myclobutanil did not photodegrade to any appreciable extent, even under the greater intensities of the shorter wavelengths. This study was done at 34 °C, somehow higher than the desired range of 20–30 °C. In this experiment the dark controls, which were maintained at the same temperature, were very stable. Comparison of the extracts from day 0 to day 30 dark controls does not indicate any substantial change in the amount of myclobutanil. The extracts from day 30 irradiated samples contained only 88.8% of phenyl ring labelled myclobutanil and 87.7% of triazole ring labelled myclobutanil. Thermal degradation of myclobutanil was slight or non-existent at this higher temperature. Comparison of studies from soil photolysis and metabolism, it is concluded that photolysis will not be a route of degradation of myclobutanil in the soil as soil metabolism will occur more rapidly. The results are shown in Tables 24 to 26.

Table 24 Distribution of radioactivity in silt loam soil after treatment with [phenyl- ^{14}C]-myclobutanil and incubation under irradiated conditions (% TAR)

Day	CO ₂	Volatiles	CHCl ₃ extract	Remaining aqueous	Non-extracted
0	0	0	95.8	0.07	4.1
3	0.02	0.004	91.7	0.07	8.2
7	0.03	0.002	89.5	0.1	10.4
16	0.3	0.008	94.1	0.2	5.5
30	1.9	0.01	89.7	0.6	7.8
30 dark	0.08	0.008	94.5	0.07	5.3

Table 25 Distribution of radioactivity in silt loam soil after treatment with [triazole- ^{14}C]-myclobutanil and incubation under irradiated conditions (% TAR)

Day	CO ₂	Volatiles	CHCl ₃ extract	Remaining aqueous	Non-extracted
0	0	0	95.8	0.06	4.3
3	0.02	0.004	93.5	0.1	6.4
7	0.02	0.002	91.7	0.2	8.1
16	0.05	0.002	94.3	0.4	5.3
30	0.2	0.003	90.8	1.4	7.7
30 dark	0.04	0	91.3	0.1	8.6

Table 26 TLC-analysis of soil extracts after treatment of silt loam soil with [^{14}C]-myclobutanil and incubation under irradiated conditions (%TAR)

Day	Phenyl ring	labelled ^a	Triazole ring	labelled ^a
	Parent	RH-9089	Parent	RH-9089
0	97.4	1.1	98.0	0.6
3	97.5	0.8	96.0	0.7
7	96.6	0.8	97.3	0.7
16	91.7	2.3	93.1	1.9
30	88.8	3.9	87.7	4.2
30 dark	96.0	0.9	96.9	0.7

^a Average from two different solvent systems.

Myclobutanil was very stable, even using these conditions which are more severe than those encountered in real sunlight. The above studies indicate that the major route of degradation of myclobutanil in the soil will be by soil metabolism and not photolysis.

Crop rotation studies

Information on the fate of myclobutanil in follow-on crop studies was made available to the Meeting.

A confined rotational crop study was conducted with [^{14}C]triazole-labelled myclobutanil (Graper, LK *et al.*, 2012, 090061). The active substance was soil-applied to confined plots of sandy loam soil at a rate of $1 \times 360 \text{ g ai/ha}$. The nature and the level of radioactive residues were investigated in lettuce (Variety: *butter crunch*), radish (Variety: *cherry belle*) and wheat (Variety: *hard red VNS*) after plant back intervals of 30, 120 and 365 days. Plant samples were harvested at maturity, and additional immature lettuce samples as well as wheat forage samples (in part dried to hay) were taken 30 days, 120 days and 365 days after planting or sowing, respectively. Soil (0.5% OM; pH 7.6) samples were taken after ploughing and after harvest of the mature crops for each plant back interval. The sampled material was stored in a freezer. All plant samples were homogenised and the radioactive residues in these samples and in the soil samples were determined by combustion analysis. Lettuce, radish and wheat samples were extracted with an accelerated solvent extractor using 80/20 acetonitrile/water. Extracts were measured with LSC and cleaned up using solid phase extraction (SPE) for HPLC analysis. The remaining tissue after ASE extraction was analysed by oxidative combustion to determine the amount of non-extractable radioactive residue.

Lettuce, radish and wheat were planted at rotational intervals of 30, 120 and 365 days after treatment and total radioactive residues ranging from 0.07 to 2.69 mg eq/kg were found in harvested crops. Residues in immature and mature lettuce and radish roots declined over time, while residues in radish tops increased. Residues in wheat hay, straw and grain did not show consistent increase or decline. All samples at all plant-back time points were subjected to extraction and characterization. For all samples, 62.9 to 104.0% of the TRR was extracted into neutral organic solvent which was analysed by SPE to give load & wash (polar) and eluate (non-polar) fractions requiring HPLC analysis. When the load & wash and eluate fractions were considered together, 63.0 to 105.8% of the sample TRR was analysed by HPLC. The three most abundant non-polar metabolites were myclobutanil at 0.43 mg eq/kg (55.2% TRR) in 30 DAT mature lettuce and MW 309 di-acid at 0.381 mg eq/kg (14.2% TRR) and RH-9090 at 0.47 mg eq/kg (17.3% TRR), both in 30 DAT wheat straw. The largest unidentified non-polar peak was 0.09 mg eq/kg (3.4% TRR) in 30 DAT wheat straw. The two most abundant polar metabolites were the triazole alanine at 0.45 mg eq/kg (30.1% TRR) and triazole acetic acid at 0.43 mg eq/kg (28.8% TRR), both in 120 DAT wheat grain. The highest concentration of the largest polar unidentified peak was 0.21 mg eq/kg (12.8% TRR) in 365 DAT wheat straw. Bound residues exceeded both 10% of the TRR and 0.05 mg eq/kg only in wheat hay, straw and grain at all plant-backs. For wheat grain, the bound residues ranged from 26.0 to 38.8% TRR and from 0.21 to 0.57 mg eq/kg.

Table 27 Total radioactive residues in crops after treatment with [^{14}C]-triazole-myclobutanil

	30 plant-back control	30 plant-back treated	120 plant-back treated	365 plant-back treated
Sample	mg eq/kg	mg eq/kg	mg eq/kg	mg eq/kg
Lettuce-immature	< 0.001	0.782	0.242	0.097
Lettuce-mature	0.001	0.938	0.240	0.067
Radish-mature tops	< 0.001	0.330	0.218	0.163
Radish-mature roots	< 0.001	0.078	0.098	0.137
Wheat Forage	< 0.001	0.533	0.468	0.228
Wheat Hay	< 0.001	1.504	1.668	1.444 ^a
Wheat Straw	0.001	2.692	1.715	1.629
Wheat Grain	< 0.001	0.559	1.500	1.457

^a As received, the ground and frozen 365 day plant-back wheat hay sample was comprised of 31.2% moisture. At this moisture, the sample residue level was calculated to be 1.168 mg eq/kg. Because OECD guidelines indicate that wheat hay should be dried to a moisture content of 10 to 20 percent, the mg eq/kg value in the table above was arithmetically adjusted as if the moisture was 15%, and the value used for the study was as shown above in the table.

Table 28 Accelerated solvent extraction (ASE) of residues in rotational crops after [¹⁴C]-triazole-myclobutanil treatment after plant back intervals of 30, 120 and 365 days

	Total	80/20 CH ₃ CN /H ₂ O, 40 °C	80/20 CH ₃ CN /H ₂ O, 40 °C	Total extractable	Non-extractable	Total recovered
Sample PBI	mg eq/kg (TRR%)	mg eq/kg (TRR%)	mg eq/kg (TRR%)	mg eq/kg (TRR%)	mg eq/kg (TRR%)	mg eq/kg (TRR%)
Immature	Lettuce					
30	0.782 (100.0)	0.770 (98.4)	0.014 (1.7)	0.784 (100.1)	0.024 (3.1)	0.807 (103.2)
120	0.242 (100.0)	0.211 (87.4)	0.005 (1.9)	0.216 (89.3)	0.017 (7.0)	0.233 (96.3)
365	0.097 (100.0)	0.098 (100.7)	0.003 (3.3)	0.101 (104.0)	0.007 (7.6)	0.109 (111.7)
Mature	Lettuce					
30	0.938 (100.0)	0.741 (78.9)	0.010 (1.0)	0.750 (80.0)	0.048 (5.1)	0.798 (85.1)
120	0.240 (100.0)	0.228 (95.2)	0.006 (2.4)	0.234 (97.6)	0.024 (10.0)	0.258 (107.6)
365	0.067 (100.0)	0.063 (93.8)	0.001 (1.2)	0.064 (95.0)	0.005 (7.1)	0.068 (102.1)
Radish	Tops					
30	0.330 (100.0)	0.305 (92.4)	0.011 (3.3)	0.316 (95.8)	0.010 (3.0)	0.326 (98.8)
120	0.218 (100)	0.203 (93.0)	0.005 (2.3)	0.208 (95.3)	0.008 (3.6)	0.216 (98.9)
365	0.163 (100.0)	0.140 (85.9)	0.007 (4.1)	0.147 (90.1)	0.008 (4.9)	0.155 (95.0)
Radish	Roots					
30	0.078 (100.0)	0.073 (93.2)	0.003 (3.6)	0.076 (96.8)	0.005 (6.3)	0.080 (103.0)
120	0.098 (100.0)	0.087 (88.5)	0.006 (6.0)	0.093 (94.5)	0.009 (9.2)	0.102 (103.7)
365	0.137 (100.0)	0.097 (70.6)	0.004 (3.0)	0.101 (73.7)	0.015 (11.2)	0.116 (84.9)
Wheat	Forage					
30	0.533 (100.0)	0.525 (98.5)	0.010 (1.9)	0.535 (100.4)	0.036 (6.8)	0.572 (107.2)
120	0.468 (100.0)	0.418 (77.9)	0.009 (1.9)	0.427 (91.1)	0.024 (5.0)	0.450 (96.2)
365	0.228 (100.0)	0.177 (77.9)	0.003 (1.4)	0.180 (79.3)	0.016 (7.1)	0.197 (86.4)
Wheat	Hay					
30	1.504 (100.0)	1.259 (83.7)	0.101 (6.7)	1.361 (90.5)	0.171 (11.4)	1.532 (101.9)
120	1.668 (100.0)	0.928 (55.7)	0.229 (13.7)	1.157 (69.4)	0.382 (22.9)	1.539 (92.3)
365	1.444 (100.0)	1.016 (70.4)	0.165 (11.4)	1.181 (81.8)	0.288 (20.0)	1.470 (101.8)
Wheat	Straw					
30	2.692 (100.0)	2.081 (77.3)	0.204 (7.6)	2.285 (84.9)	0.530 (19.7)	2.815 (104.6)
120	1.715 (100.0)	1.238 (72.2)	0.153 (8.9)	1.391 (81.1)	0.287 (16.7)	1.678 (97.8)
365	1.629 (100.0)	1.195 (73.4)	0.141 (8.7)	1.336 (82.0)	0.285 (17.5)	1.621 (99.5)
Wheat	Grain					
30	0.559 (100.0)	0.205 (36.7)	0.147 (26.2)	0.352 (62.9)	0.213 (38.1)	0.565 (101.1)
120	1.500 (100.0)	0.619 (41.2)	0.412 (27.5)	1.030 (68.7)	0.390 (26.0)	1.420 (94.7)
365	1.457 (100.0)	0.666 (45.7)	0.435 (29.9)	1.101 (75.6)	0.565 (38.8)	1.666 (114.4)

Table 29 SPE fractionation of the extractable residues in rotational crops after [¹⁴C]-triazole-myclobutanil treatment

	Analysis of extracts by SPE				Reconstituted Wash SPE for	Load & Fractions HPLC	Reconstituted SPE Fractions HPLC	Eluate for
	Total applied	Load & wash	Eluate	Total recovered	Sample for HPLC	Total recovered	Sample for HPLC	Total recovered
Sample PBI	mg eq/kg (TRR%)	mg eq/kg (TRR%)	mg eq/kg (TRR%)	mg eq/kg (TRR%)	mg eq/kg (TRR%)	mg eq/kg (TRR%)	mg eq/kg (TRR%)	mg eq/kg (TRR%)
Immature	Lettuce							
30	0.784 (100.1)	0.046 (5.9)	0.713 (91.1)	(96.9)	0.046 (5.9)	(99.2)	0.693 (88.6)	(97.2)
120	0.216 (89.3)	0.043 (18.0)	0.169 (69.9)	(98.4)	0.045 (18.8)	(104.5)	0.167 (69.3)	(99.0)
365	0.101 (104.0)	0.053 (53.9)	0.049 (50.0)	(99.9)	0.051 (52.2)	(96.8)	0.043 (43.7)	(87.4)
Mature	Lettuce							
30	0.750 (80.0)	0.099 (10.5)	0.634 (67.6)	(97.7)	0.097 (10.3)	(98.2)	0.715 (76.3)	(112.8)
120	0.234	0.079	0.152		0.075		0.153	

	Analysis of extracts by SPE				Reconstituted Wash SPE for	Load & Fractions HPLC	Reconstituted SPE Fractions HPLC	Eluate for
	Total applied	Load & wash	Eluate	Total recovered	Sample for HPLC	Total recovered	Sample for HPLC	Total recovered
Sample PBI	mg eq/kg (TRR%)	mg eq/kg (TRR%)	mg eq/kg (TRR%)	mg eq/kg (TRR%)	mg eq/kg (TRR%)	mg eq/kg (TRR%)	mg eq/kg (TRR%)	mg eq/kg (TRR%)
	(97.6)	(33.0)	(63.5)	(98.8)	(31.1)	(94.3)	(63.8)	(100.5)
365	0.064 (95.0)	0.030 (45.2)	0.032 (47.6)	(97.7)	0.028 (42.0)	(92.9)	0.030 (45.3)	(95.2)
Radish	Tops							
30	0.316 (95.8)	0.071 (21.4)	0.236 (71.7)	(97.2)	0.070 (21.1)	(98.6)	0.230 (69.7)	(97.3)
120	0.208 (95.3)	0.041 (18.9)	0.162 (74.2)	(97.7)	0.047 (21.7)	(114.9)	0.160 (73.3)	(98.8)
365	0.147 (90.1)	0.091 (55.8)	0.047 (28.6)	(93.6)	0.084 (51.5)	(92.3)	0.048 (29.1)	(101.7)
Radish	Roots							
30	0.076 (96.8)	0.025 (32.1)	0.040 (51.3)	(86.2)	0.030 (38.3)	(119.5)	0.042 (54.4)	(105.9)
120	0.093 (94.5)	0.046 (42.1)	0.031 (31.7)	(83.0)	0.045 (46.0)	(98.8)	0.030 (30.9)	(97.3)
365	0.101 (73.7)	0.058 (42.1)	0.039 (28.2)	(95.4)	0.055 (39.9)	(94.7)	0.036 (26.3)	(93.1)
Wheat	Forage							
30	0.535 (100.4)	0.060 (11.3)	0.474 (88.9)	(99.8)	0.059 (11.0)	(97.6)	0.506 (94.8)	(106.7)
120	0.427 (91.1)	0.111 (23.8)	0.293 (62.6)	(94.8)	0.105 (22.5)	(94.7)	0.262 (56.0)	(89.3)
365	0.180 (79.3)	0.093 (41.0)	0.081 (35.8)	(96.8)	0.087 (38.1)	(92.9)	0.081 (35.6)	(99.5)
Wheat	Hay							
30	1.361 (90.5)	0.225 (15.0)	1.110 (73.8)	(98.1)	0.230 (15.3)	(102.4)	1.072 (71.3)	(96.6)
120	1.157 (69.4)	0.550 (33.0)	0.531 (31.8)	(93.4)	0.530 (31.8)	(96.4)	0.520 (31.2)	(98.0)
365	1.181 (81.8)	0.614 (42.6)	0.609 (42.2)	(103.6)	0.556 (38.5)	(90.4)	0.538 (37.3)	(88.3)
Wheat	Straw							
30	2.285 (84.9)	0.293 (10.9)	1.979 (73.5)	(99.4)	0.293 (10.9)	(100.0)	1.936 (71.9)	(97.8)
120	1.391 (81.1)	0.502 (29.3)	0.854 (49.8)	(97.5)	0.523 (30.5)	(104.0)	0.904 (52.7)	(105.9)
365	1.336 (82.0)	0.562 (34.5)	0.703 (43.1)	(94.7)	0.550 (33.8)	(97.8)	0.673 (41.3)	(95.8)
Wheat	Grain							
30	0.352 (62.9)	0.321 (57.5)	0.048 (8.5)	(104.8)	0.323 (57.7)	(100.5)	0.046 (8.3)	(97.3)
120	1.030 (68.7)	0.969 (64.6)	0.057 (3.8)	(99.6)	0.958 (63.9)	(98.9)	0.057 (4.9)	(97.9)
365	1.101 (75.6)	1.006 (69.1)	0.073 (5.0)	(98.0)	0.957 (65.7)	(95.2)	0.071 (4.9)	(97.9)

Table 30 Myclobutanil and metabolite levels in SPE eluate fractions of lettuce and radish

	Immature lettuce mg eq/kg (TRR%)			Mature lettuce mg eq/kg (TRR%)			Radish tops mg eq/kg (TRR%)			Radish roots mg eq/kg (TRR%)		
Metabolites	30	120	365	30	120	365	30	120	365	30	120	365
Applied to HPLC	0.693 (88.6)	0.167 (69.3)	0.043 (43.7)	0.715 (76.3)	0.153 (63.8)	0.030 (45.3)	0.227 (68.9)	0.160 (73.3)	0.048 (29.1)	0.042 (54.4)	0.030 (30.9)	0.036 (26.3)
glu-9090	0.011 (1.4)	0.009 (3.6)	0.002 (2.3)	0.020 (2.1)	0.009 (3.7)	0.002 (3.7)	0.007 (2.0)	0.010 (4.7)	0.002 (1.3)	0.001 (1.2)	—	0.001 (0.5)

	Immature lettuce mg eq/kg (TRR%)			Mature lettuce mg eq/kg (TRR%)			Radish tops mg eq/kg (TRR%)			Radish roots mg eq/kg (TRR%)		
Metabolites	30	120	365	30	120	365	30	120	365	30	120	365
MW 309 di-acid	0.032 (4.1)	0.018 (7.5)	0.010 (10.1)	0.091 (9.7)	0.027 (11.4)	0.011 (15.7)	0.042 (12.7)	0.061 (28.1)	0.015 (9.3)	0.003 (4.3)	0.002 (1.9)	0.004 (3.0)
butyric acid	0.024 (3.0)	0.022 (9.1)	0.007 (7.4)	0.054 (5.8)	0.022 (9.3)	0.002 (2.3)	0.022 (6.6)	0.027 (12.2)	0.013 (7.9)	0.004 (5.7)	0.004 (4.1)	0.002 (1.6)
mal-glu-9090-2	0.021 (2.7)	0.014 (5.6)	0.001 (0.5)	0.039 (4.1)	—	0.001 (1.2)	0.033 (10.0)	0.006 (2.8)	0.002 (1.5)	0.001 (1.0)	0.001 (1.4)	—
RH-9090	0.018 (2.3)	0.023 (9.6)	0.004 (4.1)	0.056 (6.0)	0.021 (8.6)	0.003 (4.6)	0.065 (19.6)	0.012 (5.6)	0.002 (1.3)	0.010 (12.6)	0.005 (4.6)	0.004 (2.6)
MW-232 des-butyl	0.056 (7.2)	0.010 (4.0)	0.001 (1.5)	0.079 (8.4)	0.006 (2.3)	0.002 (2.6)	—	—	—	—	—	—
RH-9089	0.006 (0.8)	0.001 (0.3)	—	0.006 (0.7)	0.002 (0.7)	—	—	—	—	—	—	—
myclobutanil+16	0.042 (5.4)	0.012 (5.0)	0.003 (3.3)	0.067 (7.2)	0.008 (3.4)	0.002 (2.5)	0.003 (1.0)	0.001 (0.6)	—	0.001 (0.9)	—	—
MW 305 lactone	0.010 (1.3)	< 0.001 (0.1)	—	0.008 (0.9)	—	—	—	—	—	—	—	—
myclopentyl	—	0.001 (0.2)	—	0.003 (0.3)	—	—	—	—	—	—	—	0.001 (0.8)
myclobutanil	0.432 (55.2)	0.013 (5.5)	0.005 (5.1)	0.196 (20.8)	0.014 (5.9)	0.004 (6.3)	0.018 (5.3)	0.016 (7.5)	0.006 (3.5)	0.021 (26.9)	0.015 (15.6)	0.023 (16.7)
other	0.040 (5.1)	0.046 (18.8)	0.009 (9.3)	0.096 (10.2)	0.044 (18.5)	0.004 (6.5)	0.038 (11.7)	0.026 (11.8)	0.007 (4.3)	0.001 (1.8)	0.003 (3.2)	0.002 (1.1)

— = No detection.

Table 31 Myclobutanil and metabolite levels in SPE eluate fractions of wheat

	Wheat forage mg eq/kg (TRR%)			Wheat hay mg eq/kg (TRR%)			Wheat straw mg eq/kg (TRR%)			Wheat grain mg eq/kg (TRR%)		
Metabolites	30	120	365	30	120	365	30	120	365	30	120	365
Applied to HPLC	0.506 (94.8)	0.262 (56.0)	0.081 (35.6)	1.072 (71.3)	0.520 (31.2)	0.538 (37.3)	1.936 (71.9)	0.904 (52.7)	0.031 (1.9)	0.046 (8.3)	0.057 (3.8)	0.071 (4.9)
glu-9090	0.015 (2.8)	0.007 (1.5)	—	0.047 (3.1)	0.016 (1.0)	0.025 (1.8)	0.056 (2.1)	0.030 (1.7)	0.031 (1.9)	0.003 (0.5)	0.003 (0.2)	0.002 (0.1)
MW 309 di-acid	0.126 (23.7)	0.079 (17.0)	0.029 (12.8)	0.221 (14.7)	0.140 (8.4)	0.124 (8.6)	0.381 (14.2)	0.224 (13.1)	0.166 (10.2)	0.006 (1.0)	0.006 (0.4)	0.009 (0.6)
butyric acid	0.071 (13.3)	0.038 (8.2)	0.013 (5.9)	0.220 (14.6)	0.053 (3.2)	0.074 (5.1)	0.199 (7.4)	0.101 (5.9)	0.080 (4.9)	0.011 (2.0)	0.014 (0.9)	0.022 (1.5)
mal-glu-9090-1	0.061 (11.5)	—	—	0.152 (10.1)	0.040 (2.4)	0.050 (3.5)	0.138 (5.1)	0.073 (4.2)	0.054 (3.3)	—	—	—
mal-glu-9090-2	0.038 (7.1)	0.028 (6.0)	0.015 (6.4)	0.130 (8.6)	0.056 (3.4)	0.068 (4.7)	0.166 (6.2)	0.110 (6.4)	0.113 (6.9)	0.002 (0.4)	0.003 (0.2)	—
RH-9090	0.050 (9.4)	0.034 (7.3)	0.007 (3.0)	0.026 (1.7)	0.062 (3.7)	0.023 (1.6)	0.467 (17.3)	0.108 (6.3)	0.046 (2.8)	0.001 (0.2)	0.001 (< 0.1)	0.001 (0.1)
MW-232 des-butyl	0.002 (0.4)	—	—	0.009 (0.6)	—	0.013 (0.9)	—	—	0.006 (0.3)	0.003 (0.5)	0.003 (0.2)	0.005 (0.3)
RH-9089	—	—	—	0.012 (0.8)	0.003 (0.2)	0.004 (0.3)	0.063 (2.3)	0.019 (1.1)	0.015 (0.9)	< 0.001 (< 0.1)	—	—
myclobutanil+16	0.002 (0.4)	—	—	0.007 (0.5)	0.012 (0.7)	—	—	0.015 (0.9)	—	< 0.001 (0.1)	—	—
MW 305 lactone	0.003 (0.6)	0.002 (0.5)	0.001 (0.4)	0.011 (0.7)	0.002 (0.1)	—	—	0.010 (0.6)	—	< 0.001 (0.1)	—	—
myclopentyl	—	—	—	—	—	0.007 (0.5)	—	—	—	—	—	—
myclobutanil	0.065 (12.2)	0.028 (6.1)	0.007 (3.3)	0.042 (2.8)	0.008 (0.5)	0.010 (0.7)	0.058 (2.2)	0.020 (1.1)	0.008 (0.5)	—	—	—
other	0.072 (13.4)	0.044 (9.4)	0.009 (3.9)	0.196 (13.0)	0.127 (7.6)	0.140 (9.7)	0.408 (15.1)	0.196 (11.4)	0.156 (9.6)	0.019 (3.4)	0.027 (1.8)	0.033 (2.3)

— = No detection.

Table 32 Myclobutanil and metabolite levels in SPE load & wash fractions of lettuce and radish

Metabolites	Immature lettuce mg eq/kg (TRR%)			Mature lettuce mg eq/kg (TRR%)			Radish tops mg eq/kg (TRR%)			Radish roots mg eq/kg (TRR%)		
	30	120	365	30	120	365	30	120	365	30	120	365
Applied to HPLC	0.046 (5.9)	0.045 (18.8)	0.051 (52.2)	0.097 (10.3)	0.075 (31.1)	0.028 (42.0)	0.070 (21.1)	0.070 (21.7)	0.084 (51.5)	0.030 (38.3)	0.045 (46.0)	0.055 (39.9)
Triazole alanine	0.015 (1.9)	0.011 (4.5)	0.022 (22.8)	0.020 (2.1)	0.007 (2.9)	0.015 (22.6)	0.053 (16.2)	0.034 (15.5)	0.046 (28.4)	0.021 (27.2)	0.030 (31.0)	– (–)
Polar unknown	0.022 (2.8)	0.024 (10.0)	0.019 (19.9)	0.052 (5.5)	0.051 (21.1)	0.013 (19.4)	0.009 (2.6)	0.007 (3.2)	0.024 (14.4)	0.002 (3.0)	0.006 (6.1)	0.018 (13.0)
Triazole acetic acid	– (–)	0.002 (0.7)	– (–)	0.004 (0.5)	0.003 (1.2)	– (–)	– (–)	0.001 (0.6)	0.003 (2.1)	– (–)	– (–)	– (–)
Other	0.009 (1.2)	0.009 (3.7)	0.009 (9.4)	0.021 (2.3)	0.014 (5.9)	– (–)	0.008 (2.3)	0.005 (2.4)	0.011 (6.5)	0.006 (8.1)	0.009 (8.8)	0.037 (26.8)
Largest other	0.008 (1.0)	0.004 (1.6)	0.003 (3.0)	0.012 (1.3)	0.007 (2.9)	– (–)	0.003 (0.8)	0.002 (1.0)	0.003 (1.9)	0.001 (1.5)	0.003 (3.2)	0.030 (21.8)

– = No detection.

Table 33 Myclobutanil and metabolite levels in SPE load & wash fractions of wheat

Metabolites	Wheat forage mg eq/kg (TRR%)			Wheat hay mg eq/kg (TRR%)			Wheat straw mg eq/kg (TRR%)			Wheat grain mg eq/kg (TRR%)		
	30	120	365	30	120	365	30	120	365	30	120	365
Applied to HPLC	0.059 (11.0)	0.105 (22.5)	0.087 (38.1)	0.230 (15.3)	0.530 (31.8)	0.556 (38.5)	0.293 (10.9)	0.523 (30.5)	0.550 (33.8)	0.323 (57.7)	0.958 (63.9)	0.957 (65.7)
Triazole alanine	0.028 (5.3)	0.048 (10.2)	0.040 (17.8)	– (–)	0.176 (10.5)	0.215 (14.9)	0.089 (3.3)	– (–)	0.166 (10.2)	0.176 (31.5)	0.452 (30.1)	0.384 (26.4)
Polar unknown	0.018 (3.3)	– (–)	– (–)	0.075 (5.0)	0.126 (7.6)	0.171 (11.8)	0.122 (4.5)	0.180 (10.5)	0.208 (12.8)	0.009 (1.5)	0.023 (1.6)	0.069 (4.8)
Triazole acetic acid	0.005 (0.9)	0.034 (7.2)	0.026 (11.3)	0.041 (2.7)	0.186 (11.1)	0.142 (9.8)	0.076 (2.8)	0.160 (9.3)	0.135 (8.3)	0.120 (21.4)	0.432 (28.8)	0.371 (25.5)
Other	0.008 (1.5)	0.024 (5.1)	0.021 (9.0)	0.115 (7.6)	0.042 (2.5)	0.028 (1.9)	0.006 (0.2)	0.183 (10.7)	0.041 (2.5)	0.018 (3.3)	0.051 (3.4)	0.133 (9.1)
Largest other	0.007 (1.3)	0.010 (2.1)	0.009 (4.0)	0.037 (2.5)	0.021 (1.2)	0.015 (1.0)	0.006 (0.2)	0.124 (7.2)	0.017 (1.0)	0.018 (3.3)	0.036 (2.4)	0.035 (2.4)

– = No detection.

The parent compound, RH-9090 and its conjugates were found in most part of the four crops. The other two most abundant metabolites were MW 309 di-acid and butyric acid, which were not identified in the metabolism studies of crops.

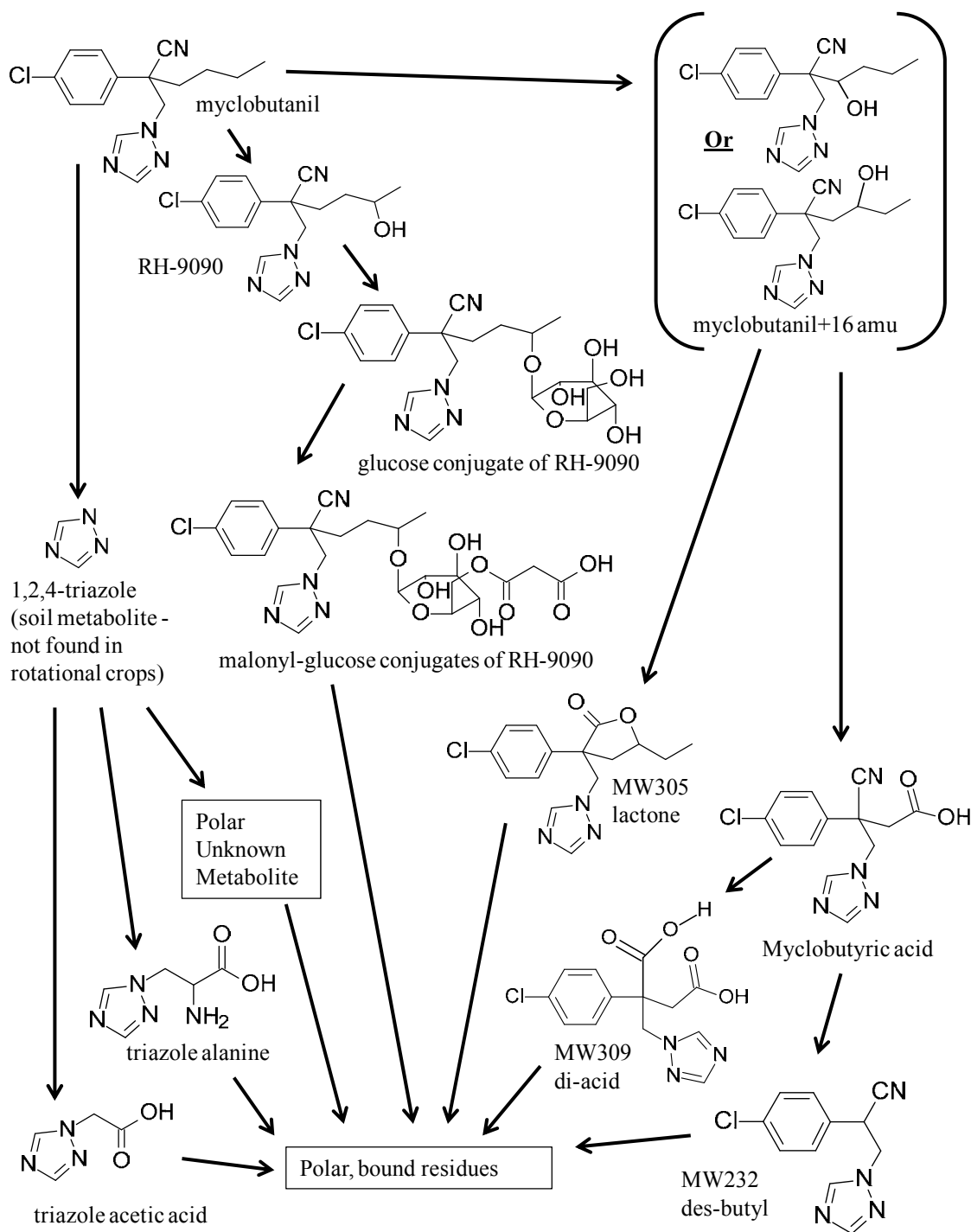


Figure 5 Proposed degradation pathway in ^{14}C -TZ labelled myclobutanil in the confined rotational crop study

RESIDUE ANALYSIS

Analytical methods

The Meeting received descriptions and validation data for analytical methods for residues of myclobutanil and RH-9090 (its alcohol metabolite) in plant and residues of myclobutanil in meat. Myclobutanil residues can be measured in most matrices to an LOQ of 0.01 to 0.05 mg/kg. No stereoselective methods were submitted for two myclobutanil enantiomers.

Analytical Method for the Measure of RH-3866 and RH-9090 Residues in Various Crops, Soil, Meat, Milk and Eggs. (Stavinski, SS *et al.*, 1988, 94356)

Analyte:	myclobutanil, RH-9090	GC/ECD or NPD
LOQ:	0.01–0.1 mg/kg	
Description	Samples are extracted by blending with methanol. However, wheat straw is extracted by soxhlet extraction with methanol and fat is extracted by refluxing in heptane. Water is added and the resulting solution is partitioned with hexane. The hexane is discarded and additional water is added to give a 50/50, v/v, water to methanol ratio. This solution is partitioned with methylene chloride. The aqueous phase is discarded and the methylene chloride is evaporated to dryness under diminished pressure. The residue is dissolved in toluene for Florisil column chromatography. After column chromatography, the eluent is evaporated to dryness and the residue is dissolved in 3% methanol/toluene for quantitation by GC-ECD. Wheat straw samples were quantitated with GC-NPD.	

Validation of the Residue Analytical Method for Myclobutanil (RH-3866) in Animal Tissues (Hen Muscle and Bovine Fat). (Wais, A, 2000, 94182)

Analyte:	myclobutanil	GC-ECD
LOQ:	0.01 mg/kg.	
Description	Step 1 (fat): The samples were extracted with n-heptane under heat reflux about 1 h. After cooling to room temperature the mixture was transferred into the separatory funnel and added methanol and sodium chloride solution. After shaking the n-heptane phase was discarded. The sample proceeded to step 2. Step 1 (muscle): The samples were extracted with methanol using high speed homogenizer. The mixture was filtered through a glass filter frit covered with Celite. The bottle and the filter cake were washed using methanol. The sample proceeded to step 2. Step 2. The sample solution was transferred into separatory funnel with addition of hexane and sodium chloride solution. The suspension was shaken for about 1 min. After shake and separation the lower aqueous layer was transferred into separatory funnel. The hexane layer was discarded. The sample was shaken with addition of sodium chloride solution and dichloromethane. The dichloromethane layer was filtered over sodium sulphate into a flask. The solution was evaporated to complete dryness. Step 3. The extraction residue was redissolved in toluene. The sample solution was transferred onto the Florisil column and repeated this procedure. Myclobutanil was eluted using a methanol/toluene mixture. The solution was evaporated to complete dryness. The residue was dissolved using a methanol/toluene mixture for determination by GC-ECD.	

Determination of Residues of Myclobutanil and Its Alcohol Metabolite (RH-9090) in Apples, Tomatoes, Grapes, Soybeans, Wheat and Radishes by Liquid Chromatography with Tandem Mass Spectrometry. (Shackelford, DD, *et al.*, 2003, 137270).

Analyte:	myclobutanil, RH-9090	LC-MS/MS	GRM 03.01
LOQ:	0.01 mg/kg.		
Description	Residues of myclobutanil and its alcohol metabolite (RH-9090) are extracted from the crop samples by homogenizing and shaking overnight with methanol. An aliquot is hydrolysed to release conjugated RH-9090 by adding concentrated hydrochloric acid and heating for 2 hours. The sample is then evaporated to dryness, and the residues reconstituted in an 80% water/20% methanol solution. The sample is applied to a SPE column. The column is washed with a 70% water/30% methanol solution, and the analytes are eluted from the column with a 80% methanol 20% water solution. The eluate is then evaporated to dryness, and the residues are reconstituted in a 97% methyl-t-butyl ether/2% methanol/1% acetic acid solution and applied to an NH ₂ SPE column. The eluate is collected and evaporated to dryness, and the residues reconstituted in a 70% water/ 30% methanol mobile phase containing 5 mM ammonium acetate and analysed with LC/MS/MS (APCI mode).		

Validation Report for Method GRM 03.01–Determination of Residues of Myclobutanil and its Alcohol Metabolite (RH-9090) in Apples, Tomatoes, Grapes, Soybeans, Wheat and Radishes by Liquid Chromatography with Tandem Mass Spectrometry. (Shackelford, DD, 2003, 137270)

Analyte:	Myclobutanil and RH-9090	HPLC/MS/MS	GRM 03.01
LOQ:	0.01 mg/kg.		

Description	The method mentioned above is applicable for the quantitative determination of residues of myclobutanil and its alcohol metabolite (RH-9090) in whole apples, whole tomatoes, whole grapes, radishes (roots and tops), wheat forage and soybean forage, which are representative of 'wet' crops. The method is also applicable for the quantitative determination of residues of myclobutanil and its alcohol metabolite (RH-9090) in soybeans (seed), a representative 'oily' crop, and in wheat grain, wheat hay and wheat straw, which are representative of 'dry' crops. The method was validated over the concentration range of 0.010–0.50 µg/g.		
Independent Laboratory Validation of Method GRM 03.01—Determination of Residues of Myclobutanil and its Alcohol Metabolite (RH-9090) in Apples, Tomatoes, Grapes, Soybeans, Wheat and Radishes by Liquid Chromatography with Tandem Mass Spectrometry. (Reed, RL, 2003, 137271)			
Analyte:	Myclobutanil and RH-9090	HPLC/MS/MS	GRM 03.01
LOQ:	0.01 mg/kg.		
Description	The method mentioned above is applicable for the quantitative determination of residues of myclobutanil and its alcohol metabolite (RH-9090) in whole tomatoes and wheat grain.		
Independent Laboratory Validation of the Multi-Residue Method-1 (MRM-1) for the Determination of Myclobutanil in Crops. (Mollica, J and West, S, 2003, 135042)			
Analyte:	myclobutanil	LC-MS/MS	The Netherland's MRM-1
LOQ:	0.2 mg/kg.		
Description	A sample of the plant material was homogenized with acetone. Dichloromethane and petroleum ether were added and the mixture was homogenized again. The sample was centrifuged and the organic extract was decanted through sodium sulphate to remove any remaining water. The extract was transferred to a volumetric flask and brought to volume with the acetone/dichloromethane/petroleum ether solution. For the apple extracts an OPC clean-up procedure was employed. Cyclohexane/ethyl acetate was used as mobile phase. Fractions were collected and then rotary evaporated to dryness under a gentle stream of nitrogen. Final extracts were reconstituted and analysed by GC/MS.		
Assessment and Validation of the Multi-Residue Enforcement Method DFG S19 for the Determination of Myclobutanil and its 1,2,4-Triazole Metabolite in Soil and the RH-9090 Metabolite in Animal Tissue. (Teasdale, R, 2003, 34429)			
Analyte:	Myclobutanil, 1,2,4-triazole metabolite and RH-9090 metabolite	LC-MS/MS	DSG S19
LOQ:	0.01 mg/kg.		
Description	Milk, meat, liver and kidney: Water was added to the sample which washomogenized followed by the addition of acetone and further homogenization. Sodium chloride and ethyl acetate/cyclohexane were added and the sample homogenized again. An aliquot of the upper organic phase was filtered through glass wool covered with sodium sulphate. The filter and the measuring cylinder were rinsed four times with portions of ethyl acetate/cyclohexane. The filtrate was evaporated. Ethyl acetate was added to the concentrated residue which was then ultrasonicated. Sodium sulphate/sodium chloride was added and the flask was swirled. Cyclohexane was added and the flask was swirled vigorously again. The extract was filtered for storage until GPC clean-up. Fat and whole egg: The sample of homogenised matrix was extracted with acetone and acetonitrile and macerated. The homogenate was then suction-filtered through filter paper in a Buchner funnel under a gentle vacuum to minimize filtrate evaporation. The filtrate was then filtered into a graduated cylinder. An aliquot of the filtrate was measured and transferred with acetone rinsings into a round bottom flask. Tri-methyl pentane was added and the solution was concentrated using rotary evaporation with a water bath. The last traces of solvent were removed using a gentle stream of air at room temperature. The residue was dissolved in ethyl acetate/cyclohexane. Once all substrates were cleaned up by GPC they were adjusted to a final volume and quantitated by GC-ECD.		
Validation of Method GRM 05.07.R1-Determination of Residues of Myclobutanil and its RH-9090 Alcohol Metabolite in Soybean Commodities using Online Solid Phase Extraction Coupled to High Performance Liquid Chromatography with Tandem Mass Spectrometry. (Shackelford, DD and Olberding, EL, 2013, 208707)			
Analyte:	Myclobutanil and RH-9090	LC-MS/MS	GRM: 05.07.R1
LOQ:	0.01 mg/kg.		

Description Residues of myclobutanil and its alcohol metabolite (RH-9090) are extracted from the sample matrix sample by homogenizing and shaking overnight with methanol. An aliquot is hydrolyzed to release conjugated RH-9090 by adding concentrated hydrochloric acid and heating for 2 hours at approximately 70 °C. The pH is adjusted to near neutral using 0.5 M aqueous Tris base. The sample is filtered, and the final solution is analysed using online solid phase extraction coupled to LC/MS/MS (APCI mode).

Method Validation Report for Method GRM 07.06—Determination of Residues of Myclobutanil and Its RH-9090 Alcohol Metabolite in Agricultural Commodities using On-line Solid Phase Extraction Coupled to Liquid Chromatography with Tandem Mass Spectrometry. (Shackelford, DD, 2008, 260120)

Analyte: Myclobutanil and RH-9090 LC-MS/MS GRM 07.06

LOQ: 0.01 mg/kg.

Description Residues of myclobutanil and its alcohol metabolite (RH-9090) are extracted from the crop samples by homogenizing and shaking overnight with methanol. An aliquot is hydrolyzed to release conjugated RH-9090 by adding concentrated hydrochloric acid and heating for 2 hours at approximately 70 °C. The pH is adjusted to near neutral using a 0.5 M aqueous Tris base solution. The sample is filtered, and the final solution is analysed using on-line solid phase extraction coupled to LC/MS/MS (APCI mode).

Summaries of analytical methods and procedural recoveries are presented in Table 34 to Table 43.

Table 34 Recoveries of myclobutanil and RH-9090 added to sample substrate (Stavinski, S and Brackett, C, 1988, 94356)

Matrix	Analyte	LOQ	Fortification Range	n =	Recovery Range	Mean
Apples	myclobutanil	0.01	0.01–0.10	8	74–100	89
Grapes	myclobutanil	0.01	0.01–0.10	6	70–94	87
Raisins	myclobutanil	0.04	0.04–0.05	2	65–88	77
Wine	myclobutanil	0.005	0.005–0.025	8	86–98	90
Wheat Grain	myclobutanil	0.01	0.01–1.0	10	83–100	89
Wheat Straw	myclobutanil	0.1	0.10–5.0	6	83–100	88
Liver Tissue	myclobutanil	0.01	0.01	2	83–87	85
Kidney Tissue	myclobutanil	0.01	0.01–0.05	2	82–91	87
Fat Tissue	myclobutanil	0.01	0.01–0.05	3	76–88	82
Muscle Tissue	myclobutanil	0.01	0.01–0.10	2	73–83	78
Milk	myclobutanil	0.01	0.01	3	68–85	79
Eggs	myclobutanil	0.01	0.01–0.05	4	85–103	91
Apples	RH-9090	0.02	0.02	2	85–92	89
Grapes	RH-9090	0.02	0.02	2	98–109	103
Raisins	RH-9090	0.04	0.04–0.05	2	65–88	77
Wine	RH-9090	0.2	0.2–0.5	4	89–96	94
Wheat Grain	RH-9090	0.01	0.01–0.2	3	85–97	93
Wheat Straw	RH-9090	0.2	0.2–1	6	76–102	91

Table 35 Recovery of myclobutanil added to sample substrate (Wais, A, 2000, 94182)

Matrix	Analyte	Fortification (mg/kg)	n =	Recovery Range (%)	Mean Recovery(%)	%RSD
Hen Muscle	myclobutanil	0.01	5	101–106	104	2
	myclobutanil	0.05	5	89–107	98	8
	myclobutanil	0.1	5	89–94	92	2

Matrix	Analyte	Fortification (mg/kg)	n =	Recovery Range (%)	Mean Recovery(%)	%RSD
Bovine Fat	myclobutanil	0.01	5	71–81	78	6
	myclobutanil	0.05	5	80–84	82	2
	myclobutanil	0.1	5	80–82	81	1

Table 36 Recovery summary of myclobutanil and RH-9090 from apples, tomatoes, grapes, soybean matrices, wheat matrices and radishes (Shackelford, DD, *et al.*, 2003, 137270)

matrix	myclobutanil					RH-9090				
	Spiked rate mg/kg	Recover range (%)	Average recovery (%)	RSD (%)	n	Spiked rate mg/kg	Recover range (%)	Average recovery (%)	RSD (%)	n
Apples	0.01	82–106	99	7.7	8	0.01	88–112	98	9.2	8
	0.05	97–103	100	3.1	3	0.05	87–92	90	2.9	3
	0.25	98–104	101	3.1	3	0.25	102–106	103	2.2	3
	0.50	97–107	103	3.5	6	0.50	85–103	95	8.8	6
	0.01–0.50	82–107	100	5.5	20	0.01–0.50	85–112	97	8.4	20
Tomatoes	0.01	86–105	98	7.4	8	0.01	85–113	98	11.3	8
	0.05	96–99	97	1.6	3	0.05	84–87	86	1.8	3
	0.25	103–108	105	2.4	3	0.25	103–105	104	1.1	3
	0.50	92–104	97	4.8	6	0.50	84–102	94	6.6	6
	0.01–0.50	86–108	99	5.9	20	0.01–0.50	84–113	96	9.6	20
Grapes	0.01	85–102	94	7.6	8	0.01	82–100	90	5.8	8
	0.05	93–97	96	2.4	3	0.05	100–102	101	1.1	3
	0.25	80–89	86	5.8	3	0.25	82–88	85	3.5	3
	0.50	82–91	88	5.1	6	0.50	81–103	92	11.8	6
	0.01–0.50	80–102	91	7.3	20	0.01–0.50	81–103	91	8.8	20
Radish	0.01	75–113	91	15.4	8	0.01	85–108	95	7.5	8
	0.05	100–104	102	1.8	4	0.05	87–94	90	4.2	4
	0.25	92–97	95	2.3	4	0.25	95–104	98	4.3	4
Root and Tops	0.50	84–102	94	6.3	8	0.50	88–105	99	5.5	8
	0.01–0.50	75–113	94	9.9	24	0.01–0.50	85–108	96	6.5	24
Wheat Grain	0.01	87–111	102	8.0	8	0.01	97–120	110	5.9	8
Soybean Seed	0.05	94–113	107	7.0	5	0.05	96–121	113	9.1	5
	0.25	101–108	105	3.4	4	0.25	82–112	100	14.7	4
Wheat and Soybean Forage	0.50	83–110	96	12.0	6	0.50	95–114	102	7.1	6
	0.01–0.50	83–113	102	8.7	23	0.01–0.50	82–121	107	9.5	23
Wheat hay and Straw	0.01	78–104	93	10.1	8	0.01	93–120	110	5.9	8
	0.05	98–102	100	1.8	4	0.05	100–114	110	6.1	4
Soybean Hay	0.25	100–102	101	1.0	4	0.25	103–106	104	1.4	4
	0.50	76–104	92	12.7	6	0.50	87–122	102	13.4	6
Wheat hay and Soybean Hay	0.01–0.50	76–104	95	9.2	22	0.01–0.50	87–122	105	8.1	22
	0.01	63–108	87	17.1	11	0.01	90–127	104	10.3	12
Wheat hay and Soybean Hay	0.05	86–98	93	6.9	3	0.05	112–120	117	3.7	3
	0.25	81–97	87	10.3	3	0.25	96–105	101	4.7	3
Soybean Hay	0.50	75–101	88	12.5	5	0.50	90–107	97	8.5	5
	0.01–0.50	63–108	88	13.8	22	0.01–0.50	90–127	104	10.1	23

Table 37 Recovery summary of myclobutanil and RH-9090 from wheat grain and tomato (Reed, RL, 2003, 137271)

Matrix	Analyte	Fortification (mg/kg)	n =	Recovery Range (%)	Mean Recovery (%)	%RSD
	myclobutanil	0.01	5	91–106	98	6
		0.02	3	102–104	103	1
Wheat Grain		0.10	5	96–106	102	4
	RH-9090	0.01	5	66–87	76	14
		0.02	3	80–91	86	6

Matrix	Analyte	Fortification (mg/kg)	n =	Recovery Range (%)	Mean Recovery (%)	%RSD
		0.10	5	67–98	87	15
	myclobutanil	0.01	5	85–94	89	4
Tomato		0.30	5	98–105	101	2
	RH-9090	0.01	5	70–82	77	6
		0.30	5	83–99	92	7

Table 38 Recovery summary of myclobutanil from apples, grapes (Mollica, J and West, S, 2003, 135042)

Matrix	Analyte	Fortification (mg/kg)	n =	Recovery Range (%)	Mean Recovery (%)	%RSD
Apples	myclobutanil	0.2	5	80–92	85	6
		2	5	83–99	94	7
Grapes	myclobutanil	0.2	5	73–86	78	6
		2	5	74–78	77	2

Table 39 Recovery summary of RH-9090 from animal commodities (meat, milk, liver, kidney, fat and eggs) (Teasdale, R., 2003, 34429)

Matrix	Analyte	Fortification (mg/kg)	n =	Recovery Range (%)	Mean Recovery (%)	%RSD
Milk	RH-9090	0.01	4	68–75	70	5
		0.10	5	69–92	77	12
Egg	RH-9090	0.01	5	74–88	81	7
		0.10	5	75–88	83	7
Meat	RH-9090	0.01	5	87–113	105	10
		0.10	5	83–104	92	9
Kidney	RH-9090	0.01	5	74–91	81	8
		0.10	5	91–109	104	7
Liver	RH-9090	0.01	5	75–97	87	10
		0.10	5	74–81	77	4
Fat	RH-9090	0.01	5	0–8	3	143
		0.10	5	1–16	9	67

Table 40 Summary of recovery of myclobutanil from agricultural commodities using the quantitation MS/MS transition Q1/Q3 m/z 289/70 and the confirmation MS/MS transition Q1/Q3 /z 289/125 (Shackelford, DD and Olberding, EL, 2013, 208707)

matrix	Q1/Q3 m/z 289/70					Q1/Q3 m/z 289/125				
	Spiked rate mg/kg	Recover range (%)	Average recovery (%)	RSD (%)	n	Spiked rate mg/kg	Recover range (%)	Average recovery (%)	RSD (%)	n
soybean	0.01	80–112	98	9.3	11	0.01	82–112	99	10.9	11
forage	0.50	101–114	107	5.4	6	0.50	101–115	106	5.7	6
	20.0	94–107	102	4.6	5	20.0	90–101	98	4.6	5
soybean	0.01	80–104	88	8.5	11	0.01	81–112	96	13.0	11
hay	0.50	94–106	100	5.2	6	0.50	92–104	99	5.2	6
	20.0	96–107	101	4.0	5	20.0	95–98	96	1.1	5
soybean	0.01	92–107	98	4.8	8	0.01	82–99	88	6.7	8
seed	0.50	101–112	107	4.1	6	0.50	100–110	105	3.9	6
processed commodities										
hulls	0.01	104–121	111	6.3	6	0.01	95–110	103	5.0	6
	0.50	107–114	111	2.6	5	0.50	107–116	111	3.1	5
meal	0.01	87–105	93	7.0	6	0.01	75–101	87	10.0	6
	0.50	94–115	106	8.8	5	0.50	92–110	101	8.5	5
oil	0.01	97–110	103	5.9	6	0.01	94–103	99	3.5	6
	0.50	108–115	111	2.3	6	0.50	108–115	112	2.6	6
aspirated	0.01	79–95	87	6.0	6	0.01	80–109	95	10.0	6

matrix	Q1/Q3 m/z 289/70					Q1/Q3 m/z 289/125				
	Spiked rate mg/kg	Recover range (%)	Average recovery (%)	RSD (%)	n	Spiked rate mg/kg	Recover range (%)	Average recovery (%)	RSD (%)	n
grain	0.50	114–130	120	4.8	6	0.50	111–128	119	4.9	6

Table 41 Summary of recovery of RH-9090 from agricultural commodities using the quantitation MS/MS transition Q1/Q3 m/z 305/125 and the confirmation MS/MS transition Q1/Q3 m/z 305/70 (Shackelford, DD and Olberding, EL, 2013, 208707)

matrix	Q1/Q3 m/z 305/125					Q1/Q3 m/z 305/70				
	Spiked rate mg/kg	Recover range (%)	Average recovery (%)	RSD (%)	n	Spiked rate mg/kg	Recover range (%)	Average recovery (%)	RSD (%)	n
soybean	0.01	80–101	91	7.7	11	0.01	72–105	87	10.1	11
forage	0.50	93–101	96	3.5	6	0.50	93–102	96	3.9	6
	20.0	90–95	92	2.3	5	20.0	91–100	97	3.7	5
soybean	0.01	81–107	92	8.3	11	0.01	74–132	93	19.0	11
hay	0.50	72–115	95	19.5	6	0.50	75–116	96	19.0	6
	20.0	93–97	95	1.9	5	20.0	92–99	94	2.7	5
soybean	0.01	64–92	78	12.3	8	0.01	66–88	76	11.0	8
seed	0.50	80–99	86	8.2	6	0.50	79–99	86	8.1	6
processed commodities										
hulls	0.01	83–101	93	7.4	6	0.01	88–112	99	9.2	6
	0.50	99–107	102	3.0	5	0.50	98–109	102	4.5	5
meal	0.01	75–90	83	5.7	6	0.01	88–101	92	5.3	6
	0.50	82–103	89	10.0	5	0.50	79–98	90	7.8	5
oil	0.01	94–113	103	7.4	6	0.01	91–116	102	8.7	6
	0.50	107–115	111	2.4	6	0.50	111–120	115	2.7	6
aspirated	0.01	74–87	81	5.5	6	0.01	74–111	92	14.1	6
grain	0.50	108–119	115	3.8	6	0.50	115–130	121	4.0	6

Table 42 Summary of recovery of myclobutanil in crops using the quantitation MS/MS transition Q1/Q3 m/z 289/70 and the confirmation MS/MS transition Q1/Q3 m/z 289/125 (Shackelford, DD, 2008, 260120)

matrix	Q1/Q3 m/z 289/70					Q1/Q3 m/z 289/125				
	Spiked rate mg/kg	Recover range (%)	Average recovery (%)	RSD (%)	n	Spiked rate mg/kg	Recover range (%)	Average recovery (%)	RSD (%)	n
	0.01	88–102	93	5.9	5	0.01	82–97	92	6.6	5
apple	2.00	94–101	99	2.8	5	2.00	94–102	98	3.3	5
	0.01–2.00	88–102	96	5.2	10	0.01–2.00	82–102	95	5.9	10
	0.01	70–95	83	12.7	5	0.01	81–115	96	13.5	5
apricot	2.00	86–104	95	8.9	5	2.00	86–100	94	6.9	5
	0.01–2.00	70–104	89	6.7	10	0.01–2.00	81–115	95	10.3	10
	0.01	81–86	84	3.0	5	0.01	83–107	93	9.9	5
cucumber	2.00	93–97	95	1.9	5	2.00	94–104	97	4.3	5
	0.01–2.00	81–97	89	6.7	10	0.01–2.00	83–107	95	7.5	10
green	0.01	96–111	103	6.8	5	0.01	93–112	100	7.8	5
pepper	2.00	96–106	102	4.0	5	2.00	103–106	104	1.1	5
	0.01–2.00	96–111	103	5.3	10	0.01–2.00	93–112	102	5.6	10
melon	0.01	81–91	88	4.8	5	0.01	67–101	89	16.2	5
(peel)	2.00	94–109	99	5.8	5	2.00	96–111	100	6.6	5
	0.01–2.00	81–109	93	8.2	10	0.01–2.00	67–111	94	12.8	10
melon	0.01	77–115	96	18.3	4	0.01	92–118	104	12.0	4
(pulp)	2.00	79–97	89	7.9	5	2.00	78–98	90	8.9	5
	0.01–2.00	77–115	92	13.5	9	0.01–2.00	78–118	96	12.4	9
	0.01	89–103	98	5.6	5	0.01	94–98	96	1.6	5
peach	2.00	76–103	91	12.6	5	2.00	76–103	91	13.6	5
	0.01–2.00	76–103	95	9.8	10	0.01–2.00	76–103	94	9.3	10

matrix	Q1/Q3 m/z 289/70					Q1/Q3 m/z 289/125				
	Spiked rate mg/kg	Recover range (%)	Average recovery (%)	RSD (%)	n	Spiked rate mg/kg	Recover range (%)	Average recovery (%)	RSD (%)	n
	0.01	76–92	84	9.2	5	0.01	76–95	88	8.6	5
strawberry	2.00	70–93	87	11.2	5	2.00	70–94	87	10.9	5
	0.01–2.00	70–93	86	9.8	10	0.01–2.00	70–95	87	9.3	10
	0.01	93–100	97	4.0	3	0.01	74–97	88	14.0	5
tomato	2.00	79–101	95	9.4	5	2.00	75–100	93	11.1	5
	0.01–2.00	79–101	96	7.5	8	0.01–2.00	74–100	91	11.7	10

Table 43 Summary of recovery of RH-9090 in crops using the quantitation MS/MS transition Q1/Q3 m/z 305/125 and the confirmation MS/MS transition Q1/Q3 m/z 305/70 (Shackelford, DD, 2008, 260120)

matrix	Q1/Q3 m/z 305/125					Q1/Q3 m/z 305/70				
	Spiked rate mg/kg	Recover range (%)	Average recovery (%)	RSD (%)	n	Spiked rate mg/kg	Recover range (%)	Average recovery (%)	RSD (%)	n
	0.01	94–107	99	5.3	5	0.01	73–96	89	10.7	5
apple	2.00	94–99	97	1.8	5	2.00	93–98	96	2.1	5
	0.01–2.00	94–107	98	3.9	10	0.01–2.00	73–98	92	8.1	10
	0.01	82–101	91	7.8	5	0.01	63–97	80	16.7	5
apricot	2.00	81–100	92	10.3	5	2.00	81–103	94	11.5	5
	0.01–2.00	81–101	92	8.6	10	0.01–2.00	63–103	87	15.5	10
	0.01	84–92	86	3.7	5	0.01	87–97	91	4.1	5
cucumber	2.00	93–102	97	3.6	5	2.00	92–105	98	4.9	5
	0.01–2.00	84–102	92	7.1	10	0.01–2.00	87–105	95	6.0	10
green	0.01	76–112	100	16.2	5	0.01	72–101	87	15.1	5
pepper	2.00	93–104	99	5.0	5	2.00	92–102	98	3.7	5
	0.01–2.00	76–112	100	11.3	10	0.01–2.00	72–102	93	11.6	10
melon	0.01	73–101	88	15.1	5	0.01	79–104	93	10.9	5
(peel)	2.00	93–109	97	6.6	5	2.00	94–106	97	5.0	5
	0.01–2.00	73–109	93	12.0	10	0.01–2.00	79–106	95	8.2	10
melon	0.01	85–93	89	4.0	4	0.01	81–98	92	8.4	4
(pulp)	2.00	78–93	88	6.7	5	2.00	81–97	89	6.4	5
	0.01–2.00	78–93	88	5.4	9	0.01–2.00	81–98	90	7.0	9
	0.01	89–104	93	6.8	5	0.01	91–108	100	6.8	5
peach	2.00	72–102	88	13.4	5	2.00	67–97	86	14.6	5
	0.01–2.00	72–104	91	10.3	10	0.01–2.00	67–108	93	12.8	10
	0.01	72–102	86	14.1	5	0.01	70–96	79	12.8	5
strawberry	2.00	71–99	88	12.8	5	2.00	71–100	89	13.2	5
	0.01–2.00	71–102	87	12.8	10	0.01–2.00	70–100	84	13.7	10
	0.01	103–109	106	2.8	3	0.01	102–114	108	5.7	3
tomato	2.00	81–95	90	6.1	5	2.00	81–100	90	7.9	5
	0.01–2.00	81–109	96	10.2	8	0.01–2.00	81–114	97	11.7	8

Stability of residues in stored analytical samples

The Meeting received information on the stability of residues of myclobutanil and its alcohol metabolite (RH-9090) in all four types of crops (wet, dry, oily and acidic). Frozen storage stability data is presented here for apples, radish root, soybean, wheat forage, wheat grain, wheat hay, wheat straw, blueberry, cucurbits and snap beans.

Homogenized 5 gram control samples were spiked with 0.1 mg/kg of myclobutanil and RH-9090 (in separate samples) and stored frozen at approximately -20 °C until analysed. Triplicate samples were analysed at 0, 42, 206, 385 and 588 days by analytical method GRM 03.01. A control sample and two fresh fortifications were run concurrently at each sampling interval to validate the analytical procedure (Dial, G., 2006, 240828). The storage stability data are summarized in Table 44.

Table 44 Recoveries for myclobutanil and RH-9090 in stored and freshly spiked plant matrix samples

Day	Apples		Radish roots		Soybean		Wheat forage		Wheat grain		Wheat hay		Wheat straw	
	A: in stored samples, % of nominal concentration; B: procedural recovery in freshly spiked sample													
	A	B	A	B	A	B	A	B	A	B	A	B	A	B
myclobutanil														
0	103	81	105	77	106	83	104	84	105	87	110	70	102	80
42	103	79	99	86	111	84	97	81	121	78	96	75	105	78
206	101	87	102	98	109	93	101	91	104	90	100	80	110	86
385	80	105	84	104	92	107	81	102	78	108	77	90	82	93
588/9	86	90	97	89	100	97	96	86	101	92	102	78	98	86
RH-9090														
0	113	81	80	85	91	101	89	107	90	107	88	99	89	106
42	100	89	80	98	99	84	117	83	108	91	104	105	96	104
206	93	110	97	108	106	102	109	110	93	106	95	79	101	97
385	85	106	98	90	100	110	104	87	97	107	94	104	89	106
588/9	94	88	105	87	110	71	104	87	113	95	91	98	101	102

Homogenized 5-gram control samples (blueberry, cucumber, snapbeans, almond meat and hulls) were spiked with 0.1–2.0 mg/kg of myclobutanil and RH-9090 (in separate samples) and stored frozen at approximately -20°C or -10°C until analysed. Triplicate samples were analysed at different sampling intervals with analytical method GRM 03.01 or TR 34S-88-10 (Malegus, R, 2011, 2017296; Batra, R, 1995, 94240; Thompson, DC, 1996, 263134; Batra, R, 1994, 94237). A control sample and one or two fresh fortification(s) were run concurrently at each sampling interval to validate the analytical procedure. The storage stability data are summarized in Table 45.

Table 45 Recoveries for myclobutanil and RH-9090 in stored and freshly spiked plant matrix samples

	Blueberry			Cucumber			Snapbean				Almond meat		Almond hulls	
	A: in stored samples, % of nominal concentration; B: procedural recovery in freshly spiked sample													
Day	A	B	Month	A	B	Day	A	B	Month	A	B	A	B	
myclobutanil														
336	91	100	0	80	78	657	94	98	0	104.5	95.9	90.7	98.2	
			3	86	94				3	81.3	84.4	95.1	91.0	
			6	74	77				6	96.8	99.2	93.1	88.4	
			12	80	107				12	82.7	89.5	108.5	112.5	
			18	104	114				18	129.5	127.0	90.2	108.0	
			24	98	80									
			30	84	93									
			36	99	100									
RH-9090														
336	87	100	0	96	92	657	94	83	0	89.4	83.7	115.3	133.0	
			3	85	86				3	68.2	71.6	77.1	91.9	
			6	90	86				6	79.7	83.4	75.5	85.7	
			12	93	117				12	71.7	79.2	69.3	66.3	
			18	84	88				18	75.4	87.1	57.7	71.3	
			24	107	84									
			30	101	112									
			36	89	96									

Liver and muscle samples were fortified at 1.0 mg/kg with myclobutanil or RH-9090 and stored frozen. At specified analysis intervals, the samples were analysed by the residue analytical method TR 31S-88-21 for myclobutanil and TR 34S-88-22 for RH-9090 ((Desai, R and Garstka, TA, 1997, 94239)). Control samples and fresh fortifications were concurrently run at each analysis interval to validate the analytical procedure. The data show no decline in frozen stored samples with time (up to three months) for both myclobutanil and RH-9090 in liver and muscle. The storage stability data are summarized in Tables 46.

Table 46 Recoveries for myclobutanil and RH-9090 in stored and freshly spiked animal matrix samples

	Muscle		Liver	
	A: in stored samples, % of nominal concentration; B: procedural recovery in freshly spiked sample			
Day	A	B	A	B
myclobutanil				
0	102	103	108	106
10	104	99.4	93.3	97.0
30	85.4	84.8	85.6	89.5
50	106	90.9	76.9	70.8
80	82.6	85.8	85.9	88.3
RH-9090				
0	104	81.6	82.0	69.7
10	85.4	75.5	87.7	76.7
30	96.2	88.5	80.3	73.4
50	88.8	79.8	98.9	101
80	102	92.2	94.6	90.2

USE PATTERN

Labels and English translations were available for all the uses. Myclobutanil is a systemic fungicide of the triazole class, providing protectant curative and eradicant activity. Mode of action of myclobutanil is inhibiting sterol biosynthesis which disrupts membrane function. A summary of the current approved label rates are provided in Table 47.

Table 47 Summary of registered uses of myclobutanil

Crop	Country ^a	Formulation		Application		Application rate per treatment			
		Type	g ai/L or g/kg	Method	Number Applications max	g ai/hL max	water (L/ha) min/max	g ai/ha max	PHI (days)
Apple	ARG	WP	400	F	3	6	2500	150	21
Apple	BRA	EC	250	F	n.s.	4.5	700–1500	68	14
Apple	BRA	WP	400		3	4.8	700 to 1500	72	14
Apple	CHN	WP	400	F	3	6.7			7
Apple	CRI, GTM, HND, PAN	WP	400	F		6	300–600	36	15
Apple	JPN	WP	20	F	3		2000–7000	280	60
Apple	MAR, DZA	EC	240	F		6.00			14
Apple	TUR	EC	245	F	3	3.675	2500	92	14
Apple	UK/IRL	EW	200	F	10	45	200–500	90	14
Apple	URY	EC	250	F	n.s.	6.25	n.s.		14
Apple	USA	EW	200	F	9	9.38	2806	263	14
Apple	USA	WP	400	F	9	8.99	2806	252	14
Apple	USA	WP	400	F,G or A	8	7.49	3742	280	14
Apple	USA	WP	400	F,G or A	8	7.49	3742	280	14
Apple, pear	HUN	EC	45		3	1.125	1200	13.5	14
Apple, pear	URY	WP	400	F	n.s.	6	n.s.		14
Apples	ARG	EC	250	F	3	6.25	2500	156	21
Apples	BGR	EC	240	F	2	4.8	1000	48	21
Apples	BGR	EC	20	F	4	0.5	1500	8	35
Apples	ITA	SC	45.5 g/L	F	3	11.3	500–1000	56.25	14
Apples	MEX	WP	400	F	n.s.	150	n.s.	n.s.	n.s.
Apples	PER	WP	400	F	2	150	n.s.	n.s.	14

Crop	Country ^a	Formulation		Application		Application rate per treatment			
		Type	g ai/L or g/kg	Method	Number Applications max	g ai/hL max	water (L/ha) min/max	g ai/ha max	PHI (days)
Apples	TWN	WP	400	F			1200	40	6
Apples/pear	ESP	EW	45	F	3	5.63		84.15	14
Apples/pear	ESP	EC	125	F	4	6.25			28
Apples/pear	ESP	EW	200	F		12.00		84.00	14
Apples/pear	ESP	EC	240	F	4	6.00			28
Apples/pear	ESP	WP	8	F		6.40			28
Apples/pear	ITA	EW	200	F	4	7.0	1000	70	15
Apples/pear	PRT	SC	45	F		7.65		67.50	14
Apples/pear	PRT	EW	45	F	3	5.63		84.15	14
Apricot	USA	EW	200	F	7	6.25	2806	175	0
Apricot	USA	WP	400	F	7	5.99	2806	168	0
Apricot	USA	WP	400	F	7	7.19	2338	168	0
Apricot	USA	WP	400	F	7	5.99	2806	168	0
Banana	Col	WP	400	F	n.s	24	n.s		n/a
Banana	CRI, GTMI, HNDI, PAN	WP	400			84.8			
Banana	DOM	WP	400			6			
Beans	Col	WP	400	F	n.s	200.00	200	16	1
Berries	MEX	WP	400	F	n.s	n.s	1200	91	n.s
Black currant	GBR/IRE	EW	200	F	6	45	200—500	90	14
Cherries	USA	WP	400	F	9	7.19	2338	168	0
Cherries	USA	WP	400	F	9	5.99	2806	168	0
Cherry	ESP	EC	125	F	5	10.00			7
Cherry	GER	EW	200	F	2				21
Cherry	HUN	EC	45	F	3	0.49	1200	5.85	14
Cherry	USA	EW	200	F	8	6.25	2806	175	0
Cherry, nectarine, peach	USA	WP	400	F	9	5.99	2806	168	0
Chili	THA	EC	240	F				24	3
Cucumber	China	WP	400	F	3	6.7			7
Cucurbits	ARG	SC	250	F	n.s	3.5	800 to 1200	42	5
Cucurbits	ARG	WP	400	F	n.s	3.2	800 to 1200	38	5
Cucurbits	CAN	WP	400	F	2		250	70	3
Cucurbits	CHL	EC	240	F	4		200—400	41	5
Cucurbits	CHL	WP	400	F	4		200—400	40	7
Cucurbits	DZA	EC	125	F		9.38			7
Cucurbits	ESP	EC	125	F		10.00			3
Cucurbits	ESP	WP	8	F		6.40		48.00	5
Cucurbits	PAN	WP	400	F	2			48	7
Cucurbits	TUN	EC	75	F				38	
Cucurbits	USA	WP	400	F	5	300	46.76	140	0
Cucurbits	USA	WP	400	F	5			140	0
Currant	CAN	WP	400	F	3		250	136	1
Currant	ESP	EC	125	F		6.25			7
Currant	ESP	EC	240	F	4	6.00			7
Currant	PAK	WP	400	F	8			140	0
Currant	USA	WP	400	F	8			140	0
Currant	USA	WP	400	F	8			140	0
Currant, red and black	ESP	EW	45	F	3	5.99		75	14
Currant, red and black	ESP	EW	200	F	3	15.00		90	14
Currant, red and black	PRT	EW	45	F	3	5.99		75	14
Fruit trees	DZA	EC	125	F		9.38			28

Crop	Country ^a	Formulation		Application		Application rate per treatment			
		Type	g ai/L or g/kg	Method	Number Applications max	g ai/hL max	water (L/ha) min/max	g ai/ha max	PHI (days)
Fruit trees	DZA	EC	125	F		9.38			28
Grape	ARG	EC	250	F		4.25	800–1200	51	21
Grape	ARG	EC	250	F		3.50	800–1200	42	21
Grape	ARG	EC	250	F		10.00	800–1200	120	21
Grape	ARG	WP	400	F		4.80	800–1200	58	21
Grape	ARG	WP	400	F		4.00	800–1200	48	21
Grape	ARG	WP	400	F		8.00	800–1200	96	21
Grape	AUS	EC	125	F	3	3.13	800–1500		14
Grape	BRA	WP	400	F		8.00	700–1000	80	7
Grape	BRA	WP	400	F	3	8	700–1000	80	7
Grape	CAN	WP	400	F	5			80	14
Grape	CHI	WP	400	F	3	10			21
Grape	CHL	EC	240	F	4	2.88	500–2500		10
Grape	CHL	WP	400	F	3	3.20	1000–3000		14
Grape	COL	WP	400	F	n.s	n.s	n.s	50	14
Grape	CRI, GTMI, HNDI, PAN	WP	400	F		13.3–26.7	300–600	80	15
Grape	CZE	EW	200	F	3			36	28
Grape	DOM	WP	400	F			1000–1500	80	15
Grape	DZA	EC	125	F		9.38			15
Grape	DZA, TUN	EC	75	F				23	21
Grape	EGY	EC	125	F		4.38			15
Grape	ESP	EW	45	F	3	5.63		72.90	14
Grape	ESP	EC	125	F		10.00			15
Grape	ESP	EW	200	F	3	14.00		68.00	14
Grape	ESP	EC	240	F		9.60			15
Grape	ESP	WP	8	F		6.40		32.00	21
Grape	GER	EW	200	F	4		400–1600	48	28
Grape	HUN	EC	45	F	3	0.49	1200	5.85	14
Grape	HUN	SC	240	F	3	25.00	1200	300	28
Grape	MAR	EC	45	F		5.63			21
Grape	MAR, DZA	EC	240	F	3	30.00			14
Grape	PRT	EC	125	F	3	3.00			21
Grape	PRT	SC	45	F	3	5.63		56.25	28
Grape	PRT	EW	45	F	3	5.63		72.90	14
Grape	TUR	SC	45	F	3	5.63	1000	56	28
Grape	TUR	EC	245	F	3	18.38	1000	184	14
Grape	URY	EC	250	F		6.25	n.s		21
Grape	URY	WP	400	F		5.60	n.s		21
Grape	USA	EW	200	F	5	n.s	n.s	146	14
Grape	USA	WP	400	F	9	n.s	n.s	73	14
Grape	USA	WP	400	F	5			140	14
Grape	USA	WP	400	F	5			140	14
Grapes	BGR	SC	45	F	3	5.6	1000	56	35
Grapes	BGR	EC	240	F	2	3.6	1000	36	15
Grapes	BGR	EC	20	F	2	0.5	1500	8	35
Grapes	ITA	SC	45.5 g/L	F	4	6.75	n.s		28
Grapes	ITA	SC	45.5 g/L	F	4	37.5	150–1000	56.25	28
Grapes	ITA	EW	200	F	4	7.0	1000	70	15
Grapes	MEX	WP	400	F	n.s	n.s	n.s	91	no

Crop	Country ^a	Formulation		Application		Application rate per treatment			
		Type	g ai/L or g/kg	Method	Number Applications max	g ai/hL max	water (L/ha) min/max	g ai/ha max	PHI (days)
									specified
Grapes	PER	WP	400	F	2	120	n.s	n.s d	14
Grapes	TWN	WP	400	F	4	3.3	1200	40	15
Grapes	TWN	SC	90	F	3	2.8	1200	34	15
Grapes	VEN	WP	400	F	n.s	n.s	n.s	20	14
Hops	ESP	EC	125	F			10.00		15
Hops	ESP	EC	240	F			9.60		15
Hops	GBR	EW	200	F	4			180	14
Hops	GER	EW	200	F	3			180	14
Hops	USA	WP	400	F	4			280.21	14
Melon	BRA	WP	400	F			300–1000	150	7
Melon	BRA	WP	400	F	3	20	300–1000	60	7
Melon	CRI, GTMI, HNDI, PAN	WP	400	F	2		150–200	48	7
Melon	DOM	WP	400	F	2			80	7
Melon	ESP	EW	45	F	3	7.52		75.15	3
Melon	ESP	EW	200	F	3	12.00		76.00	3
Melon	JAP	WP	100	F	3		1500–3000	50	1
Melon	PRT	SC	45	F	3	7.65		56.25	7
Melon	TWN	EC	125	F	3		1200	120	3
Melon, watermelon, pumpkin	MEX	WP	400	F	n.s	n.s	n.s	91	no specified
Melon, watermelon, pumpkin	MEX	WP	400	F	n.s	n.s	400	91	no specified
Melon, watermelon, pumpkin	PER	WP	400	F	2	300	n.s	no specified	0
Melon/ watermelon	ITA	SC	45.5 g/L	F	3	11.3	500–1000	56.25	7
Melon/ watermelon	ITA	EW	200	F	4	7	1000	70	3
Muskmelon	PRT	EW	45	F	3	7.52		75.15	3
Nectarine	ARG	EC	250	F		4.25	2000	85	30
Nectarine	ARG	WP	400	F		4.00	2000	80	30
Nectarine	USA	EW	200	F	8	6.25	2806	175	0
Nectarine	USA	WP	400	F	9	7.19	2338	168	0
Nectarine	USA	WP	400	F	9	5.99	2806	168	0
Peach	ARG	EC	250	F		4.25	2000	85	30
Peach	ARG	WP	400	F		4.00	2000	80	30
Peach	DZA	EC	240	F		6.00			14
Peach	TWN	WP	400	F		13.3	1000–2000	267	18
Peach	USA	EW	200	F	8	6.25	2806	175	0
Peach	USA	WP	400	F	9	7.19	2338	168	0
Peach	USA	WP	400	F	9	5.99	2806	168	0
Peach, apricot, nectarine	PRT	SC	45	F		7.65		67.50	14
Peach, apricot, nectarine, cherry	ESP	EC	240	F	5	7.20			7
Peach, apricot, nectarine, cherry	ESP	WP	8	F		6.40			7
Peach, apricot, nectarine, plum	ESP	EC	125	F	5	7.50			7
Peach, apricot, nectarine, plum, cherry	ESP	EW	200	F	2	13.00		90.00	7
Peach/ nectarine/apricot	ITA	SC	45.5 g/L	F	3	11.3	500–1000	56.25	14
Peach/ nectarine/apricot/plum	ITA	EW	200	F	4	7	1000	70	7
Peach/cherry	JAP	WP	100	F	4		2000–7000	350	1

Crop	Country ^a	Formulation		Application		Application rate per treatment			
		Type	g ai/L or g/kg	Method	Number Applications max	g ai/hL max	water (L/ha) min/max	g ai/ha max	PHI (days)
Pear	ARG	EC	250	F	4	4.25	2500	106	21
Pear	ARG	WP	400	F	4	4	2500	100	21
Pear	CHN	WP	400	F	3	5			7
Pear	UK/IRL	EW	200	F	10	45	200–500	90	14
Pear/Apple	AUS	WP	400	F		4.8			21
Pear/Apple	CAN	WP	400	F	6		500–1000	136	14
Pear/Apple	CHL	WP	400	F	4	5.0	1000–3000		15
Pear/Apple	JPN	WP	100	F	3		2000–7000	350	7
Pear/Apple	ZAF	EW	200	F	6	3			14
Pepper GH	CAN	WP	400	F	3		1000	136	3
Pepper GH	Turkey	EC	245	F	2	7.35	1000	73.50	3
Peppers	ESP	EC	125	F		10.00			3
Peppers	ESP	EW	45	F	3	7.52		75.15	3
Peppers	ESP	EW	200	F	3	12.00		76.00	3
Peppers	ESP	EC	240	F		9.60			3
Peppers	ESP	WP	8	F		6.40		64.00	5
Peppers	HUN	EC	45	F	3	0.84	800	6.75	7
Peppers	ITA	EW	200	F	4	7	1000	70	3
Peppers	JAP	WP	100	F	4		1500–3000	75	1
Peppers	PRT	EW	45	F	3	7.52		75.15	3
Peppers	USA	WP	400	F	4			140	0
Plum	TWN	WP	400	F	4	5	1000	50	6
Plum, cherry, peach, apricot	ESP	EW	45	F	2	5.99		90.00	7
Plum, cherry, peach, apricot	PRT	EW	45	F	2	5.99		90.00	7
Plum, prune	PAK	WP	400	F	7	7.19	2338	168	0
Plum, prune	USA	EW	200	F	7	6.25	2806	175	0
Plum, prune	USA	WP	400	F	7	7.19	2338	168	0
Plum, prune	USA	WP	400	F	7	5.99	2806	168	0
Plum, sour cherry	CZE	EW	200	F	3			140	21
Pome	CZE	EW	200	F	3			90	14
Pome	GER	EW	200	F	4				14
Plum, prune	USA	WP	400	F	7	5.99	2806	168	0
Snap bean	USA	WP	400	F	4			140	0
Snap bean	USA	WP	400	F	4			140	0
Soybean	ARG	SC	250	F	3	83	150 to 200	125	21
Soybean	BOL	EC	250	F	2	125	100 to 150	125	24
Soybean	BRA	EC	250	Bkp	2	125	100 to 500	125	24
Soybean	BRA	EC	250	Bkp	2	63	100 to 500	125	24
Soybean	URY	EC	250	F	3	125	100 to 150	125	21
Soybean	USA	WP	400	F	2			140	28
Soybean	USA			F	2			140	28
Stone fruit	CAN	WP	400	F	6		1000	136	1
Stone fruit	CHL	WP	400	F	4	5.0	1000–3000	150	15
Strawberry	AUS	WP	400	F			> 500	48	0
Strawberry	BEL	EC	240	F				60.00	3
Strawberry	CAN	WP	400	F	6		1000	136	3
Strawberry	CZE	EW	200	F	3			100	14
Strawberry	ESP	EW	45	F	3	5.99		59.85	3
Strawberry	ESP	EC	125	F	4	7.50			3
Strawberry	ESP	EW	200	F	3	10.00		60.00	3
Strawberry	ESP	EC	240	F	5	7.20			3
Strawberry	ESP	WP	8	F		6.40		48.00	5
Strawberry	GBR/IRE	EW	200	F	6	45	200 to 500	90	3

Crop	Country ^a	Formulation		Application		Application rate per treatment			
		Type	g ai/L or g/kg	Method	Number Applications max	g ai/hL max	water (L/ha) min/max	g ai/ha max	PHI (days)
Strawberry	GER	EW	200	F	3			100	14
Strawberry	ITA	SC	45.5 g/L	F	3	11.3	500–1000	56.25	7
Strawberry	ITA	EW	200	F	4	6	1000	60	3
Strawberry	JAP	WP	100	F	3		1500–3000	75	1
Strawberry	JAP	EC	250	F	3		1500–3000	150	1
Strawberry	MAR	EC	240	F		7.20			3
Strawberry	PRT	EC	125	F		6.25			3
Strawberry	PRT	SC	45	F	3	7.65		56.25	3
Strawberry	PRT	EW	45	F	3	5.99		59.85	3
Strawberry	PRT	EW	45	F	3	5.99	400–1000	59.85	3
Strawberry	USA	WP	400	F	6			140	0
Strawberry	USA	WP	400	F	6			140	0
Tomato	BEL	EC	240	F		7.20		38.40	3
Tomato	CAN	WP	400	F	2		1000	136	3
Tomato	CHL	EC	240	F	4		200–400	48	3
Tomato	Col	WP	400	F	n.s	400	200	32	1
Tomato	ESP	EW	45	F	3	7.52		75.15	3
Tomato	ESP	EC	125	F		10.00			3
Tomato	ESP	EW	200	F	3	12.00		76.00	3
Tomato	ESP	EC	240	F		9.60			3
Tomato	ESP	WP	8	F		6.40		64.00	5
Tomato	ITA	EW	200	F	4	7	1000	70	3
Tomato	JAP	EC	250	F	3		1500–3000	300	1
Tomato	MAR	EC	240	F		7.20			3
Tomato	MEX	WP	400	F	n.s	n.s	n.s	91	
Tomato	PRT	EW	45	F	3	7.52		75.15	3
Tomato	TUN	EC	75	F				38	
Tomatoes	BOL	EC	250	F	4	37.5	100	37.5	5
Tomatoes	USA	WP	400	G or A	4	59.94	94 to 187	112	0
Tomatoes	USA	WP	400	G or A	4	59.94	94 to 187	112	

^a Countries are indicated with their 3-letter code; A: aerial, Bkp: backpack, G: ground; GH: greenhouse n.s: not specified

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on supervised field trials for myclobutanil uses on the following crops.

Table 48 Crops and crop groups of supervised trials

Crop Group	Commodity	Country	Table No.
Citrus fruits	Orange	ESP	Table 49
	Mandarin	ESP	Table 50
Pome fruits	Apple	AUT, BRA, CAN, FRA, ESP, ITA, GRC, CHL, GER, UK, USA	Table 51
	Pear	BEL, CAN, CHL, FRA, ITA, ESP, USA	Table 52
Stone fruits	Peach	CAN, FRA, GRC, ITA, ESP, USA	Table 53
	Cherry	AUT, BEL, CAN, FRA, GER, HUN, UK, USA	Table 54
	Apricot	FRA, ITA, ESP, USA	Table 55
	Plum	AUT, CAN, C, FRA, GER, HUN, ITA, ESP, USA	Table 56

Crop Group	Commodity	Country	Table No.
Berries and other fruits	Currant	FRA, ITA, UK, USA	Table 57
	Grapes	BRA, FRA, GER, GRC, ITA, PRT, ESP, USA	Table 58
	Strawberry	FRA, ITA, GBR, ESP, USA	Table 59
Fruiting vegetable, Cucurbits	Banana	CRI, USA	Table 60, 61
	Cucumber	FRA, ITA, ESP, USA	Table 62
	Melon	FRA, GRC, ITA, ESP, USA	Table 63
	Squash	ESP, USA	Table 64
	Watermelon	ITA, USA	Table 65
	Pepper	FRA, ITA, ESP, USA	Table 66
Fruiting vegetables, Other than cucurbits	Tomato	FRA, ITA, MAR, ESP, USA	Table 67
	Snap beans	USA	Table 68
	Soybeans	ARG, BRA, USA	Table 69
Dried herbs	Hops	GER, UK, USA	Table 70
Legume animal feed	Soybean forage and hay	USA	Table 71

In addition to the description and details of the field trials, each report includes a summary of the analytical method(s) used, together with the corresponding procedural recoveries. In the field trials where multiple analyses are conducted on a single sample or where multiple samples were taken from a single plot, the average residue value is reported. Where results from separate plots with distinguishing characteristics, such as different formulations, varieties or treatment schedules were reported, results are listed for each plot.

Results have not been corrected for concurrent method recoveries unless indicated. Residues and application rates have generally been rounded to 2 or 3 significant figures or, for residues near the LOQ, to one significant figure. Residue values from the trials conducted according to the maximum GAP have been used for the estimation of maximum residue levels. For purposes of MRL estimation and risk assessment in this report, ND values in the reports were considered as < LOQ.

Citrus fruits

Oranges

A total of 15 post-harvest studies on orange were conducted in Spain 1994 and 1995. Results of orange fruits are summarized in Table 49.

Table 49 Residues of myclobutanil in oranges after post-harvest treatment in Spain

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analysed	Total of myclobutanil (mg/kg)	Reference
Post harvest										
7/95 Betera, Valencia, ESP 1995 (W. Navel)	EC (120 g ai/L)	1	—	—	50	—	0 30 45 0 30 45 0 30 45 0 30 45	Whole fruit Whole fruit Whole fruit Peel Peel Peel Pulp Pulp Pulp Juice Juice Juice	1.30 1.60 1.00 4.22 3.00 3.40 ND ND ND 0.10 ND ND	242040
8/95	EC	1	—	—	50	—	0	Whole fruit	1.06	242040

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analysed	Total of myclobutanil (mg/kg)	Reference
El Puig de Santa Maria, Valencia, ESP 1995 (W. Navel)	(120 g ai/L)						30 45 0 30 45 0 30 45 0 30 45	Whole fruit Whole fruit Peel Peel Peel Pulp Pulp Pulp Pulp Juice Juice Juice	1.16 1.10 3.50 3.40 3.30 ND ND ND 0.07 ND ND	
06/94 Betera, Valencia, ESP 1994 (W. navel)	EC (120 g ai/L)	1	—	—	50	—	0 7 14	Whole fruit Whole fruit Whole fruit	1.80 1.70 0.70	242041
08/94 El Puig, Valencia, ESP 1994 (Navel late)	EC (120 g ai/L)	1	—	—	50	—	0	Whole fruit	1.36	242041
09/94 Nules, Castellon, ESP 1994 (Navelina)	EC (120 g ai/L)	1	—	—	50	—	0 7 14	Whole fruit Whole fruit Whole fruit	1.49 1.37 0.69	242041
13/94 Betera, Valencia, ESP 1994 (Navelina)	EC (120 g ai/L)	1	—	—	50	—	0 7 14	Whole fruit Whole fruit Whole fruit	1.53 1.20 1.10	242041
14/94, Nules, Castellon, ESP 1994 (Navelina)	Wax Water Emulsion (3 g ai/L)	1	—	—	3.0	—	0	Whole fruit	2.63	242041
15/94, Cullera, Valencia, ESP 1994 (Valencia late)	Wax Water Emulsion (3 g ai/L)	1	—	—	3.0	—	0	Whole fruit	2.36	242041
20/94, Nules, Castellon, ESP 1994 (Navelina)	Wax Water Emulsion (3 g ai/L)	1	—	—	3.0	—	0	Whole fruit	2.43	242041
22/94 Oliva, Valencia, ESP 1994 (New Hall)	Wax Water Emulsion (3 g ai/L)	1	—	—	3.0	—	0	Whole fruit	2.66	242041
9/95, Oliva, Valencia, ESP 1995 (Valencia late)	Wax Water Emulsion (3 g ai/L)	1	—	—	3.0	—	0 30 60 90 0 30 60 90 0 30 60 90 0 30	Whole fruit Whole fruit Whole fruit Whole fruit Peel Peel Peel Peel Pulp Pulp Pulp Pulp Pulp Pulp Juice Juice	2.30 1.99 2.60 1.88 7.10 5.03 7.10 6.00 ND ND ND ND ND ND ND	260333

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analysed	Total of myclobutanil (mg/kg)	Reference
							60 90	Juice Juice	ND ND	
10/95, Puzol, Valencia, ESP 1995 (Valencia late)	Wax Water Emulsion (3 g ai/L)	1	—	—	3.0	—	0 30 60 90 0 30 60 90 0 30 60 90 0 30 60 90	Whole fruit Whole fruit Whole fruit Whole fruit Peel Peel Peel Peel Pulp Pulp Pulp Pulp Juice Juice Juice Juice	0.87 1.04 1.66 0.74 1.77 3.20 4.60 2.12 ND ND ND ND ND ND ND ND	260333
11/95, Carlet, Valencia, ESP 1995 (Valencia late)	Wax Water Emulsion (3 g ai/L)	1	—	—	3.0	—	0 30 60 90 0 30 60 90 0 30 60 90 0 30 60 90	Whole fruit Whole fruit Whole fruit Whole fruit Peel Peel Peel Peel Pulp Pulp Pulp Pulp Juice Juice Juice Juice	1.47 1.44 0.80 1.24 3.80 5.50 3.00 3.80 ND ND ND ND ND ND ND ND	260333
12/95, Simat de Valldigna, Valencia, ESP 1995 (Valencia late)	Wax Water Emulsion (3 g ai/L)	1	—	—	3.0	—	0 30 60 90 0 30 60 90 0 30 60 90 0 30 60 90	Whole fruit Whole fruit Whole fruit Whole fruit Peel Peel Peel Peel Pulp Pulp Pulp Pulp Juice Juice Juice Juice	2.60 2.60 2.00 1.80 6.60 6.50 6.10 2.18 ND ND ND ND ND ND ND ND	260333
13/95, Polina Del Jucar, Valencia, ESP 1995 (Valencia late)	Wax Water Emulsion (3 g ai/L)	1	—	—	3.0	—	0 30 60 90 0 30 60 90 0 30 60 90	Whole fruit Whole fruit Whole fruit Whole fruit Peel Peel Peel Peel Pulp Pulp Pulp Pulp Juice Juice Juice Juice	1.23 1.60 1.98 1.90 3.22 5.00 7.80 4.80 ND ND ND ND ND ND ND ND	260333

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analysed	Total of myclobutanil (mg/kg)	Reference
							0 30 60 90	Juice Juice Juice Juice	ND ND ND ND	

Mandarin

A total of 12 post-harvest studies on mandarin were conducted in Spain 1994 and 1995. Results of mandarin fruits are summarized in Table 50.

Table 50 Residues of myclobutanil in mandarin after post-harvest treatment in Spain

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analysed	Total of myclobutanil (mg/kg)	Reference
Post harvest										
5/95 Chilches, Castellon, ESP 1995 (Clemenules)	EC (120 g ai/ L)	1	—	—	50	—	0 30 45 0 30 45 0 30 45 0 30 45	Whole fruit Whole fruit Whole fruit Peel Peel Peel Pulp Pulp Pulp Juice Juice Juice	1.15 1.40 1.00 2.75 3.40 2.00 ND ND ND 0.10 ND ND	242040
6/95 Nules, Castellon, ESP 1995 (Hernandina)	EC (120 g ai/ L)	1	—	—	50	—	0 30 45 0 30 45 0 30 45 0 30 45	Whole fruit Whole fruit Whole fruit Peel Peel Peel Pulp Pulp Pulp Juice Juice Juice	0.94 1.50 1.10 3.35 3.80 3.10 ND ND ND 0.09 ND ND	242040
05/94 Betera, Valencia, ESP 1994 (Oroval)	EC (120 g ai/ L)	1	—	—	50	—	0 7 14	Whole fruit Whole fruit Whole fruit	2.00 1.70 1.10	242041
07/94 El Puig, Valencia, ESP 1994 (Clemenules)	EC (120 g ai/ L)	1	—	—	50	—	0	Whole fruit	1.56	242041
10/94 Nules, Castellon, ESP 1994 (Satsuma)	EC (120 g ai/ L)	1	—	—	50	—	0 7 14	Whole fruit Whole fruit Whole fruit	1.33 1.09 0.57	242041
11/94 Carlet, Valencia, ESP 1994 (Satsuma)	EC (120 g ai/ L)	1	—	—	50	—	0 7 14	Whole fruit Whole fruit Whole fruit	1.50 1.20 1.10	242041
12/94	EC	1	—	—	50	—	0	Whole fruit	1.71	242041

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analysed	Total of myclobutanil (mg/kg)	Reference
Betera, Valencia, ESP 1994 (Oroval)	(120 g ai/L)						7 14	Whole fruit Whole fruit	1.20 1.00	
16/94 Chilches, Castellon, ESP 1994 (Oroval)	Wax Water Emulsion (3 g ai/L)	1	—	—	3.0	—	0 7 14	Whole fruit Whole fruit Whole fruit	2.61 2.35 2.26	242041
17/94 Chilches, Castellon, ESP 1994 (Satsuma)	Wax Water Emulsion (3 g ai/L)	1	—	—	3.0	—	0 7 14	Whole fruit Whole fruit Whole fruit	2.80 2.54 2.36	242041
18/94 Chilches, Castellon, ESP 1994 (Satsuma)	Wax Water Emulsion (3 g ai/L)	1	—	—	3.0	—	0 7 14	Whole fruit Whole fruit Whole fruit	2.75 2.18 2.40	242041
19/94 Chilches, Castellon, ESP 1994 (Satsuma)	Wax Water Emulsion (3 g ai/L)	1	—	—	3.0	—	0 7 14	Whole fruit Whole fruit Whole fruit	2.72 2.53 2.51	242041
21/94, Oliva, Valencia, ESP 1994 (Oroval)	Wax Water Emulsion (3 g ai/L)	1	—	—	3.0	—	0	Whole fruit	2.90	242041

Pome fruits

Apples

A total of 144 trials on apple were conducted in different representative growing areas in Austria, Brazil, Canada, Chile, France, Germany, Italy, Spain, UK and USA during 1984 through 2011 growing seasons. Results of apple fruits are summarized in Table 51.

Table 51 Residues of myclobutanil in apple after treatment in various countries

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
90-0201 USA 1990 (Twenty ounce)	40 WP (40 %W/W)	10	0.224	56.1	—	7–27	14	Whole fruit	0.02	< 0.01	0.03	94312
90-0201 USA 1990 (Twenty ounce)	40 WP (40 %W/W)	10	0.224	935.3	—	7–27	14	Whole fruit	0.13	0.03	0.16	94312
90-0136 USA 1990 (Twenty ounce)	40 WP (40 %W/W)	4	0.224	93.5	—	7	14	Whole fruit	0.04	< 0.01	0.05	94312
90-0136 USA 1990 (Twenty ounce)	40 WP (40 %W/W)	4	0.224	935.3	—	7	14	Whole fruit	0.18	0.01	0.19	94312
21801070, ID, USA 2001 (Red delicious)	40 WP (40 %W/W)	12	0.19	1100	—	13–16	14	Whole fruit	0.28	< 0.01	0.29	135365
21801069 CA, USA	40 WP (40 %W/W)	12	0.19	1100	—	14–17	14	Whole fruit	0.07	< 0.01	0.08	135365

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicati on Interval (days)	DAT	Portion Analyse d	Myclob utanil (mg/kg)	RH- 9090 (mg/kg)	Total of myclob utanil (mg/kg)	Ref.
2001 (Fuji)	%W/W)											
21801068, AZ USA 2001 (Red delicious)	40 WP (40 %W/W)	12	0.19	1870	—	12–17	10	Whole fruit	0.21	< 0.01	0.22	135365
84-0313, WA USA 1984 (Jonathan)	40 WP (40 %W/W)	5	0.50	4677	—	—	129	Whole fruit	0.02	0.13	0.15	94378 94484/85
84-0238, VA USA 1984 (Rome)	40 WP (40 %W/W)	6	0.28	3741	—	—	23	Cider	0.02	< 0.01	0.03	94378 94484/85
							23	Pomace,	0.27	0.03	0.30	
							23	dry Pomace,	0.08	0.01	0.09	
							23	wet Whole fruit	0.01	0.04	0.05	
84-0238, VA USA 1984 (Rome)	40 WP (40 %W/W)	6	0.56	3741	—	—	23	Whole fruit	0.07	0.04	0.11	94378 94484/85
84-0212, SC USA 1984 (Del, wsap. grd)	40 WP (40 %W/W)	14	0.16	—	—	—	6	Whole fruit	0.32	0.12	0.44	94378 94484/85
84-0274, WI USA 1984 (McIntosh)	40 WP (40 %W/W)	9	0.43	2806	—	—	14	Whole fruit	0.17	0.04	0.21	94378 94484/85
84-0274, WI, USA 1984 (McIntosh)	40 WP (40 %W/W)	9	0.43	2806	—	—	11	Cider	0.02	0.02	0.04	94378 94484/85
							11	Pomace,	0.60	0.09	0.69	
							11	dry Pomace,	0.18	(0.04) ^a 0.03	21	
84-0241, PA USA 1984 (Multi)	40 WP (40 %W/W)	15	0.27	3701	—	—	14	Whole fruit	0.24	0.09	0.33	94378 94484/85
84-0384, PA USA 1984 (Roam beauty)	40 WP (40 %W/W)	11	0.53	2806	—	—	13	Whole fruit	0.11	0.02	0.13	94378 94484/85
84-0300, WI USA 1984 (McIntosh)	40 WP (40 %W/W)	10	0.10	2806	—	—	12	Whole fruit	0.03	0.01	0.04	94378 94484/85
85-0521, OR USA 1985 (Granny Smith)	40 WP (40 %W/W)	4	0.28	2338	—	—	139	Whole fruit	< 0.01	< 0.01	< 0.02	94300
85-0481, OR USA 1985 (Newtown)	40 WP (40 %W/W)	4	0.28	2338	—	—	124	Whole fruit	0.02	< 0.01	0.03	94300
85-0320, PA USA 1985 (G. Delicious)	40 WP (40 %W/W)	13	0.27	3741	—	—	14	Whole fruit	0.15	0.04	0.19	94311
85-0334, VA USA 1985 (Rome)	40 WP (40 %W/W)	11	0.40	1402	—	—	13	Whole fruit	0.09	0.07	0.16	94311
85-0542, OH USA	40 WP (40	14	0.28	3741	—	—	14	Whole fruit	0.19	0.06	0.25	94311

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicati on Interval (days)	DAT	Portion Analyse d	Myclob utanil (mg/kg)	RH- 9090 (mg/kg)	Total of myclob utanil (mg/kg)	Ref.
1985 (G. Delicious)	%W/W)											
85-0543, OH USA 1985 (R. Delicious)	40 WP (40 %W/W)	14	0.28	3741	–	–	14	Whole fruit	0.30	0.01	0.31	94311
85-0422, OR USA 1985 (Newtown)	40 WP (40 %W/W)	9	0.28	2338	–	–	14	Whole fruit	0.20	0.05	0.25	94311
85-0551, WA USA 1985 (Rome)	40 WP (40 %W/W)	8	0.42	5612	–	–	14	Whole fruit	0.08	0.02	0.10	94311
85-0490, MI USA 1985 (R. Delicious)	40 WP (40 %W/W)	10	0.28	2806	–	–	7	Whole fruit	0.15	0.01	0.16	94308 94472
							14 ^b	Whole fruit	< 0.01	< 0.01	< 0.02	
							21	Whole fruit	0.14	0.02	0.16	
85-0542, OH USA 1985 (G. Delicious)	40 WP (40 %W/W)	14	0.28	3741	–	–	21	Cider	0.02	0.02	0.04	94308 94472
							21	Pomace, dry	1.47	0.11	1.58	
							21	Pomace, wet	0.45	0.03	0.48	
							7	Whole fruit	0.21	0.06	0.27	
							14	Whole fruit	0.19	0.06	0.25	
							21	Whole fruit	0.15	0.08	0.23	
86-0297, OR USA 1986 (Granny Smith)	40 WP (40 %W/W)	5	0.24	786	–	13–21 and 92 at last applicatio n	0	Whole fruit	0.21	< 0.01	0.22	94309
							7	Whole fruit	0.12	< 0.01	0.13	
							14	Whole fruit	0.06	< 0.01	0.07	
							21	Whole fruit	0.03	< 0.01	0.04	
86-0260, PA USA 1986 (Macintosh)	40 WP (40 %W/W)	1	0.27	3741	–	–	0	Whole fruit	0.07	< 0.01	0.08	94309
							7	Whole fruit	0.07	< 0.01	0.08	
							14	Whole fruit	0.02	< 0.01	0.03	
							21	Whole fruit	0.03	< 0.01	0.04	
86-0297, OR USA 1986 (Granny Smith)	DF 60 (60% W/W)	5	0.24	786	–	13–21 and 92 at last applicatio n	0	Whole fruit	0.18	< 0.01	0.19	94292
							7	Whole fruit	0.10	< 0.01	0.11	
							14	Whole fruit	0.09	< 0.01	0.10	
							24	Whole fruit	0.03	< 0.01	0.04	
86-0296, OR USA 1986 (Newtown)	DF 60 (60% W/W)	5	0.24	786	–	11–19 and 80 at last applicatio n	0	Whole fruit	0.17	< 0.01	0.18	94292
							8	Whole fruit	0.09	< 0.01	0.10	
							15	Whole fruit	0.06	< 0.01	0.07	
							22	Whole fruit	0.05	< 0.01	0.06	

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
88-0319, PA USA 1988 (R. Delicious)	DF 60 (60% W/W)	8	0.17	374	–	7–18 and 91 at last application	14	Whole fruit	0.01	< 0.01	0.02	94298
88-0319, PA USA 1988 (R. Delicious)	DF 60 (60% W/W)	8	0.17	2235	–	7–18 and 91 at last application	14	Whole fruit	0.03	< 0.01	0.04	94298
88-0320, VA USA 1988 (R. Delicious)	40 WP (40 %W/W)	8	0.17	374	–	7–18 and 91 at last application	14	Whole fruit	0.04	< 0.01	0.05	94298
88-0320, VA USA 1988 (R. Delicious)	40 WP (40 %W/W)	8	0.17	2235	–	7–18 and 91 at last application	14	Whole fruit	0.04	< 0.01	0.05	94298
88-0342, WA USA 1988 (R. Delicious)	40 WP (40 %W/W)	8	0.17	468	–	11–29	14	Whole fruit	0.20	0.02	0.22	94298
88-0342, WA USA 1988 (R. Delicious)	40 WP (40 %W/W)	8	0.17	2338	–	11–29	14	Whole fruit	0.28	0.03	0.31	94298
88-0343, WA USA 1988 (R. Delicious)	DF 60 (60% W/W)	8	0.17	468	–	11–29	14	Whole fruit	0.18	0.02	0.20	94298
88-0343, WA USA 1988 (R. Delicious)	DF 60 (60% W/W)	8	0.17	2338	–	11–29	14	Whole fruit	0.20	0.02	0.22	94298
88-0331, VA USA 1988 (Rome)	DF 60 (60% W/W)	8	0.029	290	–	11–29	14	Whole fruit	0.02	< 0.01	0.03	94298
88-0331, VA USA 1988 (Rome)	DF 60 (60% W/W)	8	0.029	2002	–	11–29	14	Whole fruit	0.02	< 0.01	0.03	94298
88-0332, VA USA 1988 (Stayman)	40 WP (40 %W/W)	8	0.029	290	–	7–14 and 105 at last application	14	Whole fruit	0.03	< 0.01	0.04	94298
88-0332, VA USA 1988 (Stayman)	40 WP (40 %W/W)	8	0.029	2002	–	7–14 and 105 at last application	14	Whole fruit	0.03	< 0.01	0.04	94298
90-0029 Pelotas BRA 1989 (Gala)	EC (25.8% W/W)	4	0.0224	500	4.5	6–7	0 6 13 27	Whole fruit	0.11 0.17 0.01 0.11	0.04 0.07 0.01 0.08	0.14 0.24 0.02 0.19	94315
90-0029 Pelotas BRA 1989 (Gala)	EC (25.8% W/W)	4	0.0448	500	9	6–7	0 6 13 27	Whole fruit	0.39 0.30 0.19 0.17	0.10 0.11 0.12 0.15	0.49 0.41 0.31 0.32	94315
9325701 Dame Marie les Bois FRA 1993 (Golden Delicious)	WP (6%WP)	15	0.06	475–800	–	8–12	0 21	Whole fruit Whole fruit	0.28 0.16	0.03 0.03	0.31 0.19	241989

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
4919356 Sudanel, ESP 1993 (Golden Delicious)	EC (125 g/L)	1	0.075	1398	–	–	0	Whole fruit	0.18	< 0.01	0.19	138353
4919356 Sudanel, ESP 1993 (Golden Delicious)	EC (125 g/L)	3	0.075	1166–1398	–	–	14	Whole fruit	0.20	0.02	0.22	138353
4919356 Sudanel, ESP 1993 (Golden Delicious)	EC (125 g/L)	4	0.075	1166–1398	–	14	0 14 21	Whole fruit	0.32 0.13 0.20	0.03 0.02 0.03	0.35 0.15 0.23	138353
93 FAR RS 01 Orgueil, FRA 1993 (Golden)	WP (6%WP)	4	0.06	300	–	8–11	0 21	Whole fruit	0.09 0.04	< 0.01 0.02	0.10 0.06	241988
RAS 21/1/I Lombardia ITA 1997 (Golden Delicious)	EW (200g ai/L)	6	0.052–0.067	1165–1479	4.5	11–14	0 14	Whole fruit	0.08 0.07	< 0.01 < 0.01	0.09 0.08	241824
RAS 21/1/G Imathia region GRC 1997 (Jonagold)	EW (200 g ai/L)	6	0.068	1499–1502	4.5	10	0 14	Whole fruit	0.04 0.03	< 0.01 < 0.01	0.05 0.04	241824
AF/7780/DE/2 Calatorao ESP, 2004 (Golden)	EW (200 g ai/L)	4	0.085–0.091	1409–1517	6	8–12	14 26 48 56	Whole fruit	0.04 0.06 0.03 0.03	< 0.01 < 0.01 < 0.01 < 0.01	0.05 0.07 0.04 0.04	241776
AF/7780/DE/2 Calatorao ESP, 2004 (Golden)	EW (200 g ai/L)	6	0.070–0.073	1448–1527	6	8–12	14	Whole fruit	0.06	< 0.01	0.07	241776
AF/7780/DE/1 Barboles ESP, 2004 (Golden)	EW (200 g ai/L)	4	0.086–0.092	1433–1533	6	10–12	< 0 0 7 14 28 35	Whole fruit	0.09 0.12 0.14 0.10 0.06 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.10 0.13 0.15 0.11 0.07 0.08	241776
AF/7780/DE/1 Barboles ESP, 2004 (Golden)	EW (200 g ai/L)	6	0.086–0.095	1433–1583	6	7–12	< 0 0 7 14 28 35	Whole fruit	0.10 0.20 0.08 0.07 0.07 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.11 0.21 0.09 0.08 0.08 0.07	241776
AF/7780/DE/4 Moissac FRA, 2004 (Granny)	EW (200 g ai/L)	4	0.088–0.098	1472–1632	6	8–12	14 26 44 55	Whole fruit	0.04 0.02 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01	0.05 0.03 0.03 0.03	241776
AF/7780/DE/4 Moissac FRA, 2004 (Granny)	EW (200 g ai/L)	6	0.087–0.094	1458–1563	6	8–12	14	Whole fruit	0.07	< 0.01	0.08	241776
AF/7780/DE/3 Moissac FRA, 2004 (Granny Smith)	EW (200 g ai/L)	4	0.085–0.096	1416–1603	6	9–11	< 0 0 7 14 28 35	Whole fruit	0.09 0.13 0.04 0.05 0.05 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.10 0.14 0.05 0.06 0.06 0.04	241776
AF/7780/DE/3 Moissac FRA, 2004	EW (200 g ai/L)	6	0.085–0.092	1413–1528	6	9–12	< 0 0 7	Whole fruit	0.08 0.15 0.06	< 0.01 < 0.01 < 0.01	0.09 0.16 0.07	241776

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicati on Interval (days)	DAT	Portion Analyse d	Myclob utanil (mg/kg)	RH- 9090 (mg/kg)	Total of myclob utanil (mg/kg)	Ref.
(Granny Smith)							14 28 35		0.05 0.04 0.04	< 0.01 < 0.01 < 0.01	0.06 0.05 0.05	
85-0119 Rosario CHL, 1984 (Delicious)	40 WP (40 %W/W)	8	0.25	—	—	—	60	Whole fruit	0.03	0.05	0.08	94313
86-0279 Kentville CAN, 1985 (Mcintosh)	40 WP (40 %W/W)	8	0.125	—	—	—	45	Whole fruit	0.01	0.01	0.02	94304
R&H/205/3/G Niedersachsen GER 1996 (Winterglock-enapfel)	EW (200 g ai /L)	12	0.090–0.095	499–527	18	11–16	0 7 15	Whole fruit Whole fruit Whole fruit	0.16 0.16 0.12	0.02 0.02 0.02	0.18 0.18 0.14	241760
R&H/205/1/G Nordrhen Westfalen GER 1996 (Jonagold)	EW (200 g ai /L)	12	0.088–0.095	1429–1587	6	10–14	0 7 14	Whole fruit Whole fruit Whole fruit	0.22 0.16 0.13	< 0.01 < 0.01 < 0.01	0.23 0.17 0.14	241760
R&H/205/2/G Neidersachsen GER 1996 (Golster)	EW (200 g ai /L)	12	0.090–0.093	1497–1557	6	11–13	0 7 14	Whole fruit Whole fruit Whole fruit	0.28 0.19 0.19	0.02 0.01 0.02	0.30 0.20 0.21	241760
R&H/205/4/G Kheinland Pfalz GER 1996 (Golden Delicious)	EW (200 g ai /L)	12	0.087–0.093	481–515	18	10–14	0 7 14	Whole fruit Whole fruit Whole fruit	0.51 0.54 0.38	0.02 0.03 0.02	0.53 0.57 0.40	241760
R&H/216/1/I Lombardia ITA, 1996 (Golden Delicious)	EW (200 g ai /L)	6	0.066–0.091	1458.3–2020.8	4.5	14	0 14	Whole fruit	0.22 0.11	0.01 0.01	0.23 0.12	241831
R&H/216/2/I Lombardia ITA, 1996 (Golden Delicious)	EW (200 g ai /L)	6	0.068–0.091	1518.1–2025.4	4.5	13–15	0 14	Whole fruit	0.19 0.09	0.01 0.01	0.20 0.10	241831
AP/3201/HL/1 F, St Porchaire FRA, 1996 (Golden Delicious)	EW (200 g ai /L)	6	0.036–0.041	794–917	4.5	10–11	0 14	Whole fruit	0.04 0.04	< 0.01 < 0.01	0.05 0.05	241831
AP/3201/HL/2 F, Villemade FRA, 1996 (Red Chief)	EW (200 g ai /L)	6	0.071–0.126	1576–2791	4.5	10–14	0 14	Whole fruit	0.10 0.05	< 0.01 < 0.01	0.11 0.06	241831
R&H/214/1/I Lombardia ITA, 1996 (Golden Delicious)	EC (240 g ai /L)	6	0.067–0.091	1497.3–2020.8	—	14	0 14	Whole fruit	0.23 0.18	0.01 0.01	0.24 0.19	138355
R&H/214/2/I Lombardia ITA, 1996	EC (240 g ai /L)	6	0.066–0.106	1471–1996.4	—	13–15	0 14	Whole fruit	0.22 0.14	0.01 0.01	0.23 0.15	138355

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicati on Interval (days)	DAT	Portion Analyse d	Myclob utanil (mg/kg)	RH- 9090 (mg/kg)	Total of myclob utanil (mg/kg)	Ref.
(Golden Delicious)												
AP/3199/HL/1 F, St Porchaire FRA, 1996 (Golden Delicious)	EC (240 g ai /L)	6	0.035–0.039	781–867	–	10–11	0 14	Whole fruit	0.05 0.04	< 0.01 < 0.01	0.06 0.05	13835 5
AP/3199/HL/2 F, Villemade FRA, 1996 (Red Chief)	EC (240 g ai /L)	6	0.071–0.136	1570–3023	–	10–14	0 14	Whole fruit	0.09 0.09	< 0.01 0.01	0.10 0.10	13835 5
R&H/217/1/1 Lombardia ITA, 1996 (Golden Delicious)	EW (60 g ai/ L)	6	0.067–0.091	1486.2–2020.8	–	14	0 14	Whole fruit	0.27 0.17	0.01 < 0.01	0.28 0.18	13714 5
R&H/217/2/1 Lombardia ITA, 1996 (Golden Delicious)	EW (60 g ai/ L)	6	0.066–0.091	1463.9–2029	–	13–15	0 14	Whole fruit	0.20 0.13	< 0.01 < 0.01	0.21 0.14	13714 5
AP/3202/HL/1 F, St Porchaire FRA, 1996 (Golden Delicious)	EW (60 g ai/ L)	6	0.036–0.040	792–885	–	10–11	0 14	Whole fruit	0.07 0.04	< 0.01 < 0.01	0.08 0.05	13714 5
AP/3202/HL/2 F, Villemade FRA, 1996 (Red Chief)	EW (60 g ai/ L)	6	0.070–0.123	1561–2738	–	10–14	0 14	Whole fruit	0.16 0.10	< 0.01 < 0.01	0.17 0.11	13714 5
R&H/215/1/1 Lombardia ITA, 1996 (Golden Delicious)	EC (120 g ai /L)	6	0.068–0.092	1490.3–2041.6	4.5	14	0 14	Whole fruit	0.20 0.16	0.02 0.02	0.22 0.18	24175 8
R&H/215/2/1 Lombardia ITA, 1996 (Golden Delicious)	EC (120 g ai /L)	6	0.066–0.092	1471–2050	4.5	13–15	0 14	Whole fruit	0.18 0.11	0.01 0.01	0.19 0.12	24175 8
AP/3200/HL/1 F, St Porchaire FRA, 1996 (Golden Delicious)	EC (120 g ai /L)	6	0.038–0.041	833–906	4.5	10	0 14	Whole fruit	0.08 0.05	< 0.01 < 0.01	0.09 0.06	24175 8
AP/3200/HL/2 F, Villemade FRA, 1996 (Red Chief)	EC (120 g ai /L)	6	0.068–0.128	1512–2852	4.5	10–14	0 14	Whole fruit	0.14 0.09	< 0.01 0.01	0.15 0.10	24175 8
AK/3637/HL/1 GBR, Notting hamshire UK, 1997 (Bramley)	EW (200 g ai /L)	12	0.088–0.094	1468.2–1561.1	6	10–14	0 7 14	Whole fruit	0.16 0.12 0.15	0.01 0.01 0.02	0.17 0.13 0.17	24182 8
AK/3637/HL/2 GBR, Kent, 1997 (Jonagold)	EW (200 g ai /L)	12	0.090–0.091	1491.8–1519.4	6	10–14	0 7 14	Whole fruit	0.14 0.12 0.14	< 0.01 < 0.01 0.01	0.15 0.13 0.15	24182 8
AK/3637/HL/3 UK Gloucestershire, GBR 1997	EW (200 g ai /L)	12	0.087–0.092	480.3–506.8	18	10–14	0 7 14	Whole fruit	0.18 0.20 0.12	0.01 0.03 0.03	0.19 0.23 0.15	24182 8

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
(Worcester Permain)												
AK/3637/HL/4 UK, Kent GBR, 1997 (Cox)	EW (200 g ai /L)	12	0.091–0.093	504–519.1	18	10–14	13	Whole fruit	0.09	0.01	0.10	241828
010139_01013 5, Sao Paulo BRA, 2001)	EC (250 g ai /L)	3	0.045	1000	4.5	6–7	07 14 21 28	Whole fruit	– ^c – – – –	– – – – –	0.06 0.04 0.02 0.01 0.01	102702 150882
010139_01013 5, Sao Paulo BRA, 2001	EC (250 g ai /L)	3	0.090	1000	9	6–7	07 14 21 28	Whole fruit	– – – – –	– – – – –	0.11 0.05 0.03 0.02 0.02	102702 150882
010139_01013 5, Santa Catarina BRA 2001–2002 (Fuji)	EC (250 g ai /L)	3	0.045	1000	4.5	5–7	15	Whole fruit	–	–	0.08	102702 150882
010139_01013 5, Santa Catarina BRA 2001–2002 (Fuji)	EC (250 g ai /L)	3	0.090	1000	9	5–7	15	Whole fruit	–	–	0.25	102702 150882
010139_01013 5, Rio Grande do Sul, BRA 2001–2002 (Royal Gala)	EC (250 g ai /L)	3	0.045	1000	4.5	6–8	14	Whole fruit	–	–	0.06	102702 150882
010139_01013 5, Rio Grande do Sul, BRA 2001–2002 (Royal Gala)	EC (250 g ai /L)	3	0.090	1000	9	6–8	14	Whole fruit	–	–	0.12	102702 150882
010139_01013 5, Sao Paulo BRA, 2001 (BRA)	WP (400 g ai /kg)	3	0.048	1000	4.8	6–7	07 14 21 28	Whole fruit	– – – – –	– – – – –	0.08 0.03 0.02 0.01 < 0.01	102701 204447
010139_01013 5, Sao Paulo BRA, 2001 (BRA)	WP (400 g ai /kg)	3	0.096	1000	9.6	6–7	07 14 21 28	Whole fruit	– – – – –	– – – – –	0.14 0.07 0.04 0.02 0.02	102701 204447
010139_01013 5, Santa Catarina BRA 2001–2002 (Fuji)	WP (400 g ai /kg)	3	0.048	1000	4.8	5–7	14	Whole fruit	–	–	0.07	102701 204447
010139_01013 5 Santa Catarina BRA 2001–2002 (Fuji)	WP (400 g ai /kg)	3	0.096	1000	9.6	5–7	14	Whole fruit	–	–	0.08	102701 204447
010139_01013 5, Rio Grande do Sul, BRA 2001–2002 (Royal Gala)	WP (400 g ai /kg)	3	0.048	1000	4.8	6–8	14	Whole fruit	–	–	0.09	102701 204447
010139_01013	WP	3	0.096	1000	9.6	6–8	14	Whole	–	–	0.06	10270

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
5 Rio Grande do Sul, BRA 2001–2002 (Royal Gala)	(400 g ai/kg)							fruit				1204447
AF/8164/DE2 Uchizy, FRA 2004 (Golden)	EW (200 g ai/L)	4	0.086–0.091	1429–1518	6	9–11	14	Whole fruit	0.21	< 0.01	0.22	101405
AF/8164/DE2 Uchizy, FRA 2004 (Golden)	EW (200 g ai/L)	12	0.085–0.10	1411–1673	6	8–11	14	Whole fruit	0.16	0.02	0.18	101405
AF/8164/DE3 Corze, FRA 2004 (Fuji No. 6)	EW (200 g ai/L)	4	0.093–0.094	1545–1568	6	9–12	< 0 0 7 14 28 35	Whole fruit	0.02 0.08 0.05 0.03 0.03 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.03 0.09 0.06 0.04 0.04 0.04	101405
AF/8164/DE3 Corze, FRA 2004 (Fuji No. 6)	EW (200 g ai/L)	12	0.090–0.097	1426–1614	6	8–12	< 0 0 7 14 28 35	Whole fruit	0.05 0.10 0.05 0.06 0.04 0.03	< 0.01 < 0.01 < 0.01 0.01 0.01 0.01	0.06 0.11 0.06 0.07 0.05 0.04	101405
AF/8164/DE4 St Remy La Varenne FRA, 2004 (Granny Smith)	EW (200 g ai/L)	4	0.094–0.097	1560–1620	6	7–15	14 14 14 14 14 14 14 14 14 14	Whole fruit Whole fruit (processing) Cooked fruit washed fruit Juice Peel and seed Pomace, dry Pomace, wet Puree Raw juice Washing water	0.11 0.08 0.04 0.08 < 0.01 0.10 0.99 0.23 0.02 0.01 0.02	< 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.06 0.23 < 0.01 < 0.01 < 0.01	0.12 0.09 0.05 0.09 < 0.02 0.11 1.05 0.24 0.03 0.02 0.03	101405
AF/8164/DE4 St Remy La Varenne FRA, 2004 (Granny Smith)	EW (200 g ai/L)	12	0.089–0.098	1487–1627	6	7–15	14	Whole fruit	0.18	0.03	0.21	101405
AF/8164/DE4 St Remy La Varenne FRA, 2004 (Granny Smith)	EW (200 g ai/L)	4	0.45–0.48	1490–1593	30	7–15	14 14 14 14 14	Whole fruit Whole fruit (processing) Washed fruit Cooked fruit Apple juice	0.65 0.51 0.52 0.28 0.06	0.03 0.05 0.03 0.03 0.02	0.68 0.56 0.55 0.31 0.08	101405

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
							14	Peel and seed	0.59	0.04	0.63	
							14	Pomace, dry	6.00	0.25	6.25	
							14	Pomace, wet	1.57	0.05	1.62	
							14	Puree	0.13	0.02	0.15	
							14	Raw juice	0.06	0.02	0.08	
							14	Washing water	0.08	< 0.01	0.09	
AF/8164/DE5 Varennes Le Grand, FRA 2004 (Elstar)	EW (200 g ai/L)	4	0.089–0.093	1487–1549	6	8–12	< 0 0 7 14 28 35	Whole fruit	0.04 0.16 0.17 0.15 0.09 0.08	< 0.01 < 0.01 < 0.01 < 0.01 0.01 0.01	0.05 0.17 0.18 0.16 0.10 0.09	101405
AF/8164/DE5 Varennes Le Grand, FRA 2004 (Elstar)	EW (200 g ai/L)	12	0.082–0.095	1374–1575	6	8–11	< 0 0 7 14 28 35	Whole fruit	0.15 0.19 0.16 0.16 0.15 0.10	0.02 0.01 0.02 0.02 0.03 0.02	0.17 0.20 0.18 0.18 0.18 0.12	101405
F06W067R Languedoc-Roussillon, FRA, 2006 (Ggolden)	EW (200 g ai/L)	6	0.093–0.095	1492–1521	6.2	9–11	< 0 0 7 14 21 28	Whole fruit	0.16 0.14 0.14 0.06 0.09 0.09	0.01 < 0.01 0.01 < 0.01 0.01 0.01	0.17 0.15 0.15 0.07 0.10 0.10	2009748
CEMS-4638A Piedmont, ITA, 2010 (Red Chief)	EW (45 g ai/L)	3	0.066–0.067	1286.1–1344.2	5.1	10	< 0 0 3 8 14	Whole fruit	0.06 0.09 0.05 0.04 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.07 0.10 0.06 0.05 0.04	2017192
CEMS-4638B Langurdod–Roussillon Southern FRA, 2010 (Braeburn)	EW (45 g ai/L)	3	0.064–0.066	1053–1095	6.1	10–11	< 0 0 3 7 14	Whole fruit	0.03 0.14 0.05 0.04 0.01	0.01 < 0.01 < 0.01 0.01 < 0.01	0.04 0.15 0.06 0.05 0.02	2017192
CEMS-4638E Berkshire RG42 6EY GBR 2010 (Bramley)	EW (45 g ai/L)	3	0.066–0.067	494.5–496.7	13.4	11–12	< 0 0 3 8 15	Whole fruit	0.10 0.24 0.24 0.17 0.11	0.01 0.01 0.01 0.01 0.01	0.11 0.25 0.25 0.18 0.12	2017192
CEMS-4638F Upper AUT AUT, 2010 (Pinova)	EW (45 g ai/L)	3	0.064–0.066	960.2–986.7	6.7	11	< 0 0 3 7 14	Whole fruit	0.07 0.11 0.09 0.08 0.15	< 0.01 < 0.01 < 0.01 < 0.01 0.01	0.08 0.12 0.10 0.09 0.16	2017192
CEMS-4943A Piedmont ITA, 2011 (Red Chief)	EW (45 g ai/L)	3	0.083–0.085	1190.7–1209.3	7	9–10	< 0 0 3 7 14	Whole fruit	0.04 0.09 0.08 0.08 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.05 0.10 0.09 0.09 0.06	2019282
CEMS-4943B Langue–Roussillon Southern FRA, 2011 (Early Red)	EW (200 g ai/L)	3	0.083–0.087	991.7–1033.3	8.4	9–12	< 0 0 3 7 14	Whole fruit	0.08 0.18 0.13 0.10 0.07	< 0.01 < 0.01 0.01 0.01 0.01	0.09 0.09 0.14 0.11 0.08	2019282
CEMS-4943E	EW	3	0.067	498	13.4	10–11	< 0	Whole	0.21	< 0.01	0.22	20192

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Suffolk, IP29 5AE GBR 2011 (Jonagored)	(45 g ai/ L)						0 3 7 14	fruit	0.61 0.36 0.29 0.34	0.01 0.01 0.01 0.01	0.62 0.37 0.30 0.35	82
CEMS-4943F Upper AUT AUT, 2011 (Greenstar)	EW (200 g ai /L)	3	0.066–0.069	978.7–1036	6.7	10	< 0 0 3 7 14	Whole fruit	0.04 0.07 0.06 0.05 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.05 0.08 0.07 0.06 0.04	20192 82
F-20211 Roenne Harburg West GER 1985 (Golster)	WP (60 g ai/ kg)	12	0.041×3 0.054×2 0.068×7	900×3 1200×3 1500×7	7.5	7–14	0 5 10 14	Whole fruit	0.14 0.09 0.08 0.07	< 0.01 < 0.01 0.01 0.01	0.15 0.10 0.09 0.08	94301
F-20212 Elbstorf–Harburg West GER 1985 (Golden Delicious)	WP (60 g ai/ kg)	12	0.041×3 0.054×2 0.068×7	900×3 1200×3 1500×7	7.5	7–14	0 5 10 14	Whole fruit	0.16 0.09 0.10 0.13	< 0.01 < 0.01 < 0.01 0.01	0.17 0.10 0.11 0.14	94301
F-20221 Pfeddersheim GER 1985 (Golden Delicious)	WP (60 g ai/ kg)	12	0.041×3 0.054×2 0.068×7	900×3 1200×3 1500×7	7.5	7–14	0 5 10 14	Whole fruit	0.30 0.25 0.23 0.20	0.02 0.02 0.02 0.02	0.32 0.27 0.25 0.22	94301
F-20231 Langenau GER 1985 (Goldparmane)	WP (60 g ai/ kg)	12	0.041×3 0.054×2 0.068×7	900×3 1200×3 1500×7	7.5	7–14	0 6 10 14	Whole fruit	0.12 0.02 0.02 0.03	0.01 < 0.01 < 0.01 < 0.01	0.13 0.03 0.03 0.04	94301
F-20241 Hattersheim GER 1985 (Roter Cox)	WP (60 g ai/ kg)	12	0.041×3 0.054×2 0.068×7	900×3 1200×3 1500×7	7.5	7–14	0 5 10 14	Whole fruit	0.08 0.11 0.08 0.07	< 0.01 0.01 0.02 0.02	0.09 0.12 0.10 0.09	94301
F-20211 Roenne Harburg GER 1985 (Golster)	WP (60 g ai/ kg)	12	0.054×3 0.072×2 0.090×7	900×3 1200×3 1500×7	10	7–14	0 5 10 14	Whole fruit	0.18 0.19 0.12 0.13	0.01 0.01 0.01 0.02	0.19 0.20 0.13 0.15	94301
F-20212 Elbstorf–Harburg GER 1985 (Golden Delicious)	WP (60 g ai/ kg)	12	0.054×3 0.072×2 0.090×7	900×3 1200×3 1500×7	10	7–14	0 5 10 14	Whole fruit	0.16 0.15 0.10 0.18	< 0.01 0.01 < 0.01 0.02	0.17 0.16 0.11 0.20	94301
F-20221 Pfeddersheim West GER 1985 (Golden Delicious)	WP (60 g ai/ kg)	12	0.054×3 0.072×2 0.090×7	900×3 1200×3 1500×7	10	7–14	0 5 10 14	Whole fruit	0.42 0.38 0.24 0.30	0.02 0.02 0.02 0.02	0.44 0.40 0.26 0.32	94301
F-20231 Langenau GER 1985 (Goldparmane)	WP (60 g ai/ kg)	12	0.054×3 0.072×2 0.090×7	900×3 1200×3 1500×7	10	7–14	0 6 10 14	Whole fruit	0.14 0.09 0.17 0.10	< 0.01 0.01 0.02 0.02	0.15 0.10 0.19 0.12	94301
F-20241 Hattersheim West GER 1985 (Roter Cox)	WP (60 g ai/ kg)	12	0.054×3 0.072×2 0.090×7	900×3 1200×3 1500×7	10	7–14	0 5 10 14	Whole fruit	0.14 0.13 0.12 0.09	0.01 0.02 0.02 0.02	0.15 0.15 0.14 0.11	94301
4218531, La Reole Southern	WP (60 g ai/	16	0.043–0.050	960–1080	4.5	–	4	Whole fruit	–	–	0.05	24188 8

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
FRA, 1985 (Belle de Boskoop)	kg)		0.060–0.065		6.0			Whole fruit	–	–	0.08	
4618420 Estillac Southern FRA, 1984 (Golden Delicious)	EW (70 g ai/L)	10	0.029–0.072	130–320	22.5	14–19	23	Whole fruit	–	–	0.04	241898
4618420 Estillac Southern FRA, 1984 (Golden Delicious)	WP (400 g ai/kg)	10	0.029–0.072	130–320	22.5	14–19	23	Whole fruit	–	–	0.04	241899
FRG72F01 St Antoine de Breuilh Southern FRA, 1986 (Jonnee)	WP (22.5 g ai/L)	10	0.03	670	4.5	10–22	7	Whole fruit	–	–	0.05	242044
FRG77F4 Liguieres de Touraine Northern FRA, 1986 (Golden Delicious)	WP (22.5 g ai/L)	13	0.04	900	4.5	9–19	26	Whole fruit	–	–	0.04	242046
DEU86F21211 Elbstorf GER, 1986 (Golden Delicious)	WP (60 g ai/L)	12	0.090	500	18	8–10	0 7 14 21 28	Whole fruit	0.25 0.21 0.09 0.16 0.10	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.26 0.22 0.10 0.17 0.11	242051
DEU86F21221 Pfeddersheim GER, 1986 (Golden Delicious)	WP (60 g ai/L)	12	0.090	500	18	8–10	0 7 14 21 28 14 14	Whole fruit Juice Wet pomace	0.21 0.18 0.15 0.16 0.13 0.02 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.22 0.19 0.16 0.17 0.14 0.03 0.09	242051
DEU86F21231 Langeman GER, 1986 (Gold-parmaene)	WP (60 g ai/L)	12	0.090	500	0.018	8–10	0 7 14 21 28	Whole fruit	0.19 0.16 0.11 0.07 0.09	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.20 0.17 0.12 0.08 0.10	242051
DEU86F21241 Mainz-Drais GER, 1986 (Golden Delicious)	WP (60 g ai/L)	12	0.090	500	18	8–10	0 7 14 21 28 14 14	Whole fruit Juice Wet pomace	0.23 0.34 0.35 0.28 0.19 0.04 0.23	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.24 0.35 0.36 0.29 0.20 0.05 0.24	242051
F34.01.86 St Martin de Sescas Southern FRA, 1986 (Golden)	WP (60 g ai/L)	1	0.030	1000	3	–	0 5 9 14 21 28	Whole fruit	– – – – – –	– – – – – –	0.03 0.03 0.02 0.02 0.02 0.01	242060
F34.01.86 St Martin de Sescas Southern FRA, 1986 (Golden)	WP (35 g ai/L)	1	0.035	1000	3.5	–	0 5 9 14 21 28	Whole fruit	– – – – – –	– – – – – –	0.10 0.05 0.04 0.01 < 0.05 < 0.05	242062

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
F34.01.86 St Martin de Sescas Southern FRA, 1986 (Golden)	WP (35 g ai/L)	1	0.045	1000	4.5	—	0 5 9 14 21 28	Whole fruit	— — — — — —	— — — — — —	0.10 0.04 0.04 0.01 < 0.05 0.05	242062
F17.01.86 St Seve Southern FRA, 1986 (Jersey Mac)	WP (35 g ai/L)	9	0.035	1000	3.5	13–15	21	Whole fruit	—	—	< 0.05	242063
F17.01.86 St Seve Southern FRA, 1986 (Jersey Mac)	WP (35 g ai/L)	9	0.045	1000	4.5	13–15	21	Whole fruit	—	—	< 0.05	242063
427/84/09 Colchester Essex, England, 1984 (Cox's orange pippin)	EW (60 g ai/L)	9	0.12	2000	6.0	—	0 14 34	Whole fruit	0.22 0.25 0.04	— — —	— — —	242161
		9	0.24	2000	12	—	0 14 34		0.39 0.22 0.10	— — —	— — —	
427/84/06 Faversham Kent, England, 1984 (Cox's orange pippin)	WP (400 g ai/kg)	7	0.030	500	6.0	—	51	Whole fruit	0.07	—	—	242161
427/84/05 Ashford, Kent England, 1984 (Cox's orange pippin)	EW (60 g ai/L)	6	0.12	2000	6.0	—	0 7 14 21 28 35 42	Whole fruit	0.01 0.01 0.01 0.01 0.01 0.01 0.01	— — — — — — —	— — — — — — —	242161
		6	0.24	2000	12	—	0 7 14 21 28 35 42		0.02 0.01 0.02 0.01 0.01 0.01 0.01	— — — — — — —	— — — — — — —	
427/84/04 Faversham Kent, England 1984 (Cox's orange pippin)	WP (400 g ai/kg)	9 9	0.12 0.24	2000 2000	6.0 12	— —	33 33	Whole fruit	0.06 0.08	— —	— —	242161
427/84/04 Faversham Kent, England 1984 (Cox's orange pippin)	EC (125 g ai/L)	9	0.24	2000	12	—	33	Whole fruit	0.08	—	—	242161
427/84/03 Loughall Armagh, N. Ireland, 1984 (Bramley seedling)	WP (400 g ai/kg)	9	0.065	1100	6.0	—	48	Whole fruit	0.04	—	—	242161
427/84/02 Bristol Avon,	WP (400 g ai/kg)	8	0.17	2800	6.0	—	14 72	Whole fruit	0.09 0.02	— —	— —	242161

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicati on Interval (days)	DAT	Portion Analyse d	Myclob utanil (mg/kg)	RH- 9090 (mg/kg)	Total of myclob utanil (mg/kg)	Ref.
England 1984 (Cox's orange pippin)	/kg)											
427/84/01 Maidstone, Kent, England 1984 (Cox's orange pippin)	WP (400 g ai /kg)	7	0.12	2000	6.0	—	14 42	Whole fruit	0.19 0.11	— —	— —	24216 1
Colchester Essex, England, 1985 (Cox's orange pippin)	WP (400 g ai /kg)	10 10	0.09 0.18	2000 2000	4.5 9.0	— —	20 20	Whole fruit	0.24 0.35	0.02 0.03	0.26 0.38	24216 1
Selling, Kent England, 1985 (Cox's orange pippin)	WP (400 g ai /kg)	10 10	0.09 0.18	2000 2000	4.5 9.0	— —	25 25	Whole fruit	0.13 0.19	0.02 0.03	0.15 0.22	24216 1
Ledbury Gloucestershire, England, 1985 (Cox's orange pippin)	EW (60 g ai/ L)	10 10	0.09 0.18	2000 2000	4.5 9.0	— —	31 31	Whole fruit	0.06 0.07	0.01 0.01	0.07 0.08	24216 1
Wisbech Cambridge-shire, England, 1985 (Bramley seedling)	WP (400 g ai /kg)	10 10	0.09 0.18	2000 2000	4.5 9.0	— —	34 34	Whole fruit	0.03 0.03	0.01 0.01	0.04 0.04	24216 1
Ledbury Gloucestershire, England, 1985 (Cox's orange pippin)	WP (400 g ai /kg)	10 10	0.09	600	15	—	0 14 48	Whole fruit	0.23 0.12 0.08	0.01 0.01 0.01	0.24 0.13 0.09	24216 1
Wisbech Cambridge-shire, England, 1985 (Bramley seedling)	WP (400 g ai /kg)	10	0.09	1100	8.0	—	0 12 40	Whole fruit	0.08 0.05 0.03	0.01 0.01 < 0.01	0.09 0.06 0.04	24216 1
Colchester Essex, England, 1985 (Cox's orange pippin)	WP (400 g ai /kg)	11	0.09	500	18	—	0 14 48	Whole fruit	0.24 0.08 0.04	0.01 0.01 < 0.01	0.25 0.09 0.05	24216 1
Rainham, Kent England, 1985 (Cox's orange pippin)	WP (400 g ai /kg)	14	0.09	500	18	—	0 14 42	Whole fruit	0.18 0.09 0.04	0.01 0.01 < 0.01	0.19 0.10 0.05	24216 1
Selling, Kent England, 1985 (Cox's orange pippin)	WP (400 g ai /kg)	14	0.045– 0.09	500 500	9.0 18	— —	0 14 36	Whole fruit	0.09 0.04 0.03	0.01 0.01 < 0.01	0.10 0.05 0.04	24216 1
FRG72F06 Lignieres de Touraine, FRA	WP (225 g ai /kg)	11	0.075	1650	4.5	—	26	Whole fruit	—	—	0.03	24216 1
4048706/1 Montebello della Battaglia PV, ITA, 1987	WP (175 g ai /kg)	14	0.045– 0.063	1300– 1800	3.5	6–15	15	Whole fruit	0.13	< 0.01	0.14	24216 1

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicati on Interval (days)	DAT	Portion Analyse d	Myclob utanil (mg/kg)	RH- 9090 (mg/kg)	Total of myclob utanil (mg/kg)	Ref.
(Cooper-4)												
4048706/3 Montebello della Battaglia PV, ITA, 1987 (Cooper-4)	WP (350 g ai /kg)	14	0.045–0.063	1300–1800	3.5	6–15	15	Whole fruit	0.14	< 0.01	0.15	242161
4048706/1 Montebello della Battaglia PV, ITA, 1987 (Cooper-4)	WP (175 g ai /kg)	15	0.045–0.063	1300–1800	3.5	6–15	15	Whole fruit	0.13	< 0.01	0.14	242161
4048706/4 Montebello della Battaglia PV, ITA, 1987 (Cooper-4)	WP (350 g ai /kg)	15	0.045–0.063	1300–1800	3.5	6–15	30	Whole fruit	0.09	< 0.01	0.10	242161

^a Residues found in control sample.

^b Sampling error.

^c Not analysed.

Pears

A total of 25 trials on pear were conducted in different representative growing areas in Belgium, Canada, Chile, France, Italy, Spain and USA during 1984 through 2011 growing seasons. Results of pear fruits are summarized in Table 52.

Table 52 Supervised trials on pear in various countries

Trial ID/Location Country/Year (variety)	Form No.	No. of Appls	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	DAT	Portion Analyse d	Myclobut anil (mg/kg)	RH- 9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
85-0412 Medford, OR USA 1985 (Bartlett)	WP (40 %W/W)	8	0.22	3741	–	–	22	Whole fruit	0.07	0.07	0.14	94327
85-0393 Orefield, PA USA 1985 (Bartlett)	WP (40 %W/W)	8	0.28	3741	–	–	7 14	Whole fruit Whole fruit	0.20 0.11	0.08 0.07	0.28 0.18	94327
85-0356 Hood River OR, USA, 1985 (U'Anjuu)	WP (40 %W/W)	7	0.22	2806	–	–	17	Whole fruit	0.26	0.02	0.28	94327
85-0314 Newtown, PA USA 1985 (Bartlett)	WP (40 %W/W)	13	0.27	3741	–	–	7 14 21	Whole fruit Whole fruit Whole fruit	0.16 0.11 0.06	0.07 0.07 0.05	0.23 0.18 0.11	94327
85-0421 Lansing, MI USA 1985 (Bartlett)	WP (40 %W/W)	8	0.28	2806	–	–	7 14 21	Whole fruit Whole fruit Whole fruit	0.42 0.20 0.13	0.09 0.10 0.09	0.51 0.30 0.22	94327
4738625 Veneto, ITA 1986 (Kaiser)	EC (125 g ai/ L)	3	0.35–0.070	1000–2000	3.5	14–20	16	Whole fruit	0.23	< 0.01	0.24	94390

Trial ID/Location Country/Year (variety)	Form No.	No. of Appls	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
4738625 Veneto, ITA 1986 (Abate)	EC (125 g ai/L)	3	0.035–0.070	1000–2000	3.5	14–20	16	Whole fruit	0.23	< 0.01	0.24	94390
4918751A Valencia ESP, 1987 (Castell)	EC (125 g ai/L)	3	0.10	2000	5	26–28	27	Whole fruit	–	–	0.03	242161
4918751A Valencia ESP, 1987 (Castell)	EC (125 g ai/L)	3	0.12	2000	6	26–28	27	Whole fruit	–	–	0.04	242161
4918751B Valencia ESP, 1987 (Castell)	EC (125 g ai/L)	3	0.12	2000	6	26–28	27	Whole fruit	–	–	0.05	242161
56, Kentville, NS, CAN, 2005 (Bartlett)	WP (400 g ai/L)	6	0.134–0.139	1379–1444	9.3–9.8	6–8	14	Whole fruit	0.27	0.10	0.37	2017298
57, Jordan Station ON, CAN, 2005 (Bartlett)	WP (400 g ai/L)	6	0.135–0.137	1084–1107	12.4–12.5	6–9	14	Whole fruit	0.05	0.07	0.12	2017298
58, Jordan Station, ON, CAN, 2005 (Bosc)	WP (400 g ai/L)	6	0.135–0.136	1090–1096	12.4	6–7	14	Whole fruit	0.09	0.01	0.10	2017298
59, Vineland Station, ON CAN, 2005 (Swiss Bartlett)	WP (400 g ai/L)	6	0.139–0.142	1127–1150	12.3–12.4	6–9	14	Whole fruit	0.08	0.08	0.16	2017298
85-0120 Rosario CHL, 1984 (Triumph)	WP (40 %W/W)	8	0.12	–	–	–	60	Whole fruit	0.03	< 0.01	0.04	94327
85-0120 Rosario CHL, 1984 (Triumph)	WP (40 %W/W)	8	0.25	–	–	–	60	Whole fruit	0.05	0.01	0.06	94327
CEMS-4638C Languedoc-Roussillon, Southren FRA, 2010 (Conference)	EW (45 g ai/L)	3	0.065–0.073	1060.3–1198.4	6.1	10	14	Whole fruit	0.28	0.01	0.29	2017192
CEMS-4638D Valencia ESP, 2010 (Ercolini)	EW (45 g ai/L)	3	0.065–0.069	1151.2–1226	5.6	10	14	Whole fruit	0.03	0.02	0.05	2017192
CEMS-4638G Burgundy, FRA, 2010 (Conference)	EW (45 g ai/L)	3	0.066–0.067	491–501.6	13.4	9–11	14	Whole fruit	0.10	0.01	0.11	2017192
CEMS-4638H Nodebais, BEL, 2010 (Conference)	EW (45 g ai/L)	3	0.065–0.069	1203.7–1283.1	5.4	10–11	13	Whole fruit	0.04	0.01	0.05	2017192
CEMS-4943C Languedoc-Roussillon, FRA, 2011 (Conference)	EW (45 g ai/L)	3	0.084–0.089	1101.6–1163.5	7.6	10–11	14	Whole fruit	0.32	0.03	0.35	2019282

Trial ID/Location Country/Year (variety)	Form No.	No. of Appls	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
CEMS-4943D Valencia, ESP, 2011 (Ercolini)	EW (45 g ai/L)	3	0.066–0.084	1157.7–1216.3	5.5–6.9	10–11	13	Whole fruit	0.03	0.02	0.05	2019282
CEMS-4943G Burgundy, FRA, 2011 (Conference)	EW (45 g ai/L)	3	0.067	496.8–498.9	13.4	10	14	Whole fruit	0.05	0.01	0.06	2019282
CEMS-4943H Nodebais, BEL, 2011 (Conference)	EW (45 g ai/L)	3	0.064–0.066	1193.1–1209	5.4	10–11	14	Whole fruit	0.06	0.01	0.07	2019282
491875A Valencia ESP, 1987 (Castell)	EC (125 g ai/L)	3 3	0.10 0.12	2000 2000	5.0 6.0	26–28	27 27	Whole fruit Whole fruit	– –	– –	0.03 0.04	242147

Stone fruits

Peaches

A total of 25 trials on peach were conducted in different representative growing areas in Canada, France, Greece, Italy, Spain and USA during 1984 through 2011 growing seasons. Results of peach fruits are summarized in Table 53.

Table 53 Supervised trials on peach in various countries

Trial ID/Location Country/Year (variety)	Form	No. of Appls	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
84-0434, NC USA 1984 (Norman)	WP (40% W/W)	11	0.28	1871	–	–	1	Fruit, pitted	1.36	0.35	1.71	94349
85-0402, CA USA 1985 (Carnival)	WP (40% W/W)	4	0.28	3741	–	5–8 (app # 3 at 178 day interval)	6	Fruit, pitted	0.32	0.01	0.33	94357
87-0172, Traver, CA USA 1987 (June Lady)	DF (60% W/W)	7	0.21	3666	–	8–27	0	Fruit, pitted	0.74	0.17	0.91	94363 94328
							7	Fruit, pitted	0.36	0.25	0.61	
							14	Fruit, pitted	0.24	0.22	0.46	
							20	Fruit, pitted	0.11	0.11	0.22	
87-0235, Laton, USA, 1987 (Loadel)	DF (60% W/W)	7	0.21	3666	–	16–38	0	Fruit, Pitted (0 day PHI)	0.66	0.10	0.76	94363 94328
							7	Fruit, pitted	0.27	0.18	0.45	
87-0235, Laton, USA, 1987 (Loadel)	DF (60% W/W)	7	0.43	3666	–	16–38	0	Fruit, Pitted (0 day PHI)	1.86	0.18	2.04	94363 94328
87-0490, Ripon, CA USA 1987 (Fay Elberta)	DF (60% W/W)	9	0.21	2806	–	14–30	0	Fruit, Pitted (0 day PHI)	0.38	0.17	0.55	94363 94328
							7	Fruit, pitted	0.23	0.15	0.38	

Trial ID/Location Country/Year (variety)	Form	No. of Appl's	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
87-0165, Clarksville, AR, USA, 1987 (Redhaven)	DF (60% W/W)	9	0.21	935.3	—	6–15	0	Fruit, Pitted (0 day PHI)	0.33	0.02	0.35	94363 94328
							7	Fruit, pitted	0.21	0.10	0.31	
87-0165, Clarksville, AR, USA, 1987 (Redhaven)	DF (60% W/W)	9	0.43	935.3	—	6–15	0	Fruit, Pitted (0 day PHI)	0.35	0.09	0.44	94363 94328
87-0243, Jackson SPR, NC, USA, 1987 (Winblo)	DF (60% W/W)	10	0.21	1871	—	6–15	0	Fruit, Pitted (0 day PHI)	0.84	0.28	1.12	94363 94328
							7	Fruit, pitted	0.84	0.69	1.53	
87-0554, New Franklin USA 1987 (July Elberta)	DF (60% W/W)	11	0.21	187	—	7–14	0	Fruit, Pitted (0 day PHI)	0.62	0.20	0.82	94363 94328
87-0372 Winchester, VA, USA, 1987 (Red Haven)	DF (60% W/W)	9	0.14–0.21	1871	—	7–20	0	Fruit, Pitted (0 day PHI)	0.85	0.21	1.06	94363 94328
87-0372 Winchester, VA, USA, 1987 (Red Haven)	DF (60% W/W)	9	0.28–0.42	1871	—	7–20	0	Fruit, Pitted (0 day PHI)	1.77	0.32	2.09	94363 94328
87-0274, PA USA 1987 (Sunhigh)	DF (60% W/W)	8	0.22	2806	—	7–32	0	Fruit, Pitted (0 day PHI)	1.23	0.31	1.54	94363 94328
							7	Fruit, pitted	0.45	0.46	0.91	
87-0274, PA USA 1987 (Sunhigh)	DF (60% W/W)	8	0.45	2806	—	7–32	0	Fruit, Pitted (0 day PHI)	3.22	0.52	3.74	94363 94328
88-0142, GA USA 1988 (June Gold)	DF (60% W/W)	7	0.21	2806	—	10–11	14	Fruit, pitted	0.15	0.16	0.31	94363 94328
88-0143, GA USA 1988 (June Gold)	EC (25.8% W/W)	7	0.21	2806	—	10–11	14	Fruit, pitted	0.17	0.17	0.34	94363 94328
88-0144, GA USA 1988 (June Gold)	SYSTHANE (40 %W/W)	7	0.21	2806	—	10–11	14	Fruit, pitted	0.12	0.10	0.22	94363 94328
88-0249, Centreville, GA, USA, 1988 (Loring)	EC (25.8% W/W)	7	0.21	2806	—	14–19	14	Fruit, pitted	0.07	0.08	0.15	94363 94328
88-0250 Centreville, GA, USA, 1988 (Loring)	WP (40 %W/W)	7	0.21	2806	—	14–19	14	Fruit, pitted	0.07	0.09	0.16	94363 94328

Trial ID/Location Country/Year (variety)	Form	No. of Appl's	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
88-0251 Centreville, GA, USA, 1988 (Loring)	DF (60% W/W)	7	0.21	2806	—	14–19	14	Fruit, pitted	0.13	0.18	0.31	94363 94328
89-0319, Byron, GA USA 1989 (Rio Osa Gem)	WP (40 %W/W)	7	0.21	393	—	4–39	14	Fruit, pitted	0.14	0.14	0.28	94363 94328
89-0319, Marysville, CA, USA, 1989 (Lodell)	WP (40 %W/W)	7	0.21	2338	—	4–39	14	Fruit, pitted	0.19	0.24	0.43	94363 94328
89-0163 Byron, GA USA 1989 (Rio Osa Gem)	WP (40 %W/W)	7	0.21	468	—	9–16	14	Fruit, pitted	0.10	0.13	0.23	94363 94328
89-0163 Byron, GA USA 1989 (Rio Osa Gem)	WP (40 %W/W)	7	0.21	2338	—	9–16	14	Fruit, pitted	0.16	0.13	0.29	94363 94328
91-0033, Farmersville, CA, USA, 1991 (Queen Crest)	WP (40 %W/W)	2	0.14	196	—	14	5	Fruit, pitted	0.06	0.03	0.09	94363 94328
91-0033, Farmersville, CA, USA, 1991 (Queen Crest)	WP (40 %W/W)	2	0.14	870-889	—	14	5	Fruit, pitted	0.14	0.07	0.21	94363 94328
87-0556, New Franklin, MO USA 1987 (Red Haven)	DF (60% W/W)	11	0.43	187	—	7–14	7 14	Fruit, pitted Fruit, pitted	1.10 0.65	0.40 0.49	1.50 1.14	94353
87-0557, New Franklin, MO USA 1987 (Loring)	DF (60% W/W)	11	0.43	187	—	7–14	7 14	Fruit, pitted Fruit, pitted	0.94 0.65	0.22 0.28	1.16 0.91	94353
87-0165, Clarksville, AR, USA, 1987 (Redhaven)	DF (60% W/W)	9	0.21	935.3	—	6–15	0 7 14	Fruit, Pitted (0 day PHI) Fruit, pitted Fruit, pitted	0.34 0.18 0.10	0.03 0.04 0.04	0.37 0.22 0.14	94354
87-0172, Traver, CA USA 1987 (June Lady)	DF (60% W/W)	7	0.21	3666	—	8–27	0 7 14	Fruit, pitted Fruit, pitted Fruit, pitted	0.75 0.28 0.23	0.07 0.06 0.06	0.82 0.34 0.29	94354

Trial ID/Location Country/Year (variety)	Form	No. of Appl's	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
87-0235, Laton, USA, 1987 (Lodel)	DF (60% W/W)	7	0.43	3666		16–38	7 14	Fruit, Pitted Fruit, Pitted	0.30 0.24	0.11 0.11	0.41 0.35	94355
87-0274, PA USA 1987 (Sunhigh)	DF (60% W/W)	8	0.45	2806	–	7–32	7	Fruit, Pitted	0.48	0.21	0.69	94355
87-0243, Jackson SPR, NC, USA, 1987 (Winblo)	DF (60% W/W)	10	0.21	1871	–	6–15	0 7 14 21	Fruit, Pitted (0 day PHI) Fruit, pitted Fruit, pitted Fruit, pitted	1.40 0.90 0.38 0.63	0.18 0.25 0.29 0.37	1.58 1.15 0.67 1.00	94362
RF 2072 14-2 Rhone Southern FRA, 1992 (Gilda)	EC (75 g ai/L)	7	–	500	0.0038	7–16 (app # 6 at 82 day interval)	2	Whole fruit	–	–	0.03	241980
4839322 Verona, ITA 1993 (Maria Bianca)	EC (125 g ai/L)	4	0.063 – 0.116	1012– 1855	6.25	9–11	0 7 14	Whole fruit	0.24 0.10 0.04	0.04 0.05 0.03	0.28 0.15 0.07	242018
RAS/31/1/I Lombardia ITA, 1997 (Spring Lady)	EC (240 g ai/L)	5	0.077 – 0.117	1023– 1561	7.48– 7.60	13–15	0 3 7 0 3 7	Flesh Whole fruit	0.08 0.11 0.05 0.07 0.09 0.04	0.01 0.01 < 0.01 0.01 0.01 < 0.01	0.09 0.12 0.06 0.08 0.10 0.05	241821
RAS/31/2/I Lombardia ITA, 1997 (Maycrest)	EC (240 g ai/L)	5	0.076 – 0.117	1008– 1565	7.48– 7.49	13–16	0 3 7 0 3 7	Flesh Whole fruit	0.23 0.09 0.06 0.21 0.08 0.06	0.02 0.03 0.02 0.01 0.03 0.02	0.25 0.12 0.08 0.22 0.11 0.08	241821
RAS/31/1/G East Peloponese GRC, 1997 (Maycrest)	EC (240 g ai/L)	5	0.097 – 0.112	1299– 1503	7.46– 7.47	10–12	0 3 7 0 3 7	Flesh Whole fruit	0.34 0.24 0.14 0.30 0.22 0.13	0.02 0.03 0.04 0.02 0.02 0.04	0.36 0.27 0.18 0.32 0.24 0.17	241821
RAS/31/3/I Veneto, ITA 1997 (La Fayette)	EC (240 g ai/L)	5	0.091 – 0.114	1222– 1523	7.49	10–11	0 3 7 0 3 7	Flesh Whole fruit	0.26 0.10 0.05 0.23 0.10 0.05	0.01 0.02 0.01 0.01 0.02 0.01	0.27 0.12 0.06 0.24 0.12 0.06	241821
CEMS-4944A Piedmont ITA, 2011 (Rome star)	EW (45 g ai/L)	3	0.089 – 0.091	987.62 – 1014.29	9	10–11	< 0 0 3 6 15 < 0 0 3 6 15	Flesh Whole Fruit (calculated)	0.05 0.09 0.09 0.08 0.03 0.04 0.09 0.09 0.07 0.03	< 0.01 < 0.01 0.01 0.01 0.01 < 0.01 < 0.01 0.01 0.01 0.01 0.01	0.06 0.10 0.10 0.09 0.04 0.05 0.10 0.10 0.08 0.04	2017646

Trial ID/Location Country/Year (variety)	Form	No. of Appl's	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
CEMS-4944F Languedoc-Roussillon Southern FRA, 2011 (Western Red)	EW (45 g ai/L)	3	0.091 — 0.094	1015.6 — 1046.7	9	9–11	< 0 0 3 7 14 < 0 0 3 7 14	Flesh Whole Fruit (calculated)	0.06 0.12 0.10 0.06 0.04 0.05 0.11 0.09 0.05 0.04	0.02 0.02 0.02 0.01 0.03 0.02 0.02 0.02 0.01 0.03	0.08 0.14 0.12 0.07 0.07 0.07 0.13 0.11 0.06 0.07	2017646
RF 2072 Rhone Southern FRA, 1992 (Corine)	EC (75 g ai/L)	5	—	500	3.75	8–16	42	Whole fruit	—	—	< 0.02	241979
93FARRS02 Noves FRA, 1993 (Snow Queen)	EC (125 g ai/L)	4	0.0625	300	62.5	10–14	0 8 15	Whole fruit	0.22 0.10 0.04	0.07 0.04 0.05	0.29 0.14 0.09	241995
93FARRS03 Cabannes FRA, 1993 (Super Crimson Gold)	EC (125 g ai/L)	4	0.0625	300	62.5	12–14	0 7 14	Whole fruit	0.08 0.03 0.02	0.03 0.02 0.02	0.11 0.05 0.04	241994
97RHC09-C British Col CAN, 1997 (NA)	WP (40 %W/W)	6	0.12–0.15	2178–2480	5.4–6.1	7–9 (app # 4 at 72 day interval)	1	Fruit, pitted	0.28	—	—	135366
97RHC09-F Ontario CAN, 1997 (Viglo)	WP (40 %W/W)	6	0.13–0.14	936.5–1025	13.6	7–12 (app # 4 at 84 day interval)	1	Fruit, pitted	0.44	—	—	135366
97RHC09-G Ontario CAN, 1997 (Viglo)	WP (40 %W/W)	6	0.13–0.14	945–1034.5	13.5–13.6	9–21 (app # 4 at 48 day interval)	1	Fruit, pitted	0.24	—	—	135366
97RHC09-H Ontario CAN, 1997 (Viglo)	WP (40 %W/W)	6	0.12–0.14	905.5–1029	13.4–13.6	9–15 (app # 4 at 48 day interval)	1	Fruit, pitted	0.39	—	—	135366
S06W046R Valencia ESP, 2006 (Royal Gladys)	EW (200 g ai/L)	6	0.13	1796–1810	7.2	9–12	< 0 0 7 14 21 27 < 0 0 7 14 21 27	Flesh Whole fruit	0.18 0.44 0.19 0.11 0.08 0.05 0.15 0.37 0.16 0.09 0.07 0.05	0.04 0.05 0.04 0.04 0.03 0.03 0.03 0.04 0.03 0.04 0.03 0.03	0.22 0.49 0.23 0.15 0.11 0.08 0.18 0.41 0.19 0.13 0.10 0.08	2009589
F06W066R Languedoc-Roussillon Southern FRA, 2006 (Western Red)	EW (200 g ai/L)	6	0.13–0.14	1773–1966	7.2	9–11	7 7	Flesh Whole fruit	0.11 0.09	0.03 0.03	0.14 0.12	2009589

Trial ID/Location Country/Year (variety)	Form	No. of Appl	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
F07W074R Languedoc-Roussillon Southern FRA, 2007 (Tardibelle)	EW (200 g ai/L)	6	0.12–0.13	1583–1813	7.5	9–12	< 0 0 3 7 14 21	Whole fruit	0.09 0.28 0.22 0.11 0.07 0.04	0.03 0.03 0.04 0.03 0.03 0.02	0.12 0.31 0.26 0.14 0.10 0.06	2000789
CEMS-4639A Piedmont ITA, 2010 (Rome star)	EW (45 g ai/)	3	0.060–0.061	997.1–1017.1	6.0	10	< 0 0 3 7 14 < 0 0 3 7 14	Flesh Whole Fruit (calculated)	0.04 0.05 0.05 0.03 0.01 0.03 0.05 0.05 0.03 0.01	0.01 < 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.05 0.06 0.06 0.04 0.02 0.04 0.06 0.06 0.04 0.02	2017193
CEMS-4639B Languedoc-Roussillon Southern FRA, 2010 (Western Red)	EW (45 g ai/)	3	0.058–0.060	961.7–998.3	6.0	10	< 0 0 3 7 14 < 0 0 3 7 14	Flesh Whole Fruit (calculated)	0.06 0.18 0.12 0.08 0.05 0.05 0.15 0.10 0.07 0.04	0.02 0.01 0.02 0.02 0.02 0.02 0.01 0.01 0.02 0.02	0.08 0.19 0.14 0.10 0.07 0.07 0.16 0.11 0.09 0.06	2017193
4918518 Lleida, ESP 1985 (Catarina)	WP (60 g ai/L)	5	0.108 0.081	1800 1800	6.0 4.5	15–17	20 20	Whole fruit Whole fruit	– –	– –	0.03 0.02	241918
4318660 Ginesta Southern FRA, 1986 (Cans)	EC (125 g ai/L)	8 8 9 9	0.05 0.075 0.05 0.075	1000 1000 1000 1000	5.0 7.5 5.0 7.5	13–20	23 23 9 9	Whole fruit w/o stone	– – – –	– – – –	0.05 0.07 0.02 0.07	242050
4318740b Le Somail Southern FRA, 1987 (Cans)	EC (75 g ai/L)	7	0.056	1000	5.6	13–26	7 14 7 14	Whole fruit w/o stone Whole fruit with stone	– – – – –	– – – – –	0.25 0.12 0.20 0.10	242131
F91.01.87 St Martin de Grau, Southern FRA, 1987 (Henry)	EC (125 g ai/L)	1	0.062	1200	5.0	–	0 2 4 7 10 14	Whole fruit	– – – – – –	– – – – – –	0.50 0.20 0.20 0.15 0.10 0.10	242144
4918714 Valencia ESP, 1987 (Spring Crees)	EC (125 g ai/L)	3	0.15 0.10	2000 2000	7.5 5.0	13–16	20 20	Whole fruit Whole fruit	– –	– –	0.03 0.03	242145
4918703A Tarragona ESP, 1987 (Catherine)	EC (75 g ai/L)	8	0.045	1000	4.5	6–31	0 4 14 0 4 14	Whole fruit w/o stone Whole fruit with stone	– – – – – – – –	– – – – – – – –	0.40 0.10 0.10 0.35 0.10 0.10	242172
F60.04.87 St gilles Southern	EC (125 g ai/L)	10	0.075	400	18	13–22	11	Whole fruit w/o stone	–	–	0.11	242182

Trial ID/Location Country/Year (variety)	Form	No. of Appl s	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
FRA, 1987 (Gold 9)							11	Whole fruit with stone	—	—	0.10	
F31.02.86 Bessan Southern FRA, 1986 (O'Henry)	EC (125 g ai/L)	1	0.075	1000	7.5	—	0 5 10 14 21 28	Whole fruit w/o stone Whole fruit with stone	— — — — — —	— — — — — —	0.11 0.05 0.04 0.03 0.03 0.02	242185
4918803 Valencia ESP, 1988 (Coigua)	WP (8 g ai/L)	1 1	0.08 0.12	2500 2500	3.2 4.8	— —	7 7	Whole fruit w/o stone Whole fruit with stone	— —	— —	0.03 0.04 0.03 0.03	242196

Cherries

A total of 65 trials on cherry were conducted in different representative growing areas in Austria, Belgium, Canada, France, Germany, Hungary, UK and USA during 1984 through 2011 growing seasons. Results of cherry fruits are summarized in Table 54.

Table 54 Supervised trials on cherry in various countries

Trial ID/Location Country/Year (variety)	Form	No. of App s	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
Sour Cherries 84-0166, MI USA 1984 (Mon)	WP (40 %W/W)	5	0.43	—	—	11–15	0	Fruit, pitted	2.64	0.44	3.08	94338
Sour Cherries 84-0210, WI USA 1984 (Montgomery)	WP (40 %W/W)	6	0.42	2806	—	9–15	9	Fruit, pitted	0.56	0.39	0.95	94338
Sour Cherries 84-0210, WI USA 1984 (Montgomery)	WP (40 %W/W)	7	0.42	2806	—	9–15	1	Fruit, pitted	1.00	0.48	1.48	94338
Sour Cherries 88-0026, UT USA 1987 (Montmorency)	DF (60% W/W)	8	0.14	187	—	5–16	14	Fruit, pitted	0.16	0.07	0.23	94335
Sour Cherries 88-0026, UT USA 1987 (Montmorency)	DF (60% W/W)	8	0.28	187	—	5–16	14	Fruit, pitted	0.39	0.07	0.46	94335
Sour Cherries	DF (60%)	6	0.21	2806	—	7–19	0	Fruit,	1.04	0.57	1.61	94401

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
87-0262, MI USA 1987 (Montmorency)	W/W)						7	Pitted (0 day PHI) Fruit, pitted	0.77	0.95	1.72	110749
Sour Cherries 87-0262, MI USA 1987 (Montmorency)	DF (60% W/W)	6	0.43	2806	—	7–19	0	Fruit, Pitted (0 day PHI)	2.10	0.95	3.05	94401 110749
Sour Cherries 87-0332, WI USA 1987 (Montmorency)	DF (60% W/W)	6	0.21	1871	—	13–15	0 7	Fruit, Pitted (0 day PHI) Fruit, pitted	1.12 1.01	0.40 0.94	1.52 2.05	94401 110749
Sour Cherries 87-0332, WI USA 1987 (Montmorency)	DF (60% W/W)	6	0.43	1871	—	13–15	7	Fruit, pitted	0.56	0.72	1.28	94401 110749
Sour Cherries 89-0128, PA USA 1989 (Montmorency)	WP (40 %W/W)	6	0.21	468	—	7–14	7	Fruit, pitted	0.02	0.32	0.34	94401 110749
Sour Cherries 89-0128, PA USA 1989 (Montmorency)	WP (40 %W/W)	6	0.21	1684	—	7–14	7	Fruit, pitted	0.02	0.43	0.45	94401 110749
Sweet cherry 87-0108, CA USA 1987 (Bing)	DF (60% W/W)	5	0.21	4882	—	8–19	0 7 15 21	Fruit, pitted	0.68 0.44 0.27 0.16	0.11 0.23 0.21 0.46	0.79 0.67 0.48 0.62	94401 110749
Sweet cherry 87-0111, CA USA 1987 (Bing)	DF (60% W/W)	5	0.21	4882	—	4–15	0 7 15 21	Fruit, Pitted	0.85 0.41 0.22 0.23	0.22 0.19 0.27 0.39	1.07 0.60 0.49 0.62	94401 110749
Sweet cherry 87-0111, CA USA 1987 (Bing)	DF (60% W/W)	5	0.43	4882	—	4–15	0	Fruit, Pitted (0 day PHI)	1.09	0.26	1.35	94401 110749
Sweet cherry 87-0209, OR USA 1987 (Bing)	DF (60% W/W)	5	0.21	921	—	11–19	0 7	Fruit, Pitted	0.20 0.08	0.02 0.05	0.22 0.13	94401 110749
Sweet cherry 87-0209, OR USA 1987 (Bing)	DF (60% W/W)	5	0.43	921	—	11–19	0 7	Fruit, Pitted	0.47 0.15	0.06 0.11	0.53 0.26	94401 110749
Sweet cherry 87-0261, MI USA 1987 (Hedelfinger)	DF (60% W/W)	6	0.21	2806	—	7–19	0 7 14	Fruit, Pitted	1.44 0.44 0.90	0.61 0.69 1.51	2.05 1.13 2.41	94401 110749
Sweet cherry 88-0124, CA USA 1988 (Bing)	DF (60% W/W)	5	0.21	1057	—	7–26	14	Fruit, pitted	0.09	0.09	0.18	94401 110749
Sweet cherry 88-0125, CA USA	EC (25.8 g ai/L)	5	0.21	1057	—	7–26	14	Fruit, pitted	0.30	0.21	0.51	94401 110749

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
1988 (Bing)												
Sweet cherry 88-0126, CA USA 1988 (Bing)	WP (40 %W/W)	5	0.21	1057	—	7–26	14	Fruit, pitted	0.09	0.10	0.19	94401 110749
Sweet cherry 88-0198, WA USA 1988 (Bing)	DF (60% W/W)	5	0.21	3058	—	14–16	14	Fruit, pitted	0.11	0.15	0.26	94401 110749
Sweet cherry 88-0199, WA USA 1988 (Bing)	EC (25.8 g ai/L)	5	0.21	—	—	14–16	14	Fruit, pitted	0.05	0.08	0.13	94401 110749
Sweet cherry 88-0197, WA USA 1988 (Bing)	WP (40 %W/W)	5	0.21	3058	—	13–16	14	Fruit, pitted	0.08	0.04	0.12	94401 110749
Sweet cherry 90-0023, ID USA 1989 (Bing)	WP (40 %W/W)	6	0.21	393	—	5–12	14	Fruit, pitted	0.34	0.55	0.89	94401 110749
Sweet cherry 90-0023, ID USA 1989 (Bing)	WP (40 %W/W)	6	0.21	2338	—	5–12	14	Fruit, pitted	0.21	0.39	0.60	94401 110749
Sweet cherry 87-0261, MI USA 1987 (Hedelfinger)	DF (60% W/W)	6	0.21	187	—	7–19	7 14	Fruit, pitted	0.92 0.61	0.60 0.73	1.52 1.34	94331
Sour Cherries 87-0262, MI USA 1987 (Montmorency)	DF (60% W/W)	6	0.21	187	—	7–19	7 14	Fruit, pitted	0.84 0.35	0.58 0.60	1.42 0.95	94331
Sour Cherries 87-0332, WI USA 1987 (Montmorency)	DF (60% W/W)	6	0.21	1871	—	13–15	8 14	Fruit, Pitted Fruit, pitted	0.38 0.19	0.43 0.51	0.81 0.70	94331
Sweet cherry 87-0111, CA USA 1987 (Bing)	DF (60% W/W)	5	0.43	4882	—	4–15	7 15	Fruit, Pitted Fruit, Pitted	0.36 0.53	0.09 0.23	0.45 0.76	94332
Sweet cherry 87-0108, CA USA 1987 (Bing)	DF (60% W/W)	5	0.21	4882	—	8–19	0 7 15 21	Fruit, pitted	0.92 0.45 0.39 0.01	0.13 0.06 0.11 0.01	1.05 0.51 0.50 0.02	94332
Sweet cherry 87-0209, OR USA 1987 (Bing)	DF (60% W/W)	5	0.21	921	—	11–19	0 7 14 21	Fruit, Pitted	0.28 0.19 0.10 0.03	0.04 0.04 0.05 0.03	0.32 0.23 0.15 0.06	94402
Sweet cherry 87-0172, CA USA 1987 (June Lady)	DF (60% W/W)	7	0.21	3666	5.7	8–27	0 7 14 20	Fruit, Pitted	0.75 0.28 0.23 0.11	0.07 0.06 0.06 0.07	0.82 0.34 0.29 0.18	94402
Cherries G07W041R Lower Saxony	EW (200 g ai/L)	3	0.133 — 0.139	1470– 1539	9	10, 11	< 0 0 3	Whole fruit	0.06 0.21 0.19	0.03 0.03 0.05	0.09 0.24 0.24	200078 8

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
GER 2007 (Regina)							7 14		0.21 0.06	0.09 0.05	0.30 0.11	
Cherries G07W041R Lower Saxony GER 2007 (Regina)	EW (45 g ai/L)	3	0.128 – 0.136	1457–1545	8.8	10, 11	< 0 0 3 7 14	Whole fruit	0.06 0.19 0.25 0.16 0.07	0.04 0.04 0.06 0.06 0.05	0.10 0.23 0.31 0.22 0.12	2000788
Cherries G07W544R Lower Saxony GER 2007 (Bianca)	EW (200 g ai/L)	3	0.135 – 0.148	1490–1640	9.04	9, 11	< 0 0 3 7 14 21	Whole fruit	0.07 0.15 0.22 0.13 0.09 0.05	0.04 0.04 0.06 0.06 0.07 0.07	0.11 0.19 0.28 0.19 0.16 0.12	2000788
Cherries G07W544R Lower Saxony GER 2007 (Bianca)	EW (45 g ai/L)	3	0.132 – 0.143	1499–1629	8.8	9, 11	< 0 0 3 7 14 21	Whole fruit	0.09 0.19 0.15 0.12 0.08 0.05	0.04 0.06 0.05 0.07 0.07 0.06	0.13 0.25 0.20 0.19 0.15 0.11	2000788
Sour Cherries R&H/206/1/G Nordrhein–Westfalen GER 1996 (Morellenfeuer)	EW (200 g ai/L)	3	0.134 – 0.136	1482–1510	9	3, 44	0 14 21 21	Flesh Whole fruit	1.20 0.07 0.02 0.01	< 0.01 0.01 < 0.01 < 0.01	1.21 0.08 0.03 0.02	241759
Sour Cherries R&H/206/3/G Niedersachsen GER 1996 (Johanna)	EW (200 g ai/L)	3	0.130 – 0.146	481–538	27	3, 48	0 14 21 21	Flesh Whole fruit	3.81 0.24 0.08 0.07	< 0.01 0.03 < 0.01 < 0.01	3.82 0.27 0.09 0.08	241759
Sweet Cherries R&H/206/2/G Niedersachsen GER 1996 (Johanna)	EW (200 g ai/L)	3	0.136 – 0.142	1508–1583	9	4, 45	0 14 21 21	Flesh Whole fruit	0.75 0.12 < 0.01 < 0.01	< 0.01 0.02 < 0.01 < 0.01	0.76 0.14 < 0.02 < 0.02	241759
Sweet Cherries R&H/206/4/G Brandenburg GER 1996 (Van)	EW (200 g ai/L)	3	0.134 – 0.168	498–623	27	3, 35	0 14 21 28 21 28	Flesh Whole fruit	0.40 0.02 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.41 0.03 < 0.02 < 0.02 < 0.02 < 0.02	241759
Sour Cherries RAS/19/1/G Rheinland–Pfalz GER 1997 (Schattenmorellen)	EW (200 g ai/L)	3	0.134 – 0.139	1484–1540	9	17, 66	0 14 21 0 14 21	Flesh Whole fruit	0.57 0.13 0.03 0.50 0.11 0.03	< 0.01 0.02 < 0.01 < 0.01 0.02 < 0.01	0.58 0.15 0.04 0.51 0.13 0.04	241820
Sour Cherries RAS/19/3/G Nordrhein–Westfalen GER 1997 (Morellenfeuer)	EW (200 g ai/L)	3	0.134 – 0.138	496–511	27	15, 43	0 14 21 0 14 21	Flesh Whole fruit	0.42 0.12 0.04 0.33 0.11 0.03	< 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.43 0.13 0.05 0.34 0.12 0.04	241820
Sweet Cherries RAS/19/2/G Niedersachsen GER 1997 (Karina)	EW (200 g ai/L)	3	0.136 – 0.137	1510–1518	9	7, 60	0 14 21 0 14 21	Flesh Whole fruit	0.56 0.22 0.02 0.24 0.13 0.02	< 0.01 0.04 < 0.01 < 0.01 0.02 < 0.01	0.57 0.26 0.03 0.25 0.15 0.03	241820

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
Sweet Cherries RAS/19/4/G Brandenburg GER 1997 (Van)	EW (200 g ai/L)	3	0.134 – 0.139	496–515	27	4, 42	0 14 21 0 14 21	Flesh Whole fruit	0.51 0.17 0.01 0.26 0.11 < 0.01	< 0.01 0.03 < 0.01 < 0.01 0.02 < 0.01	0.52 0.20 0.02 0.27 0.13 < 0.02	241820
Sweet Cherries CEMS-4641F Gyorszentivan HUN 2010, (Germesdorfer)	EW (45 g ai/L)	2	90.4 90.5	1004.3 1004.6	9 9	10	< 0 0 3 7 14 < 0 0 3 7 14	Flesh Whole Fruit (calculated)	0.33 0.75 0.75 0.40 0.29 0.31 0.70 0.70 0.37 0.27	0.10 0.09 0.13 0.15 0.25 0.10 0.08 0.12 0.14 0.23	0.43 0.84 0.88 0.55 0.54 0.41 0.78 0.82 0.51 0.50	2018493
Sweet Cherries CEMS-4641D 4257 Rosoux–Crenwick BEL 2010 (Kordia)	EW (45 g ai/L)	2	90 90.5	1199.4 1207	7.5 7.5	10	6 6	Flesh Whole Fruit (calculated)	0.19 0.18	0.07 0.07	0.26 0.25	2018493
Sweet Cherries CEMS-4641A Herefordshire HR9 7UD GBR 2010 (Sweet Heart)	EW (45 g ai/L)	2	89.2 89.4	495.6 496.4	17.9 17.9	10	< 0 0 3 7 13 < 0 0 3 7 13	Flesh Whole Fruit (calculated)	0.18 0.81 0.56 0.66 0.28 0.17 0.74 0.51 0.61 0.27	0.10 0.10 0.11 0.21 0.17 0.09 0.10 0.10 0.20 0.16	0.28 0.91 0.67 0.87 0.45 0.26 0.84 0.61 0.81 0.43	2018493
Sweet Cherries CEMS-4641B Bayern GER 2010 (Regina)	EW (45 g ai/L)	2	89.2 83.9	1239 1165.3	7.2 7.2	10	< 0 0 3 7 14 < 0 0 3 7 14	Flesh Whole Fruit (calculated)	0.09 0.17 0.14 0.07 0.06 0.08 0.15 0.13 0.06 0.06	0.06 0.04 0.06 0.05 0.08 0.05 0.03 0.05 0.05 0.07	0.15 0.21 0.20 0.12 0.14 0.13 0.18 0.18 0.11 0.13	2018493
Sweet Cherries CEMS-4946C Burgundy FRA, 2011 (Sunburst)	EW (45 g ai/L)	2	0.091	504	18	10	7 7	Flesh Whole Fruit (calculated)	0.27 0.24	0.11 0.10	0.38 0.34	2018481
Sweet Cherries CEMS-4946D Gyorszentivan HUN, 2011 (Germesdorfer)	EW (45 g ai/L)	2	0.090 – 0.091	1001.2 – 1010.3	9	10	7 7	Flesh Whole Fruit (calculated)	0.38 0.31	0.14 0.11	0.52 0.42	2018481
Sweet Cherries CEMS-4946B Upper AUT AUT, 2011 (Regina)	EW (45 g ai/L)	2	0.087 – 0.089	966.2–991.2	9	10	< 0 0 3 7 14 < 0 0 3 7	Flesh Whole Fruit (calculated)	0.08 0.14 0.18 0.09 0.10 0.07 0.12 0.16 0.08	0.05 0.03 0.06 0.05 0.07 0.04 0.03 0.05 0.04	0.13 0.17 0.24 0.14 0.17 0.11 0.15 0.21 0.12	2018481

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
							14		0.09	0.06	0.15	
Sweet Cherries CEMS-4946A Herefordshire, HR9 7UD GBR 2011 (Sweet Heart)	EW (45 g ai/L)	2	0.090	498	18	11	< 0 0 3 8 14 < 0 0 3 8 14	Flesh Whole Fruit (calculated)	0.12 0.70 0.44 0.22 0.16 0.10 0.54 0.35 0.18 0.13	0.11 0.10 0.09 0.11 0.12 0.09 0.08 0.08 0.09 0.10	0.23 0.80 0.53 0.33 0.28 0.19 0.62 0.43 0.27 0.23	2018481
Sweet Cherries 97RHC09-B British Col CAN, 1997 (Sunburst)	WP (40 %W/W)	6	0.125 – 0.154	2293–2503	5.5–6.1	7 (app #4 at 39 day int)	1	Fruit, pitted	0.32	–	–	135366
Sweet Cherries 97RHC09-A British Col CAN, 1997 (Lapins)	WP (40 %W/W)	6	0.131 – 0.152	1706–1992	7.6–7.7	7–10 (app # 4 at 31 day int)	1	Fruit, pitted	0.64	–	–	135366
Sweet Cherries 97RHC09-E Ontario CAN, 1997 (690618)	WP (40 %W/W)	6	0.127 – 0.145	958.5–1065	13.6	10–15	1	Fruit, pitted	0.19	–	–	135366
DEU87F21111, Elbstorf GER 1987 (Schattenmorelle)	WP (60 g ai/L)	2	0.090	1000	9.0	–	35 49 63 83	Whole fruit	0.01 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.02 0.02 < 0.02 < 0.02	242171
DEU87F21131, Langenau GER 1987 (Schattenmorelle)	WP (60 g ai/L)	2	0.090	1000	9.0	–	73 80 87 94	Whole fruit	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02	242171
DEU87F20531, Langenau GER 1988 (Schattenmorelle)	WP (60 g ai/L)	3	0.090	1000	9.0	–	0 7 14 21 28	Whole fruit	0.38 0.09 0.04 0.02 0.01	< 0.01 0.04 < 0.01 < 0.01 < 0.01	0.39 0.13 0.05 0.03 0.02	242171
DEU87F20541, Kloppenheim GER 1988 (Schattenmorelle)	WP (60 g ai/L)	3	0.135	1500	9.0	–	0 7 14 21 27	Whole fruit	0.49 0.23 0.08 0.05 0.02	< 0.01 0.02 < 0.01 < 0.01 < 0.01	0.50 0.25 0.09 0.06 0.03	242171
DEU87F20511, Drage GER 1988 (Schattenmorelle)	WP (60 g ai/L)	3	0.135	1000	9.0	–	0 7 14 21 28	Whole fruit	0.45 0.03 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.46 0.04 0.02 < 0.02 < 0.02	242171
DEU87F20521, Bornheim GER 1988 (Schattenmorelle)	WP (60 g ai/L)	3	0.135	1500	9.0	–	0 7 14 21 28	Whole fruit	0.43 0.23 0.09 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.44 0.24 0.10 0.03 0.02	242171
DEU87F20111, Elbstorf GER 1987 (Schattenmorelle)	WP (60 g ai/L)	3	0.090	1000	9.0	–	0 7 14 21	Whole fruit	0.46 0.02 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.47 0.03 0.02 < 0.02	242171

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
morelle)												
DEU87F20131, Langenau GER 1987 (Schattenmorelle)	WP (60 g ai/L)	3	0.135	1500	9.0	–	0 7 14 21	Whole fruit	0.19 0.02 0.02 0.02	0.02 < 0.01 < 0.01 < 0.01	0.21 0.03 0.03 0.03	242171
DEU87F20021, Bornheim GER 1987 (Schattenmorelle)	WP (60 g ai/L)	3	0.135	1500	9.0	–	0 7 14 21 21	Whole fruit Juice	0.94 0.17 0.06 0.02 0.01	0.02 0.03 0.02 < 0.01 < 0.01	0.96 0.20 0.08 0.03 0.02	242171
DEU87F20041, Rosbach GER 1987 (Schattenmorelle)	WP (60 g ai/L)	3	0.135	1500	9.0	–	0 7 14 21 21	Whole fruit Juice	0.36 0.12 0.05 0.02 0.02	0.01 0.02 0.01 < 0.01 < 0.01	0.37 0.14 0.06 0.03 0.03	242171

Apricot

A total of 21 trials on apricot were conducted in different representative growing areas in France, Italy, Spain and USA during 1986 through 2011 growing seasons. Results of apricot fruits are summarized in Table 55.

Table 55 Supervised trials on apricot in various countries

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
92-0045, Bakersfield, CA, USA, 1992 (Lulubelle)	WP (40 %W/W)	7	0.17	150–151	–	4–21	0	Fruit, pitted	0.12	0.02	0.14	94310
92-0038, Laton, CA USA 1992 (Katy)	WP (40 %W/W)	7	0.17	128	–	3–21	0	Fruit, pitted	0.23	0.06	0.29	94310
92-0039, Orange Cove, CA, USA, 1992 (Castlerbright)	WP (40 %W/W)	7	0.17	101	–	3–23	0	Fruit, pitted	0.11	0.02	0.13	94310
92-0037, Winters, CA USA 1992 (Tilton)	WP (40 %W/W)	7	0.17	90–127	–	6–21	0	Fruit, pitted	0.17	0.08	0.25	94310
92-0046, Zillah, WA USA 1992 (Perfection)	WP (40 %W/W)	7	0.17	100	–	6–22	0	Fruit, pitted	0.62	0.08	0.70	94310
48395 05,	EW	5	0.071	944.2–	7.5	14	0	Pulp	0.12	0.02	0.14	242021

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
Francolinna ITA, 1995 (Palummella)	(60 g ai/L)		–0.12	1558.02			3 7 0 3 7	Whole fruit	0.04 0.03 0.11 0.04 0.03	< 0.01 0.02 0.01 < 0.01 0.01	0.05 0.05 0.12 0.05 0.04	
CEMS-4944C Piedmont, ITA, 2011 (Hargrand)	EW (45 g ai/L)	3	0.089 – 0.091	993.18– 1007.95	9	10–11	< 0 0 3 8 14 < 0 0 3 8 14	Flesh Whole Fruit (calculated)	0.04 0.27 0.13 0.08 0.05 0.04 0.25 0.12 0.07 0.05	0.01 < 0.01 0.01 0.01 0.01 0.01 < 0.01 0.01 0.01 0.01	0.05 0.28 0.14 0.09 0.06 0.05 0.26 0.13 0.08 0.06	2017646
CEMS-4944E Huesca, ESP 2011 (Farcolo)	EW (45 g ai/L)	3	0.088 – 0.091	980.9– 1010.5	9	9–11	< 0 0 3 7 13 < 0 0 3 7 13	Flesh Whole Fruit (calculated)	0.16 0.41 0.29 0.27 0.16 0.15 0.37 0.27 0.25 0.15	0.02 0.02 0.02 0.02 0.03 0.02 0.01 0.02 0.02 0.02	0.18 0.43 0.31 0.29 0.19 0.17 0.38 0.29 0.27 0.17	2017646
I06W030R Ferrara, ITA 2006 (Precoce di Imola)	EW (200 g ai/L)	6	0.128 – 0.132	1777– 1830	7.2	9–11	7 7	Flesh Whole fruit	0.05 0.04	0.02 0.02	0.07 0.06	2009590
S06W042R Murcia, ITA 2006 (Tadeo)	EW (200 g ai/L)	6	0.126 – 0.137	1744– 1891	7.2	9–11	< 0 0 7 14 < 0 0 7 14	Flesh Whole fruit	0.14 0.36 0.21 0.21 0.13 0.34 0.20 0.19	0.04 0.05 0.03 0.07 0.03 0.04 0.03 0.06	0.18 0.41 0.24 0.28 0.16 0.38 0.23 0.25	2009590
F07W075R Rhône-Alpes FRA, 2007 (Orange de Provence)	EW (200 g ai/L)	6	0.135 – 0.139	1784– 1845	7.5	9–12	< 0 0 3 7 14 22	Whole fruit	0.12 0.18 0.14 0.15 0.09 0.06	0.02 0.03 0.02 0.03 0.02 0.02	0.14 0.21 0.16 0.18 0.11 0.08	2000789
CEMS-4639D Valencia ESP, 2010 (Mitger)	EW (45 g ai/L)	3	0.061	994.3– 1004.8	6.0	10	< 0 0 3 7 15 < 0 0 3 7 15	Flesh Whole Fruit (calculated)	0.04 0.33 0.22 0.08 0.02 0.04 0.31 0.21 0.08 0.02	0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01	0.05 0.34 0.23 0.09 0.03 0.05 0.32 0.22 0.09 0.03	2017193
CEMS-4639C Piedmont ITA, 2010 (Hargrand)	EW (45 g ai/L)	3	0.058 – 0.060	971.6– 997.7	6.0	10	< 0 0 3 7 13 < 0	Flesh Whole	0.02 0.07 0.04 0.04 0.02 0.02	0.01 0.01 0.01 0.01 0.01 0.01	0.03 0.08 0.05 0.05 0.03 0.03	2017193

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
							0 3 7 13	Fruit (calculated)	0.07 0.04 0.04 0.02	0.01 0.01 0.01 0.01	0.08 0.05 0.05 0.03	
F60.03.87 Florensac Southern FRA, 1987 (Belliance)	EW (60 g ai/L)	6	0.060	600–800	7.5–10	12–16	8 8	Whole fruit Whole fruit w/o stone	– –	– –	0.04 0.04	242136
F60.01.87 Graveson Southern FRA, 1987 (Colomer)	EW (60 g ai/L)	6	0.060	1000	6.0	–	7 7	Whole fruit Whole fruit w/o stone	– –	– –	0.08 0.09	242137
F60.01.87 Graveson Southern FRA, 1987 (Colomer)	EC (125 g ai/L)	6 6	0.062 0.075	1000 1000	6.2 7.5	–	7	Whole fruit Whole fruit w/o stone	– –	– –	0.06 0.07 0.10 0.11	242138
F60.03.87 Florensac Southern FRA, 1987 (Belliance)	EC (125 g ai/L)	6 6	0.062 0.075	600–800 600–800	6.0–10 9.0–10	12–16	8	Whole fruit Whole fruit w/o stone	– – – –	– – – –	0.04 0.05 0.05 0.06	242139
F33.01.86 Camps Southern FRA, 1986 (Colomer)	EC (125 g ai/L)	1	0.075	1000	7.5	–	0 5 10 14 21 28 0 5 10 14 21 28	Whole fruit Whole fruit w/o stone	– – – – – – – – – – – –	0.40 0.40 0.17 0.10 0.05 0.02 0.55 0.50 0.20 0.11 0.06 0.02	242140	
F60.02.87 Aramon Southern FRA, 1987 (Rouge du Roussillon)	EW (60 g ai/L)	7	0.060	400	15	–	14	Whole fruit Whole fruit w/o stone	– –	– –	0.04 0.04	242174
F60.02.87 Aramon Southern FRA, 1987 (Rouge du Roussillon)	EC (125 g ai/L)	7 7	0.062 0.075	400 400	15 18	13–14 13–14	14 14	Whole fruit Whole fruit w/o stone	– – – –	– – – –	0.04 0.05 0.04 0.05	242175
4148808 Imola, BO ITA, 1988 (S. Castrese)	EC (125 g ai/L)	5 5	0.050 – 0.075 0.10– 0.15	1000–1500 1000–1500	5.0 10	8–71 8–71	18 18	Whole fruit w/o stone	0.02 0.04	< 0.01 < 0.01	0.03 0.05	242991

Plums

A total of 71 trials on plum were conducted in different representative growing areas in Austria, Canada, Czech Republic, France, Germany, Hungary, Italy, Spain and USA during 1987 through 2011 growing seasons. Results of plum fruits are summarized in Table 56.

Table 56 Supervised trials on plum in various countries

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
87-0201 Fresno, CA USA 1987 (Eldorado)	DF (60% W/W)	9	0.21	2933	—	8–16 (app # 3 at 36 day interval)	0	Fruit, pitted	0.25	0.11	0.36	94384
87-0201 Fresno, CA USA 1987 (Eldorado)	DF (60% W/W)	9	0.43	2933	—	8–16 (app # 3 at 36 day interval)	0	Fruit, pitted	0.22	0.10	0.32	94384
87-0161 Woodlake, CA USA 1987 (Red Butte)	DF (60% W/W)	7	0.21	2933	—	7–27	0	Fruit, pitted	0.09	0.02	0.11	94384
87-0260 Zillah, WA USA 1987 (Early Ital.)	DF (60% W/W)	8	0.21	1515	—	13–18	0	Fruit, pitted	0.28	0.12	0.40	94384
87-0260 Zillah, WA USA 1987 (Early Ital.)	DF (60% W/W)	8	0.43	1515	—	13–18	0	Fruit, pitted	0.83	0.32	1.15	94384
87-0366 Fennville, MI USA 1987 (Stanley)	DF (60% W/W)	6	0.21	2806	—	7–42	0	Fruit, pitted	0.59	0.14	0.73	94384
88-0017 Biglerville, PA USA 1987 (Stanley)	WP (40 %W/W)	12	0.224	991	—	7–15	0	Fruit, pitted	1.12	0.33	1.45	94384
88-0299 Nampa, ID USA 1988 (Empress)	DF (60% W/W)	6	0.21	935	—	11–42	14	Fruit, pitted	0.03	< 0.01	0.04	94385
88-0300 Nampa, ID USA 1988 (Empress)	WP (40 %W/W)	6	0.21	935	—	11–42	14	Fruit, pitted	0.06	< 0.01	0.07	94385
88-0301 Nampa, ID USA 1988 (Empress)	EC (25.8% W/W)	6	0.21	935	—	11–42	14	Fruit, pitted	0.04	0.01	0.05	94385
88-0286 Tecumseh, MI USA 1988 (Stanley)	DF (60% W/W)	11	0.21	1889–3648	—	3–14	13	Fruit, pitted	0.30	0.06	0.36	94385
88-0287	WP (40	11	0.21	1889–	—	3–14	13	Fruit,	0.16	0.02	0.18	94385

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
Tecumseh, MI USA 1988 (Stanley)	%W/W)			3648				pitted				
88-0288 Tecumseh, MI USA 1988 (Stanley)	EC (25.8% W/W)	11	0.21	1889–3648	–	3–14	13	Fruit, pitted	0.23	0.04	0.27	94385
90-0124 Orange Cove, CA, USA, 1990 (Freyer)	WP (40 %W/W)	3	0.17	187	–	14–26	14	Fruit, pitted	< 0.01	0.02	0.03	94514
90-0124 Orange Cove, CA, USA, 1990 (Freyer)	WP (40 %W/W)	3	0.17	1992	–	14–26	14	Fruit, pitted	< 0.01	0.03	0.04	94514
89-0233 Stockton, CA USA 1989 (French)	WP (40 %W/W)	6	0.21	393	–	7–17 (app # 4 at 36 day int and #5 at 54 day int)	14 14	Dried, pitted Fruit, pitted	0.45 0.52	0.19 0.03	0.64 0.55	94383
89-0233 Stockton, CA USA 1989 (French)	WP (40 %W/W)	6	0.21	2338	–	7–17 (app # 4 at 36 day int and #5 at 54 day int)	14 14	Dried, pitted Fruit, pitted	0.54 0.29	0.24 0.11	0.76 0.40	94383
89-0235 Linden, CA USA 1989 (French)	WP (40 %W/W)	6	0.21	393	–	7–12 (app # 4 at 46 day int and #5 at 54 day int)	14 14	Dried, pitted Fruit, pitted	0.43 0.13	0.24 < 0.01	0.67 0.14	94383
89-0235 Linden, CA USA 1989 (French)	WP (40 %W/W)	6	0.21	2338	–	7–12 (app # 4 at 46 day int and #5 at 54 day int)	14 14	Dried, pitted Fruit, pitted	1.05 0.15	0.21 0.01	1.26 0.16	94383
89-0234 Stockton, CA USA 1989 (French)	WP (40 %W/W)	6	0.21	393	–	4–17 (app # 4 at 36 day int and #5 at 54 day int)	14 14	Dried, pitted Fruit, pitted	0.76 0.12	0.12 < 0.01	0.88 0.13	94383
89-0234 Stockton, CA USA 1989 (French)	WP (40 %W/W)	6	0.21	2338	–	4–17 (app # 4 at 36 day int and #5 at 54 day int)	14 14	Dried, pitted Fruit, pitted	0.30 0.12	0.09 0.03	0.39 0.15	94383
90-0021 Caldwell, ID USA 1989 (Empress)	WP (40 %W/W)	6	0.21	393	–	7–45	14 14	Dried, pitted Fruit, pitted	0.30 0.06	< 0.01 0.01	0.31 0.07	94383

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
90-0021 Caldwell, ID USA 1989 (Empress)	WP (40 %W/W)	6	0.21	2338	—	7–45	14 14	Dried, pitted Fruit, pitted	0.14 0.04	0.04 < 0.01	0.18 0.05	94383
90-0022 Ontario, OR USA 1989 (Empress)	WP (40 %W/W)	6	0.21	393	—	7–52	14 14	Dried, pitted Fruit, pitted	0.37 0.09	0.31 0.03	0.68 0.12	94383
90-0022 Ontario, OR USA 1989 (Empress)	WP (40 %W/W)	6	0.21	2338	—	7–52	14 14	Dried, pitted Fruit, pitted	0.34 0.08	0.39 0.05	0.73 0.13	94383
87-0201 Fresno, CA USA 1987 (Eldorado)	DF (60% W/W)	9	0.21	2933	—	8–16 (app # 3 at 36 day interval)	0 6 13	Fruit, pitted	0.11 0.07 0.06	0.04 0.03 0.03	0.15 0.10 0.09	94513
RAS/14/4/G Nordrhein– Westfalen GER 1997 (Buhler)	EW (200 g ai/L)	5	0.11– 0.12	480– 516	22.5	10–14	0 3 7 14 0 3 7 14	Flesh Whole fruit	0.27 0.13 0.07 0.07 0.22 0.12 0.07 0.07	0.05 0.02 0.02 0.02 0.04 0.02 0.02 0.02	0.32 0.15 0.09 0.09 0.26 0.14 0.09 0.09	241826
RAS/14/4/G Nordrhein– Westfalen GER 1997 (Buhler)	EW (60 g ai/L)	5	0.11– 0.12	479– 515	22.5	10–14	0 3 7 14 0 3 7 14	Flesh Whole fruit	0.15 0.06 0.08 0.06 0.12 0.05 0.07 0.06	0.03 0.02 0.02 0.02 0.02 0.02 0.02 0.02	0.18 0.08 0.10 0.08 0.14 0.07 0.09 0.08	241826
RAS/14/1/G Rheinland– Pfalz GER 1997 (Ortenauer)	EW (200 g ai/L)	5	0.11	1492– 1525	7.5	10–12	0 3 7 0 3 7	Flesh Whole fruit	0.25 0.21 0.16 0.24 0.20 0.15	0.07 0.07 0.06 0.07 0.07 0.06	0.32 0.28 0.22 0.31 0.27 0.21	241826
RAS/14/1/G Rheinland– Pfalz GER 1997 (Ortenauer)	EW (60 g ai/L)	5	0.11	1475– 1492	7.5	10–12	0 3 7 0 3 7	Flesh Whole fruit	0.22 0.14 0.17 0.20 0.13 0.16	0.04 0.05 0.06 0.04 0.05 0.06	0.26 0.19 0.23 0.24 0.18 0.22	241826
RAS/14/3/G Rheinland– Pfalz, GER 1997 (Ortenauer)	EW (200 g ai/L)	5	0.11	1466– 1510	7.5	10–14	0 3 7 3 3 0 3 7	Flesh Prunes Puree Whole fruit	0.08 0.10 0.07 0.21 0.06 0.08 0.10 0.07	0.03 0.03 0.03 0.08 0.03 0.03 0.03 0.03	0.11 0.13 0.10 0.29 0.09 0.11 0.13 0.10	241826
RAS/14/3/G Rheinland– Pfalz GER 1997 (Ortenauer)	EW (60 g ai/L)	5	0.11– 0.12	1482– 1537	7.5	10–14	0 3 7 0 3	Flesh Whole fruit	0.10 0.08 0.08 0.09 0.08	0.04 0.02 0.04 0.04 0.02	0.14 0.10 0.12 0.13 0.10	241826

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
							7		0.08	0.04	0.12	
RAS/14/2/G Niedersachse n GER 1997 (Hauszwetsch e)	EW (200 g ai/L)	5	0.11–0.12	497–514	22.5	10–12	0 3 7 3 3 0 3 7	Flesh Prunes Puree Whole fruit	0.60 0.67 0.80 2.02 0.71 0.55 0.64 0.74	0.09 0.09 0.03 0.39 0.15 0.08 0.09 0.03	0.69 0.76 0.83 2.41 0.86 0.63 0.73 0.77	241826
RAS/14/2/G Niedersachse n GER 1997 (Hauszwetsch e)	EW (60 g ai/L)	5	0.11–0.12	501–524	22.5	10–12	0 3 7 0 3 7	Flesh Whole fruit	0.56 0.45 0.56 0.53 0.43 0.52	0.07 0.06 0.08 0.07 0.06 0.07	0.63 0.51 0.64 0.60 0.49 0.59	241826
4839504 Leno, BS ITA, 1995 (Satsuma)	EW (60 g ai/L)	5	0.096–0.12	1283.7–1557.77	7.5	14–19	0 3 7 0 3 7	Flesh Whole fruit	0.07 0.05 0.06 0.06 0.04 0.05	0.02 0.03 0.04 0.01 0.02 0.03	0.09 0.08 0.10 0.07 0.06 0.08	242020
4839501 Boara, FE ITA, 1995 (Fryar)	EW (60 g ai/L)	5	0.072–0.077	997.07–1027.77	7.5	14	0 3 7 0 3 7	Flesh Whole fruit	0.07 0.03 0.02 0.07 0.02 0.01	0.04 0.03 0.04 0.03 0.02 0.03	0.11 0.06 0.06 0.10 0.04 0.04	242020
4839503 Molinella, BO ITA, 1995 (Angeleno)	EW (60 g ai/L)	5	0.087–0.090	1165–1206	7.5	11–14	0 3 7 0 3 7	Flesh Whole fruit	0.07 0.05 0.03 0.06 0.04 0.03	0.03 0.03 0.04 0.02 0.02 0.03	0.10 0.08 0.07 0.08 0.06 0.06	242020
4839502 Boara, FE ITA, 1995 (Angeleno)	EW (60 g ai/L)	5	0.086–0.090	1141.66–1196.6	7.5	10–12	0 3 7 0 3 7	Flesh Whole fruit	0.23 0.19 0.15 0.21 0.17 0.13	0.03 0.04 0.05 0.03 0.04 0.04	0.26 0.23 0.20 0.24 0.21 0.17	242020
95FARRSP08 Sorgues Southern FRA, 1995 (Allo)	EC (125 g ai/L)	5	0.074–0.080	992–1061	7.5	10–12	0 3 7 0 3 7	Flesh Whole fruit	0.20 0.15 0.09 0.18 0.13 0.08	0.06 0.07 0.09 0.05 0.07 0.08	0.26 0.22 0.18 0.23 0.20 0.16	243827
95FARRSP07 Beaucaire Southern FRA, 1995 (Quetsche)	EC (125 g ai/L)	5	0.073–0.078	961–1041	7.5–7.61	10–12	0 3 7 0 3 7	Flesh Whole fruit	0.40 0.30 0.25 0.36 0.27 0.22	0.10 0.08 0.16 0.09 0.08 0.14	0.50 0.38 0.41 0.45 0.35 0.36	243827
95FARRSP05 Moissan Southern FRA, 1995 (Prune d'Ente)	EC (125 g ai/L)	5	0.074–0.076	913–1014	7.5–8.12	13–15	7 7 0 3 7 0 3 7	Dried, pitted Dried, whole Flesh Whole fruit	0.16 0.14 0.15 0.15 0.16 0.13 0.14 0.14	0.15 0.13 0.07 0.03 0.10 0.06 0.02 0.09	0.31 0.27 0.22 0.18 0.26 0.19 0.16 0.23	243827

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
95FARRSP06 Amans Southern FRA, 1995 (Prune d'Ente)	EC (125 g ai/L)	5	0.071 – 0.077	797– 929	8.19 – 9.11	12–14	7	Dried, pitted	0.64	0.21	0.85	243827
							7	Dried, whole	0.56	0.19	0.75	
							0	Flesh	0.28	0.07	0.35	
							3		0.24	0.06	0.30	
							7		0.22	0.10	0.32	
							0	Whole fruit	0.25	0.07	0.32	
							3		0.21	0.05	0.26	
97RHC09-D British Col CAN, 1997 (Italian Prune Plum)	WP (40 %W/W)	6	0.12– 0.15	2159– 2458	–	7 (app # 4 at 104 day interval)	1	Fruit, pitted	0.13	–	–	135366
97RHC09-I Pelham Township Ontario CAN, 1997 (Shirow)	WP (40 %W/W)	6	0.14– 0.15	996.5– 1101.5	–	10–15 (app # 4 at 48 day interval)	1	Fruit, pitted	0.20	–	–	135366
97RHC09-J Ridgeville Ontario CAN, 1997 (Italian Plum)	WP (40 %W/W)	6	0.11– 0.14	808– 1034.5	–	7–12 (app # 4 at 75 day interval)	1	Fruit, pitted	0.32	–	–	135366
97RHC09-K Kentville, Nova Scotia CAN, 1997 (Stanley)	WP (40 %W/W)	6	0.14	1000	–	7–20 (app # 2 at 70 day interval)	1	Fruit, pitted	0.97	–	–	135366
AF/4287/HL/3 Indre-Loire Northern FRA, 1998 (Stanley)	EW (200 g ai/L)	5	0.11– 0.13	1400– 1701	7.5	10–12	0	Flesh	0.12	0.01	0.13	241768
							3		0.12	0.01	0.13	
							7		0.12	< 0.01	0.13	
							0	Whole fruit	0.12	0.01	0.13	
							3		0.11	0.01	0.12	
							7		0.11	< 0.01	0.12	
AF/4287/HL/1 Loiret, Northern FRA, 1998 (Quetsche)	EW (200 g ai/L)	5	0.080 –0.11	1064– 1507	7.5	10–14	0	Flesh	0.04	< 0.01	0.05	241768
							3		0.05	< 0.01	0.06	
							7		0.04	< 0.01	0.05	
							0	Whole fruit	0.04	< 0.01	0.05	
							3		0.05	< 0.01	0.06	
							7		0.04	< 0.01	0.05	
AF/4287/HL/2 Meurthe– Moselle Northern FRA, 1998 (Quetsche)	EW (200 g ai/L)	5	0.090 –0.11	1158– 1483	7.5	14	0	Flesh	0.08	0.01	0.09	241768
							3		0.04	0.02	0.06	
							7		0.04	0.02	0.06	
							0	Whole fruit	0.08	0.01	0.09	
							3		0.04	0.02	0.06	
							7		0.04	0.02	0.06	
AF/4287/HL/4 Meurthe– Moselle FRA, 1998 (Quetsche)	EW (200 g ai/L)	5	0.090 –0.11	1179– 1522	7.5	14	0	Flesh	0.10	0.03	0.13	241768
							3		0.09	0.03	0.12	
							7		0.06	0.02	0.08	
							0	Whole fruit	0.10	0.03	0.13	
							3		0.09	0.03	0.12	
							7		0.06	0.02	0.08	
S07W024R Llombai Valencia ESP, 2007 (Larian)	EW (200 g ai/L)	6	0.13	1660– 1761	7.6	8–11	< 0	Flesh	< 0.01	< 0.01	< 0.02	2001319
							0		0.25	0.09	0.34	
							3		0.13	0.09	0.22	
							7		0.08	0.06	0.14	
							14		0.05	0.09	0.14	

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
							21 < 0 0 3 7 14 21	Whole fruit	0.01 < 0.01 0.25 0.13 0.08 0.05 < 0.01	0.04 < 0.01	0.05 < 0.02	
S07W024R Llombai Valencia ESP, 2007 (Larian)	EW (45 g ai/L)	6	0.12–0.13	1669–1746	7.4	8–11	< 0 0 3 7 14 21 < 0 0 3 7 14 21	Flesh Whole fruit	< 0.01 0.15 0.05 0.10 0.04 0.01 < 0.01 0.15 0.05 0.09 0.04 0.01	< 0.01 0.07 0.06 0.09 0.07 0.07	< 0.02 0.22 0.11 0.19 0.11 0.08	2001319
F07W073R Saint Sauveur–Gouvernet Rhone Alpes FRA, 2007 (President)	EW (200 g ai/L)	6	0.13	1702–1759	7.6	9–11	< 0 0 3 7 14 22 < 0 0 3 7 14 22	Flesh Whole fruit	0.17 0.19 0.14 0.15 0.10 0.14 0.16 0.18 0.14 0.14 0.10 0.13	0.02 0.02 0.02 0.02 0.02 0.02	0.19 0.21 0.16 0.17 0.12 0.16	2001319
F07W073R Saint Sauveur–Gouvernet Rhone Alpes FRA, 2007 (President)	EW (45 g ai/L)	6	0.13	1708–1771	7.5	9–11	< 0 0 3 7 14 22 < 0 0 3 7 14 22	Flesh Whole fruit	0.18 0.19 0.24 0.29 0.33 0.13 0.17 0.18 0.23 0.28 0.32 0.13	0.02 0.02 0.02 0.03 0.03 0.02	0.20 0.21 0.26 0.32 0.36 0.15	2001319
CEMS-4640C L'Honor de Cos, Midi-Pyrenees FRA, 2010 (Reine Claude)	EW (45 g ai/L)	3	0.059–0.062	977.8–1035	6	10	7 7	Flesh Whole Fruit (calc.)	0.03 0.03	0.01 0.01	0.04 0.04	2017194
CEMS-4640D Belgida Valencia ESP, 2010 (Black Diamond)	EW (45 g ai/L)	3	0.060–0.062	996.3–1023	6	10	8 8	Flesh Whole Fruit (calc.)	0.02 0.02	0.03 0.03	0.05 0.05	2017194
CEMS-4640B L'Honor de Cos, Midi-Pyrenees FRA, 2010	EW (45 g ai/L)	3	62.1 60.1 60.8	1035.6 1001.1 1013.3	6 6 6	10	< 0 0 3 7 14	Flesh	0.07 0.07 0.07 0.04 0.03	0.02 0.01 0.01 0.01	0.09 0.08 0.08 0.05 0.04	2017194

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
(Bavay)							< 0 0 3 7 14	Whole Fruit (calc.)	0.06 0.06 0.06 0.03 0.03	0.02 0.01 0.01 0.01 0.01	0.08 0.07 0.07 0.04 0.04	
CEMS-4640H Sayat Auvergne FRA, 2010 (Mirabelle)	EW (45 g ai/L)	2	0.087 – 0.089	481.7–491.7	18	10	7 7	Flesh Whole Fruit (calc.)	0.03 0.03	< 0.01 < 0.01	0.04 0.04	2017194
CEMS-4640F Gyorszentiván HUN, 2010 (Stanley)	EW (45 g ai/L)	2	0.090 – 0.091	978.2–991.7	9.2	10	< 0 0 3 7 14 < 0 0 3 7 14	Flesh Whole Fruit (calc.)	0.19 0.44 0.40 0.38 0.36 0.18 0.40 0.37 0.35 0.33	0.01 0.01 0.02 0.02 0.03 0.01 0.01 0.02 0.02 0.02	0.20 0.43 0.42 0.40 0.39 0.19 0.41 0.39 0.37 0.35	2017194
CEMS-4640A Piedmont ITA, 2010 (Angeleno)	EW (45 g ai/L)	3	0.058 – 0.061	958.3–1016.7	6	10	< 0 0 3 6 14 < 0 0 3 6 14	Flesh Whole Fruit (calc.)	0.03 0.06 0.06 0.05 0.02 0.03 0.05 0.06 0.05 0.02	0.02 0.01 0.02 0.01 0.01 0.02 0.01 0.02 0.01 0.01	0.05 0.07 0.08 0.06 0.03 0.05 0.06 0.08 0.06 0.03	2017194
CEMS-4640G Tlumacov Zlinsky, CZE 2010 (Svestka domaci)	EW (45 g ai/L)	2	0.091	1006.9–1009.3	9	11	8 8	Flesh Whole Fruit (calc.)	0.32 0.30	0.03 0.02	0.35 0.32	2017194
CEMS-4640I Dasenhofen Lower AUT AUT, 2010 (Cacaks–Schone)	EW (45 g ai/L)	2	0.092 – 0.094	509.4–524.2	18	11	< 0 0 3 7 14 < 0 0 3 7 14	Flesh Whole Fruit (calc.)	0.03 0.09 0.08 0.09 0.05 0.03 0.09 0.08 0.08 0.05	< 0.01 < 0.01 < 0.01 0.01 0.01 < 0.01 < 0.01 < 0.01 0.01 < 0.01	0.04 0.10 0.09 0.10 0.06 0.04 0.10 0.09 0.09 0.06	2017194
CEMS-4945C L'Honor de Cos, Midi-Pyrenees FRA, 2011 (Reine Claude)	EW (45 g ai/L)	3	0.087 – 0.090	0.097–0.10	9	10–11	7 7	Flesh Whole Fruit (calculated)	0.04 0.04	0.01 0.01	0.05 0.05	2018494
CEMS-4945D Belgida Valencia ESP, 2011 (Black Diamond)	EW (45 g ai/L)	3	0.088 – 0.090	983–994.8	9	9–11	7 7	Flesh Whole Fruit (calculated)	0.05 0.05	0.02 0.02	0.07 0.07	2018494
CEMS-4945B L'Honor de Cos, Midi-	EW (45 g ai/L)	3	0.088 – 0.090	976.7–997.8	9	10	< 0 0 3	Flesh	0.05 0.10 0.08	0.01 0.01 0.01	0.06 0.11 0.09	2018494

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
Pyrenees Southern FRA 2011 (Bavay)							7 14 < 0 0 3 7 14	Whole Fruit (calculated)	0.06 0.03 0.05 0.09 0.07 0.06 0.03	0.02 0.01 0.01 0.01 0.01 0.01 0.01	0.08 0.04 0.06 0.10 0.08 0.07 0.04	
CEMS-4945F Koszeg, Vas HUN, 2011 (Cacanska Lepotica)	EW (45 g ai/L)	2	0.089 – 0.091	991.7–1016.3	9	10	< 0 0 3 7 14 < 0 0 3 7 14	Flesh Whole Fruit (calculated)	0.12 0.20 0.14 0.12 0.16 0.11 0.18 0.13 0.12 0.16	0.02 0.02 0.02 0.02 0.03 0.02 0.02 0.02 0.02 0.03	0.14 0.22 0.16 0.14 0.19 0.13 0.20 0.15 0.14 0.19	2018494
CEMS-4945H Sayat Auvergne FRA, 2011 (Mirabelle)	EW (45 g ai/L)	2	0.088 – 0.089	489.2–492.8	18	11	6 6	Flesh Whole Fruit (calculated)	0.08 0.07	0.01 0.01	0.09 0.08	2018494
CEMS-4945A Manta, Piedmont ITA, 2011 (Angeleno)	EW (45 g ai/L)	3	0.089 – 0.091	983.3–1015	9	8–11	< 0 0 3 8 13 < 0 0 3 8 13	Flesh Whole Fruit (calculated)	0.03 0.08 0.04 0.03 0.03 0.03 0.08 0.04 0.03 0.03	0.02 0.02 0.02 0.02 0.02 0.02 0.02 0.02 0.02 0.02	0.05 0.10 0.06 0.05 0.05 0.05 0.10 0.06 0.05 0.05	2018494
CEMS-4945E Dasenhofen Lower AUT AUT, 2011 (President)	EW (45 g ai/L)	2	0.090	498.1–501	18	10	< 0 0 3 6 14 < 0 0 3 6 14	Flesh Whole Fruit (calculated)	0.10 0.22 0.20 0.21 0.13 0.09 0.21 0.19 0.20 0.12	0.01 0.01 0.01 0.02 0.02 0.01 0.01 0.01 0.02 0.02	0.11 0.23 0.21 0.23 0.15 0.10 0.22 0.20 0.22 0.14	2018494
CEMS-4945G 67128 Jaroslavice CZE 2011 (Elena)	EW (45 g ai/L)	2	0.090	1003.3–1004.7	9	10	7 7	Flesh Whole Fruit (calculated)	0.22 0.21	0.01 0.01	0.23 0.22	2018494
4148811 Ponte del Castello, ITA 1988 (Stanley)	EC (120 g ai/L)	4	0.05–0.15	1000–1500	5 10	–	14 14	fruit	0.01 0.07	0.03 0.03	0.04 0.10	262066

*Berries and other small fruits**Currants*

A total of 26 trials on currants were conducted in different representative growing areas in France, Italy, UK and USA during 1990 through 2008 growing seasons. Results of currants are summarized in Table 57.

Table 57 Supervised trials on currants in various countries

Trial ID/ Location Country Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicati on Interval (days)	DAT	Portion Analysed	Myclobut anil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
WA09 Benton county, WA USA	WP (40 %W/ W)	8	0.14	1029– 1515	–	10–17	0 3 7	Berries Berries Berries	1.48 0.83 1.17	0.27 0.26 0.30	1.75 1.09 1.47	24175 7
1995 (Wilder)	WP (40 %W/ W)	8	0.28	1029– 1515	–	10–17	0 3 7	Berries Berries Berries	4.05 3.24 2.50	0.42 0.51 0.43	4.47 3.75 2.93	
WA10, Benton county, WA USA	WP (40 %W/ W)	8	0.14	1029– 1515	–	10–17	0 3 7	Berries Berries Berries	1.92 1.06 1.10	0.26 0.28 0.25	2.18 1.34 1.35	24175 7
1995 (Wilder)	WP (40 %W/ W)	8	0.28	1029– 1515	–	10–17	0 3 7	Berries Berries Berries	3.99 2.76 3.22	0.38 0.38 0.43	4.27 3.14 3.65	
WA37, Prosser WA, USA, 2000 (Wilder)	WP (40 %W/ W)	8	0.14– 0.15	909–1018	–	13–15	0	Berries	0.76	0.18	0.94	13532 2
30-91B Newent, GBR 1991 (Ben Lomond)	EW (60 g a i/L)	5 5	0.18 0.090	2000 2000	9.0 4.5	12–18 12–18	21 0 8 17 21	Whole fruit Whole fruit	0.22 0.57 0.31 0.08 0.20	0.20 0.16 0.64 0.14 0.09	0.42 0.73 0.95 0.22 0.29	24190 4 24190 4
30-91C Ross-on- Wye Herefords hire GBR, 1991 (Ben Lomond)	EW (60 g a i/L)	5	0.090	2000	4.5	13–20	0 7 13 20	Whole fruit	0.52 0.11 0.04 0.16	< 0.01 0.03 0.04 0.05	0.53 0.14 0.08 0.21	24190 4
30-91A Ledbury Herefords hire GBR, 1991 (Ben Lomond)	EW (60 g a i/L)	5 5	0.18 0.090	2000 2000	9.0 4.5	9–18 9–18	20 0 7 13 20	Whole fruit Whole fruit	0.10 0.72 0.26 0.07 0.13	0.10 0.67 0.66 0.19 0.29	0.20 1.39 0.93 0.26 0.42	24190 4 24190 4
AK/2875/ RH/1 Stonall Staffs England,	EW (60 g a i/L)	6	0.090	500	18	–	14	Whole fruit	0.35	0.04	0.39	24176 7

Trial ID/ Location Country Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicati on Interval (days)	DAT	Portion Analysed	Myclobut anil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
1995 (Baldwin)												
AK/2875/ RH/2 Worcester shire England, 1995 (Baldwin)	EW (60 g a i/L)	6	0.090	500	18	—	14	Whole fruit	0.30	0.05	0.35	24176 7
AK/2875/ RH/3 Kent, England 1995 (Ben Alder)	EW (60 g a i/L)	6	0.090	500	18	—	14	Whole fruit	0.19	0.02	0.21	24176 7
AK/2875/ RH/4 Kent, England 1995 (Ben Lomand)	EW (60 g a i/L)	6	0.090	500	18	—	14	Whole fruit	0.24	0.04	0.28	24176 7
AK/2875/ RH/1 Stonall Staffs England, 1995 (Baldwin)	WP (6% W/W)	6	0.090	500	18	—	14	Whole fruit	0.42	0.04	0.46	24176 7
AK/2875/ RH/2 Worcester shire England, 1995 (Baldwin)	WP (6% W/W)	6	0.090	500	18	—	14	Whole fruit	0.29	0.05	0.34	24176 7
AK/2875/ RH/3 Kent, England 1995 (Ben Alder)	WP (6% W/W)	6	0.090	500	18	—	14	Whole fruit	0.31	0.03	0.34	24176 7
AK/2875/ RH/4 Kent, England 1995 (Ben Lomand)	WP (6% W/W)	6	0.090	500	18	—	14	Whole fruit	0.26	0.05	0.31	24176 7
AK/2875/ RH/1 Stonall Staffs England, 1995 (Baldwin)	EW (200 g ai/L)	6	0.090	500	18	—	14	Whole fruit	0.43	0.04	0.47	24176 7
AK/2875/ RH/2 Worcester	EW (200 g ai/L)	6	0.090	500	18	—	14	Whole fruit	0.30	0.07	0.37	24176 7

Trial ID/ Location Country Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicati on Interval (days)	DAT	Portion Analysed	Myclobut anil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
shire England, 1995 (Baldwin)												
AK/2875/ RH/3 Kent, England 1995 (Ben Alder)	EW (200 g ai/L)	6	0.090	500	18	—	14	Whole fruit	0.24	0.04	0.28	24176 7
AK/2875/ RH/4 Kent, England 1995 (Ben Lomand)	EW (200 g ai/L)	6	0.090	500	18	—	14	Whole fruit	0.26	0.05	0.31	24176 7
450/RH/9 0/BLA/A, Ledbury Herefords hire UK, 1990 (Ben Lomond)	EW (60 g a i/L)	6	0.12	2000	6	—	16 16 16	Whole fruit Juiced fruit Canned fruit	0.46 0.04 0.09	0.06 0.13 0.20	0.52 0.17 0.29	24195 9
450/RH/9 0/BLA/B, Newent Herefords hire UK, 1990 (Baldwin)	EW (60 g a i/L)	6	0.12	2000	6	—	16 16	Whole fruit Juiced fruit	0.21 0.07	0.11 0.14	0.32 0.21	24195 9
450/RH/9 0/BLA/C, Ledbury Herefords hire UK, 1990 (Baldwin)	EW (60 g a i/L)	6	0.12	2000	6	—	12 12	Whole fruit Juiced fruit	0.57 < 0.01	0.22 0.15	0.79 0.16	24195 9
SRPV Villevequ e FRA, 1992 (Tenach)	EC (125 g ai/L)	3 3 3	0.062 5	400	15.6	14 14 13–14	35 21 7	Green fruit Green fruit Maturity	0.04 0.07 0.08	— — —	— — —	24197 6
AGRI 022/08 Vigolo Vattaro, TN, ITA, 2008 (Red pool)	EW (45 g a i/L)	4	0.057 — 0.058	994–1013	—	10–11	< 0 0 3 7 14 21 28	Berries Berries Berries Berries Berries Berries Berries	0.06 0.21 0.09 0.07 0.07 0.06 0.03	— — — — — — —	— — — — — — —	20011 60
AGRI 023/08 Vigolo Vattaro, TN, ITA, 2008 (Red)	EW (45 g a i/L)	4	0.056 — 0.060	976–1053	—	10–11	14	Berries	0.09	—	—	20011 62

Trial ID/ Location Country Year (variety pool)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	DAT	Portion Analysed	Myclobut anil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.

Grapes

A total of 70 trials on grapes were conducted in different representative growing areas in Brazil, France, Germany, Greece, Italy, Portugal, Spain and USA during 1987 through 2004 growing seasons. Results of grape fruits are summarized in Table 58.

Table 58 Supervised trials on grapes in various countries

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	DAT	Portion Analysed	Myclob utanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
87-0318 McFarland, CA, USA, 1987 (Ruby), Table grapes	DF (60% W/W)	6	0.112	935.3	12	15-43	11 16	Grapes	0.03 0.05	< 0.01 0.02	0.04 0.07	94280
87-0319 Earlimart, CA USA 1987 (Ruby) Table grapes	DF (60% W/W)	6	0.112	935.3	12	10-43	7 16	Grapes	0.14 0.30	0.01 0.02	0.15 0.32	94280
87-0320 Madera, CA USA 1987 (Ruby) Table grapes	DF (60% W/W)	6	0.112	935.3	12	13-37	7 15	Grapes	0.20 0.12	0.01 0.03	0.21 0.15	94280
86-0231, Greenfield, CA USA 1986 (Muscat) Wine grapes	WP (40 %W/ W)	4	0.084	935.3	—	15-34	14	Grapes	0.09	0.04	0.13	94276
86-0233 Soledad, CA USA 1986 (Riesling) Wine grapes	WP (40 %W/ W)	4	0.084	935.3	—	15-34	14	Grapes	0.16	0.04	0.20	94276
85-0323 Reedley, CA USA 1985 (Thompson)	WP (40% W/W)	5	0.112	935.3	—	—	7 14 21 14 14	Grapes Dry pomace Wet pomace	0.16 0.14 0.09 0.74 0.07	0.03 0.05 0.06 — —	0.19 0.19 0.15 — —	94275
85-0637 Naples, NY USA 1985 (Rosette)	WP (40% W/W)	5	0.168	1871	—	—	7 14 21	Grapes	0.22 0.32 0.20	0.05 0.08 0.06	0.27 0.40 0.26	94275
85-360 Newtown, PA USA 1985 (Concord)	WP (40% W/W)	8	0.112	935.3	—	—	7 14 20	Grapes	1.30 1.08 0.33	0.18 0.17 0.07	1.48 1.25 0.40	94275
86-175, Exeter CA, USA, 1986 (Flame)	WP (40% W/W)	4	0.112	—	—	—	14	Grapes	0.10	0.03	0.13	94277
86-195, Fresno, CA	WP (40%)	5	0.082	—	—	—	7 14	Grapes	0.35 0.24	0.01 0.01	0.36 0.25	94277

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
USA 1986, (Thompson)	W/W)											
85-0360, PA USA 1985 (-)	WP (40% W/W)	8	0.112	—	—	—	7 14 20	Grapes	1.30 1.08 0.33	0.18 0.17 0.07	1.48 1.25 0.40	94443
85-0372 Soledad, CA USA 1985 (Savignon)	—	5	0.112	935.3	—	—	14	Grapes	0.31	0.03	0.34	94443
85-0373 Calistoga, CA USA 1985 (Riesling)	—	5	0.112	935.3	—	—	14	Grapes	0.48	0.10	0.58	94443
85-0374 Greenfield, CA USA 1985 (Muscat Can)	WP (40% W/W)	5	0.112	935.3	—	—	13	Grapes	0.25	0.04	0.29	94443
85-0383, CA USA 1985 (-)	WP (40% W/W)	5	0.112	—	—	—	7 14 21	Grapes	0.16 0.14 0.09	0.03 0.05 0.06	0.19 0.19 0.15	94443
85-0419 Napa, CA, USA, 1985 (Pinot Noir)	—	5	0.112	935.3	—	—	14 14 14 14 14	Grapes Dry pomace Wet pomace Juice Wine Stem	0.53 1.16 0.67 0.06 0.07 1.25	0.07 0.10 0.14 0.01 0.06 0.27	0.60 1.26 0.81 0.07 0.13 1.52	94443
85-0637 Naples, NY USA 1985 (Rosette)	WP (40% W/W)	5	0.17	1871	—	—	7 14 21	Grapes	0.22 0.32 0.20	0.05 0.08 0.06	0.27 0.40 0.26	94443
85-0323 Reedley, CA USA 1985 (Thompson)	WP (40% W/W)	5	0.112	935.3	—	—	14 14 14 14 14 14 14 14 14 14 14	Grapes Juice Dry pomace Wet pomace Wine Filtered wine A+B raisin C raisin Midget raisin Waste raisin Wine lees	0.13 0.04 0.74 0.07 0.04 0.04 0.78 0.82 0.69 3.43 0.22	0.04 0.02 0.26 0.02 0.02 < 0.01 0.16 0.25 0.24 0.80 0.02	0.17 0.06 1.00 0.09 0.06 0.05 0.94 1.07 0.93 4.23 0.24	94607
86-0262 Newtown, PA USA 1986 (Niagra)	DF (60% W/W)	1	0.14	93.53	150	—	0 7 14 21	Grapes Grapes Grapes Grapes	1.12 1.07 0.39 0.49	< 0.01 0.01 0.01 < 0.01	1.13 1.08 0.40 0.50	99505
86-0263 Newtown, PA USA 1986 (Concord)	DF (60% W/W)	1	0.14	93.53	150	—	0 7 14 21	Grapes	0.60 0.46 0.38 0.24	< 0.01 < 0.01 0.01 < 0.01	0.61 0.47 0.39 0.25	99505
86-0264 Newtown, PA	DF (60%	1	0.14	93.53	150	—	0 7	Grapes	1.38 0.69	< 0.01 0.01	1.39 0.70	99505

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	DAT	Portion Analysed	Myclob utanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
USA 1986 (Catawba)	W/W)						14 21		0.64 0.43	0.03 0.04	0.67 0.47	
R&H/202/4/G D-74360 Schozach GER, 1996 (Spatburgunder), Wine grapes	EC (240 g ai/L)	8	0.029 – 0.048	319–533	9	10–15	0 14 28	Whole fruit	0.41 0.21 0.20	0.01 0.01 0.02	0.42 0.22 0.22	94404
R&H/202/3/G D-67150 NiederkirchenG ER, 1996 (Portugieser) Wine grapes	EC (240 g ai/L)	8	0.012 – 0.047	388–1556	3	10–16	0 14 28 14 14 14	Whole fruit Young Wine Aged wine Juice	0.41 0.25 0.24 0.03 0.03 0.06	< 0.01 < 0.01 0.01 < 0.01 < 0.01 < 0.01	0.42 0.26 0.25 0.04 0.04 0.07	94404 243161
R&H/202/2/G D-67150 NiederkirchenG ER, 1996 (Muller–Thurgau) Wine grapes	EC (240 g ai/L)	8	0.030 – 0.047	334–525	9	10–16	0 14 28 14 14 14	Whole fruit Young Wine Aged wine Juice	0.48 0.27 0.29 0.05 0.04 0.07	0.01 < 0.01 0.01 < 0.01 < 0.01 < 0.01	0.49 0.28 0.30 0.06 0.05 0.08	94404 242161
R&H/202/1/G D-54518 GER, 1996 (Riesling) Wine grapes	EC (240 g ai/L)	8	0.012 – 0.061	403–2030	3	10–14	0 14 28	Whole fruit	0.70 0.47 0.35	0.02 0.02 0.02	0.72 0.49 0.37	94404
R&H/211/2/F 82290 La Ville Dieu du Temple FRA, 1996 (Syrah) Wine grapes	EC (240 g ai/L)	6	0.028 – 0.030	733–800	3.75	10–13	0 14	Whole fruit	0.04 0.03	< 0.01 < 0.01	0.05 0.04	138357
R&H/211/1/I Cazzago, lombardia, ITA, 1996 (Chardonnay) Wine grapes	EC (240 g ai/L)	6	0.036 – 0.061	980.8–1564	3.68–3.91	7–14	0 14	Whole fruit	0.16 0.06	< 0.01 < 0.01	0.17 0.07	138357
R&H/211/2/I Calvagese Lombardia ITA, 1996 (Reisling renano) Wine grapes	EC (240 g ai/L)	6	0.035 – 0.057	960–1515.9	3.6–3.79	10–14	0 14	Whole fruit	0.16 0.05	< 0.01 < 0.01	0.17 0.06	138357
R&H/211/1/F 17520 Archiac FRA, 1996 (Ugni-Blanc) Wine grape	EC (240 g ai/L)	6	0.030 – 0.036	793–947	3.75	10–11	0 14	Whole fruit	0.06 0.02	< 0.01 < 0.01	0.07 0.03	138357
R&H/212/2/F 82290 La Ville Dieu du Temple FRA, 1996 (Syrah) Wine grapes	EC (120 g ai/L)	6	0.026 – 0.030	680–810	3.75	10–13	0 14	Whole fruit	0.05 0.03	< 0.01 < 0.01	0.06 0.04	138356
R&H/212/1/F 17520 Archiac FRA, 1996 (Ugni-Blanc)	EC (120 g ai/L)	6	0.029 – 0.038	773–1000	3.75	10–11	0 14	Whole fruit	0.06 0.04	< 0.01 < 0.01	0.07 0.05	138356

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	DAT	Portion Analysed	Myclob utanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
Wine grape												
R&H/212/1/I Cazzago, lombardia, ITA, 1996 (Chardonnay) Wine grapes	EC (120 g ai/L)	6	0.037 – 0.059	993.7–1532.1	3.68–3.83	7–14	0 14	Whole fruit	0.15 0.06	< 0.01 < 0.01	0.16 0.07	138356
R&H/212/2/I Calvagese Lombardia ITA, 1996 (Reisling renano) Wine grapes	EC (120 g ai/L)	6	0.036 – 0.058	981.4–1521.3	3.68–3.80	10–14	0 14	Whole fruit	0.11 0.05	< 0.01 < 0.01	0.12 0.06	138356
R&H/213/2/F 2290 La Ville Dieu du Temple FRA, 1996 (Syrah) Wine grapes	EW (200 g ai/L)	6	0.024 – 0.030	647–787	3.75	10–13	0 14	Whole fruit	0.06 0.03	< 0.01 < 0.01	0.07 0.04	241764
R&H/213/2/I Calvagese Lombardia ITA, 1996 (Reisling renano) Wine grapes	EW (200 g ai/L)	6	0.037 – 0.057	992–1513.3	3.65–3.78	10–14	0 14	Whole fruit	0.18 0.03	< 0.01 < 0.01	0.19 0.04	241764
R&H/213/1/I Cazzago, lombardia, ITA, 1996 (Chardonnay) Wine grapes	EW (200 g ai/L)	6	0.036 – 0.058	977.6–1519.2	3.66–3.80	7–14	0 14	Whole fruit	0.14 0.07	< 0.01 < 0.01	0.15 0.08	241764
R&H/213/1/F 17520 Archiac FRA, 1996 (Ugni-Blanc) Wine grape	EW (200 g ai/L)	6	0.031 – 0.035	827–927	3.75	10–11	0 14	Whole fruit	0.04 0.03	< 0.01 < 0.01	0.05 0.04	241764
RAS/23/2/I Calvagese Lombardia ITA, 1997 (Groppello) Wine grapes	EC (240 g ai/L)	6	0.038 – 0.058	1000–1525	3.76–3.79	13–14	0 14	Whole fruit	0.18 0.10	0.01 0.01	0.19 0.11	241823
RAS/23/3/I Prato ottesole Emilia R, ITA 1997 (Barbera) Wine grapes	EC (240 g ai/L)	6	0.037 – 0.055	1000–1477	3.71	10–17	0 14	Whole fruit	0.26 0.13	0.01 0.01	0.27 0.14	241823
RAS/23/1/I Cazzago lombardia ITA, 1997 (Chardonnay) Wine grapes	EC (240 g ai/L)	6	0.038 – 0.056	1000–1500	3.72–3.76	10–11	0 14	Whole fruit	0.08 0.04	< 0.01 < 0.01	0.09 0.05	241823
RAS/23/1/G Korinthia North Peloponese GRC, 1997 (Savatiano) Wine grapes	EC (240 g ai/L)	6	0.056	1495–1512	3.7	10–11	0 14	Whole fruit	0.37 0.09	0.02 0.01	0.39 0.10	241823
R&H/203/4/G	EW	8	0.031	343–	9	10–15	0	Grapes	0.39	0.02	0.41	94403

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	DAT	Portion Analysed	Myclob utanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
D-74360 Schozach GER, 1996 (Spatburgunder), Wine grape	(200 g ai/L)		– 0.049	543			14 28		0.28 0.22	0.03 0.03	0.31 0.25	241765
R&H/203/3/G D-67150 NiederkirchenG ER 1996 (Portugisesr) Wine grape	EW (200 g ai/L)	8	0.012 – 0.048	406– 1600	3	10–16	0 14 28 14 14 14	Grapes Juice (must) Mature wine Young Wine	0.46 0.34 0.33 0.07 0.04 0.04	< 0.01 0.01 0.02 < 0.01 < 0.01 < 0.01	0.47 0.35 0.35 0.08 0.05 0.05	94403 241765
R&H/203/2/G D-67150 NiederkirchenG ER 1996 (Muller–Thurgau) Wine grape	EW (200 g ai/L)	8	0.028 – 0.048	312– 531	9	10–16	0 14 28 14 14 14	Grapes Juice (must) Mature wine Young Wine	0.45 0.41 0.35 0.09 0.07 0.06	< 0.01 0.02 0.01 < 0.01 < 0.01 < 0.01	0.46 0.43 0.36 0.10 0.08 0.06	94403 241765
R&H/203/1/G D-54518 Kesten GER, 1996 (Riesling) Wine grape	EW (200 g ai/L)	8	0.012 – 0.060	410– 2008	3	10–14	0 14 28	Grapes	0.43 0.29 0.28	0.02 0.02 0.02	0.45 0.31 0.30	94403 241765
RAS/18/2/F Roschwihr Elsaß, FRA 1997 (Spatburgunder) Wine grapes	EW (200 g ai/L)	8	0.011 – 0.049	375– 1625	3	13–15	0 7 14 21 28	Whole fruit	0.36 0.25 0.16 0.14 0.16	0.02 0.02 0.02 0.02 0.02	0.38 0.27 0.18 0.16 0.18	110206
RAS/18/4/F Wissembourg Elsaß, FRA 1997 (Spatburgunder) Wine grapes	EW (200 g ai/L)	8	0.012 – 0.048	394– 1609	3	10–15	0 7 14 21 28 14 14 14	Whole fruit Juice Mature wine Young wine	0.71 0.51 0.51 0.47 0.38 0.08 0.05 0.04	0.03 0.03 0.03 0.03 0.03 0.01 0.02 0.01	0.74 0.54 0.54 0.50 0.41 0.09 0.07 0.05	110206
RAS/18/1/F Roschwihr Elsaß, FRA 1997 (Silvaner) Wine grapes	EW (200 g ai/L)	8	0.009 – 0.030	288– 1000	3	13–15	0 7 14 21 28	Whole fruit	0.25 0.14 0.07 0.07 0.09	0.01 0.01 0.01 < 0.01 < 0.01	0.26 0.15 0.08 0.08 0.10	110206
RAS/18/3/F Wissembourg Elsaß, FRA 1997 (Muller–Thurgau) Wine grapes	EW (200 g ai/L)	8	0.009 – 0.030	306– 1006	3	10–15	0 7 14 21 28 14 14 14	Whole fruit Juice Mature wine Young wine	0.39 0.31 0.25 0.26 0.25 0.04 0.01 0.02	< 0.01 0.01 < 0.01 0.01 0.01 < 0.01 < 0.01 < 0.01	0.40 0.32 0.26 0.27 0.26 0.05 0.02 0.03	110206
AF/7409/DE/2 Gocklingen, 76831	EW (200 g ai/L)	8	0.036 – 0.084	600– 1400	6	7–18	14 28 35	Whole fruit	0.07 0.08 0.05	0.04 0.05 0.04	0.11 0.13 0.09	204469

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	DAT	Portion Analysed	Myclob utanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
GER, 2003 (Kerner) Wine grape	SC (45 g a i/L)	5	0.036 – 0.084	600–1400	6	7–42	14 28 35	Whole fruit	0.15 0.06 0.08	0.03 0.03 0.04	0.18 0.09 0.12	
AF/7409/DE/1 Gennes, 49320, FRA 2003 (Cabernet franc), Wine grape	EW (200 g ai/L)	8	0.055 – 0.061	577–985	6.1–9.5	7–35	< 0 0 7 14 28 35	Whole fruit	0.13 0.25 0.26 0.07 0.11 0.06	0.05 0.06 0.06 0.02 0.03 0.02	0.18 0.31 0.32 0.09 0.14 0.08	204469
	SC (45 g a i/L)	5	0.058 – 0.061	617–969	6.1–9.6	2–57	< 0 0 7 14 28 35	Whole fruit	0.11 0.14 0.15 0.04 0.06 0.05	0.02 0.02 0.03 0.01 0.02 0.03	0.13 0.16 0.18 0.05 0.08 0.08	
AF/7410/DE/1 Pizay, 69220, Rhone, FRA 2003 (Chardonnay) Wine grape	EW (200 g ai/L)	8	0.064 – 0.065	478–679	9.0–13	7–22	< 0 0 7 14 28 35	Whole fruit	0.04 0.04 0.03 0.02 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 0.01 < 0.01	0.05 0.05 0.04 0.03 0.03 0.03	204468
	SC (45 g a i/L)	8	0.055 – 0.061	577–985	6.1–9.5	6–36	< 0 0 7 14 28 35	Whole fruit	0.03 0.03 0.03 0.03 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 0.01 0.02	0.04 0.04 0.04 0.04 0.03 0.04	
AF/7410/DE/2 Poggio Grande, 40024 Emilia Romagna, ITA 2003 (Barbera) Wine grape	EW (200 g ai/L)	8	0.034 – 0.058	562–967	6	6–19	14 28 35	Whole fruit	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	204468
	SC (45 g a i/L)	5	0.034 – 0.059	573–990	6	8–36	14 28 35	Whole fruit	0.01 < 0.01 0.01	0.02 < 0.01 0.02	0.03 < 0.02 0.03	
AF/8165/DE/1 71700 Uchizy FRA, 2004 (Chardonnay) Wine grape	EW (200 g ai/L)	4	0.048 – 0.052	997–1083	4.8	10–11	< 0 0 7 14 28 35	Whole fruit	0.06 0.07 0.06 0.04 0.05 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.07 0.08 0.07 0.05 0.06 0.03	241777
	EW (200 g ai/L)	8	0.046 – 0.052	954–1054	4.8	8–11	< 0 0 7 14 28 35	Whole fruit	0.06 0.09 0.08 0.07 0.04 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.07 0.10 0.09 0.08 0.05 0.05	
AF/8165/DE/2 71260, St Pierre de Lanques FRA, 2004 (Chardonnay) Wine grape	EW (200 g ai/L)	4	0.046 – 0.049	958–1023	4.8	10–11	< 0 0 7 14 28 35	Whole fruit	0.12 0.22 0.08 0.07 0.05 0.07	0.02 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.14 0.23 0.09 0.08 0.06 0.08	241777
	EW (200 g ai/L)	8	0.044 – 0.052	925–1088	4.8	8–11	< 0 0 7 14 28 35	Whole fruit	0.18 0.41 0.07 0.08 0.10 0.11	0.03 0.03 < 0.01 0.01 0.02 0.01	0.21 0.44 0.08 0.09 0.12 0.12	
AF/8165/DE/3 49560 Nueil sur Layon FRA, 2004 (Gamay)	EW (200 g ai/L)	4	0.046 – 0.051	961–1061	4.8	7–10	14	Whole fruit	0.06	< 0.01	0.07	241777
	EW (200 g ai/L)	8	0.046 –	979–1061	4.8	7–12	14	Whole fruit	0.14	0.02	0.16	

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	DAT	Portion Analysed	Myclob utanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
Wine grape	ai/L)		0.051									
AF/8165/DE/4 49560 Nueil sur Layon	EW (200 g ai/L)	4	0.046 – 0.052	961–1082	4.8	8–12	14	Whole fruit	0.10	< 0.01	0.11	241777
FRA, 2004 (Cabernet Sauvignon) Wine grape	EW (200 g ai/L)	8	0.048 – 0.051	996–1056	4.8	8–12	14	Whole fruit	0.08	0.01	0.09	
GHB-P 794-03 Jundiai, SP BRA, 2001	WP (400 g ai/L)	3	0.16	1000	16	10	7	Grapes	0.31	–	–	102070
(Niagara) Table grape	WP (400 g ai/L)	3	0.080	1000	8	10	7	Grapes	0.12	–	–	
GHB-P 794-02 Marialva, PR BRA, 2002	WP (400 g ai/L)	3	0.16	1000	16	10–44	7	Grapes	0.01	–	–	102070
(Italia) Table grape	WP (400 g ai/L)	3	0.080	1000	8	10–11	7	Grapes	< 0.01	–	–	
GHB-P 794-01 Monte Mor, SP, BRA 2002 (Niagara) Table grape	WP (400 g ai/L)	3	0.16	1000	16	9	0 3 7 10 14	Grapes Grapes Grapes Grapes Grapes	0.29 0.12 0.11 0.09 0.06	– – – – –	– – – – –	102070
	WP (400 g ai/L)	3	0.080	1000	8	9	0 3 7 10 14	Grapes Grapes Grapes Grapes Grapes	0.11 0.04 0.04 0.03 0.02	– – – – –	– – – – –	
005934 Versuchsfeld Deutscher wetterdienst West GER 1984 (Kerner) Wine grapes	EC (125 g ai/L)	8	0.016	920–1440	1.6	13–15	0 14 21 28 35 42 35 35	Grapes Grapes Grapes Grapes Grapes Grapes Must Wine	0.03 0.03 0.02 0.02 0.01 0.01 0.01 0.01	– – – – –	– – – – –	94270
005928 (Trollinger) Wine grapes		7	0.032	800–1000	3.2	11–17	49	Grapes	0.01	–	–	
005931 (Muller Thurgau) Wine Grapes		8	0.048	500–667	4.8	8–15	58	Grapes	0.04	–	–	
4318546, La Tamarissiere Southern FRA, 1985 (Carignan)	EC (125 g ai/L)	8	0.030	60–100	3.0–5.0	–	14 26	Grapes	–	–	0.06 0.04	241839
4518502, Allas Bocage Southern FRA, 1985 (Ugni blanc)	EC (125 g ai/L)	12 9	0.020 0.030	250 250	8.0 12	– –	19 31 41 18 30 40	Grapes	– – – – –	– – – – –	0.05 0.05 0.04 0.05 0.05 0.04	241890
4518501, St Caprais Southern FRA, 1985 (Ugni blanc)	EC (125 g ai/L)	11 8	0.020 0.030	250 250	8.0 12	– –	19 31 41 18 30 40	Grapes	– – – – –	– – – – –	0.06 0.04 0.06 0.05 0.05 0.05	242161

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	DAT	Portion Analysed	Myclob utanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
4229203, 2580 Alenquer PRT, 1992 (Cardinal)	EC (125 g ai/L)	7	0.020 – 0.030	160– 240	12.5	13–15	21	Grapes	–	–	0.05	241981
4229207, 2580 Alenquer PRT, 1992 (Cardinal)	EC (125 g ai/L)	6	0.020 – 0.030	160– 240	12.5	12–16	28	Grapes	–	–	0.04	241983
431.86.66 Coursan FRA, 1986 (Muscat de Hambourg)	EC (75 g a i/L)	11	0.019	700	2.7	10–21	30	Grapes	–	–	0.01	242098
421.86.15B Moulon FRA, 1986 (Ugni blanc)	EC (75 g a i/L)	11	0.019	500	4.0	8–13	30	Grapes	–	–	0.05	242106
4918898 Matola Alicante, ESP 1988 (-)	EC (75 g a i/L)	3	0.023	1000	2.3	14	0 7	Grapes	– –	– –	0.10 0.02	242162
4918897 Matola Alicante, ESP 1988 (-)	EC (75 g a i/L)	3	0.023	1000	2.3	14	0 7	Grapes	– –	– –	0.10 0.03	242166
		3	0.023	1000	2.3	14	0 7 14 24	Grapes	– – – –	– – – –	0.24 0.25 0.08 0.08	
	EC (325 g ai/L)	3	0.098	1000	9.7	14	0 7	Grapes	– –	– –	0.25 0.05	
		3	0.098	1000	9.7	14	0 7 14 24	Grapes	– – – –	– – – –	< 0.10 < 0.10 < 0.10 < 0.10	

Strawberry

A total of 47 trials on strawberry were conducted in different representative growing areas in France, Italy, Spain, UK and USA during 1988 through 2007 growing seasons. Results of strawberry fruits are summarized in Table 59.

Table 59 Supervised trials on strawberry in various countries

Trial ID/Location Country/Year (variety)	Form	No. of App s	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	DA T	Portion Analyse d	Myclobutanil (mg/kg)	RH- 9090 (mg.kg)	Total of myclobutanti l (mg/kg)	Ref.
FL40 USA 1990 (-)	WP (40 %W/W)	6	0.035			13–15	0 3 7	Whole fruit	0.05 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02	0.07 < 0.04 < 0.04	94590 110951
	WP (40 %W/W)	6	0.070			13–15	0 3 7	Whole fruit	0.10 0.04 < 0.02	< 0.02 < 0.02 < 0.02	0.12 0.06 < 0.04	
	WP (40 %W/W)	6	0.14			13–15	0 3 7	Whole fruit	0.20 0.08 0.02	< 0.02 < 0.02 < 0.02	0.22 0.10 0.04	
CA39 USA 1991 (-)	WP (40 %W/W)	6	0.035			14–17	0 3 7	Whole fruit	0.04 0.02 0.02	< 0.02 < 0.02 < 0.02	0.06 0.04 0.04	94590 110951

Trial ID/Location Country/Year (variety)	Form	No. of App s	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analyse d	Myclobutanil (mg/kg)	RH- 9090 (mg.kg)	Total of myclobutanil (mg/kg)	Ref.
	WP (40 %W/W)	6	0.070			14-17	0 3 7	Whole fruit	0.08 0.06 0.04	< 0.02 < 0.02 < 0.02	0.10 0.08 0.06	
	WP (40 %W/W)	6	0.14			14-17	0 3 7	Whole fruit	0.15 0.13 0.10	< 0.02 < 0.02 < 0.02	0.17 0.15 0.12	
CA30 USA 1991 (-)	WP (40 %W/W)	6	0.035			20-21	0 4 7	Whole fruit	0.07 0.02 0.04	< 0.02 < 0.02 < 0.02	0.09 0.04 0.06	94590 110951
	WP (40 %W/W)	6	0.070			20-21	0 4 7	Whole fruit	0.07 0.06 0.05	< 0.02 0.02 < 0.02	0.09 0.08 0.07	
	WP (40 %W/W)	6	0.14			20-21	0 4 7	Whole fruit	0.23 0.12 0.12	0.02 < 0.02 < 0.02	0.25 0.14 0.14	
OH01 USA 1991 (-)	WP (40 %W/W)	6	0.035			7-14	0 4 7	Whole fruit	0.02 0.02 < 0.02	< 0.02 < 0.02 < 0.02	0.04 0.04 < 0.04	94590 110951
	WP (40 %W/W)	6	0.070			7-14	0 4 7	Whole fruit	0.06 0.09 0.04	< 0.02 < 0.02 < 0.02	0.08 0.11 0.06	
	WP (40 %W/W)	6	0.14			7-14	0 4 7	Whole fruit	0.10 0.08 0.10	< 0.02 < 0.02 < 0.02	0.12 0.10 0.12	
OR06 USA 1991 (-)	WP (40 %W/W)	6	0.035			10-22	0 3 7	Whole fruit	0.02 0.02 < 0.02	< 0.02 < 0.02 < 0.02	0.04 0.04 < 0.04	94590 110951
	WP (40 %W/W)	6	0.070			10-22	0 3 7	Whole fruit	0.03 0.03 0.02	< 0.02 < 0.02 < 0.02	0.05 0.05 0.04	
	WP (40 %W/W)	6	0.14			10-22	0 3 7	Whole fruit	0.02 0.04 0.04	< 0.02 < 0.02 < 0.02	0.04 0.06 0.06	
NC15 USA 1992 (-)	WP (40 %W/W)	6	0.035			7-14	0 3 7	Whole fruit	0.11 0.05 0.03	< 0.02 < 0.02 < 0.02	0.13 0.07 0.05	94590 110951
	WP (40 %W/W)	6	0.070			7-14	0 3 7	Whole fruit	0.16 0.08 0.05	< 0.02 < 0.02 < 0.02	0.18 0.10 0.07	
	WP (40 %W/W)	6	0.14			7-14	0 3 7	Whole fruit	0.31 0.19 0.13	< 0.02 < 0.02 < 0.02	0.33 0.21 0.15	
PA03 USA 1992 (-)	WP (40 %W/W)	6	0.035			6-11	0 3 7	Whole fruit	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02	< 0.04 < 0.04 < 0.04	94590 110951
	WP (40 %W/W)	6	0.070			6-11	0 3 7	Whole fruit	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02	< 0.04 < 0.04 < 0.04	
	WP (40 %W/W)	6	0.14			6-11	0 3 7	Whole fruit	0.03 0.02 < 0.02	< 0.02 < 0.02 < 0.02	0.05 0.04 < 0.04	
451A/RH/90/ST R, Leeds, Kent, UK 1990 (Rapells)	EW (60 g ai/L)	6	0.090	1000	9.0	9-25	0 5 8 18	Whole fruit	1.21 0.36 0.11 0.07	0.07 < 0.01 < 0.01 < 0.01	1.28 0.37 0.12 < 0.01	241905
451B/RH/90/STR , Lamberburst Kent, UK 1990 (Rapells)	EW (60 g ai/L)	6	0.090	1000	9.0	10-25	0 7 17	Whole fruit	0.25 0.23 0.03	0.08 < 0.01 0.06	0.33 0.24 0.09	241905
RES/83/93/3	EW	5	0.090	500	18	12-28	0	Whole	0.38	< 0.01	0.39	242010

Trial ID/Location Country/Year (variety)	Form	No. of App s	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analyse d	Myclobutanil (mg/kg)	RH- 9090 (mg.kg)	Total of myclobutanil (mg/kg)	Ref.
Cupar, Fife UK, 1993 (Elsanta)	(60 g ai/L)						3 7 14	fruit	0.24 0.09 0.04	< 0.01 < 0.01 < 0.01	0.25 0.10 0.05	
RES/83/93/4 Inchture Tayside Scotland, 1993 (Honeoye)	EW (60 g ai/L)	5	0.090	500	18	11–28	0 3 7 14	Whole fruit	0.83 0.50 0.27 0.06	< 0.01 < 0.01 < 0.01 < 0.01	0.84 0.51 0.28 0.07	242010
RES/83/93/1 Ross on Wye	EW (60 g ai/L)	5	0.180	500	36	11–14	0 7	Whole fruit	1.01 0.20	< 0.01 < 0.01	1.02 0.21	242010
HerefordshireUK, 1993 (Elsanta)		5	0.090			11–14	0 3 7 14	Whole fruit	0.36 0.48 0.12 0.02	< 0.01 < 0.01 < 0.01 < 0.01	0.37 0.49 0.13 0.03	
RES/83/93/2 Ledbury Herefordshire UK, 1993 (Totem)	EW (60 g ai/L)	5	0.090	500	18	11–14	0 3 7 14	Whole fruit	0.48 0.69 0.28 0.03	< 0.01 < 0.01 < 0.01 < 0.01	0.49 0.70 0.29 0.04	242010
4839312 Francolino Ferrara, ITA 1993 (Addie)	EC (125 g ai/L)	3	0.058 – 0.067	930– 1070	6.25	7	0 3 7	Whole fruit	0.07 0.07 0.05	< 0.01 < 0.01 < 0.01	0.08 0.08 0.06	242017
AP/3194/HL/3F, Meauzac Southern FRA, 1996 (Mara des bois)	EW (200 g ai/L)	6	0.064 – 0.068	853– 911	7.5	9–13	0 3 7	Whole fruit	0.13 0.05 0.04	< 0.01 < 0.01 < 0.01	0.14 0.06 0.05	241761
AP/3194/HL/1F, Saint Porcharie Southern FRA, 1996 (Elsanta)	EW (200 g ai/L)	6	0.037 – 0.055	490– 738	7.5	10–11	0 3 7	Whole fruit	0.16 0.07 0.07	< 0.01 < 0.01 < 0.01	0.17 0.08 0.08	241761
AP/3194/HL/2F, Bonnes Southern FRA, 1996 (Elsanta)	EW (200 g ai/L)	6	0.035 – 0.049	469– 659	7.5	10–12	0 3 7	Whole fruit	0.17 0.08 0.05	< 0.01 < 0.01 < 0.01	0.18 0.09 0.06	241761
AP/3194/HL/4F, Cendrieux Southern FRA, 1996 (Seascape)	EW (200 g ai/L)	7	0.062 – 0.071	829– 952	7.5	10–14	0 3 7	Whole fruit	0.31 0.26 0.19	< 0.01 0.01 < 0.01	0.32 0.27 0.20	241761
AK/3279/HL/1 Notts, UK 1996 (Elsanta)	EW (200 g ai/L)	6	0.062 – 0.076	1235 – 1527	5.0	10–11	3 3 0 3	Jam Preserve Whole fruit	0.04 0.06 0.12 0.08	< 0.01 < 0.01 < 0.01 < 0.01	0.05 0.07 0.13 0.09	241762
AK/3279/HL/2 Kent, UK 1996 (Elsanta)	EW (200 g ai/L)	6	0.067 – 0.080	1339 – 1604	5.0	10–11	3 3 0 3	Jam Preserve Whole fruit	0.04 0.07 0.15 0.08	< 0.01 < 0.01 < 0.01 < 0.01	0.05 0.08 0.16 0.09	241762
AF/3548/HL/4I, Buttapietra ITA, 1997 (Eddie)	EC (240 g ai/L)	4	0.051 – 0.075	680– 1000	8.0	10	0 3 7	Whole fruit	0.25 0.22 0.18	< 0.01 < 0.01 < 0.01	0.26 0.23 0.19	241822
AF/3548/HL/3I, Forli, ITA 1997 (Marmolada)	EC (240 g ai/L)	4	0.076 – 0.11	1018 – 1464	8.0	10	0 3 7	Whole fruit	0.75 0.60 0.48	0.01 0.02 0.02	0.76 0.62 0.50	241822
AF/3548/HL/1S, Lepe, ESP 1998 (Camarrosa)	EC (240 g ai/L)	6	0.036 – 0.050	474– 664	8.0	11–14	0 3 7	Whole fruit	0.55 0.46 0.31	0.01 0.01 0.01	0.56 0.47 0.32	241822
AF/3548/HL/2S Lepe, ESP	EC (240 g ai/L)	6	0.061 –	815– 910	8.0	10–11	0 3	Whole fruit	0.23 0.18	< 0.01 < 0.01	0.24 0.19	241822

Trial ID/Location Country/Year (variety)	Form	No. of App s	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analyse d	Myclobutanil (mg/kg)	RH- 9090 (mg.kg)	Total of myclobutanil (mg/kg)	Ref.
1998 (Camarrosa)			0.068				7		0.11	< 0.01	0.12	
RAS/180-01 Conadado ESP, 2000 (Tudla)	EC (245.2 g ai/L)	5	0.061 – 0.062	796– 807	7.7	10–13	0 3 7	Whole fruit	0.07 0.08 0.05	< 0.01 < 0.01 < 0.01	0.08 0.09 0.06	260329
RAS/180-02 Conadado ESP, 2000 (Tudla)	EC (245.2 g ai/L)	5	0.062 – 0.066	807– 866	7.7	10–13	0 3 7	Whole fruit	0.18 0.12 0.08	< 0.01 < 0.01 < 0.01	0.19 0.13 0.09	260329
S06W048R Canals Valencia ESP, 2006 (Camarosa)	EW (200 g ai/L)	6	0.069 – 0.079	947– 1088	7.2	7–8	< 0 0 3 7	Whole fruit	0.15 0.28 0.30 0.14	0.01 0.01 0.01 0.01	0.16 0.29 0.31 0.15	200974 7
S07W023R Canals Valencia ESP, 2007	EW (200 g ai/L)	6	0.073 – 0.077	962– 1020	7.5	6–8	< 0 0 3 7	Whole fruit	0.15 0.34 0.37 0.20	0.01 0.01 0.01 0.01	0.16 0.35 0.38 0.21	200190 0
(Pajaro)	EW (45 g ai/L)	6	0.070 – 0.075	934– 1006	7.5	6–8	< 0 0 3 7	Whole fruit	0.18 0.46 0.46 0.27	< 0.01 0.01 0.01 0.01	0.19 0.47 0.47 0.28	
F07W071R Carpentras Provence-Alpes- Cote d’Azur	EW (200 g ai/L)	6	0.070 – 0.80	927– 1067	7.5	7	< 0 0 3 7	Whole fruit	0.15 0.21 0.16 0.10	0.01 0.01 0.01 < 0.01	0.16 0.22 0.17 0.11	200190 0
FRA, 2007 (Pajaro)	EW (45 g ai/L)	6	0.072 – 0.076	960– 1020	7.5	7	< 0 0 3 7	Whole fruit	0.12 0.12 0.20 0.12	0.01 < 0.01 0.01 < 0.01	0.13 0.13 0.21 0.13	
S07W152R Quatretonda Valencia ESP, 2007	EW (200 g ai/L)	6	0.071 – 0.079	947– 1051	7.5	7–8	< 0 0 3 7	Whole fruit	0.10 0.30 0.19 0.08	< 0.01 0.01 0.01 < 0.01	0.11 0.31 0.20 0.09	200190 0
(Candonga)	EW (45 g ai/L)	6	0.070 – 0.074	943– 994	7.5	7–8	< 0 0 3 7	Whole fruit	0.09 0.21 0.18 0.12	0.01 0.01 0.01 0.01	0.10 0.22 0.19 0.13	
S07W153R Quatretonda Valencia ESP, 2007	EW (200 g ai/L)	6	0.071 – 0.079	942– 1049	7.5	7–8	< 0 0 3 7	Whole fruit	0.10 0.21 0.24 0.10	< 0.01 0.01 0.01 0.01	0.11 0.22 0.25 0.11	200190 0
(Camarrosa)	EW (45 g ai/L)	6	0.074 – 0.077	984– 1034	7.5	7–8	< 0 0 3 7	Whole fruit	0.07 0.15 0.13 0.09	< 0.01 < 0.01 < 0.01 < 0.01	0.08 0.16 0.14 0.10	
S07W025R Quatretonda Valencia ESP, 2007 (Pajaro)	EW (200 g ai/L)	6	0.072 – 0.078	957– 1033	7.5	7–8	< 0 0 3 7	Whole fruit	0.06 0.28 0.14 0.09	< 0.01 0.01 0.01 0.01	0.07 0.29 0.15 0.10	200132 0
AK/2876/RH/1 Nr Retford Notts, England 1995 (Elsanta)	EW (60 g ai/L)	6	0.090	500	18	–	3	Whole fruit	0.20	< 0.01	0.21	241766
AK/2876/RH/2 Southwell Notts, England 1995 (Elsanta)	EW (60 g ai/L)	6	0.090	500	18	–	3	Whole fruit	0.19	< 0.01	0.20	241766
AK/2876/RH/3 Kings Lynn Norfolk England 1995 (Elsanta)	EW (60 g ai/L)	6	0.090	500	18	–	3	Whole fruit	0.22	< 0.01	0.23	241766

Trial ID/Location Country/Year (variety)	Form	No. of App s	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DA T	Portion Analyse d	Myclobutanil (mg/kg)	RH- 9090 (mg.kg)	Total of myclobutanti l (mg/kg)	Ref.
AK/2876/RH/4 Warmington Northants England, 1995 (Elsanta)	EW (60 g ai/L)	6	0.090	500	18	—	3	Whole fruit	0.19	< 0.01	0.20	241766
AK/2876/RH/1 Nr Retford Notts, England 1995 (Elsanta)	WP (6% W/W)	6	0.090	500	18	—	3	Whole fruit	0.17	< 0.01	0.18	241766
AK/2876/RH/2 Southwell Notts, England 1995 (Elsanta)	WP (6% W/W)	6	0.090	500	18	—	3	Whole fruit	0.20	< 0.01	0.21	241766
AK/2876/RH/3 Kings Lynn Norfolk England 1995 (Elsanta)	WP (6% W/W)	6	0.090	500	18	—	3	Whole fruit	0.15	< 0.01	0.16	241766
AK/2876/RH/4 Warmington Northants England, 1995 (Elsanta)	WP (6% W/W)	6	0.090	500	18	—	3	Whole fruit	0.18	< 0.01	0.19	241766
AK/2876/RH/1 Nr Retford Notts, England 1995 (Elsanta)	EW (200 g ai/L)	6	0.090	500	18	—	3	Whole fruit	0.18	< 0.01	0.19	241766
AK/2876/RH/2 Southwell Notts, England 1995 (Elsanta)	EW (200 g ai/L)	6	0.090	500	18	—	3	Whole fruit	0.19	< 0.01	0.20	241766
AK/2876/RH/3 Kings Lynn Norfolk England 1995 (Elsanta)	EW (200 g ai/L)	6	0.090	500	18	—	3	Whole fruit	0.10	< 0.01	0.11	241766
AK/2876/RH/4 Warmington Northants England, 1995 (Elsanta)	EW (200 g ai/L)	6	0.090	500	18	—	3	Whole fruit	0.19	< 0.01	0.20	241766
4919212 Bonmati, GI ESP, 1992 (Selva)	EC (125 g ai/L)	2	0.088	800	11	7	0 3 7 13	Whole fruit	0.20 0.15 0.08 0.08	— — — —	— — — —	241969
		1	0.088	800	11	—	0 3 7 13	Whole fruit	0.20 0.07 0.12 0.03	— — — —	— — — —	
4918840 Condado, Huelva ESP, 1988 (Chandler)	EC (125 g ai/L)	5	0.093	1500	6.2	15–20	3 7 14	Whole fruit	0.12 0.07 0.04	— — —	— — —	242195
		5	0.11	1500	7.5	15–20	3 7 14	Whole fruit	0.15 0.08 0.05	— — —	— — —	
4048871 Coriano, VR ITA, 1988 (Addie)	EC (125 g ai/L)	3	0.038 — 0.075	750– 1500	5.0	14	7	Whole fruit	0.09	< 0.01	0.10	242989
		3	0.075 –0.15	750– 1500	10.0	14	7	Whole fruit	0.12	< 0.01	0.13	

*Assorted tropical fruits, inedible peel**Banana*

A total of 11 trials on banana were conducted in USA and Costa Rica according to treatment of myclobutanil at banana packing stations in Costa Rica in 1991 through 1993. All treatments consisted of one application of myclobutanil, and banana hands were sprayed or dipped at various concentrations. Results of bananas are summarized in Table 60. The total residues were calculated on the basis of peel and pulp and summarized Table 61.

Table 60 Supervised trials on banana in Costa Rica and USA

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DATE	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
91-0011 San Leandro, CA USA, 1991 (Cavendish)	EC (250 g ai/L)	1	—	—	200 Spray	—	0	Peel	0.36	0.01	0.37	94396
							7	Peel	0.30	0.01	0.31	
							14	Peel	0.33	0.01	0.34	
							21	Peel	— ^a	0.03	—	
							0	Pulp	< 0.01	< 0.01	< 0.02	
							7	Pulp	0.01	< 0.01	0.02	
							14	Pulp	0.02	< 0.01	0.03	
							21	Pulp	0.02	< 0.01	0.03	
	EC (250 g ai/L)	1	—	—	400 Spray	—	0	Peel	0.36	< 0.01	0.37	94396
							7	Peel	0.51	0.01	0.52	
							14	Peel	0.34	0.02	0.36	
							21	Peel	0.82	0.02	0.84	
							0	Pulp	< 0.01	< 0.01	< 0.02	
							7	Pulp	0.02	< 0.01	0.03	
							14	Pulp	0.02	0.01	0.03	
							21	Pulp	0.03	< 0.01	0.04	
	EC (250 g ai/L)	1	—	—	800 Spray	—	0	Peel	0.03	0.01	0.04	94396
							7	Peel	0.66	0.01	0.67	
							14	Peel	0.15	< 0.01	0.16	
							21	Peel	0.09	< 0.01	0.10	
							0	Pulp	< 0.01	< 0.01	< 0.02	
							7	Pulp	0.01	< 0.01	< 0.02	
							14	Pulp	0.02	< 0.01	0.03	
							21	Pulp	< 0.01	< 0.01	< 0.02	
91-0014 Walnut Creek CA, USA, 1991 (Not applicable)	EC (250 g ai/L)	1	—	—	200 Dip	—	0	Peel	0.67	< 0.01	0.68	94396
							7	Peel	0.93	0.02	0.95	
							14	Peel	1.12	0.02	1.14	
							21	Peel	1.00	0.02	1.02	
							0	Pulp	< 0.01	< 0.01	< 0.02	
							7	Pulp	0.03	0.01	0.04	
							14	Pulp	0.03	< 0.01	0.04	
							21	Pulp	0.03	< 0.01	0.04	
	EC (250 g ai/L)	1	—	—	200 Spray	—	0	Peel	0.03	< 0.01	0.04	94396
							7	Peel	0.02	< 0.01	0.03	
							14	Peel	0.03	< 0.01	0.04	
							21	Peel	0.02	< 0.01	0.03	
							0	Pulp	0.01	< 0.01	0.02	
							7	Pulp	< 0.01	< 0.01	< 0.02	
							14	Pulp	—	< 0.01	—	
							21	Pulp	< 0.01	< 0.01	< 0.02	
	EC (250 g ai/L)	1	—	—	400 Dip	—	0	Peel	1.33	0.01	1.34	94396
							7	Peel	1.71	0.03	1.74	
							14	Peel	1.61	0.03	1.64	
							21	Peel	1.44	0.04	1.48	
							0	Pulp	0.01	< 0.01	0.02	
							7	Pulp	0.01	0.01	0.02	
							14	Pulp	0.04	0.01	0.05	
							21	Pulp	0.05	< 0.01	0.06	
	EC	1	—	—	400	—	0	Peel	< 0.01	< 0.01	< 0.02	94396

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
	(250 g ai/L)				Spray		7 14 21 0 7 14 21	Peel Peel Peel Pulp Pulp Pulp Pulp	0.01 0.06 0.13 < 0.01 0.02 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.07 0.14 < 0.02 0.03 0.02 0.02	
91-0012 San Jose, CA USA 1991 (Cavendish)	EC (250 g ai/L)	1	—		200 Dip	—	0 7 14 21 0 7 14 21	Peel Peel Peel Peel Pulp Pulp Pulp Pulp	1.08 1.14 1.10 1.58 < 0.01 0.03 0.03 0.07	0.01 0.08 0.05 0.04 < 0.01 0.01 0.01 0.01	1.09 1.22 1.15 1.62 < 0.02 0.04 0.04 0.08	94396
	EC (250 g ai/L)	1	—		400 Dip	—	0 7 14 21 0 7 14 21	Peel Peel Peel Peel Pulp Pulp Pulp Pulp	1.64 2.06 2.34 2.54 < 0.01 0.05 0.06 —	0.01 0.08 0.07 0.06 < 0.01 0.02 0.01 0.01	1.65 2.14 2.41 2.60 < 0.02 0.07 0.07 —	94396
	EC (250 g ai/L)	1	—		800 Dip	—	0 7 14 21 0 7 14 21	Peel Peel Peel Peel Pulp Pulp Pulp Pulp	3.01 3.77 3.21 4.59 < 0.01 0.11 0.09 0.14	< 0.01 0.02 0.06 0.01 < 0.01 0.03 0.02 0.01	3.02 3.79 3.27 4.60 < 0.02 0.14 0.11 0.15	94396
91-0013 Gilroy, CA USA 1991 (Not available)	EC (250 g ai/L)	1	—		200 Spray		0 7 14 21 0 7 14 21	Peel Peel Peel Peel Pulp Pulp Pulp Pulp	0.13 0.21 0.41 0.36 < 0.01 0.02 — < 0.01	< 0.01 0.01 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.14 0.22 0.42 0.37 < 0.02 0.01 — < 0.02	94396
	EC (250 g ai/L)	1	—		400 Spray		0 7 14 21 0 7 14 21	Peel Peel Peel Peel Pulp Pulp Pulp Pulp	0.28 0.56 0.60 0.72 < 0.01 0.02 0.03 0.02	< 0.01 0.02 0.03 < 0.01 < 0.01 < 0.01 0.01 < 0.01	0.29 0.58 0.63 0.73 < 0.02 0.03 0.04 0.03	94396
	EC (250 g ai/L)	1	—		800 Spray		0 7 14 21 0 7 14 21	Peel Peel Peel Peel Pulp Pulp Pulp Pulp	0.04 0.20 0.59 0.65 < 0.01 0.02 0.02 0.02	< 0.01 0.01 0.03 0.01 < 0.01 0.01 < 0.01 < 0.01	0.05 0.21 0.62 0.66 < 0.02 0.03 0.03 0.03	94396
92-0063 Keaau Plantation, Inc. Hilo, Hawaii, USA 1992 (Williams, Grand Maine,	EC (250 g ai/L)	1	—		400 Spray		0 7 14 21 28 0 7	Peel Peel Peel Peel Peel Pulp Pulp	2.26 2.72 2.08 2.84 2.58 0.02 0.10	< 0.01 0.03 0.05 0.06 0.07 < 0.01 < 0.01	2.27 2.75 2.13 2.90 2.65 0.03 0.11	94256

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
Valerie)							14 21 28	Pulp Pulp Pulp	0.13 0.17 0.15	0.01 0.02 0.03	0.14 0.19 0.18	
	EC (250 g ai/L)	1	—		800 Spray		0 7 14 21 28 0 7 14 21 28	Peel Peel Peel Peel Peel Pulp Pulp Pulp Pulp Pulp	4.93 6.94 4.93 5.74 5.60 0.04 0.23 0.27 0.32 0.28	0.01 0.05 0.08 0.11 0.10 < 0.01 < 0.01 0.02 0.03 0.03	4.94 6.99 5.01 5.85 5.70 0.05 0.24 0.29 0.35 0.31	94256
	WP (40% W/W)	1	—		400 Spray		0 14 28 0 14 28	Peel Peel Peel Pulp Pulp Pulp	1.56 1.45 1.67 0.02 0.19 0.10	< 0.01 0.05 0.05 < 0.01 0.01 0.02	1.57 1.50 1.72 0.03 0.20 0.12	94256
	WP (40% W/W)	1	—		800 Spray		0 14 28 0 14 28	Peel Peel Peel Pulp Pulp Pulp	1.96 3.33 2.88 0.03 0.24 0.24	< 0.01 0.06 0.09 < 0.01 0.01 0.03	1.97 3.69 2.97 0.04 0.25 0.27	94256
92-0064 Robert Ha Inc. Hilo, Hawaii USA 1992 (Williams,	EC (250 g ai/L)	1	—		200 Spray		0 14 28 0 14 28	Peel Peel Peel Pulp Pulp Pulp	1.25 1.58 1.10 0.02 0.14 0.08	< 0.01 0.05 0.05 < 0.01 0.01 0.03	1.26 1.63 1.15 0.03 0.15 0.11	94256
Grand Maine, Valerie)	EC (250 g ai/L)	1	—		400 Spray		0 14 28 0 14 28	Peel Peel Peel Pulp Pulp Pulp	2.72 2.26 3.24 0.04 0.22 0.28	0.01 0.07 0.11 < 0.01 0.02 0.03	2.73 2.33 3.35 0.05 0.24 0.31	94256
	WP (40% W/W)	1	—		400 Spray		0 14 28 0 14 28	Peel Peel Peel Pulp Pulp Pulp	1.79 2.33 2.67 0.03 0.21 0.41	< 0.01 0.06 0.08 < 0.01 0.03 0.04	1.80 2.39 2.75 0.04 0.24 0.45	94256
92-0061 Valery Banana Company Kurtistown Hawaii, USA, 1992 (Williams, Grand Maine, Valerie)	EC (240 g ai/L)	1	—		200 Spray		0 7 14 21 28 0 7 14 21 28	Peel Peel Peel Peel Peel Pulp Pulp Pulp Pulp Pulp	1.02 1.14 1.14 1.28 1.16 0.01 0.09 0.11 0.09 0.07	< 0.01 0.03 0.04 0.04 0.04 < 0.01 < 0.01 < 0.01 0.05 0.04	1.03 1.17 1.18 1.32 1.20 0.02 0.10 0.12 0.14 0.11	94256
	EC (250 g ai/L)	1	—		400 Spray		0 7 14 21 28 0 7 14 21	Peel Peel Peel Peel Peel Pulp Pulp Pulp Pulp	1.93 2.21 1.76 2.25 1.99 0.03 0.19 0.19 0.22	0.01 0.06 0.06 0.08 0.08 < 0.01 0.01 0.02 0.06	1.94 2.27 1.82 2.33 2.07 0.04 0.20 0.21 0.28	94256

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
							28	Pulp	0.17	0.07	0.24	
	EC (250 g ai/L)	1	—		800 Spray		0	Peel	5.18	0.01	5.19	94256
							7	Peel	4.92	0.08	5.00	
							14	Peel	4.20	0.08	4.28	
							21	Peel	4.05	0.13	4.18	
							28	Peel	3.69	0.14	3.83	
							0	Pulp	0.03	< 0.01	0.04	
							7	Pulp	0.26	0.02	0.28	
							14	Pulp	0.33	0.03	0.36	
							21	Pulp	0.47	0.09	0.56	
							28	Pulp	0.30	0.05	0.35	
92-0062 Keaau Plantation, Inc. Hilo, Hawaii, USA 1992	EC (250 g ai/L)	1	—		200 Dip		0	Peel	1.34	< 0.01	1.35	94256
							14	Peel	1.14	0.04	1.18	
							28	Peel	0.98	0.08	1.06	
							0	Pulp	0.02	< 0.01	0.03	
							14	Pulp	0.11	< 0.01	0.12	
							28	Pulp	0.17	0.02	0.19	
(Williams, Grand Maine, Valerie)	EC (250 g ai/L)	1	—		400 Dip		0	Peel	2.04	0.01	2.05	94256
							14	Peel	1.94	0.06	2.00	
							28	Peel	1.53	0.09	1.62	
							0	Pulp	0.03	< 0.01	0.04	
							14	Pulp	0.21	0.01	0.22	
							28	Pulp	0.20	0.08	0.28	
	EC (250 g ai/L)	1	—		400 Spray		0	Peel	2.56	0.01	2.57	94256
							14	Peel	2.27	0.07	2.34	
							28	Peel	3.65	0.12	3.77	
							0	Pulp	0.05	< 0.01	0.06	
							14	Pulp	0.25	0.01	0.26	
							28	Pulp	0.27	0.05	0.32	
	WP (40% W/W)	1	—		400 Dip		0	Peel	1.58	0.01	1.59	94256
							14	Peel	1.27	0.06	1.33	
							28	Peel	1.60	0.07	1.67	
							0	Pulp	0.03	< 0.01	0.04	
							14	Pulp	0.17	0.03	0.20	
							28	Pulp	0.12	0.02	0.14	
	WP (40% W/W)	1	—		400 Spray		0	Peel	1.32	< 0.01	1.33	94256
							14	Peel	1.38	0.06	1.44	
							28	Peel	1.48	0.07	1.55	
							0	Pulp	0.02	< 0.01	0.03	
							14	Pulp	0.21	0.01	0.22	
							28	Pulp	0.18	0.04	0.22	
93-0039 Finca La Guaria, Canton de Matina Provincica de Limon, Costa Rica, 1993 (Cavendish)	EC (250 g ai/L)	1	—		100 Spray		7	Peel	0.45	0.01	0.46	94255
							14	Peel	0.63	0.02	0.65	
							21	Peel	0.53	0.03	0.56	
							7	Pulp	0.05	0.00	0.05	
							14	Pulp	0.07	0.00	0.07	
							21	Pulp	0.07	0.00	0.07	
	EC (250 g ai/L)	1	—		200 Spray		7	Peel	1.11	0.02	1.13	94255
							14	Peel	1.18	0.03	1.21	
							21	Peel	1.32	0.05	1.37	
							7	Pulp	0.10	0.00	0.10	
							14	Pulp	0.10	0.00	0.10	
							21	Pulp	0.14	0.02	0.16	
	EC (250 g ai/L)	1	—		400 Spray		7	Peel	3.26	0.04	3.30	94255
							14	Peel	3.62	0.06	3.68	
							21	Peel	3.77	0.09	3.86	
							7	Pulp	0.23	0.00	0.23	
							14	Pulp	0.31	0.02	0.33	
							21	Pulp	0.35	0.03	0.38	
93-0040 Finca Sahara	EC (250 g ai/L)	1	—		100 Spray		7	Peel	0.54	0.01	0.55	94255
							14	Peel	0.63	0.02	0.65	

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
Canton de Matina Provincia de Limon, Costa Rica, 1993 (Cavendish)	EC (250 g ai/L)	1	—		200 Spray		21	Peel	0.51	0.02	0.53	94255
							7	Pulp	0.05	0.00	0.05	
							14	Pulp	0.06	0.00	0.06	
							21	Pulp	0.05	0.00	0.05	
	EC (250 g ai/L)	1	—		400 Spray		7	Peel	1.17	0.02	1.19	94255
							14	Peel	1.07	0.01	1.08	
							21	Peel	1.57	0.03	1.60	
							7	Pulp	0.09	0.01	0.10	
							14	Pulp	0.11	0.00	0.11	94255
							21	Pulp	0.16	0.01	0.17	
							7	Peel	2.95	0.03	2.98	
							14	Peel	2.81	0.05	2.86	
							21	Peel	2.91	0.06	2.97	94255
							7	Pulp	0.17	0.00	0.17	
							14	Pulp	0.29	0.02	0.31	
							21	Pulp	0.26	0.03	0.29	
93-0041 Finca La Guaria, Canton de Matina Provincia de Limon, 1993 (Cavendish)	EC (250 g ai/L)	1	—		100 Spray		7	Peel	0.53	0.02	0.55	94255
							14	Peel	0.57	0.03	0.60	
							21	Peel	0.51	0.02	0.53	
							7	Pulp	0.05	0.00	0.05	
							14	Pulp	0.08	0.00	0.08	94255
							21	Pulp	0.09	0.00	0.09	
	EC (250 g ai/L)	1	—		200 Spray		7	Peel	0.99	0.02	1.01	
							14	Peel	1.33	0.04	1.37	
							21	Peel	1.22	0.06	1.28	94255
							7	Pulp	0.12	0.00	0.12	
							14	Pulp	0.12	0.00	0.12	
							21	Pulp	0.13	0.00	0.13	
	EC (250 g ai/L)	1	—		400 Spray		7	Peel	2.70	0.04	2.74	94255
							14	Peel	2.86	0.06	2.92	
							21	Peel	3.83	0.09	3.92	
							7	Pulp	0.32	0.00	0.32	
							14	Pulp	0.29	0.02	0.31	94255
							21	Pulp	0.39	0.02	0.41	

^a Lack of myclobutanil data in peel

Table 61 Residues of myclobutanil in banana presented on whole fruit basis from 400 g ai/hL treated plots

Trial ID	Form No.	No. of Apps	Appl Conc (ppm)	DAT	Peel Myclobutanil (mg/kg)	Pulp Myclobutanil (mg/kg)	Weight Factor ^a	Total Myclobutanil (mg/kg) ^b	Reference
91-0011	EC (250 g ai/L)	1	400	0	0.36	0	NA	0.13	94396
USA			Spray	7	0.51	0.02	NA	0.19	
1991				14	0.34	0.02	NA	0.13	
				21	0.82	0.03	NA	0.30	
91-0014	EC (250 g ai/L)	1	400	0	1.33	0.01	NA	0.47	94396
USA			Dip	7	1.71	0.01	NA	0.61	
1991				14	1.61	0.04	NA	0.59	
				21	1.44	0.05	NA	0.54	
91-0014	EC (250 g ai/L)	1	400	0	0	0	NA	0	94396
USA			Spray	7	0.01	0.01	NA	0.01	
1991				14	0.06	0.01	NA	0.03	
				21	0.13	0.01	NA	0.05	
91-0012	EC (250 g ai/L)	1	400	0	1.64	0	NA	0.57	94396
USA			Dip	7	2.06	0.05	NA	0.75	
1991				14	2.34	0.06	NA	0.86	
				21	2.54	—	NA	0.89	

Trial ID	Form No.	No. of Appls	Appl Conc (ppm)	DAT	Peel Myclobutanil (mg/kg)	Pulp Myclobutanil (mg/kg)	Weight Factor ^a	Total Myclobutanil (mg/kg) ^b	Reference
91-0013	EC (250 g ai/L)	1	400	0	0.28	0	NA	0.10	94396
USA			Spray	7	0.56	0.02	NA	0.21	
1991				14	0.60	0.03	NA	0.23	
				21	0.72	0.02	NA	0.27	
92-0063	EC (250 g ai/L)	1	400	0	2.26	0.02	0.55	1.03	94256
USA			Spray	7	2.72	0.10	0.57	1.23	
1992				14	2.08	0.13	0.58	0.95	
				21	2.83	0.17	0.61	1.21	
				28	2.58	0.15	0.64	1.02	
92-0063	WP (40% W/W)	1	400	0	1.57	0.02	0.54	0.73	94256
USA			Spray	14	1.45	0.19	0.58	0.72	
1992				28	1.67	0.10	0.65	0.65	
92-0064	EC (250 g ai/L)	1	400	0	2.71	0.04	0.55	1.24	94256
USA			Spray	14	2.26	0.22	0.62	1.00	
1992				28	3.24	0.28	0.67	1.26	
92-0064	WP (40% W/W)	1	400	0	1.79	0.03	0.55	0.82	94256
USA			Spray	14	2.33	0.21	0.64	0.97	
1992				28	2.68	0.41	0.7	1.09	
92-0061	EC (250 g ai/L)	1	400	0	1.93	0.03	0.55	0.89	94256
USA			Spray	7	2.21	0.19	0.58	1.04	
1992				14	1.76	0.19	0.6	0.82	
				21	2.25	0.22	0.62	0.99	
				28	1.99	0.17	0.66	0.79	
92-0062	EC (250 g ai/L)	1	400	0	2.04	0.03	0.57	0.89	94256
USA			Dip	14	1.94	0.21	0.6	0.90	
1992				28	1.52	0.20	0.69	0.61	
92-0062	EC (250 g ai/L)	1	400	0	2.57	0.05	0.57	1.13	94256
USA			Spray	14	2.27	0.25	0.62	1.02	
1992				28	3.65	0.28	0.7	1.29	
92-0062	WP (40% W/W)	1	400	0	1.57	0.03	0.56	0.71	94256
USA			Dip	14	1.27	0.17	0.64	0.56	
1992				28	1.6	0.12	0.67	0.61	
92-0062	WP (40% W/W)	1	400	0	1.32	0.02	0.54	0.62	94256
USA			Spray	14	1.38	0.21	0.61	0.66	
1992				28	1.48	0.18	0.65	0.63	
93-0039	EC (250 g ai/L)	1	400	7	3.26	0.23	0.58	1.50	94255
CRI			Spray	14	3.62	0.31	0.609	1.61	
1993				21	3.77	0.35	0.638	1.59	
93-0040	EC (250 g ai/L)	1	400	7	2.95	0.17	0.58	1.34	94255
CRI			Spray	14	2.81	0.29	0.609	1.28	
1993				21	2.91	0.26	0.638	1.22	
93-0041	EC (250 g ai/L)	1	400	7	2.70	0.32	0.58	1.32	94255
CRI			Spray	14	2.86	0.29	0.609	1.29	
1993				21	3.83	0.39	0.638	1.64	

^a Weight factor = Weight of pulp/ Weight of whole banana This was only available for 1992 and 1993 studies.

^b total residues were calculated from the residues measured in peel and pulp based on weight factor

Fruiting vegetables cucurbits

Cucumber

A total of 31 trials on cucumber were conducted in different representative growing areas in France, Italy, Spain and USA during 1986 through 2007 growing seasons. Results of cucumber fruits are summarized in Table 62.

Table 62 Supervised trials on cucumber in various countries

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
86-0240 Harrison, Camilla, GA USA 1986 (Pickling)	WP (40 %W/W)	6	0.071	—	—	6–8	0 5	Whole fruit	0.02 0.01	0.01 0.00	0.03 0.00	94399 94397
86-0240 Harrison, Camilla, GA USA 1986 (Pickling)	WP (40 %W/W)	6	0.14	—	—	6–8	0	Whole fruit	0.02	0.01	0.03	94399 94397
86-0240 Harrison, Camilla, GA USA 1986 (Pickling)	DF (60% W/W)	6	0.071			6–8	0 5	Whole fruit	0.02 0.00	0.01 0.00	0.03	94399 94397
86-0220 Fleischfresser, Homestead, FL USA 1986 (Poinsett 76)	WP (40 %W/W)	8	0.071	477	—	7–8	0 3 5	Whole fruit	0.05 < 0.01 0.01	0.01 0.01 0.01	0.06 0.02 0.02	94399
86-0203 Green, Cleveland, MS USA 1986 (Poinsett)	WP (40 %W/W)	5	0.071	15	—	7	5	Whole fruit	0.01	0.00	0.01	94399
86-0203 Green, Cleveland, MS USA 1986 (Poinsett)	WP (40 %W/W)	5	0.14	29	—	7	5	Whole fruit	0.02	0.00	0.02	94399
86-0269 Holowid, Celeryville, OH USA 1986 (Roadside Fan)	WP (40 %W/W)	7	0.071	—	—	7	0 3 5	Whole fruit	0.02 0.02 0.01	0.00 0.00 0.00	0.02 0.02 0.01	94399
86-0265 Laughner, Newtwn, PA USA 1986 (Marketer)	WP (40 %W/W)	3	0.071	486	—	7–9	5	Whole fruit	0.01	0.00	0.01	94399
86-0265 Laughner, Newtwn, PA USA 1986 (Marketer)	WP (40 %W/W)	3	0.14	486	—	7–9	5	Whole fruit	0.01	0.00	0.01	94399
86-0226 Fresno, CA USA 1986 (Bounty)	WP (40 %W/W)	3	0.14	281	—	6–7	0 4 6 11	Whole fruit	0.01 < 0.01 0.00 0.00	0.00 0.00 0.00 0.00	0.01 < 0.01 0.00 0.00	94438
90-0089 Fremont, OH USA 1990 (Endeavor)	WP (40 %W/W)	5	0.071	327	—	7–11	0	Whole fruit	0.05	0.01	0.06	94397
90-0089 Fremont, OH USA 1990 (Endeavor)	WP (40 %W/W)	5	0.14	327	—	7–11	0	Whole fruit	0.04	0.00	0.04	94397
90-0115	WP	5	0.071	327	—	7–10	0	Whole	0.02	0.00	0.02	94397

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
Blissfield, MI USA 1990 (Calypso)	(40 %W/W)							fruit				
90-0115 Blissfield, MI USA 1990 (Calypso)	WP (40 %W/W)	5	0.14	327	—	7–10	0	Whole fruit	0.07	0.00	0.07	94397
90-0163 Bull Tall, NC USA 1990 (Dasher II)	WP (40 %W/W)	5	0.071	309	—	6–8	0	Whole fruit	0.00	0.00	0.00	94397
90-0163 Bull Tall, NC USA 1990 (Dasher II)	WP (40 %W/W)	5	0.14	309	—	6–8	0 3 7	Whole fruit	0.02 0.03 0.02	0.00 0.01 0.01	0.02 0.04 0.03	94397
94-0109 Westfield, NY USA 1994 (Marketmore)	WP (40 %W/W)	6	0.112	274	—	5–8	0	Whole fruit	0.03	0.01	0.04	94444
94-0160 Boyntonbeach, FL, USA, 1994 (Poinsett 76)	WP (40 %W/W)	6	0.112	281	—	6–9	0	Whole fruit	0.04	0.00	0.04	94444
AP/3192/HL/2F, Villaries FRA, 1996 (Bronco)	EC (240 g ai/L)	6	0.050 — 0.062	671– 824	7.5	10–13	0 3 7	Whole fruit	0.04 0.04 0.02	< 0.01 < 0.01 < 0.01	0.05 0.05 0.03	24216 0
AP/3192/HL/1S, Los Palacios, Andalucia, ESP, 1996 (Dasher II)	EC (240 g ai/L)	6	0.096 — 0.23	1279– 3076	7.5	10–11	0 3 7	Whole fruit	0.06 0.06 0.03	< 0.01 < 0.01 < 0.01	0.07 0.07 0.04	24216 0
AP/3192/HL/3F, Sainte Livade, FRA 1996 (Tiria)	EC (240 g ai/L)	5	0.049 — 0.069	647– 923	7.5	10–11	0 3 7	Whole fruit	0.07 0.06 0.03	< 0.01 < 0.01 < 0.01	0.08 0.07 0.04	24216 0
AP/3192/HL/1F, Villaries, FRA, 1996 (Girola)	EC (240 g ai/L)	6	0.049 — 0.073	653– 973	7.5	10–11	0 3 7	Whole fruit	0.05 0.03 0.02	0.00 < 0.01 < 0.01	0.05 0.04 0.03	24216 0
AP/3192/HL/3S, Sanlucar de Barrameda, Provincia de Cadiz, ESP 1996 (Dasher II)	EC (240 g ai/L)	6	0.15– 0.20	2029– 2622	7.5	11–14	0 3 7	Whole fruit	0.05 0.04 0.01	0.00 < 0.01 < 0.01	0.05 0.05 0.02	24216 0
AP/3192/HL/2S, Los Palacios, Andalucia, ESP, 1996 (Dasher II)	EC (240 g ai/L)	5	0.16– 0.28	2075– 3688	7.5	11–14	0 3 7	Whole fruit	0.09 0.06 0.00	0.00 0.00 0.00	0.09 0.06 0.00	24216 0
S07W027R Canals, Valencia, ESP 2007 (Tipo Espanol)	EW (200 g ai/L)	8	0.095 — 0.11	945– 1051	10	6–10	< 0 0 3 7	Whole fruit	0.01 0.07 0.02 0.01	0.00 0.00 0.00 0.00	0.01 0.07 0.02 0.00	20013 21
F07W076R Pernes-les-Fontaines, Provence – Alpes-Cote d’Azur, ESP 2007 (Noa) Greenhouse	EW (200 g ai/L)	6	0.093 — 0.10	928– 1038	10	7–10	< 0 0 3 7	Whole fruit	0.03 0.09 0.04 0.02	0.01 0.01 0.01 0.01	0.04 0.10 0.05 0.03	20013 21

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicat ion Interval (days)	DAT	Portion Analyse d	Myclobut anil (mg/kg)	RH-9090 (mg/kg)	Total of myclobuta nil (mg/kg)	Ref.
S07W028R Llanera, Valencia, ESP 2007 (Tipo Espanol)	EW (200 g ai/L)	8	0.096 –0.10	974– 1040	10.1	7–8	< 0 0 3 7	Whole fruit	0.02 0.02 0.05 0.01	< 0.01 0.00 < 0.01 0.00	0.03 0.02 0.06 0.01	20013 21
I07W011R Giovinazzo, Puglia, ITA 2007 (Polignanese) Greenhouse	EW (200 g ai/L)	8	0.094 –0.10	939– 102	10	7–9	< 0 0 3 7	Whole fruit	0.05 0.06 0.04 0.05	0.01 0.01 0.01 0.01	0.06 0.07 0.05 0.06	20013 21
S06W043R Canals, Valencia, ESP 2006 (Serena)	EW (200 g ai/L)	8	0.093 – 0.098	940– 1019	9.6	7–9	< 0 0 3 7	Whole fruit	0.01 0.12 0.08 0.03	0.00 0.00 0.00 0.00	0.01 0.12 0.08 0.03	20095 86
I06W031R Granarolo, Emilia Romagna,Regio n, Bologna, ITA, 2006 (Dinero)	EW (200 g ai/L)	8	0.095 – 0.098	985– 1035	9.6	7–9	< 0 0 3 7	Whole fruit	0.00 0.04 0.03 0.01	0.00 0.00 0.00 0.00	0.00 0.04 0.03 0.01	20095 86
F06W064R Brouilla, Languedoc- Roussillon, FRA 2006 (Jazzer)	EW (200 g ai/L)	8	0.096 –0.10	998– 1042	9.6	7–9	< 0 0 3 7	Whole fruit	0.01 0.04 0.04 0.01	0.00 0.00 0.00 0.00	0.01 0.04 0.04 0.01	20095 86

Melons

A total of 21 trials on melon were conducted in different representative growing areas in France, Greece, Italy, Spain and USA during 1986 through 1997 growing seasons. Results of melon fruits are summarized in Table 63.

Table 63 Supervised trials on melon in various countries

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicat ion Interval (days)	DAT	Portion Analyse d	Myclobu tanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobu tanil (mg/kg)	Ref.
86-0271 Celeryville, OH USA 1986, (Summit) Cantaloupe	WP (40 %W/ W)	8	0.067	–	–	7	0 3 5 10	Whole fruit	0.01 0.02 0.01 0.01	0.01 0.00 0.00 0.00	0.02 0.02 0.01 0.01	94262
86-0281 Belridge, CA USA 1986, (Top Mark) Cantaloupe	WP (40 %W/ W)	2	0.28	327	–	21	11	Whole fruit	0.03	0.01	0.04	94262
86-0369 Salisbury, MO USA 1986 (Jaeger) Cantaloupe	WP (40 %W/ W)	7	0.071	468	–	5–9	0 3 5 10	Whole fruit	0.04 0.03 0.03 0.01	0.07 0.03 0.04 < 0.01	0.11 0.06 0.07 0.02	94261
88-0148 Yuma, AZ USA 1987 (Topmark) Cantaloupe	DF (60% W/W)	8	0.067	187	–	7–20	0	Whole fruit	0.16	0.00	0.16	94260
87-0249	DF	6	0.067	187	–	7	0	Whole	0.02	0.01	0.03	94260

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicat ion Interval (days)	DAT	Portion Analyse d	Myclobu tanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobu tanil (mg/kg)	Ref.
Camilla, GA USA 1987 (Planters jun) Cantaloupe	(60% W/W)						2 5	fruit	0.01 0.00	0.01 0.01	0.02 0.01	
87-0356 Von Ormy, TX USA 1987 (Grande Gold) Cantaloupe	DF (60% W/W)	7	0.067	187	—	6–8	0 5	Whole fruit	0.06 0.03	0.01 0.01	0.07 0.04	94260
86-0284 Newtown, PA USA 1986 (Harvestqueen) Cantaloupe	WP (40 %W/ W)	5	0.14	486	—	7–9	3 5	Whole fruit	0.01 0.01	0.01 0.00	0.02 0.01	94283
86-0338 Vincennes, IN USA 1986 (Superstar) Muskmelon	WP (40 %W/ W)	5	0.13	636	—	6–7	0 3 5 10	Whole fruit	0.02 0.02 0.00 0.01	0.00 0.00 0.00 0.00	0.02 0.02 0.00 0.01	94283
86-0271 Celeryville, OH USA 1986 (Summit)	WP (40 %W/ W)	8	0.067	—	—	7	0	Whole fruit	0.02	0.01	0.03	94397
87-0356 Von Ormy, TX USA 1987 (Grande gold)	DF (60% W/W)	7 7	0.067 0.13	187 187	— —	5–8 5–8	0 0	Whole fruit	0.05 0.03	0.01 0.01	0.06 0.04	94397
88-0148 Yuma, AZ USA 1988 (Topmark)	DF (60% W/W)	8	0.071	187	—	7–20	0	Whole fruit	0.03	0.00	0.03	94397
90-0128 Woodland, CA USA 1990 (PMR45)	WP (40 %W/ W)	6 6	0.071 0.14	187 187	— —	6–12 6–12	0 3 7 0 3 7	Whole fruit	0.03 0.04 0.04 0.08 0.06 0.04	0.00 0.00 0.00 0.00 0.01 0.00	0.03 0.04 0.04 0.08 0.07 0.04	94397
90-0133 Huron, CA USA 1990 (Durango)	WP (40 %W/ W)	6	0.071 0.14	— —	— —	10 10	0 0	Whole fruit	0.05 0.07	0.00 0.00	0.05 0.07	94397
94-0126 Noblesville, IN USA 1994 (Iriquois)	WP (40 %W/ W)	6	0.112	196	—	7–12	0	Whole fruit	0.05	0.00	0.05	94444
94-0177 Waller, TX USA 1994 (Magnum 45)	WP (40 %W/ W)	7	0.112	188	—	8–17	0	Whole fruit	0.02	< 0.01	0.03	94444
4839507 Franolino, Ferrara, ITA 1995 (Creso)	EC (125 g ai/L)	6	0.13– 0.14	527– 1058	13–26	10–11	0 3 7	Whole fruit	0.09 0.05 0.04	0.01 0.01 < 0.01	0.10 0.06 0.05	24202 2
4839506 Mirabello, Ferrara, ITA 1995 (Gordon)	EC (125 g ai/L)	6	0.13– 0.15	505– 1013	13–26	10–12	0 3 7	Whole fruit	0.10 0.09 0.05	< 0.01 0.01 0.02	0.11 0.10 0.07	24202 2

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicat ion Interval (days)	DAT	Portion Analyse d	Myclobu tanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobu tanil (mg/kg)	Ref.
AP/3191/HL/1F Montalzat FRA, 1996 (Manta)	EC (240 g ai/L)	6	0.084– 0.11	886– 1452	7.5	10–12	0 3 7	Whole fruit	0.06 0.05 0.02	0.00 0.00 0.00	0.06 0.05 0.02	24183 2
AP/3191/HL/2F Labarthe, FRA 1996 (Cezane)	EC (240 g ai/L)	6	0.053– 0.10	708– 1392	7.5	10–14	0 3 7	Whole fruit	0.04 0.02 0.02	0.00 0.00 0.00	0.04 0.02 0.02	24183 2
AF/345/HL/1S Sanlucar De Barrameda, Provincia de Cadiz, ESP 1997 (Melina)	EC (240 g ai/L)	5	0.088– 0.20	1171– 2673	7.5	11–14	0 3 7	Whole fruit	0.04 0.04 0.04	0.00 0.00 0.00	0.04 0.04 0.04	24181 9
RAS/28/1/G Koutala, Korinthia, North Peloponnese, GRC, 1997 (Galia)	EC (240 g ai/L)	6	0.037– 0.090	499– 1201	7.5	10–11	0 3 7	Whole fruit	0.03 0.04 0.02	0.00 0.00 0.00	0.03 0.04 0.02	24181 9

Squash

A total of 12 trials on squash were conducted in different representative growing areas in Italy, Spain and USA during 1986 through 1998 growing seasons. Results of squash fruits are summarized in Table 64.

Table 64 Supervised trials on squash in various countries

Trial ID/Location Country/Year (variety)	Form	No. of App s	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicatio n Interval (days)	DA T	Portion Analyse d	Myclobutanil (mg/kg)	RH- 9090 (mg/kg)	Total of myclobutani l (mg/kg)	Ref.
86-0239 Camilla, GA USA 1986 (Crookneck)	WP (40% W/W)	4	0.12	–	–	7	0	Whole fruit	0.05	0.00	0.05	94397
86-0270 Celeryville, OH, USA, 1986 (Elite)	WP (40% W/W)	7	0.071	–	–	7	0	Whole fruit	0.02	0.00	0.02	94397
86-0270 Celeryville, OH, USA, 1986 (Elite)	WP (40% W/W)	7	0.14	–	–	7	0	Whole fruit	0.06	< 0.01	0.07	94397
88-0147 Yuma, AZ USA 1988 (Viceroy)	DF (60% W/W)	7 7	0.071 0.14	– –	– –	7–8 7–8	0 0 5 10 15	Whole fruit	0.06 0.16 0.01 0.00 0.00	< 0.01 0.00 0.02 0.01 0.01	0.07 0.16 0.03 0.01 0.01	94397
90-0127 Waller, TX USA 1990 (Multi- pick)	WP (40% W/W)	5	0.071 0.14	374 374	– –	7 7	0 0	Whole fruit	0.02 0.04	0.00 0.00	0.02 0.04	94397
90-0134 Newtown, PA USA	WP (40% W/W)	5 5	0.071 0.14	486 486	– –	7–8 7–8	0 0	Whole fruit	0.01 0.01	0.00 0.00	0.01 0.01	94397

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
1990 (Straightneck)												
94-0091 Waller, TX USA 1994 (Table king)	WP (40% W/W)	6	0.112	192	—	5–10	0	Whole fruit	0.10	0.01	0.11	94444
94-0106 Fresno, CA USA 1994, Zucchini (Ambassador)	WP (40% W/W)	6	0.112	281	—	7	0	Whole fruit	0.01	0.01	0.02	94444
94-0110 Westfield, NY USA 1994 (Multipick)	WP (40% W/W)	6	0.112	274	—	5–8	0	Whole fruit	0.02	0.01	0.03	94444
94-0159 Zellwood, FL USA 1994 (Dixie Hybrid)	WP (40% W/W)	6	0.112	281	—	6–9	0	Whole fruit	0.02	0.01	0.03	94444
4919214 Viladecans ESP, 1992 (Diamant F1)	EC (125 g ai/L)	2	0.088	1000	8.8	7	0	Whole fruit	0.19	—	—	241971
							3		0.03	—	—	
							7		< 0.02	—	—	
							14		< 0.02	—	—	
							0		0.22	—	—	
		1	0.088	1000	8.8	—	0		0.04	—	—	
							3		< 0.02	—	—	
							7		< 0.02	—	—	
							14		< 0.02	—	—	
4048835 Asti AT, Italy 1988 (Marketer Ibrido F1)	EC (125 g ai/L)	3	0.050	1000	5.0	12, 10	7	Whole fruit	0.03	0.01	0.04	242990
			—	—	—	—	—		—	—	—	
		3	0.075	1500	10.0	12, 10	7		0.02	0.00	0.02	
			0.10–0.15	1000–1500								

Watermelon

A total of two trials on watermelon were conducted in different representative growing areas in Italy and USA during 1988 and 1994 growing seasons. Results of watermelon fruits are summarized in Table 65.

Table 65 Supervised trials on watermelon in Italy and USA

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
94-0105 Waller, TX USA 1994 (Jubilee)	WP (40% W/W)	8	0.112	188	—	8–10	0	Whole fruit	0.00	< 0.01	< 0.01	94444
4048870 Roncole, PR	EC (125 g ai/L)	2	0.10	2000	5.0	8	7	Whole fruit	0.06	0.41	0.47	242993
		2	0.20	2000	10	8	7		0.04	0.41	0.45	

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicat ion Interval (days)	DAT	Portion Analyse d	Myclobu tanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobuta nil (mg/kg)	Ref.
ITA, 1988 (Sweet favourite F1)	ai/L)											

Fruiting vegetable other than Cucurbits

Peppers

A total of 26 trials on peppers were conducted in different representative growing areas in France, Italy, Spain and USA during 1996 through 2007 growing seasons. Results of pepper fruits are summarized in Table 66.

Table 66 Supervised trials on pepper in various countries

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicat ion Interval (days)	DAT	Portion Analysed	Myclobu tanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobuta nil (mg/kg)	Ref.
CA88 Coalinga, CA USA 1997 (Excalibur pepper) Bell	WP (40 %W/W)	4	0.13– 0.14	469	–	14	0	Whole fruit	0.03	< 0.02	0.05	94387
TX39 Weslaco, TX USA 1998 (Capistrano pepper) Bell	WP (40 %W/W)	4	0.13	545– 787	–	13–15	1	Whole fruit	0.47	0.17	0.64	94387
CA83 Gilroy, CA USA 1997 (Jalapeno Grande pepper) Non-bell	WP (40 %W/W)	4	0.13– 0.14	523– 546	–	14–15	0	Whole fruit	0.09	0.03	0.12	94387
CA82 Parlier, CA USA 1997 (Copacabana pepper) Non- bell	WP (40 %W/W)	4	0.14– 0.15	492– 517	–	13–15	0 3 7 14	Whole fruit	0.16 0.18 0.15 0.11	0.04 0.05 0.06 0.07	0.20 0.23 0.21 0.18	94387
TX36 Weslaco, TX USA 1997 (Jalapeno pepper) Non- bell	WP (40 %W/W)	4	0.13	562	–	14–15	1	Whole fruit	1.19	0.20	1.39	94387
TX38 Weslaco, TX USA 1997 (Sonora Anaheim pepper) Non-bell	WP (40 %W/W)	4	0.13	562	–	14–15	1	Whole fruit	2.03	0.37	2.40	94387
AP/3205/HL/1 S, El Viso del Alcor, Provincia de Sevilla, ESP 1996 (F2-F3 of	EC (240 g ai/L)	6	0.093 –0.20	1242– 2612	7.5	10–14	0 3 7	Whole fruit	0.27 0.11 0.12	< 0.01 < 0.01 < 0.01	0.28 0.12 0.13	207056

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
Dulce Italiano) Sweet red pepper												
AP/3205/HL/2 S, Alcala del Rio, Provincia de Sevilla, ESP, 1996 (Dulce Italiano Italic) Sweet red pepper	EC (240 g ai/L)	6	0.083–0.18	1103–2350	7.5	10–14	0 3 7	Whole fruit	0.28 0.19 0.16	0.01 0.01 0.02	0.29 0.20 0.18	207056
AP/3205/HL/3 S, Sanlucar de Barrameda, Provincia ESP, 1996 (Zafio) Sweet red pepper	EC (240 g ai/L)	6	0.093–0.15	1240–1938	7.5	11–14	0 3 7	Whole fruit	0.12 0.11 0.11	0.02 0.01 0.02	0.14 0.12 0.13	207056
AP/3205/HL/4 S, Sanlucar de Barrameda, Provincia ESP, 1996 (Dulce Italiano) Sweet red pepper	EC (240 g ai/L)	6	0.060–0.12	800–1580	7.5	10–13	0 3 7	Whole fruit	0.34 0.22 0.21	< 0.01 < 0.01 0.01	0.35 0.23 0.22	207056
AP/3206/HL/1 S, El Viso del Alcor, Provincia de Sevilla, ESP 1996 (F2-F3 of Dulce Italiano) Sweet peppers	EW (200 g ai/L)	6	0.070–0.18	932–2433	7.5	10–14	0 3 7	Whole fruit	0.15 0.13 0.07	< 0.01 < 0.01 < 0.01	0.16 0.14 0.08	207055
AP/3206/HL/2 S Alcala del Rio, Provincia de Sevilla, ESP, 1996 (Dulce Italiano Italic) Sweet peppers	EW (200 g ai/L)	6	0.10–0.19	1375–2540	7.5	10–14	0 3 7	Whole fruit	0.33 0.27 0.17	0.01 0.01 0.01	0.34 0.28 0.18	207055
AP/3206/HL/3 S, Sanlucar de Barrameda, Provincia ESP, 1996 (Zafio) Sweet peppers	EW (200 g ai/L)	6	0.12–0.16	1642–2073	7.5	11–14	0 3 7	Whole fruit	0.16 0.13 0.07	0.02 0.02 0.01	0.18 0.15 0.08	207055
AP/3206/HL/4 S, Sanlucar de Barrameda, Provincia ESP, 1996 (Dulce Italiano) Sweet peppers	EW (200 g ai/L)	6	0.066–0.12	880–1575	7.5	10–13	0 3 7	Whole fruit	0.29 0.31 0.22	0.01 0.01 < 0.01	0.30 0.32 0.23	207055
RAS/26/1/G Lakonia, South Peloponnese, ITA, 1997 (Sonar) Bell pepper	EC (240 g ai/L)	6	0.060–0.11	798–1506	7.5	10–11	0 3 7	Whole fruit	0.13 0.12 0.10	< 0.01 < 0.01 < 0.01	0.14 0.13 0.11	207058
RAS/26/1/I	EC	6	0.063	833–	7.5	13–15	0	Whole	0.10	< 0.01	0.11	207058

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicat ion Interval (days)	DAT	Portion Analysed	Myclob utanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobuta nil (mg/kg)	Ref.
Villanova d'Albenga, Liguria, ITA 1997 (Quadrato giallo) Bell pepper	(240 g ai/L)		-0.23	3019			3 7	fruit	0.19 0.09	0.01 < 0.01	0.20 0.10	
RAS/26/2/1 Codogno, Lombardia, ITA, 1997 (Lamuyo) Bell pepper	EC (240 g ai/L)	6	0.061 -0.17	800- 2280	7.5	13-15	0 3 7	Whole fruit	0.11 0.10 0.06	< 0.01 < 0.01 < 0.01	0.12 0.11 0.07	207058
RAS/26/3/1 Cernusco S. N., Lombardia, ITA, 1997 (Corallo) Bell pepper	EC (240 g ai/L)	6	0.059 -0.12	785- 1526	7.5	12-15	0 3 7	Whole fruit	0.11 0.10 0.07	< 0.01 0.01 0.01	0.12 0.11 0.08	207058
S06W047R Xativa, Valencia, ESP, 2006 (Stilo) Bell peppers	EW (200 g ai/L)	6	0.096 - 0.097	1002- 1014	9.6	7-10	< 0 0 3 7	Whole fruit	0.14 0.73 0.42 0.25	0.01 0.01 0.02 0.03	0.15 0.74 0.44 0.28	200958 8
I06W033R Bitonto, Puglia ITA, 2006 (Fribello) Bell peppers	EW (200 g ai/L)	6	0.091 - 0.098	946- 1018	9.6	7-9	3	Whole fruit	0.28	0.03	0.31	200958 8
S07W051R Navarres, Valencia, ESP, 2007 (4 Cantos) Bell pepper	EW (200 g ai/L)	6	0.092 -0.11	912- 1067	10.1	7-8	< 0 0 3 7	Whole fruit	0.06 0.30 0.11 0.04	0.01 0.01 0.01 0.01	0.07 0.31 0.12 0.05	200132 5
F07W108R Brouilla, Languedoc-Roussillon, FRA, 2007 (Camayo) Bell pepper	EW (200 g ai/L)	6	0.099 -0.10	990- 1018	10.0	7-10	< 0 0 3 7	Whole fruit	0.07 0.09 0.14 0.08	0.02 0.02 0.03 0.02	0.09 0.11 0.17 0.10	200132 5
I07W012R Molfetta, Puglia, ITA 2007 (Cornetto di Molfetta) Bell pepper	EW (200 g ai/L)	6	0.10	1007- 1030	10.1	7-9	< 0 0 3 7	Whole fruit	0.08 0.38 0.17 0.17	0.02 0.02 0.03 0.04	0.10 0.40 0.20 0.21	200132 5
S07W052R Llanera, Valencia, ESP, 2007 (Italiana) Bell pepper	EW (200 g ai/L)	6	0.097 -0.11	963- 1089	10.0	7-8	3	Whole fruit	0.16	0.02	0.18	200132 5
F07W109R Alenya, Languedoc-Roussillon, FRA, 2007 (Mariniez) Bell pepper	EW (200 g ai/L)	6	0.10	992- 1038	10.0	7-8	3	Whole fruit	0.24	0.03	0.27	200132 5
I07W013R Giovinazzo, Puglia, ITA 2007	EW (200 g ai/L)	6	0.10- 0.11	989- 1076	10.1	6-9	3	Whole fruit	0.15	0.04	0.19	200132 5

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
(Frigitello) Bell pepper												

Tomato

A total of 50 trials on tomato were conducted in different representative growing areas in France, Italy, Morocco, Spain and USA during 1986 through 2007 growing seasons. Results of tomato fruits are summarized in Table 67.

Table 67 Supervised trials on tomato in some countries.

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	PHI (days)	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
93-0085 Fresno, CA USA 1993 (Dwarf)	WP (40% W/W)	4	0.070	281	—	13–14	0 5 10 15	Whole fruit	0.01 0.01 0.01 0.01	0.00 0.00 0.00 0.01	0.01 0.01 0.01 0.02	94552
(Cherry) Cherry tomato	WP (40% W/W)	4	0.14	281	—	13–14	0 5 10 15	Whole fruit	0.02 0.02 0.02 0.02	0.00 0.01 0.01 0.01	0.02 0.03 0.03 0.03	
93-0156 Fremont, OH USA	WP (40% W/W)	5	0.070	935.3	—	14–15	0	Whole fruit	0.02	0.00	0.02	94552
1993 (Sweet 100) Cherry tomato	WP (40% W/W)	5	0.14	935.3	—	14–15	0	Whole fruit	0.07	0.02	0.09	
93-0127 Carneys Point NJ, United States, 1993 (Sweet 100) Cherry tomato	WP (40% W/W)	4	0.070	327	—	14–16	0	Whole fruit	0.05	0.01	0.06	94552
(Sweet 100) Cherry tomato	WP (40% W/W)	4	0.14	327	—	14–16	0	Whole fruit	0.10	0.02	0.12	
93-0154 San Luis Rey CA, USA, 1993 (BHN 101) Cherry tomato	WP (40% W/W)	4	0.070	935.3	—	10–14	0	Whole fruit	0.03	0.01	0.04	94552
(BHN 101) Cherry tomato	WP (40% W/W)	4	0.14	935.3	—	10–14	0	Whole fruit	0.08	0.02	0.10	
94-0001 Boynton Beach FL, United States, 1993 (Cherry Grande) Cherry tomato	WP (40% W/W)	4	0.070	926	—	13–14	0 5 10 15	Whole fruit	0.02 0.01 0.01 0.01	0.01 0.01 0.01 0.01	0.03 0.02 0.02 0.02	94552
(Cherry Grande) Cherry tomato	WP (40% W/W)	4	0.14	926	—	13–14	0 5 10 15	Whole fruit	0.03 0.02 0.03 0.01	0.01 0.01 0.02 0.01	0.04 0.03 0.05 0.02	
94-0042 Ft. Pierce, FL USA 1994 (Cherry Grande) Cherry tomato	WP (40% W/W)	4	0.070	744	—	11–13	0	Whole fruit	0.02	0.00	0.02	94552
(Cherry Grande) Cherry tomato	WP (40% W/W)	4	0.14	744	—	11–13	0	Whole fruit	0.04	< 0.01	0.05	
21801050 Huron, CA USA 2001 (Hines 98-	EC (250 g ai/L)	5	0.10	373–386	—	10–11	0	Whole fruit	0.09	< 0.01	0.10	135359 135364

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	PHI (days)	Portion Analys ed	Myclob utanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
88) Tomato												
21801050 Huron, CA USA 2001 (Hines 98- 88) Tomato	WP (40 %W/ W)	5	0.10	372– 385	–	10–11	0	Whole fruit	0.10	< 0.01	0.11	135359 135364
21801049 Lemoore, CA USA 2001 (Asgro 410), Tomato	WP (40 %W/ W)	5	0.10	373– 394	–	10–11	0	Whole fruit	0.07	0.00	0.07	135359 135364
21801046 Tracy, CA USA 2001 (9775) Tomato	WP (40 %W/ W)	5	0.10	232– 239	–	14–15	0	Whole fruit	0.03	0.00	0.03	135359 135364
21801044 Dunnigan, CA USA 2001 (Heinz 9492) Tomato	WP (40 %W/ W)	5	0.10	192– 223	–	13–15	0	Whole fruit	0.07	0.00	0.07	135359 135364
21801047 Los Banos, CA USA 2001 (3155) Tomato	WP (40 %W/ W)	5	0.10	231– 239	–	12–15	0	Whole fruit	0.08	0.00	0.08	135359 135364
21801048 Hickman, CA USA 2001 (9775) Tomato	WP (40 %W/ W)	5	0.10	235– 249	–	13–14	0	Whole fruit	0.07	0.00	0.07	135359 135364
21801043 North Rose, NY, USA, 2001 (Floradade) Tomato	WP (40 %W/ W)	5	0.10	228– 240	–	14–15	0	Whole fruit	0.04	< 0.01	0.05	135359 135364
21801051 San Ardo, CA USA 2001 (Better Boy) Tomato	EC (250 g ai/L)	5	0.10	277– 315		14	0	Whole fruit	0.11	< 0.01	0.12	135359 135364
21801051 San Ardo, CA USA 2001 (Better Boy) Tomato	WP (40 %W/ W)	5	0.10	277– 302		14	0	Whole fruit	0.06	0.00	0.06	135359 135364
21801045 Williams, CA USA 2001 (3155) Tomato	WP (40 %W/ W)	5	0.10	207– 224	–	10–14	0	Whole fruit	0.07	< 0.01	0.08	135359 135364
90-0040 Homestead FL, USA, 1990 (Floradade)	WP (40 %W/ W)	4	0.094	468	–	14	0	Whole fruit	0.22	0.03	0.25	94548
89-0265 Woodland, CA	WP (40	4	0.071	281		10–12	5	Canned Catsup	0.01 0.07	0.01 0.00	0.02 0.07	94609

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	PHI (days)	Portion Analys ed	Myclob utanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
USA 1989 (Harris Moran)	%W/W)							Dry pomace Hot break Juice Paste Paste juice Puree Unwas hed Washe d Wet pomace	0.43 0.00 0.02 0.08 0.04 0.02 0.03 0.02 0.25	0.00 0.00 0.00 0.03 0.04 0.02 0.00 0.00 0.00	0.43 0.00 0.02 0.11 0.08 0.04 0.03 0.02 0.25	
89-0265 Woodland, CA USA 1989 (Harris Moran)	WP (40 %W/W)	4	0.14	281		10–12	5	Canned Catsup Dry pomace Hot break Juice Paste Paste juice Puree Unwas hed Washe d Wet pomace	0.02 0.00 1.03 0.02 0.04 0.17 0.02 0.05 0.04 0.06 0.03	0.03 0.03 0.01 0.01 0.02 0.08 0.06 0.03 0.02 0.02 0.01	0.05 0.03 1.04 0.03 0.06 0.25 0.08 0.06 0.08 0.04	94609
R&H/209/1/I Diano Marina , Liguria, ITA 1996 (Marmade) Tomato	EC (240 g ai/L)	6	0.064–0.11	850–1517	7.5	14	0 3 7	Whole fruit	0.24 0.18 0.05	< 0.01 < 0.01 < 0.01	0.25 0.19 0.06	241830
R&H/209/2/I Villanova D’Albenga, Liguria, ITA 1996 (Arlette) Tomato	EC (240 g ai/L)	6	0.061–0.11	820–1505	7.5	14	0 3 7	Whole fruit	0.24 0.18 0.06	< 0.01 < 0.01 < 0.01	0.25 0.19 0.07	241830
AP/3172/HL/2F , Bressols FRA, 1996 (Laurelia) Tomato	EC (240 g ai/L)	6	0.098–0.12	1308–1600	7.5	10–14	3 3 3 3 7	Juice Preserv e Puree Whole fruit	0.01 < 0.01 0.06 0.05 0.03 0.03	0.00 0.00 0.00 0.00 0.00	0.01 0.00 0.06 0.05 0.03 0.03	241830
AP/3172/HL/1F , Aucamville FRA, 1996 (Delfine) Tomato	EC (240 g ai/L)	6	0.093–0.11	1245–1423	7.5	10–13	3 3 3 3 7	Juice Preserv e Puree Whole fruit	0.02 < 0.01 0.06 0.05 0.02 0.03	0.00 0.00 0.00 0.00 0.00	0.02 < 0.01 0.06 0.05 0.02 0.03	241830
R&H/210/1/I Diano Marina Liguria, ITA 1996	EW (200 g ai/L)	6	0.061–0.11	810–1495	7.5	14	0 3 7	Whole fruit	0.24 0.18 0.06	< 0.01 < 0.01 < 0.01	0.25 0.19 0.07	241829

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	PHI (days)	Portion Analys ed	Myclob utanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
(Marmade) Tomato												
R&H/210/2/1 Villanova D'Albenga ITA, 1996 (Arlette) Tomato	EW (200 g ai/L)	6	0.060–0.11	800–1492	7.5	14	0 3 7	Whole fruit	0.22 0.13 0.08	< 0.01 < 0.01 < 0.01	0.23 0.14 0.09	241829
AP/3173/HL/2/ F, Bressols FRA, 1996 (Laurelia) Tomato	EW (200 g ai/L)	6	0.098–0.11	1300–1533	7.5	10–13	3 3 3 0 3 7	Juice Preserv e Puree Whole fruit	0.01 < 0.01 0.05 0.11 0.05 0.01	0.00 0.00 0.00 < 0.01 0.00 0.00	0.01 < 0.01 0.05 0.12 0.05 0.01	241829
AP/3173/HL/1/ F, Aucamville FRA, 1996 (Delfine) Tomato	EW (200 g ai/L)	6	0.097–0.11	1293–1420	7.5	10–13	3 3 3 0 3 7	Juice Preserv e Puree Whole fruit	0.01 0.01 0.04 0.07 0.04 0.02	0.00 0.00 0.00 0.00 0.00 0.00	0.01 0.01 0.04 0.07 0.04 0.00	241829
F07W067R Brouilla Languedoc-Roussillon	EW (200 g ai/L)	6	0.075–0.077	987–1024	7.5	6–11	< 0 0 3 7	Whole fruit	0.02 0.02 0.02 0.02	0.00 0.00 0.00 0.00	0.02 0.02 0.02 0.02	2001330
FRA, 2007 (Cobra) Tomato	EW (45 g ai/L)	6	0.073–0.075	980–1017	7.5	6–11	< 0 0 3 7	Whole fruit	0.02 0.02 0.01 0.03	< 0.01 0.00 0.00 < 0.01	0.03 0.02 0.01 0.04	
F07W070R Thoree les Pins Pays de Loire FRA, 2007	EW (200 g ai/L)	7	0.069–0.083	980–1093	7.5	6–9	< 0 0 3 7	Whole fruit	0.01 0.04 0.03 0.01	0.00 0.00 0.00 0.00	0.01 0.04 0.03 0.01	2001330
(Roma) Tomato	EW (45 g ai/L)	7	0.068–0.079	973–1077	7.5	6–9	< 0 0 3 7	Whole fruit	0.01 0.06 0.04 0.02	0.00 0.00 0.00 0.00	0.01 0.06 0.04 0.02	
F07W068R Alenya Languedoc-Roussillon	EW (200 g ai/L)	6	0.074–0.079	987–1047	7.5	7–11	< 0 0 3 7	Whole fruit	0.02 0.08 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.03 0.09 0.03 0.02	2001330
FRA, 2007 (Marbonne) Tomato	EW (45 g ai/L)	6	0.071–0.077	963–1043	7.5	7–11	< 0 0 3 7	Whole fruit	0.03 0.07 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.04 0.08 0.03 0.02	
F07W069R Longue, La Ptoterie FRA, 2007	EW (200 g ai/L)	6	0.075–0.080	987–1060	7.5	7–8	< 0 0 3 7	Whole fruit	0.01 0.20 0.09 0.03	0.00 < 0.01 0.01 < 0.01	0.01 0.21 0.10 0.04	2001330
(Topkapi) Tomato	EW (45 g ai/L)	6	0.072–0.076	987–1030	7.5	7–8	< 0 0 3 7	Whole fruit	0.12 0.25 0.13 0.05	0.01 0.01 0.01 0.01	0.13 0.26 0.14 0.06	
S06W041R Alberic Valencia ESP, 2006 (Bon) Tomato	EW (200 g ai/L)	6	0.10–0.11	1473–1531	7.1	7–10	< 0 0 3 7	Whole fruit	0.07 0.10 0.04 0.03	< 0.01 < 0.01 < 0.01 < 0.01	0.08 0.11 0.05 0.04	2009587
F06W062R Brouilla, Languedoc-	EW (200 g ai/L)	6	0.10–0.12	1479–1638	7.0	7–10	3	Whole fruit	0.03	< 0.01	0.04	2009587

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	PHI (days)	Portion Analys ed	Myclob utanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
Roussillon, FRA, 2006 (Cobra)												
S06W039R Teresa de Cofrentes, Valencia ESP, 2006 (Malpica) Tomato	EW (200 g ai/L)	6	0.071–0.077	991–1073	7.2	7–8	3	Whole fruit	0.01	< 0.01	0.02	2009587
I06W029R Emilia Romagna Region (Fe) ITA, 2006 (Asterix) Tomato	EW (200 g ai/L)	6	0.069–0.075	963–1025	7.2	8–9	3	Whole fruit	0.07	0.00	0.07	2009587
F06W061R Brouilla, Languedoc-Roussillon, FRA, 2006 (Cobra) Tomato	EW (200 g ai/L)	6	0.070–0.075	975–1035	7.2	7–8	< 0 0 3 7	Whole fruit	0.01 0.04 0.02 0.01	< 0.01 0.00 0.01 0.00	0.02 0.04 0.03 0.01	2009587
S06W040R Quatretonda Valencia ESP, 2006 (Malpica) Tomato	EW (200 g ai/L)	6	0.071–0.078	990–1081	7.2	7–8	< 0 0 3 7	Whole fruit	0.04 0.11 0.08 0.06	< 0.01 < 0.01 < 0.01 0.01	0.05 0.12 0.09 0.07	2009587
4918529 Premier de Dalt, Barcelone ESP, 1985 (Floritalia)	EC (125 g ai/L)	8 5 3 2	0.24 0.24 0.24 0.24	3000 3000 3000 3000	8.0 8.0 8.0 8.0	3–7 3–7 3 3	0 3 5 8 15 22 8 8 15	Whole fruit	0.06 0.04 0.03 0.02 0.02 0.02 0.06 0.03 0.03	– – – – – – – – – –	– – – – – – – – – –	241921
4219040JNL Mouton FRA, 1990 (Merveille des Marches)	EC (125 g ai/L)	8	0.075	500	15	10–13	0 2 4 7	Whole fruit	0.13 0.12 0.02 0.02	– – – –	– – – –	241953
4219040JL Mouton FRA, 1990 (Merveille des Marches)	EC (125 g ai/L)	8	0.075	500	15	10–13	0 2 4 7	Whole fruit	0.06 0.09 0.09 0.03	– – – –	– – – –	241954
4919290 Chtouka Azemmour MAR, 1992 (H236)	EC (125 g ai/L)	4	0.063	1000	6.3	2–3	0 3 5 7	Whole fruit	0.10 0.04 0.05 0.04	– – – –	– – – –	241972
4919290 Chtouka Azemmour MAR, 1992 (H236)	EC (125 g ai/L)	4	0.094	1500	6.3	2–3	0 3 5 7	Whole fruit	0.04 0.06 < 0.02 < 0.02	– – – –	– – – –	241973

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	PHI (days)	Portion Analys ed	Myclob utanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
4919354 Viladecans, B ESP, 1993 (Precolor)	EC (125 g ai/L)	3	0.13	1000–1562	3.8–13	7, 8	0 3 7 14	Whole fruit	0.16 0.24 0.16 0.04	0.01 0.02 0.03 0.02	0.17 0.26 0.19 0.06	241992
		2	0.13	1000–1562	3.8–13	7	8		0.23	< 0.01	0.24	
9319369 Muchamiel, A ESP, 1993 (Rami)	EC (125 g ai/L)	3	0.13	1077	12	7	0 7 0 4 7 14	Whole fruit	0.03 0.02 0.02 0.03 0.03 0.04	< 0.01 0.02 < 0.01 < 0.01 < 0.01 0.02	0.04 0.04 0.03 0.04 0.04 0.06	241996
4919390 Mohammedia MAR, 1993 (Hamra)	EC (125 g ai/L)	1 3 4	0.094 0.094 0.094	1500 1500 1500	6.3 6.3 6.3	– – –	0 7 0 3 7 14	Whole fruit	0.06 0.09 0.04 0.02 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.07 0.10 0.05 0.03 0.03 0.02	241997
4919391 Mohammedia MAR, 1993 (Daniella)	EC (125 g ai/L)	1 3 4	0.094 0.094 0.094	1500 1500 1500	6.3 6.3 6.3	– – –	0 7 0 3 7 14	Whole fruit	0.03 0.06 0.11 0.05 0.04 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.04 0.07 0.12 0.06 0.05 0.04	241998
4918620 Viladecans, B ESP, 1986 (Carmelo)	EC (125 g ai/L)	5 4	0.02 0.04	250 250	8.0 16	2–8 2–4	0 2 4 8 16 0 2 4 8	Whole fruit	0.10 0.15 0.07 0.10 0.05 0.25 0.30 0.40 0.10	– – – – – – – – –	– – – – – – – – –	242156
4918417 La Roqueta Almeria ESP, 1984 (–)	EC (125 g ai/L)	6	0.08	900	9.0	13–15	6	Whole fruit	0.04	–	–	242213
4918667 Premier de Dalt, Barcelona ESP, 1986 (Carmelo)	EC (125 g ai/L)	5 3	0.08 0.16	670 670	12 24	2–3 3, 4	3 5 7 9 12 5 9 12	Whole fruit	< 0.01 0.01 < 0.01 < 0.01 < 0.01 0.03 0.01 < 0.01	– – – – – – – – –	– – – – – – – – –	241988

Legume vegetables

Snap beans

A total of 10 trials on snap beans were conducted in different representative growing areas in USA during 1989 and 1999 growing seasons. Results of snap beans are summarized in Table 68.

Table 68 Supervised trials on snap beans in the USA

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	DAT	Portion Analyse d	Myclobut anil (mg/kg)	RH-9090 (mg/kg)	Total of myclobu tanil (mg/kg)	Ref.
89-0305/ TN_89/3966 Crossville, TN	WP (40 %W/W)	2	0.067	374	—	7	0 3 7	Whole bean	0.12 0.06 0.05	< 0.02 < 0.02 0.02	0.14 0.08 0.07	94516 263134 263135
USA, 1989 (Eagle)	WP (40 %W/W)	2	0.14	374	—	7	0 3 7	Whole bean	0.15 0.14 0.04	< 0.02 0.03 0.02	0.17 0.17 0.06	110950
	WP (40 %W/W)	2	0.28	374	—	7	0 3 7	Whole bean	0.36 0.30 0.14	< 0.02 < 0.02 0.02	0.38 0.32 0.16	
90-0087/ GA_90/3966 Tifton, GA, USA	WP (40 %W/W)	4	0.12	374	—	7–11	0 3 7	Whole bean	0.22 0.11 0.13	< 0.02 < 0.02 0.02	0.24 0.13 0.15	94516 263134 263135
1990 (Bountiful)	WP (40 %W/W)	4	0.24	374	—	7–11	0 3 7	Whole bean	0.61 0.48 0.18	< 0.02 0.02 0.04	0.63 0.50 0.22	110950
90-0203 NY_90/3966 Geneva, NY	WP (40 %W/W)	4	0.12	920	—	7–8	0 3 7	Whole bean	0.47 0.29 0.16	0.03 0.05 0.02	0.50 0.34 0.18	94516 263134 263135
USA, 1990 (Labrador)	WP (40 %W/W)	4	0.24	920	—	7–8	0 3 7	Whole bean	1.21 0.52 0.30	0.06 0.07 0.05	1.27 0.59 0.35	110950
90-0231 WI_90/3966 Madison, WI	WP (40 %W/W)	4	0.125	208	—	7–9	0 3 7	Whole bean	0.14 0.10 0.09	< 0.02 0.02 0.03	0.16 0.12 0.12	94516 263134 263135
USA, 1990 (root rot res line)	WP (40 %W/W)	4	0.25	208	—	7–9	0 3 7	Whole bean	0.29 0.15 0.15	0.02 0.02 0.04	0.31 0.17 0.19	110950
91-0083 TN_91/3966 Crossville, TN,	WP (40 %W/W)	4	0.125	187	—	7–8	0 3 7	Whole bean	0.20 0.09 0.07	< 0.02 < 0.02 0.02	0.22 0.11 0.09	94516 263134 263135
USA, 1991 (Tenderlake)	WP (40 %W/W)	4	0.25	187	—	7–8	0 3 7	Whole bean	0.23 0.14 0.09	< 0.02 0.03 0.03	0.25 0.17 0.12	110950
91-0085 MI_91/3966 East Lansing, MI	WP (40 %W/W)	4	0.125			8–10	0 3 7	Whole bean	0.04 0.03 0.03	< 0.02 < 0.02 < 0.02	0.06 0.05 0.05	94516 263134 263135
USA, 1991 (–)	WP (40 %W/W)	4	0.25			8–10	0 3 7	Whole bean	0.07 0.05 0.04	< 0.02 < 0.02 < 0.02	0.09 0.07 0.06	110950
91-0019 OR_91/3966 Hillsboro, OR,	WP (40 %W/W)	4	0.125	—	—	5–7	0 3 7	Whole bean	0.18 0.19 0.12	< 0.02 0.02 < 0.02	0.20 0.21 0.14	94516 263134 263135
USA, 1991 (–)	WP (40 %W/W)	4	0.25	—	—	5–7	0 3 7	Whole bean	0.33 0.23 0.15	< 0.02 < 0.02 0.02	0.35 0.25 0.17	110950

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	DAT	Portion Analyse d	Myclobut anil (mg/kg)	RH-9090 (mg/kg)	Total of myclobu tanil (mg/kg)	Ref.
92-0015 FL_92/3966 Gainesville, FL, USA, 1992 (Podsquad)	WP (40 %W/W)	4	0.125	234	—	7–11	0 3 8	Whole bean	0.37 0.26 0.12	0.02 0.02 0.02	0.39 0.28 0.14	94516 263134 263135
	WP (40 %W/W)	4	0.25	234	—	7–11	0 3 8	Whole bean	0.57 0.43 0.31	0.02 0.02 0.03	0.59 0.45 0.34	110950
99-WI08, A3966 Arlington, WI, USA, 1999 (Hystyle)	WP (40 %W/W)	4	0.12– 0.13	313– 321	—	7–8	0	Whole pods	0.09	< 0.02	0.11	104615
99-CA42, A3966 Salinas, CA, USA 1999 (Envoy)	WP (40 %W/W)	4	0.12– 0.13	519– 755	—	7–8	0	Whole pods	0.30	< 0.02	0.32	104615

Pulses

Soya beans

A total of 33 trials on soya bean were conducted in different representative growing areas in Argentina, Brazil and USA during 1999 and 2006 growing seasons. Results for soya bean seed are summarized in Table 69.

Table 69 Supervised trials on soya bean in Argentina, Brazil and USA

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	DAT	Portion Analyse d	Myclobut anil (mg/kg)	RH-9090 (mg/kg)	Total of myclobu tanil (mg/kg)	Ref.
NE York, NE, USA, 2004 (Dyna-Gro 32M32 RR)	EW (200 g ai/L)	2	0.14	186	—	15	14	Seed	0.01	0.00	0.01	220163 224527 221174
IL2 Wyoming, IL USA 2004 (Asgrow AG3302)	EW (200 g ai/L)	2	0.14	151– 153	—	14	14	Seed	0.04	0.00	0.04	220163 224527 221174
OH1 Newholland, OH USA 2004 (SC 388)	EW (200 g ai/L)	2	0.14	149– 150	—	10	14	Seed	0.03	0.00	0.03	220163 224527 221174
OH2 Atlanta, OH USA 2004 (SC 9373)	EW (200 g ai/L)	2	0.14	151– 152	—	10	14	Seed	0.02	0.00	0.02	220163 224527 221174
IA1 Richland, IA USA 2004 (Pioneer 93B87)	EW (200 g ai/L)	2	0.14	149– 173	—	14	14	Seed	0.02	0.00	0.02	220163 224527 221174
MO Kirksville, MO USA 2004 (Pioneer DG 3390)	EW (200 g ai/L)	2	0.14	167– 170	—	15	14	Seed	0.03	0.00	0.03	220163 224527 221174
IA2 Bagley, IA USA 2004 (Pioneer 92M70)	EW (200 g ai/L)	2	0.14	132– 133	—	12	14	Seed	0.01	0.00	0.01	220163 224527 221174

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicat ion Interval (days)	DAT	Portion Analyse d	Myclobut anil (mg/kg)	RH-9090 (mg/kg)	Total of myclobu tanil (mg/kg)	Ref.
IA3 Cooper, IA USA 2004 (Pioneer 92M30)	EW (200 g ai/L)	2	0.14– 0.15	136– 138	–	12	14	Seed	0.03	0.00	0.03	220163 224527 221174
WI Arkansaw, WI USA 2004 (Cropplan RT 1447)	EW (200 g ai/L)	2	0.14	185– 188	–	12	14	Seed	< 0.01	0.00	< 0.01	220163 224527 221174
ND2 Northwood, ND USA 2004 (Roughrider)	EW (200 g ai/L)	2	0.14	169– 171	–	14	14	Seed	< 0.01	0.00	< 0.01	220163 224527 221174
IL1 Carlyle, IL USA 2004 (BT 383 CR)	EW (200 g ai/L)	2	0.14– 0.15	163– 169	–	15	13	Seed	< 0.01	0.00	< 0.01	220163 224527
VA Bumpass, VA USA 2004 (Pioneer 9492 RR)	EW (200 g ai/L)	2	0.14	162– 163	–	10	14	Seed	0.15	0.02	0.17	220163 224527 221174
LA Washington, LA USA 2004 (DP 5915 RR)	EW (200 g ai/L)	2	0.14	163– 166	–	14	14	Seed	< 0.01	0.00	< 0.01	220163 224527 221174
ND1 Oakes, ND USA 2004 (DeKalb 814)	EW (200 g ai/L)	2	0.14	154– 156	–	11	14	Seed	0.00	0.00	0.00	220163 224527 221174
SD Frankfort, SD USA 2004 (Cenex 8031)	EW (200 g ai/L)	2	0.14	154– 155	–	11	14	Seed	0.00	0.00	0.00	220163 224527 221174
AR2 Tuckerman, AR USA 2004 (Garst 572 STS)	EW (200 g ai/L)	2	0.14	167– 168	–	14	14	Seed	0.00	0.00	0.00	220163 224527 221174
MN Paynesville, MN USA 2004 (Cenex 8031)	EW (200 g ai/L)	2	0.14	156	–	11	14	Seed	0.00	0.00	0.00	220163 224527 221174
IN Danville, IN USA 2004 (BT 383 CR)	EW (200 g ai/L)	2	0.14	173	–	14	14	Seed	< 0.01	0.00	< 0.01	220163 224527 221174
GA1 Chula, GA USA 2004 (DeKalb 0212647)	EW (200 g ai/L)	2	0.14– 0.15	145– 147	–	14	14	Seed	< 0.01	0.00	0.00	220163 224527 221174
GA2 Sycamore, GA USA 2004 (DeKalb 0212647)	EW (200 g ai/L)	2	0.14	144– 145	–	14	14	Seed	0.01	0.00	0.01	220163 224527 221174
AR1 Newport, AR USA 2004 (Genesisi C 444 NRR)	EW (200 g ai/L)	2	0.71 0.70	139– 141	–	14	14 14 14	Meal Oil, refined Seed	0.03 0.15 0.07	0.01 0.00 < 0.01	0.04 0.15 0.08	220163 224527 221174
ZONE 2 COMP	EW	2	0.14			14	14	Seed	0.04	0.01	0.05	220163

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicat ion Interval (days)	DAT	Portion Analyse d	Myclobut anil (mg/kg)	RH-9090 (mg/kg)	Total of myclobu tanil (mg/kg)	Ref.
40053 USA	(200 g ai/L)											224527
ZONE 4 COMP 40053 USA	EW (200 g ai/L)	2	0.14			14	14	Seed	< 0.01	0.00	< 0.01	220163 224527
ZONE 5 COMP 40053 USA	EW (200 g ai/L)	2	0.14			14	14	Seed	0.02	0.00	0.02	220163 224527
33199016 BRA 1999 (CAC-1)	EC (250 g ai/L)	1	0.125	–	–	NA	24	Seed	< 0.02	–	–	207033
	EC (250 g ai/L)	1	0.25	–	–	NA	24	Seed	< 0.02	–	–	
33199017 BRA 1999 (CAC-1)	EC (250 g ai/L)	1	0.125	–	–	NA	24	Seed	< 0.02	–	–	207033
	EC (250 g ai/L)	1	0.25	–	–	NA	24	Seed	< 0.02	–	–	
GHB-P 803 BRA 2002 (Cometa)	EC (250 g ai/L)	1	0.125	–	–	NA	24	Seed	< 0.01	–	–	102063
	EC (250 g ai/L)	1	0.25	–	–	NA	24	Seed	0.03	–	–	
050077-01 BRA 2006 (BRS-133)	EC (250 g ai/L)	2	0.125	–	–	21	24	Seed	< 0.01	–	–	240111
	EC (250 g ai/L)	2	0.25	–	–	21	24	Seed	0.03	–	–	
050077-02 BRA 2006 (BRS MG 68-Vencedora)	EC (250 g ai/L)	2	0.125	–	–	21	24	Seed	< 0.01	–	–	240108
	EC (250 g ai/L)	2	0.25	–	–	21	24	Seed	0.02	–	–	
050077-03 BRA 2006 (Silvania RR)	EC (250 g ai/L)	2	0.125	–	–	21	14 24 28 35 0	Seed Seed Seed Seed Whole pods	0.03 0.01 0.02 0.02 0.19	– – – – –	– – – – –	240109
	EC (250 g ai/L)	2	0.25	–	–	21	14 24 28 35 0	Seed Seed Seed Seed Whole pods	0.03 0.03 0.03 0.10 0.36	– – – – –	– – – – –	
050077 BRA 2006 (Codetec- 213)	EC (250 g ai/L)	2	0.125	–	–	22	14 24 28 35 0	Seed Seed Seed Seed Whole pods	0.02 0.03 < 0.01 < 0.01 0.68	– – – – –	– – – – –	240110
	EC (250 g ai/L)	2	0.25	–	–	22	14 24 28 35	Seed Seed Seed Seed	0.02 0.03 0.02 0.03	– – – –	– – – –	

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicat ion Interval (days)	DAT	Portion Analyse d	Myclobut anil (mg/kg)	RH-9090 (mg/kg)	Total of myclobu tanil (mg/kg)	Ref.
							0	Whole pods	1.12	–	–	
TRIAL02 Pdo de pergamino,	SC (261 g ai/L)	1	0.10	170		NA	24	Seed	< 0.01	–	–	136923
Buenos Aires, ARG, 2003 (Asgrow 6445)	SC (261 g ai/L)	1	0.21	170		NA	24	Seed	< 0.01	–	–	
TRIAL01 Guaiguaychu, Entre Rios,	SC (261 g ai/L)	1	0.10	170		NA	51	Seed	< 0.01	–	–	136923
ARG 2003 (Asgrow 3901)	SC (261 g ai/L)	1	0.21	170		NA	51	Seed	< 0.01	–	–	

Dried herbs

Hops

A total of 19 trials on hops were conducted in different representative growing areas in Germany, the UK and the USA during 1997 through 2007 growing seasons. Results of hops are summarized in Table 70.

Table 70 Supervised trials on hops in Germany, the UK and the USA

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicat ion Interval (days)	DAT	Portion Analyse d	Myclob utanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
98-ID18 Parma ID, USA, 1998 (Galena hops)	WP (40 %W/W)	9	0.13– 0.25	0.072– 0.103	–	8–12	0 6 14	Dried hops	8.50 7.75 4.49	< 0.50 < 0.50 < 0.33	9.00 8.25 4.82	94386
98-OR24 Hubbard, OR USA 1998 (Nugget hops)	WP (40 %W/W)	9	0.12– 0.25	0.069– 0.10	–	9–12	12	Dried hops	2.64	< 0.33	2.97	94386
98-WA44 Prosser, WA USA 1998 (Nugget hops)	WP (40 %W/W)	9	0.25– 0.42	0.094– 0.23	–	7–10	14	Dried hops	1.34	< 0.15	1.49	94386
1991-N, ADAS, Rosemaund W. Midlands UK, 1991 (N. Brewer)	EW (60 g ai /L)	6	0.059– 0.10	1300– 2250	4.5	3–22	7	Dried hops	0.50	< 0.20	0.70	24201 2
1991-Y ADAS, Rosemaund W. Midlands UK, 1991 (Yeoman)	EW (60 g ai /L)	6	0.059– 0.10	1300– 2250	4.5	3–22	13	Dried hops	0.30	< 0.20	0.50	24201 2
1992-C ADAS, Rosemaund W. Midlands UK, 1992	EW (60 g ai /L)	6	0.059– 0.10	1300– 2250	4.5	4–14	9	Dried hops	0.27	< 0.20	0.47	24201 2

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicat ion Interval (days)	DAT	Portion Analyse d	Myclob utanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
(Challenger)												
1992-Z ADAS, Rosemaund W. Midlands UK, 1992 (Zenith)	EW (60 g ai /L)	6	0.059– 0.10	1300– 2300	4.5	7–14	8	Dried hops	1.20	< 0.20	1.40	24201 2
1993-Y Perton Hereford, UK 1993 (Yeoman)	EW (60 g ai /L)	6	0.059– 0.090	1300– 2000	4.5	5–14	4	Dried hops	1.60	< 0.20	1.80	24201 2
1993-ND Brierley Leominster UK, 1993 (North Down)	EW (60 g ai /L)	6	0.059– 0.10	1300– 2300	4.5	5–21	3	Dried hops	1.80	< 0.20	2.00	24201 2
R&H/207/1/G Bayern GER 1996 (Hersbrucker)	EW (200 g ai/L)	4	0.18– 0.30	2976– 5060	6	13–14	14 14 0 7 14 14 14 14	Beer Dried hops Green hops Spent hops Trub Yeast	< 0.01 1.02 0.27 0.15 0.28 0.09 < 0.02 < 0.02	< 0.01 < 0.50 0.07 0.04 0.09 < 0.02 < 0.02 < 0.02	< 0.02 1.52 0.34 0.19 0.37 0.11 < 0.04 < 0.04	94412
R&H/207/2/G Bayern GER 1996 (Brewers Gold)	EW (200 g ai/L)	4	0.19– 0.30	3099– 5052	6	11–14	14 0 7 14	Dried hops Green hops	0.53 0.22 0.46 0.22	< 0.15 0.02 0.10 0.03	0.68 0.24 0.56 0.25	94412
R&H/207/3/G Bayern GER 1996 (Hersbrucker)	EW (200 g ai/L)	4	0.20– 0.31	2202– 3423	9	11–14	14 0 7 14	Dried hops Green hops	1.54 0.63 0.49 0.41	< 0.50 0.14 0.04 0.07	2.04 0.77 0.53 0.48	94412
R&H/207/4/G Bayern GER 1996 (Brewers Gold)	EW (200 g ai/L)	4	0.19– 0.31	2057– 3385	9	11–14	14 0 7 14	Dried hops Green hops	1.14 0.97 0.17 0.35	< 0.50 0.13 < 0.02 0.04	1.64 1.10 0.19 0.39	94412
RAS/20/3/G Bayern GER 1997 (Selekt)	EW (200 g ai/L)	4	0.18– 0.30	2000– 3300	9	11–13	14 0 7 14	Dried hops Green hops	0.63 2.41 1.00 0.16	< 0.50 0.10 0.16 0.03	1.13 2.51 1.16 0.19	94441
RAS/20/4/G Bayern GER 1997 (Tradition)	EW (200 g ai/L)	4	0.19– 0.30	2050– 3300	9	11–13	14 14 0 7 14 14 14 14	Beer Dried hops Green hops Spent hops Trub Yeast	< 0.01 0.73 0.82 0.21 0.33 0.04 < 0.02 < 0.005	< 0.01 < 0.50 0.05 0.06 0.09 < 0.005 < 0.02 < 0.02	< 0.02 1.23 0.87 0.27 0.42 0.05 < 0.04 < 0.03	94441
RAS/20/1/G Bayern GER 1997 (Hersbrucker)	EW (200 g ai/L)	4	0.18– 0.30	2975– 5025	6	11–13	14 0 7 14	Dried hops Green hops	1.06 2.42 0.25 0.35	< 0.50 0.23 0.07 0.16	1.56 2.65 0.32 0.51	94441

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica- tion Interval (days)	DAT	Portion Analyse d	Myclob- utanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
RAS/20/2/G Bayern GER 1997 (Magnum)	EW (200 g ai/L)	4	0.18– 0.30	2950– 5000	6	11–13	14 0 7 14	Dried hops Green hops	< 0.50 2.63 0.89 0.49	< 0.50 0.13 0.11 0.05	<1.00 2.76 1.00 0.54	94441
G07W386R Wolnzach Bavaria GER 2007 (Perle)	EW (200 g ai/L)	6	0.28– 0.32	4724– 5242	6.03	7	< 0 0 3 7 14 21 28 < 0 0 3 7 14 21 28	Dried hops Green hops	4.80 11.0 6.10 4.90 3.50 0.97 1.30 1.30 4.60 2.20 1.80 1.40 0.62 0.65	2.40 2.90 1.00 2.00 2.10 0.92 0.98 0.39 0.52 0.30 0.47 0.62 0.52 0.62	7.20 13.9 7.10 6.90 5.60 1.89 2.28 1.69 5.12 2.50 2.27 2.02 1.14 1.27	20013 22
G07W387R Tettngang Baden– Wurttemberg GER 2007 (Spalter)	EW (200 g ai/L)	6	0.30– 0.32	4935– 5227	6.03	6–9	14 14	Dried hops Green hops	1.80 0.98	1.40 0.80	3.20 1.78	20013 22

Legume animal feed

Soybean

A total of 24 trials on soybean were conducted in different representative growing areas in USA during 2004 growing seasons. Results of soybean forage and hay are summarized in Table 71.

Table 71 Supervised Trials on Soybean in USA

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica- tion Interval (days)	DAT	Portion Analyse d	Myclobut anil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
NE York, NE, USA, 2004 (Dyna-Gro 32M32 RR)	EW (200 g ai/L)	2	0.14	186– 187	–	14	14 14	Forage Hay	0.37 1.43	0.04 0.35	0.41 1.78	220163 224527 221174
IL2 Wyoming, IL USA 2004 (Asgrow AG3302)	EW (200 g ai/L)	2	0.14	154– 166	–	17	14 14	Forage Hay	3.01 7.31	0.23 0.78	3.24 8.09	220163 224527 221174
OH1 Newholland, OH USA 2004 (SC 388)	EW (200 g ai/L)	2	0.14	150– 151	–	14	13 13	Forage Hay	2.80 8.73	0.06 0.59	2.86 9.32	220163 224527 221174
OH2 Atlanta, OH USA 2004 (SC 9373)	EW (200 g ai/L)	2	0.14	150– 153	–	14	13 13	Forage Hay	1.30 3.20	0.21 0.86	1.51 4.06	220163 224527 221174
IA1 Richland, IA USA 2004 (Pioneer 93B87)	EW (200 g ai/L)	2	0.14	157– 180	–	13	14 14	Forage Hay	1.97 4.40	0.05 0.37	2.02 4.77	220163 224527 221174

Trial ID/Location Country/Year (variety)	Form	No. of App s	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	DAT	Portion Analyse d	Myclobut anil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
MO Kirkville, MO USA 2004 (Pioneer DG 3390)	EW (200 g ai/L)	2	0.14	157– 180	–	14	14 14	Forage Hay	1.77 7.75	0.11 1.01	1.88 8.76	220163 224527 221174
IA2 Bagley, IA USA 2004 (Pioneer 92M70)	EW (200 g ai/L)	2	0.14– 0.15	150– 175	–	14	0 7 14 21 28 14	Forage Forage Forage Forage Forage Hay	9.77 4.28 2.49 1.68 1.01 3.85	0.02 0.05 0.05 0.05 0.04 0.28	9.79 4.33 2.54 1.73 1.05 4.13	220163 224527 221174
IA3 Cooper, IA USA 2004 (Pioneer 92M30)	EW (200 g ai/L)	2	0.14– 0.15	152– 180	–	14	14 14	Forage Hay	1.97 3.68	0.03 0.40	2.00 4.08	220163 224527 221174
WI Arkansaw, WI USA 2004 (Cropplan RT 1447)	EW (200 g ai/L)	2	0.14	186– 188	–	14	14 14	Forage Hay	1.17 3.96	0.03 0.24	1.20 4.20	220163 224527 221174
ND2 Northwood, ND USA 2004 (Roughrider)	EW (200 g ai/L)	2	0.14	168– 169	–	11	14 14	Forage Hay	0.73 1.82	0.06 0.78	0.79 2.60	220163 224527 221174
IL1 Carlyle, IL USA 2004 (BT 383 CR)	EW (200 g ai/L)	2	0.12– 0.13	159– 168	–	14	14 24	Forage Hay	1.66 4.08	0.04 0.31	1.70 4.39	220163 224527
VA Bumpass, VA USA 2004 (Pioneer 9492 RR)	EW (200 g ai/L)	2	0.14	161	–	14	14 14	Forage Hay	1.28 3.38	0.18 1.46	1.46 4.84	220163 224527 221174
LA Washington, LA USA 2004 (DP 5915 RR)	EW (200 g ai/L)	2	0.14	160	–	14	14 14	Forage Hay	1.73 3.72	0.33 0.86	2.06 4.58	220163 224527 221174
ND1 Oakes, ND USA 2004 (DeKalb 814)	EW (200 g ai/L)	2	0.14	154– 155	–	11	14 14	Forage Hay	0.18 0.00	0.00 0.00	0.18 0.00	220163 224527 221174
SD Frankfort, SD USA 2004 (Cenex 8031)	EW (200 g ai/L)	2	0.14	154– 155	–	11	14 14	Forage Hay	0.05 0.00	0.00 0.00	0.05 0.00	220163 224527 221174
AR2 Tuckerman, AR USA 2004 (Garst 572 STS)	EW (200 g ai/L)	2	0.14	168	–	12	14 14	Forage Hay	2.62 8.63	0.46 2.31	3.08 10.9	220163 224527 221174
MN Paynesville, MN USA 2004 (Cenex 8031)	EW (200 g ai/L)	2	0.14	154– 156	–	12	14 14	Forage Hay	0.09 0.00	0.00 0.00	0.09 0.00	220163 224527 221174
IN Danville, IN USA 2004 (BT 383 CR)	EW (200 g ai/L)	2	0.13– 0.14	187– 202	–	10	14 14	Forage Hay	0.88 1.58	0.03 0.12	0.91 1.70	220163 224527 221174
GA1 Chula, GA USA 2004 (DeKalb 0212647)	EW (200 g ai/L)	2	0.14	144– 155	–	14	14 14	Forage Hay	1.42 3.83	0.26 1.60	1.68 5.43	220163 224527 221174
GA2 Sycamore, GA USA 2004	EW (200 g ai/L)	2	0.14	143– 157	–	14	14 14	Forage Hay	1.86 6.65	0.11 1.02	1.97 7.67	220163 224527 221174

Trial ID/Location Country/Year (variety)	Form	No. of App s	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	DAT	Portion Analyse d	Myclobut anil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
(DeKalb 0212647)												
AR1 Newport, AR USA 2004 (Genesisi C 444 NRR)	EW (200 g ai/L)	2	0.71 0.70	139– 141	–	14	14	Aspirate d grain fraction s Hulls	5.03 0.08	0.21 0.01	5.24 0.09	220163 224527 221174
	EW (200 g ai/L)	2	0.14	186	–	14	0 7 14 21 28 14	Forage Forage Forage Forage Forage Hay	16.1 4.03 2.06 1.30 0.67 7.22	0.07 0.11 0.11 0.07 0.06 0.73	16.2 4.14 2.17 1.37 0.73 7.95	
ZONE 2 COMP 40053 USA	EW (200 g ai/L)	2	0.14			14	14 14	Forage Hay	1.49 4.56	–	–	220163 224527
ZONE 4 COMP 40053 USA	EW (200 g ai/L)	2	0.14			14	14 14	Forage Hay	1.85 5.98	–	–	220163 224527
ZONE 5 COMP 40053 USA	EW (200 g ai/L)	2	0.14			14	14 14	Forage Hay	1.12 3.24	–	–	220163 224527

FATE OF RESIDUES IN STORAGE AND PROCESSING

Information and Data from Residues in Processed Commodities

The Meeting received information on the fate of myclobutanil residues during aqueous hydrolysis and also received the processing studies on apples, grapes, tomatoes, soybeans and hops.

The hydrolytic stability of myclobutanil was investigated in aqueous buffer solutions at pH 4, 7 and 9 at 50 °C over a 5 day period (94798). No degradation of myclobutanil was observed under these conditions.

The two supervised trials on apple (var. Golden delicious) were conducted in Germany to provide bulk samples for processing studies. Apple trees were treated with 12 foliar applications of a formulation of myclobutanil (containing 60 g ai/kg). The first two applications were made at the rate of 0.006 kg as/hL (0.036–0.054 kg ai/ha) when trees were near or at-flowering growth stage. The last ten applications were made at 0.018 kg as/hL (0.072–0.090 kg ai/ha) per application with the last application made at fruit maturity. Bulk samples of treated apples for the processing studies were harvested at 14 days after the last application and processed into juice and wet pomace. The analytical results are listed in Table 72.

Table 72 Residue levels in processed apple products

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applica tion Interval (days)	DAT)	Portion Analyse d	Myclobuta nil (mg/kg)	RH-9090 (mg/kg)	Total of myclobut anil (mg/kg)	Ref.
DEU86F21221 Pfeddersheim GER, 1986 (Golden Delicious)	WP (60 g ai/ L)	12	0.090	500	18	8–10	0 7 14 21 28 14 14	Whole fruit Juice Wet pomace	0.21 0.18 0.15 0.16 0.13 0.02 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.22 0.19 0.16 0.17 0.14 0.03 0.09	24205 1

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
DEU86F21241 Mainz-Drais GER, 1986 (Golden Delicious)	WP (60 g ai/L)	12	0.090	500	18	8–10	0 7 14 21 28 14 14	Whole fruit Juice Wet pomace	0.23 0.34 0.35 0.28 0.19 0.04 0.23	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.24 0.35 0.36 0.29 0.20 0.05 0.24	242051

Apple trees (Granny Smith variety) were treated with four foliar applications of a formulation of myclobutanil (EW, containing 200 g ai/L) at either 1× or 5× recommended rate. The 5× treatment rate was included in order to improve the ability to quantify residue levels in certain processed fractions of apples, such as apple juice. In the 1× treatment myclobutanil was applied at the rate of 0.006 kg ai/hL (0.094–0.097 kg ai/ha) per application, while in the 5× treatment myclobutanil was applied at the rate of 0.030 kg ai/hL (0.447–0.478 kg ai/ha) per application. The first of the four treatment applications began near the end of fruit enlargement (BBCH 78) with subsequent applications continuing on approximately 10 day intervals through ripe fruit (BBCH 85). Bulk samples of treated apples (both 1× and 5× application rates) were harvested at 14 days after the last application for processing into processed apple products (pasteurized apple juice, wet pomace and apple puree). The analytical results are listed in Table 73.

Table 73 Residue levels in processed apple products

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
AF/8164/DE4 St Remy La Varenne FRA, 2004 (Granny Smith)	EW (200 g ai/L)	4	0.094–0.097	1560–1620	6	7–15	14 14 14 14 14 14 14 14 14 14 14	Whole fruit Whole fruit (processing) Cooked fruit washed fruit Juice Peeland seed Pomace, dry Pomace, wet Puree Raw juice Washing water	0.11 0.08 0.04 0.08 < 0.01 0.10 0.99 0.23 0.02 0.01 0.02	< 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.06 0.01 < 0.01 < 0.01 < 0.01	0.12 0.09 0.05 0.09 < 0.02 0.11 1.05 0.24 0.03 0.02 0.03	101405
AF/8164/DE4 St Remy La Varenne FRA, 2004 (Granny Smith)	EW (200 g ai/L)	4	0.45–0.48	1490–1593	30	7–15	14 14 14 14	Whole fruit Whole fruit (processing) Washed fruit Cooked fruit	0.65 0.51 0.52 0.28	0.03 0.05 0.03 0.03	0.68 0.56 0.55 0.31	101405

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
							14	Apple juice	0.06	0.02	0.08	
							14	Peeland seed	0.59	0.04	0.63	
							14	Pomace, dry	6.00	0.25	6.25	
							14	Pomace, wet	1.57	0.05	1.62	
							14	Puree	0.13	0.02	0.15	
							14	Raw juice	0.06	0.02	0.08	
							14	Washing water	0.08	< 0.01	0.09	

Two supervised trials on wine grapes (white and red varieties) were conducted in Northern France. Grape vines were treated with eight foliar applications of a formulation of myclobutanil (EW, 200 g ai/L) at the rate of 0.003 kg ai/hL (0.01–0.05 kg ai/ha) per application. The first application was made at pre-bloom and continued on a 10 to 14-day interval until the grapes were harvested. Bulk samples of treated grapes for the processing studies were harvested at 14 days after the last application and transported to a processing facility in Germany. Samples of whole grapes, grape must (juice), young and mature wine were analysed for myclobutanil using an analytical method with limits of quantitation and detection of 0.01 mg/kg and 0.0025 mg/kg, respectively. Summary of data is shown in Table 74.

Table 74 Residue levels in processed grape products.

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analysed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
RAS/18/4/F Wissembourg Elsaß, FRA 1997 (Spatburgunder) Wine grapes	EW (200 g ai/L)	8	0.012–0.048	394–1609	3	10–15	0	Whole fruit	0.71	0.03	0.74	110206
							7		0.51	0.03	0.54	
							14		0.51	0.03	0.54	
							21		0.47	0.03	0.50	
							28		0.38	0.03	0.41	
							14	Juice	0.08	0.01	0.09	
							14	Wine after half year	0.05	0.02	0.07	
							14	Wine at bottling	0.04	0.01	0.05	
RAS/18/3/F Wissembourg Elsaß, FRA 1997 (Muller-Thurgau) Wine grapes	EW (200 g ai/L)	8	0.009–0.030	306–1006	3	10–15	0	Whole fruit	0.39	< 0.01	0.40	110206
							7		0.31	0.01	0.32	
							14		0.25	< 0.01	0.26	
							21		0.26	0.01	0.27	
							28		0.25	0.01	0.26	
							14	Juice	0.04	< 0.01	0.05	
							14	Wine after half year	0.01	< 0.01	0.02	
							14	Wine at bottling	0.02	< 0.01	0.03	

Two supervised trials on wine grapes (white and red varieties) were conducted in Germany to provide bulk samples for processing studies. Grapevines were treated with eight foliar applications of a formulation of myclobutanil (EW, 200 g ai/L). White grape was treated at the rate of 0.009 kg ai/hL (0.028–0.048 kg ai/ha) per application while red grapes was treated at 0.003 kg ai/hL (0.012–0.048 kg ai/ha) per application. The first application was made at pre-bloom and continued on a 10

to 14-day interval until the grapes were harvested. Bulk samples of control and treated grapes for the processing studies were harvested 14 days after the last application and transported to a processing facility in Germany. Samples of whole grapes, grape must (juice), young wine (8–10 weeks aging) and mature wine (5–6 months aging) were analysed for myclobutanil using an analytical method with limits of quantitation and detection of 0.01 mg/kg and 0.0025 mg/kg, respectively. Summary of data is shown in Table 75.

Table 75 Residue levels in processed grape products

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
R&H/203/3/G D-67150 Niederkirchen GER 1996 (Portugieser) Wine grape	EW (200 g ai/L)	8	0.012 – 0.048	406–1600	3	10–16	0	Grapes	0.46	< 0.01	0.47	94403 241765
							14		0.34	0.01	0.35	
							28		0.33	0.02	0.35	
							14	Juice (must)	0.07	< 0.01	0.08	
							14	Mature wine	0.04	< 0.01	0.05	
							14	Young Wine	0.04	< 0.01	0.05	
R&H/203/2/G D-67150 Niederkirchen GER 1996 (Muller–Thurgau) Wine grape	EW (200 g ai/L)	8	0.028 – 0.048	312–531	9	10–16	0	Grapes	0.45	< 0.01	0.46	94403 241765
							14		0.41	0.02	0.43	
							28		0.35	0.01	0.36	
							14	Juice (must)	0.09	0.00	0.09	
							14	Mature wine	0.07	< 0.01	0.08	
							14	Young Wine	0.06	< 0.01	0.07	

Grapes grown at Napa, CA, and Reedley, CA, were treated with myclobutanil five times at 0.11 kg ai/ha. Samples were harvested at 14 days. The fruit was processed at California State University at Fresno to yield juice, dry pomace, wet pomace, wine, and stems. In addition, filtered wine, A&B raisins, C raisins, midget raisins, waste raisins, and wine lees were produced. Summary of data is shown in Table 76.

Table 76 Residue levels in processed grape products

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
85-0323 Reedley, CA USA 1985 (Thompson)	WP (40% W/W)	5	0.112	935.3	–	–	14	Grapes	0.13	0.04	0.17	94607
							14	Juice	0.04	0.02	0.06	
							14	Dry pomace	0.74	0.26	1.00	
							14	Wet pomace	0.07	0.02	0.09	
							14	Wine	0.04	0.02	0.06	
							14	Filtered wine	0.04	0.00	0.04	
							14	A+B raisin	0.78	0.16	0.94	
							14	C raisin	0.82	0.25	1.07	
							14	Midget	0.69	0.24	0.93	
							14	Waste raisin	3.43	0.80	4.23	
							14	Wine lees	0.22	0.02	0.24	

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicat ion Interval (days)	DAT	Portion Analyse d	Myclobuta nil (mg/kg)	RH-9090 (mg/kg)	Total of myclobuta nil (mg/kg)	Ref.
85-0419 Napa, CA, USA, 1985 (Pinot Noir)	—	5	0.112	935.3	—	—	14	Grapes	0.53	0.07	0.60	94443
							14	Dry pomace	1.16	0.10	1.26	
							14	Wet pomace	0.67	0.14	0.81	
							14	Juice	0.06	0.01	0.07	
							14	Wine	0.07	0.06	0.13	
							14	Stem	1.25	0.27	1.52	

Supervised trials on tomatoes were conducted at two sites in France to provide bulk samples for processing studies. Tomatoes were treated with six foliar applications of a formulation of myclobutanil (EW, 200 g ai/L). The first application was made approximately 11 weeks pre-mid-commercial harvest and at 10–14 days thereafter for the next five applications. Bulk samples of tomatoes were taken at 3 days after the last application and processed into juice and preserves and puree. Samples of whole fruit, juice, preserves and puree were analysed for myclobutanil using a method with a limit of quantitation of 0.01 mg/kg. Summary of data is shown in Table 77.

Table 77 Residue levels in processed tomato products

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicat ion Interval (days)	DAT	Portion Analysed	Myclobuta nil (mg/kg)	RH-9090 (mg/kg)	Total of myclobuta nil (mg/kg)	Ref.
AP/3173/HL/2 /F, Bressols FRA, 1996 (Laurelia) Tomato	EW (200 g ai/L)	6	0.098–0.11	1300–1533	7.5	10–13	3	Juice	0.01	0.00	0.01	241829
							3	Preserve	< 0.01	0.00	< 0.01	
							3	Puree	0.05	0.00	0.05	
							0	Whole fruit	0.11	< 0.01	0.12	
							3		0.05	0.00	0.05	
AP/3173/HL/1 /F, Aucamville FRA, 1996 (Delfine) Tomato	EW (200 g ai/L)	6	0.097–0.11	1293–1420	7.5	10–13	7	Juice	0.01	0.00	0.01	241829
							3	Preserve	0.01	0.00	0.01	
							3	Puree	0.04	0.00	0.04	
							0	Whole fruit	0.07	0.00	0.07	
							3		0.04	0.00	0.04	
							7		0.02	0.00	0.02	

Supervised trials on tomatoes were conducted at two sites in France to provide bulk samples for processing studies. Tomatoes were treated with six foliar applications of a formulated product of myclobutanil (EC, 240 g ai/L). The first application was made approximately 11 weeks pre-mid-commercial harvest and at 10–14 days thereafter for the next five applications. Bulk samples of tomatoes for the processing studies were harvested at 3 days after the last application and processed into juice and preserves and puree. Samples of whole fruit, juice, preserves and puree were analysed for myclobutanil using a method with a limit of quantitation of 0.01 mg/kg. Summary of data is shown in Table 78.

Table 78 Residue levels in processed tomato products

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicat ion Interval (days)	DAT	Portion Analyse d	Myclobu tanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobuta nil (mg/kg)	Ref.
AP/3172/HL/2F, Bressols FRA, 1996 (Laurelia) Tomato	EC (240 g ai/L)	6	0.098–0.12	1308–1600	7.5	10–14	3	Juice	0.01	0.00	0.01	241830
							3	Preserve	< 0.01	0.00	< 0.01	
							3	Puree	0.06	0.00	0.06	
							0	Whole fruit	0.05	0.00	0.05	
							3		0.03	0.00	0.03	
							7		0.03	0.00	0.03	

AP/3172/HL/1F, Aucamville FRA, 1996 (Delfine) Tomato	EC (240 g ai/L)	6	0.093– 0.11	1245– 1423	7.5	10–13	3 3 3 0 3 7	Juice Preserve Puree Whole fruit	0.02 < 0.01 0.06 0.05 0.02 0.03	0.00 0.00 0.00 0.00 0.00 0.00	0.02 < 0.01 0.06 0.05 0.02 0.03	24183 0

A supervised trial on tomatoes was conducted at a field location in California, USA to provide bulk samples for processing studies. Tomatoes were treated with four foliar applications of a formulated product (WP, 40% W/W). The four applications were made at 10–15 day intervals in approximately 74 L/ha spray volumes. Bulk samples of tomatoes for the processing studies were taken at 5 days after the last application and processed into canned, wet pomace, dry pomace, juice, puree, paste, and catsup. Samples of whole fruit and tomato processed fraction were analysed for myclobutanil using a method with a limit of quantitation of 0.01 mg/kg. Summary of data is shown in Table 79.

Table 79 Residue levels in processed tomato products

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Applicat ion Interval (days)	DA T	Portion Analyse d	Myclobuta nil (mg/kg)	RH-9090 (mg/kg)	Total of myclobuta nil (mg/kg)	Ref.
89-0265 Woodland, CA USA 1989 (Harris Moran)	WP (40 %W/W)	4	0.071	281		10–12	5	Canned Catsup Dry pomace Hot break Juice Paste Paste juice Puree Unwash ed Washed Wet pomace	0.01 0.07 0.43 < 0.01 0.02 0.08 0.04 0.02 0.03 0.02 0.25	0.01 0.00 0.00 0.00 0.00 0.03 0.04 0.02 0.00 0.00 0.00	0.02 0.07 0.43 < 0.01 0.02 0.11 0.08 0.04 0.03 0.02 0.25	94609
89-0265 Woodland, CA USA 1989 (Harris Moran)	WP (40 %W/W)	4	0.14	281		10–12	5	Canned Catsup Dry pomace Hot break Juice Paste Paste juice Puree Unwash ed Washed Wet pomace	0.02 < 0.01 1.03 0.02 0.04 0.17 0.04 0.05 0.04 0.06 0.03	0.03 0.03 0.01 0.01 0.02 0.08 0.06 0.03 0.02 0.02 0.01	0.05 0.04 1.04 0.03 0.06 0.25 0.10 0.08 0.06 0.08 0.04	94609

Supervised trials on soybeans were conducted at a site in USA to provide bulk samples for processing studies. Soybeans were treated with two foliar broadcast applications of EW fungicide (208 g ai/L). The first application was made at BBCH 15 and the second application was made 14 days later at growth stage BBCH 63. Bulk samples of soybeans for the processing studies were taken at 14 days after the last application and processed into hulls, meal and oil. Aspirated grain fractions (dust) were also collected during the processing. Samples of seed, dust, hulls, meal and oil were

analysed for myclobutanil using a method with a limit of quantitation of 0.01 mg/kg. Summary of data is shown in Table 80.

Table 80 Residue levels in processed soybean products

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
AR1 Newport, AR USA 2004 (Genesisi C 444 NRR)	EW (200 g ai/L)	2	0.71–0.70	139–141	–	14	14	Aspirated grain fractions	5.03	0.21	5.24	220163 224527 221174
							14	Hulls	0.08	0.01	0.09	
							14	Meal	0.03	0.01	0.04	
							14	Oil, refined	0.15	0.00	0.15	
							14	Seed	0.07	< 0.01	0.08	

A supervised trial on hops was conducted at a site in Germany to provide bulk samples for processing studies. Hops were treated with four foliar applications of EW fungicide (200 g ai/L). Applications were made at a 1.5× rate of 9 g ai/hL applying a maximum spray volume of approximately 3400 L/ha. The first application was made at approximately 8 weeks prior to mid-commercial harvest and the second, third and fourth applications were made at 10–14 day intervals thereafter. Bulk samples of green hops for the processing studies were taken at 14 days after the last application (14 DAT), dried in the field then sent to be processed into spent hops, trub, yeast, and beer. Samples of green hops, dried hops, spent hops, trub, yeast, and beer were analysed for myclobutanil using a method with a limit of quantitation of 0.50 for dried hops, 0.02 mg/kg for green hops, spent hops, trub and yeast and 0.01 mg/kg for beer. Summary of data is shown in Table 81.

Table 81 Residue levels in processed hops products

Trial ID/Location Country/Year (variety)	Form	No. of Apps	Appl Rate (kg ai/ha)	Spray Vol (L/ha)	Appl Conc (g ai/hL)	Application Interval (days)	DAT	Portion Analyzed	Myclobutanil (mg/kg)	RH-9090 (mg/kg)	Total of myclobutanil (mg/kg)	Ref.
R&H/207/1/G Bayern GER 1996 (Hersbrucker)	EW (200 g ai/L)	4	0.18–0.30	2976–5060	6	13–14	14	Beer	< 0.01	< 0.01	< 0.02	94412
							14	Dried hops	1.02	< 0.50	1.52	
							0	Green hops	0.27	0.07	0.34	
							7	Green hops	0.15	0.04	0.19	
							14	Green hops	0.28	0.09	0.37	
							14	Spent hops	0.09	< 0.02	0.11	
							14	Trub	< 0.02	< 0.02	< 0.04	
							14	Yeast	< 0.02	< 0.02	< 0.04	
RAS/20/4/G Bayern GER 1997 (Tradition)	EW (200 g ai/L)	4	0.19–0.30	2050–3300	9	11–13	14	Beer	< 0.01	< 0.01	< 0.02	94441
							14	Dried hops	0.73	< 0.50	1.23	
							0	Green hops	0.82	0.05	0.87	
							7	Green hops	0.21	0.06	0.27	
							14	Green hops	0.33	0.09	0.42	
							14	Spent hops	0.04	0.00	0.04	
							14	Trub	< 0.02	< 0.02	< 0.04	
							14	Yeast	0.00	< 0.02	< 0.02	

Residues in animal commodities

Farm animal feeding studies

The Meeting received a lactating cow and laying hens feeding study, which provided information on likely residues resulting in animal tissues and milk from residues in the animal diet.

Lactating cows

A cow feeding study was conducted to determine the extent to which residues of myclobutanil in livestock feedstuffs transfer to edible tissues and milk. Lactating Holstein cows ranging in age from approximately 3 to 5 years and in weight from 434 to 675 kilograms were used as the test animals in this study. Sixteen cows were randomly assigned to four treatment groups based on concentration of myclobutanil in the diet. Each of the four treatment groups consisted of four cows. Each cow was tagged with a neck chain which contained a unique identification number that matched the number on a card affixed to the individual stall. The dosing levels of myclobutanil in the four feeding groups were as follows: a control group (0 ppm), low dose level (1.3× feeding level, equivalent to 1.6 ppm in feed), medium dose level (3.9× feeding level, equivalent to 4.8 ppm in feed) and a high dose level (13× feeding level, equivalent to 16.0 ppm in feed). The dose preparations were based on the highest feed consumption through day 14 of the quarantine period. The cows were orally dosed using gelatin capsules containing the test substance. The test substance was administered to the cows once daily for 28 consecutive days. Gelatin capsules containing no test substance were administered to the group of control cows during the 28 day dosing period.

The cows were weighed weekly throughout the study. Feed consumption was determined daily. Observations were made twice daily for mortality, morbidity, and pharmacotoxic signs. Milk was collected and measured gravimetrically twice daily. Milk samples were collected for analysis on test days -1, 1, 4, 7, 10, 14, 17, 21, 24, 28 and 31. A milk sample (divided into two containers each with 125 mL of milk) for each cow on each of the sample collection days was obtained by pooling the milk collected from that cow for the a.m. and p.m. milkings in proportion to the cow's total daily production. On test day 28 three cows from each of the four treatment groups were humanely sacrificed. The one remaining cow from each of the four treatment groups was placed on a three-day withdrawal period and then sacrificed on test day 31 to evaluate depuration of residues in tissues. For each cow, a representative sample of liver, kidney, peritoneal fat, and pectoral muscle tissue was taken at the scheduled sacrifices and frozen immediately.

Myclobutanil as well as the metabolite RH-9090 were quantitated in all matrices (milk, fat, muscle, kidney and liver). Additionally, the metabolite RH-0294 was quantitated in milk. Analytical methods used are listed below by matrix and analyte. The limit of quantitation (LOQ) and limit of detection (LOD) for all analytes (myclobutanil, RH-9090, and RH-0294) in the analytical methods listed below is 0.01 ppm and 0.003 ppm, respectively. The results are shown in Table 82 and Table 84.

Table 82 Residues of myclobutanil and RH-0294 in milk from cows dosed with myclobutanil in a 28-day feeding study

Treatment Group	Day	Residues, myclobutanil (mg/kg) ^a					Residues, RH-0294 (mg/kg) ^{a, b}				
		Cow A	Cow B	Cow C	Cow D	Avg. ^c	Cow A	Cow B	Cow C	Cow D	Avg. ^c
Low Dose— 1.6 ppm	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	4	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	7	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	10	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	14	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	17	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	21	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	24	ND	ND	ND	ND	ND	ND	ND	ND	< 0.01	ND
	28	ND	ND	ND	ND	ND	< 0.01	< 0.01	< 0.01	ND	< 0.01
	31 ^d	—	—	—	ND	—	—	—	—	ND	—

Treatment Group	Day	Residues, myclobutanil (mg/kg) ^a					Residues, RH-0294 (mg/kg) ^{a, b}				
		Cow A	Cow B	Cow C	Cow D	Avg. ^c	Cow A	Cow B	Cow C	Cow D	Avg. ^c
Medium Dose— 4.8 ppm	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	4	ND	ND	ND	ND	ND	< 0.01	< 0.01	< 0.01	ND	< 0.01
	7	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	10	ND	ND	ND	ND	ND	ND	ND	ND	< 0.01	ND
	14	ND	ND	ND	ND	ND	ND	ND	ND	< 0.01	ND
	17	ND	ND	ND	ND	ND	< 0.01	ND	ND	ND	ND
	21	ND	ND	ND	ND	ND	ND	< 0.01	ND	ND	ND
	24	ND	ND	ND	ND	ND	< 0.01	< 0.01	ND	ND	< 0.01
	28	ND	ND	ND	ND	ND	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	31 ^d	—	—	—	ND	—	—	—	—	ND	—
High Dose— 16.0 ppm	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	4	ND	ND	ND	ND	ND	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	7	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	10	ND	ND	ND	ND	ND	0.010	0.010	< 0.01	< 0.01	< 0.01
	14	ND	ND	ND	ND	ND	0.010	< 0.01	< 0.01	0.012	< 0.01
	17	ND	ND	ND	ND	ND	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	21	ND	ND	ND	ND	ND	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	24	ND	ND	ND	ND	ND	0.015	0.011	< 0.01	0.010	0.010
	28	ND	ND	ND	ND	ND	< 0.01	< 0.01	< 0.01	0.011	< 0.01
	31 ^d	—	—	—	ND	—	—	—	—	ND	—

^a ND = residue not detected with a limit of detection (LOD) of 0.003 mg/kg.

^b The conversion factor for RH-9090 to myclobutanil equivalents is 0.9475, based on molecular weights of 288.78 and 304.78 for myclobutanil and RH-9090, respectively.

^c For purposes of calculating an average, ND residues were assigned a value of ½ the LOD (0.003 mg/kg/2). Average residue values < 0.003 mg/kg were designated as ND. Residues below the LOQ (< 0.01 mg/kg) were assigned a value of ½ of the LOQ (0.01 mg/kg/2). Average residue values > 0.003 mg/kg, but < 0.01 mg/kg were designated as < 0.01 mg/kg.

^d Residues following a three day withdrawal period.

Table 83 Residues of myclobutanil and RH-9090 in muscle and fat tissue from cows dosed with myclobutanil in a 28-day feeding study

Treatment Group	Cow #	Residues in Muscle (mg/kg) ^a			Residues in Fat (mg/kg) ^a		
		Myclobutanil	RH-9090	RH-9090— expressed as myclobutanil ^b	Myclobutanil	RH-9090	RH-9090— expressed as myclobutanil ^b
Control	10	ND	ND	ND	ND	ND	ND
	7	ND	ND	ND	ND	ND	ND
	13	ND	ND	ND	ND	ND	ND
Average ^c		ND	ND	ND	ND	ND	ND
3-day Withdrawal	12	ND	ND	ND	ND	ND	ND
Low Dose— 1.6 ppm	15	ND	ND	ND	ND	ND	ND
	11	ND	ND	ND	ND	ND	ND
	1	ND	ND	ND	ND	ND	ND
Average ^c		ND	ND	ND	ND	ND	ND
3-day Withdrawal	2	ND	ND	ND	ND	ND	ND
Medium Dose— 4.8 ppm	9	ND	ND	ND	ND	ND	ND
	6	ND	ND	ND	ND	ND	ND
	4	ND	ND	ND	ND	ND	ND
Average ^c		ND	ND	ND	ND	ND	ND
3-day Withdrawal	5	ND	ND	ND	ND	ND	ND
High Dose— 16 ppm	3	ND	< 0.01	< 0.01	ND	ND	ND
	14	ND	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	8	ND	< 0.01	< 0.01	ND	< 0.01	< 0.01
Average ^c		ND	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
3-day	16	ND	ND	ND	ND	ND	ND

Treatment Group	Cow #	Residues in Muscle (mg/kg) ^a			Residues in Fat (mg/kg) ^a		
		Myclobutanil	RH-9090	RH-9090—expressed as myclobutanil ^b	Myclobutanil	RH-9090	RH-9090—expressed as myclobutanil ^b
Withdrawal							

^a ND = residue not detected with a limit of detection (LOD) of 0.003 mg/kg.

^b The conversion factor for RH-9090 to myclobutanil equivalents is 0.9475, based on molecular weights of 288.78 and 304.78 for myclobutanil and RH-9090, respectively.

^c For purposes of calculating an average, ND residues were assigned a value of ½ the LOD (0.003 ppm/2). Average residue values < 0.003 ppm were designated as ND. Residues below the LOQ (< 0.01 mg/kg) were assigned a value of ½ of the LOQ (0.01 mg/kg/2). Average residue values > 0.003 mg/kg, but < 0.01 mg/kg were designated as < 0.01 mg/kg.

Table 84 Residues of myclobutanil and RH-9090 in liver and kidney tissue from cows dosed with myclobutanil in a 28-day feeding study

Treatment Group	Cow #	Residues in Liver (mg/kg) ^a			Residues in Kidney (mg/kg) ^a		
		Myclobutanil	RH-9090	RH-9090—expressed as myclobutanil ^b	Myclobutanil	RH-9090	RH-9090—expressed as myclobutanil ^b
Control	10	ND	ND	ND	ND	ND	ND
	7	ND	ND	ND	ND	ND	ND
	13	ND	ND	ND	ND	ND	ND
Average ^c		ND	ND	ND	ND	ND	ND
3-day Withdrawal	12	ND	ND	ND	ND	ND	ND
Low Dose—1.6 ppm	15	ND	ND	ND	ND	ND	ND
	11	ND	ND	ND	ND	ND	ND
	1	ND	ND	ND	ND	ND	ND
Average ^c		ND	ND	ND	ND	ND	ND
3-day Withdrawal	2	ND	ND	ND	ND	ND	ND
Medium Dose—4.8 ppm	9	ND	< 0.01	< 0.01	ND	< 0.01	< 0.01
	6	ND	0.010	0.010	ND	ND	ND
	4	ND	< 0.01	< 0.01	ND	< 0.01	< 0.01
Average ^c		ND	< 0.01	< 0.01	ND	< 0.01	< 0.01
3-day Withdrawal	5	ND	ND	ND	ND	ND	ND
High Dose—16 ppm	3	< 0.01	0.014	0.013	ND	ND	ND
	14	0.011	0.032	0.030	ND	ND	ND
	8	< 0.01	0.015	0.014	ND	< 0.01	< 0.01
Average ^c		< 0.01	0.020	0.019	ND	< 0.01	< 0.01
3-day Withdrawal	16	ND	ND	ND	ND	ND	ND

^a ND = residue not detected with a limit of detection (LOD) of 0.003 mg/kg

^b The conversion factor for RH-9090 to myclobutanil equivalents is 0.9475, based on molecular weights of 288.78 and 304.78 for myclobutanil and RH-9090, respectively.

^c For purposes of calculating an average, ND residues were assigned a value of ½ the LOD (0.003 ppm/2). Average residue values < 0.003 ppm were designated as ND. Residues below the LOQ (< 0.01 mg/kg) were assigned a value of ½ of the LOQ (0.01 mg/kg/2). Average residue values > 0.003 mg/kg, but < 0.01 mg/kg were designated as < 0.01 mg/kg.

Laying Hens

A poultry feeding study was conducted to determine the extent to which residues of myclobutanil in poultry feedstuffs transfer to edible tissues and eggs.

Eighty white leghorn laying hens in top laying condition (38 weeks old) were used in this study and acclimated to the testing facility for (7) days. Ten hens were randomly assigned to each of (5) groups. Groups 6 and 7 were not utilized for residue analysis. Each hen received the radiocarbon

dose in a gelatin capsule for 28 days followed by < 24 hr, 7, and 14 day withdrawal periods (groups 1–5).

Eggs were collected each day (AM) from each hen for egg production records. All eggs for a group were kept on days -1,1,2,4,7,10,14,21,28,29,30,31,32,35 and 42, deshelled and stored frozen. Necropsy was performed for dose groups 1–5 on days 28, 35, and 42 (3–4 hens per group). At necropsy, the following tissues and whole organs were collected after exsanguinations, and immediately frozen: Breast muscle, thigh muscle, liver, kidneys, gizzard (washed, w/o contents), heart, fat. On each sacrifice day, the control hens were necropsied first, followed by the low dose group, working finally to the higher dose group hens last. Terminal body weights were taken for hens in groups 1–5.

All egg samples were homogenized by blending, aliquoted and combusted in duplicate. Tissue samples were blended with dry ice to a fine consistency and aliquoted for combustion. Combusted samples were quantitated for radiocarbon content using a liquid scintillation spectrometer in the external standardization mode. The results are shown in Table 85.

Table 85 Residues of myclobutanil in tissues from poultry dosed with myclobutanil in a 28-day feeding study

Tissue	Day	Residues, myclobutanil (mg/kg) ^a			
		1.0 ppm	3.0 ppm	10.0 ppm	30.0 ppm
Whole Eggs	–1	ND	ND	ND	ND
	1	ND	ND	ND	ND
	2	ND	0.005	0.019	0.054
	4	0.002	0.008	0.023	0.081
	7	0.005	0.011	0.034	0.118
	10	0.003	0.012	0.027	0.107
	14	0.003	0.011	0.029	0.094
	21	0.004	0.012	0.030	0.100
	28	0.003	0.013	0.031	0.122
	29	0.003	0.010	0.030	0.129
	30	ND	0.005	0.013	0.046
	31	0.003	0.003	0.012	ND
	32	ND	0.003	0.007	0.026
	35	ND	ND	ND	ND
	41	ND	ND	ND	ND
Liver	28	0.003	0.006	0.018	0.047
	35	ND	ND	ND	ND
	42	ND	ND	ND	ND
Kidney	28	ND	0.003	ND	0.021
	35	ND	ND	ND	ND
	42	ND	ND	ND	ND

The results presented in each group are the average of 10 hens. Actual feed concentrations were 1.05, 3.57, 10.2, and 28.3 ppm. Results reported as RH-3866 equivalents.

^a ND = no detectable residue. The minimum quantifiable limit for tissue except fat was 0.002 ppm for groups 1 to 3, 0.006 ppm for group 4, and 0.018 ppm for group 5. The minimum quantifiable limit for fat was 0.005 ppm for groups 1 to 3, 0.015 ppm for group 4, and 0.045 ppm for group 5.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

There are no data on market-basket monitoring studies available for myclobutanil.

NATIONAL RESIDUE DEFINITIONS

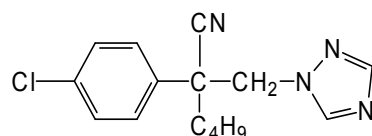
Information wasn't provided on national residue definitions for myclobutanil.

APPRAISAL

Myclobutanil was originally evaluated by the JMPR in 1992 and re-evaluated for residues several times up to 1998. Myclobutanil is a systemic protectant fungicide used to control brown rot, powdery mildew and leaf spot in stone fruit crops, and belongs to the sterol demethylation inhibitor (DMI) class of fungicides.

At the Forty-fifth Session of the CCPR (REP13/PR, Appendix XIV), myclobutanil was scheduled for periodic residue review by the 2014 JMPR. The Meeting received information on physical and chemical properties, metabolism, environmental fate, analytical methods and freezer storage stability, national registered use patterns, as well as supervised trials, processing studies and livestock feeding studies.

Myclobutanil is (R, S)-2-(4-chlorophenyl)-2-(1H-1, 2, 4-triazol-1-ylmethyl) hexanenitrile and exists as a racemate.



The following compound codes are used for the metabolites discussed below:

RH-9089	(2RS) -2-(4-chlorophenyl) -5-oxo-2-(1H-1,2,4-triazol-1-ylmethyl)hexanenitrile	
RH-9090	α -(4-chlorophenyl)- α -(3-hydroxybutyl)-1H-1,2,4-triazole-1-propanenitrile	
MW 318 Acid (butyl carboxylic acid of myclobutanil)	5-(4-chlorophenyl)-5-cyano-6-(1H-1,2,4-triazol-1-yl)hexanoic acid	
N-Glucuronic Acid Conjugate of Myclobutanil	1-[2-(4-chlorophenyl)-2-cyanoethyl]-4-hexopyranuronosyl-1H-1,2,4-triazol-4-ium	

Hydroxy-lactone	3-(4-chlorophenyl)-5-(1-hydroxyethyl)-3-(1H-1,2,4-triazol-1-ylmethyl)dihydrofuran-2(3H)-one	
RH-9090 Glucuronic Acid Conjugate	5-(4-chlorophenyl)-5-cyano-6-(1H-1,2,4-triazol-1-yl)hexan-2-yl hexopyranosiduronic acid	
RH-9090 Sulfate Conjugate	5-(4-chlorophenyl)-5-cyano-6-(1H-1,2,4-triazol-1-yl)hexan-2-yl hydrogen sulfate	
MW 334 Acid	5-(4-chlorophenyl)-5-cyano-2-hydroxy-6-(1H-1,2,4-triazol-1-yl)hexanoic acid	
RH-294 (Diol)	α -(4-chlorophenyl)- α -(3,4-dihydroxybutyl)-1H-1,2,4-triazole-1-propane-nitrile	
RH-294 Sulfate Conjugate	5-(4-chlorophenyl)-5-cyano-1-hydroxy-6-(1H-1,2,4-triazol-1-yl)hexan-2-yl hydrogen sulfate	
RH-3968 (Triazolyl Alanine, TA)	(2R)-2-amino-3-(1H-1,2,4-triazol-1-yl)propanoic acid	
RH-4098 (Triazolyl Acetic Acid, TAA)	1H-1,2,4-triazol-1-ylacetic acid	

Animal metabolism

Information was available on metabolism of myclobutanil in rats, lactating goats and laying hens.

Laboratory animals: Myclobutanil was mainly and rapidly absorbed (> 89%), extensively metabolised and rapidly and completely excreted after oral administration in rats. Peak plasma and tissue radiolabelled myclobutanil were achieved within 1 hr after oral administration and plasma elimination was biphasic. No significant tissue accumulation was observed 96 hr post-dosing. Metabolism appears to have occurred mainly through a variety of oxygenation reactions of the butyl group. The major metabolic processes involved oxygenation to the butyl group and among the

metabolites formed are the RH9090 and RH9089, the major unconjugated phenethyl triazole-containing metabolites found in plants.

Lactating goats: Myclobutanil was orally dosed to lactating goats for 5 days. Individual goats were dosed separately with myclobutanil radiolabelled in either the triazole (TZ) portion or the phenyl ring (PH) at the rate of 24 ppm and 14 ppm in the diet per day, respectively. Approximately 71% and 79% of TAR was recovered in the faeces and urine for the TZ-label and PH-label dosed animal. Most of administered dose was rapidly absorbed, metabolised and rapidly eliminated via the urine (49–58% TAR) and faeces (22% TAR). TRR levels in tissues ranged from 0.016 mg eq/kg in omental fat to 0.49 mg eq/kg in liver for the low dosed goat (PH label) and from 0.027 mg eq/kg in omental fat to 0.92 mg eq/kg in liver for the high dosed goat (TZ label). Residue levels were highest in liver and kidney and significantly lower in muscle and fat.

Milk and edible tissues were extracted with acetone, hexane and acetonitrile/water (80/20) and 78–101% radioactive residues were recovered. Unchanged parent compound was only observed in liver (2.1–6.0% TRR, 0.019–0.029 mg/kg). The metabolite RH-9090 and its sulphate and glucuronic acid conjugates were the primary residue in liver (total 59–60% TRR, 0.29–0.54 mg eq/kg), in kidney (total 58–61% TRR, 0.13–0.30 mg eq/kg), in muscle (total 47–80% TRR, 0.02–0.03 mg eq/kg) and in fat (total 44–46% TRR, 0.01–0.02 mg eq/kg). The hydroxy-lactone was the only other metabolite present in liver at levels in excess of 10% of the TRR (12–16% TRR, 0.08–0.11 mg eq/kg).

For both labels in milk, the residue levels reached a plateau within 4 days after the initiation of dosing at a level of approximately 0.033–0.079 mg eq/kg. The primary residue was RH-9090 which constituted about 28–58% of the TRR, while the only other two metabolites representing 10% or more of the TRR were RH-294 and the MW 318 carboxylic acid. No parent compound was detected in milk.

Laying hens: [^{14}C]-Myclobutanil was administered orally to groups of three laying hens once daily for 7 consecutive days at the nominal equivalent of 110 ppm in a diet. Over 95% of the dosed radioactivity was recovered in the excreta. Whole eggs and muscle were extracted with ethyl acetate, and then extracted with methanol. Fat and edible offal were extracted with n-hexane and methanol. The extracted radioactive residues accounted for 69% TRR in whole eggs, 79% TRR in fat, 83% TRR in thigh muscle, 97% TRR in breast muscle, 156% TRR in kidney (118% TRR as uncharacterized) and 72% TRR in liver (61% TRR as uncharacterized), respectively.

Total radioactive residues were observed in the liver (0.52 mg eq/kg) and the kidney (0.32 mg eq/kg), while lower residues were found in muscle (0.06 mg eq/kg) and fat (0.02 mg eq/kg). Parent myclobutanil was a main residue in fat (67% TRR) in kidney (12% TRR) in liver (4.8% TRR), and in muscle (up to 4% TRR). A band that co-chromatographed with RH-9090/RH9089 and lactone accounted for 15% TRR in kidney. The major component of the residue in muscle was RH-9089 (61–72% TRR). The major component of the residue in eggs was RH-9090 accounting for 36% TRR. No parent compound was observed in eggs.

In summary, the metabolism found in livestock was qualitatively comparable with that observed in laboratory animals. Characterization of the residues show myclobutanil together with RH-9090 and its conjugates are the major residues in the animal tissues, milk and eggs except RH-9089 as a major component in the muscle of laying hens.

Plant metabolism

The Meeting received plant metabolism studies with myclobutanil on grapes, apples, wheat, and sugar beets.

Grape seedlings in the greenhouse were placed in nutrient solution containing either ^{14}C -phenyl myclobutanil or ^{14}C -triazole myclobutanil. An average of 37% TRR remained as the parent compound. RH-9090 accounted for 6% TRR, with 11% TRR present as the RH-9090 glucoside and 14% TRR as an unknown polar component. In the 16-day uptake samples, parent compound accounted for an average of 53% TRR, RH-9090 8% TRR and RH-9090 glucoside 12% TRR.

Grape vines were sprayed five times weekly with myclobutanil, labelled with ^{14}C in the phenyl ring or the triazole ring each at a rate equivalent to 0.05 kg ai/ha. The overall recovery of identified radioactive residues ranged from 79% to 82%. TRR in whole grapes at harvest were 0.32 and 0.24 mg eq/kg for PH and TZ grapes, respectively. The major component of residue was the parent compound accounting for 66% TRR, RH-9090 for 7–9% TRR, RH-9090 glucoside for 5–6% and RH-9089 for 1%, respectively.

Apple trees received ten approximately weekly sprays of myclobutanil, labelled with ^{14}C in the phenyl ring or the triazole ring, at 0.24 kg ai/ha. After extraction with chloroform or methanol, the overall recoveries of identified radioactive residues ranged from 84% to 86% TRR. TRR in whole grapes at harvest were 0.48 and 0.32 mg eq/kg for PH and TZ grapes, respectively. The major component of the terminal residue remained parent compound accounting for 49% of TRR. Conjugated RH-9090 accounted for 21–24% TRR. Free RH-9090 accounted for 12% TRR. A minor component was RH-9089 present at 1.9% TRR. There were no differences in the metabolic profile between the two radiolabelled experiments.

Wheat seedlings were exposed to ^{14}C -myclobutanil at either 42 mg/kg (PH label) or 64 mg/kg (TZ label) in nutrient solutions for an 11 day period placed in the greenhouse. After extraction with methanol, the overall recovery including the unextracted residue ranged from 90% to 96%. In the wheat seedlings, most of the radioactivity (62–71% TRR) remained as parent compound. The total conjugates constituted the complement of the total residues (accounting for 21–30% TRR). In the excised wheat shoots, more than 72% of TRR maintained as unchanged parent compound. In the excised heads of 13-day uptake samples, parent compound accounted for up to 75% TRR, free RH-9090 for 5% TRR, RH-9090 glucoside for up to 18% TRR for both labelling forms.

The metabolism of myclobutanil, using either PH or TZ labels, was studied in wheat under field conditions and greenhouse at a rate equivalent to 0.28 kg ai/ha. After extraction with methanol, the overall recovery of identified radioactive residues ranged between 77% and 102%. TRRs in wheat grain ranged from 0.07 to 3.6 mg eq/kg, and those in wheat straw were from 2.8–69 mg eq/kg. The main components of the residue in wheat straw were parent myclobutanil (under field: 29–47% TRR), RH-9090 and its conjugates (under field: 23–41% TRR). The main components of the residue in wheat grains were RH-9090 and its conjugates (35% TRR) and parent myclobutanil (11% TRR) treated with PH label under field conditions. However, the main components of the residue in wheat grains were RH-3968 (51% TRR) and RH-4098 (25% TRR), and RH-9090 and its conjugates (8.9% TRR) treated with TZ label under field conditions while unchanged myclobutanil was a minor component of residue treated with TZ label accounting for 0.4% TRR.

Foliar applications were made to sugar beet at application rates equivalent to 0.15 kg ai/ha and 1.50 kg ai/ha using two radiolabelled forms ([^{14}C]-phenyl-myclobutanil and [^{14}C]-triazole-myclobutanil) 30 days prior to maturity. After extracted with acetonitrile, the overall recovery ranged between 91% and 105%. The main components of the residue in roots were parent myclobutanil (27–33% TRR, 0.01–0.03 mg eq/kg), conjugated RH-9090 and free RH-9090 (total 8–14% TRR, 0.006–0.007 mg eq/kg). The main components of the residue in leaves at maturity were RH-9090 (50–62% TRR, 0.26–0.43 mg eq/kg) and parent myclobutanil (16–34% TRR, 0.11–0.18 mg/kg), respectively.

In summary, the metabolism of myclobutanil in crops is qualitatively consistent and considered comparable except in wheat treatment with TZ label. The conversion of myclobutanil to RH-9090 followed by conjugation with glucose is the major metabolic pathway. Minor amounts of RH-9089 are probably a result of oxidation of RH-9090. The presence of RH-3968 and RH-4098 in wheat treated with TZ label indicates that the phenethyl triazole linkage in parent was metabolically cleaved.

Environmental fate in soil

The Meeting received information on the environment fate of myclobutanil in confined and field crop rotational studies. A study on degradation of myclobutanil in aerobic soil showed that half-life values reached up to 574 days. Myclobutanil could be a persistent compound in some soils.

Confined rotational crop

The metabolism of ^{14}C -triazole-myclobutanil in succeeding crops was investigated in wheat, radish and lettuce cultivated at three different plant back intervals for all crops (30, 120 and 365 days) at $1 \times 0.36 \text{ kg ai/ha}$. Lettuce, radish and wheat were planted at rotational intervals of 30, 120 and 365 days after soil treatment, TRRs ranged from 0.07 to 2.7 mg eq/kg were found in harvested crops. Radioactive residues in immature and mature lettuce and radish tops declined over time, while residues in radish roots increased. Residues in wheat hay, straw and grain did not show consistent increase or decline. The three most abundant non-polar components in crops planted 30 days after soil application were myclobutanil at 0.43 mg/kg (55% TRR) in mature lettuce, MW 309 di-acid at 0.38 mg eq/kg (14% TRR) and RH-9090 at 0.47 mg eq/kg (17% TRR), both in wheat straw. The two most abundant polar metabolites were the triazole alanine at 0.45 mg eq/kg (30% TRR) and triazole acetic acid at 0.43 mg eq/kg (29% TRR), both in 120 DAT wheat grain. Unextracted residues exceeded both 10% of the TRR and 0.05 mg eq/kg only in wheat hay, straw and grain at all plant-backs. In wheat hay and straw, the unextracted residues ranged from 11 to 23% TRR and from 0.17 to 0.53 mg eq/kg. For wheat grain, the unextracted residues ranged from 26 to 39% TRR and from 0.21 to 0.57 mg eq/kg.

The unchanged parent molecule was found as main component in samples of immature, mature lettuce (30-day PBI), radish roots (30, 120, 365-day PBI) and wheat forage (30 day PBI). Myclobutanil was detected as minor component in other samples. Metabolites were generally detected in lower concentrations. The parent compound, RH-9090 and its conjugates were found in most parts of the four crops. The other two most abundant metabolites were MW 309 di-acid (0.4–28% TRR) and butyric acid (0.9–15% TRR), were not identified in the metabolism studies of crops. This study indicates a potential uptake of residues for myclobutanil into plant commodities.

Field succeeding crop

Myclobutanil was applied at $6 \times 0.14 \text{ kg ai/ha}$ to zucchini in the USA (California and Georgia) approximating the estimated plateau level in soil after subsequent annual application. Within 2 days after the last application, zucchini fruit were harvested and removed from the plots. The remaining plant parts were incorporated into the soil 7–10 days after harvest and then rotational crops (soya bean, radish and wheat) were planted 30 days after the last application to the target zucchini crop. Rotational crops were sampled ranging between 71 and 258 days after final treatment. The plants were Soxhlet extracted with methanol and analysed for the parent compound and RH-9090. Residues of myclobutanil and RH-9090 occurred up to 0.36 and 0.15 mg/kg in soya bean forage, 0.093 and 0.19 mg/kg in soya bean hay, 0.052 and $< 0.01 \text{ mg/kg}$ in radish root, 0.044 and 0.12 mg/kg in radish tops, 0.071 and 0.11 mg/kg in wheat forage, 0.18 and 0.63 mg/kg in wheat straw, respectively. Myclobutanil and RH-9090 residues were higher in vegetative matrices (forage, hay and straw) than the respective seed or grain crop matrix. Residue values for soya bean seeds and wheat grains were all below LOQ (0.01 mg/kg).

Methods of analysis

The Meeting received descriptions and validation data for analytical methods for residues of myclobutanil and RH-9090 in plant and meat. Myclobutanil residues can be measured in most matrices to the LOQ range of 0.01 to 0.05 mg/kg. No stereo-selective methods were submitted for two myclobutanil enantiomers.

The crop and animal methods typically use an initial extraction with methanol or acetone or acetonitrile, and clean-up with partition and/or column steps. The final solution was analysed by GC-ECD, GC-MS or LC/MS/MS. If RH-9090 and its conjugates are determined, hydrolysis with concentrated acid and heating is applied after extraction. Myclobutanil residues can be measured in most matrices to an LOQ of 0.01 mg/kg. All methods are considered sufficiently validated. Multi-residue enforcement method DFG S19 and MRM-1 were provided and validated. The DFG S19 was valid for RH-9090 in animal tissues except fat and MRM-1 was valid for myclobutanil in crops with the LOQ of 0.2 mg/kg. Analytical methods in the feeding studies were valid for determination of

myclobutanil and total RH-9090 in milk, myclobutanil and free RH-9090 in muscle, fat, liver and kidney.

Stability of residues in stored analytical samples

The Meeting received information on the freezer storage stability of residues of myclobutanil in plant and animal commodities.

Storage stability studies were conducted on apples, radish root, soya bean, wheat forage, wheat grain, wheat hay, wheat straw, blueberry, cucurbits and snap beans. Analytical results demonstrated that myclobutanil and RH-9090 were stable in the different plant matrices for at least one year, the duration of the test period.

Storage stability studies on liver and muscle were carried out. Analytical results demonstrated that myclobutanil and its metabolite RH-9090 were stable for at least 80 days, the duration of the study.

Definition of the residue

The composition of the residue in the metabolism studies, the available residue data in the supervised trials, the toxicological significance of metabolites, the capabilities of enforcement analytical methods and the national residue definitions already operating all influence the decision on residue definition.

The metabolism studies of lactating goats showed that the unchanged parent compound was only observed in liver. RH-9090 and its conjugates were the primary residues in liver, kidney, muscle and fat. The primary residue was RH-9090, constituted about 28–58% of the TRR in milk and no parent compound was observed.

In laying hens studies, the highest ^{14}C levels were observed in liver and kidney, while fewer residues were found in muscle and fat. Parent was one of the main components detected in liver and in kidney. RH-9090-sulphate was another main residue detected in liver. A band that co-chromatographed with RH-9090/RH9089 and hydroxy-lactone accounted for 15% TRR in kidney. The major component of the residue in muscle extracts was RH-9089. The major component of residue in fat was parent compound. RH-9090 was the major component of the residue in eggs. No parent compound was observed in eggs.

RH-9090 and its conjugates are the main residues in the animal tissues, milk and eggs. Parent compound is also identified in most of tissues and as a major component in fat and kidney. Although RH-9090 and RH-9089 are found in tissues of animal metabolism study, no residues of parent compound and metabolites were expected above LOQ on the basis of dietary burden calculation and animal feeding studies. The Meeting recommended that, parent myclobutanil is the appropriate residues of concern for MRL enforcement and dietary risk assessment in animal commodities.

The octanol-water partition coefficient of myclobutanil ($\log K_{\text{OW}} = 2.56$) suggested that myclobutanil is not fat-soluble. Noting that myclobutanil residues in animal fat were less than those in muscle, the Meeting agreed that myclobutanil residue is not fat-soluble.

Metabolism studies on plants, and confined rotational crop showed that the main residues in food or feed of plant origin were myclobutanil and/or conjugated RH-9090 and free RH-9090. The Meeting decided that for plant commodities, parent myclobutanil is the appropriate residue of concern for MRL enforcement, and myclobutanil and RH-9090 and its conjugates for dietary risk assessment.

Definition of the residue (for compliance with the MRL) for plant and animal commodities: *myclobutanil*.

Definition of the residue (for estimation of dietary intake) for animal commodities: *myclobutanil*.

Definition of the residue (for estimation of dietary intake) for plant commodities: *sum of myclobutanil, α -(4-chlorophenyl)- α -(3-hydroxybutyl)-1H-1,2,4-triazole-1-propanenitrile (RH-9090) and its conjugates, expressed as myclobutanil.*

The residue is considered as not fat-soluble.

Results of supervised residue trials on crops

The Meeting received supervised trials data for myclobutanil formulations for apple, pear, peach, cherry, apricot, plum, currant, grapes, strawberry, banana, hops, tomato, squash, pepper, cucumber, melon, watermelon, snap beans and soya beans.

The method for calculation of the total residues is illustrated as follows (similar molecular weight, suggest to sum up residue of myclobutanil and RH-9090 as total residue).

RH-9090 less than LOQ (0.01 mg/kg) and more than LOD (0.0025 mg/kg)

Myclobutanil, mg/kg	RH-9090, mg/kg	Total, mg/kg
< 0.01	< 0.01	< 0.02
0.08	< 0.01	0.09

RH-9090 less LOD (0.0025 mg/kg)

Myclobutanil, mg/kg	RH-9090, mg/kg	Total, mg/kg
< 0.01	< 0.0025	< 0.01
0.08	< 0.0025	0.08

RH-9090 equal to or more than LOQ (0.01 mg/kg)

Myclobutanil, mg/kg	RH-9090, mg/kg	Total, mg/kg
0.21	0.03	0.24

Pome fruits

The critical GAP for myclobutanil on pome fruits is in Czech Republic, 3×0.09 kg ai/ha, 14-day PHI. Seven trials were available from Europe on apple against Czech GAP with myclobutanil residue of 0.03(2), 0.05, 0.07, 0.11, 0.20 and 0.34 mg/kg, with total residue of 0.03, 0.04, 0.05, 0.08, 0.12, 0.22 and 0.35 mg/kg.

Eight trials were available from Europe on pear against Czech GAP with myclobutanil residue of 0.03(2), 0.04, 0.05, 0.06, 0.10, 0.28 and 0.32 mg/kg, and with total residue of 0.05(3), 0.06, 0.07, 0.11, 0.29 and 0.35 mg/kg.

Noting the similar data population from apple and pear and medians of the datasets differed less than 5-fold, The Meeting agree to combined residues expressed in terms of myclobutanil of 0.03(4), 0.04, 0.05(2), 0.06, 0.07, 0.10, 0.11, 0.20, 0.28, 0.32 and 0.34 mg/kg. The residues expressed in terms of total residues were: 0.03, 0.04, 0.05(4), 0.06, 0.07, 0.08, 0.11, 0.12, 0.22, 0.29 and 0.35(2) mg/kg. On the basis of myclobutanil residues, the Meeting estimated a maximum residue level of 0.6 mg/kg pome fruits to replace the previous recommendation of 0.5 mg/kg. The Meeting also agreed to combine two datasets to estimate an HR of 0.35 mg/kg, an STMR of 0.07 mg/kg based on the total residue.

Stone fruits

The critical GAP for myclobutanil on peach, nectarine and cherry is in the USA, 8×0.18 kg ai/ha with PHI of 0 day. Nine trials were conducted with application number from 7 to 11 within $\pm 25\%$ GAP rate in the USA. No significant contribution should be expected from the treatments more than 3 half-lives to the final residue. Myclobutanil residues with 0 day PHI on peach were: 0.34, 0.38, 0.62, 0.66, 0.75, 0.84, 0.85 and 1.23 mg/kg and the total residues were: 0.37, 0.55, 0.76, 0.82, 0.91, 1.06, 1.12 and 1.54 mg/kg.

Eight trials were available from the USA on cherry with myclobutanil residue 0.20, 0.28, 0.75, 0.85, 0.92, 1.04, 1.12 and 1.44 mg/kg, and with total residue 0.22, 0.32, 0.82, 1.05, 1.07, 1.52, 1.61 and 2.05 mg/kg.

The Meeting estimated an HR of 2.05 mg/kg, an STMR 1.06 mg/kg, and maximum residue level of 3 mg/kg for cherry.

The critical GAP for myclobutanil on apricot, plum and prune is in the USA, 7×0.18 kg ai/ha with PHI of 0 day. Seven trials were available from the USA and Europe on apricot against US GAP with myclobutanil residue 0.11, 0.12, 0.17, 0.18, 0.23, 0.34 and 0.62 mg/kg, and with total residue 0.13, 0.14, 0.21, 0.25, 0.29, 0.38 and 0.70 mg/kg.

Eight trials were available from the USA on plum against US GAP with myclobutanil residue 0.09, 0.13, 0.20, 0.25, 0.28, 0.59, 0.97 and 1.12 mg/kg, and with total residue 0.11, 0.36, 0.40, 0.73 and 1.45 mg/kg.

The Meeting estimated an HR of 1.45 mg/kg, an STMR 0.40 mg/kg, and maximum residue level of 2 mg/kg and agreed to replace the previous recommendation of 0.2 mg/kg for plums.

Considering the higher residues came from peach, the Meeting decided not to combine datasets of peach and apricot, and estimated an HR of 1.54 mg/kg, STMR 0.865 mg/kg, and maximum residue level of 3 mg/kg for peach, nectarine and apricot on the basis of residues of peach. The Meeting agreed to withdraw the previous recommendation of 2 mg/kg for stone fruits (except plums) and 0.5 mg/kg for prunes.

Currants

The critical GAP for myclobutanil on currants is in the UK, 6×0.09 kg ai/ha with PHI of 14 days. Twelve trials were available from the UK on black currants against GAP with myclobutanil residue 0.19, 0.24(2), 0.26(2), 0.29, 0.30(2), 0.31, 0.35, 0.42 and 0.43 mg/kg, and with total residue 0.21, 0.28(2), 0.31(2), 0.34(2), 0.35, 0.37, 0.39, 0.46 and 0.47 mg/kg.

The Meeting agreed to estimate an HR 0.47 mg/kg, an STMR 0.34 mg/kg, and maximum residue level of 0.9 mg/kg for currants to replace the previous recommendation of 0.5 mg/kg on currants, black.

Grapes

The critical GAP for myclobutanil on grapes is in the USA, 5×0.15 kg ai/ha with PHI of 14 days. Nine trials were available from the USA on grapes against US GAP with myclobutanil residue in grape 0.13, 0.14(2), 0.25, 0.31, 0.32(2), 0.48 and 0.53 mg/kg, and with total residue in grape 0.17, 0.19(2), 0.29, 0.34, 0.40(2), 0.58 and 0.60 mg/kg.

The Meeting estimated an HR of 0.60 mg/kg, an STMR 0.34 mg/kg, and maximum residue level of 0.9 mg/kg for to replace the previous recommendation of 1 mg/kg for grapes.

Strawberry

The critical GAP for myclobutanil on strawberry is in the USA, 6×0.14 kg ai/ha with PHI of 0 day. Seven outdoor trials from the USA against US GAP gave residues 0.03, 0.04, 0.10, 0.15, 0.20, 0.23 and 0.31 mg/kg.

The critical GAP for myclobutanil on strawberry is in the UK, 6×0.09 kg ai/ha with PHI of 3 day. In 19 outdoor trials from Europe at UK GAP, myclobutanil residues were 0.08, 0.10, 0.14, 0.15, 0.17, 0.18(2), 0.19(4), 0.20(2), 0.22, 0.24, 0.30, 0.48, 0.50 and 0.69 mg/kg, and total residues were 0.09, 0.10, 0.15(2), 0.17, 0.18(2), 0.19(4), 0.20(2), 0.22, 0.24, 0.31, 0.48, 0.50 and 0.69 mg/kg. Eight indoor trials were available from Europe on strawberry against UK GAP with myclobutanil residue of 0.13, 0.16, 0.18, 0.19, 0.20, 0.24, 0.37 and 0.46 mg/kg, and with total residue of 0.14, 0.17, 0.19, 0.20, 0.21, 0.25, 0.38 and 0.47 mg/kg.

Considering the higher residues came from Europe, residue populations from indoor and outdoor European trials were similar, the Meeting decided to combine the two datasets. The residues in 27 trials were 0.08, 0.10, 0.13, 0.14, 0.15, 0.16, 0.17, 0.18(3), 0.19(5), 0.20(3), 0.22, 0.24(2), 0.30, 0.37, 0.46, 0.48, 0.50 and 0.69 mg/kg for myclobutanil and 0.09, 0.10, 0.14, 0.15(2), 0.17(2), 0.18(2), 0.19(5), 0.20(3), 0.21, 0.22, 0.24, 0.25, 0.31, 0.38, 0.47, 0.48, 0.50 and 0.69 mg/kg for myclobutanil and RH-9090 and its conjugates. The Meeting estimated a maximum residue level of 0.8 mg/kg on the basis of parent residues for strawberry to replace the previous recommendation of 1 mg/kg on strawberry. The Meeting also estimated an HR of 0.69 mg/kg and an STMR of 0.19 mg/kg on the basis of total residues.

Banana

Myclobutanil is registered in Costa Rica for use as a post-harvest run-off or dip application at 84.8 g ai/hL, application before packaging fruits. Three trials from Costa Rica on banana against GAP gave myclobutanil residues in whole fruits of 0.028(2) and 0.29 mg/kg. Total residues in banana pulp were 0.06, 0.07 and 0.09 mg/kg.

The Meeting agreed that there is not sufficient evidence that the proportionality principal is applicable to post-harvest treatment. Three trials on banana were not enough for estimation of an MRL, an STMR and an HR for banana. The Meeting agreed to withdraw the previous recommendation of 2 mg/kg for banana.

Fruiting vegetables, other than Cucurbits

Tomatoes

The critical GAP for myclobutanil on tomatoes is in the USA, 4× 0.11 kg ai/ha with PHI 0 day. Seventeen outdoor trials were available from the USA on tomatoes against US GAP with myclobutanil residue 0.02, 0.03(2), 0.04(2), 0.06, 0.07(5), 0.08(2), 0.09, 0.10, 0.11 and 0.22 mg/kg, and with total residue 0.02, 0.03, 0.04(2), 0.05, 0.06, 0.07(4), 0.08, 0.09, 0.10(2), 0.11, 0.12 and 0.25 mg/kg.

The Meeting decided to estimate an HR of 0.25 mg/kg, an STMR of 0.07 mg/kg based on total residues, and a maximum residue level of 0.3 mg/kg based on myclobutanil residues for tomatoes to confirm the previous recommendation.

Peppers

The critical GAP for myclobutanil on peppers is in the USA, 4× 0.14 kg ai/ha with PHI of 0 day. Two outdoor trials were available from the USA on sweet pepper against US GAP with myclobutanil residue 0.03 and 0.47 mg/kg, and with total residue 0.05 and 0.64 mg/kg. Four outdoor trials were available from the USA on chilli pepper against US GAP with myclobutanil residue 0.09, 0.18, 1.19 and 2.03 mg/kg, and with total residue 0.12, 0.23, 1.39, 2.40 mg/kg.

Considering residues from sweet and chilli peppers were similar, the Meeting decided to combine two datasets. The residues in six trials were 0.03, 0.09, 0.18, 0.47, 1.19 and 2.03 mg/kg for myclobutanil and 0.05, 0.12, 0.23, 0.64, 1.39, 2.40 mg/kg for myclobutanil and RH-9090 and its conjugates. The Meeting estimated an HR 2.40 mg/kg, an STMR 0.435 mg/kg based on total residues, and maximum residue level of 3 mg/kg based on myclobutanil residues for peppers.

On the basis of residues in peppers and dehydration factor of 7, the Meeting estimated an HR of 16.8 mg/kg, an STMR of 2.45 mg/kg and recommended a maximum residue level of 40 mg/kg for myclobutanil on peppers chilli, dried.

Fruiting vegetables, Cucurbits

The critical GAP for myclobutanil on cucurbits is in the USA, 5 applications at 0.14 kg ai/ha with a PHI of 0 days.

Summer Squash

Nine outdoor trials were available from the USA on summer squash matching US GAP. Myclobutanil residues in squash were: 0.01(2), 0.02(2), 0.04, 0.05, 0.06, 0.10 and 0.16 mg/kg, and with total residue 0.01, 0.02, 0.03(2), 0.04, 0.05, 0.07, 0.11 and 0.16 mg/kg.

Cucumber

Seven outdoor trials were available from the USA on cucumbers matching US GAP. Myclobutanil residues were: 0.02, 0.03(2), 0.04(2) and 0.07 mg/kg, and with total residue of 0.03(2), 0.04 (3) and 0.07 mg/kg.

Melons

Four outdoor trials were available from the USA on melons matching US GAP with myclobutanil residue of 0.02, 0.05, 0.07 and 0.08 mg/kg, and with total residue of 0.02, 0.05, 0.07 and 0.08 mg/kg. Two outdoor trials were available from Southern Europe on melon matching US GAP with myclobutanil residues of 0.09 and 0.10 mg/kg, and total residue of 0.10 and 0.11 mg/kg.

The US GAP is the same for cucumbers, melons and squash. The Meeting considered that the residues from trials with the foliar application on cucumber, melon and squash were similar. The Meeting agreed to propose a group maximum residue level for fruiting vegetables, cucurbits. The residues expressed in terms of myclobutanil were: 0.01(2), 0.02(4), 0.03(2), 0.04(3), 0.05(2), 0.06, 0.07(2), 0.08, 0.09, 0.10(2) and 0.16 mg/kg. The residues expressed in terms of myclobutanil and RH-9090 and its conjugates were: 0.01, 0.02(2), 0.03(4), 0.04(4), 0.05(2), 0.07(3), 0.08, 0.10, 0.11(2) and 0.16 mg/kg.

Based on the trials for cucumbers, melons and squash in the USA and Southern Europe, the Meeting estimated an HR of 0.16 mg/kg, an STMR of 0.04 mg/kg and a maximum residue level of 0.2 mg/kg for fruiting vegetables, cucurbits respectively.

*Legume vegetables**Common bean*

The critical GAP for myclobutanil on snap beans is from the USA, i.e., 4×0.14 kg ai/ha with a PHI of 0 days. Nine trials were available from the USA on snap beans matching US GAP. Myclobutanil residues found were: 0.04, 0.09, 0.14, 0.19, 0.20, 0.22, 0.30, 0.37 and 0.47 mg/kg, and with total residues of 0.06, 0.11, 0.16, 0.21, 0.22, 0.24, 0.32, 0.39 and 0.49 mg/kg.

On the basis of the trials on snap beans, the Meeting estimated an HR of 0.49 mg/kg, an STMR 0.22 mg/kg based on the total residues dataset, and a maximum residue level of 0.8 mg/kg for beans, except broad bean and soya bean (green pods and immature seeds), respectively.

Soya bean (dry)

The critical GAP for myclobutanil on soya beans is from Brazil, 2×0.125 kg ai/ha with a PHI of 24 days. Nine trials were available from Brazil on soya bean matching Brazilian GAP with parent residues of < 0.01(5), < 0.02(2), 0.02 and 0.03 mg/kg.

Noting that total residues were not available from the Brazilian trials, the Meeting decided to not estimate a maximum residue level for soya bean.

Hops

The critical GAP for myclobutanil on hops is from the USA, 4×0.28 kg ai/ha with a PHI of 14 days. Ten trials were available from Germany on hops matching US GAP, myclobutanil residues in dried hops were: < 0.50, 0.53, 0.63, 0.73, 1.02, 1.06, 1.14, 1.54, 1.80 and 3.50 mg/kg, and with total residues of 0.53, < 1.00, 1.13, 1.23, 1.52, 1.56, 2.04, 3.20 and 5.60 mg/kg.

The Meeting estimated an HR of 5.60 mg/kg, an STMR 1.52 mg/kg based on the total residue dataset, and a maximum residue level of 5 mg/kg for dry hops based on myclobutanil residues to replace the previous recommendation of 2 mg/kg on dry hops.

Animal feedstuffs

The critical GAP for myclobutanil on soya bean is in the USA, 2× 0.14 kg ai/ha with PHI of 28 days.

Soya bean forage

Two trials were available from the USA on soya bean forage against US GAP with residue 0.67 and 1.01 mg/kg.

Two trials on soya bean forage were not sufficient for estimation of a median and highest residue.

soya bean hay (dry)

Noting no trials on soya bean hay were available from the USA against US GAP, the Meeting decided not to make recommendations for soya bean hay.

Rotational crops

The Meeting noted that myclobutanil may accumulate in soil and be taken up by follow-crops in significant amounts. Residues were reported in field rotational crop studies following application to zucchini at rates of 6× 0.14 kg ai/ha. Since the foliage of the crop was incorporated into soil after harvest of the fruits, this application rate can be assumed to approximate the estimated soil plateau level following annual treatment according to the GAPs considered by the Meeting.

Residues for parent myclobutanil in respective field studies were < 0.003 mg/kg for soya bean seeds, 0.03 mg/kg to 0.36 mg/kg for soya bean forage and 0.017 mg/kg to 0.093 mg/kg for soya bean straw. The Meeting concluded that for pulses no significant transfer into seeds is expected. Median and highest residues were estimated at levels of 0.195 mg/kg and 0.36 mg/kg for legume forages and of 0.055 and 0.093 mg/kg for legume fodders.

Based on the average dry-matter content of 85% for soya bean hay, the Meeting estimated a maximum residue level of 0.2 mg/kg (DM) for legume animal feeds.

In leaves of radish grown as rotational crops myclobutanil residues were 0.015 mg/kg to 0.044 mg/kg. Extrapolating the residues found in radish leaves to all Brassica vegetables and leafy vegetables (including leafy Brassica vegetables), the Meeting estimated maximum residue levels of 0.05 mg/kg and HR and STMR values of 0.044 mg/kg and 0.030 mg/kg for Brassica vegetables and leafy vegetables (including leafy Brassica vegetables), respectively.

The roots of radish grown as a rotational crop contained myclobutanil residues of 0.026 mg/kg to 0.052 mg/kg. The Meeting decided to extrapolate the results to all bulb vegetables and all root and tuberous vegetables. The Meeting estimated a maximum residue level of 0.06 mg/kg and HR and STMR values of 0.039 mg/kg and 0.052 mg/kg for bulb vegetables and root and tuber vegetables, respectively.

Residues in wheat matrices obtained from field rotational crop studies contained myclobutanil residues < 0.003 mg/kg in the grain, 0.023 mg/kg to 0.071 mg/kg in forage and 0.015 mg/kg to 0.18 mg/kg in hay and straw. The Meeting concluded that for cereal grains no significant transfer of residues into seeds is expected. Median and highest residues were estimated at levels of 0.047 mg/kg and 0.071 mg/kg for cereal forages and of 0.098 and 0.18 mg/kg for cereal straw and fodder.

Based on the average dry-matter content of 88% for wheat straw, the Meeting estimated a maximum residue level of 0.3 mg/kg (DM) for straw and fodder (dry) of cereal grains.

The Meeting also concluded that the contribution of residues by uptake of myclobutanil from soil is insignificant compared to direct treatment for the uses evaluated.

Fate of residues during processing

The Meeting received information on the hydrolysis of myclobutanil as well as processing studies during the food processing of apples, grapes, tomatoes, soya beans and hops.

No degradation of myclobutanil was observed in a hydrolysis study at pH 4, 7 and 9 held at 50 °C, over a 5 day period.

In the following table all processing factors based on parent residues relevant for recommendation of maximum residue levels and estimation of animal dietary burden for processed commodities are summarized.

Portion Analysed	Median Processing Factor	Median residues	
		Raw commodities	Processed commodities
Apple		0.06	
Wet pomace	1.61		0.097
Dry pomace	12.1		0.726
Grapes		0.295	
Wet pomace	0.90		0.266
Dry pomace	3.94		1.16
Raisin	6.31		1.86
Tomato		0.07	
Dry pomace	20.05		1.40
Wet pomace	4.54		0.318
soya bean		0	
Hulls	1.14		0
Meal	0.43		0
Oil, refined	2.14		0
Hops		1.04	
Beer	0.0145		0.015

Based on the median processing factor of 6.31, the Meeting estimated a maximum residue level of 6 mg/kg for raisin.

In the following table all processing factors based on total residues relevant for the estimation of the dietary intake for processed commodities are summarized.

Raw and processed commodity	Median Processing Factor	STMR (mg/kg)	STMR-P (mg/kg)	HR (mg/kg)	HR-P (mg/kg)
Apple		0.07			
Juice	0.17		0.012		
Puree	0.30		0.021		
Grapes		0.34		0.60	
Juice	0.20		0.068		
Wine after half year	0.17		0.058		
Wine at bottling	0.14		0.048		
Raisin	6.29		2.14		3.77
Tomato		0.07			
Juice	0.50		0.031		
Puree	1.33		0.093		
Preserve	0.29		0.020		
Paste	3.92		0.27		
Hops Beer	0.015	1.52	0.023		

Residues in animal commodities

Estimated maximum and mean dietary burdens of farm animals

Dietary burden calculations for beef cattle, dairy cattle, broilers and layers are provided in Annex 6 to the 2014 Report. The calculations were made according to the animal diets from US-Canada, EU, Australia and Japan in the OECD Feed Table 2009.

The calculations are then summarised and the highest dietary burdens are selected for MRL and STMR estimates on animal commodities.

	Animal dietary burden, myclobutanil, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	0.046	0.26	1.22	0.83	1.47 ^a	0.96 ^b	0.096	0.055
Dairy cattle	0.63	0.45	0.86	0.58	1.31 ^c	0.88 ^d	0.18	0.10
Poultry-broiler	0	0	0.035	0.026	0	0	0.051	0.028
Poultry-layer	0	0	0.22 ^e	0.13 ^f	0	0	0	0

^a Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian meat.

^b Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian meat.

^c Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk.

^d Highest mean dairy cattle dietary burden suitable for STMR estimates for mammalian milk.

^e Highest maximum poultry dietary burden suitable for MRL estimates for poultry meat and eggs.

^f Highest mean poultry dietary burden suitable for STMR estimates for meat and eggs.

Lactating cows were orally administered myclobutanil equivalent to 0 ppm, 1.6 ppm, 4.8 ppm and 16 ppm in the feed, respectively. Residues of myclobutanil and RH-9090 in whole milk and tissues of the cows in all groups were < 0.01 mg/kg.

Dietary burden calculations showed the highest dietary burdens were less than the lowest feeding level. The Meeting decided to estimate maximum residue levels of 0.01*, and STMRs and HRs of 0 mg/kg for all milk, eggs and animal tissues. Confirming the previous recommendations of 0.01* mg/kg for cattle meat, milk and edible offal, and eggs, poultry meat and edible offal of the 1992 JMPR.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue concentrations listed below are suitable for establishing MRLs and for assessing IEDIs and/or IESTIs.

Definition of the residue (for compliance with the MRL for plant and animal commodities and for estimation of dietary intake for animal commodities): *myclobutanil*.

Definition of the residue (for estimation of dietary intake for plant commodities): *sum of myclobutanil, α -(4-chlorophenyl)- α -(3-hydroxybutyl)-1H-1,2,4-triazole-1-propanenitrile (RH-9090) and its conjugates, expressed as myclobutanil*.

CCN	Commodity	MRL, mg/kg		STMR or STMR-P, mg/kg	HP or HR-P, mg/kg
		new	previous		
FL 0327	Banana	W	2		
VP 0061	Beans, except broad bean and soya bean	0.8		0.22	0.49
VB 0040	Brassica (Cole or Cabbage) Vegetables, Head Cabbage, Flowerhead Brassicas	0.05		0.03	0.044
VA 0035	Bulb vegetables	0.06		0.039	0.052
FS 0013	Cherries	3		1.06	2.05
FB 0021	Currants, Black, Red, White	0.9	0.5	0.34	0.47

CCN	Commodity	MRL, mg/kg		STMR or STMR-P, mg/kg	HP or HR-P, mg/kg
		new	previous		
DF 0269	Dried grapes (=currants, Raisins and Sultanas)	6		2.14	3.77
MO 0105	Edible offal (mammalian)	0.01*		0	0.01
PE 0112	Eggs	0.01*		0	0.01
VC 0045	Fruiting vegetables, Cucurbits	0.2		0.04	0.16
FB 0269	Grapes	0.9	1	0.34	0.6
DH 1100	Hops, dry	5	2	1.52	5.6
VL 0053	Leafy vegetables	0.05		0.03	0.044
AL 0157	Legume animal feeds	0.2		0.055	0.093
MF 0100	Mammalian fats (except milk fats)	0.01*		0	0
MM 0095	Meat (from mammals other than marine mammals)	0.01*	0.01*	0	0.01
ML 0106	Milks	0.01*	0.01*	0	0.01
FS 2001	Peaches	3	-	0.865	1.54
VO 0051	Peppers	3	-	0.435	2.4
HS 0444	Peppers Chili, dried	30	-	4.35	24
FS 0014	Plums (including prune)	2	0.2	0.4	1.45
FP 0009	Pome fruits	0.6	0.5	0.07	0.35
PF 0111	Poultry fats	0.01*		0	0
PM 0110	Poultry meat	0.01*	0.01*	0	0.01
PO 0111	Poultry, Edible offal of	0.01*	0.01*	0	0.01
DF 0014	Prunes	W	0.5		
VR 0075	Root and tuber vegetables leaves	0.06	-	0.039	0.052
FS 0012	Stone fruits (except plums)	W	2		
AS 0081	Straw and fodder (dry) of cereal grains	0.3	-	0.098	0.18
FB 0275	Strawberry	0.8	1	0.19	0.69
VO 0448	Tomato	0.3	0.3	0.07	0.25
JF 0226	Apple juice			0.012	
	Beer			0.023	
JF 0269	Grape juice			0.068	
	Grape wine			0.048	
JF 0448	Tomato juice			0.031	
VW 0448	Tomato paste			0.27	
	Tomato preserve			0.02	
	Tomato purée			0.093	

* At or about the limit of quantification.

Animal commodities and processed foods for which no maximum residue levels were recommended:

CCN	Commodity	Median residue, mg/kg	Highest residue, mg/kg
—	Apple pomace, wet	0.097	
—	Apple pomace, dry	0.726	
—	Cereal forage	0.047	0.071
—	Grapes pomace, wet	0.266	
AB 0269	Grapes pomace, dry	1.16	
—	Legume forage	0.195	0.36
—	Tomato pomace, wet	0.318	
—	Tomato pomace, dry	1.40	

DIETARY RISK ASSESSMENT

Long term intake

The evaluation of myclobutanil resulted in recommendations for MRLs and STMR values for raw and processed commodities. Data on consumption were available for 33 food commodities and were used to calculate dietary intake. The results are shown in Annex 3 to the 2014 Report.

The International Estimated Daily Intakes (IEDIs) of myclobutanil, based on the STMRs estimated, represented 1–6% of the upper bound of the maximum ADI of 0.03 mg/kg bw for the 17 GEMS/Food cluster diets. The Meeting concluded that the long-term intake of residues of myclobutanil resulting from its uses that have been considered by JMPR was unlikely present a public health concern.

Short-term intake

The 2014 Meeting established an ARfD of 0.3 mg/kg bw for women of childbearing age only; ARfD was unnecessary for the general population.

The International Estimated Short Term Intake (IESTI) for myclobutanil was calculated for the food commodities for which STMRs, HRs and maximum residue levels were estimated by the current Meeting and for which consumption data were available. The results are shown in Annex 4 to the 2014 Report. The IESTI represented up to 10% (peach) for women of childbearing age only. The Meeting concluded that the short-term intake of residues of myclobutanil resulting from uses considered by the current Meeting was unlikely to present a public health concern.

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123466	Falconer, R	1987	Stone Fruit Fungicide Residue Trial At Fresno, California 11445
123467	Falconer, R	1987	Stone Fruit Fungicide Residue Trial At Traver, California 11446
123468	Falconer, R	1987	Stone Fruit Fungicide Residue Trial At Laton, California 11447
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123933	Harrison, WE	1988	Dithane M-45 Fungicide Peach Field Residues At Hartsfield, Georgia 12468
124717	Klauzer, JC	1988	RH-3866 60DF Cherry Field Residues At Nampa, Idaho 12668
124718	Klauzer, JC	1988	RH-3866 40WP Cherry Field Residues At Nampa, Idaho 12669
124719	Klauzer, JC	1988	RH-3866 2E Cherry Field Residues At Nampa, Idaho 12670
124753	Harrison, WE	1988	RH-3866 Fungicide Peach Field Residue At Georgia 12705
124754	Harrison, WE	1988	RH-3866 Peach Field Residues At Georgia 12704
124755	Harrison, WE	1988	RH-3866 Peach Field Residues At Georgia 12706
124764	Harrison, WE	1988	RH-3866 Peach Field Residues At Georgia 12715
124765	Harrison, WE	1988	RH-3866 Peach Field Residues At Georgia 12716
124766	Harrison, WE	1988	RH-3866 Peach Field Residues At Georgia 12717
124769	Neidlinger, TJ	1988	Cherry Residue Trial Nova At Washington 12720
124770	Neidlinger, TJ	1988	Cherry Residue Trial Nova At Washington 12721
124771	Neidlinger, TJ	1988	Cherry Residue Trial Nova At Washington 12722
124774	Neidlinger, TJ	1988	Peach Residue Trial Nova At Washington 12725
124775	Neidlinger, TJ	1988	Peach Residue Trial Nova At Washington 12726
124779	Neidlinger, TJ	1988	Cherry Residue Trial Nova At Washington 12730
124781	Neidlinger, TJ	1988	Cherry Residue Trial/Nova 2E At Zillah, Washington 12732
124800	West, LD	1988	RH-3866 Residue Trial In Peaches At California 12751
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125168	Thilsted, WE	1988	RH-3866 60DF Peach Field Residue Experiment At Pontiac, South Carolina 12819
125169	Thilsted, WE	1988	RH-3866 40W Peach Field Residue Experiment At Pontiac, South Carolina 12820
125170	Thilsted, WE	1988	RH-3866 2E Peach Field Residue Experiment At Pontiac, South Carolina 12821
125177	Holowid, JR	1988	RH-3866 60DF Peach Field Residues At Tecumseh, Michigan 12828
125178	Holowid, JR	1988	RH-3866 40W Peach Field Residues At Tecumseh, Michigan 12829
125179	Holowid, JR	1988	RH-3866 2E Peach Field Residues At Tecumseh, Michigan 12830
125187	Holowid, JR	1988	RH-3866 Cherry Field Residues At Michigan 12838
125188	Holowid, JR	1988	RH-3866 Cherry Field Residues At Michigan 12839
125189	Holowid, JR	1988	RH-3866 Cherry Field Residues At Michigan 12840
125203	West, LD	1988	RH-3866 Residue Trial In Cherries At California 12854
125204	West, LD	1988	RH-3866 Residue Trial In Cherries At California 12855
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124907	Holowid, JR	1989	RH-3866 Concentrate Vs. Dilute Cherry Residue Study At Michigan 13059
125209	West, LD	1989	RH-3866 40W Residue Trial In Cherries At Linden, California 12860
125210	Keathley, JP and West, LD	1989	RH-3866 60DF Residue Trial In Cherries At Linden, California 12861
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241909	Mestres, R	1985	Myclobutanil: Residue Data From An Individual Trial—Grapes, 1985 431.85.39; 431.85.44
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242085	Mestres, R	1986	Myclobutanil: Residue Data From An Individual Trial—Grapes, 1986 431.86.14; S-12
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242093	Mestres, R	1986	Myclobutanil: Residue Data From An Individual Trial—Grapes, 1986 431.86.66; S-25
242098	Mestres, R	1986	Myclobutanil: Residue Data From An Individual Trial—Grapes, 1986 431.86.66; S-20
242103	Mestres, R	1986	Myclobutanil: Residue Data From An Individual Trial—Grapes, 1986 431.86.65; S-30
242105	Mestres, R	1986	Myclobutanil: Residue Data From An Individual Trial—Grapes, 1986 431.86.15A; S-33
242106	Mestres, R	1986	Myclobutanil: Residue Data From An Individual Trial—Grapes, 1986 431.86.15B; S-32
242076	Mestres, R	1987	Myclobutanil: Residue Data From An Individual Trial—Grapes, 1986 491.86.13; SE-21
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94327	Burnett, TF, Deakyne, RO, Brackett, CK and Stavinski, SS	1986	RH-3866 Residue Data For Pears, 85-0120, 85-0314, 85-0393, 85-0412, 85-0421, 85-0356 31A-86-61
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94358	Burnett, TF, Deakyne, RO, Brackett, CK and Stavinski, SS	1986	RH-3866 Residue Decline Studies In Pears 31A-86-60
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124738	Jaeger, JJ	1988	RH-3866 On Apples For Residues Sprayed Dilute And Concentrate At Virginia 12688
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94951	Ackermann, IB	1984	Laboratory Leaching Study With RH-3866 TR 31O-84-09
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PHOSMET (103)

The first draft was prepared by Dr Guibiao Ye, Institute for the Control of Agrochemicals, Ministry of Agriculture, Beijing, China

EXPLANATION

Phosmet is a broad-spectrum organo-phosphorus insecticide used to control a variety of insect and mite pests which attack berries, pome, stone and citrus fruit. It is also used on field, pasture and forage crops. Phosmet is non-systemic and acts by contact and ingestion as a cholinesterase inhibitor. It is registered in a number of countries, mainly for protecting fruits and vegetables.

Phosmet was evaluated for residues several times by the JMPR from 1976 to 2007, and was evaluated under the periodic review in 1994 for toxicity and in 1997 for residue. The ADI of 0-0.01 mg/kg bw was established in 1994. The ARfD of 0.2 mg/kg bw was established in 2003. Codex MRLs for cotton seed, grapes, apricots/peaches/nectarines, potatoes, tree nuts were established in 2002, and Codex MRLs for apricot, blueberries, citrus fruits, nectarine and pome fruits were established in 2007.

GAP and supervised trials data were submitted to the 2014 JMPR for cranberry.

USE PATTERN

Information on registered uses was reported to the Meeting and is shown in Table 1.

Table 1 Registered uses for phosmet on cranberries

Crop	Country	Form.	Application					PHI days
			Method	Rate kg ai/ha	Spray conc. kg ai/hL	Water L/ha	No.	
Cranberries	USA	70 WSB	Foliar spraying	1.1-3.1	1.68-16.8		4	14

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received new information on supervised field trials on cranberries. Residue data are reported in Tables 2. Five supervised trials were conducted on cranberries in the USA in 1996 (Ref: R04625, DOC. NO.: 632-4301). A wettable powder formulation containing phosmet at 70% w/w was applied to cranberries at a nominal rate of 3.36 kg ai/ha. Four applications were made at intervals of approximately 10 days beginning after bloom with the last to mature fruits, 14 days before harvest. Duplicate samples of berries were collected 14 days after the last application. Mean residues of the duplicate samples are calculated.

The study report also includes residues of phosmet-oxon in addition to residues of phosmet. The results for phosmet-oxon are presented (all are below the LOQ) but not used further.

Results have not been corrected for concurrent method recoveries. Residues and application rates have generally been rounded to two significant figures. Although trials included control plots, residues of all controls are below LOQ.

The samples were frozen immediately after collection and stored at approximately -20 °C until extraction and analysis. The maximum storage period was 88 days (3 months). Residues of phosmet and phosmet-oxon were determined by GC-FPD using Stauffer Chemical company method WRC 73-43 (modified as Morse Laboratories Method Meth-14, Revision 3.). The LOQ of the method was 0.05 mg/kg. Mean procedural recoveries for phosmet at fortification levels of 0.05–10 mg/kg were within the acceptable range of 70–120%, RSD < 20%. The stability of phosmet in cranberry was confirmed by laboratory fortified samples (3× 1.0 mg/kg) that were stored in parallel with field samples. The average stability of phosmet after this period of 88 days was found to be 93%.

Because of the low phosmet oxon residues detected (<10% of parent), the 1997 JMPR decided that the definition of the residue for compliance with the MRL and for the estimation of dietary intake is phosmet.

Table 2 Phosmet residues in cranberries from supervised trials in USA

Country, year, variety	Application					Commodity analysed	PHI (days)	Residues ^a (mg/kg)	
	Form	kg ai/ha	Water L/ha	kg ai/hL	No.				
USA Wisconsin Rapids 1996 Ben Lear	700 WP	3.36 3.36 3.36 3.36	213 215 215 217	1.58 1.56 1.56 1.55	4	berries	14	0.82 0.88 [0.85]	
USA Biron, Wisconsin 1996 Ben Lear,	700 WP	3.36 3.36 3.36 3.36	213 215 215 217	1.58 1.56 1.56 1.55	4	berries	14	0.77 0.90 [0.84]	
USA Chatsworth, New Jersey 1996 Early black	700 WP	3.25 3.25 3.25 3.25	347 357 352 354	0.94 0.91 0.92 0.92	4	berries	14	0.12 0.14 [0.13] ^b	
USA Chatsworth, New Jersey 1996 Early black	700 WP	3.36 3.14 3.14 3.25	365 341 340 350	0.92 0.92 0.92 0.93	4	berries	14	0.085 0.083 [0.084]	
USA Long Beach, Washington 1996 McFarlin	700 WP	3.36 3.25 3.47 3.47	203 132 245 133	1.66 2.46 1.42 2.62	4	berries	14	0.91 0.84 [0.88]	

^a Residues in [] were mean of results from two duplicate sample.

^b Higher residue from two dependent trials was considered and used.

APPRAISAL

Phosmet is a broad-spectrum organo-phosphorus insecticide used to control a variety of insect and mite pests which attack berries, pome, stone and citrus fruit. It is also used on field, pasture and forage crops. Phosmet is non-systemic and acts by contact and ingestion as a cholinesterase inhibitor. It is registered in a number of countries, mainly for protecting fruits and vegetables.

Phosmet was evaluated for residues several times by the JMPR from 1976 to 2007, and was evaluated under the periodic review in 1994 for toxicity and in 1997 for residue. The ADI of 0–0.01 mg/kg bw was established in 1994. The ARfD of 0.2 mg/kg bw was established in 2003. Existing residue definitions for phosmet were set at the 1997 JMPR Meeting. For plant and animal commodities, the residue definition for enforcement and dietary risk assessment is phosmet.

Results of supervised residue trials on crops

Cranberry

GAP and supervised trials data on cranberry were received and evaluated by the current Meeting. Phosmet is registered in the USA for use on cranberry at 1.1–3.1 kg ai/ha with a PHI of 14 days. Five supervised trials were conducted on cranberries in the USA in 1996. The residues of phosmet from four independent trials were: 0.13, 0.84, 0.85, 0.88 mg/kg, with the highest residue of 0.91 mg/kg from a replicate sample in one trial.

The Meeting estimated a maximum residue level of 3 mg/kg, a HR of 0.91 mg/kg, and an STMR of 0.845 mg/kg for cranberries.

RECOMMENDATIONS

On the basis of the data from independent supervised trials, the Meeting concluded that the residue concentrations listed below are suitable for establishing MRLs and for assessing IEDIs and IESTIs.

Definition of the residue:

For compliance with MRLs and for estimation of dietary intake: Phosmet.

Commodity		MRL, mg/kg	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
CCN	Name			
FB 0265	Cranberry	3	0.845	0.91

DIETARY RISK ASSESSMENT

Long-term intake

The estimates of long-term dietary intake for phosmet (ADI 0–0.01 mg/kg bw) were calculated by the current meeting for the 17 regional diets on the basis of the STMRs estimated by the JMPR in 2002, in 2007 and in 2014. The results are shown in Annex 3 of the 2014 Report.

The International Estimated Daily Intakes (IEDI) of phosmet, based on estimated STMRs from the uses were 3–90% (rounded) of the maximum ADI (0.01 mg/kg bw). The Meeting concluded that the long-term intake of residues of phosmet from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short Term Intake (IESTI) of phosmet was calculated for cranberry. The results are shown in Annex 4 to the 2014 Report.

The IESTI of phosmet calculated on the basis of the recommendations made by current meeting represented 0–3% of the ARfD (0.2 mg/kg bw) for children, 0–2% for the general population. The Meeting concluded that the short-term intake of residues of phosmet resulting from uses in cranberry that have been considered by the JMPR is unlikely to present a public health concern.

REFERENCES

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PROPAMOCARB (148)

The first draft was prepared by Dr Guibiao Ye, Institute for the Control of Agrochemicals, Ministry of Agriculture, Beijing, China

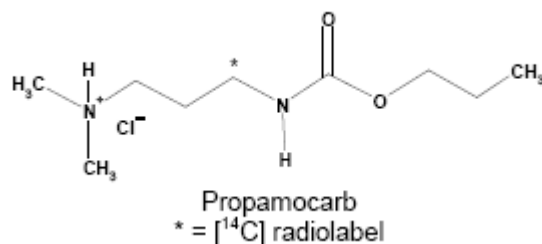
Propamocarb hydrochloride is a systemic carbamate fungicide with specific activity against Oomycete species that cause seed, seedling, root, foot and stem rots and foliar diseases in a number of edible crops. The compound was firstly evaluated by the JMPR in 1984, and was then reviewed by JMPR in 1986, 1987 and 2005, an ADI of 0–0.4 mg/kg bw and an ARfD of 2 mg/kg bw were established in 2005. Propamocarb was evaluated by the 2006 JMPR for residue and analytical aspect within the periodic review programme. The residue definition in plant for both enforcement and dietary intake purpose is propamocarb (free base), and the residue definition in animal products for both enforcement and dietary intake purpose is propamocarb, and propamocarb is not fat soluble.

The residue data in/on onion, broccoli, cauliflower, Brussels sprouts, head cabbage, kale, leek and lima bean was submitted to JMPR, and were evaluated by current Meeting. In addition, new analytical methods as well as new studies on animal metabolism and feeding, rotational crops and processed commodities are received and evaluated.

ANIMAL METABOLISM

Laying hen

The metabolism and distribution of propamocarb in laying hens were studied using [^{14}C]-propamocarb hydrochloride by Beedle and Miller (2010, MEPRX029, M-366633-01-1). The structural formula and the position of the ^{14}C label are shown below.



Twelve laying hens were dosed orally, via capsule, with [^{14}C] propamocarb hydrochloride at a rate of 1.02 mg/kg body weight (approximately 19 ppm in the feed) once daily for fourteen consecutive days. Eggs were collected once daily during the treatment period. Approximately 6 to 8 hours after the last dose, the hens were sacrificed, and edible tissues (liver, leg muscle, breast muscle, omental fat, subcutaneous fat, and renal fat) were collected. Tissue and egg samples were frozen immediately after collection and all of the samples were analysed within 6 months.

The total radioactive residue (TRR) in the egg and tissues was determined by oxidative combustion followed by liquid scintillation counter (LSC) or by direct LSC. Identification and quantitation of the residues in the egg and tissue extracts were accomplished by using reverse phase high performance liquid chromatography (HPLC) and liquid chromatography-mass spectrometry/mass spectrometry (LC-MS/MS).

Eggs, liver and muscle samples were extracted with acetonitrile/water (4:1) containing 1% acetic acid, blended, centrifuged and decanted from the solids. The extraction and centrifugation were repeated five times. The combined acetonitrile/water/acetic acid extract was radioassayed. The acetonitrile/water extracted solids were refluxed overnight in acetonitrile/water (4:1) containing 1% acetic acid. After cooling, centrifugation and decantation, the acetonitrile/water/acetic acid reflux extract was radioassayed. If un-extracted radioactivity remained in the solids (eggs), the solids were refluxed with extraction solvent followed by a dilute base (2 N KOH). Un-extracted residues in solids of the liver were enzymatically released by agitation with protease at 37 °C and pH 7.4 overnight. All

the extracts were combined and concentrated via rotary evaporator. The sample was radioassayed, and an aliquot was analysed by HPLC.

Fat samples were extracted with hexane, blended, centrifuged and decanted from the solids. The extraction and centrifugation were repeated once. The hexane extracts were combined and radioassayed. The combined hexane extract was partitioned three times with acetonitrile. The combined acetonitrile layers were radioassayed. After that, water was added to the hexane extract two times for a partition. The combined water layers and the hexane extract were radioassayed. The hexane-extracted solids were homogenized with acetonitrile/water (4:1) containing 1% acetic acid, blended, centrifuged and decanted from the solids. The extraction and centrifugation were repeated three times. The acetonitrile/water extracts were combined, radioassayed, and concentrated to an aliquot which was analysed by HPLC. The acetonitrile/water extracted solids were air-dried overnight at room temperature and aliquots were radioassayed. The hexane extract was concentrated to an oily residue via rotary evaporator. An aliquot of 2N potassium hydroxide (KOH) in ethanol was added to the concentrated sample. The sample was refluxed, cooled to room temperature and water was added to the sample which was partitioned two times with hexane. The combined hexane layers were back-extracted with water. The hexane fraction and the water fraction were radioassayed.

The TRR in eggs increased from day-1 through day-7 and reached a plateau at day-8. The TRR (expressed in Propamocarb hydrochloride equivalents) in the egg samples ranged from 0.015 mg/kg in the day-1 eggs to 0.254 mg/kg in the day-11 eggs, at maximum.

The TRR in the tissue samples from the hens dosed with Propamocarb hydrochloride for fourteen consecutive days was 0.492 mg/kg in liver, 0.135 mg/kg in leg muscle, 0.117 mg/kg in breast muscle, 0.042 mg/kg in omental fat, 0.042 mg/kg in subcutaneous fat and 0.065 mg/kg in renal fat.

The majority of the residues (92% to 99% of the TRR) in the egg and tissues were extractable and only 1% to 8% of the TRR (0.001 to 0.038 mg/kg) of the residues in egg and tissues were unextractable. The major residues in the egg and tissues were parent propamocarb (2% to 12% of the TRR; < 0.001 to 0.046 mg/kg) and desmethyl propamocarb (6% to 45% of the TRR; 0.003 to 0.113 mg/kg). The minor residues found in the egg and tissues were bis-desmethyl-propamocarb (< 1% to 7% of the TRR; < 0.001 to 0.029 mg/kg) and propamocarb-N-oxide (< 1% of the TRR; 0.003 mg/kg). The results of these analyses are presented in Table 1.

Table 1 Summary of characterization and identification of radioactive residues in hen matrices following the oral administration of [^{14}C] Propamocarb at a rate of 1.02 mg ai/kg body weight/day for fourteen consecutive days

Compound	Day-9 Egg TRR = 0.252 mg/kg		Liver TRR = 0.492 mg/kg		Muscle TRR = 0.126 mg/kg		Fat TRR = 0.043mg/kg	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
E1, F1	4	0.010	ND	ND a)	ND	ND	8	0.004
M1-a	ND	ND			16	0.020	ND	ND
M1-b, L1-a	ND	ND	1	0.007	8	0.010	ND	ND
L1-b	ND	ND	5	0.025	ND	ND	ND	ND
L1-c	ND	ND	9	0.045	ND	ND	ND	ND
L1-d	ND	ND	3	0.013	ND	ND	ND	ND
L2-a	ND	ND	<1	0.004	ND	ND	ND	ND
L2-b	ND	ND	<1	0.001	ND	ND	ND	ND
L2-c	ND	ND	<1	0.003	ND	ND	ND	ND
M2, F2	ND	ND	ND	ND	6	0.007	2	0.001
Propmaocarb-N-oxide (L2-d)	ND	ND	<1	0.003	ND	ND	ND	ND
Bis-desmethyl Propamocarb (E2, L3, F3, M3)	4	0.010	6	0.029	7	0.008	< 1	<0.001
Desmethyl-Propamocarb (E3, L3, F4, M4)	45	0.113	22	0.107	29	0.037	6	0.003
Propamocarb (E4, L5, F5, M5)	12	0.031	9	0.046	5	0.006	2	<0.001

Compound	Day-9 Egg TRR = 0.252 mg/kg		Liver TRR = 0.492 mg/kg		Muscle TRR = 0.126 mg/kg		Fat TRR = 0.043mg/kg	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
F6	ND	ND	ND	ND	ND	ND	3	0.001
E5, F7, M6	2	0.005	ND	ND	6	0.008	<1	<0.001
L6, F8	ND	ND	3	0.015	ND (a)	ND (a)	1	<0.001
L7, F9, M7	ND	ND	8	0.038	5	0.006	2	<0.001
E6	4	0.009	ND	ND	ND	ND	ND	ND
Total Identified	61	0.154	37	0.185	41	0.051	9	0.004
Total Characterized	38	0.096	55	0.269	51	0.066	89	0.038
Total extractable	99	0.250	92	0.454	92	0.117	98	0.042
Unextracted	1	0.003	8	0.038	8	0.010	2	0.001
Accountability	103		105		112		112	

ND = Not Detected

Metabolic Pathway in hen

Metabolic degradation of propamocarb is rapid and extensive, with between 2% and 12% of the TRR being observed in the egg and tissues as unmetabolized parent propamocarb. Demethylation products including desmethyl-propamocarb and bis-desmethyl-propamocarb were identified as major metabolites in egg and tissue samples, indicating that demethylation is the main route of metabolism for the parent compound. A minor route of metabolism involves oxidation of the tertiary nitrogen to form propamocarb-N-oxide. The proposed metabolic pathway for the metabolism of propamocarb in hens is shown in Figure 1 below

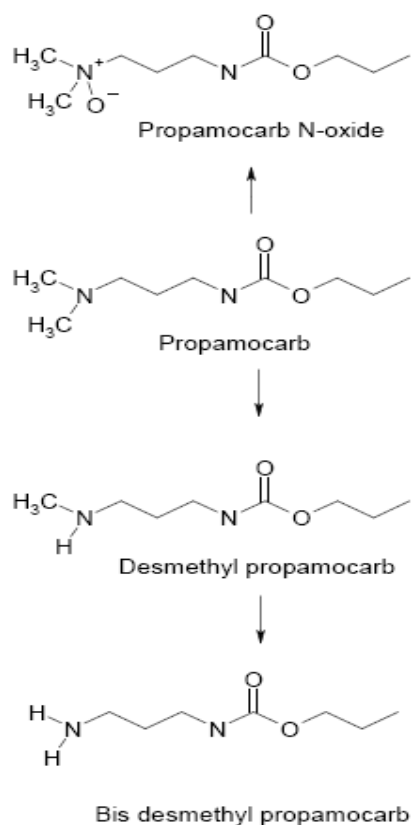


Figure 1 Proposed metabolic pathway of Propamocarb in Hens

Field crop rotational studies

Studies were conducted in Germany and France (Klein, 2004, M-226597-01-1) during 2002 and 2003, with four trials to investigate the residue level of propamocarb hydrochloride in secondary crops (Lamb's lettuce with a PBI of 52–59 days and wheat with a PBI of 81–102 days) after application to cabbages as the primary crop. No residues of propamocarb were found in any sample taken from the rotational crops, i.e., lettuce, wheat grain, wheat (green material) and wheat forage.

Cabbage plants were treated twice by drench applications to seedbeds. The first application was carried out at an application rate of 72.2 kg/ha of propamocarb hydrochloride at BBCH 00–11, directly after sowing and the second drench application 14 days after the first at an application rate of 36.1 kg/ha of propamocarb hydrochloride at BBCH 10–13. After transplanting of the young cabbage plants into the field, two additional foliar treatments were carried out at BBCH 15–45. Both spray treatments were applied at 3.61 kg/ha of propamocarb hydrochloride, the first immediately after transplanting and the second 39 to 66 days later at BBCH 41–45 (20 to 50% of the expected head size reached). Samples of cabbage were taken at day 0, 7, 14, 21 and 27–29 after the last application. After the harvest of the primary crop, the following (rotational) crops wheat and lamb's lettuce were sown in the plots.

Samples of lamb's lettuce were taken at day 111–267 (BBCH 46) and day 217–273 (BBCH 49) after the last treatment. In trial 02R460-4 in France, lamb's lettuce was abandoned due to poor germination and poor plant growth. Samples of wheat (green material) were taken at day 308–348 (BBCH 71) after the last application, wheat grain and wheat straw at day 362–369 (BBCH 89). Samples of soil were taken directly after the last application, at the time of lettuce sowing (day 52–59), at the time of wheat sowing (day 81–102), at the time of lamb's lettuce harvest (day 217–273) and at the time of wheat harvest (day 362–369).

Samples of cabbage, lamb's lettuce, wheat grain, wheat (green material) and wheat straw were analysed for residues of Propamocarb (free base) according to method 00880 by LC-MS/MS (Mende P., 2001; M-200511-02-1 or C015449) and soil samples were analysed for residues of Propamocarb (free base) according to method 20001397/01-RVS by LC-MS/MS (Mende P., 2002; M-215230-01-1 or C023560). The limit of quantification (LOQ) for Propamocarb hydrochloride was established and validated at 0.01 mg/kg in/on cabbage (head), lamb's lettuce (whole plant with root) and wheat grain, at 0.02 mg/kg for soil and 0.05 mg/kg for wheat green material and wheat straw.

Table 2 Residues in rotational crops after applications of propamocarb in the primary crop cabbage

Crop (Variety)	Country Site	Application				Residues			
		No	kg/ha (ai)	kg/hL (ai)	GS	Portion analysed	DAT ^a (days)	PCH ^b (mg/kg)	PMB ^c (mg/kg)
Cabbage, white (Perfekta) L Lettuce (Vita) Wheat (Magnus)	Germany Gersthofen	4	3.61-72.2	0.181-0.60	42	cabbage, head	0	0.07	0.06
							7	0.07	0.06
							14	0.06	0.05
							21	0.03	0.03
							28	0.03	0.03
						lettuce ^d	241	<0.01	<0.01
							264	<0.01	<0.01
							348	<0.05	<0.05
							369	<0.01	<0.01
							369	<0.05	<0.05
						soil	0	0.21	0.18
							59	<0.02	<0.02
							102	<0.02	<0.02
							264	<0.02	<0.02
							369	<0.02	<0.02

Crop (Variety)	Country Site	Application				Residues			
		No	kg/ha (ai)	kg/hL (ai)	GS	Portion analysed	DAT ^a (days)	PCH ^b (mg/kg)	PMB ^c (mg/kg)
Cabbage, white (Agressor) L Lettuce (Vit) Wheat (Drifter)	Germany Bornheim	4	3.61-72.2	0.181-0.72	41	cabbage, head	0	2.1	1.8
							7	0.09	0.08
							14	0.05	0.04
							21	0.03	0.03
							29	0.02	0.02
						lettuce ^d	111	<0.01	<0.01
							217	<0.01	<0.01
						wheat ^e	317	<0.05	<0.05
						wheat grain	362	<0.01	<0.01
						wheat straw	362	<0.05	<0.05
						soil	0	0.76	0.64
							54	<0.02	<0.02
							82	<0.02	<0.02
							217	<0.02	<0.02
							362	<0.02	<0.02
Cabbage, white (Kilor) L Lettuce (Dunkel-grüner vollherzi-ger) Wheat (Charger)	Germany Vechta-Langfoerden	4	3.61-72.2	0.181-0.60	43	cabbage, head	0	5.0	4.2
							7	0.16	0.13
							14	0.12	0.10
							21	0.06	0.05
							27	0.06	0.05
						lettuce ^d	267	<0.01	<0.01
							273	<0.01	<0.01
						wheat ^e	326	<0.05	<0.05
						wheat grain	367	<0.01	<0.01
						wheat straw	367	<0.05	<0.05
						soil	0	1.2	1.0
							52	<0.02	<0.02
							81	<0.02	<0.02
							273	<0.02	<0.02
							367	<0.02	<0.02
Cabbage, white (Destiny) L Lettuce (Dunkel-grüner vollherzi-ger) Wheat (recital)	France Soucelles	4	3.61-72.2	0.181-0.72	45	cabbage, head	0	2.6	2.2
							7	0.38	0.32
							14	0.24	0.20
							21	0.24	0.20
							28	0.07	0.06
						wheat ^e , wheat grain wheat straw	308	<0.05	<0.05
							362	<0.01	<0.01
							362	<0.05	<0.05
						soil	0	0.30	0.25
							57	0.04	0.03
							98	<0.02	<0.02
							362	<0.02	<0.02

^a DALT: Days after last treatment^b PCH = Propamocarb hydrochloride^c PMB = Propamocarb^d lettuce, whole plant with root^e wheat green material

Table 3 Recovery of Propamocarb in lamb's lettuce (whole plant with root), white cabbage (head), wheat (green material, grain and straw) and in soil*

Study/Trial No., Trial SubID, GLP, Year	Crop / plant origin	Portion analysed	Active substance	Fortification level (mg/kg)	Recovery (%) Single values		n	Mean	RSD			
02R460 02R460-1 02R460-2 02R460-3 02R460-4 GLP yes 2002	Lamb's lettuce	Whole plant with root	Propamocarb	0.010	91	95	5	93	4.1			
				0.100	94	98	5	93	8.1			
					103	88						
					84							
					91	88						
	Overall Recovery						10	93	6.1			
	White cabbage	Head	Propamocarb	0.010	78	79	2	79	-			
				0.100	80	80	2	80	-			
				6.0	103		1	103	-			
				Overall Recovery						5	84	13
				Wheat	Green material	Propamocarb	0.050	85	91	5	96	7.8
	101	100	5					99	4.0			
	102											
	99	100										
	105											
	94	98	Overall Recovery						10	98	6.1	
	Grain	Propamocarb	0.010		84	89	2	85	-			
					94	94	2	94	-			
					Overall Recovery						4	90
	Straw	Propamocarb	0.050		67	81	5	83	12			
					83	89	5	91	2.8			
					93							
					89	92						
				95	89							
	90		Overall Recovery						10	87	9.4	
Soil	-	Propamocarb	0.020	88	84	2	86	-				
			0.200	97	92	2	95	-				
			10	100	98	2	99	-				
			Overall Recovery						6	93	6.7	

* Recovery samples for Propamocarb determination were prepared by fortification of untreated samples with Propamocarb free base reference substance and calculated as Propamocarb hydrochloride.

Four rotational crop studies in The Netherlands, France, Spain and Italy (Melrose and Portet, 2010, M-349882-02-1, M-349137-02-1, M-361470-01-1, and M-349147-02-1) were conducted in/on lettuce as primary crop, followed by lettuce, carrots and winter wheat or winter barley as following rotational crops, with an intended plant black interval (PBI) of 30 days during the years 2008 to 2010.

Propamocarb was applied on lettuce as the primary crop with three spray applications at 2.5 L/ha corresponding to 1.325 kg propamocarb/ha per application. The applications were carried out with 7–13 days interval. The first treatment on lettuce (primary crop) was conducted at BBCH 14–42, whilst the last application was carried out at BBCH 19–48, 5–7 days before the anticipated commercial harvest. Lettuce (primary crop) was harvested at normal harvest stage, 32–69 days after planting. No lettuce samples were taken for analysis. Harvested primary crop lettuce was destructed and the remaining plant parts were incorporated by a mulling machine or a little rotary machine. At the 27–38 day plant back interval for carrots, 26–46 day plant back interval for lettuce and 30–38 day plant back interval for cereals the plots were prepared for crop planting/sowing (rotational crops) following normal agronomic practices for each crop type in the regions. The carrots were harvested at BBCH 46–49, the lettuce was harvested at BBCH 45–49, and the cereals were harvested at BBCH 29–89.

Lettuce, carrot and cereal samples were analysed for residues of propamocarb according to method 00880/M002 by LC-MS/MS with a LOQ of 0.01 mg/kg for carrot (root and leaf), lettuce

(head), winter wheat and winter barley (grain) and with a LOQ of 0.05 mg/kg for winter wheat and winter barley (green material and straw). Residues were determined and expressed as propamocarb.

Table 4 Recovery of Propamocarb in lettuce, carrots and cereals (secondary crops)

Crop	Portion analysed	Fortification level (mg/kg)	Recovery (%)		
			Single values	n	Mean
Carrot	leaf	0.01	87, 84, 79, 90, 84, 80, 80	7	83
		0.10	89, 90, 94, 91, 74, 77, 86, 86	8	86
	Overall Recovery			15	85
	root	0.01	84, 82, 80, 73, 84, 78, 69	7	79
		0.10	97, 98, 93, 92, 99, 87, 89, 90	8	93
	Overall Recovery			15	86
Lettuce	head	0.01	89, 86, 93, 92, 98, 83	6	90
		0.10	100, 101, 95, 97, 98, 78, 82	7	93
	Overall Recovery			13	92
Winter wheat	grain	0.01	70, 63, 82, 87, 69, 70	6	74
		0.1	83, 76, 67, 77, 76	5	76
	Overall Recovery			11	75
	green material	0.05	84, 60, 69, 92, 97, 90, 91	7	83
		0.5	73, 71, 69, 91, 92	5	79
	Overall Recovery			12	81
	straw	0.05	86, 77, 77, 93, 94, 86, 70	7	83
		0.5	76, 73, 77, 81, 80	5	77
	Overall Recovery			12	80
Winter barleyt	grain	0.01	92, 95, 98, 70	4	89
		0.1	89, 89, 91	3	90
	Overall Recovery			7	89
	green material	0.05	84, 83, 78, 83	4	82
		0.5	87, 88, 89	3	88
	Overall Recovery			7	84
	straw	0.05	75, 72, 76	3	74
		0.5	60, 74, 64	3	66
	Overall Recovery			6	70

No residues of Propamocarb could be detected (below LOQ: 0.01 mg/kg for carrots, lettuce and cereals grain, 0.05 mg/kg for cereals straw) in rotational crops at a mean plant black interval (PBI) of 31 days (from 27 to 38 days) in carrots (root and leaf), at a mean PBI of 33 days (from 26 to 46 days) in lettuce (head and leaf) and at a mean PBI of 33 days (from 30 to 38 days) in cereals (grain and straw).

RESIDUE ANALYTICAL METHODS

Analytical methods for the determination of residues of propamocarb in plant and animal matrices have been evaluated by the 2006 JMPR. The QuEChERS Method (BCS method ID 01205) for the determination of residues of Propamocarb in meat (cattle), liver (cattle), kidney (cattle), fat (cattle), milk (cattle) and egg (chicken) was developed and validated (Konrad and Neuland, 2010, M-398135-01-1 and M-387185-01-1).

The Multi Method L 00.00-115 “QuEChERS” of the Official Collection of Test Methods was performed according to § 64 LFGB with:

- extraction with water/acetonitrile (5 g homogenised specimens and 5 mL of water (7 mL for fat samples) and 10 mL of acetonitrile);
- addition of magnesium sulphate, sodium chloride and sodium citrate, liquid/liquid partition and subsequent centrifugation;
- clean up by Primary Secondary Amine (PSA) and/or by freezing-out;
- detection with liquid chromatography tandem mass spectrometry LC-MS/MS using two mass transitions.

The quantification was done by an external calibration using matrix matched standards prepared in solvents. The respective followed mass transitions are presented in Table 3 below.

Table 5 Quantification and confirmatory mass transitions

	Mass transition used for quantification (1 st MRM)	Mass transition used for confirmation (2 nd MRM)
Propamocarb	189 → 102 m/z	189 → 74 m/z

For validation of the method, recovery experiments were performed by fortifying control specimens of the matrices meat, liver, kidney, fat, milk and egg at levels of 0.01 mg/kg (LOQ) up to 0.10 mg/kg.

Matrix effects for propamocarb in meat, liver, kidney, fat, milk and egg were investigated in a preliminary test. For this purpose a calibration solution of 50 ng/mL propamocarb hydrochloride was diluted with acetonitrile / 0.05% acetic acid (1/4, v/v). The same dilution was prepared using control extracts of all matrices. The final concentrations obtained were 5 ng/mL in control matrices or in acetonitrile / 0.05% acetic acid (1/4, v/v). The results are summarized in Table 6 below.

Table 6 Results of the matrix effects assessment

Matrix	Signal of the matrix matched standard relative to the solvent standard in [%]	
	189 → 102 m/z	189 → 74 m/z
Liver	96	93
Meat	78	75
Kidney	95	100
Egg	97	98
Fat	94	90
Milk	99	98

The tests for meat showed a signal depression of up to 25%. Only slight effects were observed for liver, kidney, egg, fat and milk.

The limit of quantification (LOQ) was set at 0.01 mg/kg for propamocarb (expressed as propamocarb) in all the sample materials (meat, liver, kidney, fat, milk and egg).

The limit of determination (LOD) was 0.003 mg/kg (30% of LOQ) for propamocarb (expressed as propamocarb) in all the sample materials (meat, liver, kidney, fat, milk and egg). The chromatographic peaks of propamocarb were greater than the signal equivalent to three times the background noise.

The linearity was demonstrated by injecting standards of propamocarb hydrochloride at concentrations between 0.100 and 20.0 ng/mL. Linearity test was performed by injecting eight standard solutions. Regression coefficients R were higher than 0.999.

The mean recoveries for each fortification level should be in the range of 70–110%. Although single recoveries below 60% (e.g. for egg and fat) were noted, mean values were between 70–110% with a relative standard deviation ≤ 20%. The overall relative standard deviation and the standard deviation for each fortification level were ≤ 20%.

Table 7 Recovery results from method validation of Propamocarb in animal tissues with the quantification MRM (189 → 102 m/z)

Animal	Portion analysed	Fortification level (mg/kg)		Recovery (%)						Study No. Year	
				Single values			n	Mean	RSD ^a		
Cattle	Liver	Propamocarb	0.01	87	89	88	81	5	85	4.9	BAY-1032V 2010
			0.10	78	79	81	78	5	78	3.3	
				74							
	Overall Recovery							10	82	6.0	
Cattle	Meat	Propamocarb	0.01	84	82	85	83	5	83	1.9	
			0.10	78	78	75	78	5	77	1.8	
				76							
	Overall Recovery							10	80	4.3	
Cattle	Kidney	Propamocarb	0.01	83	83	83	84	5	82	2.9	
			0.10	68	73	73	72	5	72	3.0	
				73							
	Overall Recovery							10	77	7.6	
Cattle	Milk	Propamocarb	0.01	76	81	78	70	5	77	5.5	
			0.10	77	76	74	81	5	76	3.8	
				74							
	Overall Recovery							10	77	4.4	
Chicken	Egg	Propamocarb	0.01	74	77	77	73	5	75	2.5	
			0.10	53*	70	82	68	5	70	16	
				78							
	Overall Recovery							10	73	11	
Cattle	Fat	Propamocarb	0.01	78	81	80	63	5	78	12	
			0.10	78	78	82	77	5	78	3.7	
				74							
	Overall Recovery							10	78	8.2	

^a RSD: Relative Standard Deviation RSD = SD / Mean recovery × 100 %

*Mean value between 70 and 110% with RSD < 20%.

Table 8 Recovery results from method validation of Propamocarb in animal tissues with the confirmatory MRM (189 → 74 m/z)

Animal origin	Portion analysed	Active substance Fortification level (mg/kg)		Recovery (%)						Study Trial No. Year	
				Single values			n	Mean	RSD ^(a)		
Cattle	Liver	Propa-mocarb	0.01 0.10	86	73	76	5	79	7.1	BAY-103 2V 2010	
				83	75						
				76	78	78	5	77	2.3		
				74	78						
Overall Recovery						10	78	5.2			
Cattle	Meat	Propa-mocarb	0.01 0.10	78	83	77	5	81	3.8		
				84	81						
				78	79	78	5	78	0.6		
				78	78						
Overall Recovery						10	79	3.0			
Cattle	Kidney	Propa-mocarb	0.01 0.10	89	80	85	5	85	3.9		
				86	84						
				73	79	71	5	74	4.3		
				73	72						
Overall Recovery						10	79	8.4			
Cattle	Milk	Propa-mocarb	0.01 0.10	74	89	83	5	79	9.4		
				70	80						
				76	79	71	5	75	4.0		
				77	74						

Animal origin	Portion analysed	Active substance Fortification level (mg/kg)		Recovery (%)						Study Trial No. Year
				Single values			n	Mean	RSD ^(a)	
Chicken	Egg	Propa-mocarb	0.01 0.10	76	78	83	5	77	7.4	
				72	71					
				51*	69	86	5	70	19	
				67	78					
	Overall Recovery						10	73	13	
Cattle	Fat	Propa-mocarb	0.01 0.10	80	82	81	5	77	13	
				59	84					
				79	79	83	5	80	2.4	
				80	78					
	Overall Recovery						10	79	9.1	

An independent lab validation (ILV) was reported by Weber, 2010 (reference M-387185-1). For this ILV of the analytical method of propamocarb in muscle (cattle), liver (cattle), kidney (cattle), fat (cattle), milk (cattle) and whole egg (chicken), recovery experiments were performed at fortification levels of 0.01 mg/kg (LOQ) and 0.10 mg/kg in all matrices.

The mean recovery at each fortification level should be in the range of 70–110%. Although single recoveries above 110% (e.g. for kidney) were noted, mean values were between 70–110% with a relative standard deviation $\leq 20\%$. The overall relative standard deviation for each fortification level was $\leq 20\%$. The method was therefore considered to be valid for the determination of propamocarb residues in muscle, kidney and liver.

With slightly changed conditions in the sample preparation procedure compared to original BCS method 01205 (extraction by shaking vigorously for five minutes instead of one, no freeze out) the mean recoveries were between 70–110 % for whole milk, fat and whole egg for both levels (LOQ and ten times LOQ), and the overall relative standard deviation for each fortification level was $\leq 20\%$.

The data presented demonstrate that the QuEChERS method (BCS method 01205) is suitable and validated for the determination of residues of propamocarb in animal tissues (meat, liver, kidney, fat, milk and egg) with a LOQ of 0.01 mg/kg. Satisfactory specificity, linearity, accuracy, precision, repeatability and reproducibility have been obtained and therefore have proven its applicability as enforcement method.

Stability of residues in stored analytical samples

JMPR in 2006 has reviewed the storage stability of propamocarb (free base) and propamocarb hydrochloride in plant materials and animal tissues and products. The conclusion of the review shows that the compounds are stable in frozen storage in the tested plant commodities for 14 to 26 months and in animal commodities for at least 6 months. No evidence of residue degradation was seen in any of the studies.

A study of storage stability in commodities with high water content has been performed in cabbages (Everitt and Charter, 2000, M-198306-01-1), and was submitted to the Meeting. Cabbage samples (10 g) were fortified with Propamocarb hydrochloride at a concentration equivalent to 0.5 mg/kg propamocarb (free base) and stored in a deep-freezer at about -18 °C. Samples were removed at intervals of up to 39 months (day 0 and after 3, 7, 18, 25 and 39 months) with three replicates per interval for immediate residue analysis. A range of 80 to 116% of the added residues remained after the storage period. This indicates that residues of propamocarb hydrochloride in cabbage leaves are stable for a period of at least 39 months when stored deep frozen (-18 °C).

Table 9 Storage stability of Propamocarb hydrochloride in cabbage

Storage interval [months]	Fortification level [mg/kg] ^a	Recovered residues in stored samples							Reference
		%Residues remaining				Procedural recovery			
		Single recoveries uncorrected [%]			Mean recovery uncorrected [%]	Single recoveries [%]	Mean recovery [%]		
0	0.50	114	116	104	111	78	105	92	M-198306-01-1
3	0.50	92	96	94	94	87	85	86	
7	0.50	84	84	86	85	69	73	71	
18	0.50	84	96	80	87	66	88	77	
25	0.50	84	92	76	84	77	79	78	
39	0.50	90	82	96	89	84	92	88	

Fortified with Propamocarb hydrochloride, but fortification level equivalent Propamocarb (free base)

USE PATTERN

Formulations containing propamocarb hydrochloride, alone or co-formulated with other active ingredients, are registered for use on a wide variety of crops in many countries. Registrations cover foliar treatment of vegetable crops and potatoes, soil drench, application via drip irrigation to vegetables and ornamentals and as seed treatments. Registered uses of propamocarb and propamocarb hydrochloride in crops and countries which were relevant to this evaluation are shown in Tables 10 to 17. Representative labels covering the critical GAP were provided to the Meeting.

Table 10 Registered uses of Propamocarb in onion

Crop	Country	Application				PHI days
		Method	Rate kg ai/ha	Water L/ha	Max. No.	
Onion	Albania		0.875-1.0	400-1000	3	7
Onion	Chile		0.75-0.937	400	3	7
Onion	Colombia		0.562-0.75	500	1	7
Onion	Colombia		0.937-1.25	500	3	7
Onion	Cuba		0.75-0.937	300-600	3	7
Onion	Cuba		0.937	200-600	3	14
Onion	Cuba		0.795	200-600	3	7
Onion	Czech Republic		1.0	200-600	3	
Onion	Dominican Republic		0.75-0.937	300	3	7
Onion	Dominican Republic		0.937	200-600	3	14
Onion	Dominican Republic		0.79-1.32	200-600	3	7
Onion	El Salvador		0.75-0.937	200-600	3	7
Onion	El Salvador		0.937	200-600	3	14
Onion	El Salvador		0.79-1.32	200-600	3	7
Onion	Estonia		1.0	300-500	3	7
Onion	France		1.0	400-1000	2	7
Onion	Guatemala		0.75-0.937	300-600	3	7
Onion	Guatemala		0.937	200-600	3	14
Onion	Guatemala		0.79-1.32	200-600	3	7
Onion	Guatemala		0.795	200-600	3	7
Onion	Guyana		0.937	200-600	3	14
Onion	Honduras		0.75-0.937	300-600	3	7
Onion	Honduras		0.937	200-600	3	14
Onion	Honduras		0.795	200-600	3	7
Onion	Israel		0.75	100-300	1	3
Onion	Italy		1.0	1000	3	7
Onion	Jamaica		0.75-0.937	300-600	3	7
Onion	Korea, Republic of		0.866	1200	3	30
Onion	Latvia		1.0	300-600	3	7
Onion	Lithuania		1.0	300-600	3	7
Onion	Nicaragua		0.937	200-600	3	14
Onion	Nicaragua		0.79-1.32	200-600	3	7
Onion	Panama		0.75-0.937	300-600	3	7

Crop	Country	Application				PHI days
		Method	Rate kg ai/ha	Water L/ha	Max. No.	
Onion	Panama		0.79-1.32	200-600	3	7
Onion	Peru		0.75	400	2	14
Onion	Poland		1.0	700	3	7
Onion	Romania		0.875	600-1000	2	7
Onion	Saudi Arabia		1.083	600-1000	4	14
Onion	Switzerland		0.75	300-500	3	14
Onion	Trinidad and Tobago		0.937	200-600	3	14
Onion	Uruguay		0.124-0.156	800-1000	3	21

F: Foliar Spraying

Table 11 Registered uses of Propamocarb in Broccoli

Crop	Country	Application				PHI days
		Method	Rate kg ai/ha	Water L/ha	Max. No.	
Broccoli	Belgium	F	1.0	400-600	3	14
Broccoli	Chile	F	0.9375	400	3	17
Broccoli	Chile	F	1.325	400-600	3	14
Broccoli	Cuba	F	0.795	200-600	3	7
Broccoli	Cyprus	F	1.0	1000	3	14
Broccoli	Dominican Republic	F	1.444	200-400	1	7
Broccoli	Dominican Republic	F	1.325	200-600	3	7
Broccoli	El Salvador	F	1.325	200-600	3	7
Broccoli	Estonia	F	1.0	300-600	3	14
Broccoli	France	F	1.0	400-1000	1	14
Broccoli	Greece	F	1.0	1000	3	14
Broccoli	Guatemala	F	1.444	200-400	1	7
Broccoli	Guatemala	F	1.325	200-600	3	7
Broccoli	Guatemala	F	0.795	200-600	3	7
Broccoli	Latvia	F	1.0	300-600	3	14
Broccoli	Lithuania	F	1.0	300-600	3	14
Broccoli	Luxembourg	F	1.0	300-1000	3	14
Broccoli	Nicaragua	F	1.083	300-600	1	7
Broccoli	Nicaragua	F	1.325	200-600	3	7
Broccoli	Panama	F	1.083	300-600	1	7
Broccoli	Panama	F	1.325	200-600	3	7
Broccoli	Spain	F	1.0	500-1000	3	14
Broccoli	Switzerland	F	0.75	300-500	3	14

F: Foliar Spraying

Table 12 Registered uses of Propamocarb in Cauliflower

Crop	Country	Application				PHI days
		Method	Rate	Water	Max.	
Cauliflower	Belgium	F	1.0	400-600	3	14
Cauliflower	Chile	F	0.75-0.937	400	3	18
Cauliflower	Chile	F	1.06-1.325	400-600	3	14
Cauliflower	Cuba	F	0.795	200-600	3	7
Cauliflower	Cyprus	F	1.0	1000	3	14
Cauliflower	Dominican Republic	F	0.795-1.325	200-600	3	7
Cauliflower	El Salvador	F	0.795-1.325	200-600	3	7
Cauliflower	Estonia	F	1.0	200-400	3	14
Cauliflower	France	F	1.0	400-1000	1	14
Cauliflower	Germany	F	1.155	400-600	2	21
Cauliflower	Greece	F	1.0	1000	3	14
Cauliflower	Guatemala	F	0.795-1.325	200-600	3	7
Cauliflower	Guatemala	F	0.795	200-600	3	7

Crop	Country	Application				PHI days
		Method	Rate	Water	Max.	
Cauliflower	Honduras	F	0.795	200-600	3	7
Cauliflower	Hungary	F	0.75-1.0	400-600	3	14
Cauliflower	Latvia	F	1.0	300-600	3	14
Cauliflower	Lithuania	F	1.0	300-600	3	14
Cauliflower	Lithuania	F	1.325	1250	2	3
Cauliflower	Luxembourg	F	1.0	300-1000	3	14
Cauliflower	Nicaragua	F	0.795-1.325	200-600	3	7
Cauliflower	Panama	F	0.795-1.325	200-600	3	7
Cauliflower	Switzerland	F	0.75	300-500	3	14

F: Foliar Spraying

Table 13 Registered uses of Propamocarb in Brussels sprouts

Crop	Country	Application				PHI days
		Method	Rate kg ai/ha	Water L/ha	Max. No.	
Brussels sprouts	Belgium	F	1.0	400-600	3	14
Brussels sprouts	Chile	F	0.75-0.937	400	3	17
Brussels sprouts	Chile	F	1.06-1.325	400-600	3	14
Brussels sprouts	Estonia	F	1.0	200-400	3	14
Brussels sprouts	France	F	1.0	400-1000	1	14
Brussels sprouts	Hungary	F	0.75-1.0	500	3	14
Brussels sprouts	Latvia	F	1.0	300-600	3	14
Brussels sprouts	Lithuania	F	1.0	300-600	3	14
Brussels sprouts	Luxembourg	F	1.0	300-500	3	14

F: Foliar Spraying

Table 14 Registered uses of Propamocarb in Head cabbage

Crop	Country	Application				PHI days
		Method	Rate kg ai/ha	Water L/ha	Max. No.	
Cabbage, red	Belgium	F	1.0	400-600	3	14
Cabbage, white	Chile	F	0.75-0.937	400	3	17
Cabbage, white	Chile	F	1.06-1.325	400-600	3	14
Cabbage, white	Cuba	F	0.75-0.937	300-600	3	7
Cabbage, red	Cyprus	F	1.0	1000	3	14
Cabbage, white	Dominican Republic	F	0.75-0.937	300-600	3	7
Cabbage, white	Estonia	F	1.0	200-400	3	14
Cabbage, white	France	F	1.0	400-1000	1	14
Cabbage, red	Germany	F	1.083	400-600	2	21
Cabbage, red	Greece	F	1.0	1000	3	14
Cabbage, white	Guatemala	F	0.75-0.937	300-600	3	7
Cabbage, white	Honduras	F	0.75-0.937	300-600	3	7
Cabbage, white	Hungary	F	0.75-1.0	400-600	3	14
Cabbage, white	Jamaica	F	0.75-0.937	300-600	3	7
Cabbage, red	Lithuania	F	1.0	300-600	3	14
Cabbage, white	Lithuania	F	1.325	1250	2	3
Cabbage, white	Panama	F	0.75-0.937	300-600	3	7
Cabbage, white	Spain	F	1.0	500-1000	3	14
Cabbage, white	Switzerland	F	0.75	300-500	3	14
Cabbage, field	Trinidad and Tobago	F	0.937	200-600	3	14

F: Foliar Spraying

Table 15 Registered uses of Propamocarb in Kale

Crop	Country	Application				PHI days
		Method	Rate kg ai/ha	Rate water	Max. No.	
Kale, curly	Belgium	F	1.0	400-600	3	14
Kale, curly	Germany	Drench	15.9	2-3L/m ²	2	n.a.
Kale, curly	Slovakia	Drench	15.9	2-3L/m ²	2	n.a.
Kale, curly	Ireland	Drench	15.9	2-4 L/m ²	2	n.a.
Kale, curly	UK	Drench	15.9	2-4 L/m ²	2	n.a.
Kale, curly	Thailand	Drench plus Foliar spray	15.9 1.083	2-3L/m ² 1000	1 1	n.a. 14

*PCH = Propamocarb hydrochloride (salt); PMB = Propamocarb (free base); n.a. = not applicable

Table 16 Registered uses of Propamocarb in Leek

Crop	Country	Application				PHI days
		Method	Rate kg ai/ha	Water L/ha	Max. No.	
Leek, common	Albania	Foliar Spray	0.875-1.0	400-1000	4	14
Leek, common	Estonia	Foliar Spray	1.0	500	2	14
Leek, common	Latvia	Foliar Spray	1.0	300-600	2	14
Leek, common	Lithuania	Foliar Spray	1.0	300-600	2	14
Leek, common	Syrian Arab. Rep.	Foliar Spray	0.75-1.0	300-600	1-2	14

Table 17 Registered uses of Propamocarb in lima bean

Crop	Country	Application				PHI in days
		Method	Total Rate kg ai/ha	Water L/ha	Max. No.	
Lima bean	US	Foliar broadcast	1.68		4	0

RESIDUE FROM SUPERVISED TRIALS ON CROPS

New residue trials in broccoli, cauliflower, Brussels sprouts, head cabbage, kale, onions, leeks and Lima bean were submitted to the meeting to support establishment of new MRLs. The formulation used in trials was suspension concentrate (SC).

Onion

A total of twenty one residue trials were performed in/on onion with the propamocarb hydrochloride under field conditions in Europe in 2006 and 2007. The results are shown in Table 18.

Table 18 Residues of propamocarb following foliar application to onion (SC formulations)

Country Location Year, Variety	Application				Sample analysed	DAT (days)	Residues as Propamocarb mg/kg	Report
	kg ai/ha	Water L/ha	kg ai/hL	No.				
France, Bouafle 2006	1.0	500	0.20000	3	bulb	0	0.48	RA-2306/06
Jaunes des Cevennes						3	0.25	
						7	0.05	
Germany, Burscheid 2006	1.0	300	0.33313	3	bulb	0	0.57	RA-2306/06
Stuttgarter Riesen						3	0.13	
						7	0.05	
Belgium, Wangenies 2006	1.0	450	0.22250	3	bulb	0	0.19	RA-2306/06
Summit						3	0.01	
						7	0.02	
Germany, Kohlhof 2006	1.0	500	0.20000	3	bulb	0	0.29	RA-2306/06
Takstar						3	0.12	
						7	0.05	

Country Location Year, Variety	Application				Sample analysed	DAT (days)	Residues as Propamocarb mg/kg	Report
	kg ai/ha	Water L/ha	kg ai/hL	No.				
Germany, Schiffer-stadt 2006 Hyskin	1.05-1.08	420-430	0.250	3	bulb	0 7 10 14	2.5 <u>0.41</u> 0.24 0.09	MR- 07/212
Germany, Schiffer-stadt 2006 Savel	0.86-1.08	344-648	0.167-0.250	3	bulb	0 7 10 14	4.2 <u>1.3</u> 0.46 0.44	MR- 07/212
France, Champien 2007 Boston	1.0	300	0.333	3	bulb	0 3 7	0.07 0.02 <u>0.01</u>	RA- 2634/07
Germany, Burscheid 2007 Stuttgarter Riesen	1.0	300	0.333	3	bulb	0 3 7	0.15 0.02 <u>0.02</u>	RA- 2634/07
United Kingdom, West Row/BSE 2007, Sherpa	1.0	300	0.333	3	bulb	0 3 7	0.38 0.32 <u>0.21</u>	RA- 2634/07
The Netherlands, Wieringer-werf 2007, Hybell	1.0	800	0.125	3	bulb	0 3 7	0.12 <0.01 <u><0.01</u>	RA- 2634/07
Germany, Rotters-dorf 2007 Dacapo	1.0	400	0.250	3	bulb	0 7 10 14	3.2 <u>1.4</u> 1.1 1.2	MR- 08/050
Germany, Aholming 2007, Dacapo	1.0	400	0.250	3	bulb	7 14	<u>0.86</u> 0.52	MR- 08/050
Germany, Schiffer-stadt 2007, Kaigaro	1-1.06	400-424	0.250-0.251	3	bulb	7 14	<u>0.52</u> 0.21	MR- 08/050
France, St Jory (Midi- Pyrenees) 2006, Elodie	1.0	600	0.16688	3	bulb	0 3 7	0.38 0.15 <u>0.07</u>	RA- 2307/06
Spain, Malgrat de Mar (Barce-lona) 2006, Orion	1.0	500-600	0.16688- 0.20000	3	bulb	0 3 7	0.53 0.09 <u>0.05</u>	RA- 2307/06
Italy, Bologna (Emilia - Romagna) 2006, Density 5	1.0	500	0.20000	3	bulb	0 3 7	0.13 0.05 <u>0.02</u>	RA- 2307/06
Portugal, Alcochete (Ribatejo e Oeste) 2006, Cebola-de Alcochete	1-1.0813	500-540	0.20000	3	bulb	0 4 7	0.34 0.05 <u>0.03</u>	RA- 2307/06
France, Sathonay Village (Rhone-Alpes) 2007, Proteus	1.0	600	0.1667	3	bulb	0 3 7	<0.01 0.02 <u><0.01</u>	RA- 2635/07
Spain, Gava - Barcelona (Cataluña) 2007, Figueras	0.9188- 1.0	736-800	0.125	3	bulb	0 3 7	0.17 0.09 <u>0.05</u>	RA- 2635/07
Italy, Poggio Renatico (FE), 2007, Rossa di Toscana	1.0	600	0.1667	3	bulb	0 4 7	0.14 0.08 <u>0.02</u>	RA- 2635/07
Portugal, Francisco Alcochete (Ribatejo e Oeste) 2007, Spring Star	1.0	500	0.2000	3	bulb	0 3 7	0.30 0.04 <u>0.04</u>	RA- 2635/07

Broccoli

Ten field residue trials were conducted with the propamocarb hydrochloride on Broccoli in Europe from 2005 to 2007. The results are shown in Table 19.

Table 19 Residues of propamocarb following foliar application to Broccoli (SC formulations)

Country Location Year, Variety	Application				Sample analysed	PHI (days)	Residues as Propamocarb mg/kg	Report
	kg ai/ha	Water L/ha	kg ai/hL	No.				
France, Fondettes 2005 Monaco	1.0	300	0.33313	3	curd whole plant without roots	14 21 0 3 8	<u>0.16</u> 0.03 22 9.0 3.4	RA-2156/05
Netherlands, Zwaagdijk-Oost 2005, Montop	1.0	600	0.16688	3	curd	0 15 21	5.5 <u>0.17</u> 0.07	RA-2156/05
Belgium, Bornival 2005 Monopoly	1.0	450	0.22250	3	curd	0 3 7 14 21	2.8 2.5 0.97 <u>0.29</u> 0.07	RA-2156/05
Germany, Leichlingen 2005, Volta F1	1.0	500	0.20000	3	curd	0 14 21	7.4 <u>0.29</u> 0.07	RA-2156/05
Germany, Brühl- Schwardorf 2007, Patinon	1.0	600	0.16667	3	curd	0 14 22	2.5 <u>0.01</u> <0.01	RA-2628/07
Greece, Marathonas 2005 Marathon	1.0	500	0.20000	3	curd	0 3 7 14 21	2.8 1.3 1.1 0.20 <u>0.21</u>	RA-2157/05
France, Fenouillet 2005 Empereur	1.0	600	0.16688	3	curd	0 14 21	3.6 <u>1.7</u> 1.2	RA-2157/05
Spain, Lebrija Sevilla 2006 Marathon	1.0	500	0.20000	3	curd	0 3 7 14 21	3.6 2.5 1.4 <u>0.97</u> 0.66	RA-2157/05
Italy, Catania 2005 Cataneese	1.0	500-600	0.16688- 0.20000	3	curd	0 14 21	5.0 <u>0.32</u> 0.19	RA-2157/05
France, Castesarrasin 2007, Chevalier	1.0	600	0.16667	3	curd	0 14 21	3.9 <u>0.52</u> 0.63	RA-2628/07

Cauliflower

Ten field residue trials were conducted with the propamocarb hydrochloride on cauliflower in Europe from 2005 to 2007. The results are shown in Table 20.

Table 20 Residues of propamocarb following foliar application to cauliflower (SC formulations)

Country Location Year, Variety	Application				Sample analysed	PHI (days)	Residues as Propa- mocarb (mg/kg)	Report
	kg ai/ha	Water L/ha	kg ai/hL	No.				
France, Fondettes 2005 FARGO	1.0	600	0.16688	3	curd	0 4 7 14 21	0.24 0.09 0.05 <u>0.03</u> 0.02	RA- 2154/05
Netherlands, Zwaagdijk-Oost 2005, Freemont	1.0	600	0.16688	3	curd	14 21	<u><0.01</u> <0.01	RA- 2154/05

Country Location Year, Variety	Application				Sample analysed	PHI (days)	Residues as Propa- mocarb (mg/kg)	Report
	kg ai/ha	Water L/ha	kg ai/hL	No.				
Belgium, Bornival 2006 Thalassa	1.0	450	0.22250	3	curd	0 3 7 14 21	0.29 0.24 0.17 <u>0.04</u> 0.02	RA- 2154/05
Germany, Leichlingen 2005, Veronie F1	1.0	500	0.20000	3	curd	14 21	<u>0.08</u> 0.02	RA- 2154/05
Germany, Langenfeld- Reusrath 2007, Freedom	1.0	600	0.16667	3	curd	14 21	<u>0.02</u> <0.01	RA- 2628/07
France, Fenouillet 2005 Aviron	1.0	600	0.16688	3	curd	0 3 7 14 21	0.15 0.12 0.11 0.05 <u>0.06</u>	RA- 2155/05
Greece, Kato Souli 2005 White Magic	1.0	500	0.20000	3	curd	0 14 21	2.1 <u>0.82</u> 0.55	RA- 2155/05
Spain, Gava 2006 Flora Blanca	1.0	600	0.16688	3	curd	7 14 20	0.62 <u>0.02</u> <0.01	RA- 2155/05
Italy, Catania 2005 Violetto	1.0	600	0.16688	3	curd	0 14 21	3.1 <u>0.20</u> 0.06	RA-21 55/05
Spain, Alginet 2007, Movidick	1.0	500-800	0.125- 0.200	3	curd	14 21	<u>0.01</u> <0.01	RA- 2628/07

Brussels sprouts

Eight field residue trials were conducted with the Propamocarb hydrochloride on Brussels sprouts in Europe from 2005 to 2006. The results are shown in Table 21.

Table 21 Residues of propamocarb following foliar application to Brussels sprouts (SC formulations)

Country Location Year, Variety	Application					PHI	Residues as Propamocarb mg/kg	Report
	kg ai/ha	Water L/ha	kg ai/hL	No.				
France, Fontaine L'Etalon 2005 Maximus	1.0	300	0.33313	3	sprout	0 4 7 14 21	1.1 0.48 0.35 0.21 <u>0.25</u>	RA- 2152/05
Netherlands, Zwaagdijk-Oost 2005, Genius	1.0	600	0.16688	3	sprout	0 14 21	0.97 0.44 <u>0.49</u>	RA- 2152/05
Germany, Werl-Westönnen 2005 Cyrus	1.0- 1.06	300-318	0.33313	3	sprout	0 3 7 15 21	2.7 1.9 1.5 <u>0.99</u> <u>1.3</u>	RA- 2152/05
Germany, Langenfeld- Reusrath 2005, Genius	1.0	300	0.33313	3	sprout	0 14 21	1.4 <u>0.64</u> 0.64	RA- 2152/05
Germany, Vechta Langförden 2006, Camus	1.0	300	0.33313	3	sprout	0 14	0.67 <u>0.48</u>	RA- 2303/06
Belgium, Geer	0.94-	423-450	0.22250	3	sprout	0	0.38	RA-

Country Location Year, Variety	Application					PHI	Residues as Propamocarb mg/kg	Report
	kg ai/ha	Water L/ha	kg ai/hL	No.				
2006, Maximus	1.0					14	<u>0.20</u>	2303/06
France, Fontaine l'Etalon 2006 Louis	1.0	300	0.33313	3	sprout	0 3 7 14 21	0.64 0.83 0.51 0.41 <u>0.46</u>	RA- 2303/06
Germany, Langenfeld- Reusrath 2006 Genius	1.0	300	0.33313	3	sprout	0 3 7 14 21	0.47 0.36 0.45 <u>0.24</u> 0.23	RA- 2303/06

Head cabbage

Twelve field trials were conducted with the propamocarb hydrochloride on head cabbage in Europe from 2005 to 2006. The results are shown in Table 22.

Table 22 Residues of propamocarb following foliar application to head cabbage (SC formulations)

Country Location Year, Variety	Application				Sample analysed	PHI (days)	Residues as propamocarb mg/kg	Report
	kg ai/ha	Water L/ha	kg ai/hL	No.				
Germany, Bornheim 2005 Savoy: Alaska	1	400	0.25	3	head	0	3.7	RA-2152/05
						3	1.3	
						7	0.52	
						14	<u>0.18</u>	
						21	0.13	
Belgium, Gembloux 2005, red: Regilus	1	450	0.2225	3	head	0	0.04	RA-2150/05
						14	0.03	
						21	0.02	
France, Ennemain 2005, Savoy Ice Prince	1	300	0.33313	3	head	0	0.52	RA-2150/05
						3	0.5	
						7	0.55	
						14	<u>0.24</u>	
						21	0.19	
Netherlands, Zwaagdijk- Oost 2005, Red Integra	1	600	0.16688	3	head	0	0.37	RA-2150/05
						14	0.16	
						21	<u>0.21</u>	
Netherlands, Zwaagdijk- Oost 2006, Savoy: Produsa	1	600	0.16688	3	head	0	0.6	RA-2308/06
						14	<u>0.08</u>	
Germany, Bornheim 2006, Savoy: Visa	1	300	0.33313	3	head	0	4.4	RA-2308/06
						13	<u>0.36</u>	
France, Bouafle 2006 white: Première	1	300	0.16688	3	head	0	12	RA-2308/06
						3	4	
						7	0.39	
						14	<u>0.13</u>	
						21	0.07	
Germany,	1	600	0.33313	3	head	0	0.92	RA-2308/06

Country Location Year, Variety	Application				Sample analysed	PHI (days)	Residues as propamocarb mg/kg	Report
	kg ai/ha	Water L/ha	kg ai/hL	No.				
Leichlingen 2006 red: Rodima								
						3	0.76	
						7	0.64	
						14	<u>0.32</u>	
France, St Alban 2005 white: DELUS B285	1	600	0.16688	3	head	21	0.11	RA-2151/05
						0	0.47	
						3	0.19	
						7	0.21	
Greece, Kato Agios Ioannis 2005, white: Baner	1	300	0.33313	3	head	14	<u>0.23</u>	RA-2151/50
						21	0.08	
						0	0.73	
						14	<u>0.02</u>	
Spain, Gavà 2006 white: Megaton	1	500-600	0.16688- 0.2	3	head	21	0.1	RA-2309/06
						0	0.41	
						14	<u>0.06</u>	
Italy, Andria 2006 white: Sapalla	1	500	0.2	3	head	0	0.77	RA-2309/06
						14	<u>0.28</u>	

Kale

Nine field trials were conducted with the propamocarb hydrochloride on kale in Europe from 2006 to 2007. The results are shown in Table 23.

Table 23 Residues of propamocarb following foliar application on kale (SC formulations)

Country Location Year Variety	Application				Sample analysed	DAT (days)	Residues as Propamocarb mg/kg	Report
	kg ai/ha	Water L/ha	kg ai/hL	No.				
Germany, Oberbes- singen 2006 curly: Winnetou	0.961-1.025	600-900	0.112- 0.171	3	leaf	0	6.8	MR-07/212
						7	1.5	
						10	1.1	
						14	<u>0.46</u>	
Germany, Köln 2006 curly: Winterbor	1.0	600	0.17	3	leaf	0	13	MR-07/212
						7	9.7	
						10	6.0	
						14	<u>4.0</u> ^a	
Germany, Köln 2006 curly: Winterbor	1.0	600	0.17	3	leaf	0	14	MR-07/212
						7	6.5	
						10	6.0	
						14	<u>3.9</u>	
Germany, Schifferstadt 2006 curly: Winterbor	0.960-1.040	388-624	0.167- 0.250	3	leaf	0	24 ^b	MR-07/212
						7	10	
						10	7.0	
						14	<u>5.2</u>	
Germany, Butzbach, 2007, curly: Winnetou	1.0	600	0.17	3	leaf	14	<u>4.0</u>	MR-08/050
Germany, Reken – Hülsen, 2007, curly: Verdura	1.0	600	0.17	3	leaf	14	<u>0.33</u>	MR-08/050
Germany, Reken – Hülsen, 2007 curly: Verdura	1.0	600	0.17	3	leaf	14	<u>0.39</u>	MR-08/050
Germany, Bonn, 2007 curly: Westländer	1.0	600	0.17	3	leaf	14	<u>11.8</u>	MR-08/050

Country Location Year Variety	Application				Sample analysed	DAT (days)	Residues as Propamocarb mg/kg	Report
	kg ai/ha	Water L/ha	kg ai/hL	No.				
Germany, Bonn 2007, curly: Winnetou	1.0	600	0.17	3	leaf	14	<u>10.7</u>	MR-08/050

^a In the corresponding control sample (day 14) residues of 0.01 mg/kg were found

^b In the corresponding control sample (day 0) residues of 0.04 mg/kg were found

Leek

Twelve field residue trials were conducted with the Propamocarb hydrochloride on Leek in Europe from 2005 to 2006. The results are shown in Table 24.

Table 24 Residues of propamocarb following foliar application to Leek (SC formulations)

Country Location Year, Variety	Application				Sample analysed	PHI (days)	Residues as Propamocarb mg/kg	Report
	kg ai/ha	Water L/ha	kg ai/hL	No.				
Belgium, Wayaux 2005 Shelton	1	450	0.22250	3	whole plant without roots	0 3 7 14 21	2.3 2.6 1.9 <u>0.9</u> 0.83	RA-2111/05
Netherlands, Zwaagdijk-Oost 2005 Shelton	1	600	0.16688	3	whole plant without roots	0 3 7 14 21	6.2 7.5 4.1 <u>4.0</u> 2.4	RA-2111/05
Germany, Langenfeld- Reusrath 2005, Pandora	1	300	0.33313	3	whole plant without roots	0 14 21	2.0 <u>0.24</u> 0.15	RA-2111/05
France, Faverolles 2005 Hiverna	1	300	0.33313	3	whole plant without roots	0 13 22	15 <u>5.5</u> 0.12	RA-2111/05
France, Faverolles 2006 Diana	1	300	0.33313	3	whole plant without roots	0 14	23 <u>15</u>	RA-2302/06
Germany, Langenfeld- Reusrath 2006, Pandora	1	300	0.33313	3	whole plant without roots	0 14	5.0 <u>0.74</u>	RA-2302/06
Netherlands, Zwaagdijk-Oost 2006 Roxton	1	600	0.33313	3	whole plant without roots	0 14	9.9 <u>2.6</u>	RA-2302/06
Germany, Bornheim - Sechtem 2006, Amundo	1	300	0.33313	3	whole plant without roots	0 15	6.9 <u>2.4</u>	RA-2302/06
Italy, Lusia 2005 Sabina	1	600	0.16688	3	whole plant without roots	0 3 7 14 21	10 4.6 4.9 <u>1.1</u> 0.60	RA-2112/05
Spain, Brenes Sevilla 2005 Heracles	1	400	0.25000	3	whole plant without roots	0 3 7 14 21	15 15 <u>11</u> 4.7	RA-2112/05

Country Location Year, Variety	Application				Sample analysed	PHI (days)	Residues as Propamocarb mg/kg	Report
	kg ai/ha	Water L/ha	kg ai/hL	No.				
France, St Alban 2005 Davensy	1	600	0.16688	3	whole plant without roots	0 14 21	14 <u>4.4</u> 2.6	RA-2112/05
Portugal, Torres Novas 2005, Lancelot	1	400	0.25000	3	whole plant without roots	0 14 21	6.4 <u>1.1</u> 0.52	RA-2112/05

Lima Beans

Five field trials were conducted with the Propamocarb hydrochloride on Lima Beans (seed and forage) in United States from 2005 to 2006. The results are shown in Table 25.

Table 25 Residues of propamocarb following foliar application to Lima beans seeds

Country Location Year, Variety	Application				Sample analysed	PHI (days)	Residues as Propamocarb mg/kg	Report (Trial ID)
	Rate kg ai/ha	Water L/ha	Total Rate kg ai/ha	No.				
United States Salisbury, MD 2005 Burpee Improved Bush	1.44-1.47	35-36	6.54	4	Bean without Pods	0	0.25 0.26 0.255*	07263.05- MD15
United States Bridgeton, NJ 2005 Baby Lima	1.47-1.55	34-36	6.78	4	Bean without Pods	0	0.44 0.42 0.43*	07263.05- NJ 19
United States Clinton, NC Fordhook 2006	1.44-1.49	30-31	13.24	8	Bean without Pods	0	1.16 1.20 1.18*	07263.05- NC 03
United States Fremont, OH 2006 Fordhook 242	1.46-1.50	49-78	6.68	4	Bean without Pods	0	0.43 0.41 0.42*	07263.06- OH*01
United States Salisbury, MD 2006 Eastland	1.47-1.49	36	8.31	5	Bean without Pods	0	0.78 0.76 0.77*	07263.06- MD 02

*the average of two samples

Table 26 Residue field trials with Propamocarb conducted on Lima Beans forage

Country Location Year, Variety	Application				Sample analysed	PHI (days)	Residues as Propamocarb (mg/kg)	Report (Trial ID)
	Rate kg ai/ha	Water L/ha	Total Rate kg ai/ha	No.				
United States Salisbury, MD 2005 Burpee Improved Bush	1.44-1.47	35-36	6.54	4	Foliage	0	45.6 35.6 40.6*	07263.05- MD15
United States Bridgeton, NJ 2005 Baby Lima	1.47-1.55	34-36	6.78	4	Foliage	0	73.8 93.3 83.6*	07263.05- NJ 19
United States Clinton, NC 2006, Fordhook,	1.44-1.49	30-31	13.24	8	Foliage	0	46.1 42.9 44.5*	07263.05- NC 03

Country Location Year, Variety	Application				Sample analysed	PHI (days)	Residues as Propamocarb (mg/kg)	Report (Trial ID)
	Rate kg ai/ha	Water L/ha	Total Rate kg ai/ha	No.				
United States Fremont, OH 2006, Fordhook 242	1.46-1.50	49-78	6.68	4	Foliage	0	42.1 52.7 47.4*	07263.06- OH*01
United States Salisbury, MD 2006, Eastland	1.47-1.49	36	8.31	5	Foliage	0	151.2 136.3 143.8*	07263.06- MD 02

*the average of two samples

FATE OF RESIDUES DURING PROCESSING

Processing studies in tomato, spinach and lettuce have been conducted and submitted to the Meeting. These studies are described below. An overview of the processing factors obtained for the different processed commodities and the processing by-products is given in Table 30.

Tomatoes

Seven studies on the processing of tomatoes into juice, preserve and purée were conducted to determine the transfer of propamocarb from tomato fruits into juice, preserve and puree.

Four studies (Billian, 2008, M-307290-01-1) were conducted in Italy, Spain, Greece and Portugal with 3 sprays of propamocarb (SC formulation) at rate of 4.74 kg ai/ha with interval of 7 days and 1day PHI. Samples of tomato (fruit) to be processed were sampled at 1 day after last treatment.

The processing of tomato samples into fruit, washed; washings; raw juice; juice; fruit, peeled; peel; peeling water; preserves; strain rest, wet; raw puree and puree was performed in the Food Processing Laboratory of BCS-D-ROCS in Monheim am Rhein. The washing and peeling of tomatoes was done using household practice. The preparation of juice, preserves and puree simulated the industrial practice at a laboratory scale.

The tomatoes were washed in lukewarm standing water. The washed tomatoes were cut with a knife into small pieces and were heated after addition of water to 80–100 °C for 40 min in order to prevent enzymatic reactions. After this blanching process, the tomato pulp was passed through a strainer to separate raw juice and strain rest. Sodium chloride (0.5–0.7% (w/w)) was added to the raw juice. An aliquot of the laboratory sample of raw juice was taken. One part of the remaining raw juice was used for the processing into preserves (see below). Another portion of the remaining raw juice was filled into preserving cans and pasteurised. After pasteurisation an aliquot of the laboratory sample juice was taken.

For the processing of tomato into preserves, another portion of the deep-frozen tomatoes were washed in warm standing water. After a few minutes the peel could be taken off with a kitchen knife, but in contrast to other processing procedures the peeling was very difficult and a lot of pulp remained with the peel. The peeling water containing small pots of peel was sieved. Aliquots of the laboratory samples peel, peeling water and fruit, peeled were taken. After addition of raw juice a part of the remaining peeled tomatoes (ratio fruit/juice = 1/0.86) was filled into preserving cans and pasteurised. After pasteurisation, the tomato preserves were minced with a hand mixer. An aliquot of the obtained laboratory sample of tomato preserve was taken.

For the processing of tomato fruits into puree, tomatoes samples were washed in lukewarm standing water. The washed tomatoes were cut with a knife into small pieces and were heated after addition of water to 98–100 °C for 40 min in order to prevent enzymatic reactions. After this blanching process, the tomato pulp was passed through a strainer to separate raw juice and strain rest. The obtained tomato raw juice was mixed up with sodium chloride. An aliquot of the raw juice was concentrated (100–200 mbar, 75 °C) while stirring to obtain tomato raw puree (dry weight: 16%).

After taking an aliquot of the sample raw puree, the remaining raw puree was filled into preserving cans and pasteurised. After pasteurisation, the tomato puree was sampled.

Tomato samples and processed fractions were analysed for residues of propamocarb hydrochloride according to methods 00880/M001 (study RA-3639/07) and 00880/M002 (study 09-3235) by LC-MS/MS with an LOQ of 0.01 mg/kg. The molecular weights of propamocarb hydrochloride and Propamocarb were taken into consideration to determine and express the results as Propamocarb.

Table 27 Recovery data for propamocarb in tomato fruits and processed commodities

Portion analysed	Active substance	Fortification level (mg/kg)	Recovery (%) Single values	n	Mean	RSD
Fruit ^a	Propamocarb	0.01	104, 96, 96, 99, 104, 113	6	102	6.4
		0.1	101, 101, 102, 115	4	105	6.5
		50	90	1	-	-
Overall Recovery				11	102	7.1
Juice ^b	Propamocarb	0.01	94 95 99 117	4	101	10.6
		0.1	90 92 97 115	4	99	11.6
		5.0	94	1	-	-
Overall Recovery				9	99	9.9
Puree ^c	Propamocarb	0.01	89 91 93	3	91	2.2
		0.1	97 95	2	96	-
		10	86	1	-	-
Overall Recovery				6	92	4.4
Fruit ^d	Propamocarb	0.01	79 82 83 85	4	82	3.0
		0.1	70 71 72 73 79	5	73	4.8
Overall Recovery				9	77	7.4
Raw juice	Propamocarb	0.01	83	1	-	-
		0.1	69	1	-	-
Overall Recovery				2	76	-
Juice ^e	Propamocarb	5	96	1	-	-
Overall Recovery				1	-	-
Fruit, peeled ^f	Propamocarb	5	93	1	-	-
Overall Recovery				1	-	-
Whole fruit, washed	Propamocarb	0.01	75	1	-	-
		0.1	73	1	-	-
		0.5	94	1	-	-
Overall Recovery				3	81	14.4
Raw puree	Propamocarb	0.01	70	1	-	-
		0.1	68	1	-	-
Overall Recovery				2	69	-
Puree ^g	Propamocarb	5	101	1	-	-
Overall Recovery				1	-	-

FL = Fortification Level,

Fortified with Propamocarb-hydrochloride, determined as Propamocarb and calculated as Propamocarb

^a Tomato fruit, washed; tomato peel; preserve and tomato fruit, peeled are covered by tomato fruit.

^b Tomato washings; tomato raw juice and tomato peeling water are covered by tomato juice.

^c Tomato raw puree and tomato strain rest are covered by tomato puree.

^d The recoveries of the RAC tomato (fruit) were performed during the conduct of study 09-2235

^e Tomato, washings and tomato, peeling water are covered by tomato juice.

^f Tomato, peel and tomato, preserve are covered by tomato fruit, peeled.

^g Tomato, strain rest is covered by tomato, puree

Residues of propamocarb in tomato fruits ranged from 2.8 to 5.9 mg/kg. Residues processed fractions were between 1.2 and 3.1 mg/kg in juice, between 0.88 and 2.0 mg/kg in preserve and between 1.8 and 4.9 mg/kg in puree.

Table 28 Residues of Propamocarb in tomato fruits (RAC) and processed tomato commodities in Italy, Spain, Greece and Portugal

Sample material	Residues of Propamocarb (mg/kg)			
	Italy	Spain	Greece	Portugal
Fruit (RAC)	2.8	5.9	3.4	5.2
Fruit, washed	1.4	2.9	1.9	3.6
Washings	0.88	1.1	0.48	1.3
Raw juice	1.0	2.9	2.0	2.3
Juice	1.2	3.1	2.0	2.3
Fruit, peeled	0.86	2.5	1.2	0.73
Peel	2.8	18	8.0	5.6
Peeling water	0.98	1.5	0.77	0.94
Preserve	0.88	2.0	1.7	1.4
Strain rest, wet	1.5	2.6	2.3	2.0
Raw puree	1.8	4.4	2.5	3.9
Puree	1.8	4.9	2.2	3.7

Three studies on tomato (Schulte and Bauer, 2011, M-406443-02-1) were conducted in Northern and Southern Europe (The Netherlands, Italy, and Germany). Propamocarb (SC formulation) was sprayed three times on tomato plants with a nominal product rate of 0.62kg ai/ha and a water rate of 1000 L/ha per application. The applications were carried out with interval of 7 days and 3 days PHI. Samples of tomato (fruit) to be processed were taken at 3 days after last treatment. Residues of propamocarb in tomato fruits ranged from 0.22 to 0.59 mg/kg. Residues in processed fractions were between 0.16 and 0.40 mg/kg in juice, between 0.13 and 0.24 mg/kg in preserve and between 0.10 and 0.19 mg/kg in puree.

Table 29: Residues of Propamocarb in tomato fruits (RAC) and processed tomato commodities in Netherland, Italy, Germany

Sample material	Residues of Propamocarb (mg/kg)		
	The Netherlands	Italy	Germany
Fruit (RAC)	0.22 ^a	0.59	0.35
Raw juice	0.21 ^b	0.39	0.17
Juice	0.19 ^c	0.40	0.16
Fruit, peeled	0.07	0.13	0.05
Peel	0.64	1.8	0.96
Peeling water	0.17 ^d	0.12	0.09
Preserve	0.13	0.24	0.14
Fruit, washed	0.09	0.22	0.20
Washings	0.17	0.09	0.11
Strain rest, wet	0.10 ^e	0.21	0.12
Raw puree	0.10 ^f	0.22	0.14
Puree	0.10 ^g	0.19	0.14

*These are the calculated mean values; corresponding samples were analysed twice.

Single values:

^a 0.218 mg/kg and 0.217 mg/kg

^b 0.229 mg/kg and 0.188 mg/kg

^c 0.200 mg/kg and 0.181 mg/kg

^d 0.174 mg/kg and 0.170 mg/kg

^e 0.099 mg/kg and 0.093 mg/kg

^f 0.121 mg/kg and 0.087 mg/kg

^g 0.111 mg/kg and 0.097 mg/kg

Average processing factors (PF) of 0.57, 0.40 and 0.88 were calculated for tomato juice, preserve and puree respectively.

Table 30 Processing factors for the residue of Propamocarb in processed tomato commodities

Sample material	Processing factors for residues of Propamocarb							
	Italy	Spain	Greece	Portugal	Netherlands	Italy	Germany	Mean
Fruit, washed	0.50	0.49	0.56	0.69	0.41	0.37	0.57	0.51
Raw juice	0.36	0.49	0.59	0.44	0.95	0.66	0.49	0.57
Juice	0.43	0.53	0.59	0.44	0.86	0.68	0.46	0.57
Fruit, peeled	0.31	0.42	0.35	0.14	0.32	0.22	0.14	0.27
Peel	1.0	3.1	2.4	1.1	2.9	3.1	2.7	2.3
Preserve	0.31	0.34	0.50	0.27	0.59	0.41	0.40	0.40
Strain rest, wet	0.54	0.44	0.68	0.38	0.45	0.36	0.34	0.46
Raw puree	0.64	0.75	0.74	0.75	0.45	0.37	0.40	0.59
Puree	0.64	0.83	0.65	0.71	2.6	0.32	0.40	0.88

Spinach

Four field trials (Billian and Krussel, 2010, M-397716-01-1) were performed with spinach in Belgium, France, Germany and the Netherlands during 2009. Propamocarb was sprayed four times at rate of 3.7-3.98 kg as/ha per application and with interval of 9–11 days.

Spinach samples (leaves) to be processed were harvested at 14–15 days after the last application. The processing of the spinach leaves samples into washed leaf, washings, cooking water and cooked leaf according to household practice. The spinach leaves were washed in lukewarm standing water for about 2–5 min while moving the leaves around slowly. After washing, the leaves were centrifuged in a lettuce drainer. The collected water in the drainer was added to the rest of the washing water. Samples of washed leaves and washings were taken. The remaining washed leaves were cooked in a sieve in water steam (2 kg water/kg leaves) for about 8–10 min. After cooking, samples of cooked leaves and cooking water were taken.

Spinach samples (leaves) and processed fractions were analysed for residues of Propamocarb according to method 00880/M002 by LC-MS/MS with a LOQ of 0.01 mg/kg or 0.01 mg/L. Residues were determined and expressed as Propamocarb.

Table 31 Recovery data for Propamocarb in spinach leaves (RAC) and processed spinach commodities

Portion analysed	Active substance	Fortification level (mg/kg)	Recovery (%)			
			Single values	n	Mean	RSD
Leaf and washed leaf ^c	Propamocarb	0.01 ^a	115	1	115	-
		0.10 ^a	117	2	107	-
		0.50 ^a	98	1	98	-
		1.0 ^a	108	1	108	-
		200 ^a	97	1	97	-
		350 ^b	87	1	87	-
Overall Recovery				7	103	11

FL = Fortification Level,

^a Recoveries were determined for washed leaf.

^b Recoveries were determined for leaf.

^c These recoveries cover also washings, cooked leaf and cooking water.

Residues of propamocarb in raw leaves were between 15 and 76 mg/kg at the recommended PHI of 14-15 days. After washing, residues of propamocarb between 21 - 70 mg/kg were found in the washed leaves. The residues of propamocarb found in the washings were between 0.19 and 0.89 mg/L. The residues of propamocarb in cooked leaves were found to decrease slightly to values between 14 and 67 mg/kg. The residues of propamocarb in cooking water were found to be between 5.3 and 26 mg/L.

Table 32 Residues of propamocarb in spinach leaves (RAC) and processed spinach commodities

Country Study No. Trial No.	Crop Portion analysed	DALT (days)	Residues (mg/kg or mg/L)
			Propamocarb
Germany 09-3401 09-3401-01	Spinach leaf (RAC)	15	15
	Leaf, washed	15	21
	Washings	15	0.19
	Leaf, cooked	15	14
	Cooking water	15	5.3
France 09-3401 09-3401-02	Spinach leaf (RAC)	15	56
	Leaf, washed	15	43
	Washings	15	0.56
	Leaf, cooked	15	48
	Cooking water	15	15
Belgium 09-3401 09-3401-03	Spinach leaf (RAC)	14	76
	Leaf, washed	14	70
	Washings	14	0.68
	Leaf, cooked	14	67
	Cooking water	14	6.0
The Netherlands 09-3401 09-3401-04	Spinach leaf (RAC)	14	53
	Leaf, washed	14	42
	Washings	14	0.89
	Leaf, cooked	14	46
	Cooking water	14	26

Average processing factors (PF) of 0.97 and 0.88 were calculated for washed spinach leaves and cooked leaves respectively.

Table 33 Processing factors for the residue of Propamocarb in processed spinach commodities

Sample material	Processing factors for residues of Propamocarb				
	Germany	France	Belgium	Netherlands	Mean
Leaf, washed	1.4	0.77	0.92	0.79	0.97
Leaf, cooked	0.933	0.857	0.882	0.868	0.885

Lettuce

Two studies (Billian and Reineke, 2009, M-348419-01-1) were performed in the Netherlands and Germany on lettuce under greenhouse conditions in 2008. Propamocarb(SC formulation) was sprayed four times on lettuce plants at rate of 1.325 kg as/ha, with interval of 10 days.

Four studies (Billian and Reineke, 2010, M-397890-01-1) were performed on lettuce in the greenhouse in Belgium, France, Germany and Netherland in 2009. Propamocarb (SC formulation) was sprayed four times on lettuce at rate of 3.98 kg as/ha, with interval of 9–11 days.

Lettuce samples (head) to be processed were harvested at 13 and at 14–15 days after the last application in the 2008 and the 2009 trials, respectively. The processing of the lettuce samples (head) into the processed fractions: outer leaf, head without outer leaves, leaves without stalk, and washed inner leaves simulated household practice.

Outer leaves were removed (leaf, outer), and the remaining heads separated into leaves and stalk. The separated leaves were washed in warm water and drained. Theses fractions were analysed using the method 00880/M002 by LC-MS/MS with a LOQ of 0.01 mg/kg or 0.01 mg/L.

Table 34 Recovery data for propamocarb in lettuce head (RAC) and processed lettuce commodities

Portion analysed	Active substance	Fortification level (mg/kg)	Recovery (%)					
			Single values			n	Mean	RSD (a)
Leaf, outer	Propamocarb	0.01	93	82	94	3	90	7.4
		0.10	81	76	94	3	84	11
		50	64			1	64	-
Overall Recovery						7	83	14
Leaf, inner	Propamocarb	0.01	97	96	93	3	95	2.2
		0.10	93	93	81	3	89	7.8
		1.0	93			1	93	-
		50	82			1	82	-
Overall Recovery						8	91	6.7
Head, inner parts	Propamocarb	0.01	91	102	86	3	93	8.8
		0.10	85	69	88	3	81	13
		50	86			1	86	-
Overall Recovery						7	87	11
Leaf, inner washed	Propamocarb	0.01	94	85	93	3	91	5.4
		0.10	93	95	91	3	93	2.2
		1.0	93			1	93	-
		50	67			1	67	-
Overall Recovery						8	89	11
Washings	Propamocarb	0.01	63	68	68	3	66	4.4
		0.10	73	73	69	3	72	3.2
		50	91			1	91	-
Overall Recovery						7	72	13
Head (RAC)	Propamocarb	0.01	112			1	112	-
		0.02	96			1	96	-
		0.10	109			1	109	-
Overall Recovery						3	106	8.0
Head, inner parts ^a	Propamocarb	0.01	106			1	106	-
		0.10	102			1	102	-
		0.50	101			1	101	-
		300	97			1	97	-
Overall Recovery						4	102	3.6

^a These recoveries cover also leaf, outer; leaf, inner; washings; leaf, inner, washed

Table 35 Residues of Propamocarb in lettuce heads (RAC) and processed lettuce commodities

Country Study No. Trial No.	Crop Portion analysed	DALT (days)	Residues (mg/kg or mg/L)	
			Propamocarb	
Netherlands 08-3137 08-3137-01	Lettuce head (RAC)	13	7.7	
	Leaf, outer		5.0	
	Head, inner parts		0.27	
	Leaf, inner		0.19 ^a	
	Leaf, inner, washed		1.1 ^b	
	Washing		0.05	
Germany 08-3137 08-3137-02	Lettuce head (RAC)	13	1.6	
	Leaf, outer		4.7	
	Head, inner parts		0.03 ^c	
	Leaf, inner		0.10 ^d	
	Leaf, inner, washed		0.09 ^e	
	Washing		< 0.01	
Germany 09-3400 09-3400-01	Lettuce head (RAC)	14	26	
	Leaf, outer		57	
	Head, inner parts		1.4	
	Leaf, inner		2.7	
	Leaf, inner, washed		1.1	
	Washings		< 0.01	

Country Study No. Trial No.	Crop Portion analysed	DALT (days)	Residues (mg/kg or mg/L)
			Propamocarb
France 09-3400 09-3400-02	Lettuce head (RAC)	14	47
	Leaf, outer		110
	Head, inner parts		7.8
	Leaf, inner		10
	Leaf, inner, washed		7.7
	Washings		0.15
The Netherlands 09-3400 09-3400-03	Lettuce head (RAC)	14	12
	Leaf, outer		41
	Head, inner parts		1.2
	Leaf, inner		1.5
	Leaf, inner, washed		0.83
	Washings		< 0.01
Belgium 09-3400 09-3400-04	Lettuce head (RAC)	15	51
	Leaf, outer		120
	Head, inner parts		8.4
	Leaf, inner		9.8
	Leaf, inner, washed		4.1
	Washings		0.01

^a this result corresponds to the mean of 3 single values (0.203 mg/kg, 0.187 mg/kg and 0.17 mg/kg)

^b this result corresponds to the mean of 3 single values (1.19 mg/kg, 0.984 mg/kg and 0.993 mg/kg)

^c this result corresponds to the mean of 3 single values (0.0219 mg/kg, 0.0286 mg/kg and 0.0293 mg/kg)

^d this result corresponds to the mean of 3 single values (0.109 mg/kg, 0.101 mg/kg and 0.104 mg/kg)

^e this result corresponds to the mean of 3 single values (0.815 mg/kg, 0.097 mg/kg and 0.0813 mg/kg)

Average processing factors of 2.3, 0.09, 0.12 and 0.09 were calculated for the transfer of Propamocarb from lettuce heads into outer leaves, head inner parts, inner leaves and inner leaves washed respectively.

Table 36: Processing factors for the residue of Propamocarb in processed lettuce commodities

Sample material	Processing factors for residues of Propamocarb						
	Nether-lands	Germany	Germany	France	Nether-lands	Belgium	Mean
Leaf, outer	0.7	3.0	2.2	2.3	3.4	2.4	2.3
Head, inner parts	0.04	0.02	0.05	0.17	0.10	0.16	0.09
Leaf, inner	0.02	0.06	0.10	0.21	0.13	0.19	0.12
Leaf, inner, washed	0.10	0.06	0.04	0.16	0.07	0.08	0.09
Washings	0.01	0.01	0.01	0.01	0.01	0.01	0.01

Table 37: Summary of the processing studies in tomato, spinach and lettuce

Commodity	Processed commodity	Processing factors Residue: PCB	Processing factor(PF) (best estimate)	Study Document
Tomato (0.515mg/kg, 2006 JMPR)	Fruit, washed	0.50, 0.49, 0.56, 0.69, 0.41, 0.37, 0.57	0.51	RA-3639/07 M-307290-01-1 09-3235 M-406443-02-1
	Raw juice	0.36, 0.49, 0.59, 0.44, 0.95, 0.66, 0.49	0.57	
	Juice	0.43, 0.53, 0.59, 0.44, 0.86, 0.68, 0.46	0.57	
	Fruit, peeled	0.31, 0.42, 0.35, 0.14, 0.32, 0.22, 0.14	0.27	
	Peel	1.0, 3.1, 2.4, 1.1, 2.9, 3.1, 2.7	2.3	
	Preserve	0.31, 0.34, 0.50, 0.27, 0.59, 0.41, 0.40	0.40	
	Strain rest, wet	0.54, 0.44, 0.68, 0.38, 0.45, 0.36, 0.34	0.46	
	Raw puree	0.64, 0.75, 0.74, 0.75, 0.45, 0.37, 0.40	0.59	
	Puree	0.64, 0.83, 0.65, 0.71, 2.6, 0.32, 0.40	0.88	
	Puree	1.1, 1.4, 1.4	1.3	2006 JMPR evaluation
	Paste	3.0, 3.2, 3.0	3.1	
Spinach	Leaf, washed	1.4, 0.77, 0.92, 0.79	0.97	09-3401

Commodity	Processed commodity	Processing factors Residue: PCB	Processing factor(PF) (best estimate)	Study Document
11.2mg/kg (2006)	Leaf, cooked	0.93, 0.86, 0.88, 0.87	0.89	M-397716-01-1
Lettuce 9.9mg/kg(2006)	Leaf, outer	0.7, 3.0, 2.2, 2.3, 3.4, 2.4	2.3	08-3137
	Head, inner parts	0.04, 0.02, 0.05, 0.17, 0.10, 0.16	0.09	M-348419-01-1
	Leaf, inner	0.02, 0.06, 0.10, 0.21, 0.13, 0.19	0.12	09-3400
	Leaf, inner, washed	0.10, 0.06, 0.04, 0.16, 0.07, 0.08	0.09	M-397890-01-1

Livestock feeding studies

Dairy cow feeding study

A study on the transfer of propamocarb residues from feed to tissues and milk of dairy cows was provided to the meeting (Tew, 2003, M-240879-02-1; Tauber, 2004, M-243564-01-1).

In 3 dose groups of 3 cows, animals were dosed orally with propamocarb at nominal dose rates corresponding to 0.8ppm (1×), 2.4ppm (3×) or 8.0ppm (10×) in feed.

Animals were dosed each morning after milking and test animals were milked each morning and evening. Samples from each animal's evening and morning milk from study Days 0 (the day before the start of dosing), 1, 4, 8, 11, 15, 18, 22, 25, and 27 were pooled. The two aliquots of milk (evening and morning) were combined in volumes proportional to the total evening and morning volumes collected. Milk from one 10× cow and the control cow collected on study Day 27 was separated into milk fat and skim milk and analysed.

All test animals were sacrificed at the end of the 28-day dose period. Muscle, liver, kidney, and fat tissue samples were analysed for propamocarb (AE B039744) and its metabolites propamocarb-N-oxide (AE F155306), oxazolidine-2-one propamocarb (AE F132679), 2-hydroxy-propamocarb (AE F132675) and propamocarb glucuronide. Samples were initially analysed for all analytes except for propamocarb glucuronide. Samples were analysed for propamocarb glucuronide at a later date based on results from the method radio-validation study (conducted after the feeding study). The method used incorporates extraction with acetonitrile (propamocarb, propamocarb-N-oxide, oxazolidine-2-one-propamocarb and 2 hydroxy-propamocarb) or acidic methanol (propamocarb glucuronide) and detection and quantification by LC/MS/MS (Netzband D.J.; 2003; Document No.: M-240905-01-1). The LOQ and LOD in milk, milk fat and skim milk were 0.01 mg/kg and 0.003 mg/kg, respectively, for all matrices. The LOQ and LOD in beef tissues were 0.05 mg/kg and 0.017 mg/kg, respectively, for all matrices.

Table 38 Recoveries of Propamocarb and its metabolites in meat and milk from animals

Sample material	Analyte	Fortification level [mg/kg]	Recovered residues in stored samples					
			Single recoveries [%]			Mean recovery [%]	RSD ^(a)	n
Milk	Propamocarb	0.01	94	79	94	87	11	5
				94	74			
		0.05	90	95		92	3.8	2
	AE F132675	0.01	96	72	82	85	11	5
				81	92			
		0.05	97	81		89	13	2
	AE F155306	0.01	98	98	90	96	3.8	5
				97	99			
		0.05	94	89		92	3.9	2
	AE F132679	0.01	82	82	81	82	3.6	5
			79	87				
		0.05	81	88		84	5.9	2
	Propamocarb	0.01	129			129	-	1

Sample material	Analyte	Fortification level [mg/kg]	Recovered residues in stored samples			
			Single recoveries [%]	Mean recovery [%]	RSD ^(a)	n
	glucuronide					
Milk fat	Propamocarb	0.01	93	93	-	1
		0.05	103	103	-	1
	AE F132675	0.01	85	85	-	1
		0.05	90	90	-	1
	AE F155306	0.01	101	101	-	1
		0.05	103	103	-	1
	AE F132679	0.01	91	91	-	1
		0.05	88	88	-	1
Skim milk	Propamocarb	0.01	90	90	-	1
		0.05	88	88	-	1
	AE F132675	0.01	88	88	-	1
		0.05	82	82	-	1
	AE F155306	0.01	104	104	-	1
		0.05	106	106	-	1
	AE F132679	0.01	83	83	-	1
		0.05	87	87	-	1
Muscle	Propamocarb	0.05	83	83	-	1
		0.25	85	85	-	1
	AE F132675	0.05	86	86	-	1
		0.25	89	89	-	1
	AE F155306	0.05	86	86	-	1
		0.25	83	83	-	1
	AE F132679	0.05	73	73	-	1
		0.25	73	73	-	1
Liver	Propamocarb	0.05	89	89	-	1
		0.25	90	90	-	1
	AE F132675	0.05	76	76	-	1
		0.25	84	84	-	1
	AE F155306	0.05	76	76	-	1
		0.25	81	81	-	1
	AE F132679	0.05	76	76	-	1
		0.25	69	69	-	1
Kidney	Propamocarb	0.05	91	91	-	1
		0.25	82	82	-	1
	AE F132675	0.05	96	96	-	1
		0.25	100	100	-	1
	AE F155306	0.05	86	86	-	1
		0.25	86	86	-	1
	AE F132679	0.05	112	112	-	1
		0.25	102	102	-	1
Fat	Propamocarb	0.05	97	97	-	1
		0.25	97	97	-	1
	AE F132675	0.05	76	76	-	1
		0.25	74	74	-	1
	AE F155306	0.05	78	78	-	1
		0.25	79	79	-	1
	AE F132679	0.05	82	82	-	1
		0.25	85	85	-	1
	Propamocarb glucuronide	0.05	108 83 118	103	18	3
		0.50	103	103	-	1

Sample material	Analyte	Fortification level [mg/kg]	Recovered residues in stored samples			
			Single recoveries [%]	Mean recovery [%]	RSD ^(a)	n
	Propamocarb glucuronide	0.05	104	104	-	1

Milk samples from study days 11 through 27 were analysed. The only measurable residues found in milk were from cows dosed at the 10× level and these residues consisted entirely of metabolite 2-hydroxy-propamocarb. The average residues of 2-hydroxy-propamocarb in whole milk from 10× cows ranged from 0.012 to 0.014 mg/kg with a maximum of 0.019 mg/kg at day 22. 2-hydroxy-propamocarb residues were detectable, but not quantifiable at the 3× dose level. Residues of propamocarb-N-oxide were detectable, but not quantifiable at the 10× dose level. Residues of propamocarb and oxazolidine-2-one propamocarb were not detectable in milk at the 10× level. No detectable residues of the parent or either metabolite were found at the 1× dose level. No detectable residues of any compound were found in any control sample.

Milk from one of the 10× cows (cow #13) was collected on study Day 27 and separated into milk fat and skim milk. Residues in milk fat and skim milk were 0.013 mg/kg and 0.014 mg/kg, respectively. The residue of 2 hydroxy-propamocarb, found in the corresponding whole milk sample, was 0.014 mg/kg. These data indicate that 2 hydroxy-propamocarb does not concentrate in milk fat.

Beef tissue samples were analysed for propamocarb, 2-hydroxy-propamocarb, propamocarb-N-oxide, oxazolidine-2-one propamocarb and propamocarb glucuronide. No detectable residues of parent or metabolite were found in any control tissue. No detectable residues of parent or metabolite were found in any muscle sample. In liver, residues consisted entirely of the conjugated metabolite propamocarb glucuronide. The maximum propamocarb glucuronide residues in liver were 0.218 mg/kg (10×), 0.099 (3×) and <0.05 mg/kg (1×). In kidney, residues of propamocarb glucuronide were detected, but were below the LOQ in all 10× cows. The parent compound was detected in kidney from one 10× cow. A detectable, but not quantifiable, residue of oxazolidine-2-one propamocarb was also found in kidney from one 10× cow. In fat, the parent compound was found at levels <LOQ in two 10× cows.

Table 39 Residues of Propamocarb and its metabolites in whole milk

Day	Dose group	Residues (mg/kg)				
		Propamocarb	2-hydroxy-propamocarb	Propamocarb-N-oxide	Oxazolidine-2-one propamocarb	Propamocarb glucuronide
11	1×	ND	ND	ND	ND	not analysed
	3×	ND	< LOQ	ND	ND	not analysed
	10×	ND	0.012 ^a	< LOQ	ND	not analysed
15	1×	Not Analysed				
	3×	ND	< LOQ	ND	ND	not analysed
	10×	ND	0.013 ^b	< LOQ	ND	not analysed
18	1×	Not Analysed				
	3×	ND	< LOQ	ND	ND	not analysed
	10×	ND	0.013 ^c	< LOQ	ND	not analysed
22	1×	Not Analysed				
	3×	ND	< LOQ	ND	ND	not analysed
	10×	ND	0.013 ^d	< LOQ	ND	not analysed
25	1×	Not Analysed				
	3×	ND	< LOQ	ND	ND	not analysed
	10×	ND	0.013 ^e	< LOQ	ND	not analysed
27	1×	Not Analysed				
	3×	ND	< LOQ	ND	ND	not analysed
	10×	ND	0.014 ^f	< LOQ	ND	ND

ND: < LOD LOD = 0.003 mg/kg and LOQ = 0.01 mg/kg for all analytes in whole milk

^a average of three values : < 0.01; 0.013 and 0.012 mg/kg

^b average of three values : 0.013; 0.015 and 0.011 mg/kg

^c average of three values : 0.010; 0.015 and 0.013 mg/kg

^d average of three values : < 0.01; 0.019 and 0.011 mg/kg

^e average of three values : 0.012; 0.017 and 0.010 mg/kg

^f average of three values : 0.013; 0.016 and 0.014 mg/kg

Table 40 Residues of Propamocarb and its metabolites in skim milk

Day	Dose group	Residues (mg/kg)				
		Propamocarb	2-hydroxy-propamocarb	Propamocarb-N-oxide	Oxazolidine-2- one propamocarb	Propamocarb glucuronide
27	10×	ND	0.014	< LOQ	ND	not analysed

ND: < LOD LOD = 0.003 mg/kg and LOQ = 0.01 mg/kg for all analytes in skim milk

Table 41 Residues of Propamocarb and its metabolites in milk fat

Day	Dose group	Residues (mg/kg)				
		Propamocarb	2-hydroxy-propamocarb	Propamocarb-N-oxide	Oxazolidine-2- one propamocarb	Propamocarb glucuronide
27	10×	ND	0.013	< LOQ	ND	not analysed

ND: < LOD LOD = 0.003 mg/kg and LOQ = 0.01 mg/kg for all analytes in milk fat

Table 42 Residues of Propamocarb and its metabolites in beef muscle

Day	Dose group	Residues (mg/kg)				
		Propamocarb	2-hydroxy-propamocarb	Propamocarb-N-oxide	Oxazolidine-2- one propamocarb	Propamocarb glucuronide
28	1×	not analysed				
	3×	not analysed				
	10×	ND	ND	ND	ND	ND

ND: < LOD LOD = 0.017 mg/kg and LOQ = 0.05 mg/kg for all analytes in beef muscle

Table 43 Residues of Propamocarb and its metabolites in beef liver

Day	Dose group	Residues (mg/kg)				
		Propamocarb	2-hydroxy-propamocarb	Propamocarb-N-oxide	Oxazolidine-2- one propamocarb	Propamocarb glucuronide
28	1×	not analysed				
	3×	not analysed				0.088 ^a
	10×	ND	ND	ND	ND	0.183 ^b

ND: < LOD LOD = 0.017 mg/kg and LOQ = 0.05 mg/kg for all analytes in beef liver

^a average of three values : 0.077; 0.087 and 0.099 mg/kg

^b average of three values : 0.165; 0.165 and 0.218 mg/kg

Table 44 Residues of Propamocarb and its metabolites in beef kidney

Day	Dose group	Residues (mg/kg)				
		Propamocarb	2-hydroxy-propamocarb	Propamocarb-N-oxide	Oxazolidine-2- one propamocarb	Propamocarb glucuronide
28	1×	not analysed				
	3×	not analysed				
	10×	< LOQ	ND	ND	< LOQ	< LOQ

ND: < LOD LOD = 0.017 mg/kg and LOQ = 0.05 mg/kg for all analytes in beef kidney

Table 45 Residues of Propamocarb and its metabolites in beef fat

Day	Dose group	Residues (mg/kg)				
		Propamocarb	2-hydroxy-propamocarb	Propamocarb-N-oxide	Oxazolidine-2- one propamocarb	Propamocarb glucuronide
28	1×	not analysed				
	3×	not analysed				
	10×	< LOQ	ND	ND	ND	ND

ND: < LOD LOD = 0.017 mg/kg and LOQ = 0.05 mg/kg for all analytes in beef fat.

Laying hen feed study

The remaining three of the six subgroups of the 10× dose group hens entered into the depuration phase of the study, where eggs and tissues were analysed throughout a three week period following the administration of the last dose. On day 42, egg and tissue samples from six hens (two control and four 10× dose group hens) in the depuration phase were analysed. On study day 49, egg and tissue samples from another six hens (two control and four 10× dose group hens) in the depuration phase were analysed. On study day 56, egg and tissue samples from the remaining six hens (two control and four 10× dose group hens) in the depuration phase were collected and analysed.

Tissue and egg samples were analysed for total propamocarb residue (propamocarb and N-desmethyl propamocarb) by high performance liquid chromatography-electrospray ionization / tandem mass spectrometry (LC-MS/MS) using matrix matched standards. The limit of quantitation (LOQ) was 0.01 mg/kg for each analyte in eggs and poultry tissues. The LOD for Propamocarb were 0.0024 mg/kg in eggs, 0.0013 mg/kg in liver, 0.0010 mg/kg in muscle, and 0.0014 mg/kg in fat. The LOD for N-desmethyl propamocarb were 0.0033 mg/kg in eggs, 0.0011 mg/kg in liver, 0.0011 mg/kg in muscle, and 0.0028 mg/kg in fat.

Table 46 Recoveries of propamocarb and N-desmethyl propamocarb in eggs, fat, liver and muscle tissues

Sample material	Analyte	Fortification level [mg/kg]	Recovered residues in stored samples								
			Single recoveries [%]						Mean recovery [%]	RSD (a)	n
Eggs	N-desmethyl Propamocarb	0.010	98	82	107	108	99	82	93	12	8
				81	91						
		0.100	106	99	104	78	86	82	93	13	6
		0.250	87	88	90	76	76	77	84	6.4	13
			85	86	89	79	83	84			
			91								
	0.500	81	75	81	83	94	93	83	7.6	9	
			80	79	82						
	Propamocarb	0.010	105	90	105	104	100	87	96	8.4	8
				88	90						
0.100		85	87	85	90	92	90	88	3.3	6	
0.250		82	86	87	82	84	82	89	7.6	13	
		90	93	94	89	96	94				
		105									
	0.500	93	89	94	93	101		94	4.3	9	

Sample material	Analyte	Fortification level [mg/kg]	Recovered residues in stored samples								
			Single recoveries [%]					Mean recovery [%]	RSD (a)	n	
			100	92	91	97					
Fat	N-desmethyl Propamocarb	0.010	96	82	107	91	90	106	95	9.3	7
			96								
		0.100	90	87	97	90	88	91	4.3	5	
	0.250	85	103	91	74		88	14	4		
	Propamocarb	0.010	103	97	109	100	100	103	4.3	7	
			108	103							
0.100		81	83	90	83	93	86	6.0	5		
	0.250	81	100	92	92		91	8.6	4		
Liver	N-desmethyl Propamocarb	0.010	93	97	92	89	95	99	94	3.5	7
				94							
		0.100	100	96	97	101	90	97	4.5	5	
		0.250	81					81	-	1	
	0.500	72	72	69			71	2.4	3		
	Propamocarb	0.010	102	96	100	106	103	102	3.9	7	
			101	108							
		0.100	88	83	83	90	98	88	7.0	5	
		0.250	96					96	-	1	
0.500		80	83	78			80	3.1	3		
Muscle	N-desmethyl Propamocarb	0.010	97	95	97	100	90	98	96	3.5	7
				93							
		0.100	86	88	86	105	79	89	11	5	
	0.250	75	78	74	90		79	9.3	4		
	Propamocarb	0.010	95	98	97	101	92	99	97	3.0	7
				97							
0.100		84	88	85	101	86	89	7.9	5		
	0.250	84	85	83	90		86	3.6	4		

Propamocarb residues in the eggs were below LOD (0.0024 mg/kg) at Day 42, 49 and 56. N-desmethyl propamocarb residues in the eggs were 0.0136 at Day 42 and less than LOD (0.0033 mg/kg) at Day 49 and 56, respectively.

Propamocarb and N-desmethyl propamocarb residues in fat, liver, and muscle in the depuration phase were below the corresponding LODs.

The highest mean propamocarb residues were found in the 35 days eggs, 10× dose group. In eggs of the 35th days, the average propamocarb residue was 0.056 mg/kg in the 10× dose group, 0.016 mg/kg in the 3× dose group, 0.005 mg/kg in the 1× dose group and 0.0012 mg/kg in the 0.3× dose group. Maximum residues in anyone hen were 0.062 mg/kg in the 10× dose group, 0.016 mg/kg in the 3× dose group, 0.005 mg/kg in the 1× dose group and 0.0013 mg/kg in the 0.3× dose group. Values are presented in Table 40.

Table 47 Residues of propamocarb and its metabolite N-desmethyl propamocarb in eggs

Day	Dose group	Residues (mg/kg)						
		Propamocarb			N-desmethyl Propamocarb			Total Propamocarb
		Min	Max	Mean	Min	Max	Mean	Mean
2	10×	0.0104	0.0182	0.0140	0.0351	0.0608	0.0480	0.0620
4	10×	0.0257	0.0302	0.0283	0.1300	0.1480	0.1402	0.1685
7	10×	0.0344	0.0380	0.0363	0.2052	0.2484	0.2241	0.2604
14	10×	0.0334	0.0408	0.0355	0.2298	0.2694	0.2457	0.2811
21	10×	0.0334	0.0442	0.0388	0.2500	0.3193	0.2864	0.3252
28	10×	0.0380	0.0476	0.0428	0.2675	0.3188	0.2922	0.3350
30	10×	0.0437	0.0520	0.0491	0.2854	0.3293	0.3015	0.3506
35	0.3×	0.0010	0.0013	0.0012	0.0102	0.0132	0.0116	0.0128
	1×	0.0042	0.0050	0.0047	0.0368	0.0424	0.0389	0.0436
	3×	0.0151	0.0162	0.0157	0.0867	0.0991	0.0921	0.1078
	10×	0.0487	0.0615	0.0559	0.3201	0.4053	0.3458	0.4017

Day	Dose group	Residues (mg/kg)						
		Propamocarb			N-desmethyl Propamocarb			Total Propamocarb
42	10×	<LOD	<LOD	<LOD	0.0128	0.0142	0.0136	0.0160
49	10×	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
56	10×	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD

The LOD in eggs was 0.0024 mg/kg for propamocarb, 0.0033 mg/kg for N-desmethyl propamocarb and 0.0058 mg/kg for total propamocarb.

In the 35 day fat tissue, the highest mean propamocarb residues were 0.0097 mg/kg in the 10× dose group, 0.0021 mg/kg in the 3× dose group and below 0.0014 mg/kg (LOD) in the 1× and 0.3× dose groups. Maximum residues in anyone hen were 0.028 mg/kg in the 10× dose group, 0.0036 mg/kg in the 3× dose group, <LOD in the 1× and 0.3× dose group.

In the 35 day fat tissue, the highest mean N-desmethyl propamocarb residues were 0.0492 mg/kg in the 10× dose group, 0.0062 mg/kg in the 3× dose group, 0.0039 mg/kg in the 1× dose group and 0.0028 mg/kg in the 0.3× dose group. Values are presented in Table 41.

Table 48 Residues of propamocarb and its metabolite N-desmethyl propamocarb in fat

Day	Dose group	Residues (mg/kg)						
		Propamocarb			N-desmethyl Propamocarb			Total Propamocarb
		Min	Max	Mean	Min	Max	Mean	Mean
35	0.3×	<LOD	<LOD	<LOD	<LOD	0.0028	0.0028	0.0042
	1×	<LOD	<LOD	<LOD	0.0035	0.0046	0.0039	0.0053
	3×	<LOD	0.0036	0.0021	0.0034	0.0111	0.0062	0.0083
	10×	<LOD	0.0282	0.0097	0.0093	0.1445	0.0492	0.0589
42	10×	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
49	10×	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
56	10×	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD

The LOD in fat was 0.0014 mg/kg for Propamocarb, 0.0028 mg/kg for N-desmethyl Propamocarb and 0.0042 mg/kg for total Propamocarb.

In the 35 day liver tissue, the highest mean propamocarb residues were 0.0159 mg/kg in the 10× dose group, 0.0082 mg/kg in the 3× dose group, 0.0037 mg/kg in the 1× dose group and 0.0019 mg/kg in the 0.3× dose group. Maximum residues in anyone hen were 0.025 mg/kg in the 10× dose group, 0.015 mg/kg in the 3× dose group, 0.0055 mg/kg in the 1× dose group and 0.0021 mg/kg in the 0.3× dose group.

In the 35 day liver tissue, the highest mean N-desmethyl propamocarb residues were 0.119 mg/kg in the 10× dose group, 0.0393 mg/kg in the 3× dose group, 0.0239 mg/kg in the 1× dose group and 0.0083 mg/kg in the 0.3× dose group. Values are presented in Table 42.

Table 49 Residues of propamocarb and its metabolite N-desmethyl propamocarb in liver

Day	Dose group	Residues (mg/kg)						
		Propamocarb			N-desmethyl propamocarb (expressed as propamocarb)			Total Propamocarb
		Min	Max	Mean	Min	Max	Mean	Mean
35	0.3×	0.0015	0.0021	0.0019	0.0074	0.0101	0.0083	0.0102
	1×	0.0021	0.0055	0.0037	0.0147	0.0346	0.0239	0.0275
	3×	0.0035	0.0153	0.0082	0.0271	0.0631	0.0393	0.0476
	10×	0.0069	0.0247	0.0159	0.1051	0.1299	0.1190	0.1349
42	10×	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
49	10×	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
56	10×	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD

The LOD in liver was 0.0013 mg/kg for propamocarb, 0.0011 mg/kg for N-desmethyl Propamocarb and 0.0024 mg/kg for total Propamocarb.

In the 35 day muscle tissue, the highest mean propamocarb residues were 0.0104 mg/kg in the 10× dose group, 0.0050 mg/kg in the 3× dose group, 0.0022 mg/kg in the 1× dose group and 0.0010 mg/kg in the 0.3× dose group. Maximum residues in anyone hen were 0.013 mg/kg in the 10× dose group, 0.0086 mg/kg in the 3× dose group, 0.0028 mg/kg in the 1× dose group and 0.0010 mg/kg in the 0.3× dose group.

In the 35 day muscle tissue, the highest mean N-desmethyl propamocarb residues were 0.1017 mg/kg in the 10× dose group, 0.0250 mg/kg in the 3× dose group, 0.0149 mg/kg in the 1× dose group and 0.0053 mg/kg in the 0.3× dose group. Values are presented in Table 43.

Table 50 Residues of propamocarb and its metabolite N-desmethyl propamocarb in muscle

Day	Dose group	Residues (mg/kg)						
		Propamocarb			N-desmethyl propamocarb			Total Propamocarb
		Min	Max	Mean	Min	Max	Mean	Mean
35	0.3×	<LOD	0.0010	0.0010	0.0040	0.0065	0.0053	0.0063
	1×	0.0018	0.0028	0.0022	0.0120	0.0202	0.0149	0.0171
	3×	0.0028	0.0086	0.0050	0.0197	0.0321	0.0250	0.0300
	10×	0.0084	0.0129	0.0104	0.0940	0.1033	0.1017	0.1120
42	10×	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
49	10×	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
56	10×	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD

The LOD in muscle was 0.0010 mg/kg for propamocarb, 0.0011 mg/kg for N-desmethyl Propamocarb and 0.0021 mg/kg for total Propamocarb.

Conclusion

Feed consumption, body weights, and egg production were not adversely affected by daily oral administration of propamocarb to laying hens for 35 consecutive days at the average dose rate of 1.2 mg/kg feed (0.3×), 4.1 mg/kg feed (1×), 12.3 mg/kg feed (3×), and 41.1 mg/kg feed (10×).

The mean maximum propamocarb residues in the egg samples from the 10×, 3×, 1×, and 0.3× feeding levels were at day 35: 0.056 mg/kg, 0.016 mg/kg, 0.0047 mg/kg, and 0.0012 mg/kg respectively.

In the fat, the mean maximum propamocarb residues at day 35 was 0.0097 mg/kg in the 10× dose group, 0.0021 mg/kg in the 3× dose group, <LOD in the 1× and 0.3× dose group.

In the liver, the mean maximum propamocarb residues at day 35 was 0.016 mg/kg in the 10× dose group, 0.0082 mg/kg in the 3× dose group, 0.0037 mg/kg in the 1× dose group, and 0.0019 mg/kg in the 0.3× dose group.

In the muscle tissue, the mean maximum propamocarb residues at day 35 was 0.010 mg/kg in the 10× dose group, 0.005 mg/kg in the 3× dose group, 0.0022 mg/kg in the 1× dose group, and 0.001 mg/kg in the 0.3× dose group.

Depuration of residue (mean total Propamocarb residues) was observed from the eggs from day 35 to day 42; 0.402 mg/kg to 0.016 mg/kg respectively. No propamocarb and N-desmethyl propamocarb residue from eggs were observed at subsequent time points, <LOQ (0.01 mg/kg). In fat, liver, and muscle depuration residues were <LOQ (0.01 mg/kg) at Day 42 (and subsequent time points) indicating that propamocarb and N-desmethyl propamocarb residues were rapidly eliminated from the animals.

APPRAISAL

Propamocarb is a systemic carbamate fungicide with specific activity against Oomycete species that cause seed, seedling, root, foot and stem rots and foliar diseases in a number of edible crops. The compound was first evaluated by the JMPR in 1984, and was then reviewed by JMPR in 1986, 1987 and 2005, an ADI of 0–0.4 mg/kg bw and an ARfD of 2 mg/kg bw were established in 2005. Propamocarb was evaluated within the periodic review programme for residue and analytical aspects by JMPR in 2006.

The residue definition in plant and animal products for both enforcement and dietary intake purposes is propamocarb (free base. Propamocarb is not considered fat soluble.

The meeting received information on GAP and supervised residue trials in/on onion, leek, broccoli, cauliflower, Brussels sprouts, head cabbage, kale and lima bean. A new analytical method was provided for propamocarb in animal matrices, as well as new studies in laying hen metabolism, feeding studies on lactating cow and laying hens, rotational crops and processed commodities.

Animal metabolism

Laying hen

A metabolism study with propamocarb in laying hens was received by the Meeting. Laying hens were orally dosed via capsule with [¹⁴C] propamocarb hydrochloride at a rate of approximately 19 ppm in the feed (1.0 mg/kg body weight) once daily for fourteen consecutive days. The majority of the residues (92% to 99% of the TRR) in the egg and tissues could be extracted.

The TRR in eggs increased from day-1 through too day-7 and reached a plateau, at day-8, of about 0.25 mg eq/kg. The TRR in the tissue samples were highest in liver (0.492 mg/kg) with 0.14 mg/kg in leg muscle, 0.12 mg/kg in breast muscle and 0.042 mg/kg in fat (0.065 mg/kg in renal fat).

The major residues in the eggs and tissues were parent propamocarb (2% in fat, 5% in muscle, 9% in liver and 12% in eggs) and desmethyl propamocarb (6% in fat, 22% in liver, 29% in muscle, and 45% in eggs). The minor residues found in the eggs and tissues were bis-desmethyl-propamocarb (< 1% to 7%) and propamocarb-N-oxide (< 1%).

In summary, the metabolic degradation of propamocarb is rapid and extensive in laying hens with desmethyl-propamocarb and propamocarb being the major residues in all tissues (mostly in liver, eggs and muscle). Demethylation is the main route of metabolism for the parent compound. A minor route of metabolism involves oxidation of the tertiary nitrogen to form propamocarb-N-oxide. The pathway and metabolism profile found in laying hen is consistent to that of the lactating cow metabolism study evaluated by 2006 JMPR.

Field crop rotational studies

Rotational crop studies were conducted in Germany and France during 2002/2003. Four applications of 72.2, 36.1, 3.61 and 3.61 kg ai/ha propamocarb were applied to cabbage as the primary crop, and Lamb's lettuce and wheat were grown as rotational crops with a plant-back interval (PBI) of 52–102 days. No residues of Propamocarb were found above LOQ in Lamb's lettuce, wheat grain, wheat (green material) and wheat straw.

Four rotational crop studies were conducted in the Netherlands, France, Spain and Italy from 2008 to 2010. Three spray applications of 1.325 kg ai/ha. propamocarb with 7–13days interval were applied to lettuce as primary crop, and carrots, lettuce and wheat or barley were grown as rotational crops, with plant-back intervals (PBI) of 26–46 days. No residues of propamocarb were found above LOQ in any of these rotational crops.

In rotational field studies with wheat, sugar beet, table beet, dry bean and soya beans conducted in USA in 1997 (four applications at rates of 1.68 kg ai/ha.), which was evaluated by 2006 JMPR, there was no residues detected in wheat grain and straw, soya bean seed, beetroot and beet

tops and dry bean from a 30 day PBI, and residues were in the range of 0.229 to 0.51 mg/kg only in wheat hay and forage from a 30 day PBI.

The meeting noted that the GAP of propamocarb (3–4 applications of 1 kg ai/ha.) approximates the application rate in the new studies and was lower than in the 1997 studies. The Meeting agreed that no residue in food and feed commodities are expected from rotational crops following the registered uses.

Methods of analysis

The meeting received the information on multiresidue QuEChERS Method (BCS method ID 01205) for analysis of residues of propamocarb in meat (cattle), liver (cattle), kidney (cattle), fat (cattle), milk (cattle) and egg (chicken) (mass transitions at m/z 102 and 74). The limit of quantification (LOQ) was at 0.01 mg/kg in meat, liver, kidney, fat, milk and egg. The method was confirmed by results of an independent laboratory validation which repeated mean recovery rates of 80–88% in all matrices.

Stability of residues in stored analytical samples

The meeting received information on storage stability of propamocarb in frozen cabbage samples. Propamocarb hydrochloride was stable for a period of at least 39 months.

Results of supervised residue trials on crops

The meeting received supervised residue trial data for propamocarb in/on bulb onion, leek, broccoli, cauliflower, Brussels sprouts, head cabbage, kale and lima bean.

Bulb vegetables

Bulb onion

In 21 trials conducted in Europe according to GAP in Italy (three foliar applications of 1.0 kg ai/ha, 7days PHI), residues were < 0.01(2), 0.01, 0.02(4), 0.03, 0.04, 0.05(5), 0.07, 0.21, 0.41, 0.52, 0.86, 1.3 and 1.4 mg/kg. The Meeting estimated a maximum residue level of 2 mg/kg, a STMR of 0.05 mg/kg and a HR of 1.4 mg/kg for propamocarb in onion bulb.

Leek

In 12 trials conducted in Europe according to GAP in Albania (four foliar applications at 1.0 kg ai/ha, 14days PHI), residues were 0.24, 0.74, 0.9, 1.1(2), 2.4, 2.6, 4.0, 4.4, 5.5, 11 and 15 mg/kg. The meeting estimated a maximum residue level of 30 mg/kg, a STMR of 2.5 mg/kg and a HR of 15 mg/kg for propamocarb in leek.

Brassica vegetables

Broccoli

In ten trials conducted in Europe according to GAP in Spain (three foliar applications of 1.0 kg ai/ha, 14 days PHI), residues were 0.01, 0.16, 0.17, 0.21, 0.29(2), 0.32, 0.63, 0.97 and 1.7 mg/kg. The meeting estimated a maximum residue level of 3 mg/kg, a STMR of 0.29 mg/kg and a HR of 1.7 mg/kg for propamocarb in broccoli.

Cauliflower

In ten trials conducted in Europe according to GAP in Greece (three foliar applications of 1.0 kg ai/ha, 14 days PHI), residues were < 0.01, 0.01, 0.02(2), 0.03, 0.04, 0.06, 0.08, 0.20 and 0.82 mg/kg. The meeting estimated a maximum residue level of 2 mg/kg, a STMR of 0.035 mg/kg and a HR of 0.82 mg/kg for propamocarb in cauliflower to replace the previous recommendation of 0.2 mg/kg.

Brussels sprouts

In eight trials conducted in Europe matching the GAP of Belgium (three foliar applications of 1.0 kg ai/ha, 14 days PHI), residues were 0.20, 0.25, 0.24, 0.46, 0.48, 0.49, 0.64 and 1.3 mg/kg. The meeting estimated a maximum residue level of 2 mg/kg, a STMR of 0.47 mg/kg and a HR of 1.3 mg/kg for propamocarb in Brussels sprouts.

Head cabbage

In 12 trials conducted in Europe matching the GAP of Spain (three foliar applications at 1.0 kg ai/ha, 14 days PHI), residue were 0.02, 0.03, 0.06, 0.08, 0.13, 0.18, 0.21, 0.23, 0.24, 0.28, 0.32 and 0.36 mg/kg. The meeting estimated a maximum residue level of 1 mg/kg, a STMR of 0.195 mg/kg and a HR of 0.36 mg/kg for propamocarb in head cabbage.

*Leafy Vegetables**Kale*

In nine trials conducted in Europe matching the GAP of Belgium (three foliar applications of 1.0 kg ai/ha, 14 days PHI), residues were 0.33, 0.39, 0.46, 3.9, 4.0, 4.0, 5.2, 10.7 and 11.8 mg/kg. The meeting estimated a maximum residue level of 20 mg/kg, a STMR of 4.0 mg/kg and a HR of 11.8 mg/kg for propamocarb in kale.

*Legume vegetables**Lima Beans*

In three trials conducted in the United States according to US GAP (four foliar applications at 1.0 kg ai/ha, 0 days PHI), residues in seed without pods were 0.26, 0.42 and 0.43 mg/kg, residues in forage were 40.6, 83.6 and 47.4 mg/kg. As only three trials were conducted according to GAP the meeting could not recommend a maximum residue level for propamocarb in lima bean seed without pods.

FATE OF RESIDUES DURING PROCESSING

The Meeting received information on the fate of propamocarb residues during the processing of tomatoes, spinach and lettuce, and a study on the nature of residues in processed commodities. Propamocarb hydrochloride was stable under all conditions of processing tested (pasteurisation, baking, brewing, boiling and sterilisation). Processing factors based on the residue of propamocarb are listed in table below. Using the STMR_{RAC} obtained from propamocarb use, the Meeting estimated STMR-P values for processed commodities to be used in dietary intake calculations and/or livestock dietary burden calculations.

Summary of the processing studies in tomato, spinach and lettuce

Commodity	Processed commodity	Processing factors Residue: PCB	Processing factor(PF) (Mean or best estimate)	STMR-P= STMR _{RAC} X PF(mg/kg)
Tomato 0.515 mg/kg ^a	Juice	0.43, 0.44, 0.46, 0.53, 0.59, 0.68, 0.86	0.53	0.27
	Preserve	0.27, 0.31, 0.34, 0.40, 0.41, 0.50, 0.59	0.40	0.21
	Puree	0.32, 0.40, 0.64, 0.65, 0.71, 0.83, 1.1 ^a , 1.4 ^a , 1.4 ^a , 2.6	0.77	0.40
	Paste a	3.0, 3.2, 3.0	3.1	1.54
Spinach 11.2 mg/kg ^a	Leaf, cooked	0.86, 0.87, 0.88, 0.93	0.88	9.9

^a Data from 2006 JMPR evaluation

Farm animal feeding studies

The meeting received information on feeding studies with lactating cows and laying hens.

Dairy cow feeding study

No measurable residues of propamocarb or its metabolites 2-hydroxy-propamocarb, propamocarb-N-oxide, and oxazolidine-2-one propamocarb were detected in milk or edible tissues of cattle exposed to propamocarb at levels of 0.8, 2.4 or 8 ppm administered daily for 28 days, except for milk, where levels of up to 0.019 mg/kg of 2-hydroxy-propamocarb were found in the highest dose group (8 ppm). These residues were equally distributed between the milk fat and the skim milk. LOQs were 0.01 mg/kg (milk) and 0.05 mg/kg (tissues).

Laying hen feed study

A residue transfer study of propamocarb was conducted in laying hens. Hens were dosed orally for 35 consecutive days at the rates of 1.2 ppm (0.3×), 4.1 ppm (1×), 12.3 ppm (3×), and 41.1 ppm (10×) in the feed.

The mean (maximum) propamocarb residues in the egg samples from 1× and 0.3× feeding levels were: 0.0047 mg/kg (0.005 mg/kg) and 0.012 mg/kg (0.013 mg/kg) at Day 35.

In fat, the mean (maximum) propamocarb residues at Day 35 were < LOD (0.0014 mg/kg) in 1× and 0.3× dose groups.

In liver, the mean (maximum) propamocarb residues at Day 35 were 0.0037 mg/kg (0.0055 mg/kg) in the 1× dose group, and 0.0019 mg/kg (0.0021 mg/kg) in the 0.3× dose group.

In muscle, the mean (maximum) propamocarb residues at Day 35 were 0.0022 mg/kg (0.0028 mg/kg) in the 1× dose group, and 0.001 mg/kg (0.001 mg/kg) in the 0.3× dose group.

Animal commodity maximum residues levels

The dietary burdens were estimated using the OECD diets listed in Appendix IX of the 2009 Edition of FAO Manual. Potential cattle feed items include: cabbage, kale and potatoes (including by-products). Dietary burden calculations for beef cattle and dairy cattle and poultry are provided below.

Summary of livestock dietary burden (ppm of dry matter diet)

	US-Canada		EU		Australia		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	0.38	0.20	16.16	5.58	0.11	0.05	0.00	0.00
Dairy cattle	0.13	0.07	16.11	5.53	31.55a, b	10.70c, d	0.00	0.00
Poultry-broiler	0.00	0.00	0.10	0.04	0.00	0.00	0.00	0.00
Poultry-layer	0.00	0.00	4.03e	1.37f	0.00	0.00	0.00	0.00

^a Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian meat

^b Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk

^c Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian meat

^d Highest mean dairy cattle dietary burden suitable for STMR estimates for mammalian milk

^e Highest maximum poultry dietary burden suitable for MRL estimates for poultry meat and eggs

^f Highest mean poultry dietary burden suitable for STMR estimates for poultry meat and eggs

The propamocarb dietary burden for animal commodity MRL and STMR estimation (residue levels in animal feed expressed on dry weight) are 31.55 and 10.7 ppm for mammalian products (based primarily on the new use on kale), and the maximum and mean dietary burdens are 4.03 and 1.37 ppm for poultry products.

Mammalian products

The Meeting noted that the maximum cattle dietary burden was about 3–4 times higher than the dose level (8 ppm) in the feeding study and the metabolism study of lactating cow (11.5 ppm), and could not estimate maximum residue levels for mammalian commodities. The Meeting agreed to withdraw the previous MRLs of 0.01* mg/kg for milk, meat from mammals other than marine mammals, and edible offal, mammals.

Poultry products

The MRLs for poultry products were estimated using the maximum dietary burden and the highest residues obtained from the laying hen feeding study. For the estimation of the STMRs, the mean dietary burden and mean residues on poultry products from the feeding study were used.

The maximum dietary burden of 4.03 ppm DM matches the 1× dose level of the laying hen feeding study. Therefore, the residue levels reported in the feeding study were used as a direct estimate of the residue levels in poultry muscle, fat, liver and eggs resulting from the dietary burden. Similarly, the STMR of 1.37 ppm approximates the lowest dose level of the feeding study (1.2 ppm). Therefore, as an estimate for the STMR in the poultry products, the mean residue levels from the feeding study at this level were used. The estimated MRLs and STMRs are summarized in following table.

	Feed level	Residues	Feed level	Residues (mg/kg) in		
	(ppm) for egg residues	(mg/kg) in egg	(ppm) for tissue residues	Muscle	Liver	Fat
MRL poultry						
Feeding study ^a	4.1	0.005	4.1	0.0028	0.0055	< LOD ^c
Dietary burden and high residue	4.03	0.005	4.03	0.0028	0.0055	< LOD ^c
STMR poultry						
Feeding study ^b	1.2	0.0012	1.2	0.0010	0.0019	< LOD ^c
Dietary burden and residue estimate	1.37	0.0012	1.37	0.0010	0.0019	< LOD ^c

^a Highest residues for tissues and highest residues for eggs

^b Mean residues for tissues and mean residues for eggs

^c LOD in fat was 0.0014 for propamocarb

The meeting estimated the following STMR values: eggs 0.001 mg/kg, muscle 0.001 mg/kg, liver 0.002 mg/kg and fat 0.001 mg/kg. The following HR values are also estimated: eggs of 0.005 mg/kg, muscles of 0.003 mg/kg, liver of 0.006 mg/kg and fat of 0.001 mg/kg.

The meeting recommended the maximum residue levels of 0.01* mg/kg for poultry fat, and confirmed its previous recommendation for maximum residue levels of 0.01* mg/kg for eggs, poultry meat, and poultry, edible offal of.

RECOMMENDATIONS

Summary of the recommendations for the MRL, STMR and HR for propamocarb:

Commodity		Recommended MRL (mg/kg)		STMR (mg/kg)	HR (mg/kg)
CCN	Name	New	Previous		
VA0385	Onion	2		0.050	1.4
VB0400	Broccoli	3		0.290	1.7
VB0404	Cauliflower	2	0.2	0.035	0.82
VB0402	Brussels sprouts	2		0.47	1.3
VB0041	Cabbage, Head	1		0.195	0.36
VL0480	Kale	20		4.00	11.8
VA0384	Leek	30		2.50	15.0
PF0111	Poultry fat	0.01*		0.001	0.001
PM0110	Poultry meat	0.01*	0.01*	0.001	0.003
PO0111	Poultry, edible offal of	0.01*	0.01*	0.002	0.006
PE0112	Eggs	0.01*	0.01*	0.001	0.005

Commodity		Recommended MRL (mg/kg)		STMR (mg/kg)	HR (mg/kg)
CCN	Name	New	Previous		
ML0106	Milk	W	0.01*		
MM0095	Meat from mammals other than marine mammals	W	0.01*		
MO0515	Edible offal, mammals	W	0.01*		

DIETARY RISK ASSESSMENT

Long-term intake

The long-term dietary intake for propamocarb (ADI 0–0.4 mg/kg bw) was calculated for the 17 regional diets on the basis of the STMRs estimated by the JMPR in 2006 and the current meeting. The results are shown in Annex 3 of the 2014 Report.

The International Estimated Daily Intakes (IEDI) of propamocarb were 0–1% of the maximum ADI (0.4 mg/kg bw). The Meeting concluded that the long-term intake of residues of propamocarb from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The ARfD for propamocarb is 2 mg/kg bw. The International Estimated Short Term Intake (IESTI) of propamocarb was calculated for the commodities for the commodities for which residue levels were estimated. The results are shown in Annex 4 of the 2014 Report.

The IESTI of propamocarb calculated were up to 20% of the ARfD (2 mg/kg bw) for children and general population. The Meeting concluded that the short-term intake of residues of propamocarb resulting from uses that have been considered by current meeting is unlikely to present a public health concern.

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M-226597-01-1	Klein E. H-J.	2004	Decline of residues in white cabbage, lamb's lettuce and wheat Field Rotation Crop Study
M-349882-02-1	Melrose I.; Portet, 2010 M.,		Determination of the residues of Fosetyl and Propamocarb in/on carrot, lettuce and wheat, winter after spraying of Fosetyl & Propamocarb SL 840 in the Field in Netherlands
M-349137-02-1	Melrose I.; Portet, 2010 M.,		Determination of the residues of Fosetyl and Propamocarb in/on carrot, lettuce and barley, winter after spraying of Fosetyl & Propamocarb SL 840 in the Field in France (North)
M-361470-01-1	Melrose I.; Portet, 2010 M.,		Determination of the residues of Fosetyl and Propamocarb in/on carrot, lettuce and wheat, winter after spraying of Fosetyl & Propamocarb SL 840 in the Field in Spain
M-349147-02-1	Melrose I.; Portet, 2010 M.,		Determination of the residues of Fosetyl and Propamocarb in/on carrot, lettuce and wheat, winter after spraying of Fosetyl & Propamocarb SL 840 in the Field in Italy
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Reference Doc-ID No	Author(s)	Year	Study Title
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M-301257-02-1	Schöning R., Gnielka, A.	2008	Determination of the residues of Propamocarb and Fluopicolide in/on field samples of plant origin (onions, radish, kohlrabi, curly kale) after spray application with Infinito (687.5 SC) and Amendment
M-307731-01-	Melrose, I.; Portet, M.	2008	Determination of the residues of AE C638206 and Propamocarb hydrochloride in/on onion after spraying of AE B066752 04 SC61 A1 (687.5 SC) in the field in Southern France, Spain, Italy and Portugal
M-279693-01-1	Billian, P.	2006	Determination of the Residues of AE C638206 and Propamocarb-hydrochloride in/on broccoli after spraying of AE B066752 04 SC61 A1 (687.5 SC) in the Field in Northern France, the Netherlands, Belgium and Germany
M-307619-01-1	Melrose, I.	2008	Determination of the residues of AE C638206 and Propamocarb hydrochloride in/on broccoli and cauliflower after spraying of AE B066752 04 SC61 A1 (687.5 SC) in the field in Germany, Southern France and Spain
M-280045-01-1	Billian, P.	2006	Determination of the residues of AE C638206 and Propamocarb hydrochloride in/on broccoli after spraying of AE B066752 04 SC61 A1 (687.5 SC) in the field in Greece, Southern France, Spain and Italy
M-307619-01-1	Melrose, I.	2008	Determination of the residues of AE C638206 and Propamocarb hydrochloride in/on broccoli and cauliflower after spraying of AE B066752 04 SC61 A1 (687.5 SC) in the field in Germany, Southern France and Spain
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M-288004-01-1	Gateaud, L.; Ratajczak, M.	2007	Determination of the Residues of AE C638206 and Propamocarb-Hydrochloride in/on Savoy cabbage, Round cabbage and Red cabbage after Spraying of AE B066752 04 SC61 A1 (687.5 SC) in the Field in the Netherlands, Germany and Northern France

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M-300710-01-1	Justus, K.; Kuhnke, G.	2008	[1-14C]Propamocarb hydrochloride: Aqueous hydrolysis under conditions of processing studies
M-307290-01-1	Billian, P.	2008	Determination of the residues of AE C638206 and Propamocarb hydrochloride in/on tomato fruit and fruit for processing and the processed fractions (raw juice; washings; fruit, washed; juice; peel; preserve; fruit, peeled; peeling water; puree; raw puree; strain rest) after spraying of AE B066752 04 SC61 A1 (687.5 SC) in the field in Italy, Spain, Greece and Portugal
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M-348419-01-1	Billian, P.; Reineke, A.	2009	Determination of the residues of Fosetyl and Propamocarb in/on lettuce and processed fractions after spraying of Fosetyl & Propamocarb SL 840 in the Netherlands and Germany
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PROPICONAZOLE (160)

The first draft was prepared by Professor Eloisa Dutra Caldas, University of Brasilia, Brazil

EXPLANATION

The triazole fungicide propiconazole was last evaluated by the JMPR in 2013 for residue aspects. Residue trial data on bean (dry), bean (lima), and bean (snap), mint, pineapple following post-harvest application, barley, oat and wheat were submitted to current Meeting.

Residue definition for plant and animal commodities is *propiconazole* for compliance with the MRL and *propiconazole plus all metabolites convertible to 2,4-dichlorobenzoic acid, expressed as propiconazole* for the estimation of the dietary intakes.

METHODS OF ANALYSIS

Methods where only propiconazole is analysed involves mostly LC-MS/MS detection, with LOQ of 0.01 mg/kg. The common moiety method measure all residues convertible to 2,4-dichlorobenzoic (2,4-DCBA) and are performed using mostly GC-MSD or GC-ECD (after derivatisation of 2,4-DCBA to its methyl ester) with an LOQ of 0.05 mg/kg. These methods have been previously submitted to the JMPR for evaluation, and recovery data was also included in the study reports submitted to the present Meeting.

Samples were analysed within a storage period that was shown not to impact the residue levels, according to storage stability studies evaluated previously by the JMPR, or according to stability studies included in the reports. One exception was

USE PATTERNS

All the supervised trials submitted were conducted in the United States or in Canada. Table 1 shows the registered uses of propiconazole in USA for the crops relevant to this submission.

Table 1 Use patterns of propiconazole in USA

Crop	Formulation	Application				
		Method	Maximum rate (per application)	Number	Interval (days)	DAT (days)
Barley	SE	Foliar	0.125 kg ai/ha	2	14*	7**
Beans, dry and succulent, including lima bean and bean	SE	Foliar	0.126 kg ai/ha	3/year	7-14	7
Mint	SE	Foliar	0.126 kg ai/ha	3	14	7
Oats	SE	Foliar	0.125 kg ai/ha	2	14*	7**
Pineapple	EC	Post harvest dip	0.015 kg ai/100 L water	1	-	Na
Wheat	SE	Foliar	0.125 kg ai/ha	2	14*	7**

* second application following early application can be applied up to full head emergence (BBCH 59).

** Minimum PHI for forage and hay

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Supervised residue trials conducted with propiconazole in Canada and USA were submitted to the Meeting. Studies were conducted according to GLP, and concurrent determination of residues in untreated crops gave residues < LOQ, unless specified otherwise. Residues of propiconazole arising from use patterns where rate or PHI are $\pm 25\%$ of GAP are underlined and considered for estimation of maximum residue levels and STMRs. In each trial, two samples were analysed and the mean value used for the estimation.

Samples were analyzed for total propiconazole, as 2,4-dichlorobenzoic methyl ester, or for propiconazole only.

Pineapple

Three supervised residue trials were conducted in Hawaii in 1997 (Report: IR-4 PR No. 06585). Samples were analyzed for residues of propiconazole and its 2,4-DCBA-containing metabolites using a GC/ECD method, and expressed as total propiconazole. The results from analysis of treated samples are summarized in Table 2.

Table 2 Residues resulting from propiconazole application to pineapple in Hawaii, USA

Crop (Var)	Application			Total propiconazole, mg/kg	Trial
	No.	kg ai/100 L Water	DAT		
Pineapple (Cayenne)	1	0.015	-	<u>3.6</u> (4.1, 3.0)	HI06
	1	0.015 +Fruit Wax	-	0.50, 0.45	
	1	0.015 +Fruit Oil	-	0.56, 0.37	
Pineapple (Cayenne)	1	0.015	-	<u>1.1</u> (1.3, 0.88)	HI07
	1	0.015 +Fruit Wax	-	0.39, 0.34	
	1	0.015 +Fruit Oil	-	0.52, 0.50	
Pineapple (Cayenne)	1	0.015	-	<u>1.8</u> (1.8, 1.7)	HI08
	1	0.015 +Fruit Wax	-	0.42, 0.34	
	1	0.015 +Fruit Oil	-	0.82, 0.50	

Lima Bean (seeds)

Six supervised residue trials were conducted in USA in 2005 (Report: IR-4 PR No. 09437). Samples were analyzed for residues of propiconazole and its 2,4-DCBA-containing metabolites using a GC/ECD method, and expressed as total propiconazole. The results from analysis of treated samples are summarized in Table 3.

Table 3 Residues resulting from propiconazole application to lima bean seed in USA using Tilt 3.6 E formulation

Region	Lima vean variety	Application			Total propiconazole, mg/kg	Trial
		No.	kg ai/ha	DAT		
California	Green Baby 12-98	5	0.11	8	< 0.05 (2)	09437.05-CA163
Idaho	M15	3	0.11	8	0.07 (0.057, 0.088)	09437.05-ID19
Maryland ^a	Burpee Improved	3	0.11	7	< 0.05 (2)	09437.05-MD22
Maryland ^a	Burpee Improved	3	0.11	6	< 0.05 (2)	09437.05-MD23
New Jersey	Baby Lima	3	0.11	7	< 0.05 (2)	09437.05-NJ38
Wisconsin	909 Cyrus	3	0.11	6	< 0.05 (2)	09437.05-WI28

^a same location, same period

Snap bean

Seven supervised residue trials were conducted in USA in 2005 and 2006 (Report: IR-4 PR No. 09295). Samples were analyzed for residues of propiconazole and its 2,4-DCBA-containing metabolites using a GC/ECD method, and expressed as total propiconazole. The results from analysis of treated samples are summarized in Table 4.

Table 4 Residues resulting from propiconazole application to snap beans (pods with seeds) in USA

Region	Snap bean variety	Application			Total propiconazole, mg/kg	Trial
		No.	kg ai/ha	DAT		
California	Jade	3	0.10-0.11	7	0.38 (0.390, 0.36)	09295.05-CA88
Florida	Leon	3	0.10-0.11	7	0.23 (0.254, 0.206)	09295.05-FL34
Michigan	Hercules	3	0.10-0.11	7	0.13 (0.145, 0.124)	09295.05-MI19
N. Carolina	Bronco	3	0.10-0.11	8	0.35 (0.366, 0.329)	09295.05-NC16
Ohio	Jade	3	0.10-0.11	6	0.09 (0.090, 0.087)	09295.05-OH*10
Washington	Jade	3	0.10-0.11	6	0.11 (0.126, 0.093)	09295.05-WA*11
New York	Carson	3	0.10-0.11	7	0.27 (<u>0.290</u> , 0.247)	09295.06-NY28

Dry bean

Twelve supervised residue trials were conducted in USA in 2008 (Report: IR-4 PR No. 02008). Samples were analyzed for residues of propiconazole and its 2,4-DCBA-containing metabolites using a GC/ECD method, and expressed as total propiconazole. The results from analysis of treated samples are summarized in Table 5.

Table 5 Residues resulting from propiconazole application to dry beans in USA using Tilt 3.6 E formulation

Region	Dry bean variety	Application			Total propiconazole, mg/kg ^a	Trial
		No.	kg ai/ha	DAT		
California	Montcalm Dark Red Kidney	3	0.10-0.11	7	0.25 (0.253, 0.243)	02008.08-CA46
Colorado, Fruita	Croissant	3	0.10-0.11	8	0.05 (0.056, < 0.05)	02008.08-CO02
Colorado, Fort Collins	Vision	3	0.10-0.11	5	0.14 (0.153, 0.123)	02008.08-CO03
				7	0.18 (0.134, 0.122)	
				16	0.10 (0.104, 0.098)	
				20	0.10 (0.103, 0.098)	
Idaho	Shoshone Pinto	3	0.10-0.11	5	0.07 (0.068, 0.067)	02008.08-ID16
N. Dakota, Fargo	Ensign	3	0.10-0.11	8	0.10 (0.103, 0.10)	02008.08-ND01
N. Dakota, Fargo	Eclipse	3	0.10-0.11	8	< 0.05 (2)	02008.08-ND02
N. Dakota, Rush River Township	Maverick	3	0.10-0.11	7	0.06 (0.072, < 0.05)	02008.08-ND03
N. Dakota, Minot ^b	Maverick	3	0.10-0.11	7	< 0.05 (2)	02008.08-ND04
N. Dakota, Minot ^b	Maverick	3	0.10-0.11	7	< 0.05 (2)	02008.08-ND05
New York	Cabernet	3	0.10-0.11	8	0.13 (0.147, 0.109)	02008.08-NY04
S. Dakota, Aurora	Eclipse Black	3	0.10-0.11	8	0.09 (0.103, 0.08)	02008.08-SD03
S. Dakota, Aurora	Maverick Pinto	3	0.10-0.11	7	0.170 (2)	02008.08-SD04

^aTotal propiconazole - (TA+TAA+1,2,4 T); values <LOQ were considered at ½ LOQ; 1,2,4 T were all <LOQ.

^b same location, period and variety, only one selected;

Barley

A total of nine trials were conducted with barley in USA using 2 applications of propiconazole (Report: T004408-08;). Residues of propiconazole and total propiconazole are shown in Table 6.

Table 6 Residues from trials conducted with propiconazole in barely grain in USA in 2009/2010 using 2 application of SE formulation

Region	Barely variety	Application		DAT	Residues (mg/kg)		Trial
		kg a.i./ha	BBCH growth state		Propiconazole	Total propiconazole	
Minnesota	Spring Rawson	0.15	41 and 71	44	0.011, < 0.01	0.188, 0.57	C09-9063
N. Dakota, Northwood	Pinnacle	0.15	39 and 71	33	< 0.01	< 0.05	C13-9062
				40	< 0.01	0.0555	
				47	< 0.01 (2)	0.053, 0.066	
				54	< 0.01	0.0585	
N. Dakota, Carrington	Pinnacle	0.15	37 and 69	48	< 0.01 (2)	0.060, 0.072	C13-9065
S. Dakota,	Lacey	0.15	43-49 and 71-73	33	< 0.01 (2)	0.13 (2)	C16-9064
Nebraska	Spring, Baroness	0.15	43 and 59	29	< 0.01 (2)	< 0.05 (2)	C33-9066
Colorado	Moravian 37	0.15	55 and 71	25	0.175, 0.21	0.425, 0.516	W12-9067
Virginia	Nomini	0.15	55 and 71	30	0.144, 0.137	0.589, 0.765	E07-9061
Idaho	Ida-Gold II	0.15	55-59 and 85	16	0.020, 0.019	0.273, 0.237	W15-9070
California	UC937	0.15	69 and 71	49	1.3, 0.933	2.22, 2.02	W32-9068

Oats

A total of twelve trials were conducted with oats in USA using 2 applications of propiconazole (Report: T005494-08;). Residues of propiconazole and total propiconazole are shown in Table 7.

Table 7 Residues from trials conducted with propiconazole in oat grain in USA in 2009-2010 using 2 application of SE formulation

	Oat variety	Application		DAT (days)	Residues (mg/kg)		Trial
		kg a.i./ha	BBCH growth stage		Propiconazole	Total propiconazole	
Minnesota	Morton	0.15	41 and 71	48	0.035, 0.044	0.117, 0.31	C09-9084
N. Dakota, Ayr	Morton	0.15	52 and 70	44	0.054, 0.067	0.269, 0.173	C12-9086
N. Dakota, Gardner	Morton	0.15	72 and 70	11	0.117	0.337	C12-9087
				18	0.252	0.688	
				25	0.311, 0.355	0.862, 0.968	
				32	0.225	0.642	
N. Dakota, Northwood	Jerry	0.15	39 and 71	33	0.017, 0.022	0.086, 0.089	C13-9083 2009
N. Dakota, Northwood	Jerry	0.15	43 and 71	28	0.014, 0.017	0.063, 0.067	C13-9088 2010
N. Dakota, Carrington	Jerry	0.15	37 and 69	39	0.033 (2)	0.163, 0.143	C13-9091
S. Dakota	Stallion	0.15	43 and 71	36	0.024, 0.023	0.155, 0.208	C16-9090
Iowa	Reeves	0.15	71	32	0.086, 0.078	0.774, 0.91	C30-9085
Pensilvania	Armor	0.15	59 and 71-75	28	0.35, 0.386	1.32, 1.61	E04-9081
Colorado	Jerry	0.15	73 and 71	28	0.048, 0.05	0.184, 0.206	W12-9092
S. Caroline	Horizon 270	0.15	43 and 55	44	< 0.01 (2)	0.26 (0.257, 0.265)	E11-9082
Texas	BOB	0.15	60/71	35	0.252, 0.269	1.59, 1.52	W07-9089

Wheat

A total of twelve trials were conducted with wheat in USA using 2 applications of propiconazole (Report: T004407-08;). Residues of propiconazole and total propiconazole are shown in Table 8.

Table 8. Residues from trials conducted with propiconazole in wheat grain in USA in 2009-2010 using 2 application of SE formulation

Region	Wheat variety	Application		DAT (days)	Residues, mg/kg		Trial
		kg a.i./ha	Growth Stage		Propiconazole	"Total propiconazole"	
N. Dakota, Gardner 2009	Durum Wheat (Maier)	0.15	52 and 55	35	< 0.01 (2)	0.054 (0.052, 0.060)	C12-9035
N. Dakota, Carrington	Spring Glenn	0.15	37 and 69	55	< 0.01 (2)	< 0.05 (2)	C13-9038
S. Dakota, Lake Andes	Spring Oxen	0.15	55 – 59 and 71	34	< 0.01 (2)	0.076, 0.075	C16-9037
Iowa	Hard wheat (Briggs)	0.15	71	25	< 0.01	0.055	C30-9034
				32	< 0.01	< 0.05	
				39	< 0.01 (2)	0.058, 0.053	
				46	< 0.01	0.0684	
Texas, Raymondville	Caudillo	0.15	43 and 71	38	< 0.01 (2)	< 0.05, 0.06	W08-9036
Colorado	Winter Jagalene	0.15	53 and 71	29	< 0.01 (2)	0.054, 0.057	W12-9044
N. Dakota, Northwood	Winter wheat (Jerry)	0.15	45 and 71	43	< 0.01 (2)	< 0.05 (2)	C13-9033
N. Dakota, Carrington	Winter wheat (Jerry)	0.15	45 and 71	50	< 0.01 (2)	< 0.05 (2)	C13-9040
S. Dakota, Lake Andes	Winter wheat (Wendy)	0.15	43 and 67 - 69	64	< 0.01 (2)	< 0.05, 0.054	C16-9039
Virginia	Pioneer 26R15	0.15	65 and 75	24	< 0.01 (2)	< 0.05, 0.078	E07-9031

Region	Wheat variety	Application		DAT (days)	Residues, mg/kg		Trial
		kg a.i./ha	Growth Stage		Propiconazole	"Total propiconazole	
Texas, Groom	Winter Cutter	0.15	61 and 71	10	0.13	0.152	E13- 9041
				17	0.0762	0.125	
				24	0.084, 0.072	0.165, 0.125	
				30	0.054	0.155	
Lousiania	Winter Terral LA841	0.15	57-59 and 73	30	< 0.01 (2)	0.063, 0.169	
Idaho	Hard wheat (Klassic)	0.15	55 – 59 and 71	35	< 0.01 (2)	< 0.05 (2)	W15- 9046
Texas, Levelland	TAM 112	0.15	51 and 73	35	0.011, < 0.01	0.125, < 0.05	W39- 9042
Texas, Littlefield	Weathermaster	0.15	51 and 73	35	0.020, 0.023	0.091, 0.099	W39- 9043

Mint

Five supervised residue trials with mint were conducted in USA in 2005 (Report: IR-4 PR No. 09419). Samples were analyzed for total propiconazole as 2,4-dichlorobenzoic methyl ester, using GC/ECD. The results are summarized in Table 9.

Table 9 Residues resulting from propiconazole application to mint in USA

Region	Peppermint variety	Application			Total propiconazole, mg/kg	Trial
		No.	kg ai/ha	DAT		
Idaho	Black Mitchem	3	0.11	7	2.12, 1.98	09419.05-ID16
Wisconsin, Portage ^a	Black Mitchem	3	0.10	6	4.55, 3.43	09419.05-WI02
Wisconsin, Portage ^a	Black Mitchem	3	0.10	6	5.45, 5.44	09419.05-WI03
Washington, Prosser	Black Mitchem	3	0.11	6	4.45, 3.68	09419.05-WA03
Washington, Moxee	Black Mitchem	3	0.11	7	0.151, 0.14	09419.05-WA*04

^a same location and period, only one was considered

Lima bean foliage

Six supervised residue trials were conducted in USA in 2005 (Report: IR-4 PR No. 09437). The results from analysis of foliage samples are summarized in Table 10.

Table 10 Residues in foliage resulting from propiconazole application to lima beans in USA

Region	Lima Bean variety	Application			Total propiconazole, mg/kg	Trial
		No.	kg ai/ha	DAT, days		
California	Green Baby 12-98	5	0.643	8	4.89, 1.83	09437.05-CA163
Idaho	M15	3	0.382	8	6.67, 5.91	09437.05-ID19
Maryland*	Burpee Improved	3	0.373	7	3.69, 3.65	09437.05-MD22
Maryland*	Burpee Improved	3	0.377	6	3.90, 2.62	09437.05-MD23
New Jersey	Baby Lima	3	0.387	7	8.3, 7.4	09437.05-NJ38
Wisconsin	909 Cyrus	3	0.384	6	12.8, 12.0	09437.05-WI28

* same location, same period, only one was chosen

Animal feeds*Cereals*

Table 11 Residues from trials conducted with propiconazole in barley feed in USA in 2009/2010 using 2 application at 0.15 (Report: T004408-08;)

Region	Barley variety	Application		DAT (days)	Portion	Residues (mg/kg)		Trial
		kg a.i./ha	Growth Stage BBCH			Propiconazole	Total propiconazole	
Minnesota	Spring Rawson	0.15	30-31 + 32	7	Hay	<u>0.81</u> (0.904, 0.724)	<u>2.6</u> (2.28, 2.83)	C09-9063
				14	Hay	0.157, 0.193	2.21, 2.95	
		0.15	41 + 71	44	Straw	0.18 (0.194, 0.168)	<u>2.0</u> (1.99, 1.93)	
N. Dakota, Northwood	Pinnacle	0.15	22 + 52	0	Hay	5.98	7.75	C13-9062
				3	Hay	2.02	3.77	
				7	Hay	<u>0.44</u>	<u>2.1</u>	
				14	Hay	0.375, 0.299	2.54, 2.83	
				21	Hay	0.127	1.66	
		0.15	39 + 71	33	Straw	0.12	1.3	
				40	Straw	0.0796	0.992	
				47	Straw	0.124, 0.116	0.944, 0.891	
				54	Straw	0.14	<u>1.4</u>	
N. Dakota, Carrington	Pinnacle	0.15	16 + 59	7	Hay	<u>0.73</u> (0.848, 0.616)	<u>2.7</u> (2.85, 2.64)	C13-9065
				14	Hay	0.476, 0.49	2.84, 2.85	
		0.15	37 + 69	48	Straw	0.24 (0.25, 0.24)	<u>1.6</u> (1.8, 1.41)	
S. Dakota	Lacey	0.15	21-31 + 43-49	7	Hay	<u>0.19</u> (2.12, 1.74)	<u>9.9</u> (9.81, 10.2)	C16-9064
				15	Hay	0.546, 0.588	5.92, 5.29	
		0.15	43-49 + 71-73	33	Straw	<u>0.72</u> (0.716, 0.722)	<u>2.9</u> (3.05, 2.74)	
Nebraska	Spring Baronesse	0.15	30 + 26	8	Hay	<u>4.2</u> (4.46, 3.92)	<u>11.4</u> (11, 11.7)	C33-9066
				15	Hay	1.09, 1.05	2.94, 2.59	
		0.15	43 + 59	29	Straw	<u>0.82</u> (0.087, 0.077)	<u>1.5</u> (1.62, 1.31)	
Colorado	Moravian 37	0.15	30 + 43	7	Hay	<u>2.2</u> (2.42, 2.11)	<u>4.0</u> (4.35, 3.59)	W12-9067
				14	Hay	0, 1.01	0, 3.18	
		0.15	55 + 71	25	Straw	5.0 (5.2, 4.8)	<u>10</u> (10.2, 8.91)	
Virginia	Nomini	0.15	30 + 31	7	Hay	<u>1.7</u> (1.9, 1.49)	<u>4.3</u> (5.02, 3.52)	E07-9061
				14	Hay	0.419, 0.516	1.73, 1.82	
		0.15	55 + 71	30	Straw	3.0 (2.97, 3.06)	<u>9.9</u> (9.8, 10.3)	
Idaho	Ida-Gold II	0.15	30 + 55	7	Hay	<u>0.78</u> (0.691, 0.879)	<u>6.2</u> (5.63, 6.77)	W15-9070
				14	Hay	0.183, 0.189	4.32, 4.26	
		0.15	55-59 + 85	16	Straw	0.29 (0.289, 0.285)	<u>3.6</u> (3.76, 3.42)	
California	UC937	0.15	30 + 32	7	Hay	<u>3.0</u> (2.88, 3.19)	<u>12</u> (10.7, 13.1)	W32-9068
				14	Hay	1.34, 2.1	5.13, 6.61	
		0.15	69+ 71	49	Straw	3.8 (3.89, 3.77)	<u>5.8</u> (5.79, 5.7)	

Table 12 Residues from trials conducted with propiconazole in oat feed in USA in 2009-2010 using 2 application of SE formulation (Report: T005494-08;)

Region	Oat variety	Application		DAT (days)	Crop Part	Residues (mg/kg)		Trial
		kg a.i./ha	BBCH			Propiconazole	Total propiconazole	
Minnesota	Morton	0.15	30 + 32	7	Forage	0.85 (0.828, 0.858)	1.5 (1.47, 1.55)	C09-9084
				14	Forage	0.145, 0.151	0.899, 0.855	
				7	Hay	1.1 (1.1, 1.05)	3.2 (3.14, 3.42)	
				14	Hay	0.436, 0.458	1.79, 1.99	
	Morton	0.15	41 + 71	48	Straw	0.05 (0.047, 0.061)	1.2 (1.13, 1.32)	
N. Dakota, Ayr	Morton	0.15	31 + 49	7	Forage	1.1 (0.976, 1.23)	1.9 (1.77, 2.12)	C12-9086
				14	Forage	0.424, 0.464	1.38, 1.18	
				7	Hay	4.2 (3.91, 4.4)	7.7 (7.14, 8.33)	
				14	Hay	1.7 (2)	4.88, 3.0	
	Morton	0.15	52 + 70	44	Straw	0.25 (0.357, 0.159)	2.3 (3.2, 1.56)	
N. Dakota, Gardner	Morton	0.15	31 + 49	0	Forage	2.76	2.95	C12-9087
				3	Forage	2.42	2.82	
				7	Forage	1.3	2.0	
				14	Forage	0.428, 0.48	1.25, 1.41	
				21	Forage	0.7	1.82	
				5	Hay	7.38,	12.2	
				7	Hay	4.5	8.5	
				14	Hay	1.81, 2.69	4.07, 5.69	
				21	Hay	1.56	4.52	
	Morton	0.15	72 + 70	11	Straw	0.948	1.79	
				18	Straw	1.32	3.43	
				25	Straw	2.2 (2.4, 1.91)	6.0 (6.07, 5.86)	
				32	Straw	1.16	3.78	
N. Dakota, Northwood	Jerry	0.15	22 + 55	7	Forage	0.20 (0.203, 0.196)	0.78 (0.811, 0.746)	C13-9083
				14	Forage	0.0699, 0.0802	0.551, 0.538	
				7	Hay	0.35 (0.376, 0.332)	1.1 (0.957, 1.19)	
				14	Hay	0.171, 0.194	1.41, 1.19	
	Jerry	0.15	39 + 71	33	Straw	0.05 (0.051, 0.053)	1.2 (1.16, 1.09)	
N. Dakota, Northwood	Jerry	0.15	30 + 71	7	Forage	0.60 (0.626, 0.566)	1.4 (1.46, 1.34)	C13-9088
				14	Forage	0.359, 0.291	1.49, 1.12	
				7	Hay	1.4 (1.58, 1.19)	2.4 (2.52, 2.17)	
				14	Hay	0.499, 0.314	1.84, 1.35	
	Jerry	0.15	43 and 71	28	Straw	0.05 (0.049, 0.047)	0.30 (0.291, 0.312)	
N. Dakota, Carrington	Jerry	0.15	16 + 55	7	Forage	0.65 (0.592, 0.716)	1.5 (1.36, 1.6)	C13-9091
				14	Forage	0.237, 0.286	0.905, 1.05	
				7	Hay	1.4 (1.43, 1.33)	2.7 (2.46, 2.88)	
				14	Hay	0.864, 1.11	2.64, 3.47	
	Jerry	0.15	37 + 69	39	Straw	0.21 (0.039, 0.374)	2.9 (3.16, 2.66)	
S. Dakota	Stallion	0.15	23 + 43	6	Forage	0.82 (0.712, 0.924)	1.4 (1.31, 1.55)	C16-9090
				16	Forage	0.182, 0.171	0.588, 0.666	
				6	Hay	3.6 (3.49, 3.81)	6.2 (5.56, 6.92)	
				16	Hay	0.439, 0.64	2.59, 2.78	

Propiconazole

Region	Oat variety	Application		DAT (days)	Crop Part	Residues (mg/kg)		Trial
		kg a.i./ha	BBCH			Propiconazole	Total propiconazole	
	Stallion	0.15	43 + 71	36	Straw	0.04 (2)	<u>1.4</u> (1.43, 1.33)	
Iowa	Reeves	0.15	30	7	Forage	<u>0.16</u> (0.135, 0.176)	<u>0.50</u> (0.568, 0.431)	C30-9085
				14	Forage	0.0491, 0.0316	0.541, 0.303	
				7	Hay	<u>0.18</u> (0.258, 0.11)	<u>0.80</u> (0.967, 0.629)	
				14	Hay	0.0932, 0.069	0.96, 0.787	
	Reeves	0.15	71	32	Straw	0.02 (0.017, 0.021)	<u>0.94</u> (0.663, 1.21)	
Pensilvania	Armor	0.15	30 + 32	7	Forage	<u>1.2</u> (1.24, 1.09)	<u>1.8</u> (1.75, 1.82)	E04-9081
				14	Forage	0.42 (2)	1.2, 1.37	
				7	Hay	<u>2.0</u> (2.02, 2.07)	<u>4.8</u> (4.47, 5.17)	
				14	Hay	1.3, 1.14	3.85, 4.57	
	Armor	0.15	59 + 71-75	29	Straw	0.80 (0.872, 0.728)	<u>5.1</u> (4.12, 6.07)	
Colorado	Jerry	0.15	30 + 43	7	Forage	<u>0.15</u> (0.161, 0.146)	<u>0.50</u> (0.522, 0.476)	W12-9092
				14	Forage	0.0496, 0.0449	0.381, 0.397	
				7	Hay	<u>0.24</u> (0.243, 0.241)	<u>0.90</u> (0.859, 0.95)	
				14	Hay	0.062, 0.074	0.559, 0.624	
	Jerry	0.15	73 and 71	28	Straw	<u>0.37</u> (0.33, 0.411)	<u>2.0</u> (1.86, 2.23)	
S. Caroline	Horizon 270	0.15	30 and 32	7	Forage	0.82 (0.86, 0.78)	<u>2.3</u> (2.46, 2.16)	E11-9082
				14	Forage	0.308, 0.335	1.26, 1.51	
				7	Hay	<u>1.2</u> (1.34, 1.2)	<u>2.2</u> (3.3, 3.16)	
				14	Hay	0.645, 0.595	2.31, 1.66	
	Horizon 270	0.15	43 and 55	44	Straw	<u>0.08</u> (0.082, 0.096)	<u>2.8</u> (2.55, 2.89)	
Texas	BOB	0.15	30/33	7	Forage	<u>1.0</u> (0.87, 1.15)	<u>1.6</u> (1.42, 1.94)	W07-9089
				14	Forage	0.341, 0.408	1.0, 0.949	
				7	Hay	<u>2.4</u> (2.05, 2.83)	<u>4.8</u> (3.81, 5.7)	
				14	Hay	1.06, 0.775	2.97, 2.32	
	BOB	0.15	30/33	35	Straw	<u>0.27</u>	<u>1.9</u>	

Table 13 Residues from trials conducted with propiconazole in wheat feed in USA in 2009-2010 using 2 application of SE formulation (Report: T004407-08;)

Region	Wheat variety	Application		DAT (days)	Crop Part	Residues, mg/kg		Trial
		kg a.i./ha	Growth Stage			Propiconazole	"Total propiconazole	
N. Dakota, Gardner 2009	Durum Wheat (Maier)	0.15	52 and 55	35	Straw	<u>0.52</u> (0.632, 0.419)	<u>2.7</u> (3.55, 1.92)	C12-9035
N. Dakota, Carrington	Spring Glenn	0.15	16 and 55	7	Forage	<u>1.3</u> (1.36, 1.2)	<u>2.8</u> (2.83, 2.68)	C13-9038
				14	Forage	0.778, 0.552	2.54, 2.05	
				7	Hay	<u>2.2</u> (2.26, 2.21)	<u>12</u> (6.76, 16.9)	
				14	Hay	1.5, 1.51	5.67, 14.8	
	Spring , Glenn	0.15	37 and 69	55	Straw	0.83 (0.748, 0.912)	<u>5.0</u> (5.32, 4.83)	
S. Dakota, Lake Andes	Spring , Oxen	0.15	23 and 55 - 59	7	Forage	<u>0.92</u> (0.855, 0.975)	<u>3.0</u> (2.89, 3.13)	C16-9037
				17	Forage	0.208, 0.245	1.63, 1.61	

Region	Wheat variety	Application		DAT (days)	Crop Part	Residues, mg/kg		Trial
		kg a.i./ha	Growth Stage			Propiconazole	"Total propiconazole	
				7	Hay	<u>3.0</u> (2.96, 2.96)	<u>10</u> (12.7, 7.29)	
				17	Hay	0.728, 0.546	4.9, 5.02	
				34	Straw	0.37 (0.426, 0.319)	<u>0.19</u> (2.33, 1.56)	
	Spring , Oxen	0.15	55 – 59 and 71					
Iowa	Hard wheat (Briggs)	0.15	30 and 14 days after	0	Forage	2.56	3.6	C30-9034
				3	Forage	0.66	1.71	
				7	Forage	<u>0.27</u>	<u>1.2</u>	
				14	Forage	0.102, 0.061	1.42, 1.04	
				21	Forage	0.0524	1.3	
				0	Hay	4.9	8.92	
				3	Hay	1.15	4.1	
				7	Hay	<u>0.44</u>	<u>3.2</u>	
				14	Hay	0.149, 0.163	2.78, 2.58	
				21	Hay	0.0908	2.4	
	Hard wheat (Briggs)	0.15	71	25	Straw	0.0523	0.927	
				32	Straw	0.088	1.26	
				39	Straw	0.06 (2)	0.992, 1.83	
				46	Straw	<u>0.05</u>	<u>2.0</u>	
Texas, Raymondville	Caudillo	0.15	29 – 30 and 31	6	Forage	<u>4.8</u> (4.68, 4.9)	<u>8.4</u> (7.93, 9)	W08-9036
				14	Forage	2.09, 2.16	5.07, 5.85	
				6	Hay	<u>9.6</u> (9.82, 9.32)	<u>22</u> (22.5, 20.6)	
				14	Hay	5.34, 5.48	16.1, 18.7	
	Caudillo	0.15	43 and 71	38	Straw	0.46 (0.412, 0.498)	<u>3.6</u> (3.61, 3.5)	
Colorado	Winter, Jagalene	0.15	41 and 53	7	Forage	<u>1.0</u> (1.14, 0.992)	<u>2.2</u> (2.5, 1.82)	W12-9044
				14	Forage	0.772, 0.872	1.88, 2.29	
				7	Hay	<u>1.9</u> (1.89, 1.93)	<u>4.4</u> (4.67, 4.08)	
				14	Hay	1.08, 1.32	3.71, 3.59	
	Winter, Jagalene	0.15	53 and 71	29	Straw	0.28 (0.329, 0.232)	<u>1.0</u> (1.16, 0.847)	
N. Dakota, Gardner	Durum Wheat (Maier)	0.15	33 and 41	7	Forage	<u>1.4</u> (1.36, 1.34)	<u>1.8</u> (1.74, 2.01)	C12-9047
				13	Forage	0.452, 0.624	1.28, 1.52	
				7	Hay	<u>3.3</u> (3.08, 3.47)	<u>9.0</u> (8.12, 10.4)	
				13	Hay	1.7, 1.26	4.71, 4.29	
N. Dakota, Northwood	Winter, Jerry	0.15	30 and 37	8	Forage	<u>0.46</u> (0.486, 0.432)	<u>1.4</u> (1.59, 1.31)	C13-9033
				15	Forage	0.154, 0.144	0.839, 0.717	
				8	Hay	<u>1.1</u> (1.08, 1.13)	<u>3.4</u> (3.39, 3.36)	
				15	Hay	0.364, 0.324	1.82, 1.64	
	Winter, Jerry	0.15	45 and 71	43	Straw	0.04 (0.043, 0.049)	<u>0.60</u> (0.624, 0.57)	
N. Dakota, Carrington	Winter, Jerry	0.15	30 and 37	6	Forage	<u>1.7</u> (1.76, 1.71)	<u>4.8</u> (4.76, 4.77)	C13-9040
				15	Forage	0.308, 0.438	1.01, 1.12	
				6	Hay	<u>2.0</u> (2.04, 1.9)	<u>5.3</u> (6.12, 4.5)	
				15	Hay	0.716, 0.662	2.6 (2)	
	Winter, Jerry	0.15	45 and 71	50	Straw	0.15 (0.167, 0.142)	<u>1.6</u> (1.62, 1.57)	
S. Dakota, Lake Andes	Winter, Wendy	0.15	23 and 43	8	Forage	<u>1.6</u> (1.54, 1.58)	<u>3.4</u> (3.35, 3.39)	C16-9039
				14	Forage	0.89, 0.94	2.19, 1.88	
				8	Hay	<u>4.2</u> (4.4, 4.06)	<u>10</u> (11.5, 9.58)	

Propiconazole

	Wheat variety	Application		DAT (days)	Crop Part	Residues, mg/kg		Trial
Region		kg a.i./ha	Growth Stage			Propiconazole	“Total propiconazole	
Virginia				14	Hay	1.98, 2.06	6.55, 6.74	E07- 9031
	Winter, Wendy	0.15	43 and 67 - 69	64	Straw	0.14 (0.145, 0.132)	<u>1.6</u> (1.66, 1.47)	
	Pioneer 26R15	0.15	30	7	Forage	<u>1.4</u> (1.39, 1.44)	<u>2.1</u> (2.01, 2.21)	
				14	Forage	0.62, 0.57	1.36, 1.33	
				7	Hay	<u>2.0</u> (1.91, 2.11)	<u>4.0</u> (3.89, 4.04)	
Pioneer 26R15	0.15	65 and 75	24	Straw	3.5 (3.41, 3.61)	<u>12</u> (11.6, 13.2)		
Texas, Groom	Winter, Cutter	0.15	30 and 31	0	Forage	9.14	12.4	E13- 9041
				3	Forage	5.3	7.23	
				7	Forage	<u>0.77</u>	<u>1.6</u>	
				16	Forage	0.286, 0.345	1.08, 1.22	
				21	Forage	0.205	1.05	
				0	Hay	23	26.3	
				3	Hay	5.66	7.18	
				7	Hay	0.57	2.0	
				16	Hay	<u>0.75</u> (0.846, 0.662)	<u>2.7</u> (2.95, 2.39)	
	21	Hay	0.422	1.77				
	Winter, Cutter	0.15	61 and 71	10	Straw	4.9	<u>6.6</u>	
				17	Straw	2.44	3.81	
				24	Straw	3.78, 2.64	6.09, 4.52	
				30	Straw	0.79	2.16	
Louisiana	Winter, Terral LA841	0.15	30 and 41	7	Forage	<u>0.66</u> (0.642, 0.686)	<u>1.3</u> (1.18, 1.43)	E17- 9032
				13	Forage	0.074, 0.124	0.828, 1.04	
				7	Hay	<u>1.6</u> (1.28, 1.82)	<u>4.3</u> (4.35, 4.19)	
				13	Hay	0.216, 0.22	1.67, 1.57	
	Winter Terral LA841	0.15	57-59 and 73	14	Forage	0.696, 0.868	3.28, 2.85	
				14	Hay	1.27, 1.42	3.59, 4.6	
Idaho	Hard wheat, Klassic	0.15	30 and 33	7	Forage	<u>0.55</u> (0.526, 0.574)	<u>1.2</u> (1.31, 1.23)	W15- 9046
				13	Forage	0.262, 0.275	0.759, 1.38	
				7	Hay	<u>1.3</u> (1.39, 1.24)	<u>4.0</u> (4.03, 4.14)	
				13	Hay	0.788, 0.78	3.49, 3.64	
	Hard wheat, Klassic	0.15	55 – 59 and 71	35	Straw	0.05 (0.045, 0.051)	<u>1.5</u> (1.26, 1.67)	
Texas, Levelland	TAM 112	0.15	30 and 31	7	Forage	<u>2.2</u> (2.1, 2.43)	<u>3.8</u> (3.73, 3.8)	W39- 9042
				14	Forage	0.975, 0.98	2.63, 2.16	
				7	Hay	<u>2.8</u> (3.03, 2.5)	<u>6.7</u> (7.06, 6.31)	
				14	Hay	1.1, 1.03	4.24, 3.65	
	TAM 112	0.15	51 and 73	35	Straw	2.4 (2.73, 2.01)	<u>7.6</u> (8.24, 6.91)	
Texas, Littlefield	Weathermaster	0.15	30 and 31	7	Forage	<u>2.5</u> (2.69, 2.34)	<u>4.0</u> (3.96, 3.91)	W39- 9043
				16	Forage	1.15, 0.87	1.69, 0.97	
				7	Hay	<u>5.4</u> (5.82, 4.93)	<u>7.9</u> (8.62, 7.26)	
				16	Hay	1.85, 1.44	3.82, 4.03	
	Weathermaster	0.15	51 and 73	35	Straw	4.0 (4.36, 3.72)	<u>13</u> (13.3, 11.9)	

Processing studies

Pineapple sample from trial HI06 (Table 2), containing 0.67 mg/kg total propiconazole was peeled, with residues in the pulp at < 0.05 mg/kg and in the skin at 0.35 mg/kg. Residues in pineapple juice made from the pulp were also < 0.05 mg/kg.

APPRAISAL

The triazole fungicide propiconazole was evaluated in the periodic review program by the 2007 JMPR, when an ADI of 0–0.07 mg/kg bw and a ARfD of 0.3 mg/kg bw were established, and by the 2013 JMPR for residue aspects. Propiconazole was listed by the Forty-fifth Session of the CCPR (2013) for the review of additional uses. Residue trial data on bean (dry), bean (lima), and bean (snap), mint, pineapple following post-harvest application, barley, oat and wheat were submitted to current Meeting.

Residue definition for plant and animal commodities is *propiconazole* for compliance with the MRL and *propiconazole plus all metabolites convertible to 2,4-dichlorobenzoic acid, expressed as propiconazole* for the estimation of the dietary intakes.

Methods of analysis

Methods of analysis used in the trials submitted to this Meeting were evaluated by the 2007 JMPR and were considered fit for purpose for the estimations. Methods that used LC-MS/MS analyses propiconazole, and the results are used for the estimation of maximum residue level. The common moiety method measure all residues convertible to 2,4-dichlorobenzoic (2,4-DCBA), reported as total propiconazole, and the results were used for the estimation of STMR and HR. In the residue trials, samples were stored within the period that guarantee the integrity of the residues as shown in the storage stability studies.

Results of supervised residue trials in crops

No estimations were made for lima bean, snap bean, dry beans and mint, as only the total propiconazole residue was reported in pre-harvest treatment trials. Data for these crops will not be discussed further.

Pineapple

The GAP of the USA for propiconazole in pineapple is for one post-harvest dipping treatment in a 0.015 kg ai/hL solution. Three trials were conducted using this rate, giving total residue of 1.1, 1.8 and 3.6 mg/kg of total propiconazole (highest of 4.1 mg/kg in an individual sample). As it is unlikely that propiconazole metabolites are formed between treatment and sample preparation for analysis, the results comply with both the residue definition for MRL and dietary risk assessment.

The Meeting agreed that the number of trials was not sufficient to estimate a maximum residue level for propiconazole in pineapple following post-harvest treatment, and did not change its previous recommendation of 0.02* mg/kg.

Cereals

In the USA, GAP for propiconazole in barley, oats and wheat, is for up to 2 applications at 0.125 kg ai/ha, the second one being at least 14 days following an early season application and should not be performed after full head emergence, which corresponds to BBCH 59.

In one trial conducted in USA in barley, matching GAP, residues of propiconazole were < 0.01 mg/kg and of total propiconazole < 0.05 mg/kg. In eight other trials the second application was made after full head emergence (BBCH 71 to 85).

In one trial conducted in USA in oats according to GAP, residues of propiconazole were < 0.01 mg/kg and of total propiconazole were 0.26 mg/kg. In eleven other trials the second application was made after full head emergence (BBCH 69 to 71).

In one trial conducted in USA in wheat according to GAP residues of propiconazole were < 0.01 mg/kg and of total propiconazole 0.06 mg/kg. In fourteen other trials, the second application was made after full head emergence (BBCH 69 to 75).

The Meeting agreed that there were not enough trials matching US GAP to estimate a maximum residue level for propiconazole in barley, oats and wheat.

The Meeting did not change its previous recommendation of 0.02 mg/kg for propiconazole in wheat, rye and triticale and of 0.2 mg/kg for propiconazole in barley.

Animal feeds

Cereals

In the USA, the GAP for propiconazole in barley, oats and wheat, is for up to 2 applications at 0.125 kg ai/ha, the second at least 14 days following an early season application and should not be performed after full head emergence, corresponding to BBCH 59. No PHI is specified for straw, and the PHI is 7 days for forage and hay.

Forage

Eleven trials were conducted in oat forage in the USA matching US GAP, with total propiconazole residues of 0.50 (2), 0.78, 1.4, 1.5 (2), 1.7, 1.8, 1.9, 2.0 and 2.4 mg/kg (2.5 mg/kg highest of individual samples).

The Meeting estimated a medium and highest residue of 1.5 and 2.5 mg/kg, respectively, for propiconazole in oat forage.

Fifteen trials were conducted in wheat forage, gave total propiconazole residues of 1.2 (2), 1.3, 1.4, 1.6, 1.9, 2.1, 2.2, 2.8, 3.0, 3.4, 3.8, 3.9, 4.8 and 9 mg/kg.

The Meeting estimates a medium and highest residue of 2.2 and 9 mg/kg, respectively, for propiconazole in wheat forage.

Hay

Nine trials were conducted in barley hay with propiconazole residues of 0.19, 0.44, 0.73, 0.78, 0.81, 1.7, 2.2, 3.0 and 4.2 mg/kg. Total propiconazole residues were: 2.1, 2.6, 2.7, 4.0, 4.3, 6.2, 10, 11 and 12 mg/kg (highest 13 mg/kg).

The Meeting estimated a maximum residue level of 8 mg/kg for propiconazole in barley straw and fodder, dry. The Meeting also estimated medium and highest residue of 4.3 and 13 mg/kg, respectively, for propiconazole in barley hay.

In the 11 trials conducted with oat hay, according to US GAP, propiconazole residues were: 0.18, 0.24, 0.35, 1.1, 1.2, 1.4, 2.0, 2.4, 3.6, 4.2 and 4.5 mg/kg. Total propiconazole were: 0.80, 0.90, 1.1, 2.7, 3.2, 3.3, 4.8 (2), 6.2, 7.7 and 8.5 mg/kg.

The Meeting estimated a maximum residue level of 8 mg/kg for propiconazole in oat straw and fodder, dry. The Meeting also estimated a medium and highest residue of 3.3 and 8.5 mg/kg, respectively, for propiconazole in oat hay.

In 15 trials conducted in wheat hay, propiconazole residues were: 0.44, 0.75, 1.1, 1.3, 1.6, 1.9, 2.0 (2), 2.2, 2.8, 3.0, 3.3, 4.2, 5.4 and 9.6 mg/kg. Total propiconazole were: 2.0, 3.2, 3.4, 4.0, 4.1, 4.3, 4.4, 5.3, 6.7, 7.9, 9.3, 10 (2), 12 and 21 mg/kg (highest of 22 mg/kg).

Taking into account the trial conditions, the Meeting concluded that the residues measured in hay are applicable for straw and fodder, and recommended a maximum residue level of 15 mg/kg for

propiconazole in wheat straw and fodder, dry, and extend this estimation for rye and straw and fodder, dry. The Meeting also estimates a medium and highest residue of 5.3 and 22 mg/kg, respectively, for propiconazole in wheat hay.

The Meeting withdrew its previous maximum residue level recommendation for barley, rye, triticale and wheat straw of 2 mg/kg.

Residues in animal commodities

One cow and one poultry study were evaluated by the 2007 JMPR, and only the relevant information from these studies are summarized in this appraisal.

Lactating dairy cows

Dairy cows were dosed once daily either in the feed with propiconazole at 15 ppm, 75 ppm and 150 ppm for 14–28 consecutive days. No parent propiconazole was detected in any milk (< 0.01 mg/kg), muscle and kidney samples (< 0.05 mg/kg) at all feeding levels. The maximum level in liver was 0.14 mg/kg at the 15 ppm feeding level and 0.34 mg/kg in the 75 ppm feeding level; in fat it was < 0.05 mg/kg at the 15 ppm and 75 ppm feeding levels.

No total propiconazole (< 0.01 mg eq./kg) was found in milk at 15 ppm; at 75 ppm, the average was 0.044 mg eq./kg (maximum of 0.08 mg eq./kg). In muscle, no total propiconazole (< 0.05 mg/kg) was detected at 15 ppm and the maximum level was 0.11 mg/kg at the 75 ppm (average 0.08 mg/kg). In liver, residues were 0.81 mg/kg at the 15 ppm (average 0.63 mg/kg) and 4.3 mg/kg in the 75 ppm (average 3.7 mg/kg). In kidney, it was 0.63 mg/kg at the 15 ppm (average 0.60 mg/kg) and 4.7 mg/kg in the 75 ppm (average 3.8 mg/kg). In fat it was < 0.05 mg/kg at the 15 ppm and 0.23 mg/kg at the 75 ppm feeding level (average 0.15 mg/kg).

Laying hens

Hens were fed propiconazole at 7.5, 37.5 and 75 ppm in the feed. No propiconazole residues (< 0.05 mg/kg) were found in the eggs or the tissue sample analysed, regardless of feeding level.

At 7.5 ppm, no total propiconazole was detected in eggs, muscle, fat (< 0.05 mg/kg) and liver (< 0.1 mg/kg). At 37.5 ppm, residues can reach 0.18 mg/kg in eggs, 0.16 mg/kg in liver and 0.07 mg/kg in fat, but no residues were detected in muscle.

Farm animal dietary burden

The Meeting estimated the dietary burden of propiconazole (as total propiconazole) in farm animals on the basis of the OECD Animal Feed data published in the 2009 FAO Manual, the STMR, STMR-Ps or highest residue levels estimated at the present and by the 2007 JMPR. Dietary burden calculations are provided in Annex 6.

Commodity	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	3.9	2.1	15.9	4.3	36 ^a	12 ^c	10.1	1.62
Dairy cattle	15.8	7.2	13.5	4.0	29 ^b	12 ^d	10.5	5.56
Poultry – broiler	0.5	0.51	0.1	0.11	0.005	0.005		
Poultry – layer	0.51	0.51	5.1 ^e	1.88 ^f	0.005	0.005		

^a Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian tissues

^b Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk

^c Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

^d Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

^e Highest maximum poultry dietary burden suitable for maximum residue level estimated for poultry tissues and eggs.

^f Highest mean poultry dietary burden suitable for STMR estimated for poultry tissues and eggs

*Animal commodity estimations*Propiconazole residues, for the estimation of *maximum residue level*

	Feed level for milk or egg, ppm	Residues, mg/kg	Feed level for tissues, ppm	Residues, mg/kg			
				Muscle	Liver	Kidney	Fat
Maximum residue level for cattle							
Feeding study	15	< 0.01	15	< 0.01	0.14	< 0.01	< 0.05
	75	< 0.01	75	< 0.01	0.34	< 0.01	< 0.05
Dietary burden and highest residue	29	< 0.01	36	< 0.01	0.25	< 0.01	< 0.05
Maximum residue level for poultry							
Feeding study	7.5	< 0.05	7.5	< 0.05	< 0.05		< 0.05
Dietary burden and highest residue	5.1	< 0.05	4.6	< 0.05	< 0.05		< 0.05

Total propiconazole residues, for the estimation of HR and STMR

	Feed level for milk egg, ppm	Residues, mg/kg	Feed level for tissues, ppm	Residues, mg/kg			
				Muscle	Liver	Kidney	Fat
HR for cattle							
Feeding study			15	< 0.05	0.81	0.63	< 0.05
			75	0.11	4.3	4.7	0.23
Dietary burden and highest residue			36	0.085	1.97	1.9	0.115
STMR for cattle							
Feeding study	15	0.044	15	0.08	0.63	0.60	< 0.05
Dietary burden	12	0.035	12	0.064	0.504	0.50	< 0.05
HR for poultry							
Feeding study	7.5	< 0.05	7.5	< 0.05	< 0.05		< 0.05
Dietary burden and highest residue	5.1	< 0.05	4.6	< 0.05	< 0.05		< 0.05
STMR for poultry							
Feeding study	7.5	< 0.05	7.5	< 0.05	< 0.05		< 0.05
Dietary burden	1.88	< 0.05	4.6	< 0.05	< 0.05		< 0.05

The maximum dietary burden for the estimation of maximum residue levels in cattle tissue and milk were 36 and 29 ppm, respectively. The Meeting confirms its previous recommendation of 0.01* mg/kg for propiconazole milks and meat (from mammals other than marine mammals) and estimates a maximum residue level of 0.5 mg/kg for edible offal (mammalian) and of 0.01* mg/kg in mammalian fat (except milk fats).

The Meeting withdraw its previous recommendation of 0.01* mg/kg for propiconazole in edible offal (mammalian).

The maximum dietary burden for the estimation of HR in cattle tissues was 36 ppm. Based on the feeding levels at 15 and 75 ppm, the Meeting estimated a HR of 0.085 mg/kg in meat (from mammals other than marine mammals), of 1.97 mg/kg in edible offal (mammalian) and of 0.115 mg/kg in mammalian fat (except milk fats).

The mean dietary burden for the estimation of STMR levels in cattle tissues and milk was 12 ppm, respectively. Based on the feeding study conducted at 15 ppm, the Meeting estimates a STMR of 0.035 mg/kg in milks, 0.064 mg/kg in meat (from mammals other than marine mammals), 0.504 mg/kg in edible offal and 0.05 mg/kg in mammalian fat.

The highest calculated dietary burden for poultry is 35.1 ppm. In eggs, muscle and fat no propiconazole or total propiconazole residues were detected at the 7.5 ppm feeding level. The Meeting confirms its previous recommendation of a maximum residue level of 0.01* mg/kg for propiconazole in poultry meat and eggs, and a STMRs and HRs of 0.05 mg/kg in eggs, muscle and fat.

DIETARY RISK ASSESSMENT

Long-term intake

The IEDI of propiconazole based on the STMRs estimated by this and previous Meetings for the 17 GEMS/Food regional diets were 1–10% of the maximum ADI of 0.07 mg/kg bw (see Annex 3 of the 2014 Report). The Meeting concluded that the long-term dietary intake of residues of propiconazole is unlikely to present a public health concern.

Short-term intake

An ARfD for propiconazole is 0.3 mg/kg bw. The International Estimated Short-Term Intake (IESTI) of propiconazole for the commodities for which STMR, HR and maximum residue levels were estimated by the current Meeting. The results are shown in Annex 4 of the 2014 Report. The IESTI represented a maximum of 8% of the ARfD. The Meeting concluded that the short-term intake of propiconazole residues from uses considered by the current Meeting was unlikely to present a public health concern.

RECOMMENDATION

Residues: For plant and animal commodities is *propiconazole* for compliance with the MRL and *propiconazole plus all metabolites convertible to 2,4-dichlorobenzoic acid, expressed as propiconazole* for the estimation of the dietary intakes.

The residue if fat soluble

CCN	Commodity name	Recommended maximum residue level (mg/kg)		STMR (P) mg/kg	HR (P) mg/kg
		New	Previous		
AS 0640	Barley straw and fodder, dry	8	2		
MO 0105	Edible offal (mammalian)	0.5	0.01*	0.504	1.97
MF 0100	Mammalian fats (except milk fat)	0.01*		0.05	0.115
MM 0095	Meat (from mammals other than marine mammals)	0.01* (fat)	0.01* (fat)	0.064	0.085
MI 0106	Milks	0.01*	0.01*	0.035	
AS 0647	Oat straw and fodder, dry	8	2		
AS 0650	Rye straw and fodder, dry	15	2		
AS 0653	Triticale straw and fodder, dry	15	2		
AS 0654	Wheat straw and fodder, dry	15	2		

Feed commodities

Table entries in **bold** are new recommendations

Commodity	Median residues	Highest residue, mg/kg
Oat forage	1.5	2.5
Wheat forage	2.2	9
Barley hay	4.3	13
Oat hay	3.3	8.5
Wheat hay	5.3	22
Soya bean forage	1.87	3.45
Soya bean fodder	2.02	9.6
Sorghum forage	4.65	8.1
Sorghum stover	7.1	13.5
Rice straw	2.6	16.5
Corn forage	0.85	5
Corn stover/fodder	3.7	17
Almond hulls	1.4	
Peanut hay	7.6	14

Commodity	Median residues	Highest residue, mg/kg
Sugar beet leaves	0.3	0.96

REFERENCES

Author	Year	Study
Corley, J.	2011	Propiconazole: Magnitude of the Residue on Bean (Dry); Author: Johannes Corley, IR-4 Project HQ, Rutgers, The State University of New Jersey, Princeton, NJ 08540; dated: 7 March 2011. GLP, not published
Corley, J.	2011	Propiconazole: Magnitude of the Residue on Lima Bean; Author: Johannes Corley, IR-4 Project HQ, Rutgers, The State University of New Jersey, Princeton, NJ 08540; dated: 15 March 2011. GLP, not published
Corley, J.	2011	Propiconazole: Magnitude of the Residue on Snap Beans; Author: Johannes Corley, IR-4 Project HQ, Rutgers, The State University of New Jersey, Princeton, NJ 08540; dated: 7 March 2011. GLP, not published
Thompson, D.	2009	Propiconazole: Magnitude of the Residue on Mint; Author: Dave Thompson, IR-4 Project HQ, Rutgers, The State University of New Jersey, Princeton, NJ 08540; dated: 23 July 2009. GLP, not published
Thompson, D.	2007	Propiconazole: Magnitude of the Residue on Pineapple; Author: Dave Thompson, IR-4 Project HQ, Rutgers, The State University of New Jersey, Princeton, NJ 08540; dated: 28 November 2007. GLP, not published

PROTHIOCONAZOLE (232)

The first draft was prepared by Professor Árpád Ambrus, temporary adviser, Hungary.

EXPLANATION

Prothioconazole is a systemic triazolinthione fungicide that is used for the control of diseases caused by *Ascomycetes*, *Basidiomycetes* and *Deuteromycetes* in a range of crops. It is registered for use in a variety of crops worldwide.

Prothioconazole was evaluated for the first time by the 2008 JMPR, which established an ADI of 0–0.05 mg/kg bw and an ARfD of 0.8 mg/kg bw for woman of child bearing age for the parent prothioconazole, and an ADI of 0–0.01 mg/kg bw and an ARfD of 0.01 mg/kg bw for woman of child bearing age and 1 mg/kg bw for general population for prothioconazole-desthio. In addition, the Meeting recommended limits for a number of plant commodities and for mammalian meat and fats, edible offal and milks, based on a residue definition of ‘prothioconazole-desthio’.

The 2008 JMPR meeting concluded that the residue definition for plant commodities for enforcement and dietary risk assessment was prothioconazole-desthio. The residue definition for animal commodities for enforcement was prothioconazole-desthio and for dietary risk assessment the sum of prothioconazole-desthio, prothioconazole-desthio-3-hydroxy, prothioconazole-desthio-4-hydroxy and their conjugates, expressed as prothioconazole-desthio. The residue was considered to be not fat-soluble for the purposes of residue definition.

The 2009 JMPR evaluated residue data on additional commodities.

For the 2014 Meeting data were provided on blueberry, corn, cranberry, cucurbits, potato, sweet corn peanut and soya bean together with current use recommendations.

METHODS OF RESIDUE ANALYSIS

A number of validated analytical methods for the determination of residues in plant, animal tissue, milk and soils were evaluated by the 2007 and 2008 JMPR Meetings.

Detailed descriptions of additional analytical methods for crops that had been used in the residue trials evaluated by this Meeting are described below. References to report numbers are in parenthesis. Recovery data obtained during validation of the methods for plant and animal matrices are summarized in Table 1.

*Analytical methods for plant matrices**Method RPA JA/03/01*

Corn/sweet corn (Krolski & Harbin, 2008. RAJAP004; Krolski & Harbin, 2008. RAJAP001), soya beans (RAJAY026 and RAJAY027 in 2008/2009 JMPR)

Analyte:	Prothioconazole Prothioconazole-desthio Prothioconazole-sulfonic acid	HPLC/MS/MS	Method RPA JA/03/01
LOQ:	0.02 mg/kg		
Description:	Method RPA JA/03/01 was previously evaluated by JMPR in 2008. It has been used for the corn/sweet corn trials evaluated by this Meeting.		

Method JA-001-P04-02

Blueberry (Murphy & Dallstream, 2012, RAJAP022), cranberry (Murphy & Dallstream, 2012, RAJAP023), cucurbits (Murphy & Harbin, 2012, RAJAP024).

Analyte:	Prothioconazole	HPLC/MS/MS	Method JA-001-P04-02
	Prothioconazole-desthio		
	Prothioconazole-sulfonic acid		in report JA-001-P04-02

LOQ: 0.02 mg/kg

Description This is a modification of method RPA JA/03/01 and includes information about potential alternative analytical conditions.

The crop matrices are extracted with a mixture of methanol (MeOH), 30% hydrogen peroxide (H₂O₂), and aqueous sodium bicarbonate (NaHCO₃) at about 64°C for 2 hours. This extraction procedure converts prothioconazole to a mixture of prothioconazole sulfonic acid and desthio- prothioconazole. Residues of prothioconazole-desthio are extracted without change. Following the addition of a mixture of isotopically labelled prothioconazole sulfonic acid and prothioconazole-desthio internal standards, the sample extracts are purified by octadecyl solid phase extraction (C-18 SPE). The purified analytes are analysed by high performance liquid chromatography-electrospray/tandem mass spectrometry (LC-MS/MS).

Method 01013

Soya beans (Resende, 2013, F12-012 and Resende, 2013, F13-008), corn (Freitag & Reineke, 2009, 08-2035; Freitag & Reineke, 2009, 08-2036; Freitag, 2008, RA-2675/07; Bomke & Teubner, 2013, 11-2109; Bomke, 2012, 11-2110; Santiago, 2012, F11-034 and Resende, 2013, F12-011), potato (Bomke & Bauer, 2011, 10-2085) and oilseed rape (Freitag, Reineke & Krusell, 2010, 08-2112; Freitag & Hoffmann, 2010, 08-3112)

Analyte:	Prothioconazole	HPLC/MS/MS	Method 01013
	Prothioconazole-desthio		
			in reports MR-06/138, VM11-003 and VM12-006.

LOQ: 0.01 mg/kg

Description: Bayer method no. 01013 is suitable for the determination of the relevant residues of bixafen (BYF00587), prothioconazole, tebuconazole, trifloxystrobin and the metabolites BYF00587-desmethyl, prothioconazole-desthio (SXX0665) and CGA321113 in plant materials.

All analytes are extracted from plant materials using a mixture of acetonitrile/water (4/1; v/v, containing cysteine hydrochloride, by high-speed blending. After filtration of the extract, the stable isotopically labelled analytes are added. The solution is made up to volume, diluted and subjected to reversed phase HPLC-MS/MS without a further clean-up step. Residues are quantified using internal stable labelled standards.

Prothioconazole-desthio is detected using electrospray ionization in the positive ion mode (ESI+). The following transition ions are used for prothioconazole-desthio:

for quantification $m/z = 312 \rightarrow m/z = 70$

for confirmation $m/z = 312 \rightarrow m/z = 125$

Prothioconazole is detected using electrospray ionization in the negative ion mode (ESI-). The following transition ions are used for prothioconazole:

for quantification $m/z = 342 \rightarrow m/z = 100$
 for confirmation $m/z = 342 \rightarrow m/z = 125$

Method 01013 was developed for the determination of residues of prothioconazole and prothioconazole-desthio in/on plant materials, and validated in citrus fruit, pea seed, wheat grain, rape seed and corn green material (MR-06/138). The method has additionally been validated for prothioconazole and prothioconazole-desthio in soya bean seed and wheat grain (Resende, 2013, VM12-006) and also in bean seed, soya bean seed, oat grain, citrus fruit and sugar cane (Santiago, 2012, VM11-003) using the chromatographic conditions described in method 00765 (Sur, 2003, MR-231/02).

Method 00598/M001

Potato (Freitag & Wolters, 2007, RA-2569/05; Freitag & Wolters, 2007, RA-2604/05; Freitag & Wolters, 2007, RA-2570/05 and Freitag & Wolters, 2007, RA-2603/05)

Analyte: Prothioconazole HPLC/MS/MS Method 00598/M001
 Prothioconazole-desthio in report MR-689/99

LOQ: Wheat and barley grain, canola seed: 0.01 mg/kg
 Wheat and barley forage and straw: 0.05 mg/kg
 Potato tubers: 0.01 mg/kg

Description: Method 00598/M001 is a modification of method 00598 previously evaluated by JMPR in 2008. Method 00598/M001 was designed to measure prothioconazole and its main metabolite prothioconazole-desthio in cereal and canola matrices. It has been used in potato trials included in this evaluation.

Prothioconazole and prothioconazole-desthio are extracted from the samples with an acetonitrile/water mixture by high-speed blending after addition of cysteine-hydrochloride solution. After filtration the extract is cleaned up by liquid-liquid partition using n-hexane (saturated with acetonitrile) and dichloromethane. The analytes are chromatographed by reversed-phase HPLC on a silica-based C18-column using isocratic acetonitrile/water eluent containing acidic acid. The analytes are determined by HPLC-MS/MS in the MRM mode. Calibration is performed against internal labelled bracketing standards.

Prothioconazole-desthio is detected using electrospray ionization in the positive ion mode (ESI+) using the following transition ions for quantification: $m/z = 312 \rightarrow m/z = 70$.

Prothioconazole is detected using electrospray ionization in the negative ion mode (ESI-), using the following transition ions for quantification: $m/z = 342 \rightarrow m/z = 100$.

Method 00598/M001 was validated in wheat and barley grain, green material and straw, and canola seed, green material, straw and pod (Heinemann, 2000, MR-689/99).

Method ATM-0053

Peanut (Ellis, 2012, BCS-0232 and Ellis, 2012, BCS-0383)

Analyte: Prothioconazole HPLC/MS/MS Method ATM-0053
 Prothioconazole-desthio in report BCS-0232

- LOQ: Peanut Seeds, peanut shells: 0.01 mg/kg
Peanut forage: 0.05 mg/kg
- Description: Method ATM-0053 was used to analyse residues of prothioconazole and prothioconazole-desthio in plant material. It has been used for the peanut trials included in this evaluation.
- Residues are extracted with 20:80 water/acetonitrile after the addition of 12.5 g/L L-cysteine. The extract is filtered using a 0.45 µm filter. The analytes are determined by HPLC-MS/MS in the MRM mode. Quantitation is achieved using matrix matched analytical standards and a stable labelled internal standard for prothioconazole.
- Detection is by turbospray ionization in the positive ion mode using the following transition ions for quantification:
- Prothioconazole-desthio: $m/z = 312 \rightarrow m/z = 70$.
- Prothioconazole: $m/z = 344 \rightarrow m/z = 154$.
- Method ATM-0053 was developed for the determination of residues of prothioconazole and prothioconazole-desthio in/on cereal grain, straw and forage, canola seed and almond hull and shell. Additional recoveries in peanut matrices are provided in reports Ellis, 2012, BCS-0232 and Ellis, 2012, BCS-0383.

Analytical methods for animal matrices

Method 01009

Bovine milk, muscle, liver, kidney and fat, poultry egg (Billian & Wolters, 2006, MR-06/120 and Schulte & Oel, 2013, amendment 1 to MR-06/120)

Analyte:	JAU 6476-desthio	HPLC/MS/MS	Method: 01009
	JAU 6476-3-hydroxy-desthio		in reports MR-06/120 and
	JAU 6476-4-hydroxy-desthio		P 111G
	JAU 6476-3,4-dihydroxy-desthio		
	JAU 6476-4,5-dihydroxy-desthio		

LOQ: 0.01 mg/kg

Description: Method 01009 is for the determination of relevant residues of prothioconazole (JAU 6476-desthio, JAU 6476-3-hydroxy-desthio, JAU 6476-4-hydroxy-desthio, JAU 6476-3,4-dihydroxy-desthio, and JAU 6476-4,5-dihydroxy-desthio) in/on matrices of animal origin. This method has been independently validated (P 1111 G).

Residues are extracted from cattle (milk, muscle, kidney, liver, fat) and poultry (egg) matrices with acetonitrile / water (4/1, v/v) using a high-speed blender. Subsequently, the solutions are refluxed for 2 hours with 5 N HCl. This hydrolysis step cleaves conjugates to agylcones and converts the metabolites with diene structure back to aromatic compounds. Residues of all analytes are determined using HPLC-MS/MS. Residues are quantified against matrix-matched standards.

The following MRM transitions for quantitation and confirmation are used:

JAU 6476-desthio: m/z 312→70 or 312→125

JAU 6476-3-hydroxy-desthio: m/z 328→70 or 328→141

JAU 6476-4-hydroxy-desthio: m/z 328→70 or 328→141

JAU 6476-3,4-hydroxy-desthio: m/z 344→70 or 344→157

JAU 6476-4,5-hydroxy-desthio: m/z 344→70 or 344→157

Bovine meat, milk and egg (Bacher, 2006, P 1111 G)

Analyte: JAU 6476-desthio HPLC/MS/MS Method: 01009

JAU 6476-3-hydroxy-desthio

JAU 6476-4-hydroxy-desthio

JAU 6476-3,4-dihydroxy-desthio

JAU 6476-4,5-dihydroxy-desthio

LOQ: 0.01 mg/kg

Description: This is the independent laboratory validation (ILV) for method 01009, as described above.

Recovery data obtained during the validation of the methods described above are summarized in Table 1. In addition to these data, concurrent recoveries were also determined together with the analysis of field trial samples. Mean recoveries from both method validation and concurrent recoveries were within the acceptable range of 70%-120% with % RSD < 20%.

Multi-residue Methods

A GG/MS multi-residue method has been validated for the analysis of prothioconazole-desthio, and this method was evaluated at the 2008 JMPR Meeting.

A summary of recovery data with the methods used for plant commodities in this submission are presented in the table 1.

Table 1 Summary of recovery data obtained during method validation

Commodity	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
Plant commodities								
Blueberries	Prothioconazole-desthio	0.02-1.5	14	94-106	101	3.3	JA-001-P04-02	Murphy & Dallstream, 2012, RAJAP022
	Prothioconazole-sulfonic acid	0.02-1.5	14	75-106	97	8.7		
	Prothioconazole ^a	0.02-1.5	14	65-102	87	11.5		
Cranberries	Prothioconazole-desthio	0.02-1.5	11	84-118	103	10.3	JA-001-P04-02	Murphy & Dallstream, 2012, RAJAP023
	Prothioconazole-sulfonic acid	0.02-1.5	11	94-108	101	4.6		
	Prothioconazole ^a	0.02-1.5	12	88-103	96	6.1		
Summer squash	Prothioconazole-desthio	0.02-1.5	13	93-111	102	6.2	JA-001-P04-02	Murphy & Harbin, 2012, RAJAP024
	Prothioconazole-sulfonic acid	0.02-1.5	13	86-102	93	4.6		
	Prothioconazole ^a	0.02-1.5	15	85-105	90	5.4		
Melon	Prothioconazole-desthio	0.02-1.5	12	94-112	103	5.2	JA-001-P04-02	Murphy & Harbin, 2012, RAJAP024
	Prothioconazole-sulfonic acid	0.02-1.5	12	88-100	93	4.1		
	Prothioconazole ^a	0.02-1.5	12	70-105	92	8.9		

Commodity	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
Cucumber	Prothioconazole-desthio	0.02-1.5	10	88-113	102	7.3	JA-001-P04-02	Murphy & Harbin, 2012, RAJAP024
	Prothioconazole-sulfonic acid	0.02-1.5	10	87-97	93	3.6		
	Prothioconazole ^a	0.02-1.5	11	83-106	97	8.2		
Maize/Corn stover	Prothioconazole-desthio	0.02-20	16	79-112	93	10.7	RPA JA/03/01	Krolski & Harbin, 2008. RAJAP004
	Prothioconazole-sulfonic acid	0.02-20	16	89-103	98	4.1		
Maize/Corn grain	Prothioconazole-desthio	0.02-0.20	14	89-108	98	5.4	RPA JA/03/01	Krolski & Harbin, 2008. RAJAP004
	Prothioconazole-sulfonic acid	0.02-0.20	14	93-108	101	4.6		
Maize/Corn forage	Prothioconazole-desthio	0.02-10	18	86-110	97	6.5	RPA JA/03/01	Krolski & Harbin, 2008. RAJAP004
	Prothioconazole-sulfonic acid	0.02-10	18	90-108	97	4.7		
Sweetcorn ear without husk	Prothioconazole-desthio	0.02-0.20	13	88-104	96	5.2	RPA JA/03/01	Krolski & Harbin, 2008. RAJAP004
	Prothioconazole-sulfonic acid	0.02-0.20	13	86-103	96	5.8		
Sweetcorn forage	Prothioconazole-desthio	0.02-10	18	86-110	97	6.5	RPA JA/03/01	Krolski & Harbin, 2008. RAJAP004
	Prothioconazole-sulfonic acid	0.02-10	18	90-108	97	4.7		
Sweetcorn stover	Prothioconazole-desthio	0.02-20	16	79-112	93	10.7	RPA JA/03/01	Krolski & Harbin, 2008. RAJAP004
	Prothioconazole-sulfonic acid	0.02-20	16	89-103	98	4.1		
Popcorn stover	Prothioconazole-desthio	0.02-20	16	79-112	93	10.7	RPA JA/03/01	Krolski & Harbin, 2008. RAJAP004
	Prothioconazole-sulfonic acid	0.02-20	16	89-103	98	4.1		
Popcorn grain	Prothioconazole-desthio	0.02-0.20	14	89-108	98	5.4	RPA JA/03/01	Krolski & Harbin, 2008. RAJAP004
	Prothioconazole-sulfonic acid	0.02-20	14	93-108	101	4.6		
Maize/Corn grain	Prothioconazole-desthio	0.02-0.20	10	70-108	93	12.2	RPA JA/03/01	Krolski & Harbin, 2008, RAJAP001
	Prothioconazole-sulfonic acid	0.02-0.20	10	89-105	98	5.4		
Maize/Corn aspirated grain fractions	Prothioconazole-desthio	0.02-5.0	6	74-003	102	14.1	RPA JA/03/01	Krolski & Harbin, 2008, RAJAP001
	Prothioconazole-sulfonic acid	0.02-5.0	6	70-113	93	18.5		
Maize/Corn starch	Prothioconazole-desthio	0.02-0.50	6	77-102	90	11.9	RPA JA/03/01	Krolski & Harbin, 2008, RAJAP001
	Prothioconazole-sulfonic acid	0.02-0.50	6	84-100	90	6.1		
Maize/Corn oil (wet milled)	Prothioconazole-desthio	0.02-0.50	6	77-98	92	9.4	RPA JA/03/01	Krolski & Harbin, 2008, RAJAP001
	Prothioconazole-sulfonic acid	0.02-0.50	6	87-104	96	6.9		
Maize/Corn grits	Prothioconazole-desthio	0.02-0.50	6	80-103	94	10.3	RPA JA/03/01	Krolski & Harbin, 2008, RAJAP001
	Prothioconazole-sulfonic acid	0.02-0.50	6	77-104	90	13.1		
Maize/Corn flour	Prothioconazole-desthio	0.02-0.50	6	86-104	98	6.6	RPA JA/03/01	Krolski & Harbin, 2008, RAJAP001
	Prothioconazole-sulfonic acid	0.02-0.50	6	78-93	87	6.7		
Maize/Corn	Prothioconazole-desthio	0.02-0.50	6	89-113	98	8.5	RPA JA/03/01	Krolski &

Commodity	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
meal	Prothioconazole-sulfonic acid	0.02-0.50	6	71-96	79	11.3		Harbin, 2008, RAJAP001
Maize/Corn bran	Prothioconazole-desthio	0.02-0.50	6	80-102	92	10.3	RPA JA/03/01	Krolski & Harbin, 2008, RAJAP001
	Prothioconazole-sulfonic acid	0.02-0.50	6	79-98	86	7.7		
Maize/Corn oil (dry milled)	Prothioconazole-desthio	0.02-0.50	6	80-92	88	4.8	RPA JA/03/01	Krolski & Harbin, 2008, RAJAP001
	Prothioconazole-sulfonic acid	0.02-0.50	6	95-104	99	3.2		
Citrus fruit	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-0.10	10	84-107	100	6.6	01013	Brumhard & Stuke, 2007, MR-06/138
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-0.10	10	89-121	103	8.5		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-0.10	10	95-105	99	4.2		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-0.10	10	92-109	101	5.4		
Pea green seed	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-0.10	10	96-114	103	6.4	01013	Brumhard & Stuke, 2007, MR-06/138
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-0.10	10	95-113	104	6.1		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-0.10	10	87-96	94	2.9		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-0.10	10	93-110	97	5.4		
Rape seed	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-0.10	10	88-107	96	6.2	01013	Brumhard & Stuke, 2007, MR-06/138
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-0.10	10	88-103	95	5.4		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-0.10	10	91-106	97	5.3		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-0.10	10	90-107	97	5.3		
Wheat grain	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-0.10	10	91-103	97	3.4	01013	Brumhard & Stuke, 2007, MR-06/138
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-0.10	10	89-104	94	4.9		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-0.10	10	85-101	92	6.5		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-0.10	10	90-101	95	5.1		
Corn green material	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-0.10	10	97-119	100	7.9	01013	Brumhard & Stuke, 2007, MR-06/138
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-0.10	10	94-113	101	6.8		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-0.10	10	88-106	99	6.3		

Commodity	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-0.10	10	91-109	101	6.8		
Bean seed	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-1.0	10	90-105	99	4.6	01013 + chromatographic conditions from 00765	Santiago, 2012, VM11-003
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-1.0	10	89-107	101	5.0		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-1.0	10	98-115	107	6.8		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-1.0	10	101-120	108	6.2		
Soya bean seed	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-1.0	10	97-111	105	4.0	01013 + chromatographic conditions from 00765	Santiago, 2012, VM11-003
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-1.0	10	94-117	109	7.3		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-1.0	10	94-113	103	5.1		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-1.0	10	97-111	104	4.3		
Oat grain	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-1.0	10	86-104	93	5.9	01013 + chromatographic conditions from 00765	Santiago, 2012, VM11-003
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-1.0	10	88-111	97	10		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-1.0	10	92-106	98	5.0		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-1.0	10	93-103	98	3.6		
Citrus fruit	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-1.0	10	82-102	91	6.5	01013 + chromatographic conditions from 00765	Santiago, 2012, VM11-003
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-1.0	10	84-105	89	7.6		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-1.0	10	88-94	91	2.0		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-1.0	10	87-98	92	3.3		
Sugar cane	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-1.0	10	74-92	83	7.8	01013 + chromatographic conditions from 00765	Santiago, 2012, VM11-003
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-1.0	10	75-95	85	8.8		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-1.0	10	68-77	73	3.6		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-1.0	10	70-77	74	3.2		
Soya bean seed	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-1.0	10	107-118	112	3.9	01013	Resende, 2013, VM12-006
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-1.0	10	100-122	112	5.2		

Commodity	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-3.0	15	87-120	106	10.3		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-3.0	15	77-118	105	11.7		
Wheat grain	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-1.0	10	97-112	103	4.1	01013	Resende, 2013, VM12-006
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-1.0	10	96-110	102	4.0		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-1.0	10	96-105	99	5.0		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-1.0	10	90-103	97	5.0		
Wheat grain	Prothioconazole	0.01-0.10	10	81-106	96	7.4	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.01-0.10	10	71-96	89	7.8		
Wheat green material	Prothioconazole	0.05-5.0	15	81-100	93	5.6	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.05-5.0	15	89-97	94	2.5		
Wheat straw	Prothioconazole	0.05-5.0	14	65-90	85	8.4	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.05-5.0	14	83-96	91	3.9		
Barley grain	Prothioconazole	0.01-0.10	10	72-103	89	8.9	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.01-0.10	10	76-94	90	6.2		
Barley green material	Prothioconazole	0.05-5.0	15	87-100	95	3.6	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.05-5.0	15	87-95	91	2.1		
Barley straw	Prothioconazole	0.05-5.0	15	79-104	91	8.4	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.05-5.0	15	76-93	87	5.1		
Canola seed	Prothioconazole	0.01-0.10	20	69-118	89	14.7	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.01-0.10	20	64-95	83	11.0		
Canola green material	Prothioconazole	0.05-5.0	15	87-97	91	3.1	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.05-5.0	15	86-98	92	3.1		
Canola straw	Prothioconazole	0.05-5.0	15	84-117	101	11.1	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.05-5.0	15	80-95	91	4.4		
Canola pod	Prothioconazole	0.05-5.0	13	74-110	90	13.1	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.05-5.0	13	82-91	97	2.8		
Potato tuber (seed potato)	Prothioconazole	0.01-10	12	76-107	94	8.6	00598/M001	Freitag & Wolters, 2007, RA-2569/05 RA-2604/05 RA-2570/05 RA-2603/05
	Prothioconazole-desthio	0.01-10	12	89-100	94	3.7		
Potato tuber	Prothioconazole	0.01-0.10	18	78-107	97	7.1		
	Prothioconazole-desthio	0.01-0.10	18	80-110	91	6.7		
Triticale straw	Prothioconazole	0.01-1.0	10	72-102	84	12.5	ATM-0053	ATM-0053 in

Commodity	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
	Prothioconazole-desthio	0.01-1.0	10	91-119	99	8.4		Ellis, 2012, BCS-0232
Canola seed	Prothioconazole	0.01-1.0	10	84-106	91	6.5	ATM-0053	ATM-0053 in Ellis, 2012, BCS-0232
	Prothioconazole-desthio	0.01-1.0	10	94-102	99	2.6		
Wheat grain	Prothioconazole	0.01-1.0	10	78-106	94	11.0	ATM-0053	ATM-0053 in Ellis, 2012, BCS-0232
	Prothioconazole-desthio	0.01-1.0	10	87-108	93	8.8		
Barley forage	Prothioconazole	0.01-1.0	9	71-99	80	14.0	ATM-0053	ATM-0053 in Ellis, 2012, BCS-0232
	Prothioconazole-desthio	0.01-1.0	10	97-108	102	4.2		
Almond hull and shell	Prothioconazole	0.01-1.0	10	71-91	91	9.6	ATM-0053	ATM-0053 in Ellis, 2012, BCS-0232
	Prothioconazole-desthio	0.01-1.0	10	93-102	98	3.1		
Peanut Seeds	Prothioconazole	0.01-1.0	12	73-88	80	7.1	ATM-0053	Ellis, 2012, BCS-0232
	Prothioconazole-desthio	0.01-1.0	12	76-96	88	8.4		
Peanut shells	Prothioconazole	0.01-1.0	6	73-81	75	4.9	ATM-0053	Ellis, 2012, BCS-0232
	Prothioconazole-desthio	0.01-1.0	6	82-115	98	15.9		
Peanut fodder	Prothioconazole	0.01-1.0	6	70-98	82	16.0	ATM-0053	Ellis, 2012, BCS-0232
	Prothioconazole-desthio	0.01-1.0	6	81-89	84	3.2		
Peanut Seeds	Prothioconazole	0.01-1.0	17	82-109	93	10.2	ATM-0053	Ellis, 2012, BCS-0383
	Prothioconazole-desthio	0.01-1.0	17	74-111	95	11.4		
Peanut shells	Prothioconazole	0.01-1.0	14	74-118	92	13.6	ATM-0053	Ellis, 2012, BCS-0383
	Prothioconazole-desthio	0.01-1.0	17	76-108	95	10.2		
Peanut fodder	Prothioconazole	0.01-1.0	10	60-101	77	21.8	ATM-0053	Ellis, 2012, BCS-0383
	Prothioconazole-desthio	0.01-1.0	12	93-109	102	4.7		
Animal commodities								
Cattle milk	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-0.10	10	84-105	94	7.9	01009	Billian & Wolters, 2006, MR-06/120
Cattle muscle		0.01-0.10	10	82-98	91	6.8		
Cattle kidney		0.01-0.10	10	80-97	90	6.0		
Cattle liver		0.01-0.10	10	93-101	97	2.8		
Cattle fat		0.01-0.10	10	83-94	88	3.8		
Poultry egg		0.01-0.10	10	86-94	90	3.0		
Cattle milk	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-0.10	10	85-104	93	6.8	01009	Billian & Wolters, 2006, MR-06/120
Cattle muscle		0.01-0.10	10	83-99	92	6.6		
Cattle kidney		0.01-0.10	10	82-100	89	5.9		
Cattle liver		0.01-0.10	10	88-99	95	3.4		
Cattle fat		0.01-0.10	10	84-97	89	5.0		
Poultry egg		0.01-0.10	10	84-93	88	3.0		
Cattle milk	JAU 6476-3-hydroxy- desthio $m/z = 328 \rightarrow m/z = 70$	0.01-0.10	10	80-104	95	9.0	01009	Billian & Wolters, 2006, MR-06/120
Cattle muscle		0.01-0.10	10	82-99	91	6.8		
Cattle kidney		0.01-0.10	10	82-109	92	8.5		
Cattle liver		0.01-0.10	10	88-105	99	5.3		
Cattle fat		0.01-0.10	10	87-97	93	3.1		

Commodity	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
Poultry egg		0.01-0.10	10	88-99	93	4.6		
Cattle milk	JAU 6476-3-hydroxy- desthio $m/z = 328 \rightarrow m/z = 141$	0.01-0.10	10	79-106	93	9.9	01009	Billian & Wolters, 2006, MR-06/120
Cattle muscle		0.01-0.10	10	82-101	92	7.2		
Cattle kidney		0.01-0.10	10	85-105	94	6.9		
Cattle liver		0.01-0.10	10	94-105	101	3.9		
Cattle fat		0.01-0.10	10	83-102	92	6.3		
Poultry egg		0.01-0.10	10	87-99	93	4.7		
Cattle milk		0.01-0.10	10	76-103	92	10.9		
Cattle muscle	JAU 6476-4-hydroxy- desthio $m/z = 328 \rightarrow m/z = 70$	0.01-0.10	10	83-101	92	7.3	01009	Billian & Wolters, 2006, MR-06/120
Cattle kidney		0.01-0.10	10	80-105	90	7.6		
Cattle liver		0.01-0.10	10	91-105	99	5.8		
Cattle fat		0.01-0.10	10	90-100	95	3.1		
Poultry egg		0.01-0.10	10	85-99	91	5.4		
Cattle milk		0.01-0.10	10	78-102	91	9.8		
Cattle muscle		0.01-0.10	10	82-101	92	7.4		
Cattle kidney	JAU 6476-4-hydroxy- desthio $m/z = 328 \rightarrow m/z = 141$	0.01-0.10	10	86-104	93	6.1	01009	Billian & Wolters, 2006, MR-06/120
Cattle liver		0.01-0.10	10	90-107	98	5.6		
Cattle fat		0.01-0.10	10	86-103	93	4.8		
Poultry egg		0.01-0.10	10	88-94	91	2.3		
Cattle milk		0.01-0.10	10	78-105	95	10.6		
Cattle muscle		0.01-0.10	10	66-89	76	10.1		
Cattle kidney		0.01-0.10	10	86-104	93	5.5		
Cattle liver	JAU 6476-3,4-dihydroxy- desthio $m/z = 344 \rightarrow m/z = 70$	0.01-0.10	10	82-102	92	8.0	01009	Billian & Wolters, 2006, MR-06/120
Cattle fat		0.01-0.10	10	78-117	94	12.8		
Poultry egg		0.01-0.10	10	87-103	92	5.4		
Cattle milk		0.01-0.10	10	75-107	93	12.0		
Cattle muscle		0.01-0.10	10	66-88	75	9.1		
Cattle kidney		0.01-0.10	10	85-101	93	6.8		
Cattle liver		0.01-0.10	10	90-104	96	4.9		
Cattle fat	JAU 6476-3,4-dihydroxy- desthio $m/z = 344 \rightarrow m/z = 157$	0.01-0.10	10	79-115	90	13.5	01009	Billian & Wolters, 2006, MR-06/120
Poultry egg		0.01-0.10	10	87-100	92	5.4		
Cattle milk		0.01-0.10	10	77-111	94	11.3		
Cattle muscle		0.01-0.10	10	77-97	85	7.2		
Cattle kidney		0.01-0.10	10	82-103	92	6.7		
Cattle liver		0.01-0.10	10	88-107	96	5.2		
Cattle fat		0.01-0.10	10	84-124	97	13.4		
Poultry egg	JAU 6476-4,5-dihydroxy- desthio $m/z = 344 \rightarrow m/z = 70$	0.01-0.10	10	83-100	89	6.2	01009	Billian & Wolters, 2006, MR-06/120
Cattle milk		0.01-0.10	10	81-107	92	9.4		
Cattle muscle		0.01-0.10	10	76-97	85	7.4		
Cattle kidney		0.01-0.10	10	77-104	92	8.3		
Cattle liver		0.01-0.10	10	89-101	95	3.8		
Cattle fat		0.01-0.10	10	85-123	98	12.7		
Poultry egg		0.01-0.10	10	83-93	88	4.1		

Commodity	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
Bovine meat	Prothioconazole-desthio	0.01-0.10	10	98-100	98	1	01009	Bacher, 2006, P 1111 G
Milk	$m/z = 312 \rightarrow m/z = 70$ (quantitation)	0.01-0.10	10	98-105	101	2		
Egg		0.01-0.10	10	84-91	88	3		
Bovine meat	Prothioconazole-desthio	0.01-0.10	10	95-99	97	1	01009	Bacher, 2006, P 1111 G
Milk	$m/z = 312 \rightarrow m/z = 125$ (confirmation)	0.01-0.10	10	98-106	101	3		
Egg		0.01-0.10	10	82-92	87	3		
Bovine meat	JAU 6476-3-hydroxy- desthio	0.01-0.10	10	96-102	98	2	01009	Bacher, 2006, P 1111 G
Milk	$m/z = 328 \rightarrow m/z = 70$ (quantitation)	0.01-0.10	10	96-103	101	3		
Egg		0.01-0.10	10	84-96	89	4		
Bovine meat	JAU 6476-3-hydroxy- desthio	0.01-0.10	10	96-104	100	3	01009	Bacher, 2006, P 1111 G
Milk	$m/z = 328 \rightarrow m/z = 141$ (confirmation)	0.01-0.10	10	98-108	103	3		
Egg		0.01-0.10	10	87-98	91	4		
Bovine meat	JAU 6476-4-hydroxy- desthio	0.01-0.10	10	93-108	99	5	01009	Bacher, 2006, P 1111 G
Milk	$m/z = 328 \rightarrow m/z = 70$ (quantitation)	0.01-0.10	10	94-109	101	5		
Egg		0.01-0.10	10	85-101	89	5		
Bovine meat	JAU 6476-4-hydroxy- desthio	0.01-0.10	10	93-108	100	5	01009	Bacher, 2006, P 1111 G
Milk	$m/z = 328 \rightarrow m/z = 141$ (confirmation)	0.01-0.10	10	94-107	101	4		
Egg		0.01-0.10	10	85-100	89	5		
Bovine meat	JAU 6476-3,4-dihydroxy- desthio	0.01-0.10	10	86-94	90	4	01009	Bacher, 2006, P 1111 G
Milk	$m/z = 344 \rightarrow m/z = 70$ (quantitation)	0.01-0.10	10	93-109	99	5		
Egg		0.01-0.10	10	94-102	96	4		
Bovine meat	JAU 6476-3,4-dihydroxy- desthio	0.01-0.10	10	81-93	88	4	01009	Bacher, 2006, P 1111 G
Milk	$m/z = 344 \rightarrow m/z = 157$ (confirmation)	0.01-0.10	10	89-107	98	6		
Egg		0.01-0.10	10	91-101	96	3		
Bovine meat	JAU 6476-4,5-dihydroxy- desthio	0.01-0.10	10	84-93	89	4	01009	Bacher, 2006, P 1111 G
Milk	$m/z = 344 \rightarrow m/z = 70$ (quantitation)	0.01-0.10	10	94-102	97	2		
Egg		0.01-0.10	10	85-96	90	5		
Bovine meat	JAU 6476-4,5-dihydroxy- desthio	0.01-0.10	10	84-92	89	3	01009	Bacher, 2006, P 1111 G
Milk	$m/z = 344 \rightarrow m/z = 157$ (confirmation)	0.01-0.10	10	94-105	97	4		
Egg		0.01-0.10	10	87-99	92	5		

^a Prothioconazole recovery is the sum of prothioconazole-desthio and prothioconazole-sulfonic acid values from recovery samples fortified with prothioconazole only.

Storage Stability under Frozen Conditions

Plant commodities

Information relating to storage stability of residues in wheat, canola (seed, pod, straw), spinach (leaves), sugar beet (root, leaf with root collar), tomato (fruit) and field pea (dried) was evaluated at the 2008 JMPR Meeting. This showed that residues of prothioconazole-desthio were stable over a frozen storage period of up to 36 months in wheat and at least 24 months for the other crops. Residues

of prothioconazole and prothioconazole-desthio were stable in wheat hay and straw, canola seeds, mustard greens, turnip root and tomato fruit over a frozen storage period of 36-42 months.

Subsequently, a freezer storage stability study for residues of prothioconazole and prothioconazole-desthio (JAU6476-desthio) in wheat (forage, straw, grain, bran and flour), canola (seed and oil), mustard greens, tomato (fruit and paste) and turnip roots was performed over a 36-month period (Gould and Timberlake, 2008; Report RAJAY016), and the final results are summarized below.

Wheat, canola, mustard greens, tomato, turnip

Untreated control samples were fortified individually with prothioconazole or its metabolite prothioconazole-desthio at a concentration of 0.25 mg/kg and then frozen at < -10°C. Samples were analysed with method RPA JA/03/01 immediately after fortification (0 day) and after storage intervals up to 36 months. At the day 0, three fortified samples for each compound and one control were analysed. At subsequent analysis intervals, two stored samples and two freshly fortified samples for each compound and one control were analysed.

Prothioconazole is measured indirectly as the sum of the two oxidation products (prothioconazole-sulfonic acid and prothioconazole-desthio). Prothioconazole-desthio is measured directly. The limits of quantification (LOQ) were 0.01 mg/kg for prothioconazole-sulfonic acid and prothioconazole-desthio.

The stability of residues expressed as the mean of two replicate analyses in the stored samples is summarized in Table 2.

Table 2 Storage stability data and concurrent recovery data for prothioconazole and prothioconazole-desthio in various matrices

Storage Interval (days)	Prothioconazole		Prothioconazole-desthio	
	Procedural recovery (%)	Residues remained (%)	Procedural recovery (%)	Residues remained (%)
Canola oil				
0	90	-	95	-
64	92	89	99	99
132	93	86	93	90
190	90	80	93	90
380	91	79	93	89
771	101	74	98	99
1084	92	67	97	99
Canola seed				
0	88	-	91	-
68	87	82	94	96
134	74	66	95	90
197	72	63	93	90
386	71	57	95	90
778	63	53	96	96
1091	89	69	98	96
Mustard greens				
0	82	-	95	-
58	88	78	95	96
127	88	75	99	94

Storage Interval (days)	Prothioconazole		Prothioconazole-desthio	
	Procedural recovery (%)	Residues remained (%)	Procedural recovery (%)	Residues remained (%)
185	82	68	93	90
375	77	61	98	94
766	82	65	99	99
1079	90	71	98	96
Tomato fruit				
0	81	-	93	-
57	83	73	96	95
126	71	62	93	90
184	51	48	91	91
374	62	54	94	92
765	74	51	98	101
1078	89	69	98	98
Tomato paste				
0	91	-	94	-
58	87	84	98	97
126	89	79	92	91
184	86	64	92	90
374	88	59	93	89
765	93	54	96	98
1078	89	49	95	99
Turnip roots				
0	87	-	95	-
58	84	82	93	93
126	74	70	95	93
184	58	63	94	91
374	39	48	96	94
765	80	70	99	101
1078	95	90	98	100
Wheat bran				
0	84	-	96	-
63	89	75	97	99
131	84	69	97	92
189	82	63	94	91
378	86	55	87	88
770	84	57	95	97
1082	87	57	97	98
Wheat flour				
0	88	-	98	-
57	89	88	98	96
127	80	82	95	93
185	83	76	94	92
374	79	70	95	91

Storage Interval (days)	Prothioconazole		Prothioconazole-desthio	
	Procedural recovery (%)	Residues remained (%)	Procedural recovery (%)	Residues remained (%)
766	79	68	98	101
1078	90	73	98	97
Wheat forage				
0	76	-	92	-
59	82	75	96	97
130	78	70	94	90
188	69	62	91	89
377	57	48	93	90
769	53	50	98	99
1078	84	71	99	98
Wheat grain				
0	88	-	96	-
61	90	80	99	94
131	86	76	91	89
189	84	72	94	93
378	89	65	80	78
769	90	59	99	100
1083	91	56	96	98
Wheat straw				
0	92	-	95	-
60	89	85	91	94
130	87	76	91	87
188	84	72	91	88
377	85	73	94	91
769	116	101	122	122
1082	88	74	94	93

USE PATTERN

Prothioconazole is a broad spectrum systemic triazolinthione fungicide that is used for the control of diseases caused by *Ascomycetes*, *Basidiomycetes* and *Deuteromycetes* in a range of crops. It has protective, curative and eradicated activity and can be used as both a seed treatment and a foliar treatment. Its mode of action is interference with the synthesis of ergosterol in the target fungi by inhibition of CYP51, which catalyses demethylation at C14 of lanosterol or 24-methylene dihydrolanosterol, leading to morphological and functional changes in the fungal cell membrane.

Prothioconazole is registered for use in a variety of crops worldwide. In this evaluation the national registered uses are summarised where supervised trials have been conducted or in countries with similar GAPs to where the supervised trials were carried out. Table 3 represents a summary of these relevant GAPs.

Table 3 Registered uses of prothioconazole

Crop	Country	Formulation	Application				PHI days
			Method	Rate, g ai/ha	Spray conc. g ai/hL	No. or max (g ai/ha/ season)	
Berries and small fruit							
Bushberry subgroup (blueberry) ^a	USA	Proline SC 480 (480 g/L SC)	Foliar	200	213	2 (400 g ai/ha)	7
Bushberry subgroup (blueberry) ^a	Canada	Proline SC 480 (480 g/L SC)	Foliar	200	150-200	2 (400 g ai/ha)	7
Low growing berry subgroup, except strawberry (cranberry) ^a	USA	Proline SC 480 (480 g/L SC)	Foliar	175	186	2 (350 g ai/ha)	45
Low growing berry subgroup, except strawberry (cranberry) ^a	Canada	Proline SC 480 (480 g/L SC)	Foliar	175	175	2 (350 g ai/ha)	45
Fruiting vegetables, cucurbits							
Cucurbits ^a	USA	Proline SC 480 (480 g/L SC)	1 Soil + 2 Foliar	200	212	1 soil + 2 foliar (600 g ai/ha)	7
Cucurbits ^a	Canada	Proline SC 480 (480 g/L SC)	1 Soil + 2 Foliar	200 + 2 x 200	200	1 soil + 2 foliar (600 g ai/ha)	7
Cucurbits ^a	Canada	Proline SC 480 (480 g/L SC)	1 Soil + 4 Foliar	200 + 4 x 100	100-200	1 soil + 4 foliar (600 g ai/ha)	7
Fruiting vegetables, other than cucurbits							
Sweet corn	USA	Stratego YLD (125 g/L SC)	Foliar	46	39-246	4	0 ^e 14 ^f
Sweet corn	USA	Prosaro 421 SC (211 g/L SC)	Foliar	100	106-535	4 (400 g ai/ha)	7 ^e 49 ^f
Sweet corn	USA	ProlineSC 480	Foliar	200	106-535	4	0 ^e 14 ^{e,f}
Sweet corn	Canada	Proline SC 480 (480 g/L SC)	Foliar	200	200-400	4	14 ^{e,f}
Pulses							
Soya bean	USA	Proline SC 480 (480 g/L SC)	Foliar	88-175	94-583	3 (450 g ai/ha)	21
Soya bean	USA	Stratego YLD (125 g/L SC)	Foliar	42	39-225	3	21 ^b
Soya bean	USA	Provost 433 SC (145 g/L SC)	Foliar	32	23-171	3 (95 g ai/ha)	21
Soya bean	Canada	Proline SC 480 (480 g/L SC)	Foliar	100	100-200	1	20
Soya bean	Brazil	FOX SC 325 (175 g/L SC)	Foliar	70	35-350	2	30
Soya bean	Brazil	Proline (250 g/L EC)	Foliar	75	17-250	2	30
Soya bean	Brazil	FOX Xpro (175 g/L SC)	Foliar	70-87.5	46.7-438	4	30
Root and tuber vegetables							
Potato	USA	Emesto Silver (18 g/L SC)	Seed treatment	0.36 g ai/ 100 kg seed pieces	-	3 (160 g ai/ha)	-
Potato	Canada	Emesto Silver (18 g/L SC)	Seed treatment	0.36 g ai/ 100 kg seed pieces	-		-

Crop	Country	Formulation	Application				PHI days
			Method	Rate, g ai/ha	Spray conc. g ai/hL	No. or max (g ai/ha/ season)	
Potato	Germany (EU)	Monceren Pro FS 258 (8 g/L FS)	Seed treatment	0.48-0.64 g ai/100 kg seed pieces (12-16 g ai/ha)	-	1	-
Potato	Netherlands (EU)	Moncereen Pro FS 258 (8 g/L FS)	Seed treatment	0.48 g ai/100 kg seed pieces (12-24 g ai/ha)	-	1	-
Potato	Switzerland	Monceren Pro FS 258 (8 g/L FS)	Seed treatment	0.48 g ai/100 kg seed pieces (12 g ai/ha)	Undiluted or 12-20	1	-
Cereal grains							
Corn (field corn and popcorn)	USA	Proline SC 480 (480 g/L SC)	Foliar	200	213-714	4 (800 g ai/ha)	0 ^c 14 ^d
Corn	USA	Stratego YLD (125 g/L SC)	Foliar	46	19-246	2	0 ^c 14 ^d
Corn	USA	Prosaro 421 SC (211 g/L SC)	Foliar	100	106-535	4 (400 g ai/ha)	21 ^c 36 ^d
Corn	Canada	Proline SC 480 (480 g/L SC)	Foliar	200	200-400	1	14
Corn	Brazil	FOX SC 325 (175 g/L SC)	Foliar	70-87.5	35-438	2	15
Corn	Brazil	FOX XPRO (175 g/L SC)	Foliar	70-87.5	47-438	2	15
Corn	Belarus	Lamador FS 400 (250 g/L SC)	Seed treatment	50 g ai/tonne	-		-
Corn	Belarus	Prosaro 250 EC (125 g/L EC)	Foliar	125	33-63	1	30
Corn	Czech Republic (EU)	Prosaro 250 EC (125 g/L EC)	Foliar	125	14.6-62.5	1	35
Corn	France (EU)	Feuver FS 300 (300 g/L SC)	Seed treatment	4.5 g ai/ 50000 seeds		1	-
Corn/maize	Italy (EU)	Prosaro 250 EC (125 g/L EC) ⁱ	Foliar	125	25	2	-
Corn/maize	Slovak Republic (EU)	Prosaro 250 EC (125 g/L EC)	Foliar	125		2	-
Oilseeds							
Peanuts ^a	Australia	Brumby 480 SC (480 g/L SC)	Foliar	120-192	27-128	4	28 ^h
Peanuts	USA	Proline SC 480 (480 g/L SC) ⁱ	In-furrow, banded and foliar	175-200	94-426	4 (800 g ai/ha)	14 ^g
Peanuts	USA	Provost 433 SC (145 g/L SC)	Foliar	73-112	78-238	4 (450 g ai/ha)	14 ^g
Peanuts	Canada	Proline SC 480 (480 g/L SC)	Foliar	175-200	175-200	4 (800 g ai/ha)	14 ^g

^a Proposed new use^b For soya bean seed. Do not graze or feed soya bean forage or hay.^c For field corn forage^d For field corn/popcorn grain and fodder^e For sweet corn forage and ears^f For sweet corn fodder^g Do not feed hay or threshings or allow livestock to graze in treated areas^h Do not graze or cut for stock food for 4 weeks after application

The bush berry subgroup includes: Aronia berry; blueberry (highbush and lowbush); Chilean guava; highbush cranberry; currant (black, buffalo, and red); elderberry; European barberry; gooseberry; edible honeysuckle; huckleberry; jostaberry; juneberry (Saskatoon berry); lingonberry; native currant; salal; sea buckthorn; and cultivars, varieties, and/or hybrids of these.

Low growing berry subgroup, except strawberry, includes: Bearberry; bilberry; cloudberry; cranberry; muntries; partridgeberry; and cultivars, varieties, and/or hybrids of these.

RESIDUES RESULTING FROM SUPERVISED TRIALS

Supervised trials were conducted to support MRLs for the following crops or groups of crops: blueberry, cranberry, cucurbits (cucumber, summer squash and melon), sweet corn, soya bean, potato, maize (field corn, popcorn) and peanuts. The results of these supervised trials are summarized in the following tables.

Crop Group	Commodity	Country, year of trials	Table No.
Berries and other small fruits	Blueberry	USA and Canada, 2010	4
	Cranberry	USA, 2010	5
Fruiting vegetables, cucurbits	Cucurbits	USA and Canada, 2010	6
Fruiting vegetables, other than cucurbits	Sweet corn	USA, 2006	7
Pulses	Soya bean	USA, 2004-2005	8
		Brazil, 2012-2013	9
Root and tuber vegetables	Potato	Europe, 2005, 2010	10
Cereal grains	Corn/Maize Popcorn	USA, 2006	11
	Corn/Maize Corn/Maize	USA, 2006	12
		Brazil, 2011-2012	13
		Europe, 2007, 2008, 2011	14
		Australia, 2011-2012	15
	Peanuts	Australia, 2011-2012	15
Oilseeds	Sweetcorn forage and fodder	USA	16
		USA	17
		USA	18
		EU	19
		Australia	20
Animal feed	Field corn forage	USA	17
		USA	18
		USA	19
		EU	19
		Australia	20

In addition to the description and details of the field trials and analytical methods, each report includes procedural recoveries and in some cases a summary of the method validation.

The residue definition for monitoring and risk assessment is prothioconazole-desthio only. However for consistency with previous JMPR evaluations, for trials conducted in the USA, Canada and Brazil, the tables show residues of prothioconazole sulfonic acid (JAU 6476 sulfonic acid), prothioconazole-desthio (JAU 6476 desthio) and total prothioconazole (JAU 6476). For trials conducted in Australia, the tables show residues of prothioconazole (JAU 6476), prothioconazole-desthio (JAU 6476 desthio) and total prothioconazole (JAU 6476). Unless otherwise indicated: the prothioconazole (JAU 6478), JAU 6476 sulfonic acid and total prothioconazole (JAU6478 total) residues are expressed as mg prothioconazole equivalents/kg. The prothioconazole-desthio (JAU 6476 desthio) residue is expressed as mg prothioconazole-desthio/kg.

Only the residues of prothioconazole-desthio have been used for calculation of MRL, HR and STMR values.

The prothioconazole recovery was calculated as the sum of prothioconazole-desthio and prothioconazole-sulfonic acid from recovery samples fortified with prothioconazole only.

In a number of studies, residues of triazole-derived metabolites (TDMs) and hydroxylated metabolites were determined in addition to residues of prothioconazole, prothioconazole desthio and prothioconazole sulfonic acid. The results of these analyses are not summarised here as these analytes are not included in the residue definition for monitoring or risk assessment.

The maximum periods between sampling and analyses were within the tested storage stability period of 3 years for prothioconazole-desthio.

In the trials where multiple analyses were conducted on a single sample, the mean value is reported. Where multiple samples were taken from a single plot, the mean residue value is reported. Where results from separate plots with distinguishing characteristics such as different formulations, varieties or treatment schedules were reported, results are listed for each plot.

The performance parameters of the methods used in the trials are summarised in section on Methods of residue analysis. Results have not been corrected for concurrent method recoveries. Residues and application rates have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Residue values from the trials conducted according to the maximum GAP were used for the estimation of maximum residue levels. Those results included in the tables are underlined.

Berries and Other Small Fruit

Bushberries - Blueberry

Supervised trials in USA and Canada: Eleven supervised trials on blueberry were conducted in 2010 in the major blueberry-producing states in the USA and Canada (Murphy & Dallstream, 2012, RAJAP022). The trials were carried out according to the GAP in the USA/Canada. Two applications of an SC formulation containing 480 g/L prothioconazole were made at 6–7 day intervals and samples of blueberries were taken from each plot at 5–7 days after last application (DALA). In the two decline trials, additional samples were collected at 0, 3, 13 or 14, and 21 days after the last application.

The blueberries were frozen after collection and stored frozen (< -20 °C) until extraction and analysis, with the exception of the bulk ‘washing’ sample from trial JA007-10DA, which was shipped unfrozen. A sub-sample of blueberry RAC was taken from the bulk ‘washing’ sample and frozen after collection. A second sub-sample was washed with water, drained and then frozen.

Residues of prothioconazole on blueberries were determined by the method, JA-001-P04-02. Procedural recoveries at fortification levels of 0.02–1.5 mg/kg were within 70–120% for prothioconazole-desthio, prothioconazole-sulfonic acid and prothioconazole, with the exception of one recovery for prothioconazole at 0.02 mg/kg which was 65. The results of the trials are summarized in Table 4.

Table 4 Prothioconazole residues in blueberry resulting from supervised trials in the USA and Canada

BLUEBERRY Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
GAP, USA and Canada	480 g/L SC	200			2	7					
JA001-10HA Pennsylvania, USA, 2010 (Bluecrop)	480 g/L SC	197- 201	44	449- 458	2	6	Berry	0.05	<u>0.28</u>	0.32	Murphy & Dallstream, 2012, RAJAP022
JA002-10HA Pennsylvania, USA, 2010 (Elliot)	480 g/L SC	204- 207	54	381- 385	2	5	Berry	0.09	<u>0.70</u>	0.79	Murphy & Dallstream, 2012, RAJAP022
JA003-10HA New York, USA, 2010 (Duke)	480 g/L SC	197- 199	72	274- 276	2	7	Berry	0.16	<u>0.52</u>	0.67	Murphy & Dallstream, 2012, RAJAP022

BLUEBERRY Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
JA004-10DA New York, USA, 2010 (Blue ray)	480 g/L SC	203	72	282- 282	2	0 3 7 14 21	Berry	0.22 0.12 0.09 0.06 0.05	0.58 0.42 <u>0.26</u> 0.16 0.11	0.80 0.54 0.35 0.22 0.16	Murphy & Dallstream, 2012, RAJAP022
JA005-10HA North Carolina, USA, 2010 (Blue Haven)	480 g/L SC	194- 200	70	276- 283	2	7	Berry	0.02	<u>0.15</u>	0.17	Murphy & Dallstream, 2012, RAJAP022
JA006-10HA Georgia, USA, 2010 (Climax)	480 g/L SC	201- 203	47	424- 429	2	7	Berry	0.05	<u>0.56</u>	0.61	Murphy & Dallstream, 2012, RAJAP022
JA007-10DA Georgia, USA, 2010 (Bright Blue)	480 g/L SC	201	47	423- 425	2	0 3 7 13 21 7	Berry Berry, washed	0.06 0.03 0.02 < 0.02 < 0.02 < 0.02	0.40 0.27 <u>0.22</u> 0.12 0.08 0.18	0.46 0.29 0.25 0.13 0.08 0.19	Murphy & Dallstream, 2012, RAJAP022
JA008-10HA Illinois, USA, 2010 (Duke)	480 g/L SC	202- 206	106- 108	190- 192	2	7	Berry	0.18	<u>0.87</u>	1.0	Murphy & Dallstream, 2012, RAJAP022
JA009-10HA Illinois, USA, 2010 (Duke)	480 g/L SC	201	46-47	201	2	7	Berry	0.06	<u>0.60</u>	0.66	Murphy & Dallstream, 2012, RAJAP022
JA010-10HA Ontario, Canada, 2010 (Blue Crop)	480 g/L SC	206- 213	80	256- 266	2	7	Berry	0.07	<u>0.65</u>	0.72	Murphy & Dallstream, 2012, RAJAP022
JA011-10HA Oregon, USA, 2010 (Blue Crop)	480 g/L SC	198- 202	50-57	198- 202	2	7	Berry	0.04	<u>0.42</u>	0.45	Murphy & Dallstream, 2012, RAJAP022

Low growing berry subgroup, except strawberry

Cranberry

Six supervised trials were conducted according to the GAP for cranberries in the USA/Canada in the major cranberry-producing states in the USA (Murphy & Dallstream, 2012, RAJAP023) in 2010. Two applications of an SC formulation containing 480 g/L prothioconazole were made at 7–8 day intervals and samples of cranberries were taken from each plot at 43–46 DALA. In the decline trial, additional samples were collected at 35, 40, 50, and 55 days after the last application. The cranberries were frozen after collection and stored frozen (< -20 °C) until extraction and analysis, with the exception of the bulk ‘washing’ sample from trial JA015-10DA, which was shipped unfrozen. A sub-sample of

cranberry RAC was taken from the bulk 'washing' sample and frozen after collection. A second sub-sample was washed with water, drained and then frozen.

Residues of prothioconazole on cranberries were determined with method, JA-001-P04-02. Procedural recoveries at fortification levels of 0.02–1.5 mg/kg were within 70–120% for prothioconazole-desthio, prothioconazole-sulfonic acid and prothioconazole. The results of the trials are summarized in Table 5.

Table 5 Prothioconazole residues in cranberry resulting from supervised trials in the USA

CRANBERRY Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/l)	g ai/ha	g ai/hL	Water (L/ha)	No						
GAP, USA and Canada	480 g/L SC	175			2	45					
JA012-10HA New Jersey, USA, 2010 (Stevens)	480 g/L SC	174- 176	41	421- 430	2	46	Fruit	< 0.02	<u>< 0.02</u>	< 0.04	Murphy & Dallstream, 2012, RAJAP023
JA013-10HA New York, USA, 2010 (Pilgrim)	480 g/L SC	179- 180	62-63	281- 290	2	45	Fruit	< 0.02	<u>< 0.02</u>	< 0.04	Murphy & Dallstream, 2012, RAJAP023
JA014-10HA Wisconsin, USA, 2010 (Grygleski GHI)	480 g/L SC	169- 175	70-75	234- 243	2	44	Fruit	< 0.02	<u>0.03</u>	< 0.04	Murphy & Dallstream, 2012, RAJAP023
JA015-10DA Wisconsin, USA, 2010 (Stevens)	480 g/L SC	170- 173	70-74	234- 243	2	35	Fruit	< 0.02	0.02	< 0.04	Murphy & Dallstream, 2012, RAJAP023
						40		< 0.02	< 0.02	< 0.04	
						43		< 0.02	<u>< 0.02</u>	< 0.04	
						50		< 0.02	< 0.02	< 0.04	
						55		< 0.02	< 0.02	< 0.04	
						43	Whole fruit, washed	< 0.02	< 0.02	< 0.04	
JA016-10HA British Columbia, USA, 2010 (Pilgrims)	480 g/L SC	168- 170	87-90	187- 196	2	43	Fruit	< 0.02	<u>0.09</u>	0.09	Murphy & Dallstream, 2012, RAJAP023
JA017-10HA Washington, USA, 2010 (Stevens)	480 g/L SC	172- 173	74	232- 233	2	46	Fruit	< 0.02	<u>0.03</u>	< 0.04	Murphy & Dallstream, 2012, RAJAP023

Fruiting Vegetables, Cucurbits

Cucumber, melon and summer squash

In 2010, eight supervised trials were conducted on cucumber, eight trials on musk melon and eight trials on summer squash according to the GAP for cucurbits in the USA/Canada in the USA (Murphy & Harbin, 2012, RAJAP024). One application was made by drip irrigation and two foliar applications

were made using an SC formulation containing 480 g/L prothioconazole, with a 5-8 days interval between the two foliar applications.

Samples of cucumbers, melon or summer squash were taken from each plot prior to the 3rd application (shown as -0 days in the table below), at 2-3 days and at 5-8 DALA. In the decline trials, additional samples were collected at 0, 13-14 and 19-21 days after the last application. The samples were frozen after collection and stored frozen (< -20°C) until extraction and analysis, with the exception of the bulk samples from trials JA032-10HA and JA033-10HA (melon) and JA039-10DA (summer squash), which were shipped unfrozen. Sub-samples of melon and summer squash RAC were taken from the bulk samples and frozen after collection. Second sub-samples of melon were peeled and the pulp frozen. A second sub-sample of summer squash was washed with water and cooked, and samples of washed and cooked summer squash were stored frozen.

Residues of prothioconazole on cucurbits were determined with the method JA-001-P04-02. Procedural recoveries at fortification levels of 0.02-1.5 mg/kg were within 70-120% for prothioconazole-desthio, prothioconazole-sulfonic acid and prothioconazole in each matrix. The results of the trials are summarized in Table 6.

Table 6 Prothioconazole residues in cucurbits resulting from supervised trials in the USA and Canada

CUCURBITS Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/l)	g ai/ha	g ai/hL	Water (L/ha)	No						
GAP, USA and Canada	480 g/L SC	200			3	7					
CUCUMBER											
JA019-10DA Georgia, USA, 2010 (Thunder)	480 g/L SC	197- 200	86-88	224- 230	3	-0 0 3 7 14 21	Fruit	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.02 0.04 0.04 <u>0.03</u> 0.02 < 0.02	< 0.04 0.05 0.04 < 0.04 < 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA020-10HA North Carolina, USA, 2010 (Long Green)	480 g/L SC	197- 204	85-138	146- 240	3	-0 3 8	Fruit	< 0.02 < 0.02 < 0.02	0.03 0.04 <u>0.03</u>	< 0.04 0.05 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA021-10HA Florida, USA, 2010 (Alibi)	480 g/L SC	197- 201	71	279- 281	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	< 0.02 0.02 <u>0.02</u>	< 0.04 < 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA022-10HA Ohio, USA, 2010 (Intimidator F1)	480 g/L SC	198- 200	119	166- 168	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	0.04 0.07 <u>0.05</u>	0.04 0.07 0.06	Murphy & Harbin, 2012, RAJAP024
JA023-10HA Illinois, USA, 2010 (Eureka)	480 g/L SC	200- 203	66-70	291- 303	3	-0 2 7	Fruit	< 0.02 < 0.02 < 0.02	0.02 0.05 <u>0.04</u>	< 0.04 0.05 0.04	Murphy & Harbin, 2012, RAJAP024
JA024-10HA Ontario, Canada, 2010 (Talladega)	480 g/L SC	198- 203	84-88	231- 235	3	-0 3 6	Fruit	< 0.02 < 0.02 < 0.02	0.06 0.12 <u>0.06</u>	0.06 0.14 0.07	Murphy & Harbin, 2012, RAJAP024

CUCURBITS Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/l)	g ai/ha	g ai/hL	Water (L/ha)	No						
JA025-10HA Illinois, USA, 2010 (Talladega)	480 g/L SC	200- 208	62	326- 338	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	0.02 0.04 <u>0.02</u>	< 0.04 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA026-10DA Texas, USA, 2010 (Pointsett)	480 g/L SC	199- 202	129- 133	152- 154	3	-0 0 3 7 14 21	Fruit	< 0.02 0.03 < 0.02 < 0.02 < 0.02 < 0.02	0.04 0.08 0.08 <u>0.05</u> 0.03 0.02	0.05 0.11 0.10 0.05 < 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
MELON, NETTED (MUSK)											
JA028-10HA Georgia, USA, 2010 (Athena)	480 g/L SC	200- 203	96-99	204- 212	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	0.03 0.08 <u>0.03</u>	0.04 0.09 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA029-10HA Illinois, USA, 2010 (Hales Best)	480 g/L SC	200- 203	72-73	277- 278	3	-0 2 7	Fruit	< 0.02 0.02 < 0.02	0.03 0.13 <u>0.06</u>	< 0.04 0.15 0.07	Murphy & Harbin, 2012, RAJAP024
JA030-10DA Ontario, Canada, 2010 (Earlisweet)	480 g/L SC	198- 200	84-86	230- 236	3	-0 0 3 7 14 19	Fruit	< 0.02 0.05 < 0.02 < 0.02 < 0.02 < 0.02	0.05 0.18 0.10 <u>0.06</u> 0.06 0.05	0.06 0.23 0.11 0.07 0.07 0.06	Murphy & Harbin, 2012, RAJAP024
JA031-10HA Ontario, Canada, 2010 (Primo)	480 g/L SC	197- 201	84	233- 235	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	0.07 0.18 <u>0.07</u>	0.08 0.20 0.08	Murphy & Harbin, 2012, RAJAP024
JA032-10HA Texas, USA, 2010 (Mainstream)	480 g/L SC	200- 202	133- 140	143- 152	3	-0 3 7 3	Fruit Pulp	< 0.02 < 0.02 < 0.02 < 0.02	0.03 0.09 <u>0.06</u> < 0.02	< 0.04 0.10 0.07 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA033-10HA California, USA, 2010 (Jumbo Cantaloupe)	480 g/L SC	200- 202	69-71	287- 292	3	-0 3 7 3	Fruit Pulp	< 0.02 < 0.02 < 0.02 < 0.02	0.06 0.19 <u>0.15</u> < 0.02	0.07 0.20 0.17 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA034-10HB California, USA, 2010 (Honeydew)	480 g/L SC	198- 204	80-81	248- 252	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	0.04 0.10 <u>0.06</u>	0.05 0.11 0.07	Murphy & Harbin, 2012, RAJAP024

CUCURBITS Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/l)	g ai/ha	g ai/hL	Water (L/ha)	No						
JA035-10DA California, USA, 2010 (Hal's Best Jumbo)	480 g/L SC	200- 201	59	337- 342	3	-0 0 2 7 14 21	Fruit	< 0.02 0.04 < 0.02 0.02 < 0.02 < 0.02	0.07 0.22 0.09 <u>0.15</u> 0.12 0.07	0.08 0.25 0.11 0.17 0.13 0.07	Murphy & Harbin, 2012, RAJAP024
SUMMER SQUASH											
JA036-10HA New York, USA, 2010 (Sunray)	480 g/L SC	200- 204	72	282- 283	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 <u>< 0.02</u>	< 0.04 < 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA037-10DA North Carolina, USA, 2010 (Yellow Straight Neck)	480 g/L SC	198- 201	111- 138	143- 181	3	-0 0 3 7 13 20	Fruit	< 0.02 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.03 0.10 0.02 <u>< 0.02</u> < 0.02 < 0.02	< 0.04 0.12 0.03 < 0.04 < 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA038-10HA Florida, USA, 2010 (Early Summer Crookneck)	480 g/L SC	197- 201	71	280- 281	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	< 0.02 0.02 <u>< 0.02</u>	< 0.04 < 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA039-10DA Illinois, USA, 2010 (Golden Dawn III)	480 g/L SC	200- 201	59-74	271- 343	3	-0 0 3 6 13 21 3 3	Fruit Fruit, washed Fruit, cooked	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.02 0.07 0.04 <u>0.03</u> < 0.02 < 0.02 0.02 0.02	< 0.04 0.09 0.05 < 0.04 < 0.04 < 0.04 < 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA040-10HA Ontario, Canada, 2010 (Senator)	480 g/L SC	197- 201	80-84	235- 251	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	< 0.02 0.03 <u>< 0.02</u>	< 0.04 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA041-10HA Ontario, Canada, 2010 (Sunray)	480 g/L SC	199- 200	82-84	237- 369	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 <u>< 0.02</u>	< 0.04 < 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA042-10HA California, USA, 2010 (Dark green Zucchini)	480 g/L SC	199- 202	54	369- 374	3	-0 2 6	Fruit	< 0.02 < 0.02 < 0.02	0.03 0.08 <u>0.05</u>	< 0.04 0.09 0.05	Murphy & Harbin, 2012, RAJAP024

CUCURBITS Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/l)	g ai/ha	g ai/hL	Water (L/ha)	No						
JA043-10HA British Columbia, USA, 2010 (Noche)	480 g/L SC	199- 201	100	201	3	-0 2 5	Fruit	< 0.02 < 0.02 < 0.02	< 0.02 0.07 <u>0.06</u>	< 0.04 0.08 0.06	Murphy & Harbin, 2012, RAJAP024

Fruiting Vegetables, Other Than Cucurbits

Sweet corn

Supervised trials in the USA: Twelve supervised trials were conducted in 2006 on sweet corn in the USA (Krolski & Harbin, 2008, RAJAP004). The trials were carried out with 4×200 g ai/ha. The currently supported GAP in the USA is 4×100 g ai/ha. Four foliar applications were made to each treated plot of an SC formulation containing 480 g/L prothioconazole with a 2–11-day intervals between the each application. Each spray included a non-ionic surfactant.

In the sweet corn trials, ears, including seeds plus cob with husk removed, were harvested at 0 and 5–7 DALA (BBCH 73–79). In the decline trial, ears samples were collected at 0, 3, 6, 10 and 14 days. The samples were frozen after collection and stored frozen (< -20 °C) until extraction and analysis. Residues of prothioconazole were determined with method RPA JA/03/01. Procedural recoveries at fortification levels of 0.02–20 mg/kg were within 70–120% for prothioconazole-desthio, prothioconazole-sulfonic acid and prothioconazole in each matrix. The results of the trials are summarized in Table 7.

Table 7 Prothioconazole residues in sweet corn resulting from supervised trials in the USA

SWEET CORN Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/l)	g ai/ha	g ai/hL	Water (L/ha)	No						
GAP, USA	480 g/L SC	100			4	0/14					
JA002-06HA Virginia, USA, 2006 (Devotion)	480 g/L SC	201- 208	128	156- 163	4	0 6	Ear Ear	< 0.02 < 0.02	<u>< 0.018</u> < 0.018	< 0.02 < 0.02	Krolski & Harbin, 2008, RAJAP004
JA003-06HA New York, USA, 2006 (Gourmet Sweet 372A)	480 g/L SC	204- 208	122- 123	167- 170	4	0 7	Ear Ear	< 0.02 < 0.02	<u>< 0.018</u> < 0.018	< 0.02 < 0.02	Krolski & Harbin, 2008, RAJAP004
JA005-06HA Georgia, USA, 2006 (Sweet G-90)	480 g/L SC	200	142- 159	125- 141	4	0 7	Ear Ear	< 0.02 < 0.02	<u>< 0.018</u> < 0.018	< 0.02 < 0.02	Krolski & Harbin, 2008, RAJAP004

SWEET CORN Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/l)	g ai/ha	g ai/hL	Water (L/ha)	No						
JA007-06DA Florida, USA, 2006 (Silver Queen)	480 g/L SC	195- 200	167- 181	109- 120	4	0	Ear	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
						3	Ear	< 0.02	< 0.018	< 0.02	
						6	Ear	< 0.02	< 0.018	< 0.02	
						10	Ear	< 0.02	< 0.018	< 0.02	
						14	Ear	< 0.02	< 0.018	< 0.02	
JA008-06HA Missouri, USA, 2006 (Peaches & Cream)	480 g/L SC	200- 204	115- 149	137- 174	4	0	Ear	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
						6	Ear	< 0.02	< 0.018	< 0.02	
JA009-06HA Illinois, USA, 2006 (BC 0805)	480 g/L SC	196- 204	150- 160	123- 132	4	0	Ear	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
						7	Ear	< 0.02	< 0.018	< 0.02	
JA010-06HA Nebraska, USA, 2006 (Serendipity)	480 g/L SC	199- 204	153- 157	129- 131	4	0	Ear	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
						5	Ear	< 0.02	< 0.018	< 0.02	
JA011-06HA Minnesota, USA, 2006 (39H85 Pioneer)	480 g/L SC	196- 202	111- 118	171- 181	4	0	Ear	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
						7	Ear	< 0.02	< 0.018	< 0.02	
JA012-06HA Iowa, USA, 2006 (Mirai 131)	480 g/L SC	198- 206	156- 182		4	0	Ear	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
						7	Ear	< 0.02	< 0.018	< 0.02	
JA031-06HA California, USA, 2006 (Silver Queen)	480 g/L SC	197- 201	117- 119	168- 170	4	0	Ear	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
						7	Ear	< 0.02	< 0.018	< 0.02	
JA032-06HA Washington, USA, 2006 (Golden Jubilee)	480 g/L SC	202- 204	143- 145	140- 142	4	0	Ear	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
						7	Ear	< 0.02	< 0.018	< 0.02	
JA033-06HA Oregon, USA, 2006 (Serendipity)	480 g/L SC	195- 205	120- 126	163- 165	4	0	Ear	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
						5	Ear	< 0.02	< 0.018	< 0.02	

Pulses

Soya beans

Supervised trials in the USA: Residue data on soya beans from 19 trials conducted in the USA were previously evaluated by the 2008 JMPR and 2009 JMPR. The current USA GAP is 3×88 –175 g ai/ha, PHI 21 days.

Three foliar spray applications of an SC formulation were made using ground-based equipment to

soya beans at 145 to 188 g ai/ha. Intervals between applications were from 7 to 11 days (Duah, F. K.; Harbin, A. M, 2006, Report No.: RAJAY026). A non-ionic surfactant was added as a spray adjuvant at the lowest labelled rate. Duplicate treated samples of seed were collected at PHIs ranging from 19 to 23 days. Residues of prothioconazole, prothioconazole-desthio and prothioconazole sulfonic acid were analysed using Method RPA JA/03/01 with LOQ of 0.05 mg/kg.

Table 8 Prothioconazole residues in soya bean seed after three foliar treatments in supervised trials in the USA

SOYA BEAN (Variety) Location Year	Application				Residues (mg/kg) in soya bean seed			Report No Trial No. Doc No
	kg ai/ha	kg ai/hL	GS	DAT	JAU 6476 -sulfonic acid	JAU 6476- desthio	JAU 6476- total	
Soya bean (USA)	3 x 0.15			21	Min 140 L/ha (ground), 47 L/ha (air) Max 0.45 kg ai/ha/year			SC 480 (JMPR08)
Soya (Pioneer 96M20) Molino, Florida 2004	0.145- 0.151	0.1- 0.103	GS88	7 14 21 28 35	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 <u>< 0.05</u> < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	RAJAY026 JA014-04D-P2 M-270206-01-1
Soya (Stine 2788) Seymour, Illinois 2004	0.151- 0.154	0.115- 0.117	GS89	7 13 19 27 34	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 <u>< 0.05</u> < 0.05 < 0.05	< 0.05, < 0.05 < 0.05 < 0.05 < 0.05	RAJAY026 JA019-04D-P2 M-270206-01-1
Soya (Pioneer RR 97B52) Tifton, Georgia 2004	0.142- 0.15	0.101- 0.105	GS80	21	< 0.05	<u>< 0.05, < 0.05</u>	< 0.05, < 0.05	RAJAY026 JA015-04H-P2 M-270206-01-1
Soya (Pioneer 9492RR) Leland, Mississippi 2004	0.15- 0.157	0.097- 0.102	GS79	20	< 0.05, < 0.05	<u>< 0.05</u>	< 0.05	RAJAY026 JA016-04H-P2 M-270206-01-1
Soya (DP 5915 RR) Washington, Louisiana 2004	0.15- 0.157	0.077- 0.096	GS81	21	< 0.05, < 0.05	<u>< 0.05</u>	< 0.05	RAJAY026 JA017-04H-P2 M-270206-01-1
Soya (DP5634RR) Proctor, Arkansas 2004	0.149- 0.15	0.107- 0.108	GS79	21	< 0.05, < 0.05	<u>0.055</u>	0.06	RAJAY026 JA018-04H M-270206-01-1
Soya (Fontanelle 431RR) Stilwell, Kansas 2004	0.149- 0.153	0.103- 0.109	GS87	23	< 0.05, < 0.05	<u>0.051</u>	0.07	RAJAY026 JA020-04H M-270206-01-1
Soya (NKS28W2) Springfield, Nebraska 2004	0.15- 0.15	0.109- 0.111	GS80	19	< 0.05, < 0.05	<u>< 0.05</u>	< 0.05	RAJAY026 JA021-04H-P2 M-270206-01-1

SOYA BEAN (Variety) Location Year	Application				Residues (mg/kg) in soya bean seed			Report No Trial No. Doc No
	kg ai/ha	kg ai/hL	GS	DAT	JAU 6476 -sulfonic acid	JAU 6476- desthio	JAU 6476- total	
Soya (Croplan RT0907) Britton, South Dakota 2004	0.149- 0.149	0.159- 0.159	GS83	19	< 0.05, < 0.05	<u>< 0.05</u>	< 0.05	RAJAY026 JA024-04H-P2 M-270206-01-1
Soya (Croplan RT1447) Dumfries, Minnesota 2004	0.15- 0.151	0.085- 0.085	GS79	21	< 0.05, < 0.05	<u>< 0.05</u>	< 0.05	RAJAY026 JA025-04H-P2 M-270206-01-1
Soya (SC 9373) New Holland, Ohio 2004	0.151- 0.155	0.01- 0.105	GS93	20	< 0.05, < 0.05	<u>0.105</u>	0.14	RAJAY026 JA026-04H M-270206-01-1
Soya (92M70) Bagley, Iowa 2004	0.148- 0.151	0.11- 0.118	GS79	19	< 0.05	<u>< 0.05</u>	< 0.05	RAJAY026 JA027-04H-P2 M-270206-01-1
Soya (DynaGro DG 32M32RR) York, Nebraska 2004	0.149- 0.15	0.08- 0.08	GS80	19	< 0.05	<u>< 0.05</u>	< 0.05	RAJAY026 JA028-04H-P2 M-270206-01-1
Soya (Pioneer RR) USA Sheridan, Indiana 2004	0.146- 0.148	0.094- 0.095	GS97	21	< 0.05	<u>< 0.05</u>	< 0.05	RAJAY026 JA029-04H M-270206-01-1
Soya (Pioneer 93M80) USA Richland, Iowa 2004	0.15- 0.152	0.093- 0.118	GS83	21	< 0.05, < 0.05	<u>< 0.05</u>	< 0.05	RAJAY026 JA030-04H-P2 M-270206-01-1
Soya (Pioneer 91M50) USA Geneva, Minnesota 2004	0.15- 0.152	0.094- 0.097	GS80	20	< 0.05, < 0.05	<u>< 0.05</u>	< 0.05	RAJAY026 JA031-04H-P2 M-270206-01-1
Soya (Pioneer 93B85) USA Geneva, Minnesota 2004	0.149- 0.15	0.088- 0.089	GS77	21	< 0.05, < 0.05	<u>< 0.05</u>	< 0.05	RAJAY026 JA032-04H-P2 M-270206-01-1

SOYA BEAN (Variety) Location Year	Application				Residues (mg/kg) in soya bean seed			Report No Trial No. Doc No
	kg ai/ha	kg ai/hL	GS	DAT	JAU 6476 -sulfonic acid	JAU 6476- desthio	JAU 6476- total	
Soya (BT-383CR) USA Carlyle, Illinois 2004	0.15- 0.15	0.155- 0.161	GS79	21	< 0.05, < 0.05	<u>≤ 0.05</u>	< 0.05	RAJAY026 JA033-04H M-270206-01-1
Soya (RG 200 RR) USA Sabin, Minnesota 2005	0.148- 0.15	0.093- 0.099	GS69	19	< 0.05, < 0.05	<u>≤ 0.05</u>	< 0.05	RAJAY026 JA022-04HA M-270206-01-1

GS = growth stage at last application

Supervised trials in Brazil: Ten supervised trials have been conducted in 2012–2013 on soya bean in Brazil (Resende, 2013, F12-012 and Resende, 2013, F13-008). The trials were carried out according to the GAP for soya bean in Brazil (4 x 87.5 g ai/ha, PHI 30 days). Eight of the trials were decline trials and two were at harvest trials. Four foliar applications were made to each treated plot with an SC formulation containing 175 g/L prothioconazole + 150 g/L trifloxystrobin + 125 g/L bixafen at 87.5 g prothioconazole/ha with a 14 day interval between applications. The applications were made at growth stages BBCH 51-79.

The samples were frozen after collection and stored frozen (< -20°C) until extraction and analysis. The maximum period of storage was 213 days (7 months). Residues of prothioconazole-desthio and prothioconazole were determined by HPLC/MS/MS method 01013. The LOQ was 0.01 mg/kg for each analyte.

Table 9 Prothioconazole residues in soya bean resulting from supervised trials in Brazil

SOYA BEAN Trial year (Variety)	Application						Commodity	JAU 6476 mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No	days					
GAP, BRAZIL	175 g/L SC	87.5			4	30					
F12-012-01 Brazil, 2012 (TMG 7161 RR)	450 g/L SC ^a	81.7- 93.5	41-47	200	4	25 30 35	seed	< 0.01 < 0.01 < 0.01	0.02 <u>0.02</u> 0.01	0.03 0.03 0.02	Resende, 2013, F12-012
F12-012-02 Brazil, 2012 (Monsoy 7808 RR)	450 g/L SC ^a	87.5- 107.8	44-54	200	4	25 30 34	seed	< 0.01 < 0.01 < 0.01	0.09 <u>0.10</u> 0.04	0.10 0.11 0.05	Resende, 2013, F12-012
F12-012-03 Brazil, 2012 (M 7908)	450 g/L SC ^a	86.4- 95.8	43-48	200	4	25 30 35	seed	< 0.01 < 0.01 < 0.01	< 0.01 <u>< 0.01</u> < 0.01	< 0.02 < 0.02 < 0.02	Resende, 2013, F12-012
F12-012-04 Brazil, 2012 (NA 5909 RG)	450 g/L SC ^a	79.0- 97.5	40-49	200	4	30	seed	< 0.01	<u>0.01</u>	0.02	Resende, 2013, F12-012

SOYA BEAN Trial year (Variety)	Application					PHI days	Commodity	JAU 6476 mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
F12-012-05 Brazil, 2012 (M 7908)	450 g/L SC ^a	91.4- 99.5	44-50	200	4	30	seed	< 0.01	<u>0.01</u>	0.02	Resende, 2013, F12-012
F13-008-01 Brazil, 2013 (TMG 7161 RR)	450 g/L SC ^a	87.5- 92.4	44-46	200	4	15	seed	< 0.01	0.01	0.02	Resende, 2013, F13-008
						20		< 0.01	0.02	0.03	
						25		< 0.01	0.02	0.03	
						30		< 0.01	<u>0.03</u>	0.04	
						35		< 0.01	0.02	0.03	
F13-008-02 Brazil, 2013 (TMG 7161 RR)	450 g/L SC ^a	84.7- 89.2	42-45	200	4	15	seed	< 0.01	0.02	0.03	Resende, 2013, F13-008
						20		< 0.01	0.02	0.03	
						25		< 0.01	0.01	< 0.02	
						30		< 0.01	<u>< 0.01</u>	< 0.02	
						35		< 0.01	< 0.01	< 0.02	
F13-008-03 Brazil, 2013 (TMG 7161 RR)	450 g/L SC ^a	81.0- 91.5	41-46	200	4	15	seed	< 0.01	< 0.01	< 0.02	Resende, 2013, F13-008
						20		< 0.01	< 0.01	< 0.02	
						25		< 0.01	< 0.01	< 0.02	
						30		< 0.01	<u>< 0.01</u>	< 0.02	
						35		< 0.01	< 0.01	< 0.02	
F13-008-04 Brazil, 2013 (NA 5909)	450 g/L SC ^a	85.9- 90.7	43-45	200	4	15	seed	< 0.01	0.03	0.04	Resende, 2013, F13-008
						20		< 0.01	0.05	0.06	
						25		< 0.01	0.04	0.05	
						30		< 0.01	<u>0.04</u>	0.05	
						35		< 0.01	0.03	0.04	
F13-008-05 Brazil, 2013 (Monsoy 7905)	450 g/L SC ^a	87.5- 90.4	44-45	200	4	15	seed	< 0.01	0.01	0.02	Resende, 2013, F13-008
						20		< 0.01	0.03	0.04	
						25		< 0.01	0.02	0.03	
						30		< 0.01	<u>< 0.01</u>	< 0.02	
						35		< 0.01	< 0.01	< 0.02	

^a 450 g/L SC formulation containing 175 g/L prothioconazole + 150 g/L trifloxystrobin + 125 g/L bixafen

Root and tuber vegetables

Potato

Supervised trials in Europe (Belgium, UK, Netherlands, Germany, France, Spain and Italy): A total of twenty supervised trials were conducted in 2005 and 2010 on potato (Ref: Bomke & Bauer, 2011, 10-2085; Freitag & Wolters, 2007, RA-2569/05; Freitag & Wolters, 2007, RA-2604/05; Freitag & Wolters, 2007, RA-2570/05 and Freitag & Wolters, 2007, RA-2603/05). All trials were harvest trials.

In the study of Bomke & Bauer, (10-2085), an FS formulation containing 18 g/L prothioconazole and 100 g/L penflufen was applied as a seed treatment to potato tubers at 0.72 g prothioconazole/100 kg seed (equivalent to 36 g prothioconazole/ha). This is a higher application rate than the GAP in the EU (up to 0.64 g ai/100 kg seed, equivalent to 16 g ai/ha), but the trials demonstrate that residues in tubers at harvest are <LOQ, even at the higher application rate. Treated tubers for planting were collected after treatment (shown as 0 or -1 day DAT in the table below, where a 0 day corresponds to the day of planting treated tubers), and then tubers collected at maturity (90 day DAT). Residues of prothioconazole and prothioconazole-desthio were determined by HPLC/MS/MS using method 01013. The LOQ was 0.01 mg/kg for each analyte.

In the studies of Freitag & Wolters, (RA-2569/05) and Freitag & Wolters, (RA-2604/05) an FS formulation containing 8 g/L prothioconazole and 248 g/L penflufen was applied as a seed piece treatment using a roller table prior to planting at 0.60 g prothioconazole/100 kg seed pieces. Treated tubers for planting were collected after treatment (0 day), and then tubers collected at BBCH 43–48 (90–91 DAT) and at maturity/BBCH 49 (110–148 DAT). Residues of prothioconazole and prothioconazole-desthio were determined by HPLC/MS/MS using method 00598/M001. The LOQ was 0.01 mg/kg for each analyte

In the studies of Freitag & Wolters, (RA-2570/05) and Freitag & Wolters (RA-2603/05), an FS formulation containing 8 g/L prothioconazole and 248 g/L penflufen was applied to potato tubers at planting using an in-furrow method at 30.1–32.0 g prothioconazole/ha. Treated tubers were collected after treatment (0 day), then at BBCH 43–48 (90–91 DAT) and at maturity/BBCH 49 (110–148 DAT). Residues of prothioconazole and prothioconazole-desthio were determined by HPLC/MS/MS using method 00598/M001. The LOQ was 0.01 mg/kg for each analyte.

The samples were frozen after collection and stored frozen (< -20 °C) until extraction and analysis. The maximum period of storage was 318 days (10.6 months).

Table 10 Prothioconazole residues in potato resulting from supervised trials in Europe

POTATO Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 mg/kg	JAU 6476 desthio mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No					
GAP, EU	8 g/L FS	12-16	-	-	1	F - The PHI is covered by the vegetation time between application and harvest, no PHI is set.				
10-2085-01 UK, 2010 (Nicola)	118 g/L FS ^a	36 (0.72 g ai/ 100 kg seed)	-	-	1	-1	Tuber for planting	0.89	0.57	Bomke & Bauer, 2011, 10-2085
						90	Tuber		<u>≤ 0.01</u>	
10-2085-02 France, 2010 (Nicola)	118 g/L FS ^a	36 (0.72 g ai/ 100 kg seed)	-	-	1	0	Tuber for planting	1.21	0.97	Bomke & Bauer, 2011, 10-2085
						90	Tuber		<u>≤ 0.01</u>	
10-2085-03 Belgium, 2010 (Nicola)	118 g/L FS ^a	36 (0.72 g ai/ 100 kg seed)	-	-	1	0	Tuber for planting	1.17	1.07	Bomke & Bauer, 2011, 10-2085
						90	Tuber		<u>≤ 0.01</u>	
10-2085-04 Netherlands, 2010 (Nicola)	118 g/L FS ^a	36 (0.72 g ai/ 100 kg seed)	-	-	1	0	Tuber for planting	1.34	0.78	Bomke & Bauer, 2011, 10-2085
						90	Tuber		<u>≤ 0.01</u>	
R 2005 0466/7 Netherlands, 2005 (Bintje)	258 g/L FS ^b	8.4 (0.60 g ai/ 100 kg seed)	3000	-	1	0	Tuber for planting	0.52	0.95	Freitag & Wolters, 2007, RA- 2569/05
						90 124	Tuber Tuber	< 0.01 < 0.01	<u>≤ 0.01</u> < 0.01	
R 2005 0873/5 UK, 2005 (Bintje)	258 g/L FS ^b	7.2 (0.60 g ai/ 100 kg seed)	3000	-	1	0	Tuber for planting	0.74	0.33	Freitag & Wolters, 2007, RA- 2569/05
						91 133	Tuber Tuber	< 0.01 < 0.01	<u>≤ 0.01</u> < 0.01	
R 2005 0874/3	258 g/L FS ^b	5.6 (0.60 g ai/	3000	-	1	0	Tuber for planting	0.38	1.20	Freitag & Wolters,

POTATO Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 mg/kg	JAU 6476 desthio mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No					
Germany, 2005 (Bintje)		100 kg seed)				90 136	Tuber Tuber	< 0.01 < 0.01	<u>< 0.01</u> < 0.01	2007, RA- 2569/05
R 2005 0875/1 France, 2005 (Bintje)	258 g/L FS ^b	4.8 (0.60 g ai/ 100 kg seed)	3000	-	1	0	Tuber for planting	0.28	0.97	Freitag & Wolters, 2007, RA- 2569/05
						90 148	Tuber Tuber	< 0.01 < 0.01	<u>< 0.01</u> < 0.01	
R 2005 0881/6 France, 2005 (Bintje)	258 g/L FS ^b	4.0 (0.60 g ai/ 100 kg seed)	3000	-	1	0	Tuber for planting	0.20	0.79	Freitag & Wolters, 2007, RA- 2604/05
						90 122	Tuber Tuber	< 0.01 < 0.01	<u>< 0.01</u> < 0.01	
R 2005 0882/4 Italy, 2005 (Bintje)	258 g/L FS ^b	10.8 (0.60 g ai/ 100 kg seed)	3000	-	1	0	Tuber for planting	0.70	0.48	Freitag & Wolters, 2007, RA- 2604/05
						90 118	Tuber Tuber	< 0.01 < 0.01	<u>< 0.01</u> < 0.01	
R 2005 0883/2 France, 2005 (Bintje)	258 g/L FS ^b	5.7 (0.60 g ai/ 100 kg seed)	3000	-	1	0	Tuber for planting	1.10	0.47	Freitag & Wolters, 2007, RA- 2604/05
						90 110	Tuber Tuber	< 0.01 < 0.01	<u>< 0.01</u> < 0.01	
R 2005 0884/0 Spain, 2005 (Bintje)	258 g/L FS ^b	10.8 (0.60 g ai/ 100 kg seed)	3000	-	1	0	Tuber for planting	0.74	0.34	Freitag & Wolters, 2007, RA- 2604/05
						91 128	Tuber Tuber	< 0.01 < 0.01	<u>< 0.01</u> < 0.01	
R 2005 0467/5 France, 2005 (Bintje)	258 g/L FS ^b	32.0	16.0	200	1	0	Tuber, treated	0.16	0.13	Freitag & Wolters, 2007, RA- 2570/05
						90 110	Tuber Tuber	< 0.01 < 0.01	<u>< 0.01</u> < 0.01	
R 2005 0877/8 Italy, 2005 (Bintje)	258 g/L FS ^b	32.0	10.7	300	1	0	Tuber, treated	0.11	0.11	Freitag & Wolters, 2007, RA- 2570/05
						90 118	Tuber Tuber	< 0.01 < 0.01	<u>< 0.01</u> < 0.01	
R 2005 0878/6 France, 2005 (Bintje)	258 g/L FS ^b	32.0	10.7	300	1	0	Tuber, treated	0.04	0.05	Freitag & Wolters, 2007, RA- 2570/05
						90 122	Tuber Tuber	< 0.01 < 0.01	<u>< 0.01</u> < 0.01	
R 2005 0879/4 Spain, 2005 (Bintje)	258 g/L FS ^b	32.0	10.7	300	1	0	Tuber, treated	0.03	0.03	Freitag & Wolters, 2007, RA- 2570/05
						91 128	Tuber Tuber	< 0.01 < 0.01	<u>< 0.01</u> < 0.01	
R 2005 0880/8	258 g/L FS ^b	32.0	16.0	200	1	0	Tuber, treated	0.09	0.06	Freitag & Wolters,

POTATO Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 mg/kg	JAU 6476 desthio mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No					
Germany, 2005 (Bintje)						90 116	Tuber Tuber	< 0.01 < 0.01	≤ 0.01 < 0.01	2007, RA- 2603/05
R 2005 0885/9 France, 2005 (Bintje)	258 g/L FS ^b	32.0	16.0	200	1	0	Tuber, treated	0.06	0.09	Freitag & Wolters, 2007, RA- 2603/05
						90 148	Tuber Tuber	< 0.01 < 0.01	≤ 0.01 < 0.01	
R 2005 0886/7 Germany, 2005 (Bintje)	258 g/L FS ^b	32.0	16.0	200	1	0	Tuber, treated	0.06	0.05	Freitag & Wolters, 2007, RA- 2603/05
						90 136	Tuber Tuber	< 0.01 < 0.01	≤ 0.01 < 0.01	
R 2005 0887/5 Netherlands, 2005 (Bintje)	258 g/L FS ^b	32.0	16.0	200	1	0	Tuber, treated	0.03	0.04	Freitag & Wolters, 2007, RA- 2603/05
						90 124	Tuber Tuber	< 0.01 < 0.01	≤ 0.01 < 0.01	

^a 118 g/L FS formulation containing 18 g/L prothioconazole + 100 g/L penflufen.

^b 258 g/L SE formulation containing 8 g/L prothioconazole + 248 g/L pencycuron.

Cereal grains

Maize (field corn) and popcorn

Supervised trials in the USA: Twenty supervised trials were conducted in 2006 on field corn and three trials on popcorn in the USA (Krolski & Harbin, 2008, RAJAP004). The trials were carried out according to the GAP for corn in the USA. Four foliar applications were made to each treated plot with an SC formulation containing 480 g/L prothioconazole with 2–11-day intervals between the application. Each spray included a non-ionic surfactant.

In the field corn trials, grain was harvested at 14 days (BBCH 89) after last application. In the decline trials, grain samples were collected at PHIs of 0, 7, 13–14, 20–21 and 27–28 days.

In the popcorn trials, grain was sampled at harvest at 14 DAT (BBCH 89).

The samples were frozen after collection and stored frozen (< -20 °C) until extraction and analysis. The maximum period of storage was 365 days (12 months). Residues were determined by high performance liquid chromatography – triple stage quadrupole mass spectrometry (LC/MS/MS) with LOQ of 0.02 mg/kg equivalent to 0.0018 mg/kg prothioconazole desthio.

Table 11 Prothioconazole residues in field corn (maize) resulting from supervised trials in the USA

FIELD CORN (MAIZE) Trial year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
GAP, USA	480 g/L SC	200			4	0/14					
JA004-06HA Pennsylvania, 2006 (TA5750)	480 g/L SC	203- 210	111- 112	181- 197	4	14 14	Grain	< 0.02	≤ 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004

FIELD CORN (MAIZE) Trial year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
JA006-06HA Georgia, USA, 2006 (31N26)	480 g/L SC	200- 201	142- 160	125- 141	4	14	Grain	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA013-06DA Iowa, USA, 2006 (33N30LL)	480 g/L SC	192- 202	160- 178	115- 120	4	0	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004
						7	Grain	< 0.02	< 0.018	< 0.02	
						13	Grain	< 0.02	<u>< 0.018</u>	< 0.02	
						20	Grain	< 0.02	< 0.018	< 0.02	
						27	Grain	< 0.02	< 0.018	< 0.02	
JA014-06DA Minnesota, USA, 2006 (Pioneer 39H85)	480 g/L SC	196- 201	110- 119	169- 178	4	0	Grain	< 0.02	0.04	0.05	Krolski & Harbin, 2008, RAJAP004
						7	Grain	< 0.02	0.05	0.06	
						14	Grain	< 0.02	<u>0.05</u>	0.06	
						21	Grain	< 0.02	0.03	0.03	
						28	Grain	< 0.02	0.05	0.07	
JA015-06HA Kansas, USA, 2006 (Garst 8881RR)	480 g/L SC	195- 202	149- 150	130- 136	4	14	Grain	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA016-06HA Illinois, USA, 2006 (Garst 8568 CB/LL)	480 g/L SC	193- 209	116- 143	145- 175	4	13	Grain	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA017-06HA Nebraska, USA, 2006 (NK38B4)	480 g/L SC	197- 202	151- 154	130- 132	4	11	Grain	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA018-06HA Iowa, USA, 2006 (NK65C5)	480 g/L SC	200- 202	151- 154	130- 132	4	12	Grain	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA019-06HA Missouri, USA, 2006 (34B20)	480 g/L SC	200- 205	112- 118	170- 178	4	14	Grain	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA020-06HA North Dakota, USA, 2006 (DKC35-51)	480 g/L SC	197- 205	107- 131	156- 184	4	14	Grain	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA021-06HA North Dakota, USA, 2006 (Pioneer 39D81)	480 g/L SC	193- 200	129- 142	136- 154	4	14	Grain	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA022-06HA Iowa, USA, 2006 (FA6508)	480 g/L SC	204- 205	126- 143	143- 163	4	14	Grain	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004

FIELD CORN (MAIZE) Trial year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
JA023-06HA Illinois, USA, 2006 (B-T 6516 RR2YG)	480 g/L SC	198- 203	110- 120	167- 184	4	14	Grain	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA024-06HA Minnesota, USA, 2006 (38G16)	480 g/L SC	196- 202	113- 114	172- 177	4	14	Grain	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA025-06HA Nebraska, USA, 2006 (Pioneer 34N45YG/RR)	480 g/L SC	200- 202	107- 108	185- 188	4	14	Grain	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA026-06HA Indiana, USA, 2006 (DKC 57-59 RR/plus)	480 g/L SC	197- 202	109- 120	165- 182	4	14	Grain	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA027-06HA Iowa, USA, 2006 (Pioneer 34N50)	480 g/L SC	200- 204	108- 168	122- 184	4	14	Grain	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA028-06HA Ohio, USA, 2006 (Crows 7R154)	480 g/L SC	201- 205	133- 136	149- 153	4	14	Grain	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA029-06HA Minnesota, USA, 2006 (Pioneer 38H66)	480 g/L SC	195- 203	123- 125	156- 163	4	14	Grain	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA030-06HA Texas, USA, 2006 (Pioneer 31G97)	480 g/L SC	193- 201	146- 153	127- 137	4	14	Grain	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004

Table 12 Prothioconazole residues in popcorn resulting from supervised trials in the USA

POPCORN Trial Country, year (Variety)	Application					PHI days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (l/ha)	No						
GAP, USA	480 g/L SC	200			4	0/14					
JA034-06HA Kansas, USA, 2006 (Yellow)	480 g/L SC	195- 206	142- 145	136- 143	4	14	Grain	< 0.02	<u>< 0.018</u>	< 0.02	Krolski & Harbin, 2008, RAJAP004

POPCORN Trial Country, year (Variety)	Application					PHI days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (l/ha)	No						
JA035-06HA Illinois, USA, 2006 (M2101)	480 g/L SC	197- 207	115- 143	145- 174	4	12 12	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA036-06HA Nebraska, USA, 2006 (AP2501)	480 g/L SC	198- 200	153- 155	128- 130	4	12 12	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004

Supervised trials in Brazil: Ten supervised trials were conducted in 2011-2012 on corn in Brazil matching the GAP for corn in Brazil.

In the study of Santiago (F11-034) two foliar applications were made to each treated plot with an SC formulation containing 175 g/L prothioconazole + 150 g/L trifloxystrobin at 87.5 g prothioconazole/ha with a 15-day interval between applications. The applications were made at growth stages BBCH 55–67 and BBCH 60–71.

In the study of Resende (F12-011) two foliar applications were made to each treated plot with an SC formulation containing 175 g/L prothioconazole + 125 g/L bixafen + 150 g/L trifloxystrobin at 87.5 g prothioconazole/ha with a 14-day interval between applications. The applications were made at growth stages BBCH 55-61 and BBCH 65-71.

In the decline trials, seeds were harvested at 10, 14–15 and 19–20 days (BBCH 73–79, 75–79 and 75–79) after last treatment. In the at harvest trials, seeds were harvested at 14–15 days DAT (BBCH 75–79). The samples were frozen after collection and stored frozen (< -20 °C) until extraction and analysis. The maximum period of storage was 88 days (3 months).

Residues of prothioconazole-desthio and prothioconazole were determined by HPLC/MS/MS using method 01013 with the chromatographic conditions described in method 00765. The LOQ was 0.01 mg/kg for each analyte. Procedural recoveries at fortification levels of 0.01–1.0 mg/kg were within the acceptable range of 70–120%, RSD <20%, for prothioconazole and prothioconazole-desthio.

Table 13 Prothioconazole residues in corn (maize) resulting from supervised trials in Brazil

FIELD CORN Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
GAP, BRAZIL	175 g/L SC	87.5			2	15					
F11-034-01 Brazil, 2011-2012 (Pioneer 30F35H)	325 g/L SC ^a	87.5	44	200	2	10 15 20	seed	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	Santiago, 2012, F11-034
F11-034-02 Brazil, 2011-2012 (Maximus TL)	325 g/L SC ^a	87.5- 88.9	44-45	200	2	10 15 20	seed	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	Santiago, 2012, F11-034

FIELD CORN Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
F11-034-03 Brazil, 2012 (DKB 350)	325 g/L SC ^a	86.5- 89.4	43-45	200	2	10 15 20	seed	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	Santiago, 2012, F11-034
F11-034-04 Brazil, 2012 (Pioneer 30F53)	325 g/L SC ^a	93.3- 96.3	47-48	200	2	15	seed	< 0.01	< 0.01	< 0.02	Santiago, 2012, F11-034
F11-034-05 Brazil, 2012 (DKB 350)	325 g/L SC ^a	94.2- 95.2	47-48	200	2	15	seed	< 0.01	< 0.01	< 0.02	Santiago, 2012, F11-034
F12-011-01 Brazil, 2012 (DKB 350)	450 g/L SC ^b	77.6- 101.7	39-51	200	2	10 14 19	seed	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	Resende, 2013, F12-011
F12-011-02 Brazil, 2012 (Pioneer 30F53)	450 g/L SC ^b	93.5- 97.5	47-49	200	2	10 14 20	seed	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	Resende, 2013, F12-011
F12-011-03 Brazil, 2012 (Maximum TL)	450 g/L SC ^b	95.0- 102.8	48-51	200	2	10 15 20	seed	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	Resende, 2013, F12-011
F12-011-04 Brazil, 2012 (P30F35)	450 g/L SC ^b	87.5- 99.8	44-50	200	2	14	seed	< 0.01	< 0.01	< 0.02	Resende, 2013, F12-011
F12-011-05 Brazil, 2012 (DKB 350)	450 g/L SC ^b	84.7- 102.7	42-51	200	2	14	seed	< 0.01	< 0.01	< 0.02	Resende, 2013, F12-011

^a 325 g/L SC formulation containing 175 g/L prothioconazole + 150 g/L trifloxystrobin

^b 450 g/L SC formulation containing 175 g/L prothioconazole + 125 g/L bixafen + 150 g/L trifloxystrobin

Supervised trials in Europe (Belgium, UK, Netherlands, Germany, France, Spain, Italy, Portugal and Greece): A total of twenty supervised trials were conducted in 2007, 2008 and 2011 on maize/corn in Europe. All trials were decline trials.

In the first set of studies (Freitag & Reineke, 2009, 08-2035 and Freitag & Reineke, 2009, 08-2036) an EC formulation containing 125 g/L prothioconazole and 125 g/L tebuconazole was applied twice to maize at 125 g prothioconazole/ha, with an interval of 23–54 days between applications. The applications were made at growth stages BBCH 33–34 and BBCH 69. Immature (milk ripe) seeds were collected at BBCH 75–79 (8–26 DAT), ear without husk was collected at BBCH 85 (28–56 day PHI) and mature seeds at BBCH 89 (PHI 52–83 DAT). Residues of prothioconazole-desthio were determined by HPLC/MS/MS using method 01013. The LOQ was 0.01 mg/kg. Procedural recoveries at fortification levels of 0.01–3.0 mg/kg were within the acceptable range of 70–120%, RSD < 20%, for prothioconazole-desthio in each matrix.

In the second study (Freitag, 2008, RA-2675/07), an EC formulation containing 250 g/L prothioconazole was applied twice to maize at 200 g prothioconazole/ha, with an interval of 23–43 days between applications. The applications were made at growth stages BBCH 33 and BBCH 69. Ear without husk was collected at BBCH 85 (23–46 DAT) and mature seeds at BBCH 89 (56–69 DAT). Residues of prothioconazole-desthio were determined by HPLC/MS/MS using method 01013 (LOQ was 0.01 mg/kg). Procedural recoveries at fortification levels of 0.01–2.0 mg/kg were within the acceptable range of 70–120%, RSD < 20%, for prothioconazole-desthio in each matrix.

In the other studies (Bomke & Teubner, 2013, 11-2109 and Bomke, 2012, 11-2110) an EC formulation containing 125 g/L prothioconazole and 125 g/L fluopyram was applied twice to maize at 130 g prothioconazole/ha, with an interval of 13–16 days between applications. The applications were made at growth stages BBCH 51–65 and BBCH 67–71. Immature (milk ripe) seeds were collected at BBCH 79 (14–31 DAT), ear without husk was collected at BBCH 79 (14–31 DAT) and BBCH 85 (28–56 DAT) and mature seeds and rest of plant at BBCH 89 (48–82 DAT). Residues of prothioconazole-desthio were determined by HPLC/MS/MS using method 01013. The LOQ was 0.01 mg/kg. Procedural recoveries at fortification levels of 0.01–1.0 mg/kg were in the range of 70–120%, RSD < 20%, for prothioconazole-desthio in each matrix.

The samples were frozen after collection and stored frozen (< -20 °C) until extraction and analysis. The maximum period of storage was 406 days (13.3 months).

Table 14 Prothioconazole residues in maize/corn resulting from supervised trials in Europe

MAIZE Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 desthio mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No				
GAP, EU	250 g/L EC	125			2	-			
08-2035-01 UK, 2008 (Cixxom)	250 g/L EC ^a	125	63	200	2	19	seed, immature (milk ripe)	< 0.01	Freitag & Reineke, 2009, 08-2035
						56	ear w/o husk	< 0.01	
						69	seed	< 0.01	
08-2035-02 Germany, 2008 (Justina)	250 g/L EC ^a	125	42	300	2	24	seed, immature (milk ripe)	< 0.01	Freitag & Reineke, 2009, 08-2035
						49	ear w/o husk	< 0.01	
						64	seed	< 0.01	
08-2035-03 Germany, 2008 (Seleno)	250 g/L EC ^a	125	32-42	300- 400	2	26	seed, immature (milk ripe)	< 0.01	Freitag & Reineke, 2009, 08-2035
						37	ear w/o husk	< 0.01	
						57	seed	< 0.01	
08-2035-04 Netherlands, 2008 (Rosalie)	250 g/L EC ^a	125	32	400	2	18	seed, immature (milk ripe)	< 0.01	Freitag & Reineke, 2009, 08-2035
						40	ear w/o husk	< 0.01	
08-2036-01 France, 2008 (PR33Y74)	250 g/L EC ^a	125	42	300	2	11	seed, immature (milk ripe)	< 0.01	Freitag & Reineke, 2009, 08-2036
						34	ear w/o husk	< 0.01	
						83	seed	< 0.01	
08-2036-02 Spain, 2008 (DKC6451YG)	250 g/L EC ^a	125-132	42	300- 316	2	19	seed, immature (milk ripe)	< 0.01	Freitag & Reineke, 2009, 08-2036
						28	ear w/o husk	< 0.01	
						53	seed	< 0.01	

MAIZE Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 desthio mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No				
08-2036-03 Italy, 2008 (PR33A46)	250 g/L EC ^a	125	31	400	2	13	seed, immature (milk ripe)	< 0.01	Freitag & Reineke, 2009, 08-2036
						34	ear w/o husk	< 0.01	
						55	seed	< 0.01	
08-2036-04 Portugal, 2008 (A46)	250 g/L EC ^a	125	25-42	300- 500	2	8	seed, immature (milk ripe)	< 0.01	Freitag & Reineke, 2009, 08-2036
						30	ear w/o husk	< 0.01	
						52	seed	< 0.01	
R 2007 0829/7 Germany, 2007 (Nescio)	250 g/L EC	200	67	300	2	29 43	ear w/o husk	< 0.01 < 0.01	Freitag, 2008, RA-2675/07
						56	seed	< 0.01	
R 2007 0837/8 Germany, 2007 (DK3745)	250 g/L EC	200	67	300	2	19 46	ear w/o husk	< 0.01 < 0.01	Freitag, 2008, RA-2675/07
						68	seed	< 0.01	
R 2007 0838/6 Spain, 2007 (PR 31G98)	250 g/L EC	186-200	50	372- 400	2	16 23	ear w/o husk	< 0.01 < 0.01	Freitag, 2008, RA-2675/07
						64	seed	< 0.01	
R 2007 0839/4 Portugal, 2007 (PR33N09)	250 g/L EC	200	67	300	2	15 33	ear w/o husk	< 0.01 < 0.01	Freitag, 2008, RA-2675/07
						69	seed	< 0.01	
11-2109-01 France, 2011 (Cobalt)	250 g/L SE ^b	125	42	300	2	20	seed, immature	< 0.01	Bomke & Teubner, 2013, 11-2109
						20 40	ear without husk	< 0.01 < 0.01	
						82	seed	< 0.01	
11-2109-02 Germany, 2011 (Saludo)	250 g/L SE ^b	125	42	300	2	31	seed, immature	< 0.01	Bomke & Teubner, 2013, 11-2109
						31 51	ear without husk	< 0.01 < 0.01	
						65	seed	< 0.01	
11-2109-03 UK 2011 (Cougar)	250 g/L SE ^b	125	50	250	2	31	seed, immature	< 0.01	Bomke & Teubner, 2013, 11-2109
						31 49	ear without husk	< 0.01 < 0.01	
						60	seed	< 0.01	
11-2109-04 Belgium, 2011 (Delitop)	250 g/L SE ^b	125	45	275	2	29	seed, immature	< 0.01	Bomke & Teubner, 2013, 11-2109
						29 42	ear without husk	< 0.01 < 0.01	
						64	seed	< 0.01	
11-2110-02	250 g/L SE ^b	125	31	400	2	17	seed, immature	< 0.01	Bomke, 2012,

MAIZE Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 desthio mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No				
Italy, 2011 (Constanza)						17 29	ear without husk	< 0.01 < 0.01	11-2110
						62	seed	< 0.01	
11-2110-03 Spain, 2011 (DKC66 67YG)	250 g/L SE ^b	125	42	300	2	20	ear without husk	< 0.01	Bomke, 2012, 11-2110
						69	seed	< 0.01	
11-2110-04 Greece, 2011 (Dekalp, 5276)	250 g/L SE ^b	125	31	400	2	18	seed, immature	< 0.01	Bomke, 2012, 11-2110
						18 31	ear without husk	< 0.01 < 0.01	
						48	seed	< 0.01	
11-2110-05 France, 2011 (PR 33 A46)	250 g/L SE ^b	125	42	300	2	14	seed, immature	< 0.01	Bomke, 2012, 11-2110
						14 29	ear without husk	< 0.01 < 0.01	
						64	seed	< 0.01	

ear w/o husk = ear (Seeds plus cob) without husk

^a 250 g/L EC formulation containing 125 g/L prothioconazole + 125 g/L tebuconazole

^b 250 g/L SE formulation containing 125 g/L prothioconazole + 125 g/L AE C656948 (fluopyram)

Oilseeds

Peanuts

Eight supervised trials were conducted in 2011–2012 on peanut in Australia (Ellis, 2012, BCS-0232 and Ellis, 2012, BCS-0383). Four trials included plots which were treated according to the GAP for peanut in Australia (4 times 120–192 g ai/ha at 10–14 days apart, 28-day PHI), and four trials included plots which were treated at total application rates within 25% of the GAP rate, but shorter PHI (up to 21 days). These trials are submitted in support of the use on peanuts as they show that residues in peanut seeds are <LOQ at all sampling intervals. All of the trials were decline trials.

In study of Ellis, (BCS-0232), 3, 3 or 5 foliar applications were made to each treated plot with an SC formulation containing 480 g/L prothioconazole at nominally 180 or 120 g ai/ha with a 10 day interval between applications. Seeds were harvested immediately before the last application and after 0, 7, 14–15 and 20–21 days. For the plot treated at 5 × 120 g ai/ha samples of seeds were collected after field drying following pulling at 7 DAT.

In another study of Ellis, (BSC-0383), each trial consisted of up to 7 treated plots. Plots were treated with an SC formulation containing 480 g/L prothioconazole. One plot received an in-furrow treatment at 192 g ai/ha at planting, and the plants lifted 117–167 DAT. The remaining plots received 3 or 4 foliar applications at 192 or 288 g ai/ha, or an in-furrow application followed by 3 or 4 foliar applications at 192 or 288 g ai/ha, and the plants lifted at 14–15, 21–22 and 27–29 DAT. In furrow applications were made at growth stage BBCH 00, and foliar applications were made at growth stages BBCH 59–89. For all plots, plants were left to dry for 7–13 days before samples of seeds were collected.

Residues of prothioconazole-desthio and prothioconazole were determined by HPLC/MS/MS using method ATM-0053. The LOQ was 0.01 mg/kg for each analyte and matrix in study BCS-0232, and 0.01 mg/kg for each analyte in seeds and shells and 0.05 mg/kg for each analyte in forage in study

BCD-0383. Procedural recoveries at fortification levels of 0.01 (or 0.05)–1.0 mg/kg were within the acceptable range of 70–120%, RSD < 20%, for prothioconazole and prothioconazole-desthio.

The samples were frozen after collection and stored frozen (< -20 °C) until extraction and analysis. The maximum period of storage was 612 days (20 months), which falls within the confirmed stability period of 3 years.

In peanut seeds, residue levels of prothioconazole-desthio were < 0.01 mg/kg, residues of prothioconazole were < 0.01 mg/kg and residues of total prothioconazole (sum of prothioconazole-desthio and prothioconazole, expressed as prothioconazole) were < 0.02 mg/kg at all sampling intervals in all trials.

The results of the trials relevant to the Australian GAP are summarized in Table 15.

Table 15 Prothioconazole residues in peanut resulting from supervised trials in Australia

PEANUTS	Application					DAT	Commodity	JAU 6476	JAU 6476 desthio	JAU 6476 total	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No	days		mg/kg	mg/kg	mg/kg	
GAP, AUSTRALIA	480 g/L SC	192			4	28					
C351 Australia, 2009 (SO05R)	480 g/L SC	176- 177	79-80	221- 223	4	-0	seed	< 0.01	< 0.01	< 0.02	Ellis, 2012, BCS-0232
						0		< 0.01	< 0.01	< 0.02	
						7		< 0.01	< 0.01	< 0.02	
						14		< 0.01	< 0.01	< 0.02	
						21		< 0.01	< 0.01	< 0.02	
		176- 177	74-80	221- 239	5	-0	seed	< 0.01	< 0.01	< 0.02	
						0		< 0.01	< 0.01	< 0.02	
						7		< 0.01	< 0.01	< 0.02	
						14		< 0.01	< 0.01	< 0.02	
						21		< 0.01	< 0.01	< 0.02	
C352 Australia, 2009 (Menzies)	480 g/L SC	175- 178	73-79	223- 239	4	-0	seed	< 0.01	< 0.01	< 0.02	Ellis, 2012, BCS-0232
						0		< 0.01	< 0.01	< 0.02	
						7		< 0.01	< 0.01	< 0.02	
						14		< 0.01	< 0.01	< 0.02	
						21		< 0.01	< 0.01	< 0.02	
		175- 178	73-79	223- 239	5	-0	seed	< 0.01	< 0.01	< 0.02	
						0		< 0.01	< 0.01	< 0.02	
						7		< 0.01	< 0.01	< 0.02	
						14		< 0.01	< 0.01	< 0.02	
						21		< 0.01	< 0.01	< 0.02	
C353 Australia, 2009 (Holt)	480 g/L SC	177- 178	50-53	334- 358	4	-0	seed	< 0.01	< 0.01	< 0.02	Ellis, 2012, BCS-0232
						0		< 0.01	< 0.01	< 0.02	
						7		< 0.01	< 0.01	< 0.02	
						14		< 0.01	< 0.01	< 0.02	
						21		< 0.01	< 0.01	< 0.02	
		177- 178	50-54	328- 358	5	-0	seed	< 0.01	< 0.01	< 0.02	
						0		< 0.01	< 0.01	< 0.02	
						7		< 0.01	< 0.01	< 0.02	
						14		< 0.01	< 0.01	< 0.02	
						21		< 0.01	< 0.01	< 0.02	

days. Green material was collected prior to the final application (shown as -0 day in the table) and immediately after the final application (0 DAT), and then at grows stages ranging from BBCH 33 to BBCH 85.

Table 16 Prothioconazole residues in sweet corn forage and fodder from US trials

SWEET CORN Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
GAP, USA	480 g/L SC	100			4	0/14					
JA002-06HA Virginia, USA, 2006 (Devotion)	480 g/L SC	201- 208	128	156- 163	4	0	Forage	0.95	<u>4.08</u>	5.45	Krolski & Harbin, 2008, RAJAP004
						6	Forage	0.14	1.52	1.82	
						14	Stover	0.09	<u>0.82</u>	0.99	
JA003-06HA New York, USA, 2006 (Gourmet Sweet 372A)	480 g/L SC	204- 208	122- 123	167- 170	4	0	Forage	0.63	<u>3.20</u>	4.15	Krolski & Harbin, 2008, RAJAP004
						7	Forage	0.21	1.40	1.75	
						14	Stover	0.11	<u>0.71</u>	0.89	
JA005-06HA Georgia, USA, 2006 (Sweet G-90)	480 g/L SC	200	142- 159	125- 141	4	0	Forage	1.09	<u>3.75</u>	5.22	Krolski & Harbin, 2008, RAJAP004
						7	Forage	0.54	1.70	2.42	
						14	Stover	0.37	<u>1.45</u>	1.96	
JA007-06DA Florida, USA, 2006 (Silver Queen)	480 g/L SC	195- 200	167- 181	109- 120	4	0	Forage	0.46	<u>2.08</u>	2.76	Krolski & Harbin, 2008, RAJAP004
						3	Forage	0.33	1.56	2.08	
						6	Forage	0.18	0.75	1.00	
						10	Forage	0.19	0.98	1.28	
						14	Forage	0.17	0.80	1.06	
						10	Stover	0.30	1.38	1.82	
						14	Stover	0.39	<u>1.70</u>	2.26	
						20	Stover	0.28	1.48	1.91	
						28	Stover	0.37	1.47	1.99	
						34	Stover	0.35	1.27	1.75	
JA008-06HA Missouri, USA, 2006 (Peaches & Cream)	480 g/L SC	200- 204	115- 149	137- 174	4	0	Forage	0.51		3.13	Krolski & Harbin, 2008, RAJAP004
						6	Forage	0.09		0.93	
						13	Stover	0.14		1.99	
JA009-06HA Illinois, USA, 2006 (BC 0805)	480 g/L SC	196- 204	150- 160	123- 132	4	0	Forage	0.34		2.00	Krolski & Harbin, 2008, RAJAP004
						7	Forage	0.04		0.43	
						14	Stover	0.09		0.83	
JA010-06HA Nebraska, USA, 2006 (Serendipity)	480 g/L SC	199- 204	153- 157	129 131	4	0	Forage	0.43	<u>1.89</u>	2.51	Krolski & Harbin, 2008, RAJAP004
						5	Forage	0.12	0.83	1.04	
						13	Stover	0.14	<u>0.98</u>	1.21	
JA011-06HA Minnesota, USA, 2006 (39H85 Pioneer)	480 g/L SC	196- 202	111- 118	171- 181	4	0	Forage	0.56	<u>2.12</u>	2.95	Krolski & Harbin, 2008, RAJAP004
						7	Forage	0.29	1.43	1.86	
						14	Stover	0.93	<u>5.88</u>	7.41	

SWEET CORN Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
JA012-06HA Iowa, USA, 2006 (Mirai 131)	480 g/L SC	198- 206	156- 182		4	0	Forage	0.45	<u>1.98</u>	2.63	Krolski & Harbin, 2008, RAJAP004
						7	Forage	0.07	0.37	0.47	
						12	Stover	0.16	<u>0.90</u>	1.15	
JA031-06HA California, USA, 2006 (Silver Queen)	480 g/L SC	197- 201	117- 119	168- 170	4	0	Forage	0.56	<u>2.82</u>	3.67	Krolski & Harbin, 2008, RAJAP004
						7	Forage	0.34	2.19	2.76	
						12	Stover	0.52	<u>3.61</u>	4.50	
JA032-06HA Washington, USA, 2006 (Golden Jubilee)	480 g/L SC	202- 204	143- 145	140- 142	4	0	Forage	0.39	<u>1.94</u>	2.53	Krolski & Harbin, 2008, RAJAP004
						7	Forage	0.04	0.57	0.67	
						12	Stover	0.08	<u>1.68</u>	1.93	
JA033-06HA Oregon, USA, 2006 (Serendipity)	480 g/L SC	195- 205	120- 126	163- 165	4	0	Forage	< 0.24	<u>1.31</u>	1.69	Krolski & Harbin, 2008, RAJAP004
						5	Forage	0.06	0.65	0.78	
						13	Stover	0.04	<u>0.67</u>	0.78	

Table 17 Prothioconazole residues in field corn (maize) resulting from supervised trials in the USA

FIELD CORN (MAIZE) Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
GAP, USA	480 g/L SC	200			4	0/14					
JA004-06HA Pennsylvania, USA, 2006 (TA5750)	480 g/L SC	203- 210	111- 112	181- 197	4	14	Stover	0.26	<u>2.22</u>	2.71	Krolski & Harbin, 2008, RAJAP004
		208- 214	112	185- 191	4	0 6	Forage Forage	0.50 0.14	<u>1.90</u> 1.06	2.59 1.30	
JA006-06HA Georgia, USA, 2006 (31N26)	480 g/L SC	200- 201	142- 160	125- 141	4	14	Stover	0.85	<u>4.05</u>	5.31	Krolski & Harbin, 2008, RAJAP004
		200	141- 159	125- 141	4	0 7	Forage Forage	0.85 0.27	<u>3.44</u> 1.82	4.64 2.27	
JA013-06DA Iowa, USA, 2006 (33N30LL)	480 g/L SC	192- 202	160- 178	115- 120	4	0	Stover	0.97	4.39	5.81	Krolski & Harbin, 2008, RAJAP004
						7	Stover	0.19	1.16	1.47	
						13	Stover	0.32	2.22	2.76	
						20	Stover	< 0.02	0.05	0.06	
						27	Stover	0.37	<u>2.42</u>	3.04	
		195- 205	160- 176	115- 124	4	0	Forage	0.46	<u>2.20</u>	2.89	
						7	Forage	0.15	1.08	1.34	
						14	Forage	0.14	1.04	1.28	
						22	Forage	0.10	0.65	0.81	
						27	Forage	0.10	0.70	0.87	

FIELD CORN (MAIZE) Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
JA014-06DA Minnesota, USA, 2006 (Pioneer 39H85)	480 g/L SC	196- 201	110- 119	169- 178	4	0	Stover	3.15	11.90	16.27	Krolski & Harbin, 2008, RAJAP004
						7	Stover	1.37	5.69	7.65	
						14	Stover	0.91	<u>4.66</u>	6.05	
						21	Stover	0.45	2.40	3.11	
						28	Stover	0.71	3.68	4.77	
		194- 204	114- 122	164- 179	4	0	Forage	0.69	<u>2.15</u>	3.06	
						7	Forage	0.16	0.98	1.24	
						14	Forage	0.16	1.13	1.40	
						22	Forage	0.14	1.00	1.24	
						27	Forage	0.13	0.86	1.08	
JA015-06HA Kansas, USA, 2006 (Garst 8881RR)	480 g/L SC	195- 202	149- 150	130- 136	4	14	Stover	0.58	<u>6.70</u>	7.97	Krolski & Harbin, 2008, RAJAP004
		197- 203	149- 151	131- 137	4	0 7	Forage Forage	0.73 0.25	<u>3.06</u> 2.38	4.11 2.87	
JA016-06HA Illinois, USA, 2006 (Garst 8568 CB/LL)	480 g/L SC	193- 209	116- 143	145- 175	4	13	Stover	0.60	<u>3.18</u>	4.10	Krolski & Harbin, 2008, RAJAP004
		195- 208	115- 143	142- 175	4	0 7	Forage Forage	0.88 0.62	<u>3.60</u> 3.34	4.84 4.30	
JA017-06HA Nebraska, USA, 2006 (NK38B4)	480 g/L SC	197- 202	151- 154	130- 132	4	11	Stover	1.21	<u>6.16</u>	8.01	Krolski & Harbin, 2008, RAJAP004
		200- 203	153- 155	129- 131	4	0 5	Forage Forage	0.54 0.17	<u>2.27</u> 1.11	3.04 1.39	
JA018-06HA Iowa, USA, 2006 (NK65C5)	480 g/L SC	200- 202	151- 154	130- 132	4	12	Stover	0.33	<u>2.32</u>	2.89	Krolski & Harbin, 2008, RAJAP004
		198- 201	151- 153	130- 132	4	0 7	Forage Forage	0.43 0.12	<u>1.84</u> 0.96	2.46 1.17	
JA019-06HA Missouri, USA, 2006 (34B20)	480 g/L SC	200- 205	112- 118	170- 178	4	14	Stover	0.41	<u>3.89</u>	4.69	Krolski & Harbin, 2008, RAJAP004
		188- 202	112- 155	130- 176	4	0 7	Forage Forage	0.45 0.08	<u>2.62</u> 1.20	3.34 1.40	
JA020-06HA North Dakota, USA, 2006 (DKC35-51)	480 g/L SC	197- 205	107- 131	156- 184	4	14	Stover	0.27	<u>2.35</u>	2.87	Krolski & Harbin, 2008, RAJAP004
		198- 208	107- 138	152- 185	4	0 6	Forage Forage	0.42 0.28	<u>2.19</u> 2.12	2.83 2.62	
JA021-06HA North Dakota, USA, 2006 (Pioneer 39D81)	480 g/L SC	193- 200	129- 142	136- 154	4	14	Stover	0.65	<u>3.68</u>	4.71	Krolski & Harbin, 2008, RAJAP004
		189- 203	119- 142	134- 170	4	0 7	Forage Forage	0.53 0.30	<u>1.75</u> 1.25	2.46 1.68	
JA022-06HA Iowa, USA, 2006 (FA6508)	480 g/L SC	204- 205	126- 143	143- 163	4	14	Stover	0.57	<u>3.78</u>	4.74	Krolski & Harbin, 2008, RAJAP004
		200- 207	137- 138	145- 153	4	0 7	Forage Forage	0.36 0.05	<u>1.96</u> 0.61	2.52 0.73	

FIELD CORN (MAIZE) Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
JA023-06HA Illinois, USA, 2006 (B-T 6516 RR2YG)	480 g/L SC	198- 203	110- 120	167- 184	4	14	Stover	0.36	<u>1.09</u>	1.57	Krolski & Harbin, 2008, RAJAP004
		198- 203	112- 124	163- 177	4	0 7	Forage Forage	0.54 0.19	<u>2.65</u> 2.14	3.46 2.54	
JA024-06HA Minnesota, USA, 2006 (38G16)	480 g/L SC	196- 202	113- 114	172- 177	4	14	Stover	0.59	<u>3.59</u>	5.64	Krolski & Harbin, 2008, RAJAP004
		198- 216	113- 114	174- 191	4	0 7	Forage Forage	0.47 0.08	<u>2.58</u> 1.75	3.31 2.01	
JA025-06HA Nebraska, USA, 2006 (Pioneer 34N45YG/RR)	480 g/L SC	200- 202	107- 108	185- 188	4	14	Stover	0.52	<u>2.90</u>	3.71	Krolski & Harbin, 2008, RAJAP004
		200- 203	108- 110	181- 186	4	0 7	Forage Forage	0.31 0.13	<u>1.67</u> 0.93	2.15 1.16	
JA026-06HA Indiana, USA, 2006 (DKC 57-59 RR/plus)	480 g/L SC	197- 202	109- 120	165- 182	4	14	Stover	0.17	<u>0.99</u>	1.27	Krolski & Harbin, 2008, RAJAP004
		197- 201	110- 118	170- 180	4	0 7	Forage Forage	0.16 0.03	<u>0.71</u> 0.12	0.94 0.17	
JA027-06HA Iowa, USA, 2006 (Pioneer 34N50)	480 g/L SC	200- 204	108- 168	122- 184	4	14	Stover	0.84	<u>4.47</u>	5.77	Krolski & Harbin, 2008, RAJAP004
		200- 201	128- 139	144- 156	4	0 7	Forage Forage	0.42 0.14	<u>2.41</u> 1.28	3.08 1.55	
JA028-06HA Ohio, USA, 2006 (Crows 7R154)	480 g/L SC	201- 205	133- 136	149- 153	4	14	Stover	0.31	<u>3.48</u>	4.14	Krolski & Harbin, 2008, RAJAP004
		198- 206	135- 139	143- 152	4	0 7	Forage Forage	0.64 0.18	<u>2.99</u> 1.44	3.94 1.78	
JA029-06HA Minnesota, USA, 2006 (Pioneer 38H66)	480 g/L SC	195- 203	123- 125	156- 163	4	14	Stover	0.48	<u>3.22</u>	4.03 < 0.02	Krolski & Harbin, 2008, RAJAP004
		198- 200	125	159- 160	4	0 7	Forage Forage	0.40 0.14	<u>2.14</u> 1.16	2.76 1.41	
JA030-06HA Texas, USA, 2006 (Pioneer 31G97)	480 g/L SC	193- 201	146- 153	127- 137	4	14	Stover	0.33	<u>3.42</u>	4.10	Krolski & Harbin, 2008, RAJAP004
		200- 205	146- 153	131- 138	4	0 5	Forage Forage	0.55 0.23	<u>2.99</u> 1.91	3.84 2.34	

Table 18 Prothioconazole residues in popcorn resulting from supervised trials in the USA

POPCORN Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
GAP, USA	480 g/L SC	200			4	0/14					
JA034-06HA Kansas, USA, 2006 (Yellow)	480 g/L SC	195- 206	142- 145	136- 143	4	14	Stover	0.40	<u>2.46</u>	3.11	Krolski & Harbin, 2008, RAJAP004
JA035-06HA Illinois, USA, 2006 (M2101)	480 g/L SC	197- 207	115- 143	145- 174	4	12	Stover	1.24	<u>4.43</u>	6.12	Krolski & Harbin, 2008, RAJAP004
JA036-06HA Nebraska, USA, 2006 (AP2501)	480 g/L SC	198- 200	153- 155	128- 130	4	12	Stover	0.36	<u>2.51</u>	3.13	Krolski & Harbin, 2008, RAJAP004

Table 19 Prothioconazole residues in maize/corn forage resulting from supervised trials in Europe

MAIZE Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 desthio mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No				
GAP, EU	250 g/L EC	125			2	-			
08-2035-01 UK, 2008 (Cixxom)	250 g/L EC ¹	125	63	200	2	0* 0 19 56	green material	< 0.01 0.64 0.12 <u>0.03</u>	Freitag & Reineke, 2009, 08-2035
08-2035-02 Germany, 2008 (Justina)	250 g/L EC ¹	125	42	300	2	0* 0 24 49	green material	< 0.01 0.31 0.03 <u>≤ 0.01</u>	Freitag & Reineke, 2009, 08-2035
08-2035-03 Germany, 2008 (Seleno)	250 g/L EC ¹	125	32-42	300-400	2	0* 0 26 37	green material	0.06 0.40 0.04 <u>0.03</u>	Freitag & Reineke, 2009, 08-2035
08-2035-04 Netherlands, 2008 (Rosalie)	250 g/L EC ¹	125	32	400	2	0* 0 18 40	green material	0.05 0.55 0.11 <u>0.05</u>	Freitag & Reineke, 2009, 08-2035
08-2036-01 France, 2008 (PR33Y74)	250 g/L EC ¹	125	42	300	2	0* 0 11 34	green material	0.02 0.42 0.09 <u>0.02</u>	Freitag & Reineke, 2009, 08-2036

MAIZE Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 desthio mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No				
08-2036-02 Spain, 2008 (DKC6451YG)	250 g/L EC ¹	125-132	42	300-316	2	0* 0 19 28	green material	0.02 0.43 0.08 <u>0.05</u>	Freitag & Reineke, 2009, 08-2036
08-2036-03 Italy, 2008 (PR33A46)	250 g/L EC ¹	125	31	400	2	0* 0 13 34	green material	0.03 0.35 0.17 <u>0.07</u>	Freitag & Reineke, 2009, 08-2036
08-2036-04 Portugal, 2008 (A46)	250 g/L EC ¹	125	25-42	300-500	2	0* 0 8 30	green material	< 0.01 0.26 0.31 <u>0.07</u>	Freitag & Reineke, 2009, 08-2036
R 2007 0829/7 Germany, 2007 (Nescio)	250 g/L EC	200	67	300	2	-33 0* 0 29 43	green material	0.90 < 0.01 0.76 0.05 <u>0.03</u>	Freitag, 2008, RA-2675/07
R 2007 0837/8 Germany, 2007 (DK3745)	250 g/L EC	200	67	300	2	-39 0* 0 19 46	green material	1.3 < 0.01 0.49 0.03 <u>0.01</u>	Freitag, 2008, RA-2675/07
R 2007 0838/6 Spain, 2007 (PR 31G98)	250 g/L EC	186-200	50	372-400	2	-25 0* 0 16 23	green material	0.94 0.03 0.40 0.18 <u>≤ 0.01</u>	Freitag, 2008, RA-2675/07
R 2007 0839/4 Portugal, 2007 (PR33N09)	250 g/L EC	200	67	300	2	-43 0* 0 15 33	green material	1.7 0.34 0.42 0.05 <u>0.02</u>	Freitag, 2008, RA-2675/07
11-2109-01 France, 2011 (Cobalt)	250 g/L SE ²	125	42	300	2	0* 0 20 40	green material	0.18 0.44 0.16 <u>0.08</u>	Bomke & Teubner, 2013, 11-2109
						82	rest of plant	<u>0.06</u>	
11-2109-02 Germany, 2011 (Saludo)	250 g/L SE ²	125	42	300	2	0* 0 31 51	green material	0.13 0.65 0.05 <u>0.02</u>	Bomke & Teubner, 2013, 11-2109
						65	rest of plant	<u>0.03</u>	
11-2109-03 UK 2011 (Cougar)	250 g/L SE ²	125	50	250	2	0* 0 31 49	green material	0.34 0.55 0.16 <u>0.12</u>	Bomke & Teubner, 2013, 11-2109
						60	rest of plant	<u>0.22</u>	

MAIZE Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 desthio mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No				
11-2109-04 Belgium, 2011 (Delitop)	250 g/L SE ²	125	45	275	2	0* 0 29 42	green material	0.04 0.28 0.05 <u>0.03</u>	Bomke & Teubner, 2013, 11-2109
						64	rest of plant	<u>0.04</u>	
11-2110-02 Italy, 2011 (Constanza)	250 g/L SE ²	125	31	400	2	0* 0 17 29	green material	0.04 0.50 0.06 <u>0.03</u>	Bomke, 2012, 11-2110
						62	rest of plant	<u>0.05</u>	
11-2110-03 Spain, 2011 (DKC66 67YG)	250 g/L SE ²	125	42	300	2	0* 0 20	green material	0.05 0.39 <u>0.06</u>	Bomke, 2012, 11-2110
						69	rest of plant	<u>0.05</u>	
11-2110-04 Greece, 2011 (Dekalp, 5276)	250 g/L SE ²	125	31	400	2	0* 0 18 31	green material	0.14 0.82 0.27 <u>0.16</u>	Bomke, 2012, 11-2110
						48	rest of plant	<u>0.13</u>	
11-2110-05 France, 2011 (PR 33 A46)	250 g/L SE ²	125	42	300	2	0* 0 14 29	green material	0.15 0.87 0.24 <u>0.14</u>	Bomke, 2012, 11-2110
						64	rest of plant	<u>0.24</u>	

Soya bean forage and hay

Supervised trials in the USA: Residue data on soya beans from 19 trials conducted in the USA were previously evaluated by the 2008 JMPR and 2009 JMPR. In these trials the time between last application and sampling was too short (7 days) and therefore cannot be used to estimate residue levels.

Peanut fodder

Eight supervised trials were conducted on peanut in Australia. Peanut fodder was analysed in four trials at 28 DAT. The residue values are summarised in Table 20. Residues in hay are not reported because the use instructions indicate: 'Do not feed hay or threshings or allow livestock to graze in treated areas.'

Table 20 Prothioconazole residues in peanut resulting from supervised trials in Australia

PEANUTS Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (l/ha)	No						
GAP, AUSTRALIA	480 g/L SC	192			4	28					
C668 Australia 2011-2012 (HOJ-E Holt)	480 g/L SC	193- 194	96	201- 202	4	15 22 29	Fodder	0.10 0.07 0.30	3.64 6.86 <u>11.60</u>	4.12 7.64 13.09	Ellis, 2012, BCS-0383
C697 Australia 2012 (Holt)	480 g/L SC	192- 193	96	199- 201	4	14 21 28	Fodder	< 0.05 < 0.05 < 0.05	2.72 3.43 <u>1.15</u>	3.00 3.78 1.27	Ellis, 2012, BCS-0383
C680 Australia 2011-2012 (HOJ-L-2)	480 g/L SC	192- 194	26-29	673- 754	4	15 21 28	Fodder	< 0.05 < 0.05 < 0.05	0.52 1.57 <u>1.14</u>	0.58 1.73 1.26	Ellis, 2012, BCS-0383
C682 Australia 2011-2012 (WH2 04401)	480 g/L SC	192- 193	25-37	517- 778	4	14 21 27	Fodder	0.34 0.26 0.06	17.10 22.10 <u>7.00</u>	19.19 24.62 7.78	Ellis, 2012, BCS-0383

FATE OF RESIDUES IN STORAGE AND IN PROCESSING

Information and Data from Residues in Processed Commodities

Processing studies on corn, peanut and oilseed rape are summarized in this section.

Processing Corn into Corn Flour, Starch, Grits, Oil, Aspirated Grain Fractions and Bran

A corn processing study (Krolski & Harbin, 2008, RAJAP001) has been conducted in which field corn was treated with an SC formulation containing 480 g/L prothioconazole at an exaggerated total application rate of 4.0 kg ai/ha, equivalent to 5× the maximum total seasonal rate for prothioconazole on corn. Bulk samples of corn grain were collected at maturity (BBCH 89) at 13 DAT, and frozen prior to analysis/processing. Subsamples of corn grain were removed for analysis, aspirated grain fractions were generated, and the remaining grain was used to generate the required processed commodities of starch, oil (wet and dry milled, refined, bleached, and deodorized), grits, flour, meal and bran. Processing was performed using procedures that simulated commercial processing practices.

Grain samples were dried in an oven until the moisture content was 10–13%. The corn grain was then aspirated and the material that passed through the 2360 micron sieve was recombined to produce an aspirated grain fraction. After generation of aspirated grain fractions, whole corn was cleaned by aspiration and screening. A subsample of cleaned corn grain was dry milled to form large grits and grits, meal, flour and bran.

The germ material was conditioned and flaked and then extracted with hexane to give crude oil and solvent extracted germ flakes. The crude oil was neutralised with 16 degree Baumé sodium hydroxide to give alkali refined oil and soapstock. The soapstock was discarded. The refined oil was bleached using activated bleaching earth and deodorised using 0.5% citric acid solution to give deodorized oil (dry milled) and distillates.

A second subsample of dried cleaned corn grain was wet milled to form hull, germ, starch and gluten fractions. The germ material was conditioned, flaked and pressed to form crude oil and presscake. The presscake was extracted with hexane to form solvent extracted presscake and crude oil. The combined crude oil was refined using 16 degree Baumé sodium hydroxide to give refined oil and soapstock. The soapstock was discarded. The refined oil was bleached and deodorised as described above to give deodorized oil (wet milled) and distillates.

The samples were frozen after collection and stored frozen ($< -12^{\circ}\text{C}$) until extraction and analysis. The maximum period of storage was 429 days (14 months) for grain and 85 days (3 months) for aspirated grain fractions and processed commodities, which falls within the stability period of 3 years as confirmed by previous studies.

Residues of prothioconazole were determined by high performance liquid chromatography – triple stage quadrupole mass spectrometry (LC/MS/MS). The method, RPA JA/03/01, which was used for the analysis was validated with an LOQ of 0.02 mg/kg for prothioconazole-sulfonic acid, 0.02 mg/kg for prothioconazole-desthio and 0.02 mg/kg for total prothioconazole.

Residues of the metabolites 1,2,4-triazole, triazolylalanine and triazolylacetic acid were also determined in this study, but the results are not summarised here as these analytes are not included in the residue definition for monitoring or risk assessment.

Procedural recoveries at fortification levels of 0.02–5 mg/kg were within the acceptable range of 70–120% for prothioconazole-desthio, prothioconazole-sulfonic acid and prothioconazole in each matrix. The prothioconazole recovery was the sum of prothioconazole-desthio and prothioconazole-sulfonic acid from recovery samples fortified with prothioconazole only.

Residues of prothioconazole were almost exclusively on the outside of the corn grain, and were strongly concentrated ($90\times$) in the aspirated grain fraction, and slightly concentrated in the bran ($1.3\times$). Prothioconazole residues do not concentrate in starch, oil, grits, flour or meal ($< 1\times$) during processing of corn grain. The results are summarised in Table 30.

Table 21 Residues of prothioconazole in corn processed fractions

Processed Fractions	Total prothioconazole residue (mean) mg/kg	Processing factor	Reference
Corn grain	0.072		Krolski & Harbin, 2008, RAJAP001
Aspirated grain fractions	6.50	90	
Starch	< 0.02	< 0.28	
Oil (wet milled)	< 0.02	< 0.28	
Grits	< 0.02	< 0.28	
Flour	0.041	0.57	
Meal	0.031	0.43	
Bran	0.094	1.31	
Oil (dry milled)	< 0.02	< 0.28	

Processing Oilseed Rape to Meal and Refined Oil

A field trial to measure the magnitude of prothioconazole residues in canola seed, canola meal and canola refined oil was previously evaluated by the 2008 JMPR and reported in the 2008 JMPR Evaluation (Prothioconazole – Table 81). The 2008 meeting concluded that no concentration ($< 0.7\times$) of the total prothioconazole derived residues was seen in canola meal and refined oil. The processing factors derived from this study on canola are summarized in Table 22.

Table 22 Residues of prothioconazole in canola processed fractions

Processed Fractions	Total prothioconazole residue mg/kg	Processing factor	Reference
Canola seed	0.03		2008 JMPR
Canola meal	< 0.02	< 0.7	
Refined oil	< 0.02	< 0.7	

An additional oilseed rape processing study (Freitag & Hoffmann, 2010, 08-3112) was submitted. The oilseed rape seed samples originate from a trial (08-2112-02) conducted as part of study of Freitag, Reineke & Krusell, 2010, 08-2112.

Seed samples were processed into screw pressed oil, pomace, meal, solvent extracted oil, crude oil, pre-clarified crude oil, neutralised crude oil and refined oil, simulating commercial practices.

After defrosting, rape seed samples were dried at 35 °C for 30 hours to a moisture content of 6–10%. The conditioned seed was cleaned manually using a sieve to remove parts of coarse stalks and weed seeds. The conditioned and cleaned rape seeds were pressed in a screw press yielding screw-pressed oil, and pomace.

An aliquot of the pomace was milled (distance of the rolls: 2.0 mm) and sub-samples extracted using a small technical extraction device. The extraction was conducted in two steps. The first extraction step started with addition of n-hexane to the pomace. The n-hexane circulated through the pomace for about 2 hours. During the circulation, the n-hexane was heated up to approx. 60 °C. Residual solvent was removed from the solvent-oil-mixture (miscella) using a rotary evaporator at approx. 50 °C, yielding solvent extracted oil. The solvent-extracted meal was sampled after storing at room temperature for approximately one day.

The two oil fractions obtained (screw-pressed oil and solvent extracted oil) were mixed yielding the sample of crude oil. The crude oil was filtered, yielding the sample of pre-clarified crude oil. The pre-clarified crude oil was heated up to 60–70 °C while stirring. After addition of 10% water, the mixture of water and crude oil was heated up to 85–90 °C and maintained at 85 °C for approximately 45 minutes while stirring. After phase separation, the watery phase including mucilage was removed. To the oily phase approximately 1% of concentrated phosphorus acid was added at 60 °C while stirring. The mixture was heated up to approximately 85 °C and maintained at that temperature for approximately 45 minutes while stirring. Immediately before switching off the stirrer, 10% water was added. The mixture of water and oily phases remained at approximately 85 °C until separation of the phases, and the watery phase including flocculated precipitation was then removed. After determination of the acid-number, the oily phase was heated up to approximately 90 °C while stirring. A few grams of a 7% sodium hydroxide solution (amount depending on acidity of the oil phase) were added and the mixture was stirred for approximately 20 minutes. After addition of 10% water and further 5 minutes of stirring the stirrer was switched off, the phases were allowed to separate and the watery phase was removed. The process was repeated as the acid-number of the oil was above 0.12. After the second neutralisation process, the acid value was below 0.12 and an aliquot of neutralised crude oil was sampled.

The remaining neutralised crude oil was heated up to 90 °C while stirring and washed by the addition of 10% water. The mixture was stirred for 20 min at 90 °C. After phase separation the watery phase was removed. These steps were repeated until the pH of the washing water was in the range 7-8 and the soap content of the oil was $\leq 0.01\%$. The oil was heated up to approximately 95 °C while stirring. After addition of citric acid (60 mg/kg oil), the oil was dried under vacuum. The oil was heated up to 95 °C while stirring. After addition of 1% podsol (referring to the used oil) the oil was bleached for 5 min without vacuum and 20 min with vacuum. The podsol was removed by filtration under vacuum. The oil was heated under vacuum up to 240 °C while stirring. After reaching 160 °C,

steam was transferred through the oil in order to expel fatty acids, odour and taste influencing compounds as well as other volatile compounds. The mixture stayed at 240°C for about 20 minutes. After cooling to 160 °C, the steam supply was stopped and then the oil was dried under vacuum until a temperature of ≤ 80 °C was reached. The refined oil was sampled.

All processed fractions were stored frozen (-18 °C) prior to analysis. seed was analysed within 645 days of sampling, and all processed fractions were analysed within 349 days of processing, which fall within the stability period of 3 years as confirmed by previous studies. Residues of prothioconazole-desthio were determined by HPLC/MS/MS using method 01013. The LOQ was 0.01 mg/kg in all matrices. Procedural recoveries in seed and oil at fortification levels of 0.01–0.10 mg/kg were within the acceptable range of 70–120%, RSD < 20%, for prothioconazole-desthio in each matrix.

In this study, samples were also analysed for residues of the hydroxyl metabolites (prothioconazole- α -hydroxy-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio and prothioconazole-6-hydroxy-desthio) and for residues of 1,2,4-triazole, triazole acetic acid, triazole lactic acid and triazole alanine. The results are not summarised here as none of these analytes are included in the residue definition for monitoring or risk assessment.

Results show that prothioconazole-desthio residues do not concentrate in meal, screw-pressed oil or refined oil ($\leq 1\times$), but do concentrate in crude oil and solvent extracted oil ($2\times$) during processing of rape seed. The results are summarised in Table 23.

Table 23 Residues of prothioconazole in oilseed rape processed fractions

Processed Fractions	Prothioconazole-desthio residue mg/kg	Processing factor	Reference
Oilseed rape seed	0.01		Freitag & Hoffmann, 2010, 08-3112
Screwpressed oil	0.01	1	
Pomace	0.01	1	
Meal	< 0.01	<1	
Solvent extracted oil	0.02	2	
Crude oil	0.02	2	
Preclarified crude oil	0.02	2	
Neutralised crude oil	0.01	1	
Refined oil	< 0.01	<1	

Based on the available processing studies, the processing factors that have been calculated are summarized in Table 24.

Table 24 Summary of processing factors

Commodity	Processed fraction	Processing factor	Reference
Peanut	Peanut meal	1.8	2008 JMPR
	Refined oil	< 0.1	
	Roasted peanuts	0.5	
	Peanut butter	0.6	
Wheat	Aspirated grain fraction	250	2008 JMPR
	Bran	2.4	
	Flour	< 0.4	
	Middlings	0.6	

Commodity	Processed fraction	Processing factor	Reference
	Shorts	1	
	Wheat germ	2	
Soya bean	Aspirated grain fraction	75	2008 JMPR
	Meal	0.2	
	Hull	0.5	
	Refined oil	< 0.2	
Oilseed rape	Meal	<1	Freitag & Hoffmann, 2010, 08-3112
	Refined oil	<1	
Corn	Aspirated grain fractions	90	Krolski & Harbin, 2008, RAJAP001
	Starch	<1	
	Oil (wet milled)	<1	
	Grits	<1	
	Flour	<1	
	Meal	<1	
	Bran	1.3	
	Oil (dry milled)	<1	

RESIDUE ON ANIMAL PRODUCTS

Residues in Animal Commodities

In addition to those commodities which had been evaluated by the previous Meetings, the following commodities could result in residues in animal tissues, milk, and eggs: sweet corn stover, field corn stover, popcorn stover, soya bean hay and peanut fodder.

APPRAISAL

Prothioconazole was evaluated first time by the 2008 JMPR. The residue definition for plant commodities for enforcement and dietary risk assessment was prothioconazole-desthio. The residue definition for animal commodities for enforcement was prothioconazole-desthio and for dietary risk assessment was the sum of prothioconazole-desthio, prothioconazole-desthio-3-hydroxy, prothioconazole-desthio-4-hydroxy and their conjugates, expressed as prothioconazole-desthio. The residue was considered to be not fat-soluble for the purposes of residue definition.

The 2008 JMPR established, for prothioconazole-desthio, an ADI of 0–0.01 mg/kg bw and ARfD of 0.01 mg/kg bw for women of child bearing age and 1 mg/kg bw for the general population.

The Forty-fifth Session of CCPR scheduled prothioconazole for periodic re-evaluation of residues by the 2014 JMPR. For the 2014 Meeting data were provided on blueberry, corn, cranberry, cucurbits, potato, sweet corn peanut and soya bean together with current use recommendations and analytical methods used in supervised trials.

Methods of analysis

A number of validated analytical methods for the determination of residues in plant, animal tissue, milk and soils were evaluated by the 2007 and 2008 JMPR Meetings. The Meeting received the basic analytical methods with minor modifications which were validated to determine residues in crops that

had been used in the residue trials. An analytical method was also provided for the determination of residues of prothioconazole in animal matrices.

The trials carried out in the USA/Canada used method RPA JA/03/01 or the modified method JA-001-P04-02 which involve extraction with methanol, hydrogen peroxide and sodium bicarbonate at an elevated temperature. This converts prothioconazole to a mixture of prothioconazole-sulfonic acid, while prothioconazole-desthio is extracted unchanged. After purification steps, the residues are analysed by LC-MS/MS. The LOQ of these methods is 0.02 mg/kg for prothioconazole-sulfonic acid and 0.02 mg/kg for prothioconazole-desthio.

The trials carried out in Brazil, EU and Australia used methods 01013, 00598/M001 and ATM-0053 in which residues of prothioconazole and prothioconazole-desthio are extracted with acetonitrile/water containing cysteine hydrochloride. After purification steps, dependent on the method, the analytes are analysed by LC-MS/MS. The LOQ of these methods is 0.01 mg/kg (or 0.05 mg/kg for forage and straw matrices) for prothioconazole and prothioconazole-desthio.

Storage Stability under Frozen Conditions

Information relating to storage stability of residues in wheat, canola (seed, pod, straw), spinach (leaves), sugar beet (root, leaf with root collar), tomato (fruit) and field pea (dried) was evaluated at the 2008 JMPR Meeting. This showed that residues of prothioconazole-desthio were stable over a frozen storage period of up to 36 months in wheat and at least 24 months for the other crops. Residues of prothioconazole and prothioconazole-desthio were stable in wheat hay and straw, canola seeds, mustard greens, turnip root and tomato fruit over a frozen storage period of 36–42 months.

An additional freezer storage stability study for residues of prothioconazole and prothioconazole-desthio (JAU 6476-desthio) in wheat (forage, straw, grain, bran and flour), canola (seed and oil), mustard greens, tomato (fruit and paste) and turnip roots was performed.

No significant degradation of prothioconazole-desthio was observed in any matrix analysed over the 3-year frozen storage period.

The stability of parent prothioconazole varied from 57 days up to 378 days without showing any dependency on the sample matrix. However, metabolism studies indicated that after foliar application prothioconazole rapidly degraded to prothioconazole-desthio and polar metabolites being converted to the sulfonic acid derivative, which is measured by the analytical method. Therefore the relative instability of parent compound does not affect the validity of the desthio residues measured in supervised trials.

Results of supervised residue trials on crops

Supervised trials have been conducted to support MRLs for the following crops or groups of crops: blueberry, cranberry, cucurbits (cucumber, summer squash and melon), sweet corn, soya bean, potato, maize (field corn, popcorn) and peanuts.

In trials conducted in the USA, Canada and Brazil, residues of prothioconazole sulfonic acid (JAU 6476 sulfonic acid), prothioconazole-desthio (JAU 6476 desthio) and total prothioconazole (JAU 6476) were determined and reported. In trials conducted in Australia, residues of prothioconazole (JAU 6476), prothioconazole-desthio (JAU 6476 desthio) and total prothioconazole (JAU 6476) were determined and reported. However, only the residues of prothioconazole-desthio have been used for calculation of MRL, HR and STMR values in accordance with the residue definition. The residues of prothioconazole-desthio are expressed as mg prothioconazole-desthio equivalents/kg.

Bush berries

Blueberry

Eleven supervised trials were conducted in 2010 in the USA and Canada matching the US GAP (2 × 200 g ai/ha nominal rate at 6–7 days intervals, 7-day PHI) for the bush berry subgroup.

Prothioconazole-desthio residues from trials on blueberries were: 0.15, 0.22, 0.26, 0.28, 0.42, 0.52, 0.56, 0.60, 0.65, 0.70 and 0.87 mg/kg.

Taking into account that blueberry is the representative commodity for the subgroup; the Meeting estimated a maximum residue level of 1.5 mg/kg, a HR of 0.87 mg/kg and a STMR of 0.52 mg/kg for prothioconazole-desthio residues for the bush berry subgroup.

Low growing berries

Cranberry

Six supervised trials were conducted in 2010 in the USA according to the US GAP (2 × 186 g ai/ha nominal rate at 7-10 days intervals, 45-day PHI)

Prothioconazole-desthio residues from trials on cranberries were: ≤ 0.02 (3), 0.03 (2) and 0.09 mg/kg.

The Meeting estimated maximum residue level of 0.15 mg/kg, HR of 0.09 mg/kg and STMR of 0.025 mg/kg for prothioconazole-desthio residues for cranberry.

Fruiting vegetables, Cucurbits

Eight supervised trials were conducted in 2010 on *cucumber*, eight trials on *musk melon* and eight trials on *summer squash* in the USA according to US GAP (one soil +2 × 200 g ai/kg nominal rate at 7-10 days intervals, max 600 g ai/season, 7-day PHI).

Prothioconazole-desthio residues from trials on cucumber were: 0.02, 0.02, 0.03, 0.03, 0.04, 0.05, 0.05, 0.06 mg/kg.

Prothioconazole-desthio residue concentrations from trials on musk melon were: 0.03, 0.06, 0.06, 0.06, 0.07, 0.15, 0.15 mg/kg.

Prothioconazole-desthio residue concentrations from trials on summer squash were: < 0.02 (5), 0.03, 0.05 and 0.06 mg/kg.

The Meeting noted that the GAP in USA is for cucurbit vegetables, the residue populations were not significantly different and the median residues were within the 5 times range, and agreed to combine the datasets. Residues in the 24 trials were: < 0.02 (5), 0.02 (2), 0.03 (4), 0.04, 0.05 (3), 0.06 (6), 0.07 and 0.15 (2) mg/kg.

The Meeting noted that the ARfD would be exceeded by 150% for watermelon. As no alternative GAP for fruiting vegetables was available, watermelon was not included in the recommendation for the crop group fruiting vegetables, cucurbits.

The Meeting estimated a maximum residue level of 0.2 mg/kg, HR of 0.15 mg/kg and STMR of 0.045 mg/kg prothioconazole-desthio residues for fruiting vegetables, cucurbits (except watermelon).

Fruiting vegetables, Other Than Cucurbits

Sweet corn

Twelve supervised trials were conducted on sweet corn in the USA at 4 × 200 g ai/ha matching the current US GAP. The PHI for sweet corn forage and ears is 0 days and 14 days for fodder.

Prothioconazole-desthio residue concentrations in corn ears were: < 0.018 in all samples.

The Meeting estimated a maximum residue level of 0.02 mg/kg, and HR and STMR values of 0.018 mg/kg for sweet corn.

*Pulses**Soya beans*

Residue data on soya beans from 19 trials conducted in the USA were previously evaluated by the 2008 and 2009 JMPR Meetings. The current US GAP for soya beans permit 3 applications at 88–175 g ai/ha rate at 10–21 day intervals with a PHI of 21 days.

The average prothioconazole-desthio residues in soya bean samples derived from trials corresponding to the current US GAP were: < 0.05 (16), 0.051, 0.055 and 0.105 mg/kg

Ten supervised trials were conducted in 2012–2013 season on soya bean in Brazil matching the GAP for Brazil (4×87.5 g ai/ha, PHI 30 days). The prothioconazole-desthio residues were reported as the parent compound. Applying the conversion factor of 0.907 (312.20/344.26) the residues expressed as prothioconazole-desthio were: $< 0.009 \times 4$, 0.009×2 , 0.018, 0.027, 0.036 and 0.091 mg/kg.

The Meeting noted that the US trials resulted in higher residues and used those values. The meeting estimated a maximum residue level of 0.2 mg/kg and STMR value of 0.05 mg/kg.

*Root and tuber vegetables**Potato*

A total of twenty supervised trials were conducted in 2005 and 2010 as potato seed-piece treatments in Belgium, France, Germany, Italy, the Netherlands, Spain and the UK at maximum GAP (up to 0.64 g ai/100 kg seed, equivalent to 16 g ai/ha) or higher rate. The potatoes were harvested at maturity. The prothioconazole-desthio residues were < 0.01 mg/kg in all samples take 90–136 days after seed treatment.

Taking into account that the LOQ of the enforcement method is 0.02 mg/kg, the Meeting estimated a maximum residue level of 0.02* mg/kg, HR and STMR values of 0.01 mg/kg for potato.

*Cereal grains**Maize*

Twenty supervised trials were conducted in 2006 on field corn and three trials on popcorn in the USA (4×200 g ai/ha at 7–14 days apart, 0 day PHI for forage and 14 days for grain and fodder).

The prothioconazole-desthio residues in maize grain samples collected at 14 days after last application were: < 0.018 (17), and 0.05 mg/kg,

Ten supervised trials were conducted in 2011–2012 on corn in Brazil according to the GAP (2×70 –87.5 g ai/ha, at minimum 15 days, 15 days PHI).

The prothioconazole-desthio residues in maize grain samples collected at 14–15 days after last application were: < 0.01 (10) mg/kg.

A total of twenty supervised trials were conducted in 2007, 2008 and 2011 on maize/corn in Europe matching the GAP of the Czech Republic (2×125 g ai/ha, PHI 35 days) and Italy (2×125 g ai/ha, retreatment at a minimum of 14 days, 35 days or non-specified PHI).

The prothioconazole-desthio residues in all of immature and mature maize grain samples collected from 17 days onward were: < 0.01 (20) mg/kg.

The Meeting considered that all maize grain/seed samples (47) contained non-detectable residues regardless of maturity and time between last application and sampling, except in one trial where residues of up to 0.05 mg/kg residue was measured.

The Meeting estimated a maximum residue level of 0.1 mg/kg and STMR value of 0.018 mg/kg for maize and popcorn.

*Oilseeds**Peanuts*

Residue data on peanuts from trials conducted in the USA had been evaluated by the 2009 JMPR. Residues of prothioconazole-desthio were < 0.02 mg/kg in 12 trials matching the USA GAP (4 × 200 g ai/ha, PHI 14 days).

Eight supervised trials were conducted in 2011–2012 in Australia. Four trials included plots which were treated according to the GAP for peanut in Australia (4 times 120–192 g ai/ha at 10–14 days apart, 28-day PHI), and four trials included plots which were treated with higher number of applications and a shorter PHI (up to 21 days). Samples were taken immediately before the last application and 0, 7, 14–15 and 20–21 days after.

The prothioconazole-desthio residues in all samples collected from Day 0 to day 29 days were: < 0.01 mg/kg.

The Meeting confirmed its previous recommendations of 0.01* mg/kg.

Primary feed commodities

Descriptions of trial conditions and residues are described under relevant food commodities. The residue data relevant to the feed commodities are summarized below.

Maize and sweet corn forage and fodder

Prothioconazole-desthio residue concentrations in sweet corn forage from trials conducted at 4 × 200 g ai/ha were: 1.31, 1.50, 1.89, 1.94, 1.98, 2.08, 2.12, 2.37, 2.82, 3.20, 3.75 and 4.08 mg/kg.

Prothioconazole-desthio residue concentrations in field corn forage from trials based on the USA GAP for corn are: 0.71, 1.67, 1.75, 1.84, 1.90, 1.96, 2.14, 2.15, 2.19, 2.20, 2.27, 2.41, 2.58, 2.62, 2.65, 2.99 (2), 3.06, 3.44 and 3.60 mg/kg.

The Meeting noted that the GAP in USA for sweet corn and maize is the same and that the residue populations were not significantly different, and agreed to combine the datasets.

Residues in the trials were: 0.71, 1.31, 1.50, 1.67, 1.75, 1.84, 1.89, 1.90, 1.94, 1.96, 1.98, 2.08, 2.12, 2.14, 2.15, 2.19, 2.20, 2.27, 2.37, 2.41, 2.58, 2.62, 2.65, 2.82, 2.99 (2), 3.06, 3.44, 3.60, 3.75 and 4.08 mg/kg.

Residues in forage from the European trials were much lower (< 0.2 mg/kg) than from the trials conducted according to the USA GAP, and therefore the Meeting used the US trial data for the estimation of residue levels.

The Meeting estimated highest residue of 4.08 mg/kg and median residue of 2.15 mg/kg for sweet corn and filed maize forage.

Prothioconazole-desthio residue concentrations in sweet corn stover from trials conducted at 4 × 200 g ai/ha were: 0.67 (2), 0.71, 0.82, 0.90, 0.98, 1.45, 1.68 (2), 1.70, 3.61 and 5.88 mg/kg.

Prothioconazole-desthio residue concentrations in field corn stover from trials based on the USA GAP for corn were: 0.67 (2), 0.71, 0.82, 0.90, 0.98, 0.99, 1.09, 1.45, 1.68 (2), 1.70, 2.22, 2.32, 2.35, 2.42, 2.90, 3.18, 3.22, 3.42, 3.48, 3.59, 3.61, 3.68, 3.78, 3.89, 4.05, 4.47, 4.66, 5.88, 6.16 and 6.70 mg/kg.

Prothioconazole-desthio residue concentrations in popcorn stover from trials based on the USA GAP for corn were: 2.46, 2.51 and 4.43 mg/kg.

The Meeting noted that the GAP in the USA is for sweet corn and maize is the same and the residue populations were not significantly different, and agreed to combine the datasets. Residues in the trials were: 0.99, 1.09, 2.22, 2.32, 2.35, 2.42, 2.46, 2.51, 2.90, 3.18, 3.22, 3.42, 3.48, 3.59, 3.68, 3.78, 3.89, 4.05, 4.43, 4.47, 4.66, 6.16 and 6.70 mg/kg.

The Meeting estimated a maximum residue level of 15 mg/kg (dry weight basis) highest residue of 6.7 mg/kg and median residue of 3.48 mg/kg for sweet corn and field maize stover.

Soya bean forage and hay

The Meeting received information on residues in soya bean forage and hay. However, the samples were taken at 7 days PHI, which did not correspond to the current US GAP and therefore could not be used for the estimation of residue levels.

Peanut fodder

Prothioconazole-desthio residue concentrations in peanut hay from trials based on the Australian GAP for peanuts were: 1.14, 1.15, 7.00 and 11.60 mg/kg.

The Meeting estimated highest and median residues of 11.6 mg/kg and 4.08 mg/kg, respectively, for peanut hay.

Fate of residue during processing

Processing Corn

Field corn was treated at 5× total seasonal rate. Subsamples of corn grain were removed for analysis, aspirated grain fractions were generated, and the remaining grain was used to generate the required processed commodities of starch, oil (wet and dry milled, refined, bleached, and deodorized), grits, flour, meal and bran. Processing was performed using procedures that simulated commercial practices.

The processing factors calculated from total prothioconazole residues were: starch (< 0.28), oil, wet milled (< 0.28), grits (< 0.28), flour (0.57), meal (0.43), bran (1.31), oil, dry milled (< 0.28).

The Meeting noted that the major part of the residue is prothioconazole-desthio and concluded that the results of processing study can be used for estimation of the processing factors for prothioconazole-desthio. The Meeting estimated STMR values (mg/kg) for maize starch (0.0050), maize flour (0.010), and refined maize oil (0.0050).

Processing rape seed

Rape seed was sampled following treatment with prothioconazole, 2×120 g/ha. The conditioned and cleaned rape seeds were pressed in a screw press, yielding screw pressed oil, and pomace. An aliquot of pomace was extracted twice with hexane. The screw pressed oil and extracted oil were combined to obtain crude oil, which was neutralized and processed to obtain refined oil.

The processing factors calculated were: screw pressed oil (1), pomace (1), meal (< 1), solvent extracted oil (2), crude oil (2), pre-clarified crude oil (2), neutralized crude oil (1) and refined oil (< 1)

The Meeting estimated STMR-P values for rape seed oil, edible of 0.02 mg/kg based on the STMR of 0.02 estimated by the 2009 JMPR

Residues in Animal Commodities

The following commodities evaluated by the present and previous Meetings could result in residues in animal tissues, milk, and eggs: sweet corn forage and stover, field corn forage and stover, popcorn forage and stover, soya bean hay and peanut fodder (dry), grains, straw and fodder (dry) of cereal grains.

Livestock dietary burden

The maximum and mean dietary burdens were calculated using the highest residues or median residues of prothioconazole-desthio estimated by the current Meeting on a basis of the OECD Animal Feeding Table. The calculated maximum and mean animal burdens are summarized below

Summary of livestock dietary burdens (ppm of dry matter diet)

	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	4.04	3.32	12.36	5.47	21.6 ^a	5.50 ^c	3.15	3.15
Dairy cattle	10.63	4.85	10.89	4.94	18.42 ^b	5.50 ^d	7.66	5.25
Broilers	2.86	2.86	1.17	1.17	1.18	1.18	0.29	0.29
Layers	2.86	2.86 ^f	3.33 ^e	1.71	1.18	1.18	1.72	1.72

^a Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian meat

^b Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk

^c Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian meat.

^d Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

^e Highest maximum poultry dietary burden suitable for MRL estimates for poultry meat and eggs.

^f Highest mean poultry dietary burden suitable for STMR estimates for poultry meat and eggs.

Animal commodity maximum residue levels

The 2009 JMPR summarized the total prothioconazole desthio residues (mg/kg) in edible tissues of dairy cattle after 28 days of dosing with prothioconazole-desthio:

Tissue	4 ppm dose		25 ppm dose		100 ppm dose	
	Range	Mean	Range	Mean	Range	Mean
Liver	0.02-0.05	0.04	0.18-0.26	0.22	0.61-1.6	0.95
Kidney	0.01-0.04	0.02	0.11-0.17	0.14	0.41-1.1	0.65
Muscle	< 0.01	< 0.01	< 0.01	< 0.01	0.01-0.03	0.02
Fat	< 0.01	< 0.01	0.01-0.02	0.01	0.03-0.14	0.07
Milk	< 0.004	< 0.004	< 0.004	< 0.004	0.013-0.02	0.017

The maximum dietary burden for beef cattle remained the same as was estimated by the 2009 JMPR. The maximum dietary burden for dairy cattle is 18.42 ppm which is smaller than the 25 ppm dose and no detectable residue can be expected in milk.

The mean dietary burden for beef and dairy cattle is 5.5 ppm, which is higher than the 4.8 ppm estimated by the 2009 JMPR. However the STMR values estimated by the 2009 JMPR cover the likely median residues.

The Meeting confirmed its previous recommendations for maximum residue levels, HR and median residues for mammalian meat, and edible offal and milk.

The 2008 Meeting concluded that the feeding study on poultry did not reflect the residue composition in feed and could not be used for estimation of residue levels.

RECOMMENDATIONS

On the basis of the data from supervised trials and farm animal feeding studies reported by the 2006 JMPR, the Meeting concluded that the residue levels listed below are appropriate for establishing maximum residue limits and for IEDI assessment.

Definition of the residue (for enforcement and dietary risk assessment) for plant commodities: *prothioconazole-desthio*.

Definition of the residue (for enforcement and dietary risk assessment) for animal commodities: *the sum of prothioconazole-desthio. (M04), prothioconazole-desthio-3-hydroxy (M14), prothioconazole-desthio-4-hydroxy (M15), expressed as prothioconazole-desthio after correction for molecular weight.*

Commodity		MRL mg/kg	STMR mg/kg	HR mg/kg
CCN	Name			
FB 2006	Bush berries	1.5	0.52	0.87
FB0265	Cranberry	0.15	0.025	0.9
VC 0046	Fruiting vegetables, Cucurbits, except watermelon	0.2	0.045	0.15
VO 0447	Sweet corn	0.02	0.018	0.018
VD 0541	Soya bean	0.2	0.05	
GC 0656	Popcorn	0.1	0.018	
VR 0589	Potato	0.02(*)	0.01	0.01
GC 0645	Maize	0.1	0.01	
AF 0645	Maize forage		2.15	4.08
	Sweet corn forage		2.15	4.08
AS 0645	Maize fodder	15	3.48	6.7
	Sweet corn fodder	15	3.48	6.7
AL 0697	Peanut fodder	15	4.08	11.6
CF 1255	Maize flour		0.0057	
OR 0645	Maize oil		0.0028	
	Maize starch		0.0028	
OR 0495	Rape oil refined		0.02	

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intake (IEDI) for prothioconazole-desthio was calculated from the recommendations for STMR-s for raw agricultural commodities in combination with consumption data for corresponding food commodities. The results are shown in Annex 3 to the 2014 Report.

The IEDI of the 17 GEMS/Food cluster diets were in the range of 0–3 % of the maximum ADI of 0.01 mg/kg bw. The Meeting concluded that the long-term intake of residues from uses of prothioconazole considered by the Meeting is unlikely to present a public health concern.

Short-term intake

The International Estimated Short-term Intake (IESTI) for prothioconazole-desthio was calculated from the recommendations for HR and STMR-s for raw agricultural commodities in combination with consumption data for corresponding food commodities. The results are shown in Annex 4 to the 2014 Report.

The IESTI for women of child bearing age is 0–100% of the ARfD of 0.01 mg/kg bw. The IESTI for the rest of the population is 0–1 % of the ARfD of 1 mg/kg bw.

For watermelon the IESTI represented 150% of the ARfD of 0.01 mg/kg bw. No alternative GAP was available. On the basis of information provided to the JMPR it was not possible to conclude that the estimate of short-term intake of prothioconazole, from the consumption of watermelon was less than the ARfD. The Meeting did not estimate MRL for watermelon.

The other commodities considered by the JMPR were within 0–100% of ARfD for women of child bearing age and 1% of general population. The Meeting concluded that the short-term intake of prothioconazole when used in ways that have been considered by the MPR is unlikely to present public health concern.

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Resende, G.	2013	Determinação de resíduos de Bixafen, Prothioconazole e Trifloxystrobin e seus respectivos metabólitos na cultura da Soja após aplicação de Bixafen & Prothioconazole & Trifloxystrobin 450 SC, juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil, Bayer CropScience, Sao Paulo, Brazil, Bayer CropScience, , Report No.: F13-008, Report includes Trial Nos.: F13-008-01, F13-008-02, F13-008-03, F13-008-04, F13-008-05, Edition Number: M-465974-01-1, Date: 2013-10-01, GLP/GEP: yes, unpublished.
Anon.	2013	Prothioconazole & trifloxystrobin & bixafen; 450SC (175 + 150 +125); Soybean; Brazil; BBA Bayer CropScience, Bayer CropScience, , Report No.: M-472259-01-1, Report includes Trial Nos.: F13-008-01, F13-008-02, F13-008-03, F13-008-04, F13-008-05, Edition Number: M-472259-01-1, Date: 2013-10-01, GLP/GEP: yes, unpublished
Santiago, L.	2012	Determinação de resíduos de prothioconazole e trifloxystrobin e seus respectivos metabólitos na cultura do milho após a pulverização de Fox (325 SC) juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil, Bayer S.A., Bayer CropScience, Sao Paulo, Brazil, Bayer CropScience, Report No.: F11-034, Report includes Trial Nos.: F11-034-01, F11-034-02, F11-034-03, F11-034-04, Edition Number: M-435783-01-1, Date: 2012-07-31, GLP/GEP: yes, unpublished.
Anon	2012	JAU 6476 & CGA 279202; SC 325; maize; Brazil; BBA, Bayer S.A., Bayer CropScience, Sao Paulo, Brazil, Bayer CropScience, Report No.: M-444409-01-1, Report includes Trial Nos.: F11-034-01, F11-034-02, F11-034-03, F11-034-04, F11-034-05, Edition Number: M-444409-01-1, Date: 2012-07-31, GLP/GEP: no, unpublished.
Santiago, L.	2013	Determinação de resíduos de bixafen, prothioconazole e trifloxystrobin e seus respectivos metabólitos na cultura do milho após a pulverização de bixafen & prothioconazole & trifloxystrobin 450 SC juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil, Bayer S/A, Sao Paulo, Brazil, Bayer CropScience, Report No.: F12-011, Report includes Trial Nos.: F12-011-01, F12-011-02, F12-011-03, F12-011-04, F12-011-05, Edition Number: M-448104-01-1, Date: 2013-03-01, GLP/GEP: yes, unpublished.
Anon.	2013	CGA 279202 & BYF 00587 & JAU 6476; SC 450; maize/corn; Brazil; BBA, Bayer S/A, Sao Paulo, Brazil, Bayer CropScience, Report No.: M-451489-01-1, Report includes Trial Nos.: F12-011-01, F12-011-02, F12-011-03, F12-011-04, F12-011-05, Edition Number: M-451489-01-1, Date: 2013-03-15, GLP/GEP: yes, unpublished.
Teubner, L.; Bomke, S.	2013	Determination of the residues of AE C656948 and prothioconazole in/on maize/corn after spray application of AE C656948 & JAU 6476 SE 250 in northern France, Germany, United Kingdom and Belgium, Bayer CropScience, Report No.: 11-2109, Report includes Trial Nos.: 11-2109-01, 11-2109-02, 11-2109-03, 11-2109-04, Edition Number: M-445030-01-1 Date: 2013-01-18, GLP/GEP: yes, unpublished.

Author(s)	Year	Title, Source , Report No., Date,
Freitag, T.; Reineke, A.; Krusell, L.	2010	Amendment No. 0002 to Report No. 08-2112 - Determination of the residues of BYF 00587 and prothioconazole in/on winter rape after spraying of bixafen & prothioconazole EC 225 in the field in Belgium, France (North) and Germany, Bayer CropScience, Report No.: 08-2112, Report includes Trial Nos.: 08-2112-01, 08-2112-02, 08-2112-03, 08-2112-04, Edition Number: M-385740-03-1, Date: 2010-07-07, .Amended: 2012-04-10, GLP/GEP: yes, unpublished.
Freitag, Th.; Hoffmann, M.	2010	Determination of the residues of BYF 00587 and prothioconazole in the processed fractions of winter rape (oil, screwpressed; pomace; meal; oil, solvent extracted; oil, crude; crude oil, preclarified; crude oil, neutralized and oil, refined) after spraying of Bixafen & Prothioconazole EC 225 in the field in Germany, Bayer CropScience, , Report No.: 08-3112, Report includes Trial Nos.: 08-3112-01, Edition Number: M-392874-02-1 Date: 2010-10-12, .Amended: 2011-05-02, GLP/GEP: yes, unpublished.
Krolski, M. E.; Harbin, A. M.	2008	JAU6476 480 SC - Magnitude of the residue in/on field corn processed commodities, Bayer CropScience LP, Stilwell, KS, USA, Bayer CropScience, Report No.: RAJAP001, Edition Number: M-304727-01-1, EPA MRID No.: 47521903, Date: 2008-07-25, GLP/GEP: yes, unpublished.

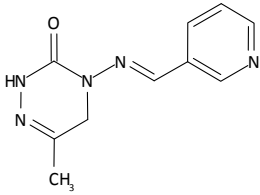
PYMETROZINE (279)

The first draft was prepared by Mr Christian Sieke, Federal Institute for Risk Assessment, Berlin, Germany

EXPLANATION

Pymetrozine is a pyridine azomethine insecticide used to control homopteran insects (aphids and whiteflies) as well as pollen beetles selectively. Although it has no knockdown effect, pymetrozine rapidly affects the feeding behaviour of the insect pests. It was considered for the first time by the 2014 JMPR for toxicology and residues.

IDENTITY

ISO common name	Pymetrozine
Chemical name	
IUPAC	(E)-4,5-dihydro-6-methyl-4-(3-pyridylmethyleneamino)-1,2,4-triazin-3(2H)-one
CA	4,5-dihydro-6-methyl-4-[(E)-(3-pyridinyl-methylene)amino]-1,2,4-triazin-3(2H)-one
CAS No.	123312-89-0
CIPAC No.	593
Structural formula	
Molecular formula	C ₁₀ H ₁₁ N ₅ O
Molecular mass	217.2 g/mol

The pymetrozine molecule contains a double bond about which E/Z isomerism is possible. However, pymetrozine technical material is manufactured by a process that yields almost exclusively the E isomer.

Specifications

Specifications for pymetrozine were not yet developed by FAO.

Physical and chemical properties

Physical and chemical properties

Property	Results	Method (test material)	Reference
Melting point	217 °C with thermal decomposition	OECD Guideline for	Rodler, 1993,
Boiling point & temperature of decomposition	Not measurable (decomposes) Decomposition starts to occur at about 190 °C	Testing of Chemicals 102 (99.4% AI)	PYMET_001
Relative density	1.37 g cm ⁻³ at 20 °C	OECD Guideline for Testing of Chemicals 109 (99.7% pure AI)	Fueldner, 1995, PYMET_002

Property	Results	Method (test material)	Reference
Vapour pressure	$< 4 \times 10^{-6}$ Pa at 25 °C	OECD Guideline for Testing of Chemicals 104A (99.4% pure AI)	Geoffroy, 1993, PYMET_003
Henry's Law Coefficient	$< 3.0 \times 10^{-6}$ Pa m ³ mol ⁻¹ (calculated)	Calculation	Burkhard, 1995, PYMET_004
Physical state, colour	White fine powder	Visual (99.7% pure AI)	Das, 1995, PYMET_005
Odour	Odourless		
Spectra active substance	<u>UV/vis</u> ϵ V/vis a L mol ⁻¹ cm ⁻¹ (245.6 nm) ϵ m) 45.6 a L mol ⁻¹ cm ⁻¹ (299.2 nm) No absorption max between 400 nm and 750 nm observed <u>IR</u> 3300–3500 cm ⁻¹ N-H stretch Approx. 3080 cm ⁻¹ aromatic C-H stretch 1660–1700 cm ⁻¹ aromatic C-C, C=N, C=O stretch 1461 cm ⁻¹ aromatic C-C 1338 cm ⁻¹ C-N stretch	UV Spectrometry IR Spectrometry (purified AI)	Muller, 1995, PYMET_006
Solubility in water including effect of pH	290 mg L ⁻¹ pH 6.5, 25 °C, pure water 320 mg L ⁻¹ pH 5.0, 25 °C, buffer 270 mg L ⁻¹ pH 7.0, 25 °C, buffer	OECD Guideline for Testing of Chemicals 105 (99.4% AI)	Stulz, 1995, PYMET_007
Solubility in organic solvents	n-hexane < 1 mg L ⁻¹ toluene 34 mg L ⁻¹ dichloromethane 1 200 mg L ⁻¹ ethanol 2400 mg L ⁻¹ n-octanol 450 mg L ⁻¹ acetone 940 mg L ⁻¹ ethyl acetate 1200 mg L ⁻¹	SOP 433.1.209 (99.4% AI)	Stulz, 1993, PYMET_008
Partition coefficient n-octanol / water	log P _{ow} -0.18, 25 °C, pure water log P _{ow} -0.24, 25 °C, buffer pH 5.0 log P _{ow} -0.19, 25 °C, buffer, pH 7.0 log P _{ow} -0.20, 25 °C, buffer pH 9.0	OECD Guideline for Testing of Chemicals 107 (99.4% AI)	Rodler, 1993, PYMET_009 & Stulz, 1995, PYMET_010
Hydrolysis rate	pH 9, 25 °C stable pH 7, 25 °C stable pH 5, 25 °C t _{1/2} 5–10 days pH 1, 25 °C t _{1/2} 2.8 hours	OECD Guideline for Testing Chemicals 111 ([Pyridine-5- ¹⁴ C]-pymetrozine, radiochemical purity 97.9%)	Kirkpatrick, 1995, PYMET_011
Photochemical degradation in water	pH 7, 25 °C (buffer) t _{1/2} 4.3–6.8 days (natural sunlight at 40°N, 12:12 photocycle)	UBA Draft Test Guideline "Phototransformation of Chemicals in Water, Berlin, FRG 1990. [Pyridine-5- ¹⁴ C]-pymetrozine, RP 97.2% [Triazine-6- ¹⁴ C]-pymetrozine, RP > 97%	Kirkpatrick, 1995, PYMET_012 & Kirkpatrick, 1995, PYMET_013
Quantum yield of direct photo-transformation	quantum yield of direct photo-k = 0.233 min ⁻¹ for E → Z isomerisation and 0.0026 min ⁻¹ for irreversible thermal decay of Z isomer	UBA Draft Test Guideline "Phototransformation of Chemicals in Water, Part A, Direct Phototransformation", Berlin, FRG 1990 (Pure AI—99.4%)	Abildt, 1994, PYMET_014

Property	Results	Method (test material)	Reference
Dissociation constant	$pK_{a,1} = 4.06$ (basic) $pK_{a,2} = < 1$ (basic)	OECD Guideline for Testing Chemicals 112 (purified AI)	Jakel, 1993, PYMET_015 & Stulz, 1995, PYMET_016
Stability in air, photochemical oxidative degradation	Estimated half-life by hydroxyl radical oxidation is between 4 and 28 hours	Calculated (Atkins method) with 1.5×10^6 OH radicals cm^{-3} and 12:12 photocycle	Stamm, 1995, PYMET_017

Formulations

Pymetrozine is primarily available as a 500 g/kg WG formulation.

METABOLISM AND ENVIROMENTAL FATE

Metabolism studies were conducted using [Pyridine-5- ^{14}C]-pymetrozine (pyridine-label) and [Triazine-6- ^{14}C]-pymetrozine (triazine-label). The position of the label for both substances is presented in the following figures:

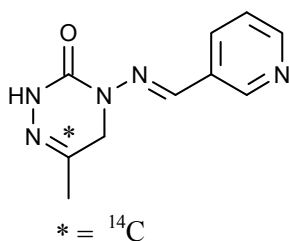


Figure 1 [Pyridine-5- ^{14}C]-pymetrozine

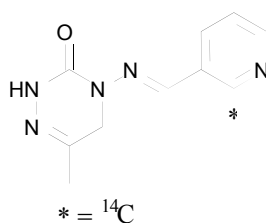


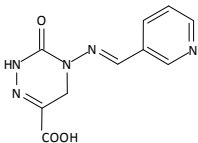
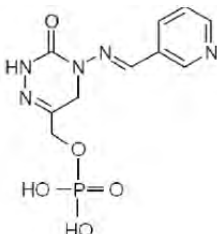
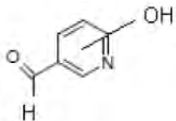
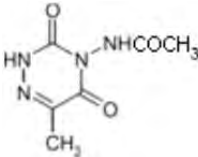
Figure 2 [Triazine-6- ^{14}C]-pymetrozine

Chemical names, structures and code names of metabolites and degradation products of pymetrozine are shown below.

Known metabolites of pymetrozine

<i>Code Names</i>	<i>Chemical Abstracts Name (IUPAC Name), molecular formula, molar mass</i>	<i>Structure</i>	<i>Where found</i>
Parent pymetrozine, CGA 215944	4,5-dihydro-6-methyl-4-[(3-pyridinylmethylene)-amino]-1,2,4-triazin-3[2H]-one		Rat, goat, hen Rice, cotton, potato, tomato, rotational crops
CGA 359009, 2U/IA12	4,5-dihydro-5-hydroxy-6-methyl-4-[(3-pyridinylmethylene)-amino]-1,2,4-triazin-3(2H)-one		Rat, goat, hen Rice, potato, tomato
CGA 323584	4,5-dihydro-5-oxy-6-methyl-4-[(3-pyridinylmethylene)-amino]-1,2,4-triazin-3(2H)-one		Rice, tomato
CGA 300407	3-pyridinecarboxaldehyde (nicotinealdehyde)		Rat, goat, hen Rice, potato, tomato
CGA 128632 incl. conjugates	3-pyridinemethanol		Hen Rice, cotton, potato, tomato, rotational crops
CGA 180777	3-pyridinecarboxylic acid (nicotinic acid)		Rat, goat, hen Rice, cotton, potato, tomato, rotational crops
CGA 180778	3-pyridinecarboxamide (nicotinamide)		Rat, goat, hen Rice, potato, tomato, rotational crops
CGA 96956	1-methyl-3-pyridinecarboxylic acid (trigonelline)		Rice, cotton, potato, tomato, rotational crops
CGA 319251	6-hydroxynicotinic acid		Tomato

Code Names	Chemical Abstracts Name (IUPAC Name), molecular formula, molar mass	Structure	Where found
CGA 294849	4-amino-6-methyl-1,2,4- triazine-3,5(2H,4H)-dione		Rat, goat, hen Rice, cotton, potato, tomato, rotational crops
GS 23199 incl. conjugates	6-methyl-1,2,4-triazine- 3,5(2H,4H)-dione		Rat, goat, hen Rice, cotton, potato, tomato, rotational crops
CGA 266591	2,3,4,5-tetrahydro-3,5-dioxo- 1,2,4-triazine-6-carboxylic acid		Rotational crops
CGA 215525	4-amino-6-methyl-1,2,4-triazin- 3(2H)-one		Rat Potato, tomato
CGA 249257	4,5-dihydro-6-methyl-1,2,4- triazin-3(2H)-one		Rat, goat, hen Rice, potato, tomato
I _{A7}	4,5-dihydro-6-methyl-4-[(3-(1- methyl-6-oxo-1,6- dihydropyridinylmethylene)- amino]-1,2,4-triazin-3(2H)-one		Hen
CGA 245342	4,5-dihydro-6-methyl-4-[(3-(1- oxy)-pyridinyl-methylene)- amino]-1,2,4-triazin-3(2H)-one		Goat
CGA 313124	4,5-dihydro-6-hydroxymethyl- 4-[(3-pyridinyl- methylene)amino]-1,2,4- triazin-3(2H)-one		Rat, goat

Code Names	Chemical Abstracts Name (IUPAC Name), molecular formula, molar mass	Structure	Where found
5U/I _{A2}	4,5-dihydro-6-carboxy-4-[(3-pyridinyl methylene)-amino]-1,2,4-triazin-3(2H)-one		Rat, goat
II _{A2}	phosphate conjugate of CGA 313124		Goat
I _{A17}	hydroxylated 3-pyridinecarboxaldehyde		Goat
CGA259168	N-(4,5-dihydro-6-methyl-3,5-dioxo-1,2,4-triazin-4(2H)-yl)-acetamide		Rat, hen

Animal metabolism

The Meeting received metabolism studies on laboratory animals, poultry and lactating goats using the pyridine- and the triazine-label of pymetrozine.

The metabolism of pymetrozine in livestock animals was intensive, showing only minor amounts of unchanged parent substance in all matrices. The major degradation steps are:

- oxidation at the triazine-methyl group leading to alcohol CGA313124, and subsequently to the corresponding carboxylic acids (A2 and 5U)
- oxidation at the triazine methylene group leading to metabolite CGA359009, and subsequently after cleavage between the triazine and pyridine ring to triazine-specific CGA 294849 and GS23199
- cleavage reaction between the triazine and pyridine ring systems leading by various reaction mechanisms to triazine-specific compounds CGA215525, CGA249257, CGA259168, and pyridine-specific metabolites CGA180777, CGA 180778 and CGA300407.

Laboratory animals

In the rat the extent of oral absorption is high (> 80%), based on urinary and biliary data. Pymetrozine is widely distributed in the body. High concentrations of both triazine- and pyridine-labelled material were found in the liver and kidney. The labelled material was rapidly excreted via urine (50–75% in 24 hours). Absorbed pymetrozine was extensively metabolized, with unmetabolized parent compound representing approximately 10% of the excreted radiolabel. Compounds containing both ring structures represented over 50% of the identified metabolites. The kinetics, excretion pattern, tissue distribution of radioactivity and metabolite profile were similar for both radiolabelled sites and administered dose levels as well as when the administration of radiolabelled pymetrozine was preceded by 14 days of administration of the unlabelled material (see WHO Monograph).

Lactating goats

The metabolic fate of [pyridine-5-¹⁴C]-pymetrozine in lactating goats was investigated by Rümbeili (1994, PYMET_018). The compound was administered to two lactating goats (35 kg bw and 49 kg bw) in gelatine capsules at 0.39 mg/kg body weight, corresponding to a level of 7.5 ppm in the feed, for four consecutive days. Excreta and milk were collected daily. The animals were slaughtered approximately 6 hours after the last dose. Muscle, omental fat, peritoneal fat, liver, kidney, blood, bile and content of gastrointestinal tract/rumen were collected.

Radioactivity was measured using liquid scintillation counting. Thin-layer chromatography, high performance liquid chromatography and high voltage electrophoresis were used to identify and characterise radioactive components in sample extracts.

The total recovery of the administered radioactivity was 86.5 %. The majority of the recovered radioactivity was found in the urine (52.4%) and faeces (14.7%). Of the administered radioactivity 3.1% were excreted via milk.

Radioactive residues in the edible tissues, muscle, omental fat, peritoneal fat, liver and kidney were between 0.03–1.95% of the administered dose. There was 0.58% of the dose remaining in leg muscle, 0.03% in tenderloin, 1.95% in liver and 0.15% in kidney.

A summary of the recovered radioactivity is presented in Table 1.

Table 1 Recovered radioactivity from goat tissues and excreta after administration of [pyridine-5-¹⁴C]-pymetrozine

Tissue	Mean radioactivity (mg/kg or mg/L pymetrozine eq.)	Mean radioactivity (% of total dose)
Milk (0–78 h)	0.326	3.14
Leg muscle	0.068	0.58
Tenderloin	0.069	0.03
Omental fat	0.026	0.03
Perirenal fat	0.032	0.02
Kidney	0.718	0.15
Liver	1.479	1.95
Faeces (0–78 h)	1.693	14.73
Urine (0–78 h)	7.927	52.37
Cage debris	–	0.65
Cage wash	–	2.43
Blood	0.069	0.25
Bile	1.303	0.02
GIT/rumen	0.669	10.18
Total excreted		73.33
Total recovery		86.54

Unchanged pymetrozine was present in small amounts ranging between 1.6% in liver tissue and 10.8% in muscle and were equivalent to 0.01 mg eq/kg or less in all samples except liver and kidney where the equivalent residues was 0.02 mg eq/kg and 0.07 mg eq/kg, respectively.

In tissue nicotinamide (CGA180778) was a major metabolite found in muscle (44.2%), fat (23.7%), liver (36.5%) and kidney (27.4%).

The hydroxylated pymetrozine derivative CGA313124 was found as a residue in muscle (10.2%), fat (6.8%), liver (3.0%) and kidney (11.3%) with residues greater than 0.01 mg eq/kg in liver and kidney. CGA313124 was also the major metabolite in milk at 36.3% of the radioactive residue. The phosphate conjugate of CGA313124 was also found in milk, accounting for an additional 38.9% of the radioactive residue.

For the composition of radioactive residues in milk and tissues please see Table 2.

Table 2 Composition of radioactive residues in goats tissues and milk after administration of [pyridine-5-¹⁴C]-pymetrozine

	Metabolite Fractions in mg eq/kg (% TRR) ^a				
	Muscle	Fat	Milk (0–78 h)	Liver ^b	Kidney
TRR	0.068	0.027	0.326	1.479	0.718
Identified					
Pymetrozine	0.0073 (10.8)	0.0013 (4.9)	0.011 (3.4)	0.024 (1.6)	0.07 (9.8)
5U/1 _{A2}	0.0008 (1.2)	0.0006 (2.2)	–	0.064 (4.3)	0.038 (5.3)
CGA180777	–	–	–	–	0.051 (7.1)
CGA313124	0.0069 (10.2)	0.0018 (6.8)	0.118 (36.3)	0.044 (3.0)	0.081 (11.3)
CGA 313124 phosphate conjugate	–	–	0.127 (38.9)	–	–
CGA180778	0.03 (44.2)	0.0064 (23.7)	0.002 (0.7)	0.54 (36.5)	0.197 (27.4)
CGA359009	–	–	0.0036 (1.1)	–	–
CGA300407	0.0006 (0.9)	–	0.0082 (2.5)	–	–
IA17	–	–	0.0039 (1.2)	0.148 (10.0)	–
Characterised					
IA1 (start)	0.0128 (18.8)	0.0015 (5.5)	–	0.232 (15.7)	0.046 (6.4)
IIA1	–	–	0.01 (3.2)	–	–
Others					
Unresolved	0.0082 (12.0)	0.0023 (8.6)	0.025 (7.7)	0.37 (25.2)	0.105 (14.6)
Hexane phase	0.0007 (1.1)	0.0106 (39.2)	0.0003 (0.1)	0.003 (0.2)	–
Sub-total	0.0675 (99.2)	0.0245 (90.9)	0.31 (95.1)	1.43 (96.5)	0.588 (81.9)
Unextracted	0.0024 (3.5)	0.0014 (5.1)	0.0095 (2.9)	0.021 (1.4)	0.089 (12.4)
Total	0.0698 (102.7)	0.0259 (96.0)	0.319 (98.0)	1.45 (97.9)	0.677 (94.3)

^a Means of both animals^b After microwave extraction

For the investigation of the stability of residues during freezer storage, samples of liver, milk and milk extract were re-analysed after 13–32 months by TLC and the pattern was compared to the first analysis:

Table 3 Storage stability of liver, milk and milk extract samples from lactating goats dosed with [pyridine-¹⁴C]-pymetrozine

Fraction	% TRR				
	Liver 9 months storage	Liver 32 months storage	Milk 10 month storage	Milk extract 13 additional month storage	Milk 23 month storage
I _{A1}	18.1	18.3	–	–	–
CGA 313124 phosphate conjugate	5.8	7.7	32.0	27.1	4.8
CGA313124	4.1	3.7	37.1	33.3	72.9
pymetrozine	2.2	2.2	3.8	6.8	4.5
CGA300407	–	–	3.5	3.5	3.3
CGA180778	49.9	49.5	–	–	–
unresolved	19.9	18.6	23.6	29.3	14.5

The metabolism of lactating goats using [triazine-6-¹⁴C]-pymetrozine was investigated by Rumbeli (1994, PYMET_019). Two animals (37 & 41 kg bw) were administered doses equivalent to 0.54 mg/kg bw or 10 ppm in the diet for a period of four consecutive days. Excreta and milk were collected daily. The animals were slaughtered approximately 6 hours after the last dose. Muscle, omental fat, peritoneal fat, liver, kidney, blood, bile and content of gastrointestinal tract/rumen were collected.

Radioactivity was measured using liquid scintillation counting. Thin-layer chromatography, high performance liquid chromatography and high voltage electrophoresis were used to identify and characterise radioactive components in sample extracts.

The total recovery of the administered radioactivity was 83.4 %. The majority of the recovered radioactivity was found in the urine (47.1%) and faeces (16.6%). Of the administered radioactivity 3.7% were excreted via milk.

Radioactive residues in the edible tissues, muscle, omental fat, peritoneal fat, liver and kidney were between 0.02–1.09% of the administered dose. There was 0.38% of the dose remaining in leg muscle, 0.02% in tenderloin, 1.09% in liver and 0.09% in kidney.

A summary of the recovered radioactivity is presented in Table 4.

Table 4 Recovered radioactivity from goat tissues and excreta after administration of [triazine-6-¹⁴C]-pymetrozine at 10 ppm

Tissue	Mean radioactivity (mg/kg or mg/L pymetrozine eq.)	Mean radioactivity (% of total dose)
Milk (0–78 h)	0.447	3.71
Leg muscle	0.072	0.38
Tenderloin	0.074	0.02
Omental fat	0.109	0.06
Perirenal fat	0.069	0.01
Kidney	0.573	0.09
Liver	1.057	1.09
Faeces (0–78 h)	2.19	16.58
Urine (0–78 h)	11.235	47.08
Cage debris	–	0.27
Cage wash	–	2.46
Blood	0.082	0.18
Bile	1.917	0.06
GIT/rumen	1.035	11.37
Total excreted		70.11
Total recovery		83.35

Unchanged pymetrozine was present in small amounts ranging between 2.3% TRR in liver tissue and 10% TRR in muscle.

In tissue the hydroxylated pymetrozine derivative CGA313124 was found as a major residue in fat (24.7% TRR) and kidney (15.1% TRR) and in concentrations of 0.051 mg eq/kg in liver. CGA313124 was also the major metabolite in milk with 40% of the TRR. The phosphate conjugate of CGA313124 was also found in milk, accounting for an additional 40.7% TRR.

In kidney, 5U/I_{A2}, which represents the carboxylic acid of pymetrozine, was also identified as a major metabolite (11.7% TRR, 0.067 mg eq/kg).

For the composition of radioactive residues in milk and tissues please refer to Table 5.

Table 5 Composition of radioactive residues in goats tissues and milk after administration of [triazine-6-¹⁴C]-pymetrozine at 10 ppm

	Metabolite Fractions in mg eq/kg (% TRR) ^a				
	Muscle	Fat	Milk (0–78 h)	Liver ^b	Kidney
TRR	0.047	0.098	0.447	1.06	0.573
Identified					
Pymetrozine	0.0047 (10)	0.0071 (7.2)	0.015 (3.3)	0.024 (2.3)	0.052 (9.0)
5U/I _{A2}	–	0.0067 (6.8)	–	0.08 (7.5)	0.067 (11.7)
CGA249257	0.0012 (2.6)	–	–	0.098 (9.2)	–
CGA245342	–	0.0012 (1.2)	–	–	–
CGA313124	0.0045 (9.5)	0.0024 (24.7)	0.179 (40)	0.051 (4.8)	0.087 (15.1)
CGA 313124 phosphate conjugate	–	–	0.182 (40.7)	–	–
CGA294849	0.0014 (3.0)	0.0024 (2.4)	–	0.032 (3.0)	0.02 (3.5)
CGA359009	–	–	0.003 (0.7)	–	–
GS23199	0.0016 (3.3)	0.0024 (2.4)	–	0.023 (2.2)	0.018 (3.2)
Characterised					
IA1 (start)	0.013 (27.6)	0.0026 (2.7)	–	0.26 (24.7)	0.064 (11.2)

	Metabolite Fractions in mg eq/kg (% TRR) ^a				
	Muscle	Fat	Milk (0–78 h)	Liver ^b	Kidney
IIA1	–	–	0.011 (2.5)	–	–
Others					
Unresolved	0.0086 (18.2)	0.034 (34.8)	0.038 (8.4)	0.45 (42.2)	0.155 (27)
Hexane phase	0.0023 (4.9)	0.0075 (7.6)	–	0.003 (0.3)	–
Sub-total	0.037 (79.1)	0.09 (92.1)	0.429 (96)	1.05 (98.8)	0.46 (80.7)
Unextracted	0.01 (22.3)	0.0046 (4.7)	0.03 (6.6)	0.01 (0.9)	0.074 (12.9)
Total	0.0477 (101.4)	0.0949 (96.8)	0.459 (102.6)	1.06 (99.7)	0.536 (93.6)

^a: Means of both animals

^b After microwave extraction

For the investigation of the stability of residues during freezer storage, samples of liver, milk and milk extract were re-analysed after 9–32 months by TLC and the pattern was compared to the first analysis:

Table 6 Storage stability of liver, milk and milk extract samples from lactating goats dosed with [triazine-¹⁴C]-pymetrozine

Fraction	% TRR				
	Liver 10 months storage	Liver 32 months storage	Milk 11 month storage	Milk extract 9 additional month storage	Milk 20 month storage
I _{A1}	7.0	13.8	–	–	–
5U/I _{A2}	15.0	16.3	–	–	–
CGA313124	9.5	12.5	–	–	–
CGA 313124 phosphate conjugate	–	–	32.7	31.9	12.5
pymetrozine	4.5	5.1	–	–	–
CGA249257	6.6	6.2	42.0	41.5	68.8
CGA294849	5.9	2.1	–	–	–
GS23199	4.3	1.1	–	–	–
unresolved	47.2	42.9	25.3	26.6	18.7

Laying hens

The metabolic fate of [pyridine-5-¹⁴C]-pymetrozine was studied in five laying leghorn hens by Rümblei (1994, PYMET_020). The compound was administered in gelatine capsules at 0.82 mg/kg body weight, corresponding to a level of 10.8 ppm in the diet, for four consecutive days. Excreta and eggs were collected daily. The animals were slaughtered approximately 6 hours after the last dose. Muscle, skin with attached fat, peritoneal fat, liver, kidney and content of gastrointestinal tract were collected.

Radioactivity was measured using liquid scintillation counting. Thin-layer chromatography, high performance liquid chromatography and high voltage electrophoresis were used to identify and characterise radioactive components in sample extracts.

The total recovery of the administered radioactivity was 79.0%. The gastrointestinal tract was not analysed, which may account for most of the remaining administered radioactivity. The majority of the radioactivity (77.0%) was found in the excreta. Radioactive residues in the edible tissues were 0.27% TRR in lean meat, 0.05% TRR in skin and fat, 0.77% TRR in liver, 0.11% TRR in kidney and 0.02% TRR in eggs. A summary of the recovered radioactivity is presented in Table 7.

Table 7 Radioactive residues in eggs and tissues after oral administration of [pyridine-5-¹⁴C]-pymetrozine for 4 consecutive days at a dose of 0.82 mg/kg body weight day (10.8 ppm)

Tissue	Mean radioactivity (mg/kg or mg/L pymetrozine eq.)	Mean radioactivity (% of total dose)
Egg white (0–78 h)	0.007	0.016
Egg yolk (0–78 h)	0.004	0.005

Tissue	Mean radioactivity (mg/kg or mg/L pymetrozine eq.)	Mean radioactivity (% of total dose)
Total egg (0–78 h)	0.006	0.021
Lean meat	0.043	0.269
Skin + fat	0.024	0.052
Peritoneal fat	0.011	0.009
Liver	0.986	0.769
Kidney	0.544	0.108
Blood	0.029	0.03
Gizzard	1.674	0.772
Excreta	–	76.261
Cage wash	–	0.758
Total excreted		77.04
Total recovery		79.048

Unchanged pymetrozine was present in very small amounts only detectable in egg white (4.9% TRR) and kidney (0.2% TRR).

In all tissues and eggs CGA180777 (nicotinic acid) & CGA180778 (nicotinamide) were the major residue present at 8.6 % TRR to 76.5% TRR.

The only other major metabolites identified were IA7 (skin + fat: 16.6% TRR, egg white: 15.4% TRR and kidney: 12.5% TRR) and CGA300407 (egg white: 11.1% TRR).

For the composition of radioactive residues in milk and tissues please refer to Table 8.

Table 8 Identification of radioactivity in hens after oral administration of [pyridine-5-¹⁴C]-pymetrozine for 4 consecutive days at a dose of 0.82 mg/kg body weight day (10.8 ppm)

	Metabolite Fractions in mg eq/kg (% TRR) ^a					
	Lean meat	Skin + fat	Egg white ^b	Egg yolk ^b	Liver	Kidney
TRR	0.043	0.019	0.011	0.010	0.927	0.519
Identified						
pymetrozine	–	–	0.001 (4.9)	–	–	0.001 (0.2)
CGA180777	–	–	–	–	0.015 (1.6)	0.336 (64.7)
IA7	0.002 (5.7)	0.008 (16.6)	0.002 (15.4)	–	0.022 (2.4)	0.065 (12.5)
CGA180778	0.033 (76.5)	0.012 (62.6)	0.008 (28.2)	0.001 (8.6)	0.652 (70.3)	0.007 (1.3)
CGA359009	–	–	–	< 0.001 (1.3)	–	–
CGA128632	–	–	< 0.001 (2.2)	–	–	–
CGA300407	–	< 0.001 (1.8)	0.001 (11.1)	–	–	–
Characterised						
IA1 (start)	< 0.001 (1.0)	0.0011 (5.9)	< 0.001 (8.1)	0.0026 (26.1)	0.044 (4.8)	0.0088 (1.7)
IA5	–	–	–	–	0.064 (6.9)	0.036 (7.0)
Others						
Unresolved	0.0034 (7.8)	0.0008 (4.2)	0.0012 (10.7)	0.0028 (27.8)	0.056 (6.0)	0.0046 (8.9)
Hexane phase	< 0.001 (1.1)	< 0.001 (1.9)	–	< 0.001 (7.8)	–	0.0016 (0.3)
Soxhlet	–	–	–	–	0.044 (4.8)	–
Sub-total	0.04 (92.1)	0.018 (93.0)	0.0089 (80.6)	0.0072 (71.6)	0.897 (96.8)	0.501 (96.6)
Unextracted	0.0029 (6.7)	0.002 (10.8)	0.001 (9.6)	0.0019 (19.1)	0.036 (3.9)	0.0078 (1.5)
Total	0.042 (98.8)	0.0197 (103.8)	0.009 (90.2)	0.0091 (90.7)	0.933 (100.7)	0.509 (98.1)

^a Mean of animals

^b 72–78 h interval

For the investigation of the stability of residues during freezer storage, samples of lean meat and liver were re-analysed after 32 months by TLC and the pattern was compared to the first analysis:

Table 9 Storage stability of lean meat and liver samples from laying hens dosed with [pyridine-¹⁴C]-pymetrozine

Fraction	% TRR			
	Lean meat 10 month storage	Lean meat 32 month storage	Liver 7 month storage	Liver 32 month storage
I _{A1}	—	—	5.2	2.7
CGA180777	—	—	1.8	1.9
I _{A5}	—	—	7.5	6.5
I _{A7}	6.3	1.0	2.6	2.9
CGA180778	85.0	82.4	76.5	78.7
unresolved	8.7	16.6	6.4	7.3

The metabolic fate of [triazine-6-¹⁴C]-pymetrozine was studied in five laying leghorn hens by Rümblei (1994, PYMET_021). The compound was administered in gelatine capsules at 0.79 mg/kg body weight, corresponding to a level of 10.4 ppm in the diet, for four consecutive days. Excreta and eggs were collected daily. The animals were slaughtered approximately 6 hours after the last dose. Muscle, skin with attached fat, peritoneal fat, liver, kidney and content of gastrointestinal tract were collected.

Radioactivity was measured using liquid scintillation counting. Thin-layer chromatography, high performance liquid chromatography and high voltage electrophoresis were used to identify and characterise radioactive components in sample extracts.

The total recovery of the administered radioactivity was 83.8%. The gastrointestinal tract was not analysed, which may account for most of the remaining administered radioactivity. The majority of the radioactivity (82%) was found in the excreta. Radioactive residues in the edible tissues were 0.14% TRR in lean meat, 0.034% TRR in skin and fat, 0.086% TRR in liver, 0.032% TRR in kidney and 0.055% TRR in eggs. A summary of the recovered radioactivity is presented in Table 10.

Table 10 Recovery of applied radioactivity in hens after oral administration of [triazine-6-¹⁴C]-pymetrozine for 4 consecutive days at a dose of 0.79 mg/kg body weight day (10.4 ppm)

Tissue	Mean radioactivity (mg/kg or mg/L pymetrozine eq.)	Mean radioactivity (% of total dose)
Egg white (0–78 h)	0.02	0.049
Egg yolk (0–78 h)	0.006	0.006
Total egg (0–78 h)	0.016	0.055
Lean meat	0.021	0.144
Skin + fat	0.019	0.034
Peritoneal fat	0.007	0.004
Liver	0.107	0.086
Kidney	0.162	0.032
Blood	0.032	0.024
Gizzard	2.5	1.471
Excreta	—	81.701
Cage wash	—	0.28
Total excreted		82.036
Total recovery		83.831

Unchanged pymetrozine was present in small amounts representing 1% to 4.8% of the TRR. In all tissues and eggs CGA259168 was the major residue present at 6.6 % TRR to 44.8% TRR. In addition I_{A7} was also a major metabolite in all matrices (5.1% to 48.5% of the TRR).

In meat and egg white CGA294849 represented 11.1% TRR and 10.4% TRR, respectively, while GS23199 was a major metabolite in egg white only (10.4% TRR).

For the composition of radioactive residues in milk and tissues please refer to Table 11.

Table 11 Identification of radioactivity in hens after oral administration of [triazine-6-¹⁴C]-pymetrozine for 4 consecutive days at a dose of 0.79 mg/kg body weight day (10.4 ppm)

	Metabolite Fractions in mg eq/kg (% TRR) ^a					
	Lean meat	Skin + fat	Egg white ^b	Egg yolk ^b	Liver	Kidney
TRR	0.021	0.015	0.025	0.013	0.106	0.162
Identified						
pymetrozine	< 0.001 (1.3)	< 0.001 (1.0)	0.001 (2.9)	–	0.002 (1.9)	0.008 (4.8)
CGA259168	0.008 (38.9)	0.004 (24.4)	0.011 (44.8)	0.002 (13.3)	0.007 (6.6)	0.018 (11.0)
I _{A7}	0.001 (5.1)	0.008 (22.3)	0.003 (11.1)	–	0.028 (26.6)	0.079 (48.5)
CGA294849	0.002 (11.1)	0.001 (6.2)	0.003 (10.4)	< 0.001 (3.5)	0.002 (2.3)	0.006 (3.9)
GS23199	< 0.001 (2.2)	< 0.001 (2.6)	< 0.001 (10.4)	< 0.001 (3.5)	0.002 (2.3)	0.006 (3.9)
Characterised						
I _{A1} (start)	0.001 (5.0)	0.0019 (12.5)	0.0026 (10.4)	< 0.001 (6.1)	0.024 (22.5)	0.0028 (1.7)
Others						
Unresolved	0.0052 (24.8)	0.0014 (9.5)	0.0025 (10)	0.0056 (44.3)	0.029 (27.1)	0.034 (20.7)
Hexane phase	< 0.001 (2.3)	< 0.001 (3.7)	< 0.001 (0.1)	< 0.001 (4.5)	0.0028 (2.6)	0.0015 (0.9)
Soxhlet	–	–	–	–	0.0051 (4.8)	–
Sub-total	0.019 (90.7)	0.012 (82.2)	0.023 (91.1)	0.0095 (73.3)	0.099 (93.6)	0.152 (93.8)
Unextracted	0.0019 (9.2)	0.0025 (16.5)	0.001 (3.9)	0.0014 (11.1)	0.0065 (6.1)	0.007 (4.4)
Total	0.021 (99.9)	0.0148 (98.7)	0.024 (95)	0.0097 (74.4)	0.106 (99.7)	0.159 (98.2)

^a Mean of animals

^b 72–78 h interval

For the investigation of the stability of residues during freezer storage, samples of lean meat and liver were re-analysed after 32 months by TLC and the pattern was compared to the first analysis:

Table 12 Storage stability of lean meat and liver samples from laying hens dosed with [triazine-¹⁴C]-pymetrozine

Fraction	% TRR			
	Lean meat 11 month storage	Lean meat 32 month storage	Liver 8 month storage	Liver 32 month storage
I _{A1}	–	–	25.9	27.7
CGA259168	46.7	30.3	7.6	8.8
I _{A7}	6.1	15.2	30.6	34.0
pymetrozine	1.5	2.0	2.2	0.9
CGA294849 + GS23199	16.0	12.1	2.6	5.9
unresolved	29.7	40.4	31.1	22.7

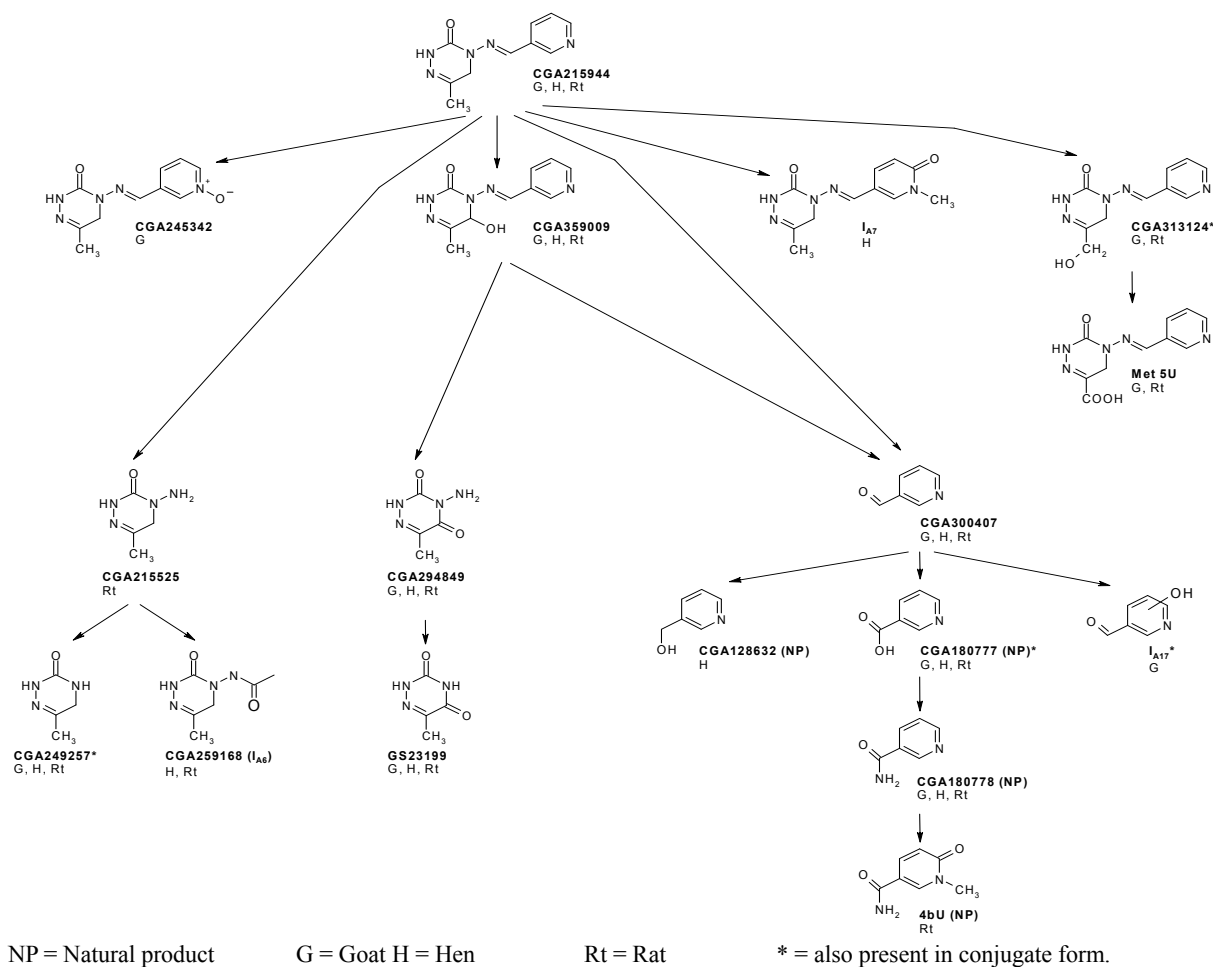


Figure 3 Metabolic pathway of pymetrozine in animals

Plant metabolism

The fate of pymetrozine in plants was investigated following foliar and granular application of [pyridine-5-¹⁴C]- and/or [triazine-6-¹⁴C]-radiolabelled active substance to tomato, potato, cotton and rice.

In the first weeks after treatment most of the residue was found on the leaf surface, consisting of unchanged parent substance. Within 4 weeks most of the residue is taken up by the plant and metabolised. Main metabolic steps are oxidation to CGA359009 and CGA323584, cleavage of the parent showing both pyridine and triazine metabolites and finally incorporation into glucose, lignin and protein. A proposed metabolic pathway scheme is presented in Figure 4.

Tomato

The metabolism of pymetrozine was investigated in tomatoes after three spray applications with [triazine-6-¹⁴C]-pymetrozine by Sandmeier (1999, PYMET_022). Fruit bearing plants were treated with rates equivalent to 0.47 kg ai/ha per application with one week intervals. The plants were protected by a plastic housing and watered by irrigation to avoid run-off of the residues. Samples (mature fruit and leaves) were harvested 3, 7 and 14 days after the last treatment. All samples were stored for a period of 6 months or less.

Tomatoes were dipped and rinsed in methanol. The washings were combined prior to analysis by radio-assay and TLC. Washed tomatoes homogenised in the presence of liquid nitrogen. The total

radioactive residues in tomatoes and leaves were determined by liquid scintillation counting after combustion of the homogenised samples.

The homogenised plant material was extracted with methanol:water (8:2). This procedure was repeated twice with tomato samples and three times with leaf samples until the last extract contained less than 5% of the radioactivity of the first extract. A Soxhlet extraction with methanol was carried out and the unextracted radioactivity was determined by combustion analysis. Characterisation and identification of radioactive components was performed with HPLC-MS using electro spray ionisation (ESI) and atmospheric pressure ionisation (APCI) techniques.

The TRR in fruit was similar at the three sampling intervals: 0.58 mg/kg pymetrozine equivalents after 3 days, 0.57 mg/kg pymetrozine equivalents after 7 days and 0.51 mg/kg pymetrozine equivalents after 14 days. The TRR in leaves decreased from 27.9 mg/kg pymetrozine equivalents, 3 days after the final treatment to 21.6 and 17 mg/kg pymetrozine equivalents after 7 and 14, respectively. Surface washings of the fruit contained 68, 59 and 48.6% of the TRR at intervals of 3, 7 and 14 days, respectively, after the final application (see Table 13).

Table 13 Summary of the distribution of radioactivity and residual [triazine-6-¹⁴C]-pymetrozine in tomato plants

Days after final application	Crop part	Total residues (mg eq/kg)	Surface radioactivity (% TRR)	Extractable radioactivity		Unextracted (% TRR)	Total (% TRR)
				Cold (% TRR)	Soxhlet (% TRR)		
3	Tomatoes	0.582	68.1	28.1	1.2	4.4	101.8
	Leaves	27.935	N/A	79.4	1.7	15.7	96.8
7	Tomatoes	0.568	59.3	34.8	1.6	5.6	101.3
	Leaves	21.585	N/A	74.8	1.7	21.1	97.6
14	Tomatoes	0.511	48.6	42.5	1.2	7.7	100.0
	Leaves	17.046	N/A	73.3	2.0	28.5	103.8

Pymetrozine was the major residue in fruits ranging from 56.8% TRR after 3 days to 31.9% TRR after 14 days. No other fraction accounted for more than 10% of TRR in fruit except CGA 294849 after 14 days (13.5% TRR, 0.0691 mg eq/kg).

Table 14 Quantification of metabolite fraction in tomato plants 3 days after the final [triazine-6-¹⁴C]-pymetrozine application

Fraction	3 days after final application			
	Fruit		Leaves	
	Washing + extract		Extract	
	% TRR	mg eq/kg	% TRR	mg eq/kg
TRR	–	0.582	–	27.935
Identified				
pymetrozine	56.8	0.3309	49.5	13.829
CGA 215525	1.8	0.0107	2.0	0.557
CGA 249257	0.3	0.0019	0.4	0.124
CGA 359009	2.8	0.0160	1.9	0.535
CGA 294849	7.4	0.0429	3.3	0.920
GS 23199	3.3	0.0189	2.3	0.634
GS 23199 n-Gluc	0.3	0.0015	1.0	0.293
CGA 323584	0.6	0.0033	0.4	0.118
Characterised				
I ₁	10.5	0.0609	4.5	1.247
I ₂	1.9	0.0112	1.1	0.317
I ₃	2.00	0.0118	1.6	0.459
I ₄	1.2	0.0069	1.6	0.439
I ₆	0.5	0.0027	0.7	0.204
I _{6a}	0.5	0.0032	0.5	0.144
I _{6b}	0.9	0.0050	0.5	0.138

Fraction	3 days after final application			
	Fruit		Leaves	
	Washing + extract		Extract	
	% TRR	mg eq/kg	% TRR	mg eq/kg
I _{10a}	—	—	1.5	0.417
I ₁₃	—	—	0.4	0.120
I _{13b}	—	—	0.2	0.055
Other				
Unresolved	5.5	0.0321	5.8	1.632
Soxhlet	1.2	0.007	1.7	0.475
Unextracted	4.4	0.0256	15.7	4.386
Total	101.8	0.582	96.8	27.935

Table 15 Quantification of metabolite fraction in tomato plants 7 days after the final [triazine-6-¹⁴C]-pymetrozine application

Fraction	7 days after final application			
	Fruit		Leaves	
	Washing + extract		Extract	
	% TRR	mg eq/kg	% TRR	mg eq/kg
TRR	—	0.568	—	21.585
Identified				
pymetrozine	50.1	0.2847	37.7	8.137
CGA 215525	1.9	0.0107	2.1	0.444
CGA 249257	0.5	0.003	0.7	0.158
CGA 359009	2.3	0.0133	2.3	0.489
CGA 294849	7.9	0.0448	4.3	0.930
GS 23199	4.0	0.0226	3.4	0.743
GS 23199 n-Gluc	0.3	0.0018	2.0	0.436
CGA 323584	0.3	0.0016	0.4	0.089
Characterised				
I ₁	11.6	0.0657	6.2	1.334
I ₂	1.9	0.0107	1.8	0.391
I ₃	2.3	0.0133	2.3	0.488
I ₄	1.7	0.0099	1.9	0.413
I ₆	0.6	0.0033	0.9	0.199
I _{6a}	0.6	0.0032	0.5	0.111
I _{6b}	0.8	0.0043	0.5	0.102
I _{10a}	—	—	1.4	0.308
I ₁₃	—	—	0.5	0.108
I _{13b}	—	—	0.2	0.042
Other				
Unresolved	7.3	0.0416	5.7	1.222
Soxhlet	1.6	0.0091	1.7	0.367
Unextracted	5.6	0.0318	21.1	4.554
Total	101.3	0.568	97.6	21.585

Table 16 Quantification of metabolite fraction in tomato plants 14 days after the final [triazine-6-¹⁴C]-pymetrozine application

Fraction	14 days after final application			
	Fruit		Leaves	
	Washing + extract		Extract	
	% TRR	mg eq/kg	% TRR	mg eq/kg
TRR	—	0.511	—	17.046
Identified				
pymetrozine	31.9	0.1631	31.3	5.333
CGA 215525	2.0	0.0102	1.6	0.270
CGA 249257	0.7	0.0033	0.6	0.104
CGA 359009	2.3	0.0115	1.5	0.261
CGA 294849	13.5	0.0691	3.7	0.631

Fraction	14 days after final application			
	Fruit		Leaves	
	Washing + extract		Extract	
	% TRR	mg eq/kg	% TRR	mg eq/kg
GS 23199	6.8	0.035	3.7	0.628
GS 23199 n-Gluc	0.7	0.0034	2.9	0.496
CGA 323584	0.2	0.0011	0.4	0.070
Characterised				
I ₁ (start)	14.1	0.0719	8.0	1.358
I ₂	3.5	0.0178	2.3	0.386
I ₃	3.3	0.017	3.1	0.530
I ₄	2.0	0.0104	2.5	0.434
I ₆	1.3	0.0065	1.2	0.204
I _{6a}	0.8	0.0039	0.6	0.099
I _{6b}	1.0	0.0051	0.7	0.122
I _{10a}	–	–	1.6	0.269
I ₁₃	–	–	0.1	0.110
I _{13b}	–	–	0.1	0.019
Other				
Unresolved	7.1	0.0362	6.9	1.173
Soxhlet	1.2	0.0061	2.0	0.341
Unextracted	7.7	0.0393	28.5	4.858
Total	100.0	0.511	103.8	17.046

In an older set of studies also conducted by Sandmeier (1992, PYMET_023 & 1993, PYMET_024) the distribution and degradation of [triazine-6-¹⁴C] pymetrozine was studied in field grown tomatoes after foliar application. Three months old tomato plants (with green tomatoes, approx. 2 cm in diameter) were twice treated in an interval of 7 days with a WP formulation at a rate of about 0.25 kg ai/ha each.

The sampling followed two strategies. In a short-term experiment plant material (tomatoes and leaves) was sampled about 1 and 4 hours; 1, 2 and 7 days after the last application. The long-term experiment involved sampling 26 and 49 days. All samples were analysed within 22 months.

Leaves and tomatoes were shortly washed with MeOH/H₂O. After cold extraction a soxhlet extraction with methanol was performed. The unextracted radioactivity was determined by combustion of the dry material. The radioactivity was measured with a scintillation counter or by autoradiography. Besides the TLC Liquid Chromatography, HPLC, chemical reactions and enzyme cleavage were used to characterise fractions. Quantification of metabolites was performed by 2D TLC.

In the short-term experiment about 50% of the initial radioactivity was found after seven days in the treated leaves and fruits. Most of the residue was present on the surface consisting of unchanged parent substance. However, a quick and steady absorption into the plants was observed. In the extracts, the amount of pymetrozine was lower than on the surface, indicating a quick degradation within the plants. An overview of the radioactivity found in tomato samples is presented below. All samples were analysed only for the amount of pymetrozine present.

Table 17 Penetration of radioactivity and residual [triazine-6-¹⁴C]-pymetrozine in tomato leaves and fruits

Interval	Plant part	Total residue	Pymetrozine	Surface wash		Extracts			Total
		mg eq/kg	mg/kg	% TRR	% pymetrozine	% TRR	% pymetrozine	% unextracted	% TRR
1 hr	leaves	20.442	16.178	91.3	74.5	6.0	4.6	2.8	100.1
	fruits	1.221	1.05	96.8	83.4	3.1	2.5	0.5	100.4
4 hrs	leaves	21.807	15.181	93.2	66.7	4.8	2.9	2.3	100.3
	fruits	0.953	0.727	91.8	71.9	5.9	4.3	1.9	99.6
1 day	leaves	19.421	12.279	80.2	57.2	12.7	6.02	7.7	100.6
	fruits	0.87	0.611	82.9	60.9	14.8	9.3	3.7	101.4

2 days	leaves	17.733	10.215	76.9	52.4	12.9	5.2	7.4	97.2
	fruits	0.384	0.255	73.7	49.4	19.1	9.1	5.2	98.0
7 days	leaves	10.717	3.232	52.1	25.7	21.9	4.4	23.4	97.4
	fruits	0.255	0.092	47.1	24.1	41.6	11.9	12.7	101.4

In the long-term experiment the composition of the radioactive residues in tomato leaves and fruits was investigated. After 26 and 49 days the parent substance was extensively metabolized. Although pymetrozine was still present as the only major metabolite in treated plant parts (8.6–17.2% TRR), most of the recovered radioactivity was distributed into minor metabolites or characterised.

Table 18 Quantification of metabolite fractions in various tomato plant parts after spray application of [triazine-6-¹⁴C]-pymetrozine

Fraction	Metabolite Fractions in mg eq/kg (% TRR)							
	26 days				49 days			
	upper fruit, newly grown	treated lower fruit	upper leaves, newly grown	treated lower leaves	upper fruit, newly grown	treated lower fruit	upper leaves, newly grown	treated lower leaves
TRR	0.025	0.355	0.087	13.3	0.053	0.229	1.35	6.37
Identified								
pymetrozine	–	0.061 (17.3)	–	1.622 (12.2)	< 0.001 (0.3)	0.022 (9.8)	0.7 (7.2)	0.547 (8.6)
CGA215525	–	0.003 (0.9)	–	0.146 (1.1)	–	< 0.001 (0.3)	–	0.012 (0.2)
CGA249257	–	0.005 (1.4)	–	0.16 (1.2)	0.001 (2.2)	0.005 (2.1)	0.016 (1.2)	0.089 (1.4)
CGA359009	–	< 0.01 (1.9)	–	0.239 (1.8)	0.005 (8.9)	< 0.01 (1.4)	0.052 (3.9)	0.089 (1.4)
CGA294849	–	0.03 (8.5)	–	0.279 (2.1)		0.02 (8.9)		0.121 (1.9)
GS23199	–	0.018 (5.2)	–	0.439 (3.3)		0.016 (7.1)		0.133 (2.1)
GS23199 n-Gluc	–	0.005 (1.5)	–	0.611 (4.6)	0.001 (2.2)	0.004 (1.8)	0.116 (8.6)	0.42 (6.6)
Characterised								
I ₁ (start)	–	0.079 (22.2)	–	1.82 (13.7)	0.025 (47.4)	0.063 (27.5)	0.163 (12.1)	0.815 (12.8)
I ₂	–	< 0.01 (2.1)	–	0.279 (2.1)	< 0.01 (2.2)	< 0.01 (1.8)	< 0.01 (0.3)	0.267 (4.2)
I ₃	–	< 0.01 (2.2)	–		< 0.01 (1.3)	< 0.01 (1.5)		
I ₄	–	< 0.01 (0.7)	–	0.1 (0.8)	–	< 0.01 (0.4)	–	0.03 (0.5)
I ₆	–	< 0.01 (1.1)	–	0.12 (0.9)	< 0.01 (4.9)	< 0.01 (0.7)	0.02 (1.8)	0.08 (1.3)
I ₁₃	–	< 0.01 (0.5)	–	0.252 (1.9)	–	< 0.01 (0.4)	< 0.01 (0.5)	0.7 (1.1)
Soxhlet & unresolved	–	0.062 (17.4)	–	2.42 (18.2)	0.0086 (16.2)	0.04 (17.3)	0.204 (15.1)	0.81 (12.7)
Unextracted	–	0.045 (12.7)	–	4.56 (34.3)	0.008 (15.3)	0.034 (15)	0.62 (45.8)	2.8 (44.2)
Total	0.025 (100)	0.34 (95.6)	0.087 (100)	13.1 (98.2)	0.053 (101)	0.22 (96)	1.3 (96.5)	6.3 (99)

Distribution and degradation of [pyridine-5-¹⁴C]-pymetrozine were studied by Gross (1994, PYMET_025) in field-grown tomatoes protected by a plastic housing. Three month old tomato plants (with green tomatoes approximately 2 cm diameter) were treated twice at 14 days interval with approximately 0.25 kg ai/ha formulated in a WP product. Whole plants were sampled approximately 3 hours and 15 days after the first application, and at 1 hour, 7 days (first mature fruit) and 27 days (maturity) after the second application.

Tomatoes were rinsed in methanol:water (1:1 v/v). The total radioactive residues were determined by liquid scintillation counting after combustion of the homogenised samples. The homogenised plant material was extracted thrice with methanol:water (8:2). The combined extracts were used for determination of the parent by TLC. A Soxhlet extraction with methanol was also carried out and the unextracted radioactivity was determined by combustion analysis.

Characterisation and identification of radioactive components was performed with TLC, ion exchange LC, HPLC using authentic standards of metabolites as reference compounds, and GC-MS.

The first mature tomato fruits were harvested 7 days after the second treatment. Total residues were 0.229 mg eq/kg with a parent content of 0.072 mg eq/kg. Total residues in leaves were 7.432 mg eq/kg with a parent content of 2.221 mg eq/kg.

At harvest (27 days after the second treatment), total residues reached 0.173 mg eq/kg in tomato fruit and 2.433 mg eq/kg in leaves. Parent residues were 0.016 mg eq/kg and 0.286 mg eq/kg in fruit and leaves respectively. The radioactivity of the surface of tomato fruit decreased from nearly 90% 1 hour after the first treatment to less than 10% 15 days later.

An overview of the total radioactive residues and their extractability is presented in Table 19.

Table 19 Summary of the distribution of radioactivity and residual [pyridine-5-¹⁴C]-pymetrozine in tomato plants

Days after final application	Crop part	Total residues (mg eq/kg)	Pymetrozine (mg eq/kg)	Surface radioactivity (% TRR)	Extractable radioactivity		Unextracted (% TRR)	Total (% TRR)
					Cold (% TRR)	Soxhlet (% TRR)		
3 h after 1 st application	Leaves	10.837	6.440	88.5	78.6	4.1	9.5	92.2
	Fruit	0.538	0.461		9.6	0.1	0.8	99.0
15 days after first application	Leaves	3.823	0.665	9.7	79.1	1.1	14.3	94.5
	Fruit	0.131	0.015		84.7	0.5	3.7	98.6
15 days (1 h after 2 nd application)	Leaves	13.608	7.773	82.7	80.0	4.3	7.5	91.8
	Fruit	1.029	0.829		15.6	0.1	0.7	99.1
23 days (7 days after 2 nd application)	Leaves	7.432	2.221	25.0	79.9	1.2	13.7	94.8
	Fruit	0.229	0.072		68.1	0.8	5.4	99.3
42 days (27 days after 2 nd application)	Leaves	2.433	0.286	5.8	76.4	1.1	17.1	94.6
	Fruit	0.173	0.016		81.9	0.4	4.2	92.3

The composition of radioactive residues showed unchanged pymetrozine as the only significant residue directly after application. Most of the parent was located on the fruit and leaf surface. Within the first days after treatment the active substance is absorbed and in plants degraded into various minor metabolites. The major metabolite identified was CGA96956, representing up to 70% of the TRR in fruits and leaves. The only other major metabolite found in fruits and leaves was CGA128632 present up to 19.5% of the TRR.

Table 20 Quantification of metabolite fraction in tomato plants 3 hours and 15 days after first application of [pyridine-6-¹⁴C]-pymetrozine

Fraction	3 h after the first application				15 days after the first application			
	Fruit			Leaves	Fruit			Leaves
	Surface	Extract	Total		Surface	Extract	Total	
	% TRR	% TRR	mg eq/kg (% TRR)	mg eq/kg (% TRR)	% TRR	% TRR	mg eq/kg (% TRR)	mg eq/kg (% TRR)
TRR			0.538	10.837			0.131	3.823
Identified								

Fraction	3 h after the first application				15 days after the first application			
	Fruit			Leaves	Fruit			Leaves
	Surface	Extract	Total		Surface	Extract	Total	
	% TRR	% TRR	mg eq/kg (% TRR)	mg eq/kg (% TRR)	% TRR	% TRR	mg eq/kg (% TRR)	mg eq/kg (% TRR)
pymetrozine	78.4	6.4	0.456 (84.8)	6.55 (60.4)	5.9	3.8	0.013 (9.7)	0.62 (16.3)
CGA96956	–	1.0	0.005 (1.0)	0.78 (7.2)	1.6	68.5	0.092 (70.1)	1.0 (26.4)
CGA128632 (sugar conj)	–	0.6	0.003 (0.6)	0.52 (4.8)	–	8.6	0.011 (8.6)	0.25 (6.6)
CGA319251	–	–	–	0.04 (0.4)	–	–	–	0.057 (1.5)
CGA180777	–	–	–	–	0.4	–	< 0.001 (0.4)	–
CGA180778	–	–	–	–	–	0.8	< 0.001 (0.6)	–
CGA128632	–	1.2	0.006 (1.2)	0.23 (2.1)	–	0.8	0.001 (0.8)	0.092 (2.4)
CGA300407	3.5	0.2	0.02 (3.7)	0.16 (1.5)	0.2	–	< 0.001 (0.2)	0.027 (0.7)
Characterised								
IP2a	–	–	–	–	0.3	–	< 0.001 (0.3)	–
IP5	–	–	–	–	–	–	–	–
IP8	–	–	–	–	–	0.8	0.001 (0.8)	–
2U	2.7	–	0.015 (2.7)	–	0.5	–	< 0.001 (0.5)	–
Unresolved	2.7	0.2	0.016 (2.9)	0.28 (2.6)	0.7	1.5	0.003 (2.2)	0.15 (3.9)
Soxhlet	–	0.1	< 0.001 (0.1)	0.44 (4.1)	–	0.5	< 0.001 (0.5)	0.042 (1.1)
Unextracted	–	0.8	0.004 (0.8)	1.03 (9.5)	–	3.7	0.005 (3.7)	0.55 (14.3)
Total			0.526 (97.8)	10.04 (92.6)			0.129 (98.4)	3.61 (94.5)

Table 21 Quantification of metabolite fraction in tomato plants 1 hour and 7 days after second application of [pyridine-6-¹⁴C]-pymetrozine

Fraction	1 h after the second application				7 days after the second application			
	Fruit			Leaves	Fruit			Leaves
	Surface	Extract	Total		Surface	Extract	Total	
	% TRR	% TRR	mg eq/kg (% TRR)	mg eq/kg (% TRR)	% TRR	% TRR	mg eq/kg (% TRR)	mg eq/kg (% TRR)
TRR			1.029	13.608			0.229	7.432
Identified								
pymetrozine	77.8	2.3	0.824 (80.1)	7.96 (58.5)	10.1	10.8	0.068 (29.9)	2.1 (28.2)
CGA96956	0.7	11.8	0.129 (12.5)	1.27 (9.3)	1.2	39.3	0.093 (40.5)	1.76 (23.7)
CGA128632 (sugar conj)	–	0.7	0.007 (0.7)	0.83 (6.1)	–	11.5	0.026 (11.5)	1.4 (19.1)
CGA319251	–	–	–	0.12 (0.9)	–	–	–	0.13 (1.8)
CGA180777	–	–	–	–	0.9	–	0.002 (0.9)	–
CGA180778	–	0.1	0.001 (0.1)	–	–	1.0	0.002 (1.0)	–
CGA128632	–	0.3	0.003 (0.3)	0.19 (1.4)	–	2.0	0.005 (2.0)	0.14 (1.9)
CGA300407	1.7	0.1	0.019 (1.8)	0.22 (1.6)	0.6	1.3	0.005 (1.9)	0.052 (0.7)
Characterised								
IP5	–	–	–	–	0.2	–	< 0.001 (0.2)	–
IP8	–	0.1	0.001 (0.1)	–	–	0.4	< 0.001 (0.4)	–
2U	1.0	–	0.01 (1.0)	–	1.3	–	0.003 (1.3)	–
Unresolved	1.5	0.2	0.017 (1.7)	0.3 (2.2)	1.5	1.8	0.008 (3.3)	0.31 (4.2)
Soxhlet	–	0.1	0.001 (0.1)	0.59 (4.3)	–	0.8	0.002 (0.8)	0.089 (1.2)
Unextracted	–	0.7	0.007 (0.7)	1.02 (7.5)	–	5.4	0.012 (5.4)	1.02 (13.7)

Fraction	1 h after the second application				7 days after the second application			
	Fruit			Leaves	Fruit			Leaves
	Surface	Extract	Total		Surface	Extract	Total	
	% TRR	% TRR	mg eq/kg (% TRR)	mg eq/kg (% TRR)	% TRR	% TRR	mg eq/kg (% TRR)	mg eq/kg (% TRR)
Total			1.02 (99.1)	12.49 (91.8)			0.227 (99.1)	7.02 (94.5)

Table 22 Quantification of metabolite fraction in tomato plants 27 days after second application of [pyridine-6-¹⁴C]-pymetrozine

Fraction	27 d after the second application			
	Fruit			Leaves
	Surface	Extract	Total	
	% TRR	% TRR	mg eq/kg (% TRR)	mg eq/kg (% TRR)
TRR			0.173	2.443
Identified				
pymetrozine	3.3	3.5	0.012 (6.8)	0.256 (10.5)
CGA96956	1.1	64.0	0.11 (65.1)	0.80 (32.9)
CGA128632 (sugar conj)	–	–	0.013 (7.6)	0.65 (8.6)
CGA319251	–	–	–	0.044 (1.8)
CGA180777	0.3	–	< 0.001 (0.3)	–
CGA180778	–	1.0	0.002 (1.0)	–
CGA128632	–	1.1	0.002 (1.1)	0.042 (1.7)
CGA300407	0.1	–	< 0.001 (0.1)	0.012 (0.5)
Characterised				
IP2a	0.2	–	< 0.001 (0.2)	–
IP5	0.1	–	< 0.001 (0.1)	–
IP8	–	0.3	0.001 (0.8)	–
2U	0.2	–	< 0.001 (0.2)	–
Unresolved	0.4	3.8	0.007 (4.2)	0.068 (2.8)
Soxhlet	–	0.4	< 0.001 (0.4)	0.027 (1.1)
Unextracted	–	4.2	0.007 (4.2)	0.418 (17.1)
Total			0.159 (92.1)	2.31 (94.7)

Potato

For the investigation of the plant metabolism of pymetrozine in potato a study was conducted by Fleischmann (2000, PYMET_026) involving treatment either with [triazine-¹⁴C]-pymetrozine or [pyridine-¹⁴C]-pymetrozine formulated as a WP product. The plants were treated with three foliar sprays in one week intervals. The applications using [triazine-¹⁴C]- and [pyridine-¹⁴C]-pymetrozine were conducted with 0.15 kg ai/ha each (total rate 0.45 kg ai/ha) or at exaggerated rates of 1.05 kg ai/ha each (total rate 3.1 kg ai/ha). The first of the three foliar spray applications was 61 days after planting. Potato foliage and tubers were harvested 7, 14 and 29 days after the third application.

Samples were extracted with acetonitrile:water (8:2) or methanol:water (8:2) and purified using C₁₈ SPE techniques. Unextracted radioactivity in tubers were refluxed with neutral solvent acetonitrile/water or methanol/water. Additional radioactive residues were released after treatment with enzymes and then refluxing with acid or an alternative sequence refluxing with H₂O and then refluxing with base. Extracts were characterised by reverse phase HPLC and 2D-TLC. Radioactivity in liquid samples and extracts were determined by LSC.

The TRR in potatoes treated at a rate of 3 × 0.15 kg ai/ha with [triazine-¹⁴C]-pymetrozine were 0.13, 0.12 and 0.11 mg eq/kg pymetrozine equivalents in tubers and 11.7, 8.5 and 6.4 mg eq/kg pymetrozine equivalents in foliage harvested at 7, 14 and 29 days after the final application, respectively. The TRR in potatoes treated with rates of 3 × 1.05 kg ai/ha were 0.34, 0.41 and 0.37 mg eq/kg in tubers and 46.2, 37.2 and 40.4 mg eq/kg in foliage harvested after 7, 14 and 29 days, respectively.

Potatoes treated at a rate of 3×0.15 kg ai/ha with [pyridine- ^{14}C]-pymetrozine had TRRs of 0.26, 0.31 and 0.36 mg eq/kg in tubers and 8.2, 6.8 and 7.8 mg eq/kg in foliage harvested at 7, 14, and 29 days, respectively. The treatments with 3×1.05 kg ai/ha using [pyridine- ^{14}C]-pymetrozine resulted in tuber residues of 0.8, 0.8 and 1.1 mg eq/kg and foliage residues of 29.4, 31.4 and 35.6 mg eq/kg at the same respective harvest intervals.

A summary of the total radioactive residues is presented in Table 23.

Table 23 Summary of extraction of potato samples following treatment with [pyridine-5- ^{14}C]- and [triazine-6- ^{14}C]-pymetrozine

Substrate	Interval	TRR	Extracted residues (ACN:H ₂ O)		Extracted residues (MeOH:H ₂ O)		Unextracted		Total
		mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Triazine- ¹⁴ C-label									
Tubers, 3 × 0.15	7 DAT	0.128	65.8	0.084	7.87	0.01	30.66	0.039	104.33
kg ai/ha	14 DAT	0.117	59.38	0.069	8.17	0.01	34.48	0.04	102.03
	29 DAT	0.112	62.82	0.07	6.48	0.007	31.83	0.036	101.13
Foliage, 3 × 0.15	7 DAT	11.736	51.83	6.083	—	—	43.32	5.084	95.15
kg ai/ha	14 DAT	8.542	55.81	4.767	—	—	37.82	3.307	94.52
	29 DAT	6.43	50.07	3.22	—	—	45.35	2.916	95.42
Tubers, 3 × 1.05	7 DAT	0.343	63.11	0.216	5.12	0.018	31.25	0.107	99.48
kg ai/ha	14 DAT	0.412	70.25	0.289	3.79	0.016	30.29	0.125	104.33
	29 DAT	0.369	68.33	0.252	3.39	0.013	37.16	0.137	108.88
Foliage, 3 × .105	7 DAT	46.249	44.19	20.437	14.54	6.723	38.18	17.66	96.91
kg ai/ha	14 DAT	37.205	51.33	19.096	9.73	3.622	36.91	13.733	97.97
	29 DAT	40.366	49.64	20.037	12.56	5.068	41.37	16.7	103.57
Pyridine- ¹⁴ C-label									
Tubers, 3 × 0.15	7 DAT	0.259	86.62	0.224	8.43	0.022	4.38	0.011	99.43
kg ai/ha	14 DAT	0.312	91.56	0.286	5.42	0.017	5.06	0.016	102.04
	29 DAT	0.356	98.82	0.352	—	—	6.19	0.022	105.01
Foliage, 3 × 0.15	7 DAT	8.186	38.02	3.112	—	—	59.86	4.9	97.88
kg ai/ha	14 DAT	6.826	38.7	2.642	—	—	62.32	4.254	101.02
	29 DAT	7.756	42.57	3.302	—	—	55.2	4.281	97.77
Tubers, 3 × 1.05	7 DAT	0.8	85.3	0.682	16.89	0.135	3.67	0.029	105.86
kg ai/ha	14 DAT	0.848	90.15	0.764	8.86	0.075	5.34	0.045	104.35
	29 DAT	1.072	96.57	1.035	7.14	0.077	4.72	0.051	108.43
Foliage, 3 × .105	7 DAT	29.38	51.0	14.984	—	—	53.73	15.786	104.73
kg ai/ha	14 DAT	31.405	41.7	13.096	—	—	60.53	19.009	102.23
	29 DAT	35.552	35.54	12.635	—	—	70.18	24.95	105.72

DAT: days after treatment

The triazine radiolabelled tuber extracts showed a complex mixture of aqueous soluble polar moieties separated into at least 11 fractions by reverse phase HPLC. Approximately 30% of the tuber TRR eluted in four early eluting HPLC regions. The largest early eluting HPLC region (T3) was 13% to 15% TRR and was further characterised as a mixture of multiple components.

In tubers the major metabolites identified were pymetrozine (1–4% TRR), GS-23199 (approximately 2% TRR) and its glycoside conjugate (3–5% TRR), CGA-249257 (1–2% TRR) and CGA-294849 (1–3% TRR). The major residues in foliage extracts were pymetrozine (3–9% TRR) and the glycoside of GS-23199 (9–16% TRR). Other identified components in the foliage were GS-23199 (1–2% TRR), CGA-249257 (1–2% TRR) and CGA-294849 (1–3% TRR).

The pyridine radiolabelled residues from tubers were primarily identified as CGA96956 (“trigonelline”, 54–75% TRR), monosaccharide conjugates of CGA-180777 (12–22% TRR), CGA-180777 (1–5% TRR), CGA-180778 (< 2% TRR), CGA-128632 (approximately 1% TRR) and pymetrozine ($\leq$ 2% TRR).

The major residues in foliage were identified as CGA96956 ("trigonelline", 3–6% TRR), CGA180777 (< 1% TRR), CGA180778 (< 2% TRR), glycoside conjugate of CGA128632 (10–17% TRR), CGA128632 ($\leq$ 2% TRR) and pymetrozine (5–18% TRR).

Table 24 Distribution of metabolites in potato tubers and foliage after application of [triazine-6-¹⁴C]-pymetrozine at rates of 3×0.15 kg ai/ha

Fraction	Tuber						Foliage					
	7 day DAT		14 day DAT		29 day DAT		7 day DAT		14 day DAT		29 day DAT	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
TRR		0.128		0.117		0.112		11.736		8.542		6.43
Identified												
pymetrozine	4.0	0.005	1.1	0.001	1.2	0.001	6.7	0.790	8.7	0.741	6.3	0.402
CGA294849 & CGA215525	2.0	0.003	1.1	0.001	2.8	0.003	1.5	0.175	2.8	0.243	1.1	0.070
CGA249257	2.0	0.003	1.0	0.001	0.72	0.001	1.4	0.163	1.5	0.126	0.55	0.035
GS23199	2.0	0.003	1.9	0.002	1.8	0.002	1.5	0.178	1.4	0.119	1.8	0.114
GS23199 gluc.	3.9	0.005	3.2	0.004	5.0	0.006	9.2	1.080	13.1	1.120	15.3	0.984
Characterised												
T1	6.6	0.008	5.1	0.006	7.3	0.008	0.35	0.041	0.95	0.081	0.65	0.042
T2	6.1	0.008	6.0	0.007	8.2	0.009	0.32	0.038	0.35	0.030	0.69	0.044
T3 ^a	13.0	0.017	11.5	0.013	12.4	0.014	1.1	0.125	1.2	0.105	1.4	0.087
T4	7.5	0.010	5.5	0.006	3.5	0.004	2.1	0.249	2.0	0.170	1.3	0.085
T5	–	–	1.1	0.001	1.3	0.002	–	–	–	–	–	–
T6	3.0	0.004	1.7	0.002	2.3	0.003	0.60	0.070	0.65	0.056	2.7	0.174
T7	0.78	0.001	0.63	0.001	0.62	0.001	0.20	0.024	0.37	0.032	–	–
T9	–	–	–	–	–	–	–	–	–	0.000	0.92	0.059
T11	0.93	0.001	0.88	0.001	0.43	0.000	1.8	0.211	0.56	0.048	0.33	0.021
T12	–	–	0.37	0.000	0.69	0.001	–	–	0.42	0.036	–	–
T14	1.0	0.001	0.53	0.001	0.62	0.001	–	–	0.43	0.037	–	–
T15	–	–	–	–	–	–	–	–	0.85	0.072	–	–
T17	0.84	0.001	1.5	0.002	0.95	0.001	1.6	0.193	3.0	0.255	2.5	0.163
T18	0.60	0.001	1.2	0.001	2.3	0.003	4.1	0.476	2.6	0.223	0.85	0.055
T19	1.2	0.001	3.0	0.004	1.8	0.002	3.0	0.352	2.1	0.179	3.3	0.210
T21	2.6	0.003	0.35	0.000	0.31	0.000	2.9	0.334	1.7	0.147	4.7	0.304
T22	0.38	0.000	0.27	0.000	0.27	0.000	1.5	0.174	0.84	0.072	–	–
T23	–	–	0.22	0.000	–	–	–	–	–	–	–	–
T24	1.4	0.002	0.51	0.001	0.63	0.001	2.8	0.325	4.4	0.377	3.3	0.212
T25	0.24	0.000	–	–	0.12	0.000	0.9	0.106	1.1	0.095	0.44	0.028
T26	–	–	–	–	–	–	0.8	0.094	0.79	0.067	0.5	0.032
T27	–	–	–	–	–	–	0.91	0.107	–	–	–	–
Other												
Unextracted	39.1	0.050	42.2	0.049	43.5	0.049	–	–	–	–	–	–
MeOH:H ₂ O Reflux	7.9	0.010	8.2	0.010	6.5	0.007	–	–	–	–	–	–
Final unextracted	30.7	0.039	34.5	0.040	31.8	0.036	43.3	5.1	38.7	3.1	45.4	2.916
Total characterised	73.6	0.094	61.6	0.072	60.9	0.068	50.3	5.9	53.3	4.6	49.1	3.16
Total identified	13.9	0.018	8.3	0.01	11.5	0.013	20.3	2.4	27.5	2.4	25.0	1.6

^a Characterised as multiple components

Table 25 Distribution of metabolites in potato tubers and foliage after application of [triazine-6-¹⁴C]-pymetrozine at rates of 3 × 1.05 kg ai/ha

Fraction	Tuber						Foliage					
	7 day DAT		14 day DAT		29 day DAT		7 day DAT		14 day DAT		29 day DAT	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
TRR		0.343		0.412		0.369		46.249		37.205		40.366
Identified												
pymetrozine	3.5	0.012	4.9	0.020	0.17	0.001	10.3	4.740	9.2	3.408	7.0	2.812
CGA294849 & CGA215525	2.0	0.007	0.64	0.003	2.1	0.008	2.8	1.312	2.8	1.037	1.9	0.755
CGA249257	2.4	0.008	0.83	0.003	2.4	0.009	1.0	0.479	1.8	0.655	1.4	0.576
GS23199	2.7	0.009	3.0	0.012	2.2	0.008	2.0	0.942	2.4	0.907	1.9	0.777
GS23199 gluc.	5.1	0.017	5.4	0.022	5.7	0.021	7.6	3.534	11.8	4.374	16.1	6.482
Characterised												
T1	6.1	0.021	9.0	0.037	11.2	0.041	0.25	0.112	0.66	0.244	1.0	0.408
T2	6.9	0.024	7.2	0.030	9.2	0.034	0.12	0.058	0.32	0.119	0.39	0.158
T3 ^a	15.0	0.051	12.3	0.051	13.5	0.050	0.88	0.407	1.4	0.527	1.4	0.572
T4	5.5	0.019	1.4	0.006	3.9	0.014	2.6	1.196	2.9	1.083	1.3	0.516
T5	—	—	3.6	0.015	—	—	—	—	—	—	—	—
T6	2.8	0.010	3.3	0.014	2.0	0.007	0.36	0.167	0.84	0.313	1.5	0.606
T7	0.56	0.002	0.54	0.002	2.1	0.008	—	—	0.37	0.136	—	—
T9	—	—	—	—	0.58	0.002	—	—	—	—	—	—
T11	0.55	0.002	2.4	0.010	0.74	0.003	—	—	0.15	0.056	0.12	0.048
T12	0.33	0.001	0.28	0.001	1.0	0.004	—	—	—	—	—	—
T14	0.68	0.002	0.65	0.003	0.98	0.004	—	—	0.50	0.186	1.9	0.773
T15	—	—	—	—	0.78	0.003	—	—	—	—	—	—
T17	1.4	0.005	1.4	0.006	—	—	1.8	0.812	2.3	0.851	0.99	0.399
T18	1.0	0.003	1.9	0.008	0.52	0.002	—	—	1.9	0.710	1.1	0.438
T19	4.2	0.015	3.9	0.016	—	—	1.2	0.574	2.3	0.849	1.2	0.496
T21	—	—	1.6	0.007	—	—	2.2	1.013	1.3	0.483	2.9	1.157
T22	—	—	0.74	0.003	0.16	0.001	0.64	0.296	0.93	0.344	—	—
T23	—	—	—	—	—	—	1.3	0.587	—	—	—	—
T24	0.93	0.003	1.1	0.005	—	—	1.3	0.610	1.1	0.397	3.2	1.295
T25	—	—	—	—	—	—	—	—	1.5	0.540	0.60	0.240
T26	—	—	—	—	—	—	—	—	—	—	0.75	0.304
T27	—	—	—	—	—	—	—	—	—	—	0.64	0.257
Other												
Unextracted	39.1	0.134	37.3	0.154	41.9	0.140	56.3	26.045	50.5	18.775	55.9	22.563
MeOH:H ₂ O Reflux	5.1	0.018	3.8	0.016	3.4	0.013	14.5	6.723	9.7	3.622	21.6	5.068
Enzyme	8.5	0.029	9.5	0.039	11.1	0.041	—	—	—	—	—	—
0.5 N HCl Reflux	14.4	0.049	16.8	0.069	18.3	0.068	—	—	—	—	—	—
Final unextracted	3.7	0.013	3.1	0.013	4.6	0.017	38.2	17.660	36.9	13.732	41.4	16.7
Total characterised	89.4	0.307	97.8	0.40	89.7	0.33	54.7	25.3	58.4	21.7	58.7	23.7
Total identified	15.6	0.054	14.7	0.06	12.4	0.047	23.8	11.0	27.9	10.4	28.3	11.4

^a Characterised as multiple components

Table 26 Distribution of metabolites in potato tubers and foliage after application of [pyridine-5-¹⁴C]-pymetrozine at rates of 3 × 0.15 kg ai/ha

Fraction	Tuber						Foliage					
	7 day DAT		14 day DAT		29 day DAT		7 day DAT		14 day DAT		29 day DAT	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
TRR		0.224		0.286		0.352		8.168		6.826		7.756
Identified												
pymetrozine	2.2	0.006	0.97	0.003	1.1	0.004	5.8	0.476	6.6	0.452	5.1	0.393
CGA96956	54.1	0.140	60.1	0.187	66.8	0.238	6.4	0.525	4.5	0.310	2.7	0.211
CGA180777	0.64	0.002	0.81	0.003	1.0	0.004	0.75	0.061	0.65	0.045	0.22	0.017
CGA180777-gly	21.0	0.054	22.1	0.069	16.4	0.058	0.61	0.050	0.63	0.043	1.1	0.082
CGA180778	1.2	0.003	1.0	0.003	0.5	0.002	1.9	0.156	1.4	0.094	1.8	0.138
CGA128632	1.1	0.003	1.3	0.004	1.2	0.004	1.3	0.109	1.6	0.109	2.0	0.157
CGA128632-gly	1.9	0.005	0.86	0.003	0.86	0.003	15.9	1.297	14.7	1.000	17.5	1.355
Characterised												
P1	1.3	0.003	1.6	0.005	1.9	0.007	0.14	0.011	0.21	0.014	0.35	0.027
P4	–	–	0.09	0.000	0.51	0.002	0.03	0.002	–	–	0.13	0.010
P6	0.48	0.001	0.74	0.002	0.79	0.003	–	–	–	–	–	–
P7	–	–	0.08	0.000	–	–	–	–	–	–	–	–
P8	–	–	–	–	–	–	0.11	0.009	0.05	0.004	0.67	0.052
P9	–	–	0.08	0.000	–	–	–	–	–	–	0.15	0.012
P11	–	–	0.49	0.002	–	–	0.35	0.029	0.41	0.028	0.21	0.016
P12	–	–	–	–	–	–	0.32	0.026	1.5	0.105	2.6	0.204
P13	–	–	0.39	0.001	0.08	0.000	0.42	0.034	–	–	–	–
P16	–	–	–	–	0.42	0.002	–	–	–	–	–	–
P20	–	–	0.13	0.000	–	–	0.89	0.073	0.30	0.021	–	–
P21	–	–	–	–	0.12	0.000	–	–	–	–	–	–
P22	0.11	0.000	0.96	0.003	1.2	0.004	–	–	–	–	–	–
P23	–	–	–	–	–	–	1.7	0.135	1.7	0.117	2.1	0.164
P24	–	–	–	–	0.12	0.000	–	–	–	–	0.29	0.022
P25	–	–	–	–	0.15	0.001	0.18	0.015	0.07	0.005	0.17	0.013
P26	–	–	–	–	0.19	0.001	–	–	0.05	0.004	–	–
P27	–	–	–	–	0.07	0.000	0.02	0.002	–	–	–	–
P28	–	–	–	–	–	–	0.02	0.002	–	–	–	–
Other												
Unextracted	12.2	0.031	10.1	0.032	6.2	0.022						
MeOH:H ₂ O Reflux	8.4	0.022	5.4	0.017	–	–						
Final unextracted	4.4	0.011	5.1	0.016	–	–	60.0	4.900	62.3	4.254	55.2	4.281
Total characterised	93.1	0.21	94.3	0.27	94.1	0.33	37.9	3.1	37.7	2.6	41.4	3.2
Total identified	81.9	0.18	87.1	0.25	87.9	0.31	32.7	2.7	30.1	2.1	30.4	2.4

Table 27 Distribution of metabolites in potato tubers and foliage after application of [pyridine-5-¹⁴C]-pymetrozine at rates of 3 × 1.05 kg ai/ha

Fraction	Tuber						Foliage					
	7 day DAT		14 day DAT		29 day DAT		7 day DAT		14 day DAT		29 day DAT	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
TRR		0.800		0.848		1.072		29.380		31.405		35.552
Identified												
pymetrozine	1.2	0.010	1.4	0.012	1.2	0.013	17.6	5.181	14.1	4.4	8.9	3.168
CGA96956	69.5	0.556	71.7	0.608	74.5	0.798	6.2	1.86	3.5	1.1	2.2	0.765
CGA180777	0.09	0.001	3.1	0.027	4.5	0.048	–	–	0.64	0.2	0.49	0.175
CGA180777-gly	–	–	–	–	12.4	0.1332	0.07	0.020	0.44	0.14	0.31	0.111
CGA180778	0.88	0.007	1.8	0.015	0.34	0.004	3.0	0.869	1.2	0.39	2.0	0.700
CGA128632	1.2	0.010	1.4	0.012	1.1	0.012	3.1	0.919	3.4	1.1	1.7	0.597
CGA128632-gly	0.75	0.006	0.83	0.007	0.99	0.011	12.3	3.605	9.5	2.98	10.6	3.782
Characterised												
P4	0.44	0.003	3.3	0.028	0.81	0.009	0.25	0.074	–	–	–	–
P6	–	–	–	–	1.7	0.018	0.02	0.006	0.12	0.036	0.21	0.076
P7	–	–	0.05	0.000	–	–	0.12	0.035	0.08	0.025	0.09	0.033
P8	–	–	0.18	0.001	–	–	0.06	0.019	0.15	0.046	0.15	0.052
P9	–	–	–	–	–	–	0.05	0.016	0.16	0.51	0.19	0.068
P10	–	–	–	–	–	–	0.02	0.007	–	–	–	–
P11	–	–	–	–	–	–	0.03	0.008	0.05	0.017	0.70	0.248
P12	–	–	–	–	0.1	0.001	1.3	0.373	0.22	0.070	1.2	0.419
P13	–	–	–	–	–	–	–	–	1.7	0.543	–	–
P14	–	–	–	–	–	–	–	–	0.67	–	–	–
P16	0.56	0.004	–	–	0.47	0.005	–	–	–	–	–	–
P20	0.22	0.002	–	–	–	–	1.7	0.503	–	–	–	–
P21	0.09	0.001	–	–	–	–	–	–	–	–	–	–
P22	0.16	0.001	–	–	0.06	0.001	–	–	–	–	–	–
P23	2.5	0.020	0.35	0.003	–	–	3.0	0.894	2.9	0.897	2.2	0.772
P24	0.10	0.001	–	–	–	–	–	–	–	–	–	–
P25	0.38	0.003	–	–	–	–	–	–	–	–	0.21	0.073
P26	–	–	–	–	–	–	–	–	0.52	0.164	–	–
P27	–	–	–	–	–	–	–	–	–	–	0.09	0.032
Other												
Unextracted	21.3	0.170	15.1	0.128	13.3	0.142	–	–	–	–	–	–
MeOH:H ₂ O Reflux	16.9	0.135	8.9	0.075	7.1	0.077	–	–	–	–	–	–
Final unextracted	3.7	0.029	5.3	0.045	4.7	0.051	53.7	15.8	60.5	19.0	70.2	24.950
Total characterised	96.6	0.77	103.2	0.87	103.7	1.1	47.8	14.0	38.5	12.1	34.6	12.3
Total identified	73.7	0.59	80.2	0.68	95.0	1.0	42.2	12.4	32.8	10.3	26.2	9.3

The storage stability of residues in potato matrices was investigated by re-analysing the samples after 19–22 weeks. Recovered residues are presented in the following table.

Table 28 Storage stability of potato samples treated with [pyridine-¹⁴C]- or [triazine-¹⁴C]-pymetrozine

	% TRR					
	Pyridine-label				Triazine-label	
Application rate	3 × 150 g ai/ha				3 × 150 g ai/ha	
Sample	Foliage		Tuber		Tuber	
	Analysis	Re-analysis	Analysis	Re-analysis	Analysis	Re-analysis
Storage interval	5 weeks	25 weeks	6 weeks	25 weeks	5 weeks	27 weeks
pymetrozine	5.8	4.9	2.2	2.5	3.96	2.7
CGA96956	6.4	7.0	54.1	45.1	–	–
gly-CGA180777	0.6	0.19	21.0	10.0	–	–
CGA180777	0.75	1.5	0.64	8.5	–	–
gly-CGA128632	15.9	16.0	1.8	1.4	–	–
CGA294849					1.97	1.3
CGA249257					1.99	1.1
GS23199					2.0	2.2
gly-GS23199					3.9	3.4
Unextracted	59.9	60.5	12.2	13.5	39.1	43.1

In two older studies the behaviour and metabolism of [triazine-6-¹⁴C]-pymetrozine (Nicollier, 1994, PYMET_027) and [pyridine-5-¹⁴C]-pymetrozine (Nicollier, 1994, PYMET_028) were studied in field grown potato plants. Forty-one day old potato plants were treated with one of the labelled compounds twice at 20 day interval at a rate of 0.2 kg ai/ha each.

Samples of the foliage were collected 1 h and 20 days after the first application and 1 h, 29 d and 55 d after the last application. The tubers were collected at maturity 55 d after the final treatment.

For the analysis the tubers were peeled. All samples were homogenized and extracted with methanol:water (8:2). The remaining residues were soxhlet extracted with methanol. The identification of residues was performed by TLC and high voltage electrophoresis. Total radioactive residues were determined by combustions and LSC.

Following foliar application of pymetrozine to potatoes most of the residue was recovered from the leaves. Directly after treatment the parent substance was the major residue (42–57% TRR). However, the rates quickly declined within the first 20 days after treatment to less than 5% of the TRR in the leaves. In tubers the TRR was very low (0.051–0.072 mg eq/kg). No parent substance was identified in tuber peel or flesh (see Table 29).

Table 29 Composition and extractability of radioactive residues from potatoes following application of pymetrozine at rates of 2 × 0.2 kg ai/ha

Substrate	Interval	TRR mg eq/kg	Parent		Extracted		unextracted	Total
			mg eq/kg	% TRR	Cold (% TRR)	Soxhlet (% TRR)	% TRR	% TRR
Pyridine-5- ¹⁴ C-label								
Leaves, total	1 h after 1 st application	23.326	9.837	42.2	57.8	2.2	33.8	93.8
Upper leaves	20 d after 1 st application	0.762	0.007	0.9	52.7	0.6	38.0	91.3
Lower leaves	20 d after 1 st application	3.164	0.026	0.8	40.3	2.8	50.3	93.4
Leaves, total	1 h after 2 nd application	11.422	3.534	30.9	50.0	3.8	35.4	89.2
Upper leaves	29 d after the 2 nd application	0.675	0.003	0.4	52.3	2.5	40.4	95.2
Lower	29 d after the 2 nd	2.186	0.035	1.6	44.0	2.7	44.1	90.8

Substrate	Interval	TRR	Parent		Extracted		unextracted	Total
		mg eq/kg	mg eq/kg	% TRR	Cold (% TRR)	Soxhlet (% TRR)	% TRR	% TRR
leaves	application							
Foliage	55 d after the 2 nd application	1.287	0.041	3.2	17.7	2.2	84.6	104.5
Tuber, flesh	55 d after the 2 nd application	0.068	< 0.003	< 4.4	99.4	1.4	6.9	107.7
Tuber, peel	55 d after the 2 nd application	0.095	< 0.005	< 5.2	76.2	2.4	14.9	93.5
Tuber, whole	55 d after the 2 nd application	0.072 (calc.)	–	–	–	–	–	–
Triazine-6- ¹⁴ C-label								
Leaves, total	1 h after 1 st application	17.169	9.74	56.7	78.1	2.6	19.3	100
Upper leaves	20 d after 1 st application	0.367	–	–	26.6	2.5	73.2	102.3
Lower leaves	20 d after 1 st application	3.627	0.014	< 0.4	37.9	2.5	56.0	96.4
Leaves, total	1 h after 2 nd application	9.52	5.24	55.0	78.2	1.2	11.9	91.3
Upper leaves	29 d after the 2 nd application	0.56	0.005	8.9	22.5	1.8	74.8	99.1
Lower leaves	29 d after the 2 nd application	2.386	0.06	2.5	32.6	2.4	66.4	101.4
Foliage	55 d after the 2 nd application	1.821	0.038	2.1	8.1	2.3	80.0	90.4
Tuber, flesh	55 d after the 2 nd application	0.049	< 0.001	0.4	76.0	0.9	25.6	102.5
Tuber, peel	55 d after the 2 nd application	0.062	–	–	46.5	3.3	57.6	107.4
Tuber, whole	55 d after the 2 nd application	0.051 (calc.)	–	–	–	–	–	–

The identification of the residue following application of [pyridine-5-¹⁴C]-pymetrozine revealed CGA180777 (22.2% TRR) and its conjugates (29.1% TRR) as the major residue in the tubers. CGA96956 was also present as a major metabolite (25.1% TRR), while only traces of unchanged parent were detected. In the foliage the parent was the major residue directly after treatment. After 20–55 days, pymetrozine was strongly degraded, leaving only 0.4–0.9% of the TRR in the leaves. Most of the radioactivity was recovered as conjugated of CGA168632.

After application of [triazine-6-¹⁴C]-pymetrozine GS23199 was the only major metabolite in the tubers (11% TRR). Identical to the pyridine-label, the parent substance was only detectable in traces. In the foliage pymetrozine was mainly present after treatment. The degraded residue after 20–55 days was the glucose conjugate of GS23199 (0.3–18.5% TRR).

A summary of the characterised and identified radioactive residues is presented in Tables 30 and 31.

Table 30 Identification of radioactive residues from potatoes following application of [pyridine-5-¹⁴C]-pymetrozine at rates of 2 × 0.2 kg ai/ha

Fraction	Radioactive residues in mg eq/kg (% TRR)									
	1 h after 1 st appl.	20 d after 1 st appl.		1 h after 2 nd appl.	29 d after 2 nd appl.	55 d after 2 nd appl.				
	Leaves, total	Upper leaves	Lower leaves	Leaves, total	Upper leaves	Lower leaves	Foliage	Tuber, peel	Tuber, flesh	Tuber, whole
TRR	23.326	0.762	3.164	11.422	0.675	2.186	1.287	0.095	0.068	0.072
Identified										
pymetrozine	9.84 (42.2)	0.007 (0.9)	0.025 (0.8)	6.57 (57.5)	0.011 (1.6)	0.009 (0.4)	0.041 (3.2)	< 0.001 (0.8)	–	< 0.001 (0.2)
CGA180777	–	–	–	–	–	–	0.059 (4.6)	0.002 (2.3)	0.018 (27.0)	0.016 (22.2)

Table 31 Identification of radioactive residues from potatoes following application of [triazine-6-¹⁴C]-pymetrozine at rates of 2×0.2 kg ai/ha

[illegible]

Fraction	Radioactive residues in mg eq/kg (% TRR)									
	1 h after 1 st appl.	20 d after 1 st appl.		1 h after 2 nd appl.	29 d after 2 nd appl.		55 d after 2 nd appl.			
	Leaves, total	Upper leaves	Lower leaves	Leaves, total	Upper leaves	Lower leaves	Foliage	Tuber, peel	Tuber, flesh	Tuber, whole
II ₁ (start)	0.53	0.057	0.25	0.28	0.049	0.13	0.099	0.002 (3.5)	0.001 (2.0)	0.001 (2.3)
II ₂	(3.1)	(15.6)	(7.0)	(2.9)	(8.7)	(5.5)	(5.4)	0.014 (22.7)	0.011 (22.8)	0.012 (22.7)
II _{3a}									0.002 (3.5)	0.002 (3.0)
II _{3b}								< 0.001 (0.5)	0.006 (12.1)	0.005 (10.1)
I ₂					0.026 (4.6)	0.14 (5.9)	0.011 (0.6)	< 0.001 (0.3)	< 0.001 (1.9)	< 0.001 (1.6)
I ₃	–	–	–	–	–	–	–	–	–	–
I ₆	–	–	–	–	–	–	0.007 (0.4)	–	< 0.001 (1.2)	< 0.001 (1.0)
I _{6a}	–	–	–	–	–	–	–	< 0.001 (0.9)	< 0.001 (0.8)	< 0.001 (0.8)
I ₁₀	0.19 (1.1)	–	–	0.3 (3.1)	–	–	0.038 (2.1)	–	< 0.001 (0.9)	< 0.001 (0.8)
I ₁₃	–	–	–	0.067 (0.7)	–	–	0.084 (4.6)	0.002 (2.8)	< 0.001 (0.8)	< 0.001 (1.2)
I ₁₄	–	–	–	–	–	–	0.056 (3.1)	–	–	–
Unresolved	2.4 (14)	0.015 (4.0)	0.37 (10.1)	1.2 (12.8)	0.03 (5.4)	0.21 (9.0)	0.35 (19.2)	0.011 (18.2)	0.007 (13.4)	0.007 (14.2)
Soxhlet	0.45 (2.6)	0.009 (2.5)	0.09 (2.5)	0.11 (1.2)	0.01 (1.8)	0.057 (2.4)	0.042 (2.3)	0.002 (3.3)	< 0.001 (0.9)	< 0.001 (1.3)
Unextracted	3.3 (19.3)	0.27 (73.2)	2.0 (56)	1.1 (11.9)	0.42 (74.8)	1.58 (66.4)	0.65 (35.6)	0.022 (35.9)	0.013 (25.6)	0.014 (27.3)
Total	17.151 (99.9)	0.375 (102.3)	3.5 (96.4)	8.7 (91.3)	0.555 (99.1)	2.42 (101.3)	(90.5)	0.064 (103.7)	0.05 (102.1)	0.052 (102.4)

Rice

The metabolism of pymetrozine in paddy rice following foliar treatment was investigated using [pyridine-5-¹⁴C]-pymetrozine (Sandmeier, 1994, PYMET_029) and [triazine-6-¹⁴C]-pymetrozine (Sandmeier, 1994, PYMET_030). The rice plants grown in containers with paddy water were treated once with a foliar application equivalent to 0.25 kg ai/ha 45 days before harvest. Samples of rice foliage were taken 19 days (approx. 75% maturity), and samples of grains, husks and straw 45 days after application (full maturity).

The homogenised samples were extracted with methanol/water five times. The combined extracts were used for TLC analysis. After cold extraction a hot microwave extraction with 1-propanol/water (80:20) was performed. Besides the TLC with various solvent systems the Liquid Chromatography with different columns was used. Total radioactive residue were measured by combustion and LSC.

TRR levels found in the samples were present at comparable concentrations for both labels. In the foliage 1.72–2.09 mg eq/kg were found. In the grain and in straw TRR levels were 0.137–0.243 mg eq/kg and 5.31–6.34 mg eq/kg, respectively. Husks contained 0.57–1.7 mg eq/kg, but were not further analysed for the composition of the residue.

The identification of the radioactivity revealed unchanged pymetrozine as the major residue in foliage (86–89% TRR) and in straw (63–74% TRR). No other major metabolites were found. In the grain the unextracted residue was high, representing 63–86% of the TRR. No further characterisation of the unextracted residue was conducted. Most of the radioactivity identified for the pyridine-label was present as CGA180777 and its conjugates (6.7% TRR each). For the triazine-label only minor metabolites were found (< 2.3% TRR each).

A summary on the TRR levels found in the samples and on their composition is presented below.

Table 32 Radioactive residues of [pyridine-¹⁴C]- and [triazine-¹⁴C]-pymetrozine after foliar application of 0.25 kg ai/ha to paddy rice

Fraction	Radioactive residues in mg eq/kg (% TRR)							
	[pyridine- ¹⁴ C]-pymetrozine				[triazine- ¹⁴ C]-pymetrozine			
	foliage 19 DAT	grain 45 DAT	straw 45 DAT	husks 45 DAT	foliage 19 DAT	grain 45 DAT	straw 45 DAT	husks 45 DAT
TRR	1.72 (100)	0.243 (100)	5.31 (100)	1.707 (100)	2.09 (100)	0.137 (100)	6.34 (100)	0.57 (100)
Identified								
pymetrozine	1.53 (88.9)	0.002 (0.8)	3.95 (74.4)	n.a.	1.79 (85.5)	0.003 (2.3)	3.99 (63.0)	n.a.
CGA96956	0.1 (5.8)	0.021 (8.7)	0.101 (1.9)		–	–	–	
CGA180777	–	0.016 (6.7)	0.143 (2.7)		–	–	–	
CGA180777 conj	–	0.016 (6.7)	0.18 (3.4)		–	–	–	
CGA180778	–	0.001 (0.4)	0.085 (1.6)		–	–	–	
CGA359009	0.057 (3.3)	< 0.001 (0.2)	0.14 (2.6)		0.071 (3.4)	0.001 (0.7)	0.184 (2.9)	
CGA128632			0.021 (0.4)		–	–	–	
CGA215525	–	–	–		0.014 (0.7)	< 0.01 (0.1)	0.101 (1.6)	
CGA300407	0.014 (0.8) ^a	–	0.08 (1.5)		–	–	–	
CGA294849	–	–	–		0.014 (0.7)	< 0.001 (0.5)	0.07 (1.1)	
GS23199	–	–	–		0.016 (0.8)	< 0.001 (0.2)	0.07 (1.1)	
Characterised								
I ₁	–	–	–	n.a.	0.033 (1.6)	0.004 (2.8)	0.127 (2.0)	n.a.
I _{2/3}	–	–	–		–	< 0.01 (0.2)	–	
I ₅	–	–	–		–	< 0.01 (0.3)	0.044 (0.7)	
I _{6/6a}	–	–	–		–	< 0.01 (0.2)	–	
I _{p8}	–	< 0.01 (0.3)	–		–	–	–	
I _{p10}	–	< 0.01 (0.3)	–		–	–	–	
I ₁₃	–	–	–		–	< 0.001 (0.2)	0.044 (0.7)	
I _{13a}	–	–	0.12 (2.3)		–	–	–	
Microwave and unresolved	0.11 (6.3)	0.023 (9.6)	0.42 (7.9)	n.a.	0.19 (8.9)	0.06 (4.4)	0.78 (12.3)	n.a.
Unextracted	0.065 (3.8)	0.15 (62.5)	0.44 (8.2)	n.a.	0.14 (6.6)	0.12 (86.2)	1.15 (18.1)	n.a.
Total	1.87 (108.9)	0.234 (96.2)	5.68 (106.9)	n.a.	2.26 (108.2)	0.134 (98.1)	6.56 (103.5)	n.a.

n.a. = Not analysed

DAT = Days after treatment

^a co-elution with I_{13a}

For the granular application of pymetrozine to paddy rice two studies were conducted using [pyridine-5-¹⁴C]-pymetrozine (Sandmeier, 1995, PYMET_031) and [triazine-6-¹⁴C]-pymetrozine

(Sandmeier, 1995, PYMET_032). Two weeks old rice plants in a small seedling box were treated with 5% granules (GR 5) at application rates corresponding to 0.6 kg ai/ha. One day after application the seedlings (including the soil) were transferred into six containers. Foliage was sampled 1, 41 (25% maturity) and 69 days (50% maturity) after application, and grains, husks and straw 116 days (maturity) after application. Samples were analysed within two months after sampling.

The extraction of samples was made with methanol (80%). After the cold extraction a hot microwave extraction in 1-propanol was performed. The extracts were used for TLC, liquid chromatography and hydrolysis or enzymatic cleavage. Unextracted residues in straw were further addressed by means of acidic microwave extraction (n-propanol/ 0.1 N HCl, 80:20), further residue hydrolysis by boiling 2 N NaOH and subsequent extract acidification and concentration to precipitate the lignin fraction. TLC analysis was performed with the extracts. In grain, unextracted radioactivity was released with 0.05 N NaOH. After neutralisation and precipitation of protein/starch with ethanol, the resulting extract was subjected to TLC analysis after evaporation and solubilisation in methanol/water. Remaining unextracted radioactivity was determined by combustion.

Total radioactive residues were highest in foliage directly after treatment with 33.15–42.4 mg eq/kg, but declined to 0.72–0.82 mg eq/kg within 69 days. At harvest dry straw gave TRR levels of 2.59–2.63 mg eq/kg. In the grain the radioactivity was much lower (0.205–0.523 mg eq/kg).

The identification of the residues showed unchanged parent as the major residue only in foliage directly after application (37.6–59.7% TRR). In all other samples pymetrozine was present at low amounts not exceeding 8.3% of the TRR. The pyridine-label specific metabolites CGA96956 and CGA180777 including its glucose-conjugate were the major residues identified. Following application of the triazine-labelled pymetrozine no major metabolites were found.

For both labels a major part of the radioactivity remained unextracted, representing 40–50% TRR in straw and 56–86% TRR in the grain. The characterisation of the unextracted radioactivity revealed incorporation into glucose and related natural components like starch.

A summary of the radioactive residues found and their composition is presented below.

Table 33 Radioactive residues of [pyridine-¹⁴C]- and [triazine-¹⁴C]-pymetrozine after granular application of 0.6 kg ai/ha to paddy rice

Fraction	Radioactive residues in mg eq/kg (% TRR)											
	[pyridine- ¹⁴ C]-pymetrozine						[triazine- ¹⁴ C]-pymetrozine					
	foliage 1 DAT	foliage 41 DAT	foliage 69 DAT	straw 116 DAT	husks 116 DAT	grain 116 DAT	foliage 1 DAT	foliage 41 DAT	foliage 69 DAT	straw 116 DAT	husks 116 DAT	grain 116 DAT
TRR	33.15 (100)	1.402 (100)	0.82 (100)	2.631 (100)	3.661 (100)	0.523 (100)	42.411 (100)	1.183 (100)	0.723 (100)	2.59 (100)	0.477 (100)	0.205 (100)
Identified												
pymetrozine	19.8 (59.7)	0.081 (5.8)	0.031 (3.8)	0.089 (3.4)	–	–	15.9 (37.6)	0.098 (8.3)	0.035 (4.9)	0.109 (4.2)	0.01 (2.0)	< 0.001 (0.2)
CGA96956	3.2 (9.5) ^a	0.275 (19.6)	0.196 (23.9)	0.3 (11.2)	1.025 (28)	0.055 (10.6)	–	–	–	–	–	–
CGA180777	–	0.062 (4.4)	0.033 (4.0)	0.288 (10.9)	0.533 (15.1)	0.044 (8.4)	–	–	–	–	–	–
CGA180777 conj	^a	0.129 (9.3)	0.156 (19)	0.168 (6.4)	0.754 (20.6)	0.09 (17.2)	–	–	–	–	–	–
CGA180778	–	0.049 (3.5)	0.016 (1.9)	0.176 (6.7)	0.026 (0.7)	0.003 (0.6)	–	–	–	–	–	–
CGA359009	6.0 (18)	0.08 (5.7)	0.03 (3.2)	–	–	–	6.2 (14.7)	0.084 (7.1)	0.032 (4.5)	0.041 (1.6)	0.012 (2.5)	0.001 (0.7)
CGA128632- conj.	–	–	0.005 (0.6)	0.047 (1.8)	–	–	–	–	–	–	–	–
CGA128632	–	0.013 (0.9)	0.1 (1.2)	0.061 (2.3)	–	–	–	–	–	–	–	–
CGA249257	–	–	–	–	–	–	0.55 (1.3)	0.008 (0.7)	0.005 (0.7)	–	–	< 0.001 (< 0.03)
CGA300407	2.1	0.025	0.021	0.061	–	–	–	–	–	–	–	–

Fraction	Radioactive residues in mg eq/kg (% TRR)											
	[pyridine- ¹⁴ C]-pymetrozine						[triazine- ¹⁴ C]-pymetrozine					
	foliage 1 DAT	foliage 41 DAT	foliage 69 DAT	straw 116 DAT	husks 116 DAT	grain 116 DAT	foliage 1 DAT	foliage 41 DAT	foliage 69 DAT	straw 116 DAT	husks 116 DAT	grain 116 DAT
	(6.3)	(1.8) ^b	(2.5) ^b	(2.3)								
CGA294849	–	–	–	–	–	–	1.5 (3.5)	0.056 (4.7)	0.04 (5.5)	0.236 (9.1)	0.013 (2.8)	< 0.001 (0.2)
GS23199	–	–	–	–	–	–	7.1 (16.8)	0.013 (1.1)	0.017 (2.3)	0.127 (4.9)	0.014 (2.9)	< 0.001 (0.1)
GS23199-gluc.	–	–	–	–	–	–	1.2 (2.9)	0.022 (1.9)	0.025 (3.5)	0.145 (5.6)	0.016 (3.3)	< 0.01 (0.5)
Characterised												
I ₁	–	–	–	–	–	–	–	0.125 (10.6)	0.092 (12.7)	0.308 (11.9)	0.071 (14.9)	0.007 (3.3)
I _{2/3}	–	–	–	–	–	–	–	–	–	–	–	< 0.01 (0.4)
I _{P4a}	–	–	–	0.03 (1.0)	–	–	–	–	–	–	–	–
I _{8a}	–	–	–	–	–	–	–	–	–	< 0.02 (0.6)	–	< 0.01 (< 0.3)
I _{6/6a}	–	–	–	–	–	–	–	–	–	0.028 (1.1)	< 0.01 (0.9)	< 0.01 (< 0.3)
I _{P10}	–	< 0.01 (0.4)	< 0.01 (0.4)	–	0.06 (1.7)	< 0.01 (1.5)	–	–	–	–	–	–
I ₁₃	–	–	–	–	–	–	–	0.026 (2.2)	< 0.01 (1.5)	0.088 (3.4)	< 0.01 (0.7)	< 0.01 (0.3)
I _{13a}	–	b	b	0.042 (1.6)	–	–	3.6 (8.4)	0.031 (2.6)	0.015 (2.1)	0.013 (0.5)	0.004 (0.8)	–
I ₁₄	–	–	–	–	–	–	–	< 0.01 (0.8)	< 0.01 (0.4)	0.02 (0.9)	–	–
Microwave and unresolved	–	0.14 (9.8)	0.1 (12.0)	0.24 (9.0)	0.45 (12.3)	0.031 (5.9)	3.6 (8.4)	0.09 (7.6)	0.05 (7.1)	0.19 (7.2)	0.045 (9.4)	< 0.01 (4.5)
Unextracted ^c	0.66 (2.0)	0.43 (30.9)	0.28 (33.8)	1.07 (40.6)	0.86 (23.5)	0.29 (55.9)	2.7 (6.4)	0.62 (52.2)	0.4 (55.4)	1.28 (49.6)	0.26 (58.4)	0.18 (85.9)
Total	33.15 (100)	1.29 (92.1)	0.87 (106.1)	2.58 (98.1)	3.73 (101.9)	0.53 (101.1)	42.4 (100)	1.18 (99.8)	0.73 (100.6)	2.6 (100.6)	0.47 (98.6)	0.2 (96.4)

DAT = Days after treatment

^a Sum of CGA96956 and CGA18077 conj.^b Sum of CGA300407 and I_{13a}^c Characterised as glucose and correlated products like starch

For the investigation of the stability of residues during freezer storage, grain and straw extracts were re-analysed after 13 months by TLC and the pattern was compared to the first analysis:

Table 34 Storage stability of rice grain and straw extracts after treatment with [pyridine-¹⁴C]- or [triazine-¹⁴C]-pymetrozine

Fraction	% TRR				triazine- ¹⁴ C- label	
	pyridine- ¹⁴ C- label					
	grain extract fresh	grain extract stored 13 months	straw extract fresh	straw extract stored 13 months	straw extract fresh	straw extract stored 11 months
I _{P1} / I ₁ (Start)	–	–	54.2	53.8	32.4	30.5
CGA180777	15.3	18.5	11.6	11.8	–	–
CGA128632	–	–	4.5	2.4	–	–
IP4a	–	–	2.5	2.5	–	–
pymetrozine	–	–	5.8	8.1	11.4	9.9
GS23199	–	–	–	–	11.2	10.0
GS23199-gluc.	–	–	–	–	17.1	16.7
CGA180778	–	–	9.0	6.2	–	–
CGA359009 &	–	–	5.7	3.5	6.2	6.8

CGA128632						
CGA300407 & I _{13a}	–	–	2.3	4.5	2.1	2.7
I ₁₃	–	–	–	–	4.4	5.1
CGA294849	–	–	–	–	3.2	5.0
CGA96956	27.7	30.8	–	–	–	–
gly-CGA180777	9.9	11.1	–	–	–	–
I _{6/6a} & I _{8a}	–	–	–	–	6.4	5.1
II _{P6} +I _{P10}	39.7	33.5	–	–	–	–
unresolved	7.5	6.1	4.5	7.4	5.8	8.3

Cotton

The investigation on the metabolism of pymetrozine in cotton under glasshouse conditions was reported in two studies by Gentile (1995, PYMET_033 and 1996, PYMET_034). [Pyridine-5-¹⁴C] or [triazine-6-¹⁴C] pymetrozine formulated as WP 25 product were applied twice with over-the-top spraying involving application rates equivalent to 0.2 kg ai/ha per treatment. The applications were performed with one week intervals ending directly before blooming of the plants.

One hour after each application leaf samples were collected. At green boll stage, 52 days after the second application, bolls and leaves were taken. At maturity, 93 days after the second application, each plant was separated into old-leaves, new leaves, stems, hulls, fibres and seeds.

Each of the plant parts collected after 93 days were homogenised and five times extracted with methanol-water. Enzymatic cleavage (overnight with cellulase) of the methanolic seeds extract was performed. The combined extracts were used for parent and metabolite quantification by 1-dimensional or 2-dimensional TLC. After the cold extraction a soxhlet extraction with methanol was performed. The non-extractable radioactivity was determined by combustion. The analytical procedure was identical for all plant parts except seeds for which the ground material was first extracted twice with hexane to remove oil. Unextracted radioactivity of new-leaves, fibres and seeds was further extracted with 2 N HCl. The released radioactivity was derivated (methylation and acetylation) and analysed by PEI-cellulose- and silica gel-TLC.

Identification of the radioactivity in the samples collected after 93 days revealed pymetrozine as the major residue in leaves, stems, hulls and the extracted oil. In addition CGA96956 was a major residue in most matrices being present at levels up to 50% of the TRR in the seeds. The oil extraction showed that it remains in the seed meals and is not present in oil. A summary of the residues found in cotton is presented in Tables 35 and 36.

Table 35 Quantitation of metabolite fractions in cotton plant parts from harvest, 93 days after the second application of [pyridine-5-¹⁴C] pymetrozine

Fraction	Radioactive residues in mg eq/kg (% TRR)							
	treated leaves	new leaves	stems	hulls	fibres	seeds	seed oil ^a	seed meal ^b
TRR	5.85	0.198	1.56	4.79	0.166	0.21	–	–
Identified								
pymetrozine	4.85 (83)	0.086 (43.5)	1.16 (74.4)	2.61 (54.4)	0.079 (47.5)	0.019 (9.0)	(57.9)	(6.0)
CGA96956	0.205 (3.5)	0.046 (23.2)	0.009 (0.6)	0.805 (16.8)	0.03 (18.1)	0.105 (50)	–	(54.5)
CGA180777	–	–	–	–	0.007 (4.4)	0.017 (8.1)	(11.4)	(8.1)
CGA128632	–	–	–	–	0.002 (1.5)	0.004 (1.8)	–	(2.0)
Characterised								
Start	–	–	–	–	–	0.002 (0.8)	(13.1)	–
I _{P10}	–	–	–	–	–	< 0.001 (0.1)	(1.6)	–
Soxhlet and unresolved	0.56 (9.6)	0.03 (13.7)	0.15 (9.8)	0.85 (17.7)	0.004 (2.5)	0.033 (15.7)	(9.4)	(16.5)

Unextracted	2.9 (5.0)	0.03 (15.9)	0.16 (10.5)	0.46 (9.7)	0.037 (22.4)	0.024 (11.4)	–	(12.4)
Total	5.9 (101.1)	0.19 (96.3)	1.5 (95.3)	4.7 (98.6)	0.16 (96.4)	0.2 (96.9)	(93.4)	(99.5)

^a Oil fraction contained 6.1% of the TRR

^b Meal fraction contained 91.7% of the TRR

Table 36 Quantitation of metabolite fractions in cotton plant parts from harvest, 93 days after the second application of [triazine-6-¹⁴C] pymetrozine

Fraction	Radioactive residues in mg eq/kg (% TRR)							
	treated leaves	new leaves	stems	hulls	fibres	seeds	seed oil ^a	seed meal ^b
TRR	0.623	0.033	1.719	2.673	0.065	0.043	–	–
Identified								
pymetrozine	0.408 (65.5)	–	1.071 (62.3)	1.547 (57.9)	0.018 (27.7)	0.003 (7.4)	(34.5)	(0.9)
CGA294849	0.014 (2.2)	–	0.021 (1.2)	0.064 (2.4)	0.002 (3.4)	0.001 (2.2)	(11.2)	–
GS23199	0.006 (0.9)	–	0.005 (0.3)	0.011 (0.4)	0.002 (3.0)	–	–	–
Characterised								
I _{1A}	–	0.005 (14.1)	–	–	0.002 (3.0)	0.014 (32)	–	(39.3)
I _{1B}	–	–	–	–	–	0.001 (3.1)	–	(3.8)
I _{1C}	–	–	–	–	–	0.002 (4.9)	–	(6.0)
I _{1D}	–	0.005 (14.1)	–	–	–	0.002 (4.3)	–	(5.3)
I _{1E}	–	0.001 (4.3)	–	–	–	0.003 (7.1)	–	(8.7)
I _{1F}	–	0.001 (4.8)	–	–	–	0.004 (8.8)	–	(10.8)
III ₀	–	–	–	–	–	–	(15.5)	–
III ₂	0.012 (1.9)	–	0.021 (1.2)	0.051 (1.9)	–	–	–	–
III ₃	0.008 (1.3)	–	0.027 (1.6)	0.045 (1.7)	–	–	–	–
III ₄	–	–	–	–	–	0.001 (2.5)	(12.9)	–
Soxhlet and unresolved	(12.7)	(22.5)	(10.7)	(16.6)	(8.7)	(15.8)	(21.3)	(14.3)
Unextracted	(17.4)	(40.2)	(12.9)	(20.7)	(54.5)	(11.4)	–	(14.0)
Total	(98.4)	(102.1)	(87.4)	(98.2)	(100.1)	(102.5)	(95.4)	(103)

^a Oil fraction contained 19.4% of the TRR

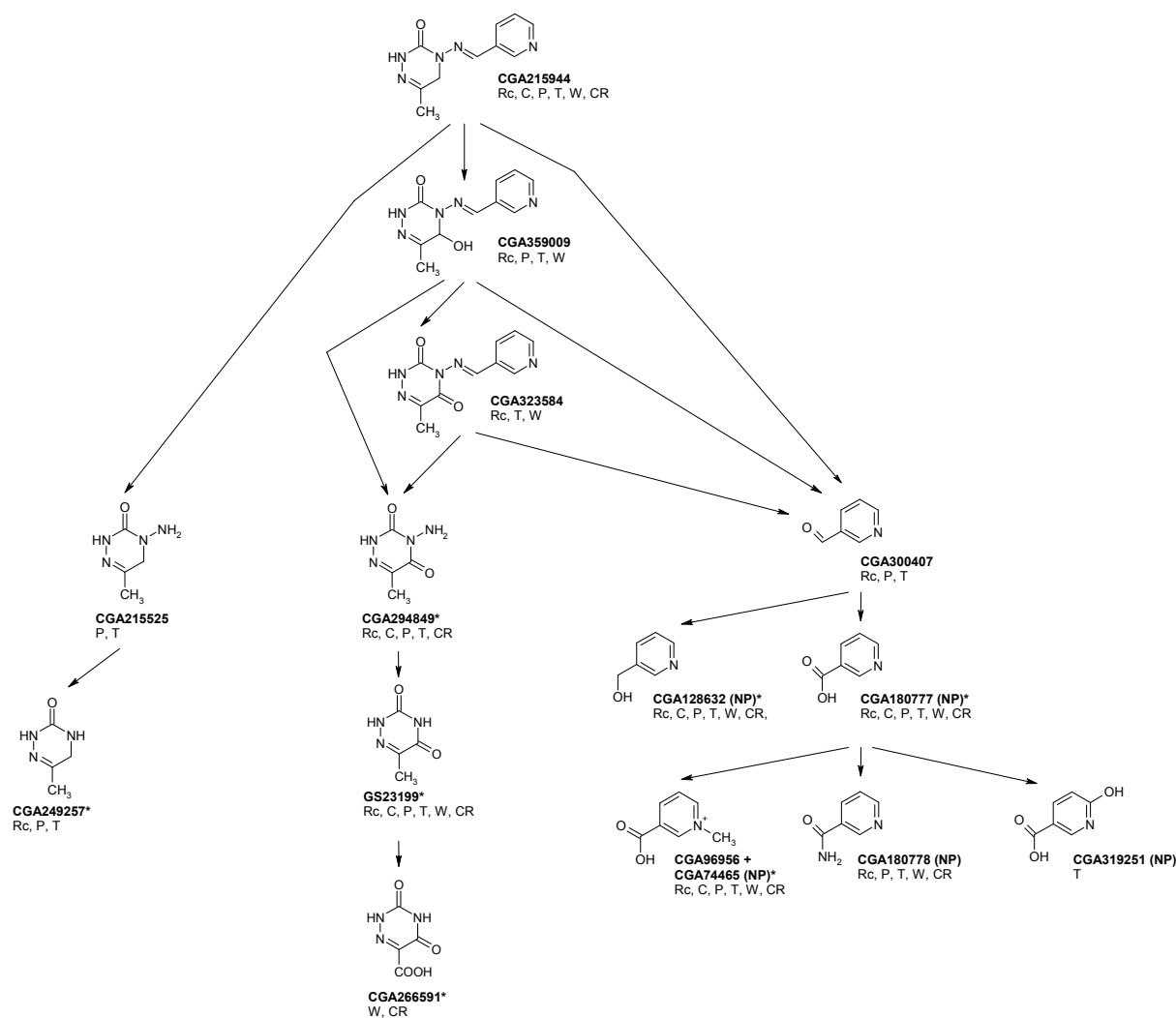
^b Meal fraction contained 86% of the TRR

For the investigation of the stability of residues during freezer storage, the seed meal sample treated with [pyridine-¹⁴C]-pymetrozine was re-analysed after 25 months by TLC and the pattern was compared to the first analysis.

For the [triazine-¹⁴C]-pymetrozine sample, TLC fractions were not attributed to known reference compounds.

Table 37 Storage stability of cotton seed meal treated with [pyridine-¹⁴C]-pymetrozine

Fraction	% TRR	
	Seed meal fresh	Seed meal stored additional 25 months
CGA96956	70.7	81.6
CGA180777	11.2	7.6
pymetrozine	8.4	4.9
Fraction 4	2.8	1.2
Unresolved	6.9	4.7



NP = Natural product
rotation

Rc = Rice C = Cotton

P = Potato

T = Tomato

CR = Crop

Figure 4 Proposed metabolic pathway of pymetrozine in plants

Environmental fate in soil

For the investigation of the environmental fate of pymetrozine the Meeting received studies on hydrolysis and the behaviour in confined and field rotational crops.

In buffer solutions pymetrozine is quickly degraded within hours to days at pH values below 7. The main degradation product was CGA215525.

In confined rotational crop studies conducted at rates equivalent to 0.41 kg ai/ha pymetrozine was only found in traces. Major metabolites found were CGA96956, CGA266591 and GS23199, mostly conjugated with glucose.

In field rotational crop studies conducted with four applications of 0.1 kg ai/ha each for tomato, lettuce or peppers, no detectable residues of pymetrozine or GS23199 were found (LOQ: 0.02 mg/kg).

Hydrolysis

The stability of pymetrozine in sterile buffer solutions was investigated using [pyridine-5-¹⁴C]-pymetrozine (Kirkpatrick, 1995, PYMET_036) and [triazine-6-¹⁴C]-pymetrozine (McDonald, 1996, PYMET_037).

For the pyridine-label buffer solutions at pH 1, 5, 7 and 9 were incubated at 25 °C for up to 35 days. The concentrations used were 5 mg pymetrozine per litre. Total recovery rates of the applied radioactivity were always near 100%.

The samples incubated at 25 °C the nature of the radioactive residues were further investigated. CGA300407 was the only identified degradation product.

Table 38 Proportion of [pyridine-5-¹⁴C]-pymetrozine and its degradation products in pH 1 buffer at 25 °C

Fraction	% of applied activity							
	0	0.5 h	1 h	2 h	3 h	6 h	24 h	48 h
Identified								
pymetrozine	94.3	87.5	80.0	68.0	57.5	38.6	22.2	22.0
CGA300407	0.6	10.1	16.3	29.2	40.7	61.6	76.1	77.1
Diffuse Radioactivity	2.7	2.9	2.2	2.1	2.1	1.7	2.4	1.8
Total	97.4	100.5	98.5	99.3	100.4	101.9	100.7	98.9

Table 39 Proportion of [pyridine-5-¹⁴C]-pymetrozine and its degradation products in pH 5 buffer at 25 °C

Fraction	% of applied activity							
	0	3 d	6 d	10 d	15 d	20 d	25 d	32 d
Identified								
pymetrozine	98.1	77.7	62.1	50.9	41.5	37.5	36.5	36.1
CGA300407	0.8	20.2	35.8	47.9	57.1	60.7	62.3	62.8
Diffuse Radioactivity	1.2	2.1	2.2	1.3	1.5	1.9	1.3	1.2
Total	100.1	100	100.1	100.1	100.1	100.1	100.1	100.1

Table 40 Proportion of [pyridine-5-¹⁴C]-pymetrozine and its degradation products in pH 7 buffer at 25 °C

Fraction	% of applied activity									
	0	3 d	6 d	10 d	14 d	17 d	21 d	24 d	27 d	30 d
Identified										
pymetrozine	98.1	97.4	97.3	96.7	96.4	96.4	96.4	95.8	95.7	95.1
CGA300407	0.6	0.7	1.4	1.3	1.7	1.8	2.1	2.3	2.5	2.8
Diffuse Radioactivity	1.3	1.9	1.4	2.0	2.0	1.9	1.6	2.1	1.9	2.1
Total	100	100	100.1	100	100.1	100.1	100.1	100.1	100.1	100

Table 41 Proportion of [pyridine-5-¹⁴C]-pymetrozine and its degradation products in pH 9 buffer at 25 °C

Fraction	% of applied activity									
	0	3 d	6 d	10 d	14 d	17 d	21 d	24 d	27 d	30 d
Identified										
pymetrozine	97.8	97.3	96.8	96.2	95.7	95.2	95.1	94.3	94.2	93.9
CGA300407	0.5	1.0	2.0	1.8	2.6	2.7	3.2	3.5	3.7	4.3
Diffuse Radioactivity	1.7	1.8	1.3	2.0	1.7	2.1	1.9	2.2	2.2	1.9
Total	100	100.1	100.1	100	100	100	100.2	100	100.1	100.1

The triazine-label aqueous buffered solutions with pH 1, 5, 7 and 9 were incubated at 25, 50 and 70 °C. The nominal concentration of pymetrozine in all buffered solutions was 5 mg/L. Samples were collected at day zero and varying intervals up to 30 days.

Total recovery rates of the applied radioactivity were always near 100%. However, the proportion of the parent substance declined depending on the pH and the incubation temperature.

Table 42 Proportion of [triazine-6-¹⁴C]-pymetrozine in pH 1 buffer (mean of % applied)

Temp.	% pymetrozine remaining after incubation							
	0	0.5 h	1 h	2	3 h	6 h	24 h	48 h
25 °C	95.6	88.8	81.2	68.0	57.2	38.0	22.5	21.8
50 °C	95.0	54.0	26.4	11.8	10.7	9.8	10.7	–
70 °C	96.1	9.9	7.5	7.4	7.3	7.6	7.8	–

Table 43 Proportion of [triazine-6-¹⁴C]-pymetrozine in pH 5 buffer (mean of % applied)

Temp.	% pymetrozine remaining after incubation																	
	0	0.5 h	1 h	3 h	6 h	1 d	2 d	3 d	4 d	5 d	6 d	7 d	10 d	15 d	20 d	25 d	30 d	32 d
25 °C	96.0	–	–	–	–	–	–	87.6	–	78.3	79.1	–	67.6	58.3	52.2	46.7	42.4	44.7
50 °C	96.4	–	–	–	90.7	74.2	59.2	–	36.0	–	–	22.1	17.1	–	–	–	–	–
70 °C	96.4	93.5	90.3	78.5	63.8	19.7	8.8	–	–	–	–	–	–	–	–	–	–	–

Table 44 Proportion of [triazine-6-¹⁴C]-pymetrozine in pH 7 buffer (mean of % applied)

Temp.	% pymetrozine remaining after incubation							
	0	5 d	9 d	10 d	15 d	20 d	25 d	30 d
25 °C	97.2	96.6	95.9	–	95.6	94.8	94.5	93.9
50 °C	96.8	92.8	–	88.1	84.6	82.3	78.6	73.4
70 °C	93.6	78.4	–	64.0	52.2	41.6	30.6	26.1

Table 45 Proportion of [triazine-6-¹⁴C]-pymetrozine in pH 9 buffer (mean of % applied)

Temp.	% pymetrozine remaining after incubation							
	0	5 d	9 d	10 d	15 d	20 d	25 d	30 d
25 °C	97.2	96.7	96.4	–	96.2	95.6	95.9	95.4
50 °C	96.6	90.2	–	85.6	75.3	74.2	63.3	61.9
70 °C	96.	83.3	–	64.6	37.6	25.6	18.9	10.0

For the pH1 and pH5 samples incubated at 25 °C the nature of the radioactive residues was further investigated. CGA215525 was identified as the major degradation products.

Table 46 Proportion of [triazine-6-¹⁴C]-pymetrozine and its degradation products in pH 1 buffer at 25 °C

Fraction	% of applied activity							
	0	0.5 h	1 h	2 h	3 h	6 h	24 h	48 h
Identified								
pymetrozine	94.3	87.5	80.0	68.0	57.5	38.6	22.2	22.0
CGA294849	^a	1.1	2.0	2.2	2.7	3.3	2.9	2.9
CGA215525 + CGA249257	^a	3.9	7.2	14.0	19.2	30.6	47.0	46.6
Characterised								
Unknown 1 ^b	^a	2.0	3.6	6.7	9.2	13.3	12.2	14.4
Unknown 2	^a	3.0	4.3	5.9	7.7	9.4	11.7	9.8

Fraction	% of applied activity							
	0	0.5 h	1 h	2 h	3 h	6 h	24 h	48 h
Diffuse Radioactivity	4.0	2.6	3.0	3.4	3.8	4.9	4.2	4.4
Total	100.2	100.1	100.1	100.2	100.1	100.1	100.2	100.1

^a Combined peaks representing 1.9% applied activity

^b Multiple minor components with < 4% AR each

Table 47 Proportion of [triazine-6-¹⁴C]-pymetrozine and its degradation products in pH 5 buffer at 25 °C

Fraction	% of applied activity							
	0	3 d	6 d	10 d	15 d	20 d	25 d	32 d
Identified								
pymetrozine	95.8	87.6	79.1	69.9	61.4	55.1	48.9	44.7
CGA215525	0.7	8.7	16.4	24.2	31.5	38.7	42.8	47.7
CGA249257	0.2	0.5	1.0	1.1	1.7	1.2	2.6	1.8
Characterised								
Unknown 1	0.2	0.8	0.9	0.9	1.0	1.1	1.1	1.0
Unknown 2	–	0.2	–	0.2	0.8	0.8	0.4	0.8
Unknown 3	0.3	0.2	0.2	0.3	0.4	0.4	0.3	0.4
Diffuse Radioactivity	2.9	2.4	2.5	3.5	3.3	2.8	4.0	3.8
Total	99.9	100	100.1	100.1	99.9	100	99.9	100

Confined rotational crop studies

For the investigation of pymetrozine in rotational crops several plots were conducted in the USA by Fleischmann (1998, PYMET_035). Each plot received one treatment to the bare soil of either [pyridine-5-¹⁴C]- or [triazine-6-¹⁴C]-pymetrozine at rates equivalent to 0.41 kg ai/ha. Wheat, radish and mustard were planted in the treated soil 30, 60, 95, 122 and 361 days after test substance application.

Total radioactive residues (TRR) in plant samples were determined by radiocombustion/LSC. Samples were extracted with acetonitrile:water (4:1 v/v) or methanol:water (4:1 v/v). Homogenates were filtered and the filter cake re-extracted at least 2× with solvent. Total extractable radioactivity was determined by summing the radioactivity in the combined filtrates and unextracted radioactivity in the residue was determined by combustion/LSC.

Acetonitrile:water extracts were cleaned-up on C₁₈ SPE cartridges. After concentration, solubilised residues were partitioned between water and water-immiscible solvents. Selected liquid and solid samples were treated with glucosidase, cellulase or cellulase/cellobiase in acetate buffer, with 0.1 M to 6 M HCl under reflux, or with acetylating or methylating agents for component characterisation and identification.

The post-extraction solids were sequentially subjected to neutral solvent reflux, 1% sodium chloride reflux, cellulase/cellobiase treatment, dilute acid reflux, dilute base reflux, strong acid reflux and strong base reflux to further characterise the extracted radioactivity.

Total radioactive residues in edible commodities at their respective harvest intervals were in the range of 0.018–0.049 mg eq/kg (wheat grain), 0.025–0.133 mg eq/kg (mustard leaves) and 0.026–0.061 mg eq/kg (radish tubers) for [triazine-6-¹⁴C]-pymetrozine and of 0.036–0.109 mg eq/kg (wheat grain), 0.011–0.053 mg eq/kg (mustard leaves) and 0.014–0.042 mg eq/kg (radish tubers) for [pyridine-5-¹⁴C]-pymetrozine.

In potential feed items residues were 0.047–0.48 mg eq/kg and 0.021–0.228 mg eq/kg in wheat forage and fodder for triazine-6-¹⁴C]- and [pyridine-5-¹⁴C]-label, respectively.

Table 48 Total radioactive residues (TRR) in rotational crop fractions after soil application of [pyridine-5-¹⁴C]-pymetrozine

Crop	Sample	30 day plant-back		60 day plant-back		90 day plant-back		120 day plant-back		360 day plant-back	
		DAA	mg eq/kg	DAA	mg eq/kg	DAA	mg eq/kg	DAA	mg eq/kg	DAA	mg eq/kg
Wheat	25% mature	96	0.068	152	0.041	189	0.068	203	0.089	413	0.024
Wheat	50% mature	121	0.052	189	0.029	203	0.102	229	0.074	448	0.021
Wheat	Mature fodder	248	0.115	253	0.132	254	0.228	254	0.157	568	0.044
Wheat	Mature grain	248	0.079	253	0.057	254	0.109	254	0.048	568	0.036
Mustard	Mature leaves	96	0.053	189	0.014	207	0.023	229	0.011	413	0.019
Radish	Mature leaves	96	0.060	189	0.018	207	0.019	229	0.021	413	0.127
Radish	Mature tubers	96	0.021	189	0.022	207	0.015	229	0.014	414	0.042

DAA = Days after application

Table 49 Total radioactive residues (TRR) in rotational crop fractions after soil application of [triazine-6-¹⁴C]-pymetrozine

Crop	Sample	30 day plant-back		60 day plant-back		90 day plant-back		120 day plant-back		360 day plant-back	
		DAA	mg eq/kg	DAA	mg eq/kg	DAA	mg eq/kg	DAA	mg eq/kg	DAA	mg eq/kg
Wheat	25% mature	96	0.145	152	0.073	189	0.072	203	0.133	413	0.075
Wheat	50% mature	121	0.095	189	0.059	203	0.106	229	0.245	448	0.047
Wheat	Mature fodder	248	0.249	253	0.418	254	0.448	254	0.482	568	0.078
Wheat	Mature grain	248	0.033	253	0.049	254	0.043	254	0.028	568	0.018
Mustard	Mature leaves	96	0.133	189	0.025	207	0.060	229	0.072	413	0.056
Radish	Mature leaves	96	0.159	189	0.015	207	0.043	229	0.100	413	0.078
Radish	Mature tubers	96	0.061	189	0.027	207	0.039	229	0.037	414	0.026

DAA = Days after application

The identification of the radioactive residues in wheat revealed only minor levels of pymetrozine present in forage and fodder after application of the triazine-label (0.003–0.01 mg eq/kg, 2.2–4.0% TRR). In grain sown after a 30 day plantback interval CGA96956 was the only major metabolite detected (0.013 mg eq/kg, 16.1% TRR). No other metabolite exceeded a level of 0.01 mg eq/kg or 10% TRR for both labels.

Following application of the triazine-label major residues in forage and fodder were CGA96956 (up to 0.021 mg eq/kg, 31.7% TRR) and CGA180777 including its glucose-conjugate (up to 0.016 mg eq/kg, 24% TRR). For the triazine-label CGA266591 and GS23199 and the glucose-conjugates of both represented major residues in forage and fodder.

Table 50 Distribution of [pyridine-5-¹⁴C]-labelled residues in wheat planted 30 days after soil application

Fraction	25% mature forage		50% mature forage		Mature wheat fodder		Wheat grain	
	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)
TRR	0.068		0.052		0.115		0.079	
Identified								
pymetrozine	–	–	–	–	–	–	–	–
CGA96956	0.017	25.4	0.016	31.7	0.021	18.4	0.013	16.1
CGA180777	0.011	16.4	0.003	6.4	0.001	1.2	0.001	1.7
CGA180777-gluc.	0.001	1.0	0.005	10.0	–	–	0.001	1.3
CGA180778	0.005	6.7	0.005	10.0	0.001	1.1		
CGA128632-gluc.	0.004	4.4	0.003	5.8	0.011	9.9	0.001	0.9
Characterised								
	0.002	2.3	–	–	0.002	1.7	–	–
	0.001	1.0	–	–	0.001	0.5	–	–
	0.001	1.8	–	–	< 0.001	0.2	–	–
	–	–	–	–	< 0.001	0.2	–	–
	–	–	–	–	< 0.001	0.6	–	–
	–	–	–	–	0.001	1.0	–	–
	0.002	2.6	–	–	0.001	0.8	–	–
	0.002	2.9	–	–	–	–	< 0.001	0.4
	0.001	2.0	–	–	–	–	0.001	1.0
	< 0.001	0.7	–	–	0.004	3.1	0.003	4.4
	< 0.001	0.4	–	–			0.002	3.0
	< 0.001	0.6	–	–	–	–	0.001	0.7
	< 0.001	0.6	< 0.001	0.4	–	–	< 0.001	0.2
Unextracted	0.017	25.1	0.015	28.9	0.069	60.1	0.061	77.3
% characterised		74.0		72.4		40.6		28.9
% identified		51.7		61.3		24.4		19.2

Table 51 Distribution of [pyridine-5-¹⁴C]-labelled residues in wheat planted 122 days after soil application

Fraction	25% mature forage		50% mature forage		Mature wheat fodder		Wheat grain	
	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)
TRR	0.089		0.074		0.157		0.048	
Identified								
pymetrozine	–	–	–	–	–	–	–	–
CGA96956	0.020	22.7	0.016	21.6	0.012	7.7	0.006	12.7

Fraction	25% mature forage		50% mature forage		Mature wheat fodder		Wheat grain	
	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)
CGA180777	0.012	13.7	0.008	4.7	0.009	5.6	0.001	1.7
CGA180777-gluc.	0.001	1.7	0.008	10.4	0.004	2.5	0.001	1.7
CGA180778	0.004	5.0	0.003	3.5	0.002	1.3	0.001	1.3
CGA128632-gluc.	0.01	10.9	0.006	7.5	0.01	6.5	< 0.001	0.8
Characterised								
	–	–	0.003	3.6	–	–	–	–
	0.002	2.6	0.003	3.8	0.004	2.3	< 0.001	0.3
	0.001	0.6						
	–	–	–	–	< 0.001	0.2	–	–
	–	–	–	–	0.001	0.6	–	–
	–	–	–	–	0.001	0.8	–	–
	–	–	0.001	1.9	–	–	–	–
	0.005	5.8	0.004	5.7	0.004	2.7	0.002	3.3
			0.001	1.6	0.004	2.6		
	< 0.001	0.5			0.001	0.8		
	0.003	3.3	0.003	3.6	–	–	0.002	3.2
					–	–		
					–	–	0.000	0.8
	< 0.001	0.3	–	–	0.004	2.3	< 0.001	0.1
Unextracted	0.031	34.8	0.024	32.2	0.101	64.5	0.037	77.6
% characterised		68.9		71.2		39.1		28.4
% identified		53.9		47.6		23.6		18.2

Table 52 Distribution of [pyridine-5-¹⁴C]-labelled residues in wheat planted 361 days after soil application

Fraction	25% mature forage		50% mature forage		Mature wheat fodder		Wheat grain	
	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)
TRR	0.024		0.021		0.044		0.036	
Identified								
pymetrozine	–	–	–	–	–	–	–	–
CGA96956	0.006	24.7	0.006	26.3	0.006	14.7	0.006	15.6
CGA180777	0.005	22.5	0.002	8.7	0.003	6.6	0.001	2.8
CGA180777-gluc.	< 0.001	1.6	< 0.001	1.0	0.001	1.6	< 0.001	1.2
CGA180778	0.001	6.0	0.001	3.3	0.003	5.9	< 0.001	1.0
CGA128632-gluc.	0.003	9.6	< 0.001	2.4	0.002	5.1	0.001	2.1
Characterised								
	–	–	–	–	0.001	2.2	–	–
	< 0.001	1.0	–	–			< 0.001	0.3
	< 0.001	0.5	–	–	–	–	–	–

Fraction	25% mature forage		50% mature forage		Mature wheat fodder		Wheat grain	
	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)
	—	—	< 0.001	1.3	—	—	—	—
	0.001	3.8	0.001	2.6	0.002	4.3	0.001	2.7
	< 0.001	1.1	< 0.001	0.7	0.001	1.3	0.001	2.3
	—	—	—	—	—	—		
	0.001	2.2	< 0.001	1.2	0.001	2.4	0.001	3.1
	< 0.001	0.4	< 0.001	0.7	—	—	< 0.001	0.5
Unextracted	0.006	25.2	0.008	36.0	0.024	55.3	0.018	51.3
% characterised		78.3		58.9		47.6		30.2
% identified		64.6		41.8		34.0		22.8

Table 53 Distribution of [triazine-6-¹⁴C]-labelled residues in wheat planted 30 days after soil application

Fraction	25% mature forage		50% mature forage		Mature wheat fodder		Wheat grain	
	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)
TRR	0.145		0.095		0.249		0.033	
Identified								
pymetrozine	0.004	3.0	0.003	3.6	0.006	2.2	< 0.001	1.0
CGA294849	0.001	0.8	—	—	0.003	1.3	< 0.001	1.3
CGA266591	0.010	7.0	0.026	27.6	0.009	3.6	0.001	1.5
GS23199	0.003	2.0	—	—	0.002	0.6	< 0.001	1.0
GS23199-gluc.	0.053	36.2	0.007	7.5	0.023	9.1	0.003	7.7
CGA266591-gluc.	0.003	2.4	0.002	1.9	0.008	3.1	< 0.001	1.2
Characterised								
G1	0.003	1.9	0.002	2.4	0.010	4.0	0.001	2.8
G2a	0.004	2.6	0.003	3.2	0.011	4.3		
	—	—	0.001	1.2				
G2b	0.006	4.0	0.003	3.3			0.001	3.1
G2c	0.001	0.4	0.002	2.6			< 0.001	1.0
	—	—	—	—	—	—	< 0.001	0.6
	0.002	1.1	0.001	1.1	—	—	< 0.001	0.3
G7 ^a	0.006	4.4	0.005	5.2	0.009	3.8	0.001	1.6
	—	—	0.001	0.9	—	—	< 0.001	0.6
	0.001	0.9	—	—	0.002	0.8	—	—
G10	0.002	1.5	0.002	2.0	0.003	1.2	< 0.001	0.2
Other	0.000	0.2	< 0.001	0.2	< 0.001	0.2	< 0.001	0.04
Unextracted	0.039	27.1	0.030	31.6	0.174	69.8	0.024	73.36
% characterised		76.3		68.8		36.9		23.41
% identified		55.0		45.6		22.4		14.14

^a G7—mixture of a glycoside of CGA266591 (G7b) and a glycoside of a 1,2,4-triazine-3-one-6-carboxylic acid (G7a)

Table 54 Distribution of [triazine-6-¹⁴C]-labelled residues in wheat planted 122 days after soil application

Fraction	25% mature forage		50% mature forage		Mature wheat fodder		Wheat grain	
	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)
TRR	0.133		0.245		0.482		0.028	
Identified								
Pymetrozine	0.005	3.5	0.010	4.0	0.011	2.4	—	—
CGA294849	0.001	1.0	—	—	0.008	0.6	—	—
GS23199	0.008	5.8	0.005	2.0	0.004	0.8	0.000	1.6
Gly-GS23199	0.008	5.7	0.027	11.2	0.020	4.2	0.001	2.8
CGA266591	0.003	2.6	0.011	4.6	0.030	6.3		
CGA266591-gluc.	0.014	10.3	0.013	5.5	0.013	2.8	0.000	0.4
Characterised								
G1	0.004	3.2	0.004	1.5	0.011	2.4	0.000	0.6
	—	—	0.001	0.5	—	—	0.000	0.3
G2a	0.003	2.4	0.004	1.6	0.008	1.7	0.000	0.8
G2b	0.004	3.0	0.006	2.5	0.022	4.6	0.001	4.5
G2c	—	—	0.002	1.0	0.003	0.7	0.001	4.6
G2d	—	—	0.009	3.6	—	—	0.003	10.2
	—	—	—	—	0.002	0.5	—	—
	0.001	0.6	0.001	0.3	—	—	0.000	1.0
	—	—	—	—	—	—	0.000	1.5
	—	—	0.001	0.3	0.002	0.4	—	—
G7 ^a	0.015	11.1	0.029	11.8	0.013	2.8	0.001	4.9
	—	—	—	—	0.004	0.8	—	—
	0.003	2.1	—	—	0.004	0.8	0.000	0.7
G10	0.008	2.3	0.005	1.9	0.007	1.4	—	—
	0.002	1.5	—	—	0.002	0.4	0.000	0.6
Unextracted	0.068	51.0	0.130	53.0	0.310	64.3	0.018	66.0
% characterised		53.9		58.1		37.0		38.6
% identified		40.0		38.9		17.8		9.6

^a G7—mixture of a glycoside of CGA266591 (G7b) and a glycoside of a 1,2,4-triazine-3-one-6-carboxylic acid (G7a)

Table 55 Distribution of [triazine-6-¹⁴C]-labelled residues in wheat planted 361 days after soil application

Fraction	25% mature forage		50% mature forage		Mature wheat fodder		Wheat grain	
	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)
TRR	0.075		0.047		0.078		0.018	
Identified								
pymetrozine	—	—	—	—	—	—	—	—

Fraction	25% mature forage		50% mature forage		Mature wheat fodder		Wheat grain	
	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)
CGA294849	—	—	—	—	—	—	0.000	1.1
GS23199	—	—	—	—	—	—	0.000	0.8
GS23199-gluc.	0.019	25.5	0.012	25.2	0.005	6.8	0.001	5.0
CGA266591	0.009	11.5			—	—	—	—
CGA266591-gluc.	0.002	3.0	0.002	3.4	0.003	3.3	0.000	2.2
Characterised								
G1	0.001	1.8	0.001	2.2	0.003	3.8	0.000	2.6
G2a	0.004	4.9	0.003	6.1	0.004	5.1	0.000	2.2
G2b	—	—	—	—	0.001	1.3	0.000	1.2
	—	—	—	—	0.000	0.3	—	—
	—	—	—	—	0.000	0.5	—	—
	—	—	—	—	0.001	1.1	—	—
	—	—	—	—	0.001	1.0	—	—
G7 ^a	0.003	4.4	0.002	4.4	0.003	3.6	0.000	0.8
	0.001	1.7	0.000	0.8	0.002	2.1	0.000	0.8
	0.002	3.0	—	—	0.001	1.7	—	—
	0.001	1.3	0.001	1.9	0.000	0.4	—	—
	0.001	1.1	—	—	0.001	1.4	0.000	0.2
	0.001	1.3	0.001	1.8	0.000	0.6	—	—
Unextracted	0.023	30.3	0.016	34.9	0.048	61.9	0.014	76.6
% characterised		67.5		64.6		32.8		18.7
% identified		44.5		32.9		12.5		9.8

^a G7—mixture of a glycoside of CGA266591 (G7b) and a glycoside of a 1,2,4-triazine-3-one-6-carboxylic acid (G7a)

In mustard the identification of radioactive residues showed only traces of unchanged pymetrozine (0.001–0.003 mg eq/kg).

Following application of the pyridine-label, no identified residue exceeded a level of 0.01 mg eq/kg. Major residues were CGA180777 including its glucose conjugate (up to 0.008 mg eq/kg, 16.7% TRR) and CGA180778 (up to 0.004 mg eq/kg, 17% TRR).

After treatment with triazine-labelled pymetrozine the glucose conjugate of GS23199 was the major residue for the 30d plant-back interval (0.05 mg eq/kg, 37.5% TRR) and the 361 d plant-back interval (0.008 mg eq/kg, 13.5% TRR). After a plant-back interval of 122 d the glucose conjugate of CGA266591 (0.024 mg eq/kg, 33.7% TRR) and CGA266591 itself (0.006 mg eq/kg, 8.2% TRR) were the major residues.

Table 56 Distribution of [pyridine-5-¹⁴C]-labelled residues in mustard planted 30, 122 and 361 days after soil application

Fraction	30 day mustard		122 day mustard		361 day mustard	
	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)
TRR	0.053		0.011		0.019	
Identified						

Fraction	30 day mustard		122 day mustard		361 day mustard	
	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)
pymetrozine	–	–	–	–	–	–
CGA96956	0.001	2.8	0.002	13.9	0.002	8.0
CGA180777	0.001	2.3	< 0.001	2.0	0.002	10.6
CGA180777-gluc.	0.004	6.7	< 0.001	4.4	–	–
CGA180778	0.004	10.0	0.001	12.7	0.003	17.0
Gly-CGA128632	0.003	5.2	0.001	5.8	0.001	8.4
Characterised						
	–	–	–	–	< 0.001	2.4
	0.001	2.3	< 0.001	4.5	–	–
	0.001	2.0	–	–	< 0.001	1.4
	0.001	1.0	–	–	–	–
	0.001	1.0	< 0.001	1.5	–	–
	< 0.001	0.7	< 0.001	0.5	< 0.001	1.0
	< 0.001	0.8	< 0.001	1.9	–	–
	0.001	2.1	–	–	–	–
	0.001	1.1	–	–	0.001	2.8
	0.011	21.3	–	–	–	–
	0.002	3.3	< 0.001	3.3	–	–
			< 0.001	1.4	< 0.001	1.4
	0.001	1.7	< 0.001	1.5	< 0.001	1.2
	< 0.001	0.4	< 0.001	0.7	–	–
Unextracted	0.021	38.9	0.006	50.5	0.007	38.6
% characterised		64.6		49.4		48.7
% identified		25.9		38.8		37.3

Table 157 Distribution of [triazine-6-¹⁴C]-labelled residues in mustard planted 30, 122 and 361 days after soil application

Fraction	30 day mustard		122 day mustard		361 day mustard	
	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)
TRR	0.133		0.072		0.056	
Identified						
Pymetrozine	0.001	1.1	–	–	0.003	5.1
CGA266591	0.003	2.0	0.006	8.2	0.003	4.9
Gly-CGA266591	0.001	0.4	0.024	33.7	0.005	9.2
GS23199	0.005	3.9	0.004	5.8	–	–
Gly-GS23199	0.050	37.5	0.006	8.9	0.008	13.5
CGA294849	0.003	2.2	< 0.001	1.0	–	–
Characterised						

Fraction	30 day mustard		122 day mustard		361 day mustard	
	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)
G1	–	–	0.001	1.2	0.001	2.2
G2a	0.015	11.0	0.006	8.0	0.004	7.8
G2b	0.005	4.1	0.001	1.7	0.002	2.9
G2c	0.001	0.9	< 0.001	0.5	–	–
G7	0.008	6.2	0.012	16.9	0.007	12.6
	–	–	0.003	4.4	< 0.001	0.4
	–	–	0.001	1.4	0.001	1.6
	0.001	0.6	< 0.001	0.3	0.001	1.3
Unextracted	0.035	26.5	0.015	20.5	0.016	29.4
% characterised		91.6		90.6		68.6
% identified		50.9		73.4		40.2

In radish the composition of the radioactivity was comparable to the other crops investigated. Unchanged pymetrozine was only present in traces (< 0.001–0.006 mg eq/kg, up to 4.6% TRR). Major residues identified were CGA96956 (up to 0.005 mg eq/kg, 27.7% TRR) for the pyridine-label and as CGA266591-gluc. (up to 0.029 mg eq/kg, 28.9% TRR) and GS23199-gluc. (up to 0.032 mg eq/kg, 30.1% TRR) for the triazine-label.

Table 58 Distribution of [pyridine-5-¹⁴C]-labelled residues in radishes planted 30, 122 and 361 days after soil application

Fraction	30 day foliage		30 day tubers		122 day foliage		122 day tubers		361 day foliage		361 day tubers	
	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)
TRR	0.060		0.021		0.021		0.014		0.127		0.042	
Identified												
CGA96956	0.005	9.1	0.002	10.5	0.005	22.7	0.001	6.7	0.013	10.4	0.005	12.1
CGA180777	0.003	5.5	0.001	4.9	0.001	5.0	< 0.001	3.5	0.008	6.7	0.004	9.9
Gly-CGA180777	–	–	< 0.001	1.7	–	–	< 0.001	2.2	0.002	1.6	0.001	2.2
Characterised												
Fractions 8-10	0.002	3.2	–	–	–	–	–	–	–	–	0.001	2.0
	0.002	3.1	< 0.001	1.8	< 0.001	0.5	–	–	< 0.001	0.3	0.002	5.7
	–	–	< 0.001	1.0	< 0.001	0.5	< 0.001	0.4	–	–	–	–
	–	–	–	–	–	–	–	–	0.002	1.5	< 0.001	1.2
	< 0.001	0.8	–	–	–	–	–	–	0.001	1.2	< 0.001	0.46
	0.001	1.2	0.001	6.3	< 0.001	0.8	< 0.001	0.8	0.015	11.8	0.003	7.5
	0.001	2.3	< 0.001	1.3	< 0.001	2.1	< 0.001	0.7	–	–	–	–
	0.001	1.5	–	–	–	–	–	–	–	–	–	–
	0.004	6.2	0.001	5.8	0.001	2.6	< 0.001	0.7	0.004	3.4	0.001	3.2
	0.003	5.3	0.001	3.2	< 0.001	0.8	< 0.001	0.6	0.007	5.5	0.001	3.2
	–	–	–	–	< 0.001	1.7	< 0.001	2.2	–	–	–	–
	< 0.001	0.7	–	–	< 0.001	0.9	< 0.001	0.8	0.002	1.7	–	–

Fraction	30 day foliage		30 day tubers		122 day foliage		122 day tubers		361 day foliage		361 day tubers	
	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)	mg eq/kg	TRR (%)
	< 0.001	0.7	–	–	< 0.001	0.9	–	–	–	–	–	–
	< 0.001	0.1	< 0.001	0.5	< 0.001	0.3	0.011	75.6	< 0.001	0.2	< 0.001	0.3
Unextracted	0.031	51.8	0.014	64.5	0.010	47.3		18.5	0.061	47.8	0.021	49.4
% characterised		43.2		38.6		37.2		14.6		50.4		53.7
% identified		24.3		29.9		33.1				33.8		27.3

Table 59 Distribution of [triazine-6-¹⁴C]-labelled residues in radishes planted 30, 122 and 361 days after soil application

Fraction	30 day foliage		30 day tubers		122 day foliage		122 day tubers		361 day foliage		361 day tubers	
	mg/kg	TRR (%)	mg/kg	TRR (%)	mg/kg	TRR (%)	mg/kg	TRR (%)	mg/kg	TRR (%)	mg/kg	TRR (%)
TRR	0.159		0.061		0.100		0.037		0.078		0.026	
Identified												
pymetrozine	0.006	3.5	0.001	1.6	0.003	3.1	0.002	4.2	0.004	4.6	0.001	4.4
CGA294849	0.001	0.9	–	–	0.001	1.2	< 0.001	0.2	0.001	1.1	< 0.001	0.6
CGA266591	0.002	1.0	0.001	0.9	0.003	2.8	< 0.001	0.8	0.004	5.5	< 0.001	0.9
CGA266591-gluc.	0.010	6.5	0.003	4.2	0.029	28.9	0.001	3.0	0.005	6.6	0.001	4.0
GS23199	0.012	7.3	0.003	5.5	0.010	9.6	0.002	4.9	–	–	0.001	4.8
GS23199-gluc.	0.032	30.1	0.002	3.0	0.006	5.7	< 0.001	0.6	0.005	6.1	< 0.001	1.0
Characterised												
G1	0.002	1.4	0.002	2.8	0.001	1.4	0.001	3.2	0.001	1.4	0.001	4.2
	–	–	0.001	1.0	–	–	< 0.001	1.0	–	–	–	–
G2a	0.006	3.6	0.001	1.5	0.004	3.6	< 0.001	0.7	0.001	1.2	< 0.001	1.8
G2b	0.006	4.0	0.003	5.0	0.003	3.0	< 0.001	0.8	0.002	2.6	< 0.001	1.0
G2c	0.010	6.1	< 0.001	0.6	0.001	1.2	0.001	2.4	0.002	2.1	< 0.001	1.8
G2d	–	–	0.002	2.9	0.001	0.5	< 0.001	0.3	–	–	< 0.001	0.3
	–	–	< 0.001	0.5	–	–	< 0.001	0.4	–	–	–	–
	0.002	1.0	–	–	–	–	< 0.001	0.3	< 0.001	0.4	–	–
G7	0.012	7.5	0.002	2.5	0.012	12.2	0.001	1.5	0.007	9.2	0.001	2.1
	–	–	–	–	–	–	–	–	0.001	1.0	–	–
G10	0.002	1.2	< 0.001	0.3	–	–	< 0.001	0.4	0.002	2.9	< 0.001	0.6
	0.001	0.6	–	–	–	–	–	–	–	–	–	–
	< 0.001	0.3	< 0.001	0.65	0.003	3.4	< 0.001	0.2	< 0.001	0.4	< 0.001	0.4
Unextracted	0.053	33.4	0.032	53.0	0.029	29.0	0.028	75.9	0.042	53.8	0.016	60.3
% characterised		69.8		38.0		79.1		25.0		48.0		31.3
% identified		46.6		20.6		63.5		15.1		33.0		17.9

Two additional study on the uptake of residues from soil were conducted by Gross (1995, PYMET_038) and Sandmeier (1993, PYMET_039). [Pyridine-5-¹⁴C]- or [triazine-6-¹⁴C]-pymetrozine were applied to bare soil at application rates equivalent to 0.5 kg ai/ha and 0.46 kg ai/ha, respectively. After selected plantback intervals (PBI) lettuce (PBI 36–63 d), wheat (PBI 91–106 d), sugar beets (PBI 285–307 d) and maize (PBI 285–307 d) were planted and grown to maturity.

Total radioactive residues in the samples were determined by combustion and LSC. Except for stalks of maize and wheat and wheat husks, which all contained no parent residues above the LOQ of the method (0.001–0.007 mg eq/kg), no identification of the radioactive residue was performed.

In the following table the TRR levels found are summarised.

Table 60 Total radioactive residues in rotational crops after application of 0.5 kg [pyridine-5-¹⁴C]- or 0.46 kg [triazine-6-¹⁴C]-pymetrozine per ha to bare soil

Crop	Description	[pyridine-5- ¹⁴ C]-pymetrozine			[triazine-6- ¹⁴ C]-pymetrozine		
		Plantback interval (DAT)	Harvest (DAT)	mg eq/kg	Plantback interval (DAT)	Harvest (DAT)	mg eq/kg
Lettuce	50% mature heads	63	91	0.006	36	61	0.019
	mature heads	63	116	0.005	36	77	0.007
Wheat	25% mature plant	91	216	0.004	106	178	0.013
	50% mature plant	91	307	0.007	106	284	0.005
	mature stalk	91	334	0.016	106	313	0.061
	mature husks	91	383	0.012	106	368	0.023
Sugar beet	mature grain	91	383	0.01	106	368	0.005
	25% mature tops	307	392	0.004	285	313	0.05
	25% mature roots	307	392	0.004	285	313	0.009
	50% mature tops	307	438	0.008	285	368	0.003
	50% mature roots	307	438	0.006	285	368	0.003
	mature tops	307	453	0.004	285	424	0.002
Maize	mature roots	307	453	0.004	285	424	0.003
	25% mature plants	307	363	0.012	285	313	0.006
	50% mature plants	307	392	0.007	285	336	0.004
	mature stalks	307	453	0.036	285	424	0.027
	mature cobs	307	453	0.008	285	424	0.004
	mature grain	307	453	0.009	285	424	0.002

DAT = Days after treatment

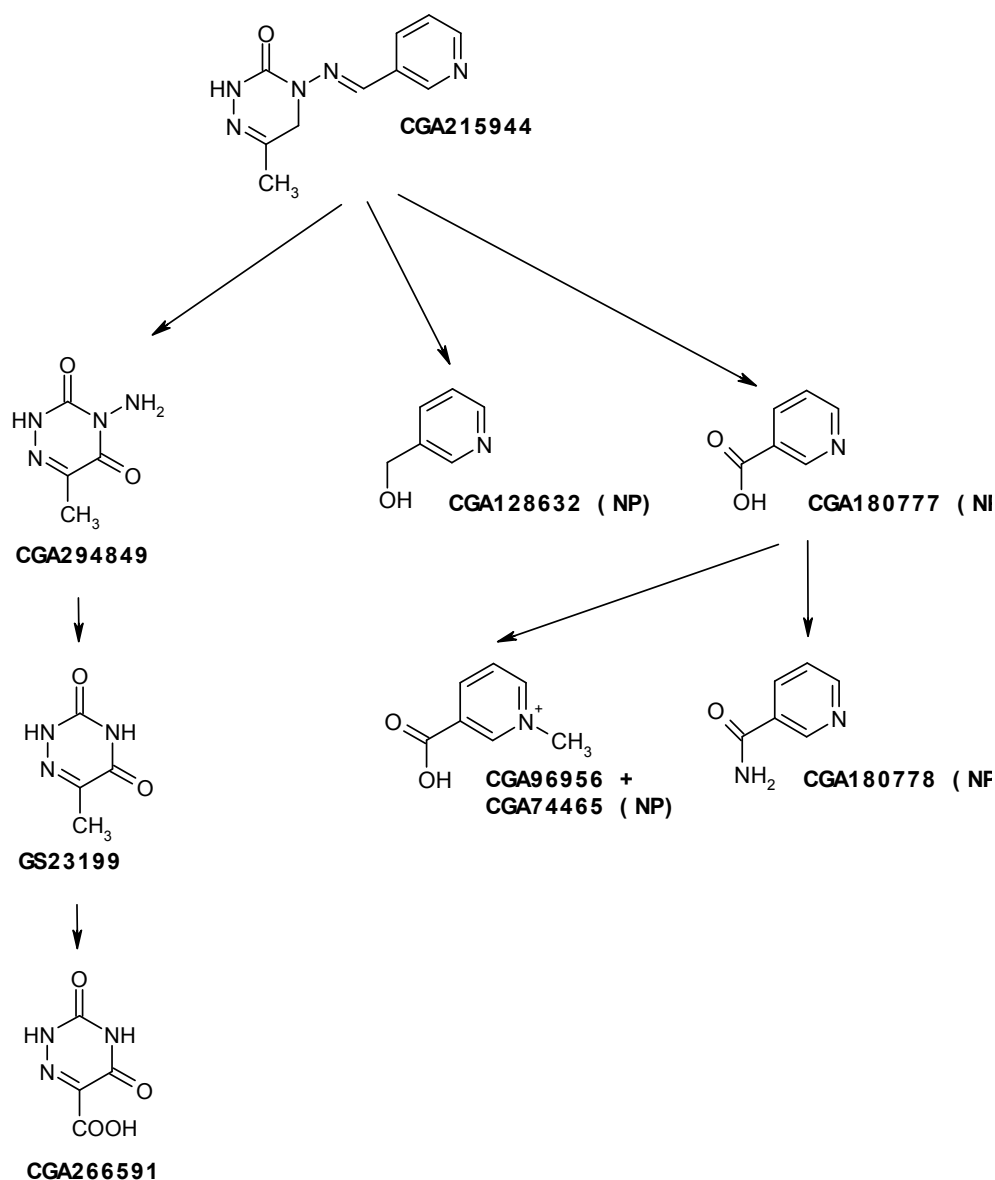


Figure 5 Proposed metabolic pathway of pymetrozine in rotational crops

Field rotational crop studies

In the USA (California, Florida and New York) four supervised field rotational crop studies have been performed by Joseph (1998, PYMET_040). Pymetrozine as a 50% WP formulation was applied four times at 7-day intervals to target crops of tomatoes, peppers, cucumbers and lettuce 0.1 kg ai/ha. Treated crops were harvested immediately after the final application and rotational crops wheat, turnips and lettuce were planted approximately 30 days after the final application and grown to maturity. All samples collected were analysed for parent pymetrozine and the metabolite GS23199.

Table 61 Residues of pymetrozine and GS23199 in rotational crops following foliar application of pymetrozine

Trial	Primary crop	Location	Application rate	Plantback interval (DAT)	Harvest interval (DAT)	Sample	pymetrozine (mg/kg)	GS23199 (mg/kg)
Trial: 02-IR-036-	Tomato	USA, CA	4 × 0.1 kg ai/ha 7 day	29	141	Immature wheat forage	< 0.02	< 0.02

Trial	Primary crop	Location	Application rate	Plantback interval (DAT)	Harvest interval (DAT)	Sample	pymetrozine (mg/kg)	GS23199 (mg/kg)
97/CA			interval					
					203	Immature wheat forage (hay)	< 0.02	< 0.02
					306	Mature wheat straw	< 0.02	< 0.02
					306	Mature wheat grain	< 0.02	< 0.02
Trial: 02-IR-037-97/CA	Pepper	USA, CA	4 × 0.1 kg ai/ha 7 day interval	31	136	Mature turnip tops	< 0.02	< 0.02
					136	Mature turnip roots	< 0.02	< 0.02
Trial: 07-IR-008-97/FL	Cucumber	USA, FL	4 × 0.1 kg ai/ha 7 day interval	32	80	Mature lettuce	< 0.02	< 0.02
					95	Mature turnip tops	< 0.02	< 0.02
					95	Mature turnip roots	< 0.02	< 0.02
					96	Immature wheat forage	< 0.02	< 0.02
					119	Immature wheat forage (hay)	< 0.02	< 0.02
					178	Mature wheat straw	< 0.02	< 0.02
					178	Mature wheat grain	< 0.02	< 0.02
Trial: 05-IR-005-97/NY	Lettuce	USA, NY	4 × 0.1 kg ai/ha 7 day interval	27	97	Mature lettuce	< 0.02	< 0.02
					97	Mature turnip tops	< 0.02	< 0.02
					97	Mature turnip roots	< 0.02	< 0.02
					89	Immature wheat forage	< 0.02	< 0.02
					287	Immature wheat forage	< 0.02	< 0.02
					343	Immature wheat forage (hay)	< 0.02	< 0.02
					372	Mature wheat straw	< 0.02	< 0.02
					372	Mature wheat grain	< 0.02	< 0.02

DAT = Days after last treatment

RESIDUE ANALYSIS

Analytical methods

For pymetrozine, analytical methods were provided following a comparable pattern. The samples (plants and animals) are extracted using aqueous borate buffer/methanol or in case of fatty matrices n-hexane. After filtration and a clean-up with C18 SPE the residue is measured by HPLC-MS/MS with two specific mass transition for each analyte. All methods were validated with an LOQ of 0.01 mg/kg.

The applicability of multi residue methods was not investigated.

Table 62 Overview of analytical methods for pymetrozine

Method	Matrix	Extraction	Clean-Up	Detection, LOQ
REM 154.09	high water acidic high oil dry	aqueous borate buffer (pH 9)/methanol (24/1 v/v)	C18 SPE	HPLC-MS/MS m/z: 218.1 → 105.1 & m/z: 218.1 → 78.2 LOQ: 0.01 mg/kg
REM 154.04	high water high oil dry	0.05 M borate buffer (pH 9) and methanol	C18 SPE	HPLC-UV, 300 nm LOQ: 0.02 mg/kg
AG-643	high water	0.05 M borate buffer	C18 SPE	HPLC-UV, 300 nm

Method	Matrix	Extraction	Clean-Up	Detection, LOQ
	high oil	(pH 9) and methanol		LOQ: 0.02 mg/kg
GRM018.01A	high water acidic	aqueous borate buffer (pH 9)/methanol (24/1 v/v)	none	HPLC-MS/MS m/z 218.1 → 105.1 LOQ: 0.005 mg/kg
Specialised rice method	dry	methanol:aqueous borate buffer	none	UPLC-MS/MS m/z: 218 → 105 & m/z: 218 → 79 LOQ: 0.001– 0.005 mg/kg
GRM018.03A	all animal matrices	Fat: hexane Others: acetonitrile/water (9/1, v/v)	C18 SPE	HPLC-MS/MS m/z: 218 → 105 & m/z: 218 → 78 LOQ: 0.01 mg/kg
GRM018.06A	all animal matrices	methanol/water (90/10 v/v)	C18 SPE	HPLC-MS/MS m/z: 234.2 → 105.1 & m/z: 234.2 → 78.1 LOQ: 0.01 mg/kg
AR-644	all animal matrices	acetonitrile/water (9+1, v/v)	C18 SPE	HPLC-UV (300 nm) LOQ: 0.01 mg/kg

Table 63 Overview of analytical methods for pymetrozine metabolites

Method, Analyte	Matrix	Extraction	Clean-Up	Detection, LOQ
GRM018.01A CGA300407	high water acidic	aqueous borate buffer (pH 9)/methanol (24/1 v/v)	none	HPLC-MS/MS m/z 108 → 80 & 108 → 53 LOQ: 0.005 mg/kg
GRM018.06A CGA313124	all animal matrices	methanol/water (90/10 v/v)	C18 SPE	HPLC-MS/MS m/z: 234.2 → 105.1 & m/z: 234.2 → 78.1 LOQ: 0.01 mg/kg
AG-658 CGA313124	Meat Liver Kidney (fat and milk not provided with acceptable recovery data)	methanol/water (90/10 v/v)	C18 SPE	HPLC-UV (300 nm) LOQ: 0.01 mg/kg

Plant materials

Method REM 154.09 (Crook, 2007, PYMET_041; Jones, 2012, PYMET_042; Amic, 2012, PYMET_043)

The analytical method REM 159.09 using HPLC-MS/MS was developed for the determination of pymetrozine in plant matrices with an LOQ of 0.01 mg/kg in high water content, acidic, high oil content and dry commodities.

Samples are extracted by homogenisation with aqueous borate buffer (pH 9)/methanol (24/1 v/v) for 3–5 minutes. Extracts are centrifuged for 5 minutes. For oilseed rape, 0.3 g C₁₈ silica is added to each aliquot (5 mL) and samples are shaken manually, vortexed and then centrifuged. Aliquots were transferred into appropriate vessels, evaporated to dryness and then reconstituted in 5 mM ammonium acetate solution. Final determination is performed by LC-MS/MS monitoring the m/z 218.1–105.1 for the primary transition and the m/z 218.1–78.2 for the confirmatory transition.

Table 64 Recovery data for method REM 159.09 in plant matrices (LC-MS/MS, m/z: 218.1 → 105.1 & m/z: 218.1 → 78.2)

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Analyte, reference, MRM transition
Cabbage	0.01	5	93–107	99	6	Pymetrozine, Crook (2007, PYMET_041)
	0.1	5	92–105	101	5	m/z: 218.1 → 105.1
Lettuce	0.01	5	103–112	107	3.5	Pymetrozine, Jones (2012, PYMET_042)
	0.5	5	103–110	106	2.9	m/z: 218.1 → 105.1
Oilseed rape seeds	0.01	5	102–120	108	6.6	
	0.5	5	101–111	105	3.8	
Oranges	0.01	5	87–94	92	3.7	
	0.5	5	101–117	110	5.4	
Wheat grain	0.01	5	82–107	98	9.9	
	0.5	5	102–108	104	2.2	
Lettuce	0.01	5	97–110	103	4.6	Pymetrozine, Jones (2012, PYMET_042)
	0.5	5	101–111	106	3.9	m/z: 218.1 → 78.2
Oilseed rape seeds	0.01	5	96–120	104	9.3	
	0.5	5	102–110	106	2.8	
Oranges	0.01	5	99–129	112	11.9	
	0.5	5	103–117	110	4.7	
Wheat grain	0.01	5	77–117	100	14.4	
	0.5	5	101–106	104	2.1	
Lettuce	0.01	5	85–91	88	3	Pymetrozine, Amic (2012, PYMET_043)
	0.1	5	89–104	96	7	m/z: 218.1 → 105.1
Oilseed rape seeds	0.01	5	100–122	108	8	
	0.1	5	96–103	98	3	
Lettuce	0.01	5	60–86	76	14	Pymetrozine, Amic (2012, PYMET_043)
	0.1	5	87–104	94	7	m/z: 218.1 → 78.2
Oilseed rape seeds	0.01	5	104–127	111	8	
	0.1	5	87–94	92	3	

Method REM 154.04 (Tribolet, 1995, PYMET_044; Tribolet, 1998, PYMET_045)

The analytical method REM 154.04 using HPLC-UV was developed for the determination of pymetrozine in plant matrices with an LOQ of 0.02 mg/kg in high water content, high oil content and dry commodities.

The crop matrix is extracted by homogenising and shaking with 0.05 M borate buffer (pH 9) and methanol. The extract is filtered and made to volume with methanol. An aliquot is diluted with borate buffer and evaporated to approximately 6 mL volume at 40 °C (high oil content crops are extracted with hexane at this stage). The extract is cleaned up on an SPE silica cartridge, and the eluate evaporated to dryness. The residue is taken up in acetone and cleaned up on a C₁₈ SPE column. The elute is evaporated to dryness and the residue reconstituted in water for analysis by HPLC with UV detection at 300 nm.

Table 65 Recovery data for method REM 159.04 in plant matrices (HPLC-UV, 300 nm)

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Analyte, reference
Tomato	0.02	6	89–111	88	10	Pymetrozine,
	0.04	2	89–91	90	–	Tribolet (1995, PYMET_044)
	0.2	3	88–100	95	7	
Bell pepper	0.02	2	77	77	–	
	0.04	1	71	71	–	
	0.2	2	74–75	75	–	
Cucurbits	0.02	2	75–85	80	–	

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Analyte, reference
	0.04	1	73	73	–	
	0.2	2	80–86	83	–	
Potatoes	0.02	2	78–86	72	–	
	0.04	1	69	69	–	
	0.2	2	64–76	69	–	
Green tobacco leaves	0.02	2	82–89	86	–	
	0.04	1	90	90	–	
	0.2	2	78–92	85	–	
Rice grain	0.02	2	72–95	84	–	
	0.04	1	57	57	–	
	0.2	2	69–86	78	–	
Cotton seed	0.02	6	67–85	76	9	
	0.04	2	69–82	76	–	
	0.2	3	75–95	84	12	
Rice straw	0.02	5	88–115	101	10	
	0.04	1	102	102	–	
	0.2	2	62–80	71	–	
Lettuce	0.02	15	71–105	92	11	Pymetrozine,
	0.2	15	69–102	86	12	Tribolet (1998, PYMET_045)
Cabbage	0.02	15	70–122	86	17	
	0.2	15	73–108	89	10	
Sweet peppers	0.02	10	53–102	82	18	
	0.2	10	77–97	88	9	
Tomatoes	0.02	14	72–102	89	11	
	0.2	14	70–103	85	14	
Potatoes	0.02	4	81–95	89	7	
	0.2	4	81–88	86	4	
Melons	0.02	6	68–99	91	13	
	0.2	6	61–105	85	18	
Peaches	0.02	5	81–108	92	13	
	0.2	5	71–91	80	10	
Eggplant	0.02	2	85–99	92	–	
	0.2	2	78–84	81	–	
Cucumbers	0.02	3	80–100	90	10	
	0.2	3	72–83	84	11	
Squash	0.02	2	95–111	103	–	
	0.2	2	92–99	96	–	
Sugar beet leaves	0.02	2	93–94	94	–	
	0.2	2	82–85	84	–	
Sugar beet roots	0.02	2	86–98	92	–	
	0.2	2	83–85	84	–	

Method AG-643 (Joseph, 1996, PYMET_046; Mayer, 1996, PYMET_047; Joseph, 1999, PYMET_048)

The analytical method AG-643 using HPLC-UV was developed for the determination of pymetrozine in plant matrices with an LOQ of 0.02 mg/kg in high water content and high oil content commodities.

Crop matrix is extracted by shaking with 0.05 M borate buffer in methanol. The extract is diluted and an aliquot reduced in volume by evaporation. The extract is cleaned up on a diatomaceous earth SPE cartridge and then on a silica SPE cartridge. The eluate is reconstituted in mobile phase and analysed by HPLC with UV detection (300 nm).

Table 66 Recovery data for method AG-643 in plant matrices (HPLC-UV, 300 nm)

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Analyte, reference
Cucumber	0.02	2	118–122	120	–	Pymetrozine,
	0.05	2	83–93	88	–	Joseph (1996, PYMET_046)

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Analyte, reference
	0.1	1	67	67	–	
	0.2	2	64–71	68	–	
Cantaloupes	0.02	2	100–122	111	–	
	0.05	2	74–86	80	–	
	0.1	2	73–80	77	–	
	0.2	1	73	73	–	
Tomatoes	0.02	2	114–116	115	–	
	0.05	2	86–88	87	–	
	0.1	2	80	80	–	
	0.2	2	72–72	73	–	
Tomato pomace, wet	0.02	2	103–113	108	–	
	0.05	2	85–87	86	–	
	0.1	2	77–79	78	–	
	0.2	1	68	68	–	
Tomato pomace, dry	0.02	2	97–98	98	–	
	0.05	2	70–84	77	–	
	0.1	2	79–81	80	–	
	0.2	1	67	67	–	
Peppers	0.02	2	65–65	65	–	
	0.05	2	71–78	75	–	
	0.1	2	63–67	65	–	
	0.2	1	74	74	–	
Tobacco, green	0.02	2	81–84	83	–	
	0.05	2	65–85	75	–	
	0.1	2	73–77	75	–	
	0.2	1	73	73	–	
Tobacco, cured	0.02	2	65–77	71	–	
	0.05	2	69–75	72	–	
	0.1	2	62–73	68	–	
	0.2	2	62–64	63	–	
Pecan	0.02	12	69–117	90	15	Pymetrozine,
	0.05	4	79–82	86	7	Mayer (1996, PYMET_047)
	0.1	4	70–90	82	11	
	0.2	4	64–98	83	17	
Tomato paste	0.02	2	66–87	76	–	Pymetrozine,
	0.2	2	78–81	79	–	Joseph (1999, PYMET_048)
Tomatoes	0.02	2	95–119	107	–	
	0.2	2	86–96	91	–	
Cucumbers	0.02	2	85–98	92	–	
	0.2	2	90–92	91	–	

Method GRM018.01A (Crook, 2007, PYMET_049)

The analytical method GRM018.01A using HPLC-MS/MS was developed for the determination of pymetrozine and CGA300407 in plant matrices with an LOQ of 0.005 mg/kg in high water content and acidic commodities.

The crop matrix is extracted with methanol:aqueous borate buffer pH 9 (36:4 v/v). The homogenate is filtered and the filtrate washed with methanol to give an extract concentration of 0.1 g/mL. The extract is evaporated under an air stream and the residue made up to 2 mL with 5 mM ammonium acetate. This final extract is analysed by LC-MS/MS for pymetrozine (m/z 218 $\rightarrow$ 105) and CGA300407 (m/z 108 $\rightarrow$ 80 & m/z 108 $\rightarrow$ 53) residues.

Table 67 Recovery data for method GRM018.01A in plant matrices

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Analyte, reference
Lettuce	0.005	5	85–108	95	10	Pymetrozine,
	0.05	5	91–98	95	3	Crook (2007, PYMET_049)
Oranges	0.005	5	82–101	96	8	m/z: 218.1 → 105.1
	0.05	5	88–94	91	2	
Sweet pepper	0.01	4	67–104	89	19	
	0.1	4	73–108	92	19	
Cucumber	0.01	4	89–108	101	9	
	0.1	4	93–106	100	5	
Lettuce	0.005	5	71–92	83	9	CGA300407,
	0.05	5	67–83	74	8	Crook (2007, PYMET_049)
Oranges	0.005	5	104–114	108	3	m/z 108 → 80 &
	0.05	5	91–96	94	3	m/z 108 → 53
Sweet pepper	0.01	4	63–98	77	20	
	0.1	4	70–80	72	10	
Cucumber	0.005	2	69–86	78	–	
	0.1	4	60–82	72	13	
	0.1	4	69–90	77	13	

Method for rice (Wu, 2009; PYMET_050)

This specialised method was developed for data generation purposes in supervised field trials on rice. Parent pymetrozine is analysed by UPLC-MS/MS (m/z 218 → 105 & 218 → 79) with a LOQ of 0.001–0.005 mg/kg in dry matrices.

The crop matrix is extracted with methanol:aqueous borate buffer. The homogenate is filtered and the filtrate washed with methanol. The extract is evaporated by rotary evaporator. This final extract is analysed for pymetrozine residue by UPLC-MS/MS to an LOQ of 0.001–0.005 mg/kg (m/z 218 → 105 & 218 → 79).

Table 68 Recovery data for a specialised rice method (UPLC-MS/MS, m/z: 218 → 105 & m/z: 218 → 79)

Matrix	Fortification level (mg/kg)	Recovery, mean (%)	RSD (%)	Analyte, reference
Rice straw	0.001	89	6	Pymetrozine,
	0.01	93	4	Wu (2009, PYMET_050)
	0.5	98	5	m/z: 218.1 → 105 &
Rice husks	0.005	105	6	m/z: 218.1 → 79
	0.01	98	4	
	0.5	85	5	
Rice grain	0.005	84	6	
	0.01	92	7	
	0.5	88	4	

Animal materials

Method GRM018.03A (Class, 2011, PYMET_051; Schwarz & Class, 2011, PYMET_052; Jones, 2012, PYMET_053)

The analytical method GRM018.03A using HPLC-MS/MS was developed for the determination of pymetrozine in animal commodities with an LOQ of 0.01 mg/kg in all tissues, eggs and milk.

Samples of milk, eggs, meat, liver and kidney are extracted by homogenisation with acetonitrile/water (9/1, v/v). Extracts are centrifuged and the supernatant is decanted. Sodium tetraborate solution (0.2 M) is added and the volume of the sample is made up to 100 mL with acetonitrile/water (9/1, v/v). The extract is purified using C₁₈-treated silica product and centrifuged. An aliquot of the resulting solution is taken, diluted in acetonitrile: ammonium acetate (1/4, v/v).

Samples of fat are dissolved in hexane and sonicated. The samples are then extracted with acetonitrile. Sodium tetraborate solution (0.2M) is added and the volume of the sample is made up to 50 mL with acetonitrile/water (9/1, v/v) prior to being frozen at -18 °C for 2 hours. The extract is then cleaned up using C₁₈-treated silica product and centrifuged. An aliquot of the resulting solution is diluted in acetonitrile/ ammonium acetate (1/4, v/v).

Final determination is performed by HPLC-MS/MS (m/z: 218 → 105 & m/z: 218 → 79).

Table 69 Recovery data for method GRM018.03A in animal matrices (LC-MS/MS, m/z: 218 → 105 & m/z: 218 → 78)

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Analyte, reference, MRM transition
Milk	0.01	5	96–100	98	1	Pymetrozine,
	0.1	5	103–107	105	2	Class (2011, PYMET_051) &
Egg	0.01	5	98–109	104	4	Schwarz & Class (2011, PYMET_052)
	0.1	5	107–111	109	1	m/z: 218 → 105
Meat	0.01	5	98–102	101	2	
	0.1	5	99–102	101	1	
Liver	0.01	5	93–104	98	4	
	0.1	5	93–105	99	5	
Kidney	0.01	5	81–89	86	4	
	0.1	5	80–93	88	6	
Fat	0.01	5	80–87	84	4	
	0.1	5	84–91	87	3	
Milk	0.01	5	101–105	103	2	Pymetrozine,
	0.1	5	104–107	105	1	Class (2011, PYMET_051) &
Egg	0.01	5	104–109	107	2	Schwarz & Class (2011, PYMET_052)
	0.1	5	107–110	109	1	m/z: 218 → 78
Meat	0.01	5	97–105	102	3	
	0.1	5	98–102	100	1	
Liver	0.01	5	90–110	99	7	
	0.1	5	93–105	100	5	
Kidney	0.01	5	82–86	85	2	
	0.1	5	81–93	87	5	
Fat	0.01	5	78–86	82	4	
	0.1	5	82–89	85	3	
Milk	0.01	5	99–118	113	7	Pymetrozine,
	0.5	5	83–101	92	8	Jones (2012, PYMET_053)
Bovine liver	0.01	5	99–111	104	4	m/z: 218 → 105
	0.5	5	91–108	102	7	
Milk	0.01	5	89–113	106	9	Pymetrozine,
	0.5	5	84–101	93	7	Jones (2012, PYMET_053)
Bovine liver	0.01	5	107–112	109	2	m/z: 218 → 78
	0.5	5	89–108	102	7	

Method GRM018.06A (Amic, 2011, PYMET_054; Jones, 2012, PYMET_053)

The analytical method GRM018.06A using HPLC-MS/MS was developed for the determination of CGA313124 and its phosphate conjugate in animal commodities with an LOQ of 0.01 mg/kg in all tissues, eggs and milk.

Samples are extracted by homogenisation twice with methanol/ultra pure water (90/10 v/v). Milk samples and whole blood samples are extracted for the first homogenisation with methanol only. Extracts are filtered and made up to a volume of 150 mL with methanol. An aliquot (30 mL) is then evaporated to an aqueous residue. Milk samples are then placed in an oven at 60 °C for 30 minutes. The extract is purified on a C₁₈ solid phase extraction (SPE) cartridge using methanol as the eluent. The eluent is collected and then evaporated to dryness. The dry extract is dissolved in methanol/ultra pure water (10/90, v/v), followed by final determination of the pymetrozine metabolite CGA313124 by LC-MS/MS (m/z 234.2 → 105.1 & 234.2 → 78.1).

Table 70 Recovery data for method GRM018.06A determining CGA313124 in animal matrices (LC-MS/MS, m/z: 234.2 → 105.1 & m/z: 234.2 → 78.1)

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Analyte, reference, MRM transition
Milk	0.01	5	90–97	92	3	CGA313124,
	0.1	5	93–98	95	2	Amic (2011, PYMET_054)
Egg	0.01	5	94–100	98	3	m/z: 234.2 → 105.1
	0.1	5	93–99	96	3	
Bovine muscle	0.01	5	84–91	87	4	
	0.1	5	89–92	91	1	
Bovine fat	0.01	5	81–95	87	8	
	0.1	5	71–84	77	6	
Bovine liver	0.01	5	88–95	92	3	
	0.1	5	89–95	92	3	
Bovine kidney	0.01	5	99–103	101	1	
	0.1	5	98–104	101	3	
Milk	0.01	5	90–96	94	3	CGA313124,
	0.1	5	89–94	92	2	Amic (2011, PYMET_054)
Egg	0.01	5	98–101	99	1	m/z: 234.2 → 78.1
	0.1	5	93–98	95	2	
Bovine muscle	0.01	5	81–92	87	5	
	0.1	5	90–93	91	1	
Bovine fat	0.01	5	83–99	90	7	
	0.1	5	76–82	78	3	
Bovine liver	0.01	5	86–95	89	5	
	0.1	5	86–95	90	4	
Bovine kidney	0.01	5	93–101	97	3	
	0.1	5	95–103	100	3	
Milk	0.01	5	86–94	91	4	CGA313124,
	0.1	5	88–92	90	2	Jones (2012, PYMET_053)
Eggs	0.01	5	79–84	81	3	m/z: 234.2 → 105.1
	0.1	5	75–83	78	4	
Bovine liver	0.01	5	72–87	80	9	
	0.1	5	80–88	83	4	
Milk	0.01	5	88–93	91	3	CGA313124,
	0.1	5	86–90	88	2	Jones (2012, PYMET_053)
Eggs	0.01	5	79–84	82	3	m/z: 234.2 → 78.1

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Analyte, reference, MRM transition
	0.1	5	75–81	77	4	
Bovine liver	0.01	5	75–89	82	8	
	0.1	5	81–89	83	4	

Method AG-644 (Joseph, 1996, PYMET_055; Wang, 1997, PYMET_056)

Method AG-644 was developed for the determination of pymetrozine in animal products (meat, fat, liver, kidney, milk and eggs) using HPLC-UV (300 nm).

The sample was homogenised twice with acetonitrile/water (9 + 1, v/v), followed by filtration and passing of the extract through a C₁₈ SPE cartridge. After addition of Borax buffer to the eluate, the solution is evaporated of solvent until only an aqueous phase remains. The analyte is partitioned into ethyl acetate on an Extrelut cartridge, followed by evaporation of the organic phase to dryness. Cleanup of residue is performed on a silica SPE cartridge, eluted with methanol. The final determination was performed by HPLC on a two-column switching system (column 1: CN; column 2: C₁₈ reversed phase) with UV-detection (300 nm).

Table 71 Recovery data for method AG-644 in animal matrices (HPLC-UV, 300 nm)

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Analyte, reference, Detection
Meat ^a	0.01	5	97–90	93	3	Pymetrozine,
	0.1	2	82–68	75	–	Joseph (1996, PYMET_055)
	0.2	4	82–50	67	16	
	0.5	4	83–44	67	17	
Fat ^a	0.01	5	115–68	90	18	
	0.1	2	83–81	82	–	
	0.2	4	79–71	76	4	
	0.5	4	82–76	78	3	
Liver ^a	0.01	5	93–77	85	6	
	0.1	2	75–74	75	–	
	0.2	4	77–68	73	4	
	0.5	4	81–48	66	16	
Kidney ^a	0.01	3	104–87	95	9	
	0.2	3	73–60	67	7	
	0.5	1	75	75	–	
	1.0	2	64–56	60	–	
Milk ^a	0.01	3	99–87	94	6	
	0.2	3	85–74	79	6	
	0.5	3	72–67	70	3	
Eggs ^a	0.01	2	89–75	82	–	
	0.1	2	62–61	62	–	
	0.2	2	64–62	63	–	
	0.5	2	66–66	66	–	
Liver	0.01	4	46–105	75	25	Pymetrozine,
	0.1	4	76–91	82	7	Wang (1997, PYMET_056)
Milk	0.01	4	90–98	94	4	
	0.1	4	60–68	65	4	
Eggs	0.01	4	76–94	83	8	

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Analyte, reference, Detection
	0.1	4	68–88	82	9	

^a Tested on bovine, goat and poultry tissues, milk and eggs

Method AG-658 (Joseph, 1996, PYMET_057)

Method AG-658 was developed for the determination of CGA313124 in animal tissues (meat, fat, liver and kidney) and of CGA 313124 and its phosphate conjugate in milk using HPLC-UV (300 nm).

Samples are extracted by homogenisation twice with methanol/water (90/10 v/v). Milk samples are extracted for the first homogenisation with methanol only. Extracts are filtered and passed through a C₁₈ SPE cartridge. Methanol is then evaporated from the resultant extract. Milk samples are placed in an oven at 60 °C for 30 minutes. The aqueous extract is filtered using a phenyl SPE cartridge and the eluate is then diluted with water. A C₁₈ SPE cartridge is used to perform a solvent exchange into methanol before the methanol solution is evaporated to dryness. The analyte is dissolved in methylene chloride/methanol (90/10 v/v), followed by final determination of CGA313124 by HPLC with UV detection (300 nm).

Table 72 Recovery data for method AG-658 determining CGA313124 in animal matrices (HPLC-UV, 300 nm)

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Analyte, reference, Detection
Meat ^a	0.01	2	93–125	109	–	CGA313124, Joseph (1996, PYMET_057)
	0.1	2	61–84	73	–	
	0.2	2	83–90	87	–	
Fat ^a	0.01	3	22–131	70	56	
	0.1	3	49–82	60	19	
	0.2	3	38–73	52	19	
Liver ^a	0.01	2	136–141	139	–	
	0.1	2	96–100	98	–	
	0.2	2	87–99	93	–	
Kidney ^a	0.01	2	102–107	105	–	
	0.1	2	82–83	83	–	
	0.2	2	73–92	78	–	
Milk ^a	0.01	2	57–69	63	–	
	0.02	1	102	102	–	
	0.05	1	72	72	–	
	0.1	2	61–65	63	–	
	0.2	1	63	63	–	
	0.5	1	69	69	–	

^a Validated for bovine and goat tissue and milk

Stability of pesticides in stored analytical samples

Plant matrices

Tribolet (1996, PYMET_058)

Samples of tomatoes, potatoes and cottonseed were separately fortified with pymetrozine at a nominal rate of 0.5 mg/kg. A sample of 2.5–2.8 kg was homogenised under addition of dry-ice to keep the sample frozen. During the homogenization a solution containing pymetrozine was added to reach the desired final concentration. The sample was split into sub-samples for storage.

Two sub-samples were taken and analysed for residues of the fortified material immediately following fortification. The results of these samples revealed an insufficient homogenization, resulting

in a recombination of all samples and further homogenization after 8 days. The rehomogenized samples were stored deep frozen below -18 °C for up to 24 months with duplicate sub-samples being taken at intervals of 1, 2, 4, 6, 12, 18 and 25/26 months and analysed for residues of pymetrozine.

Table 73 Recovered pymetrozine residues in stored plant commodities after storage up to 25 months

Matrix	Fortification level (mg/kg)	Storage period (months)	Residue level in stored samples ^a				Procedural recovery	
			Individual values (mg/kg)	Mean (mg/kg)	Mean (%)	RSD (%)	Individual values (%)	Mean (%)
Tomato	0.5	0	0.45, 0.43, 0.48, 0.47, 0.44, 0.47	0.456	100	5	84, 85	85
		1	0.39, 0.37, 0.43	0.396	87	8	70, 89	80
		2	0.37, 0.47, n.a.	0.42	92	–	76, 78	77
		4	0.3, 0.41, n.a.	0.36	79	–	89, 89	89
		6	0.36, 0.38, n.a.	0.37	81	–	89, 94	92
		12	0.28, 0.28, 0.35	0.3	66	13	87, 66	76
		18	0.25, 0.26, 0.25	0.25	55	2	86, 83	85
		25	0.25, 0.22, 0.21	0.23	50	9	96, 89	93
Potato	0.5	0	0.41, 0.4, 0.4	0.4	100	1	95, 92	94
		1	0.28, 0.27, 0.33	0.29	73	11	84, 75	80
		2	0.23, 0.3, n.a.	0.22	55	–	76, 79	78
		4	0.24, 0.25, n.a.	0.25	63	–	86, 71	89
		6	0.25, 0.25, 0.27	0.26	65	4	76, 77	77
		12	0.22, 0.19, 0.22	0.21	53	8	75, 85	80
		18	0.22, 0.21, 0.22	0.22	55	3	77, 70	74
		25	0.21, 0.22, 0.24	0.22	55	7	86, 90	88
Cotton seed	0.5	0	0.33, 0.41, 0.45	0.4	100	15	68, 81	75
		1	0.34, 0.34, 0.32	0.33	83	3	66, 73	70
		2	0.41, 0.37, n.a.	0.39	98	–	83, 76	80
		4	0.4, 0.37, n.a.	0.39	98	–	82, 76	79
		6	0.42, 0.42, n.a.	0.42	105	–	91, 97	94
		12	0.34, 0.31, 0.31	0.32	80	5	77, 67	72
		18	0.4, 0.4, 0.38	0.39	98	3	75, 69	72
		25	0.32, 0.34, 0.36	0.34	85	6	69, 66	68

^a Not corrected for procedural recovery

n.a. = Not analysed

Boyette (1998, PYMET_059)

Samples of cucumbers, tomatoes, tomato paste, cottonseed and cottonseed oil were separately fortified with pymetrozine and GS23199 at a nominal rate of 1.0 mg/kg each. Two sub-samples were immediately taken and analysed for residues of the fortified material. The remaining samples were stored deep frozen at approximately -20 °C for up to 24 months with duplicate sub-samples being taken at intervals and analysed for residues.

Table 74 Recovered pymetrozine and GS23199 residues in stored plant commodities after storage up to 24 months

Matrix	Fortification level (mg/kg)	Storage period (months)	Pymetrozine		GS23199			
			Residue level in stored samples ^a		Procedural recovery	Residue level in stored samples ^a		Procedural recovery
			Individual values in mg/kg (mean)	% Mean	Individual values in % nominal (mean)	Individual values in mg/kg (mean)	% Mean	Individual values in % nominal (mean)
Cucumbers	1	0	0.78, 0.79 (0.79)	100	81, 80 (81)	0.89, 0.91 (0.9)	100	90, 87 (89)
		2	0.63, 0.59 (0.61)	77	82, 92 (87)	0.87, 0.86 (0.87)	97	94, 89 (92)

Matrix	Fortification level (mg/kg)	Storage period (months)	Pymetrozine			GS23199		
			Residue level in stored samples ^a		Procedural recovery	Residue level in stored samples ^a		Procedural recovery
			Individual values in mg/kg (mean)	% Mean	Individual values in % nominal (mean)	Individual values in mg/kg (mean)	% Mean	Individual values in % nominal (mean)
		4	0.49, 0.5 (0.5)	63	84, 80 (82)	0.83, 0.83 (0.83)	92	86, 86 (86)
		6	0.39, 0.37 (0.38)	48	82, 76 (79)	0.84, 0.82 (0.83)	92	83, 86 (85)
		10	0.17, 0.13 (0.15)	19	89, 85 (87)	0.83, 0.66 (0.75)	83	86, 86 (86)
		12	0.1, 0.22 (0.16)	20	78, 91 (85)	0.7, 0.75 (0.73)	81	87, 91 (89)
		14	0.2, 0.05 (0.13)	16	79, 70 (75)	0.85, 0.84 (0.85)	94	97, 95 (96)
		18	0.11, 0.17 (0.14)	18	76, 80 (78)	0.83, 0.84 (0.84)	93	92, 91 (92)
		24	0.12, 0.03 (0.08)	10	83, 79 (81)	0.85, 0.81 (0.83)	92	90, 89 (90)
Tomato,	1	0	0.83, 0.81 (0.82)	100	85, 82 (84)	0.85, 0.89 (0.87)	100	86, 86 (86)
fruit		2	0.8, 0.79 (0.8)	98	86, 72 (79)	0.83, 0.82 (0.83)	95	85, 85 (85)
		4	0.7, 0.75 (0.73)	89	82, 74 (78)	0.87, 0.86 (0.87)	100	90, 92 (91)
		6	0.85, 0.85 (0.85)	104	97, 93 (95)	0.8, 0.8 (0.8)	92	80, 81 (81)
		10	0.76, 0.73 (0.75)	91	87, 87 (87)	0.85, 0.79 (0.82)	94	87, 91 (89)
		12	0.72, 0.78 (0.75)	91	91, 94 (93)	0.91, 0.86 (0.89)	102	83, 85 (84)
		14	0.63, 0.63 (0.63)	77	81, 68 (75)	0.83, 0.81 (0.82)	94	88, 90 (89)
		18	0.48, 0.55 (0.52)	63	77, 74 (76)	0.82, 0.82 (0.82)	94	94, 87 (91)
		24	0.56, 0.79 (0.68)	83	95, 84 (90)	0.84, 0.81 (0.83)	95	90, 84 (87)
Tomato,		0	0.77, 0.75 (0.76)	100	82, 81 (82)	0.82, 0.82 (0.82)	100	78, 83 (81)
paste		2	0.57, 0.62 (0.6)	79	85, 77 (81)	0.9, 0.9 (0.9)	110	82, 88 (85)
		4	0.7, 0.66 (0.68)	89	72, 75 (74)	0.74, 0.75 (0.75)	91	74, 85 (80)
		6	0.62, 0.54 (0.58)	76	83, 81 (82)	0.76, 0.73 (0.75)	91	79, 80 (80)
		10	0.51, 0.53 (0.52)	68	86, 73 (80)	0.78, 0.76 (0.77)	94	75, 84 (80)
		12	0.47, 0.38 (0.43)	57	95, 108 (102)	0.82, 0.79 (0.81)	99	83, 90 (87)
		14	0.46, 0.45 (0.46)	61	71, 73 (72)	0.72, 0.71 (0.72)	88	76, 78 (77)
		18	0.39, 0.46 (0.43)	57	83	0.76, 0.78 (0.77)	94	86, 85 (86)
		24	0.27, 0.33 (0.3)	39	75, 77 (76)	0.59, 0.68 (0.64)	78	83, 78 (81)
Cotton,	1	0	0.83, 0.84 (0.84)	100	90, 85 (88)	0.88, 0.86 (0.87)	100	90, 87 (89)

Matrix	Fortification level (mg/kg)	Storage period (months)	Pymetrozine			GS23199		
			Residue level in stored samples ^a		Procedural recovery	Residue level in stored samples ^a		Procedural recovery
			Individual values in mg/kg (mean)	% Mean	Individual values in % nominal (mean)	Individual values in mg/kg (mean)	% Mean	Individual values in % nominal (mean)
seed		2	0.7, 0.66 (0.68)	81	82, 78 (80)	0.82, 0.84 (0.83)	95	84, 88 (86)
		4	1.25, 1.07 (1.16)	138	101, 121 (111)	0.98, 0.98 (0.98)	113	104, 99 (102)
		6	0.68, 0.74 (0.71)	85	82, 82 (82)	0.8, 0.79 (0.8)	92	83, 85 (84)
		10	1.03, 1.11 (1.07)	127	130, 123 (127)	0.82, 0.82 (0.82)	94	82, 78 (80)
		12	0.83, 0.67 (0.75)	89	89, 96 (93)	0.74, 0.8 (0.77)	89	78, 82 (80)
		14	0.65, 0.69 (0.67)	80	63, 71 (67)	0.81, 0.81 (0.81)	93	85, 86 (86)
		18	0.83, 0.81 (0.82)	98	96, 89 (93)	0.79, 0.7 (0.75)	86	90, 87 (89)
		24	0.79, 0.7 (0.75)	89	95, 89 (92)	0.77, 0.79 (0.78)	90	86, 83 (85)
Cotton,		0	0.88, 0.82 (0.85)	100	82, 92 (87)	0.93, 0.94 (0.94)	100	93, 93 (93)
oil		2	1.19, 1.17 (1.18)	139	114, 107 (111)	0.93, 0.9 (0.92)	98	90, 94 (92)
		4	0.87, 0.92 (0.9)	106	80, 92 (86)	0.85, 0.83 (0.84)	89	90, 93 (92)
		6	0.82, 0.83 (0.83)	98	87, 86 (87)	0.82, 0.81 (0.82)	87	85, 88 (87)
		10	0.83, 0.76 (0.8)	94	80, 90 (85)	0.84, 0.82 (0.83)	88	91, 93 (92)
		12	0.71, 0.61 (0.66)	78	76, 73 (75)	0.87, 0.82 (0.85)	90	93, 87 (90)
		14	0.68, 0.59 (0.64)	75	79, 71 (75)	0.89, 0.96 (0.93)	99	100, 97 (99)
		18	0.71, 0.62 (0.67)	79	71, 88 (80)	0.79, 0.84 (0.82)	87	94, 86 (90)
		24	0.73, 0.59 (0.66)	78	85, 85 (85)	0.66, 0.77 (0.72)	77	75, 72 (74)

^a Not corrected for procedural recovery

Tribolet (2001, PYMET_060)

Specimens of dried hops were individually fortified with pymetrozine and GS23199 at a rate of 5 mg/kg for each analyte. Three replicate specimens of each plant matrix were analysed at zero time and two replicate specimens of each plant matrix were removed after 0, 3, 6, 9 and 12 months storage at a nominal temperature of $\leq -18^{\circ}\text{C}$ and analysed. In parallel analysis of two freshly prepared fortified specimens was performed on each date of analysis.

Table 75 Recovered pymetrozine and GS23199 residues in stored hops after storage up to 12 months

Matrix	Fortification level (mg/kg)	Storage period (months)	Pymetrozine		GS23199	
			Residue level in stored samples ^a	Procedural recovery	Residue level in stored samples ^a	Procedural recovery

			Individual values in mg/kg (mean)	% Mean	Individual values in % nominal (mean)	Individual values in mg/kg (mean)	% Mean	Individual values in % nominal (mean)
Hops, dry	5	0	3.4, 3.7, 3.6 (3.6)	100	74, 74, 65 (71)	3.5, 3.3, 4.0 (3.6)	100	66, 70 (68)
		3	2.4, 2.9, 2.8, 3.1, 3.7, 3.5 (3.1)	86	68, 71, 68, 69 (69)	3.2, 3.6, 3.8 (3.5)	97	69, 62 (66)
		6	3.0, 3.2, 2.4 (2.9)	81	64, 80 (67)	3.4, 3.8, 3.7 (3.6)	100	80, 78 (79)
		9	2.3, 2.8, 2.8 (2.6)	72	67, 64 (66)	3.6, 3.5, 3.9 (3.7)	103	78, 68 (73)
		12	3.2, 3.2, 2.7 (3.0)	83	66, 81 (74)	3.5, 3.3, 4.0 (3.6)	100	67, 67 (67)

^a Not corrected for procedural recovery

Schulz (2011, PYMET_061)

Samples of peach (fruit without stone), melons, cucumber, lettuce and oranges were homogenized under addition of dry ice and separately fortified with pymetrozine at a nominal rate of 0.5 mg/kg. Two sub-samples were immediately taken and analysed for residues of pymetrozine. The remaining samples were stored deep frozen at approximately -18 °C for up to 24 months with duplicate sub-samples being taken at intervals and analysed for residues.

Table 76 Recovered pymetrozine residues in stored plant commodities after storage up to 24 months

Matrix	Fortification level (mg/kg)	Storage period (months)	Residue level in stored samples ^a			Procedural recovery	
			Individual values (mg/kg)	Mean (mg/kg)	Mean (%)	Individual values (%)	Mean (%)
Orange	0.5	0	0.49, 0.51, 0.51	0.5	100	102, 102	102
		1	0.5, 0.51	0.51	102	106, 101	104
		3	0.43, 0.43	0.43	86	95, 94	95
		6	0.46, 0.48	0.47	94	104, 102	103
		12	0.42, 0.41	0.42	84	95, 95	95
		18	0.36, 0.36	0.36	72	91, 90	91
		24	0.37, 0.34	0.36	72	97, 96	97
Peaches	0.5	0	0.5, 0.52, 0.47	0.5	100	105, 102	104
		1	0.5, 0.51	0.51	102	97, 99	98
		3	0.29, 0.3	0.3	60	91, 89	90
		6	0.29, 0.32	0.31	62	98, 99	99
		12	0.3, 0.29	0.3	60	97, 98	98
		18	0.28, 0.28	0.28	56	91, 87	89
		24	0.24, 0.23	0.24	48	95, 96	96
Cucumber	0.5	0	0.49, 0.52, 0.51	0.51	100	100, 100	100
		1	0.49, 0.48	0.49	96	102, 102	102
		3	0.33, 0.31	0.32	63	91, 88	90
		6	0.19, 0.18	0.19	37	100, 103	102
		12	0.1, 0.09	0.1	20	93, 95	94
		18	0.06, 0.06	0.06	12	95, 95	95
		24	0.03, 0.03	0.03	6	90, 90	90
Melon	0.5	0	0.54, 0.53, 0.51	0.53	100	106, 112	109
		1	0.47, 0.48	0.48	91	99, 100	100
		3	0.43, 0.42	0.43	81	93, 87	90
		6	0.44, 0.48	0.46	87	99, 98	99
		12	0.51, 0.49	0.5	94	99, 102	101
		18	0.39, 0.42	0.41	77	91, 97	94
		24	0.4, 0.4	0.4	75	95, 84	90
Lettuce	0.5	0	0.51, 0.5, 0.53	0.51	100	98, 106	102

Matrix	Fortification level (mg/kg)	Storage period (months)	Residue level in stored samples ^a			Procedural recovery	
			Individual values (mg/kg)	Mean (mg/kg)	Mean (%)	Individual values (%)	Mean (%)
		1	0.42, 0.42	0.42	82	101, 103	102
		3	0.27, 0.29	0.28	55	91, 94	93
		6	0.28, 0.28	0.28	55	105, 101	103
		12	0.27, 0.23	0.25	49	95, 97	96
		18	0.18, 0.2	0.19	37	95, 96	96
		24	0.12, 0.14	0.13	25	91, 91	91

^a Not corrected for procedural recovery

Joseph (2000, PYMET_062)

Samples of broccoli, cabbage, mustard greens, head lettuce, leaf lettuce, spinach and celery taken from field trials with incurred residues of pymetrozine were reanalysed for residues of pymetrozine after freezer storage at approximately -20 °C for an additional period of 8 to 13 months depending on the crop.

Table 77 Incurred residues of pymetrozine in crop commodities after storage at -20 °C

Matrix	Sample ID	Storage interval (months)	Corrected residues before storage (mg/kg) ^a	Uncorrected Residue after storage (mg/kg)	Procedural recovery (%)	Corrected Residue after storage (mg/kg)	Corrected Recovery (% remaining)
Broccoli	259541	13	0.08	0.05	90	0.055	68
	259542	13	0.18	0.16	90	0.176	98
	Mean						73
Cabbage	259544	10	0.27	0.21	75	0.28	104
	259545	10	0.18	0.25	75	0.33	183
	Mean						144
Mustard greens	259547	13	3.4	2.2	79	2.78	82
	259548	13	4.2	2.0	79	2.53	60
	Mean						71
Head lettuce	259550	8	0.37 ^b	0.19 ^b	94	0.202	55
	259551	8	0.39 ^b	0.19 ^b	94	0.202	52
	Mean						54
Leaf lettuce	259553	10	1.1	0.6	73	0.822	75
	259554	10	0.9	0.81	73	1.11	123
	Mean						99
Spinach	259556	9	4.8	3.6	75	4.8	100
	259557	9	5.3	2.3	75	3.07	58
	Mean						79
Celery	259559	11	0.63	0.38	90	0.422	67
	259560	11	0.49	0.34	90	0.378	77
	Mean						72

^a Uncorrected values not reported in this study

^b Mean of duplicate analysis

Animal matrices

Boyette (1998, PYMET_063)

Samples of beef muscle, beef liver and milk were separately fortified with pymetrozine or CGA313124 at a nominal rate of 0.2 mg/kg. Two sub-samples were immediately taken and analysed for residues of the fortified materials. The remaining samples were stored deep frozen at approximately -20 °C for up to 18 months with duplicate sub-samples being taken at intervals and analysed for residues.

Table 78 Residues of pymetrozine and CGA313124 in animal commodities after storage at -20 °C

Matrix	Forti fication level (mg/kg)	Storage period (months)	Pymetrozine			CGA313124		
			Residue level in stored samples ^a		Procedural recovery	Residue level in stored samples ^a		Procedural recovery
			Individual values in mg/kg (mean)	% Mean	Individual values in % nominal (mean)	Individual values in mg/kg (mean)	% Mean	Individual values in % nominal (mean)
Muscle, bovine	0.2	0	0.17, 0.16 (0.17)	100	80, 87 (84)	0.18, 0.19 (0.19)	100	90, 87 (89)
		3	0.17, 0.13 (0.15)	88	102, 97 (100)	0.1, 0.15 (0.13)	68	82, 99 (91)
		6	0.12, 0.11 (0.12)	71	77, 75 (76)	0.16, 0.11 (0.13)	68	92, 82 (87)
		12	0.05, 0.06 (0.06)	35	84, 83 (84)	0.06, 0.09 (0.08)	42	87, 81 (84)
		18	0.05, 0.05 (0.05)	29	87, 89 (88)	0.07, 0.08 (0.08)	42	80
Liver, bovine	0.2	0	0.17, 0.13 (0.15)	100	90, 81 (86)	0.19, 0.19 (0.19)	100	77, 93 (85)
		3	0.17, 0.18 (0.18)	120	80, 102 (91)	0.19, 0.2 (0.2)	105	94, 89 (92)
		6	0.15, 0.13 (0.14)	93	85, 80 (83)	0.16, 0.19 (0.18)	95	105
		12	0.11, 0.12 (0.12)	80	86, 89 (88)	0.1, 0.07 (0.09)	47	95, 89 (92)
		18	0.05, 0.07 (0.06)	40	71, 80 (86)	0.01, < 0.01 (0.01)	5	55
Milk	0.2	0	0.16, 0.16 (0.16)	100	83, 79 (81)	0.16, 0.16 (0.16)	100	85, 78 (82)
		3	0.16, 0.16 (0.16)	100	84, 72 (78)	0.16, 0.14 (0.15)	94	70, 67 (69)
		6	0.12, 0.1 (0.11)	69	77, 64 (71)	0.17, 0.15 (0.16)	100	82, 75 (79)
		12	0.1, 0.14 (0.12)	75	76, 76 (76)	0.17, 0.17 (0.17)	106	76, 77 (77)
		18	0.13, 0.14 (0.14)	88	77, 80 (79)	0.15, 0.12 (0.14)	88	116, 62 (89)

^a Not corrected for procedural recovery

USE PATTERN

Pymetrozine is a systemic neuroactive insecticide; however, its mode of action in the nervous system is unknown. It is used in a large variety of fruit and vegetable crops, oilseeds and rice against aphids and other suckling insects close to harvest.

Table 79 List of uses of pymetrozine

Crop	Country	Application detail					
		Form	Type	kg ai/ha	Growth stage at last treatment	No.	Pre harvest interval (PHI) in days
Citrus fruit							
Citrus	Portugal	WG 50	foliar, F	0.01 kg ai/hL		1	21
Clementine	Italy	WG 50	foliar, F	0.25		2	21
Lemon	Italy	WG 50	foliar, F	0.25		2	21
Lemon	Spain	WG 50	foliar, F	0.25		2	21
Mandarin	Italy	WG 50	foliar, F	0.25		2	21
Mandarin	Spain	WG 50	foliar, F	0.25		2	21
Orange	Greece	WG 50	foliar, F	0.3		1	21
Orange	Italy	WG 50	foliar, F	0.25		2	21
Orange	Spain	WG 50	foliar, F	0.25		2	21
Pome fruit							
Apple	Italy	WG 50	foliar, F	0.25		1	14
Apple	Spain	WG 50	foliar, F	0.25		2	14
Pear	Italy	WG 50	foliar, F	0.25		1	14
Pear	Spain	WG 50	foliar, F	0.25		2	14
Stone fruit							
Apricot	Belgium	WG 50	foliar, F/P	0.1		2	21
Apricot	Germany	WG 50	foliar, F	0.02 kg ai/hL		2	21
Nectarine	Spain	WG 50	foliar, F	0.25		2	14
Peach	Belgium	WG 50	foliar, F	0.1		2	21

Crop	Country	Application detail					
		Form	Type	kg ai/ha	Growth stage at last treatment	No.	Pre harvest interval (PHI) in days
Peach	France	WG 50	foliar, F	0.02 kg ai/hL		2	14
Peach	Germany	WG 50	foliar, F	0.02 kg ai/hL		2	21
Peach	Greece	WG 50	foliar, F	0.15		2	14
Peach	Italy	WG 50	foliar, F	0.25		2	14
Peach	Portugal	WG 50	foliar, F	0.01 kg ai/hL		3	14
Peach	Spain	WG 50	foliar, F	0.25		2	14
Berries and other small fruits							
Blackberry	Belgium	WG 50	foliar, F/P	0.1		3	3
Blueberry	Belgium	WG 50	foliar, F/P	0.1		2	14
Currants	Belgium	WG 50	foliar, F/P	0.1		2	14
Currants	Germany	WG 50	foliar, F	0.2		2	14
Foxberry	Belgium	WG 50	foliar, F/P	0.1		2	14
Gooseberry	Belgium	WG 50	foliar, F/P	0.1		2	14
Raspberry	Belgium	WG 50	foliar, F/P	0.1		3	3
Strawberry	Belgium	WG 50	foliar, F	0.2		3	3
Strawberry	Belgium	WG 50	foliar, P	0.2		3	3
Strawberries	Czech Republic	WG 50	foliar, F	0.2	before flowering, after harvest	3	Plant growth
Strawberry	Germany	WG 50	foliar, F/P	0.2	before flowering, after harvest	3	plant growth
Strawberry	Portugal	WG 50	foliar, P	0.2		3	3
Brassica vegetables							
Brassica vegetables (broccoli, Brussels sprouts, cabbage, cauliflower, Chinese broccoli, Chinese cabbage, Chinese mustard cabbage, Collards, Kale, Kohlrabi, mizuna, mustard greens, mustards spinach, rape greens, turnip greens)	USA	WG 50	foliar, F	0.1		2	7
Broccoli	Belgium	WG 50	foliar, F	0.2		3	14
Broccoli	Netherlands	WG 50	foliar, F	0.2		2	14
Broccoli	United Kingdom	WG 50	foliar, F	0.2		3	14
Brussels sprouts	Belgium	WG 50	foliar, F	0.2		3	14
Brussels sprouts	Germany	WG 50	foliar, F	0.2		2	21
Brussels sprouts	United Kingdom	WG 50	foliar, F	0.2		3	14
Cabbages	United Kingdom	WG 50	foliar, F	0.2		3	14
Cauliflower	Belgium	WG 50	foliar, F	0.2		3	14
Cauliflower	Czech Republic	WG 50	foliar, F	0.2		3	14
Cauliflower	Netherlands	WG 50	foliar, F	0.2		2	14
Cauliflower	United Kingdom	WG 50	foliar, F	0.2		3	14
Chinese cabbage	Belgium	WG 50	foliar, F	0.2		2	7
Chinese cabbage	Netherlands	WG 50	foliar, F	0.2		2	14
Flowering brassicas	Germany	WG 50	foliar, F	0.2		3	14
Head cabbage	Belgium	WG 50	foliar, F	0.2		3	14
Head cabbage	Czech Republic	WG 50	foliar, F	0.2		3	7
Head cabbage	Germany	WG 50	foliar, F	0.2		3	7
Head cabbage	Netherlands	WG 50	foliar, F	0.2		2	14

Crop	Country	Application detail					
		Form	Type	kg ai/ha	Growth stage at last treatment	No.	Pre harvest interval (PHI) in days
Head cabbage	Portugal	WG 50	foliar, F	0.2		3	14
Kale	France	WG 50	foliar, F/P	0.2		2	14
Kale, curly	Belgium	WG 50	foliar, F	0.2		2	7
Kohlrabi	Germany	WG 50	foliar, F/P	0.2		3	14
Leafy brassicas	Germany	WG 50	foliar, F	0.2		2	7
Fruiting vegetables, cucurbits							
Courgette	Netherlands	WG 50	foliar, F/P	0.03 kg ai/hL		2	1
Courgette	Spain	WG 50	foliar, F/P	0.25		2	3
Courgette	Spain	WG 50	foliar, F/P	0.5		1	3
Cucumber	Belgium	WG 50	foliar, P	0.15		2	1
Cucumber	Czech Republic	WG 50	foliar, F	0.36		3	3
Cucumber	France	WG 50	foliar, F/P	0.2		3	3
Cucumber	Germany	WG 50	foliar, P	0.03 kg ai/hL		3	7
Cucumber	Greece	WG 50	foliar, F/P	0.45		2	3
Cucumber	Italy	WG 50	foliar, F/P	0.25		2	3
Cucumber	Netherlands	WG 50	foliar, F/P	0.03 kg ai/hL		2	1
Cucumber	Portugal	WG 50	foliar, F/P	0.3		2	3
Cucumber	Spain	WG 50	foliar, F/P	0.25		2	3
Cucumber	Spain	WG 50	foliar, F/P	0.5		1	3
Cucurbits (Chayote, Chinese waxgourd, citron melon, cucumber, gourd, gherkin, muskmelon, pumpkins, summer squash, winter squash, watermelon)	USA	WG 50	foliar, F	0.1		2	0
Gherkin	Belgium	WG 50	foliar, P	0.28		2	1
Gherkin	France	WG 50	foliar, F/P	0.2		3	3
Gherkin	Netherlands	WG 50	foliar, F/P	0.03 kg ai/hL		2	1
Melon	Belgium	WG 50	foliar, P	0.05		2	1
Melon	France	WG 50	foliar, F/P	0.2		3	3
Melon	Greece	WG 50	foliar, F/P	0.45		2	3
Melon	Italy	WG 50	foliar, F/P	0.25		2	3
Melon	Netherlands	WG 50	foliar, F/P	0.03 kg ai/hL		2	1
Melon	Portugal	WG 50	foliar, F/P	0.3		3	3
Melon	Spain	WG 50	foliar, F/P	0.25		2	3
Melon	Spain	WG 50	foliar, F/P	0.5		1	3
Pumpkins	France	WG 50	foliar, F/P	0.2		3	3
Pumpkins	Netherlands	WG 50	foliar, F/P	0.03 kg ai/hL		2	1
Pumpkins	Portugal	WG 50	foliar, F/P	0.3		3	3
Summer squash	Netherlands	WG 50	foliar, F/P	0.03 kg ai/hL		2	1
Watermelons	France	WG 50	foliar, F/P	0.2		3	3
Watermelons	Italy	WG 50	foliar, F/P	0.25		2	3
Watermelons	Portugal	WG 50	foliar, F/P	0.3		3	3
Watermelons	Spain	WG 50	foliar, F/P	0.25		2	3
Watermelons	Spain	WG 50	foliar, F/P	0.5		1	3
Zucchini	Belgium	WG 50	foliar, P	0.1		2	1
Zucchini	Belgium	WG 50	foliar, F	0.1		3	1
Zucchini	France	WG 50	foliar, F/P	0.2		3	3

Crop	Country	Application detail					
		Form	Type	kg ai/ha	Growth stage at last treatment	No.	Pre harvest interval (PHI) in days
Zucchini	Italy	WG 50	foliar, F/P	0.25		2	3
Fruiting vegetables, other than cucurbits							
Aubergine	Belgium	WG 50	foliar, P	0.3		2	1
Aubergine	Czech Republic	WG 50	Foliar, P	0.36		3	3
Bell pepper	Belgium	WG 50	foliar, P	0.3		2	1
Bell pepper	Italy	WG 50	foliar, F/P	0.25		2	3
Bell pepper	Netherlands	WG 50	foliar, P	0.03 kg ai/hL		2	1
Chilli pepper	Netherlands	WG 50	foliar, P	0.03 kg ai/hL		2	1
Eggplant	France	WG 50	foliar, F/P	0.2		3	3
Eggplant	Germany	WG 50	foliar, P	0.03 kg ai/hL		3	3
Eggplant	Greece	WG 50	foliar, F/P	0.45		2	3
Eggplant	Italy	WG 50	foliar, F/P	0.25		2	3
Eggplant	Netherlands	WG 50	foliar, P	0.03 kg ai/hL		2	1
Eggplant	Portugal	WG 50	foliar, F/P	0.25		2	3
Eggplant	Spain	WG 50	foliar, F/P	0.25		2	3
Eggplant	Spain	WG 50	foliar, F/P	0.25		1	3
Fruiting vegetables (eggplant, pepino, peppers, tomatillo, tomato)	USA	WG 50	foliar, F	0.1		2	0
Okra	Netherlands	WG 50	foliar, P	0.03 kg ai/hL		2	1
Pepper	Czech Republic	WG 50	foliar, F	0.36		3	3
Pepper	France	WG 50	foliar, F/P	0.2		3	3
Pepper	Greece	WG 50	foliar, F/P	0.15		2	3
Pepper	Portugal	WG 50	foliar, F/P	0.3		3	3
Pepper	Spain	WG 50	foliar, F/P	0.25		2	3
Pepper	Spain	WG 50	foliar, F/P	0.25		1	3
Sweet corn	Czech Republic	WG 50	foliar, F	0.2		1	14
Sweet corn	Germany	WG 50	foliar, F	0.2		1	14
Sweet pepper	Czech Republic	WG 50	foliar, P	0.36		3	3
Sweet pepper	Germany	WG 50	foliar, P	0.03 kg ai/hL		3	14
Tomato	Belgium	WG 50	foliar, P	0.15		2	1
Tomato	Czech Republic	WG 50	foliar, F	0.36		3	3
Tomato	Germany	WG 50	foliar, P	0.03 kg ai/hL		3	3
Tomato	Greece	WG 50	foliar, F/P	0.45		2	3
Tomato	Italy	WG 50	foliar, F/P	0.25		2	3
Tomato	Netherlands	WG 50	foliar, P	0.45		3	1
Tomato	Portugal	WG 50	foliar, F/P	0.3		3	3
Tomato	Spain	WG 50	foliar, F/P	0.25		2	3
Tomato	Spain	WG 50	foliar, F/P	0.25		1	3
Leafy vegetables							
Endive	Belgium	WG 50	foliar, P	0.1		3	14
Endive	Belgium	WG 50	foliar, F	0.1		3	7
Endive	Czech Republic	WG 50	foliar, F	0.2		3	7
Endive	France	WG 50	foliar, F	0.2		2	7
Endive	France	WG 50	foliar, P	0.2		2	14
Endive	Germany	WG 50	foliar, F/P	0.2		3	7
Endive	Netherlands	WG 50	foliar, F/P	0.2		2	14
Fresh herbs	Germany	WG 50	foliar, F	0.2		3	14
Fresh herbs	Netherlands	WG 50	foliar, F/P	0.2		2	14
Head lettuce	Germany	WG 50	foliar, F	0.2		3	7

Crop	Country	Application detail					
		Form	Type	kg ai/ha	Growth stage at last treatment	No.	Pre harvest interval (PHI) in days
Lamb's lettuce	Portugal	WG 50	foliar, F	0.25		2	7
Lamb's lettuce	Portugal	WG 50	foliar, P	0.25		2	14
Leaf lettuce	Germany	WG 50	foliar, F/P	0.2		3	7
Leafy vegetables (Amaranth, rucola, cardoon, celery, fresh herbs, endive, head lettuce, leaf lettuce, radicchio, rhubarb, spinach, Swiss chard)	USA	WG 50	foliar, F	0.1		2	7
Lettuce	Belgium	WG 50	foliar, P	0.1		3	7
Lettuce	Belgium	WG 50	foliar, F	0.1		3	14
Lettuce	Czech Republic	WG 50	foliar, F	0.2		3	7
Lettuce	France	WG 50	foliar, F	0.2		2	7
Lettuce	France	WG 50	foliar, P	0.2		2	14
Lettuce	Italy	WG 50	foliar, F/P	0.25		2	14
Lettuce	Netherlands	WG 50	foliar, F/P	0.2		2	14
Lettuce	Portugal	WG 50	foliar, F	0.25		2	7
Lettuce	Portugal	WG 50	foliar, P	0.25		2	14
Lettuce	Spain	WG 50	foliar, F	0.25		2	7
Lettuce	Spain	WG 50	foliar, P	0.25		2	14
Mustard green	Portugal	WG 50	foliar, F	0.25		2	7
Mustard green	Portugal	WG 50	foliar, P	0.25		2	14
Rocket-salad	Belgium	WG 50	foliar, P	0.2		3	14
Rocket-salad	Belgium	WG 50	foliar, F	0.2		3	7
Rocket-salad	Portugal	WG 50	foliar, F	0.25		2	7
Rocket-salad	Portugal	WG 50	foliar, P	0.25		2	14
Scarole	France	WG 50	foliar, F	0.2		2	7
Scarole	France	WG 50	foliar, P	0.2		2	14
Spinach	Belgium	WG 50	foliar, F	0.2		2	14
Swiss chard	Belgium	WG 50	foliar, F	0.2		2	14
Watercress	Portugal	WG 50	foliar, F	0.25		2	7
Watercress	Portugal	WG 50	foliar, P	0.25		2	14
Legume vegetables							
Legume beans	Germany	WG 50	foliar, F/P	0.2		2	7
Peas with pods	Germany	WG 50	foliar, F	0.12	59	2	14
Root and tuber vegetables							
Celeriac	Germany	WG 50	foliar, F	0.2		3	14
Potato	Belgium	WG 50	foliar, F	0.15		3	plant growth
Potato	Czech Republic	WG 50	Foliar, F	0.1		2	7
Potato	France	WG 50	foliar, F	0.15		3	14
Potato	Germany	WG 50	foliar, F	0.1		2	7
Potato	Italy	WG 50	foliar, F/P	0.25		2	7
Potato	Netherlands	WG 50	foliar, F	0.15		2	7
Potato	Spain	WG 50	foliar, F/P	0.25		2	3
Potato	Spain	WG 50	foliar, F/P	0.5		1	3
Potato	United Kingdom	WG 50	foliar, F	0.15		2	7
Potato and other tuberous roots ^a	USA	WG 50	foliar, F	0.19		2	14
Root vegetables	Czech Republic	WG 50	foliar, F	0.2		3	14
Turnips	Germany	WG 50	foliar, F	0.2		3	14
Stalk and stem vegetables							
Artichoke	France	WG 50	foliar, F/P	0.2		2	14
Asparagus	USA	WG 50	foliar, F	0.1		6	170
Celery	Czech Republic	WG 50	Foliar, F	0.2		3	14
Celery	Germany	WG 50	foliar, P	0.2		1	35
Fennel	Germany	WG 50	foliar, F	0.2		3	14

Crop	Country	Application detail					
		Form	Type	kg ai/ha	Growth stage at last treatment	No.	Pre harvest interval (PHI) in days
Cereal grains							
Rice	China		foliar, F	0.15		2	14
Treenuts							
Chestnut	France	WG 50	foliar, F	0.1		2	14
Hazelnut	France	WG 50	foliar, F	0.1		2	14
Pecan	USA	WG 50	foliar, F	0.14		2	14
Walnut	France	WG 50	foliar, F	0.1		2	14
Oilseeds							
Cotton	Greece	WG 50	foliar, F	0.2		2	35
Cotton	USA	WG 50	foliar, F	0.1		2	21
Mustard	France	WG 50	foliar, F	0.075	59	1	plant growth
Rape	Belgium	WG 50	foliar, F	0.075	59	1	plant growth
Rape	Czech Republic	WG 50	foliar, F	0.075	59	1	Plant growth
Rape	France	WG 50	foliar, F	0.075	59	1	plant growth
Rape	Germany	WG 50	foliar, F	0.075	59	1	plant growth
Rape	United Kingdom	WG 50	foliar, F	0.15 + 0.075	26 + 59	2	growth stage
Herbs							
Hops	Belgium	WG 50	foliar, F/P	0.1		2	n.s.
Hops	Czech Republic	WG 50	foliar, F	0.4		2	14
Hops	France	WG 50	foliar, F	0.03 kg ai/hL		3	14
Hops	Germany	WG 50	foliar, F	0.4		2	21
Hops	USA	WG 50	foliar, F	0.21		3	14

^a The group includes: (Arracacha, Arrowroot, Bean yam, Cassava, Chayote, Chinese artichoke, Chufa, Dasheen, Edible canna, Ginger, Jerusalem artichoke, Leren, Sweet potato, Tanier, Yam, Turmeric)

Residues resulting from supervised trials on crops

Residue levels were reported as measured. Application rates were always reported as pymetrozine equivalents. When residues were not detected they are shown as below the LOQ, e.g., < 0.01 mg/kg. Application rates and spray concentrations have generally been rounded to two significant figures. HR and STMR values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. These results are underlined.

Laboratory reports included method validation including batch recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Field reports provided data on the sprayers used and their calibration, plot size, residue sample size and sampling date. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Residue data are recorded unadjusted for % recovery.

Pymetrozine—supervised residue trials

Commodity	Indoor/Outdoor	Treatment	Countries	Table
Lemon	outdoor	foliar	Spain	80
Mandarins	outdoor	foliar	Spain	81
Oranges	outdoor	foliar	Spain	82
Apples	outdoor	foliar	Italy	83
Apricots	outdoor	foliar	France, Spain	84
Peaches	outdoor	foliar	France, Italy, Spain	85
Strawberries	outdoor	foliar	United Kingdom	86
Strawberries	protected	foliar	United Kingdom	87

Commodity	Indoor/Outdoor	Treatment	Countries	Table
Broccoli	outdoor	foliar	Switzerland, United Kingdom	88
Cauliflower	outdoor	foliar	France, Switzerland	89
Head cabbage	outdoor	foliar	France, Germany, Portugal, Spain	90
Cucumber	protected	foliar	France, Netherlands, Spain, Switzerland	91
Melons	protected	foliar	France, Italy, Spain	92
Sweet peppers	protected	foliar	France, Italy, Netherlands, Spain, Switzerland	93
Tomatoes	protected	foliar	France, Germany, Netherlands, Switzerland	94
Head lettuce	outdoor	foliar	USA	95
Leaf lettuce	outdoor	foliar	USA	96
Spinach	outdoor	foliar	USA	97
Potatoes	outdoor	foliar	France, Germany, United Kingdom	98
Asparagus	outdoor	foliar	USA	99
Celery	outdoor	foliar	USA	100
Globe artichoke	outdoor	foliar	France	101
Rice	outdoor	foliar	China	102
Pecan	outdoor	foliar	USA	103
Walnuts	outdoor	foliar	USA	104
Oilseed rape	outdoor	foliar	France, Germany, Italy, United Kingdom	105
Cotton seeds	outdoor	foliar	Greece, Spain	106
Rice straw	outdoor	foliar	China	107
Cotton hulls	outdoor	foliar	Greece, Spain	108

Citrus fruits

Table 80 Residues of pymetrozine following foliar application to lemons

Location, Year (variety)	Application					Residues, mg/kg			Storage interval	Trial No.,
	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Spain, Crevillente 1994 (Berna)	WP	1	0.14	0.01	89	whole fruit	0 7 14 21 28 ^a	0.19 0.05 0.03 <u>0.02</u> < 0.02	10	CGA215944/0286 Tribolet (1995, PYMET_068)
						peel	28	< 0.02		
						flesh	28	< <u>0.02</u>		
						juice	28	< 0.005		
Spain, Librilla 1995 (Eureka)	WP	1	0.3	0.01	83	whole fruit	0 7 14 21 ^a	0.19 0.05 0.03 <u>0.03</u>	9	CGA215944/0422 Tribolet (1996, PYMET_079)
						peel	21	0.04		

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
						flesh	21	< 0.02		
						juice	21	< 0.005		
Spain, Librilia	WP	1	0.3	0.01	83	whole fruit	0 7 14 21 ^a	0.26 0.11 0.05 0.07	9	CGA215944/0423
1995 (Berna)						peel	21	0.12		Tribolet (1996, PYMET_080)
						flesh	21	< 0.02		Replicate plot
						juice	21	0.005		
Spain, Santomera	WP	1	0.3	0.01	82	whole fruit	0 7 14 21 ^a	0.36 0.11 < 0.02 0.02	9	CGA215944/0424
1995 (Berna)						peel	21	0.03		Tribolet (1996, PYMET_081)
						flesh	21	< 0.02		
						juice	21	< 0.005		

^a Calculated

DAT = Days after last treatment

BBCH 82/83 = Fruit ripe for picking; fruit has not yet developed variety-specific colour

BBCH 89 = Fruit ripe for consumption; fruit has typical taste and firmness; beginning of senescence and fruit abscission

Table 81 Residues of pymetrozine following foliar application to mandarins

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Spain, Sagunto	WP	1	0.41	0.01	77	whole fruit	0 3 7 14 21 ^a	0.29 0.17 0.04 0.02 < 0.02	8	CGA215944/0273
1994 (Clemenules)						peel	21	0.02		Tribolet (1995, PYMET_069)
						flesh	21	< 0.02		
Spain, Rinconada	WP	1	0.29	0.01	83	whole fruit	0 3 7 14 21 ^a	0.28 0.07 0.06 0.02 0.02	8	CGA215944/0274
1994 (Clemenules)						peel	21	0.03		Tribolet (1995, PYMET_070)
						flesh	21	< 0.02		
Spain, Benacazon	WP	1	0.3	0.01	89	whole fruit	0 3 7 14 21 ^a	0.22 0.14 0.06 0.05 0.03	3	CGA215944/0420
1995 (Clemenules)						peel	21	0.04		Tribolet (1996, PYMET_072)
						flesh	21	< 0.02		

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Spain, Sagunto 1995 (Clemenules)	WP	1	0.3	0.01	81	whole fruit	0 3 7 14 21 ^a	0.16 0.18 0.13 0.07 <u>0.03</u>	4	CGA215944/0421 Tribolet (1996, PYMET_078)
						peel flesh	21 21	0.04 < <u>0.02</u>		

^a Calculated

DAT = Days after last treatment

BBCH 77 = Fruits about 70% of final size

BBCH 81 = Beginning of fruit colouring (colour-break)

BBCH 82/83 = Fruit ripe for picking; fruit has not yet developed variety-specific colour

BBCH 89 = Fruit ripe for consumption; fruit has typical taste and firmness; beginning of senescence and fruit abscission

Table 82 Residues of pymetrozine following foliar application to oranges

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Spain, Amposta 1994 (Navelina)	WP	1	0.48	0.01	89	whole fruit	0 3 6 13 20 ^a	0.03 0.04 0.03 0.06 <u>0.02</u>	9	CGA215944/0283 Tribolet (1995, PYMET_064)
						peel flesh	20 20	0.03 < <u>0.02</u>		
Spain, Rinconada 1994 (Newhall)	WP	1	0.27	0.01	83	whole fruit	0 3 7 14 21 ^a	0.09 0.02 < 0.02 0.02 <u>0.02</u>	9	CGA215944/0284 Tribolet (1995, PYMET_065)
						peel flesh	21 21	0.03 < <u>0.02</u>		
Spain, Masalfasar 1994 (Thomson)	WP	1	0.17	0.01	81	whole fruit	0 3 7 14 21 ^a	0.12 0.11 0.07 0.05 <u>0.05</u>	9	CGA215944/0285 Tribolet (1995, PYMET_066)
						peel flesh	21 21	0.08 < <u>0.02</u>		
	WP	1	0.16	0.01	81	whole fruit	0 3 7 14 21 ^a	0.1 0.07 0.08 0.04 0.05	9	CGA215944/0286 Tribolet (1995, PYMET_067) Replicate plot
						peel flesh	21 21	0.08 < <u>0.02</u>		
Spain, Amposta	WP	1	0.46	0.01	85	whole fruit	0 3 7	0.35 0.14 0.19	6	CGA215944/0480 Tribolet (1996,

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
1995 (Navelina)						peel flesh juice	14 21 ^a 21 21 21	0.05 <u>0.04</u> 0.08 < <u>0.02</u> < 0.005		PYMET_071)
Spain, Benacazon 1996 (Navelino)	WP	1	0.4	0.01	87	whole fruit peel flesh juice	0 3 7 14 21 ^a 21 21 21	0.23 0.07 0.05 0.04 <u>0.02</u> 0.02 < <u>0.02</u> 0.005	6	CGA215944/0481 Tribolet (1996, PYMET_073)
Spain, Masalfasar 1996 (Navel Thomson)	WP	1	0.4	0.01	81	whole fruit peel flesh juice	0 3 7 14 22 ^a 22 22 22	0.29 0.3 0.06 0.04 <u>0.02</u> 0.02 < <u>0.02</u> < 0.005	6	CGA215944/0482 Tribolet (1996, PYMET_074)
Spain, Torrente 1996 (Navelino)	WP	1	0.4	0.01	83	whole fruit peel flesh juice	0 3 7 14 22 ^a 22 22 22	0.12 0.08 0.04 0.02 < <u>0.02</u> < 0.02 < <u>0.02</u> < 0.005	6	CGA215944/0483 Tribolet (1996, PYMET_075)
Spain, Picana 1995 (New Hall)	WP	1	0.4	0.01	85	whole fruit peel flesh juice	0 3 7 15 21 ^a 21 21 21	0.27 0.2 0.18 0.14 <u>0.06</u> 0.15 < <u>0.02</u> < 0.005	6	CGA215944/0484 Tribolet (1996, PYMET_076)
Spain, Sagunto 1995 (New Hall)	WP	1	0.4	0.01	81	whole fruit peel flesh juice	0 3 7 15 21 ^a 21 21 21	0.25 0.18 0.09 0.02 <u>0.03</u> 0.06 < <u>0.02</u> < 0.005	6	CGA215944/0485 Tribolet (1996, PYMET_077)

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Spain, Palomares 2006 (Navelina)	WP	1	0.32	0.01	79	whole fruit	0 ^a 7 ^a 13 ^a 22 ^a 29 ^a	0.11 0.009 0.007 <u>0.006</u> 0.005	1	ES-IR-06-0162 Mason (2007, PYMET_082) Remark : CGA300407 in all samples < 0.005 mg/kg
						peel	0 7 13 22 29	0 0.29 0.018 0.012 0.009 0.006		
						flesh	0 7 13 22 29	0 0.008 < 0.005 < 0.005 < <u>0.005</u> < 0.005		
Spain, Los Palacios 2006 (Navelina)	WP	1	0.31	0.01	79	whole fruit	0 ^a 7 ^a 13 ^a 22 ^a 29 ^a	0.13 0.019 0.013 <u>0.007</u> 0.005	1	ES-IR-06-0163 Mason (2007, PYMET_082) Remark : CGA300407 in all samples < 0.005 mg/kg
						peel	0 7 13 22 29	0 0.34 0.048 0.028 0.01 0.006		
						flesh	0 7 13 22 29	0 0.013 < 0.005 < 0.005 < <u>0.005</u> < 0.005		
Spain, El Coronil 2006 (Navelina)	WP	1	0.31	0.01	81	whole fruit	0 ^a 7 ^a 13 ^a 22 ^a 29 ^a	0.52 0.037 0.018 0.009 <u>0.012</u>	1	ES-IR-06-0164 Mason (2007, PYMET_082) Remark : CGA300407 in all samples < 0.005 mg/kg
						peel	0 7 13 22 29	0 1.6 0.092 0.038 0.016 0.025		
						flesh	0 7 13 22 29	0 0.029 < 0.005 < 0.005 < <u>0.005</u> < 0.005		
Spain, Olivares 2006 (Navelina)	WP	1	0.29	0.01	81	whole fruit	0 ^a 8 ^a 14 ^a 21 ^a 28 ^a /a	0.17 0.02 0.013 <u>0.01</u> 0.01	1	ES-IR-06-0165 Mason (2007, PYMET_082) Remark : CGA300407 in all samples
						peel	0 8	0.57 0.047		

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
						flesh	14 21 28 0 8 14 21 28	0.029 0.021 0.021 < 0.005 < 0.005 < 0.005 < 0.005 < 0.005		< 0.005 mg/kg

^a Calculated

DAT = Days after last treatment

BBCH 77 = Fruits about 70% of final size

BBCH 79 = Fruits about 90% of final size

BBCH 81 = Beginning of fruit colouring (colour-break)

BBCH 82/83 = Fruit ripe for picking; fruit has not yet developed variety-specific colour

BBCH 85/87 = Advanced ripening; increase in intensity of variety-specific colour

BBCH 89 = Fruit ripe for consumption; fruit has typical taste and firmness; beginning of senescence and fruit abscission

Pome fruits

Table 83 Residues of pymetrozine following foliar application to apples

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Italy, Belfiore 2002 (Golden)	WG	1	0.25	0.02	83	fruit	0 3 7 14 21	0.03 0.02 < 0.02 < 0.02 < 0.02	6	CGA215944/4883 Sole (2003, PYMET_083)
Italy, Pernumia 2002 (Golden)	WG	1	0.25	0.019	79	fruit	0 3 7 14 21	0.05 0.02 < 0.02 < 0.02 < 0.02	6	CGA215944/4884 Sole (2003, PYMET_084)
Italy, Borgo d'Ale 2002 (Jonagold)	WG	1	0.26	0.025	85	fruit	0 14	0.02 < 0.02	6	CGA215944/4882 Sole (2003, PYMET_085)
Italy, Trebiano di Ponte Nizza 2003 (Golden)	WG	1	0.25	0.017	83	fruit	0 3 7 14 21	0.12 0.12 < 0.02 < 0.02 < 0.02	8	CGA215944/4925 Sole (2004, PYMET_086)
Italy, San Bonifacio 2003 (Red Chief)	WG	1	0.25	0.025	83	fruit	0 3 7 14 21	0.14 0.06 < 0.02 < 0.02 < 0.02	8	CGA215944/4926 Sole (2004, PYMET_087)
Italy, Belfiore 2003 (Golden Delicious)	WG	1	0.25	0.019	83	fruit	0 14	0.13 < 0.02	8	CGA215944/4924 Sole (2004, PYMET_088)

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Italy, Badia Polesine 2003 (Red Chief)	WG	1	0.26	0.019	85	fruit	0 15	0.06 < 0.02	8	CGA215944/4922 Sole (2004, PYMET_089)
Italy, Gaibana 2003 (Red Chief)	WG	1	0.25	0.025	85	fruit	0 14	0.09 < 0.02	8	CGA215944/4923 Sole (2004, PYMET_090)

DAT = Days after last treatment

BBCH 79 = Fruits about 90% of final size

BBCH 83/85 = Advanced ripening: increase in intensity of cultivar-specific colour

Stone fruits

Table 84 Residues of pymetrozine following foliar application to apricots

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
France (South), Lafrancaise 2011 (Fantasme)	WG	3	0.25 0.26 0.26	n.s. n.s. n.s.	81 81 85	whole fruit ^a	0 3 7 15 21	0.09 0.06 0.02 0.02 <u>0.01</u>	1	S11-00627-01 Betson (2012, PYMET_092)
						flesh	0 3 7 15 21	0.1 0.07 0.02 0.02 0.01		
France (South), Pyrénées-Orientales 2011 (Royal Roussillon)	WG	3	0.23 0.26 0.25	n.s. n.s. n.s.	81 85 85	whole fruit ^a	-0 0 3 7 14 21	< 0.01 0.02 0.02 < 0.01 < 0.01 <u>< 0.01</u>	1	S11-00627-02 Betson (2012, PYMET_092)
						flesh	-0 0 3 7 14 21	< 0.01 0.02 0.02 < 0.01 < 0.01 < 0.01		
Spain, Fontanars del Alforins 2011 (Mixer de Castelló)	WG	3	0.24 0.24 0.23	n.s. n.s. n.s.	74 76 78	whole fruit ^a	0 3 7 14	0.18 0.09 0.04 <u>< 0.01</u>	1	S11-00627-03 Betson (2012, PYMET_092)
						flesh	0 3 7 14	0.19 0.09 0.04 < 0.01		
Spain, Quatretonda	WG	3	0.25 0.25	n.s. n.s.	75 76	whole fruit ^a	0 3	0.05 0.02	1	S11-00627-04

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
2011 (Tadeo)			0.24	n.s.	78	flesh	7 14 21 0 3 7 14 21	0.01 0.01 < 0.01 0.06 0.02 0.01 0.01 < 0.01		Betson (2012, PYMET_092)

^a Calculated

n.s. = Not stated

DAT = Days after last treatment

BBCH 73 Second fruit fall

BBCH 74 Fruits about 40% of final size

BBCH 75 Fruits about 50% of final size

BBCH 76 Fruits about 60% of final size

BBCH 77 Fruits about 70% of final size

BBCH 78 Fruits about 80% of final size

BBCH 85 Colouring advanced

Table 85 Residues of pymetrozine following foliar application to peaches

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
France (South), Corbère 2010 (Corindon)	WG	3	0.25 0.26 0.25	n.s. n.s. n.s.	75 76 77	whole fruit ^a flesh	0 3 7 14 21 0 3 7 14 21	0.02 < 0.01 < 0.01 < 0.01 < 0.01 0.03 < 0.01 < 0.01 < 0.01 < 0.01	1	S10-01588-01 Allen (2011, PYMET_91)
Italy, Altedo 2010 (Spring Bell)	WG	3	0.24 0.24 0.26	n.s. n.s. n.s.	74 75 77	whole fruit ^a flesh	0 3 7 14 21 0 3 7 14 21	< 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01 < 0.01	1	S10-01588-02 Allen (2011, PYMET_91)
Italy, Barbiano 2010 (Big Bang)	WG	3	0.24 0.24 0.23	n.s. n.s. n.s.	73 73 78	whole fruit ^a flesh	0 3 7 14 21 0 3 7	0.05 0.02 0.02 < 0.01 < 0.01 0.08 0.02 0.03	1	S10-01588-03 Allen (2011, PYMET_91)

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
							14 21	< 0.01 < 0.01		
Spain, Carlet 2010 (Spring Crest)	WG	3	0.26 0.25 0.24	n.s. n.s. n.s.	75 78 85	whole fruit ^a flesh	0 3 7 14 0 3 7 14	0.02 0.02 0.02 <u>0.04</u> 0.03 0.03 0.02 0.05	1	S10-01588-04 Allen (2011, PYMET_91)
Spain, Quatretonda 2010 (Baby Gold)	WG	3	0.28 0.27 0.25	n.s. n.s. n.s.	77 78 78	whole fruit ^a flesh	0 3 7 14 21 0 3 7 14 21	0.03 0.03 0.02 0.02 <u>0.04</u> 0.04 0.04 0.02 0.02 0.05	1	S10-01588-05 Allen (2011, PYMET_91)
Spain, El Coronil 2010 (Red Robin)	WG	3	0.23 0.25 0.25	n.s. n.s. n.s.	75 76 77	whole fruit ^a flesh	0 3 7 14 21 0 3 7 14 21	0.09 0.02 0.01 < <u>0.01</u> < 0.01 0.11 0.02 0.02 < 0.01 < 0.01	1	S10-01588-06 Allen (2011, PYMET_91)

^a Calculated

n.s. = Not stated

DAT = Days after last treatment

BBCH 73 = Second fruit fall

BBCH 74 = Fruits about 40% of final size

BBCH 75 = Fruits about 50% of final size

BBCH 76 = Fruits about 60% of final size

BBCH 77 = Fruits about 70% of final size

BBCH 78 = Fruits about 80% of final size

BBCH 85 = Colouring advanced

Strawberries

Table 86 Residues of pymetrozine following foliar application to strawberries in the field

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
United Kingdom, Gnosall 2004	WG	3	0.21 0.21 0.2	0.1 0.1 0.1	73 85 87	fruit	0 3 7 10	0.07 0.1 0.02 < 0.02	3	HD/6 Sole (2005, PYMET_094)

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
(Elsanta)										
United Kingdom, Ashford Carbonell 2004 (Symphony)	WG	3	0.21 0.2 0.21	0.1 0.1 0.1	85 85 85	fruit	3 7	0.05 < 0.02	2	HD/7 Sole (2005, PYMET_094)
United Kingdom, North Wheatley 2004 (Flamenco)	WG	3	0.2 0.2 0.2	0.1 0.1 0.1	85 85 85	fruit	3 7	< 0.02 0.02	1	HD/8 Sole (2005, PYMET_094)
United Kingdom, Handsacre 2004 (Everest)	WG	3	0.2 0.2 0.2	0.1 0.1 0.1	73 85 87	fruit	0 3 7 10	0.21 0.06 < 0.02 0.02	1	HD/9 Sole (2005, PYMET_094)

DAT = Days after last treatment

BBCH 73 = Seeds clearly visible on receptacle tissue

BBCH 85 = First fruits have cultivar specific colour

BBCH 87 = Main harvest: most fruits coloured

Table 87 Residues of pymetrozine following foliar application to strawberries in glasshouse

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
United Kingdom, Gnosall 2005 (Elsanta)	WG	3	0.2 0.2 0.21	0.1 0.1 0.1	73 81 85	fruit	0 3 7 10	0.09 0.08 0.05 0.02	2	AK/8866/HD/1 Bour (2005, PYMET_093)
United Kingdom, Southwell 2005 (Elsanta)	WG	4	0.2 0.21 0.21 0.22	0.1 0.1 0.1 0.1	73 81 81 85	fruit	0 3 7 10	0.1 0.12 0.12 0.1	1	AK/8866/HD/2 Bour (2005, PYMET_093)
United Kingdom, Chilcote 2005 (Elsanta)	WG	3	0.2 0.21 0.21	0.1 0.1 0.1	73 81 85	fruit	3 7	0.08 0.06	2	AK/8866/HD/3 Bour (2005, PYMET_093)
United Kingdom, Friday Bridge 2005 (Elsanta)	WG	3	0.2 0.2 0.21	0.1 0.1 0.1	73 81 81	fruit	3 7	0.11 0.05	2	AK/8866/HD/4 Bour (2005, PYMET_093)
United Kingdom, Gnosall 2004 (Elsanta)	WG	3	0.21 0.2 0.19	0.1 0.1 0.1	73 81 85	fruit	0 3 7 10	0.14 0.05 < 0.02 0.09	3	HD/1 Sole (2005, PYMET_094)

Location, Year (variety)	Application					Residues, mg/kg			Storage interval	Trial No., Reference
	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	
United Kingdom, Southwell 2004 (Elsanta)	WG	3	0.21 0.21 0.21	0.1 0.1 0.1	73 81 85	fruit	0 3 7 10	0.16 0.12 0.07 0.06	4	HD/2 Sole (2005, PYMET_094)
United Kingdom, Burntwood 2004 (Elsanta)	WG	3	0.2 0.2 0.21	0.01 0.01 0.01	73 73 81	fruit	3 7	0.09 < 0.02	3	HD/3 Sole (2005, PYMET_094)
United Kingdom, Chilcote 2004 (Elsanta)	WG	3	0.2 0.2 0.21	0.1 0.1 0.1	73 81 85	fruit	3 7	0.09 < 0.02	3	HD/4 Sole (2005, PYMET_094)

DAT = Days after last treatment

BBCH 73 = Seeds clearly visible on receptacle tissue

BBCH 81 = Beginning of ripening: most fruits white in colour

BBCH 85 = First fruits have cultivar specific colour

Broccoli

Table 88 Residues of pymetrozine following foliar application to broccoli

Location, Year (variety)	Application					Residues, mg/kg			Storage interval	Trial No., Reference
	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	
Switzerland, Fully 2003 (Fiesta)	WG	3	0.21 0.21 0.22	0.02 0.02 0.02	19 41 43	flower heads	0 14	0.38 < 0.02	7	03-5006 Sole (2004, PYMET_110)
United Kingdom, Hoffleet Stow 2004 (Iron)	WG	3	0.2 0.2 0.2	0.03 0.03 0.03	18 33 33	flower heads	0 3 7 14 21	2.2 0.15 0.01 < 0.01 < 0.01	17	AF/7929/SY1 Mason (2009, PYMET_111)
	WG +adj.	3	0.2 0.2 0.2	0.03 0.03 0.03	18 33 33	flower heads	0 3 7 14 21	1.8 0.1 < 0.01 < 0.01 < 0.01	17	
United Kingdom, Banks 2004 (Emerald pride)	WG	3	0.19 0.2 0.2	0.03 0.03 0.03	43 47 47	flower heads	0 3 7 14 21	1.1 0.02 0.02 < 0.01 < 0.01	17	AF/7929/SY2 Mason (2009, PYMET_111)
	WG +adj.	3	0.2 0.2	0.03 0.03	43 47	flower heads	0 3	1.5 0.02	17	

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
			0.2	0.03	47		7 14 21	0.02 0.01 0.01		

DAT = Days after last treatment

BBCH = 18 8 true leaves unfolded

BBCH = 19 9 or more true leaves unfolded

BBCH 33 = Main shoot has reached 30% of the expected height typical for the variety

BBCH 41 = Lateral buds begin to develop

BBCH 43 = First sprouts tightly closed

Cauliflower

Table 89 Residues of pymetrozine following foliar application to cauliflower

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
France (North), St. Genouph 2000 (Scirente)	WG	3	0.21 0.21 0.21	0.05 0.05 0.05	19 41 43	flower heads	0 2 7 9 14	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02, < 0.02	2	0030602 Pointurier (2001, PYMET_112) Remark : DAT 14 samples were cut into segments before storage
France (North), St. Pierre des Corps 2000 (Ardego)	WG	3	0.19 0.19 0.9	0.05 0.05 0.05	19 41 45	flower heads	14	< 0.02, < 0.02	2	0030702 Pointurier (2001, PYMET_113) Remark Samples were cut into segments before storage
France (North), Poncey les Athée 2000 (Vinson)	WG	3	0.2 0.2 0.21	0.05 0.05 0.05	19 19 43	flower heads	0 3 7 10 14	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02, < 0.02	2	0030601 Pointurier (2001, PYMET_114) Remark : DAT 3,7, 10 and 14 samples were cut into segments before storage
France (North), Labergement les Auxonne 2000 (Frémont)	WG	3	0.22 0.2 0.21	0.05 0.05 0.05	19 19 43	flower heads	14	< 0.02, < 0.02	2	0030701 Pointurier (2001, PYMET_115) Remark : Samples were cut into segments before storage

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Switzerland, Fully 2001 (Fremont)	WG	3	0.2 0.2 0.2	0.02 0.02 0.02	17 39 43	flower heads	0 11	0.12 < 0.02, < 0.02	6	1011/01 Gasser (2002, PYMET_116) Remark : Samples were cut into segments before storage
France (North), Labergement les Auxonne 2001 (Fremont)	WG	3	0.2 0.22 0.22	0.05 0.05 0.05	19 19 43	flower heads	0 3 7 10 14	0.07 < 0.02 < 0.02 < 0.02, < 0.02 < 0.02	3	0131201 Pointurier (2002, PYMET_117) Remark : DAT 7, 10 and 14 samples were cut into segments before storage
France (North), Poncey les Athee 2001 (Nautilus)	WG	3	0.2 0.2 0.21	0.05 0.05 0.05	19 19 43	flower heads	10	< 0.02, < 0.02	7	0131301 Pointurier (2002, PYMET_118) Remark : Samples were cut into segments before storage
Switzerland, Fully 2001 (Fremont)	WG	3	0.2 0.2 0.2	0.02 0.02 0.02	17 19 42	flower heads	0 3 7 10 14	0.02 < 0.02 < 0.02 < 0.02, < 0.02 < 0.02	6	1012/01 Gasser (2002, PYMET_119) Remark : Samples were cut into segments before storage

DAT = Days after last treatment

BBCH = 17 7 true leaves unfolded

BBCH 19 9 = or more true leaves unfolded

BBCH 39–41 = Cauliflower heads begin to form; width of growing tip > 1 cm

BBCH 43 = 30% of the expected head diameter reached

BBCH 45 = 50% of the expected head diameter reached

Head cabbage

Table 90 Residues of pymetrozine following foliar application to head cabbage

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Northern Europe										
Germany,	WP	3	0.17	0.03	—	whole plant	0	0.08	3	1128/95

[illegible]

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Portugal, Lourinha 1994 (Capitata L.)	WP	3	0.18 0.19 0.2	0.03 0.03 0.03	— 41 45	head	0 7 14 21 28	5.5 0.56 0.02 < 0.02 < 0.02	13	1024/94 Bohm (1996, PYMET_102) Day 0 control : 0.05 mg/kg
	WP	3	0.18 0.2 0.19	0.03 0.03 0.03	— 41 45	head	0 7 14 21 28	4.4 0.26 0.02 < 0.02 < 0.02	13	1025/94 Bohm (1996, PYMET_103) Replicate plot
Portugal, Escaroupim 1994 (Capitata L.)	WP	3	0.19 0.2 0.21	0.03 0.03 0.03	— 41 45	head	0 7 14 21 28	15.5 1.1 0.5 < 0.02 < 0.02	13	1026/94 Bohm (1996, PYMET_104)
Portugal, Lourinha 1994 (Capitata L.)	WP	3	0.16 0.25 0.2	0.03 0.03 0.03	— 41 45	head	0 7 14 21	2.9 0.04 0.03 < 0.02	17	1191/94 Bohm (1996, PYMET_105)
Portugal, Lourinha 1995 (Sabauda L.)	WP	3	0.18 0.16 0.17	0.03 0.03 0.03	— 41 45	head	0 6 14 21 28	0.63 < 0.02 < 0.02 < 0.02 < 0.02	8	1030/95 Bohm (1996, PYMET_106)
Portugal, Lourinha 1995 (Capitata L.)	WP	3	0.17 0.16 0.18	0.03 0.03 0.03	— 41 45	head	0 6 14 21 28	1.3 0.21 0.03 < 0.02 < 0.02	8	1031/95 Bohm (1996, PYMET_107) Replicate plot (Distance ~1km)
Portugal, Lourinha 1995 (Capitata L.)	WP	3	0.16 0.19 0.17	0.03 0.03 0.03	— 41 45	head	0 6 14 21 28	1.4 0.23 < 0.02 < 0.02 < 0.02	8	1035/95 Bohm (1996, PYMET_108) Replicate plot (Distance ~5km)
France (South), Canals 2004 (Rigoletto)	WG	3	0.2 0.2 0.2	0.03 0.03 0.03	41 44 45	head	0 3 7 14 21	2.5 0.04 < 0.01 < 0.01 < 0.01	8	AF/7932/SY/1 Mason (2005, PYMET_109)
	WG +adj.	3	0.2 0.2 0.2	0.03 0.03 0.03	41 44 45	head	0 3 7 14 21	3.7 0.04 0.01 < 0.01 < 0.01	8	
France (South), Vesines 2004	WG	3	0.2 0.2 0.2	0.03 0.03 0.03	19 19 42	head	0 3 7 14	2.3 1.2 0.02 < 0.01	6	AF/7932/SY/2 Mason (2005, PYMET_109)

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
(Castello)							21	< 0.01		
	WG +adj.	3	0.2 0.2 0.2	0.03 0.03 0.03	19 19 42	head	0 3 7 14 21	3.5 2.0 0.05 < 0.01 < 0.01	6	
Spain, Zaragoza 2004 (Taler)	WG	3	0.2 0.2 0.2	0.03 0.03 0.03	43 44 47	head	0 3 7 14 21	1.8 0.1 0.09 0.05 < 0.01	3	AF/7932/SY/3 Mason (2005, PYMET_109)
	WG +adj.	3	0.2 0.2 0.2	0.03 0.03 0.03	43 44 47	head	0 3 7 14 21	1.8 0.12 0.13 0.04 < 0.01	3	
Spain, Alagón 2004 (Dama)	WG	3	0.2 0.2 0.2	0.03 0.03 0.03	43 44 45	head	0 3 7 14 21	5.0 2.3 1.6 0.03 0.04	3	AF/7932/SY/5 Mason (2005, PYMET_109)
	WG +adj.	3	0.2 0.2 0.2	0.03 0.03 0.03	43 44 45	head	0 3 7 14 21	7.5 2.0 1.5 0.03 0.03	3	

+adj. = With adjuvant

DAT = Days after last treatment

BBCH 18 8 = True leaves unfolded

BBCH 41 = Heads begin to form: the two youngest leaves do not unfold

BBCH 42 = 20% of the expected head size reached

BBCH 43 = 20% of the expected head size reached

BBCH 47 = 70% of the expected head size reached

Cucumbers

Table 91 Residues of pymetrozine following foliar application to cucumbers in glasshouse

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Spain, Calahonda 1995 (Marumba)	WP	3	0.45 0.45 0.45	0.03 0.03 0.03	89 89 89	fruit	0 3 7 14 21	0.21 0.17 0.07 0.02 < 0.02	3	1133/95 Tribolet (1996, PYMET_120)
Spain, Carchuna 1995 (Aurelia)	WP	3	0.45 0.45 0.45	0.03 0.03 0.03	89 89 89	fruit	0 3 7 14 21	0.51 0.39 0.17 0.04 < 0.02	3	1134/95 Tribolet (1996, PYMET_121)
Spain,	WP	3	0.45	0.03	89	fruit	0	0.22	3	1135/95

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
La Aldeilla 1995 (Brunes)			0.45 0.45	0.03 0.03	89 89		3 7 14 21	0.18 0.06 < 0.02 < 0.02		Tribolet (1996, PYMET_122)
Switzerland, Füllinsdorf 1997 (Enigma)	WG	3	0.45 0.45 0.45	0.09 0.09 0.09	51 61 74	fruit	-0 0 1 3 7	0.07 0.1 0.11 0.06 0.04	10	1111/97 Tribolet (1998, PYMET_123)
Netherlands, Siebengewald 2006 (Fitnes)	WP	3	0.47 0.47 0.48	0.45 0.45 0.45	83 85 89	fruit	-0 0 1 3 7 10	0.009 0.29 0.22 <u>0.088</u> 0.03 0.008	1	NL-IR-06-0156 Mason (2007, PYMET_124)
France, Fournes en Weppes 2006 (Aramon)	WP	3	0.46 0.42 0.43	0.45 0.45 0.45	72 74 76	fruit	-0 0 1 3 7 10	0.014 0.4 0.35 <u>0.21</u> 0.029 0.014	1	NL-IR-06-0157 Mason (2007, PYMET_124)
Spain, Utrera 2006 (Suxo)	WP	3	0.44 0.44 0.44	0.45 0.45 0.45	70 73 75	fruit	-0 0 1 3 7 10	0.016 0.034 0.035 <u>0.045</u> 0.035 0.01	1	ES-IR-06-0282 Mason (2007, PYMET_125)
France, Chateaufort 2006 (Avalon)	WP	3	0.45 0.43 0.49	0.45 0.45 0.45	82 84 84	fruit	-0 0 1 3 7 10	0.024 0.15 0.094 <u>0.083</u> 0.036 0.014	1	FR-IR-06-0159 Mason (2007, PYMET_125)
United Kingdom, Cottingham 2013 (Bon Bon)	WG	3	0.43 0.49 0.49	0.05 0.05 0.05	60 61 69	fruit	1 3	0.24 <u>0.11</u>	1	S13-02540-01 Dorange (2014, PYMET_181) Remark: CGA294849 & CGA324284 < 0.01 mg/kg, respectively, in all samples
Poland, Wielkopolskie 2013 (Colonel)	WG	3	0.45 0.45 0.45	0.08 0.08 0.08	69 72 74	fruit	1 3	0.21 <u>0.09</u>	1	S13-02540-02 Dorange (2014, PYMET_181) Remark: CGA294849 & CGA324284 < 0.01 mg/kg, respectively, in all samples
Spain,	WG	3	0.46	0.09	79	fruit	1	0.17	1	S13-02540-05

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Elche 2013 (Neptuno)			0.49 0.43	0.06 0.07	81 82		3	<u>0.07</u>		Dorange (2014, PYMET_181) Remark: CGA294849 & CGA324284 < 0.01 mg/kg, respectively, in all samples
Spain, Xativa 2013 (Potomac)	WG	3	0.41 0.41 0.41	0.04 0.04 0.04	81 82 85	fruit	1 3	0.13 <u>0.08</u>	1	S13-02540-06 Dorange (2014, PYMET_181) Remark: CGA294849 & CGA324284 < 0.01 mg/kg, respectively, in all samples

DAT = Days after last treatment

BBCH 51 = First flower initial with elongated ovary visible on main stem

BBCH 61 = First flower open on main stem

BBCH 70–79 = First to 9 or more fruits on main stem have reached typical size and form

BBCH 81–89 = 10–90% of fruits show typical fully ripe colour

Melons

Table 92 Residues of pymetrozine following foliar application to melons in glasshouse

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Italy, Serravalle a Po 1999 (Harper)	WG	3	0.3 0.3 0.3	0.03 0.03 0.03	74 78 86	whole fruit ^a	0 3	0.03 0.025	7	1072/99 Tribolet (2000, PYMET_126) Remarks: - Samples were cut into segments before storage.
	WP	3	0.3 0.3 0.3	0.03 0.03 0.03	74 78 86	whole fruit ^a	0 3	0.02 0.025	7	- Due to segmentation peel and pulp came into contact, causing contamination.
						peel	0 3	0.025 0.02		
						pulp	0 3	0.025 0.02		
Italy, Serravalle a Po 1999	WG	3	0.3 0.3 0.3	0.03 0.03 0.03	74 78 86	whole fruit ^a	0 3	0.02 0.025	5	1071/99 Tribolet (2000, PYMET_127)
						peel	0	0.035		

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
(Capitol)						pulp	3 0 3	0.04 0.02 0.02		Remarks: Samples were cut into segments before storage.
	WP	3	0.3 0.3 0.3	0.03 0.03 0.03	74 78 86	whole fruit ^a peel pulp	0 3 0 3 0 3	0.03 < 0.02 0.065 0.02 0.02 < 0.02	5	Due to segmentation peel and pulp came into contact, causing contamination.
France, Lansargues 1999 (Lunastar)	WG	3	0.3 0.3 0.3	0.03 0.03 0.03	64 68 81	whole fruit	3	0.05, 0.04 (0.045)	4	9931602 Pointurier (1999, PYMET_128)
	WP	3	0.3 0.3 0.3	0.03 0.03 0.03	64 68 81	whole fruit	3	0.15, 0.16 (0.16)	4	
France, Le Cellier 1999 (Lunastar)	WG	3	0.3 0.3 0.3	0.03 0.03 0.03	72 74 85	whole fruit	2	0.08, 0.08 (0.08)	4	9931601 Pointurier (1999, PYMET_129)
	WP	3	0.3 0.3 0.3	0.03 0.03 0.03	72 74 85	whole fruit	2	0.11, 0.11 (0.11)	4	
Spain, Carchuna 1998 (Galia)	WP	3	0.3 0.3 0.3	0.04 0.04 0.04	73 89 89	whole fruit	0 3 7 14	0.06 0.02 0.02 0.03	8	1075/98 Tribolet (1999, PYMET_130) Remarks: Samples were halved before storage. Residues of GS23199 were < 0.02 mg/kg in all samples
	WP	3	0.3 0.3 0.3	0.04 0.04 0.04	73 89 89	whole fruit	3	0.02, 0.04 (0.03)	9	1077/98 Tribolet (1999, PYMET_132) Replicate plot Remarks: Samples were halved before storage. Residues of GS23199 were < 0.02 mg/kg in

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
										all samples
Spain, Los Banos 1998 (Blanco portugués)	WP	3	0.3 0.3 0.3	0.05 0.05 0.05	89 89 89	whole fruit	0 3 7 14	0.03 < 0.02 < 0.02 < 0.02	8	1076/98 Tribolet (1999, PYMET_131) Remarks: Samples were halved before storage. Residues of GS23199 were < 0.02 mg/kg in all samples
	WP	3	0.3 0.3 0.3	0.05 0.05 0.05	89 89 89	whole fruit	3	0.02, 0.02 (0.02)	8	1078/98 Tribolet (1999, PYMET_133) Replicate plot Remarks: Samples were halved before storage. Residues of GS23199 were < 0.02 mg/kg in all samples

^a Calculated

DAT = Days after last treatment

BBCH 61–69 = 1 to 9 or more flowers open on the main stem

BBCH 71–79 = 1 to 9 or more fruit on main stem have reached typical size and form

BBCH 81–89 = 10–90% of fruits show typical fully ripe colour

Peppers, sweet

Table 93 Residues of pymetrozine following foliar application to sweet peppers in glasshouse

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Netherlands, Etten Leur 1999 (Spirit)	WG	3	0.43 0.43 0.42	0.03 0.03 0.03	71–79 71–79 71–79	fruits	0 3	0.39, 0.23 (0.31) 0.19, 0.28 (0.24)	8	1091/99 Tribolet (2000, PYMET_134)
	WP	3	0.43 0.43 0.41	0.03 0.03 0.03	71–79 71–79 71–79	fruits	0 3	0.21, 0.24 (0.23) 0.26, 0.16 (0.21)	8	Remark: Procedural recovery : 66 %
Italy, Camisano	WG	3	0.45 0.45 0.45	0.03 0.03 0.03	76 77 78	fruits	0 3	0.48, 0.67 (0.58) 0.69, 0.27	8	1073/99 Tribolet (2000,

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
1999 (Stratos)								(0.58)		PYMET_135)
	WP	3	0.45 0.45 0.45	0.03 0.03 0.03	76 77 78	fruits	0 3	0.15, 0.37 (0.26) 0.2, 0.27 (0.24)	8	
Italy, S. Dona di Piave-ve 1999 (Stratos)	WG	3	0.45 0.45 0.45	0.03 0.03 0.03	77 77 77	fruits	0 3	0.3, 0.44 (0.37) 0.12, 0.22 (0.17)	5	1074/99 Tribolet (2000, PYMET_136)
	WP	3	0.45 0.45 0.45	0.03 0.03 0.03	77 77 77	fruits	0 3	0.33, 0.35 (0.34) 0.16, 0.17 (0.17)	5	
Spain, Utrera 2006 (Italico)	WP	3	0.47 0.47 0.45	0.05 0.05 0.05	73 75 77	fruits	-0 0 1 3 7 10	1.0 1.8 1.4 1.4 1.1 0.65	1	ES-IR-06-0150 Mason (2007, PYMET_137) Remark: Residues of CGA300407 were < 0.005 mg/kg in all samples
France, Mauguio 2006 (Goal F1)	WP	3	0.45 0.44 0.44	0.05 0.05 0.05	73 81 82	fruits	-0 0 1 3 7 10	0.32 0.56 0.49 0.54 0.18 0.34	1	FR-IR-06-0151 Mason (2007, PYMET_137) Remark: Residues of CGA300407 were < 0.005 mg/kg in all samples
Netherlands, Meterik 2006 (Corsica)	WP	3	0.46 0.47 0.46	0.05 0.05 0.05	81 85 87	fruits	-0 0 1 3 7 10	0.34 0.54 0.9 0.64 0.54 0.37	1	NL-IR-06-160 Mason (2007, PYMET_138) Remark: Residues of CGA300407 were < 0.005 mg/kg in all samples
Netherlands, Belfeld 2006 (Derby)	WP	3	0.45 0.46 0.46	0.05 0.05 0.05	81 85 87	fruits	-0 0 1 3 7 10	0.34 0.63 0.57 0.43 0.28 0.23	1	NL-IR-06-161 Mason (2007, PYMET_138) Remark: Residues of CGA300407 were < 0.005 mg/kg in all samples

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
France, L'Isle sur Sorgue 1998 (Lima)	WP	3	0.3 0.3 0.3	0.03 0.03 0.03	85 85 85	fruits	0 3	0.36 0.32	2	9831702 Pointurier (1999, PYMET_139) Remark: Residues of GS23199 were < 0.02 mg/kg in all samples
Netherlands, Made 1997 (Blondy)	WG	3	0.3 0.3 0.3	0.03 0.03 0.03	75 75 75	fruits	-0 0 3 7 14	0.17 0.29 0.27 0.19 0.07	10	IR2297 Tack (1998, PYMET_140)
	WP	3	0.3 0.3 0.3	0.03 0.03 0.03	75 75 75	fruits	-0 0 3 7 14	0.15 0.2 0.15 0.19 0.05	10	
Spain, Los Palacios 1998 (Italico)	WP	3	0.29 0.29 0.31	0.03 0.03 0.03	81 85 85	fruits	0 3	0.72, 0.73 (0.73) 0.58, 0.61 (0.6)	10	1055/98 Tribolet (1999, PYMET_141) Remark: Residues of GS23199 were < 0.02 mg/kg in all samples
France, Monteux 1998 (Lipari)	WP	3	0.3 0.3 0.3	0.03 0.03 0.03	85 85 85	fruits	0 3	0.73 0.56	2	9831701 Pointurier (1999, PYMET_142) Remark: Residues of GS23199 were < 0.02 mg/kg in all samples
Spain, Mareny de Barraquetes 1998 (Italiano)	WP	3	0.3 0.3 0.3	0.05 0.03 0.03	72 73 73	fruits	0 3	0.51, 0.73 (0.62) 0.25, 0.34 (0.3)	9	1054/98 Tribolet (1999, PYMET_143) Remark: Residues of GS23199 were < 0.02 mg/kg in all samples
Switzerland, Aesch 1997 (not reported)	WG	3	0.3 0.3 0.3	0.06 0.06 0.06	73 77 79	fruits	-0 0 1 3 7	0.54 0.67 0.26 0.33 0.29	9	1110/97 Tribolet (1998 PYMET_144)
United	WG	3	0.42	0.05	75	fruits	1	0.55	1	S13-02500-01

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Kingdom, Newbourne 2013 (Atris)			0.45 0.45	0.05 0.04	75 77		3	0.83		Gemrot (2014, PYMET_182) Remark: CGA294849 & CGA324284 < 0.01 mg/kg, respectively, in all samples
France, Allones 2013 (Snackor F1)	WG	3	0.46 0.49 0.45	0.07 0.08 0.07	81 83 87	fruit	1 3	0.28 0.16	1	S13-02500-02 Gemrot (2014, PYMET_182) Remark: CGA294849 & CGA324284 < 0.01 mg/kg, respectively, in all samples
France, Elne 2013 (Remus)	WG	3	0.45 0.44 0.46	0.07 0.07 0.07	81 83 87	fruits	1 3	0.38 0.43	1	S13-02500-03 Gemrot (2014, PYMET_182) Remark: CGA294849 & CGA324284 < 0.01 mg/kg, respectively, in all samples
France, Rivesaltes 2013 (Joselito)	WG	3	0.45 0.46 0.45	0.07 0.07 0.07	81 83 87	Fruits	1 3	1.2 1.1	1	S13-02500-04 Gemrot (2014, PYMET_182) Remark: CGA294849 & CGA324284 < 0.01 mg/kg, respectively, in all samples

DAT = Days after last treatment

BBCH 71–79 = 1st to 9th or more fruit cluster: fruit has reached typical size

BBCH 81–89 = 10–90% of fruits show typical fully ripe colour

Tomatoes

Table 94 Residues of pymetrozine following foliar application to tomatoes in glasshouse

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Switzerland,	WP	3	0.4	0.02	82	fruits	0	0.16	12	1083/94

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Fully 1994 (Merano)			0.4 0.4	0.02 0.02	83 84		1 3 8 14	0.15 0.16 0.18 0.11		Tribolet (1995, PYMET_145) Last appl. : 25-JUL-1994
Switzerland, Fully 1994 (Savor)	WP	3	0.4 0.4 0.4	0.02 0.02 0.02	80 82 84	fruits controls	0 1 3 7 14 0 14	0.47 0.09 0.08 0.08 0.09 0.62 0.11	14	1084/94 Tribolet (1995, PYMET_146) Last appl. : 04-JUL-1994
Switzerland, Fully 1994 (Savor)	WP	3	0.4 0.4 0.4	0.02 0.02 0.02	82 83 84	fruits	0 1 3 7 15	0.4 0.25 0.23 0.23 0.02	12	1085/94 Tribolet (1995, PYMET_147) Last appl. : 18-JUL-1994
Switzerland, Fully 1994 (Paola)	WP	3	0.4 0.4 0.4	0.02 0.02 0.02	82 83 84	fruits	0 1 3 7 14	0.24 0.22 0.17 0.09 0.08	10	1086/94 Tribolet (1995, PYMET_148) Last appl. : 26-JUL-1994
Switzerland, Füllinsdorf 1997 (Frontera)	WG	3	0.45 0.45 0.45	0.09 0.09 0.09	79 81 85	fruits	-0 0 1 3 7	0.11 0.2 0.24 0.22 0.11	10	1112/97 Tribolet (1998, PYMET_149)
Netherlands, Bleiswijk 1998 (Aromata)	WP	3	0.44 0.45 0.45	0.03 0.03 0.03	71-79 71-79 71-79	fruits	0 3	0.17, 0.2 (0.19) 0.17, 0.2 (0.19)	5	1093/98 Tribolet (1999, PYMET_150) Remark: Residues of GS23199 were < 0.02 mg/kg in all samples
Germany, Upstadt-Weiher 2010 (Providance)	WG	3	0.45 0.43 0.45	0.08 0.08 0.08	82 84 85	fruits	0 1 3 7 14	0.81 <u>0.77</u> 0.38 0.4 0.56	1	S10-01586-01 Allen (2011, PYMET_151)
France, Allonnes 2010 (Sadique)	WG	3	0.45 0.46 0.47	0.08 0.08 0.08	81 83 87	fruits	0 1 3 7 14	0.21 <u>0.08</u> 0.05 0.05 0.05	1	S10-01586-02 Allen (2011, PYMET_151)
France, Elne	WG	3	0.46 0.49 0.45	0.15 0.15 0.15	82 85 85	fruits	0 1 3	0.34 <u>0.39</u> 0.2	1	S10-01587-01 Allen (2011,

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
2010 (Sartigys)							7 14	0.17 0.35		PYMET_152)
France, Orgueil 2010 (Brenda)	WG	3	0.47 0.44 0.46	0.11 0.11 0.11	81 82 89	fruits	0 1 3 7 14	0.28 0.27 0.2 <u>0.3</u> 0.11	1	S10-01587-03 Allen (2011, PYMET_152)
United Kingdom, Newbourne 2013 (Douglas)	WG	3	0.41 0.44 0.41	0.15 0.15 0.15	76 78 81	fruits	1 3	<u>0.33</u> 0.3	2	S13-02535-01 Gemrot (2014, PYMET_183) Remark: CGA294849 & CGA324284 < 0.01 mg/kg, respectively, in all samples
France, Langu� Mercier 2013 (Altesse)	WG	3	0.41 0.43 0.45	0.09 0.09 0.09	85 85 87	fruits	1 3	<u>0.14</u> 0.13	2	S13-02535-02 Gemrot (2014, PYMET_183) Remark: CGA294849 & CGA324284 < 0.01 mg/kg, respectively, in all samples
Spain, Torrellano 2013 (Anairis)	WG	3	0.46 0.41 0.45	0.05 0.04 0.04	85 85 85	fruits	1 3	<u>0.27</u> 0.17	2	S13-02535-03 Gemrot (2014, PYMET_183) Remark: CGA294849 & CGA324284 < 0.01 mg/kg, respectively, in all samples
Spain, Xativa 2013 (Optima)	WG	3	0.47 0.45 0.44	0.05 0.04 0.04	81 82 82	fruits	1 3	<u>0.18</u> 0.13	2	S13-02535-04 Gemrot (2014, PYMET_183) Remark: CGA294849 & CGA324284 < 0.01 mg/kg, respectively, in all samples

DAT = Days after last treatment

BBCH 71–79 = 1st to 9th or more fruit cluster: fruit has reached typical size

BBCH 81–89 = 10–90% of fruits show typical fully ripe colour

*Leafy vegetables, except brassica leafy vegetables**Head lettuce*

Table 95 Residues of pymetrozine following foliar application to head lettuce

Location,	Application					Residues, mg/kg				Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	GS23199	months	Reference
USA, Fresno (CA) 1998 (Great Lakes 659-700)	WG	2	0.1 0.1	0.03 0.03	mature mature	head	0 1 3 5 9	0.37, 0.39 (0.38) 0.11, 0.12 (0.12) 0.05, 0.06 (0.06) 0.03, 0.17 (0.1) < 0.02, 0.03 (0.03)	< 0.02(2) 0.03, 0.03 (0.03) 0.02, 0.05 (0.04) < 0.02(2) < 0.02, 0.03 (0.03)	1	02-IR-049-97 Campbell (1998, PYMET_153)
						head w/o wrapper leaves	0	< 0.02, 0.05 (0.04)	< 0.02(2)		
						wrapper leaves	0 7	0.23, 0.27 (0.25) < 0.02, 0.06 (0.04)	0.04, 0.06 (0.05) 0.05, 0.06 (0.06)		
USA, Indian River (FL) 1998 (Michigan Peto)	WG	2	0.1 0.1	0.03 0.03	mature mature	head	0 7	0.89, 0.92 (0.91) 0.06, 0.06 (0.06)	< 0.02, 0.02 (0.02) < 0.02(2)	5	07-IR-012-97 Campbell (1998, PYMET_153)
						head w/o wrapper leaves	0 7	< 0.02(2) < 0.02(2)	< 0.02(2) < 0.02(2)		
						wrapper leaves	0 7	1.2, 1.7 (1.5) 0.05, 0.11 (0.08)	0.03, 0.03 (0.03) < 0.02(2)		
USA, Wayne (NY) 1997 (Crispino)	WG	2	0.1 0.1	0.03 0.03	mature mature	head	0 7	0.3, 0.34 (0.32) 0.02, 0.03 (0.03)	< 0.02(2) < 0.02(2)	10	NE-IR-807-97 Campbell (1998, PYMET_153)
						head w/o wrapper leaves	0 7	0.14, 0.15 (0.15) < 0.02, 0.03 (0.03)	< 0.02(2) < 0.02(2)		
						wrapper leaves	0 7	0.48, 0.81 (0.65) 0.06, 0.07 (0.07)	0.03, 0.03 (0.03) < 0.02(2)		
USA, Monterey (CA) 1997 (Great Lakes)	WG	2	0.1 0.1	0.03 0.03	mature mature	head	0 7	< 0.02, 0.04 (0.03) < 0.02(2)	< 0.02(2) < 0.02(2)	1	0W-IR-504-97 Campbell (1998,
						head	0	< 0.02, 0.02	< 0.02(2)		

Location,	Application					Residues, mg/kg				Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	GS23199	months	Reference
						w/o wrapper leaves	7	(0.02) < 0.02(2)	< 0.02(2)		PYMET_153)
						wrapper leaves	0	0.91, 1.4 (1.2)	0.04, 0.04 (0.04)		
							7	0.07, 0.08 (0.08)	< 0.02(2)		
USA, San Luis Obispo (CA)	WG	2	0.1 0.1	0.03 0.03	mature mature	head	0	0.25, 0.35 (0.3)	< 0.02(2)	6	0W-IR-505-97
1997 (Pipus)							6	< 0.02(2)	< 0.02(2)		Campbell (1998, PYMET_153)
						head w/o wrapper leaves	0	< 0.02, 0.03 (0.03)	< 0.02(2)		
							6	< 0.02(2)	< 0.02(2)		
						wrapper leaves	0	0.75, 0.81 (0.78)	< 0.02(2)		
							6	0.11, 0.23 (0.17)	< 0.02(2)		
USA, Yuma (AZ)	WG	2	0.1 0.1	0.03 0.03	mature mature	head	0	0.27, 0.36 (0.32)	< 0.02(2)	6	0W-IR-506-97
1997 (Raider)							7	< 0.02(2)	< 0.02(2)		Campbell (1998, PYMET_153)
						head w/o wrapper leaves	0	0.07, 0.11 (0.09)	< 0.02(2)		
							7	0.07, 0.12 (0.1)	< 0.02(2)		
						wrapper leaves	0	1.4, 1.4 (1.4)	< 0.02, 0.03 (0.03)		
							7	0.73, 0.79 (0.76)	0.05, 0.06 (0.06)		

DAT = Days after last treatment

Leaf lettuce

Table 96 Residues of pymetrozine following foliar application to leaf lettuce

Location,	Application					Residues, mg/kg				Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	GS23199	months	Reference
USA, Fresno (CA)	WG	2	0.1 0.1	0.03 0.03	mature mature	leaves	0	0.66, 0.87 (0.77)	< 0.02(2)	2	02-IR-050-97
1998 (Waldsmanns Green M1)							1	0.18, 0.5 (0.34)	< 0.02(2)		Campbell (1998, PYMET_153)
							3	0.19, 0.27 (0.23)	0.02, 0.04 (0.03)		
							5	0.03, 0.16 (0.1)	< 0.02, 0.02 (0.02)		
							7	0.09, 0.15 (0.12)	< 0.02(2)		
							9	0.05, 0.05 (0.05)	< 0.02(2)		

Location, Year (variety)	Application					Residues, mg/kg				Storage interval months	Trial No., Reference
	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	GS23199		
USA, Indian River (FL) 1997 (Black Seeded)	WG	2	0.1 0.1	0.03 0.03	mature mature	leaves	0 7	1.9, 2.8 (2.4) < 0.02(2)	< 0.02, 0.03 (0.03) < 0.02(2)	14	07-IR-003-97 Campbell (1998, PYMET_153)
USA, Wayne (NY) 1997 (Black Seeded Simpson)	WG	2	0.1 0.1	0.03 0.03	mature mature	leaves	0 7	1.4, 1.5 (1.5) 0.06, 0.08 (0.07)	< 0.02(2) < 0.02(2)	10	NE-IR-806- 97 Campbell (1998, PYMET_153)
USA, Monterey (CA) 1997 (Prize Head)	WG	2	0.1 0.1	0.03 0.03	mature mature	leaves	0 7	1.1, 1.7 (1.4) 0.28, 0.46 (0.37)	< 0.02(2) 0.02, 0.02 (0.02)	7	0W-IR-507- 97 Campbell (1998, PYMET_153)
USA, San Luis Obispo (CA) 1997 (Green Vision)	WG	2	0.1 0.1	0.03 0.03	mature mature	leaves	0 6	0.46, 0.71 (0.59) 0.05, 0.08 (0.07)	< 0.02(2) < 0.02(2)	6	0W-IR-508- 97 Campbell (1998, PYMET_153)
USA, Yuma (AZ) 1998 (Crisp and Green)	WG	2	0.1 0.1	0.03 0.03	mature mature	leaves	0 7	0.87, 1.1 (1.0) 0.09, 0.26 (0.18)	< 0.02(2) < 0.02(2)	3	0W-IR-509- 97 Campbell (1998, PYMET_153)

DAT = Days after last treatment

Spinach

Table 97 Residues of pymetrozine following foliar application to spinach

Location, Year (variety)	Application					Residues, mg/kg				Storage interval months	Trial No., Reference
	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	GS23199		
USA, Fresno (CA) 1998 (St. Helens)	WG	2	0.1 0.1	0.03 0.03	mature mature	leaves	0 7	4.8, 5.3 (5.1) 0.21, 0.32 (0.27)	< 0.02, 0.02 (0.02) 0.03, 0.05 (0.04)	2	02-IR-051-97 Campbell (1998, PYMET_153)
USA, San Patricio (TX) 1997 (Cascade)	WG	2	0.1 0.1	0.03 0.03	mature mature	leaves	0 7	3.8, 3.9 (3.9) 0.14, 0.15 (0.15)	< 0.02(2) < 0.02(2)	16	0S-IR-307-97 Campbell (1998, PYMET_153)
USA, Boulder (CO) 1997	WG	2	0.1 0.1	0.03 0.03	mature mature	leaves	0 7	2.4, 2.5 (2.5) < 0.02, 0.02 (0.02)	0.03, 0.04 (0.04) < 0.02(2)	12	NE-IR-302- 97 Campbell

Location,	Application					Residues, mg/kg				Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	GS23199	months	Reference
(Melody)											(1998, PYMET_153)
USA, Montgomery (VA) 1997 (Bloomsdate Long)	WG	2	0.1 0.1	0.03 0.03	mature mature	leaves	0 7	3.1, 3.2 (3.2) 0.09, 0.1 (0.1)	< 0.02(2) < 0.02(2)	11	MW-IR-302-97 Campbell (1998, PYMET_153)
USA, Hunterdon (NJ) 1997 (Tyee F1)	WG	2	0.1 0.1	0.03 0.03	mature mature	leaves	0 8	2.2, 2.8 (2.5) 0.11, 0.11 (0.11)	< 0.02(2) < 0.02(2)	12	NE-IR-502-97 Campbell (1998, PYMET_153)
USA, San Luis Obispo (CA) 1997 (not reported)	WG	2	0.1 0.1	0.03 0.03	mature mature	leaves	0 1 3 5 7 9	3.2, 5.9 (4.6) 3.2, 3.3 (3.3) 0.39, 0.39 (0.39) 0.24, 0.3 (0.27) 0.08, 0.17 (0.13) 0.1, 0.16 (0.13)	< 0.02(2) < 0.02(2) < 0.02(2) < 0.02(2) < 0.02(2) < 0.02(2)	8	OW-IR-514-97 Campbell (1998, PYMET_153)

DAT = Days after last treatment

Potatoes

Table 98 Residues of pymetrozine following foliar application to potatoes

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
United Kingdom, Driffield 2010 (Markies)	WG	3	0.21 0.2 0.21	0.1 0.1 0.1	85 85 89	tubers	-0 0 1 3 7	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	1	S10-01590-01 Allen (2011, PYMET_154)
United Kingdom, Driffield 2010 (Estima)	WG	3	0.21 0.2 0.2	0.1 0.1 0.1	85 92 95	tubers	-0 0 1 3 7	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	1	S10-01590-02 Allen (2011, PYMET_154)
Germany, Vaihingen 2010 (Selma)	WG	3	0.23 0.19 0.21	0.1 0.1 0.1	88 91 91	tubers	-0 0 1 3 7	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	1	S10-01590-03 Allen (2011, PYMET_154)

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
France (North), Audeville 2010 (Caesar)	WG	3	0.19 0.19 0.19	0.1 0.1 0.1	81 81 81	tubers	-0 0 1 3 7	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	1	S10-01590-04 Allen (2011, PYMET_154)

DAT = Days after last treatment

BBCH 81–89 = Ripening of fruit and seed

BBCH 91 = Beginning of leaf yellowing

BBCH 92–93 = Most of the leaves yellowish

BBCH 95 = 50% of the leaves brownish

Asparagus

Table 99 Residues of pymetrozine following foliar application to asparagus (vegetative)

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
USA, Soledad (CA) 1999 (UC157)	WG	3	0.19 0.19 0.19	0.04 0.03 0.02	veg. veg. veg.	spears	172	< 0.02, < 0.02	3	CA113 Samoil (2002, PYMET_159) Residues of GS23199 were < 0.02 mg/kg for all samples
USA, Salinas (CA) 1999 (UC157)	WG	3	0.19 0.19 0.19	0.04 0.03 0.02	veg. veg. veg.	spears	177	< 0.02, < 0.02	3	CA114 Samoil (2002, PYMET_159) Residues of GS23199 were < 0.02 mg/kg for all samples
USA, Bridgeton (NJ) 1999 (RRDC male hybrid)	WG	3	0.19 0.19 0.19	0.07 0.07 0.07	veg. veg. veg.	spears	225	< 0.02, < 0.02	4	NJ39 Samoil (2002, PYMET_159) Residues of GS23199 were < 0.02 mg/kg for all samples
USA, Prosser (WA) 1999 (Jersey Giant)	WG	3	0.19 0.19 0.19	0.04 0.04 0.04	veg. veg. veg.	spears	224	< 0.02, < 0.02	2	WA50 Samoil (2002, PYMET_159) Residues of GS23199 were < 0.02 mg/kg for all samples

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
	WG	3	0.19 0.19 0.19	0.04 0.04 0.04	veg. veg. veg.	spears	220	< 0.02, < 0.02	2	WA51 Samoil (2002, PYMET_159) Replicate trial Residues of GS23199 were < 0.02 mg/kg for all samples
USA, Wapato (WA) 1999 (Mary Washington)	WG	3	0.19 0.19 0.19	0.04 0.04 0.04	veg. veg. veg.	spears	221	< <u>0.02</u> , < <u>0.02</u>	2	WA52 Samoil (2002, PYMET_159) Residues of GS23199 were < 0.02 mg/kg for all samples
USA, Oregon (WI) 1999 (Mary Washington)	WG	3	0.19 0.19 0.19	0.05 0.05 0.05	veg. veg. veg.	spears	267	< <u>0.02</u> , < <u>0.02</u>	4	WI22 Samoil (2002, PYMET_159) Residues of GS23199 were < 0.02 mg/kg for all samples
	WG	3	0.19 0.19 0.19	0.05 0.05 0.05	veg. veg. veg.	spears	254	< 0.02, < 0.02	4	WI23 Samoil (2002, PYMET_159) Replicate trial Residues of GS23199 were < 0.02 mg/kg for all samples

DAT = Days after last treatment

veg. = Vegetative

Celery

Table 100 Residues of pymetrozine following foliar application to celery

Location,	Application					Residues, mg/kg				Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	GS23199	months	Reference
USA, Orange County (FL) 1997 (June Bell 1622)	WG	2	0.1 0.1	0.03 0.03	mature mature	sticks	0 7	0.38, 0.47 (0.43) < 0.02(2)	< 0.02(2) < 0.02(2)	11	FL-IR-411-97 Campbell (1998, PYMET_153)
USA, Newaygo (MI) 1997 (Florida 683K)	WG	2	0.1 0.1	0.03 0.03	mature mature	sticks	0 7	0.88, 1.0 (0.94) 0.03, 0.05 (0.04)	< 0.02(2) < 0.02(2)	10	NE-IR-703-97 Campbell (1998, PYMET_153)
USA, San Luis Obispo (CA) 1997 (Conquistador)	WG	2	0.1 0.1	0.03 0.03	mature mature	sticks	0 1 3 5 7 9	0.49, 0.63 (0.56) 0.25, 0.42 (0.34) 0.19, 0.21 (0.2) 0.11, 0.16 (0.14) 0.03, 0.04 (0.04) < 0.02(2)	< 0.02(2) < 0.02(2) < 0.02(2) < 0.02(2) < 0.02(2) < 0.02(2)	7	0W-IR-510-97 Campbell (1998, PYMET_153)
USA, San Luis Obispo (CA) 1997 (Conquistador)	WG	2	0.1 0.1	0.03 0.03	mature mature	sticks	0 7	0.33, 0.39 (0.36) 0.03, 0.03 (0.03)	< 0.02(2) < 0.02(2)	7	0W-IR-511-97 Campbell (1998, PYMET_153) Replicate plot
USA, Santa Barbara (CA) 1997 (Conquistador)	WG	2	0.1 0.1	0.03 0.03	mature mature	sticks	0 7	0.29, 0.41 (0.35) < 0.02(2)	< 0.02(2) < 0.02(2)	7	0W-IR-512-97 Campbell (1998, PYMET_153)
USA, Santa Barbara (CA) 1997 (5275)	WG	2	0.1 0.1	0.03 0.03	mature mature	sticks	0 7	0.72, 0.93 (0.82) < 0.02(2)	< 0.02(2) < 0.02(2)	7	0W-IR-513-97 Campbell (1998, PYMET_153) Replicate plot

DAT = Days after last treatment

Artichoke, globe

Table 101 Residues of pymetrozine following foliar application to globe artichokes

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
France (North), Plouzevede 2003 (Camus)	WG	2	0.2 0.2	0.07 0.07	74 77	Flower head	0 7 14 21	0.16 0.06 < 0.02 < 0.02	8	03-5060 Sole (2004, PYMET_155)
France (North), Cleder 2003 (Castel)	WG	2	0.2 0.2	0.07 0.07	72 79	Flower head	0 14	0.17 < 0.02	8	03-5061 Sole (2004, PYMET_156)
France (North), Treflaouenan 2002 (Castel)	WG	2	0.2 0.2	0.07 0.07	45 49	Flower head	0 3 7 14 21	0.12 0.02 < 0.02 < 0.02 < 0.02	8	02-1129 Sole (2003, PYMET_157)
France (North), Treflaouenan 2002 (Camus)	WG	2	0.2 0.2	0.07 0.07	41 44	Flower head	0 14	0.14 < 0.02, < 0.02	8	02-1130 Sole (2003, PYMET_158)

DAT = Days after last treatment

BBCH 41–49 = 10–90% of the expected root diameter reached

BBCH 71–79 = 10–90% of fruits have reached typical size

Rice

Table 102 Residues of pymetrozine in rice grain and husks following foliar application

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
China, Fujian 2007 (n.r.)	WG	1	0.15	n.r.	n.r.	grain husks	14 14 21	< 0.001 < 0.001 < 0.001	n.r.	Wu (2009, PYMET_050)
		2	0.15	n.r.	n.r.	grain husks	14 14 21	< 0.001 0.0036 < 0.001	n.r.	
		1	0.23	n.r.	n.r.	grain husks	14 14 21	< 0.001 0.0038 < 0.001	n.r.	
		2	0.23	n.r.	n.r.	grain husks	14 14 21	< 0.001 0.0061 < 0.001	n.r.	
China,	WG	1	0.15	n.r.	n.r.	grain	14	< 0.001	n.r.	Wu (2009,

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Fujian 2008 (n.r.)						husks	14 21	0.0038 < 0.001		PYMET_050)
		2	0.15	n.r.	n.r.	grain	14	< 0.001	n.r.	
						husks	14 21	0.0061 0.0034		
		1	0.23	n.r.	n.r.	grain	14	< 0.001	n.r.	
						husks	14	0.0018		
		2	0.23	n.r.	n.r.	grain	14	< 0.001	n.r.	
						husks	14 21	0.0036 0.0014		
China, Zhejiang 2007 (n.r.)	WG	1	0.15	n.r.	n.r.	grain	14	< 0.001	n.r.	Wu (2009, PYMET_050)
						husks	14 21	0.0014 < 0.001		
		2	0.15	n.r.	n.r.	grain	14	< 0.001	n.r.	
						husks	14 21	0.0016 < 0.001		
		1	0.23	n.r.	n.r.	grain	14	< 0.001	n.r.	
						husks	14 21	0.0036 < 0.001		
		2	0.23	n.r.	n.r.	grain	14	< 0.001	n.r.	
						husks	14 21	0.0058 < 0.001		
China, Zhejiang 2008 (n.r.)	WG	1	0.15	n.r.	n.r.	grain	14	< 0.001	n.r.	Wu (2009, PYMET_050)
						husks	14 21	0.0016 < 0.001		
		2	0.15	n.r.	n.r.	grain	14	< 0.001	n.r.	
						husks	14 21	0.0018 < 0.001		
		1	0.23	n.r.	n.r.	grain	14	< 0.001	n.r.	
						husks	14 21	0.0019 < 0.001		
		2	0.23	n.r.	n.r.	grain	14	< 0.001	n.r.	
						husks	14 21	0.0029 < 0.001		
China, Guangxi 2009	WG	1	0.15	n.r.	n.r.	grain	14 21 28	0.0054 0.002 0.0017	n.r.	Wu (2009, PYMET_050)

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
(n.r.)						husks	14 21 28	0.013 0.029 0.0032		
		2	0.15	n.r.	n.r.	grain	14 21 28	0.0065 0.0013 < 0.001	n.r.	
						husks	14 21 28	0.0084 0.0069 0.0043		
		1	0.23	n.r.	n.r.	grain	14 21 28	0.0074 0.0025 0.0014	n.r.	
						husks	14 21 28	0.006 0.0032 0.003		
		2	0.23	n.r.	n.r.	grain	14 21 28	0.01 0.0053 0.0011	n.r.	
						husks	14 21 28	0.034 0.014 0.0011		
China, Hunan	WG	1	0.15	n.r.	n.r.	grain	14 21 28	0.0086 0.0051 0.003	n.r.	Wu (2009, PYMET_050)
2009 (n.r.)						husks	14 21 28	0.13 0.048 < 0.001		
		2	0.15	n.r.	n.r.	grain	14 21 28	0.0079 0.0026 < 0.001	n.r.	
						husks	14 21 28	0.2 0.3 0.013		
		1	0.23	n.r.	n.r.	grain	14 21 28	0.017 0.016 0.0085	n.r.	
						husks	14 21 28	0.3 0.12 0.0094		
		2	0.23	n.r.	n.r.	grain	14 21 28	0.017 0.026 0.011	n.r.	
						husks	14 21 28	0.37 0.2 0.12		

n.r. = Not reported

DAT = Days after last treatment

Tree nuts

Pecan

Table 103 Residues of pymetrozine following foliar application in pecans

Location,	Application					Residues, mg/kg				Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	GS23199	months	Reference
USA, Mitchell (GA) 1998 (Desirable)	WG	2	0.15 0.15	0.03 0.03	hulls splitting	nutmeat	0	< 0.02(2)	< 0.02(2)	5	0S-IR-832-98/GA Joseph (1999, PYMET_048)
							3	< 0.02(2)	< 0.02(2)		
							6	< 0.02(2)	< 0.02(2)		
							10	< 0.02(2)	< 0.02(2)		
							14	< 0.02(2)	< 0.02(2)		
							18	< 0.02(2)	< 0.02(2)		
						Control	0	< 0.02	0.02		
							6	< 0.02	0.03		
							14	< 0.02	0.02		
USA, Chambers (AL) 1998 (Cape Fear)	WG	2	0.15 0.15	0.38 0.38	Shuck loose	nutmeat	14	< 0.02(2)	< 0.02(2)	5	0S-IR-841-98/AL Joseph (1999, PYMET_048)
USA, Dona Ana (NM) 1998 (Western Schley)	WG	2	0.15 0.15	0.05 0.05	shuck split	nutmeat	12	< 0.02(2)	< 0.02(2)	3	0S-IR-721-98/NM Joseph (1999, PYMET_048)
	WG	2	0.15 0.15	0.03 0.03	shuck split	nutmeat	12	< 0.02(2)	< 0.02(2)	3	
USA, Rapides Parish (LA) 1998 (Jackson)	WG	2	0.15 0.15	0.38 0.38	shell harden.	nutmeat	14	< 0.02(2)	< 0.02(2)	5	0S-IR-901-98/LA Joseph (1999, PYMET_048)
	WG	2	0.15 0.15	0.02 0.02	shell harden.	nutmeat	14	< 0.02(2)	< 0.02(2)	5	
USA, Wharton (TX) 1998 (Pawnee)	WG	2	0.15 0.15	0.05 0.05	nut fill	nutmeat	0	< 0.02(2)	< 0.02(2)	6	0S-IR-202-98/TX Joseph (1999, PYMET_048)
							3	< 0.02(2)	< 0.02(2)		
							6	< 0.02(2)	< 0.02(2)		
							10	< 0.02(2)	< 0.02(2)		
							14	< 0.02(2)	< 0.02(2)		
							18	< 0.02(2)	< 0.02(2)		
	WG	2	0.15 0.15	0.03 0.03	nut fill	nutmeat	0	< 0.02(2)	< 0.02(2)	6	
							3	< 0.02(2)	< 0.02(2)		
							6	< 0.02(2)	< 0.02(2)		
							10	< 0.02(2)	< 0.02(2)		
							14	< 0.02(2)	< 0.02(2)		
							18	< 0.02(2)	< 0.02(2)		

DAT = Days after last treatment

Walnuts

Table 104 Residues of pymetrozine following foliar application in walnuts

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
France (South), Camarsac 2002 (Franquette)	WG	2	0.1 0.1	0.02 0.02	7 7	nut + shell nutmeat	14 14	< 0.05 < 0.05	9	RE02049 Malet (2004, PYMET_160)
France (South), Gensac 2002 (Franquette)	WG	2	0.1 0.09	0.01 0.01	7 7	nut + shell nutmeat	14 14	< 0.05 < 0.05	9	RE02057 Malet (2004, PYMET_160)
France (South), Camarsac 2003 (Franquette)	WG	2	0.1 0.1	0.01 0.01	7 7	nut + shell nutmeat	14 14	< 0.05 < 0.05	9	RE03007 Malet (2004, PYMET_161)
France (South), Gensac 2003 (Lara)	WG	2	0.11 0.1	0.02 0.02	7 7	nut + shell nutmeat	14 14	< 0.05 < 0.05	9	RE03012 Malet (2004, PYMET_161)

BBCH 7 fruit development

DAT = Days after last treatment

Rapeseed

Table 105 Residues of pymetrozine following foliar application in rape seeds

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
European North										
United Kingdom, Owthorpe 2008 (Excalibur)	WG	1	0.1	0.05	59	seeds	110	< 0.01	9	S08-00624-01 Schulz (2009, PYMET_162)
United Kingdom, Isley Walton 2008 (Fortis)	WG	1	0.1	0.05	59	seeds	124	< 0.01	9	S08-00624-02 Schulz (2009, PYMET_162)
France (North), Stotzheim 2008 (Grizzly)	WG	1	0.1	0.05	59	seeds	124	< 0.01	9	S08-00624-03 Schulz (2009, PYMET_162)
Germany,	WG	1	0.1	0.05	59	seeds	75	< 0.01	9	S08-00624-04

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Wiesloch 2008 (Trabant)										Schulz (2009, PYMET_162)
European South										
France (South), Montauban 2008 (Barrel)	WG	1	0.1	0.05	59	seeds	110	< 0.01	9	S08-00625-01 Schulz (2009, PYMET_163)
France (South), Verdun Sur Garonne 2008 (Porthos)	WG	1	0.1	0.05	59	seeds	118	< 0.01	9	S08-00625-02 Schulz (2009, PYMET_163)
Italy, San Lazzaro di Savena 2008 (Bel Canto)	WG	1	0.1	0.05	59	seeds	83	< 0.01	9	S08-00625-03 Schulz (2009, PYMET_163)
Italy, San Lazzaro di Savena 2008 (Avvenir)	WG	1	0.1	0.05	59	seeds	83	< 0.01	9	S08-00625-04 Schulz (2009, PYMET_163) Replicate trial

DAT = Days after last treatment

BBCH 59 = First petals visible, flower buds still closed

Cottonseed

Table 106 Residues of pymetrozine following foliar application in cotton seeds

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Spain, Coria del Rio 1997 (Vulcano)	WG	3	0.2 0.18 0.21	0.2 0.24 0.22	75 76 79	delinted seeds	35 50	< 0.02 < 0.02	9	1162/97 Tribolet (1998, PYMET_164)
Greece, Roumeli 1999 (506 Stoneville)	WP	3	0.2 0.2 0.2	0.04 0.04 0.04	79 81 83	delinted seeds	0 7 14 21 28 35	0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	13	1099/99 Tribolet (2001, PYMET_165)
	WG	3	0.2 0.2 0.2	0.04 0.04 0.04	79 81 83	delinted seeds	0 7 14	0.03 < 0.02 < 0.02	13	

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
							21 28 35	< 0.02 < 0.02 < 0.02		
Greece, Kato Glines 1999 (453 Stoneville)	WP	3	0.2 0.2 0.2	0.04 0.04 0.04	77 79 83	delinted seeds	0 7 14 21 28 35	0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	12	1100/99 Tribolet (2001, PYMET_166)
	WG	3	0.2 0.2 0.2	0.04 0.04 0.04	77 79 83	delinted seeds	0 7 14 21 28 35	0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	12	
Greece, Kamaroula 1999 (453 Stoneville)	WP	3	0.2 0.2 0.2	0.04 0.04 0.04	69 73 80	delinted seeds	35	< 0.02	13	1101/99 Tribolet (2001, PYMET_167)
	WG	3	0.2 0.2 0.2	0.04 0.04 0.04	69 73 80	delinted seeds	35	< 0.02	13	
Greece, Mavrogia 1999 (506 Stoneville)	WP	3	0.2 0.2 0.2	0.04 0.04 0.04	69 73 80	delinted seeds	35	< 0.02	15	1102/99 Tribolet (2001, PYMET_168)
	WG	3	0.2 0.2 0.2	0.04 0.04 0.04	69 73 80	delinted seeds	35	< 0.02	15	
Greece, Ipodromos 1999 (453 Stoneville)	WG	3	0.2 0.2 0.2	0.04 0.04 0.04	69 73 80	delinted seeds	35	< 0.02	13	1103/99 Tribolet (2001, PYMET_169)

DAT = Days after last treatment

BBCH 69 = End of flowering

BBCH 71–79 = 10–90% of bolls have attained their final size

BBCH 81–89 = 10–90% of bolls open

Animal feeds**Rice straw**

Table 107 Residues of pymetrozine in rice straw and stalks following foliar application

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
China, Fujian	WG	1	0.15	n.r.	n.r.	straw	14 21	0.0063 < 0.001	n.r.	Wu (2009, PYMET_050)

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
2007 (n.r.)										
		2	0.15	n.r.	n.r.	straw	14 21	0.0052 < 0.001	n.r.	
		1	0.23	n.r.	n.r.	straw	14 21	0.01 0.0015	n.r.	
		2	0.23	n.r.	n.r.	straw	14 21	0.01 0.016	n.r.	
		1	0.23	0.05	n.r.	Stalks	3 7 14 28 45	0.097 0.059 0.015 0.0061 0.0028	n.r.	
China, Fujian 2008 (n.r.)	WG	1	0.15	n.r.	n.r.	straw	14 21	0.0059 0.0023	n.r.	Wu (2009, PYMET_050)
		2	0.15	n.r.	n.r.	straw	14 21	0.01 0.0038	n.r.	
		1	0.23	n.r.	n.r.	straw	14 21	0.0091 0.0013	n.r.	
		2	0.23	n.r.	n.r.	straw	14 21	0.0013 < 0.001	n.r.	
		1	0.23	0.05	n.r.	stalks	0 1 3 5 7 10 14 28 35 45	0.16 0.15 0.095 0.055 0.051 0.017 0.0096 0.0056 0.0019 0.001	n.r.	
China, Zhejiang 2007 (n.r.)	WG	1	0.15	n.r.	n.r.	straw	14 21	0.0024 < 0.001	n.r.	Wu (2009, PYMET_050)
		2	0.15	n.r.	n.r.	straw	14 21	0.0063 0.0029	n.r.	
		1	0.23	n.r.	n.r.	straw	14 21	0.0052 0.0026	n.r.	
		2	0.23	n.r.	n.r.	straw	14 21	0.01 0.0048	n.r.	
		1	0.23	0.05	n.r.	stalks	0 1 3 7 10 14 21 28	0.18 0.15 0.096 0.057 0.033 0.011 0.0062 0.0051	n.r.	

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
							35 45	0.0027 0.0019		
China, Zhejiang 2008 (n.r.)	WG	1	0.15	n.r.	n.r.	straw	14 21	0.0048 < 0.001	n.r.	Wu (2009, PYMET_050)
		2	0.15	n.r.	n.r.	straw	14 21	0.0061 < 0.001	n.r.	
		1	0.23	n.r.	n.r.	straw	14 21	0.0089 < 0.001	n.r.	
		2	0.23	n.r.	n.r.	straw	14 21	0.0011 < 0.001	n.r.	
		1	0.23	0.05	n.r.	stalks	0 1 3 5 7 14 21 28 35 45	0.11 0.058 0.049 0.021 0.0083 0.0027 0.0015 0.0018 < 0.001 < 0.001	n.r.	
China, Guangxi 2009 (n.r.)	WG	1	0.15	n.r.	n.r.	straw	14 21 28	0.016 0.0016 0.0076	n.r.	Wu (2009, PYMET_050)
		2	0.15	n.r.	n.r.	straw	14 21 28	0.0032 0.0017 < 0.001	n.r.	
		1	0.23	n.r.	n.r.	straw	14 21 28	0.0058 0.0024 0.001	n.r.	
		2	0.23	n.r.	n.r.	straw	14 21 28	0.0027 0.0029 0.0019	n.r.	
		1	0.23	0.05	n.r.	stalks	0 1 3 5 7 10 14 21 28	0.12 0.095 0.082 0.014 0.0062 0.0032 0.0024 0.0019 0.0011	n.r.	
China, Hunan 2009 (n.r.)	WG	1	0.15	n.r.	n.r.	straw	14 21 28	0.041 0.0021 < 0.001	n.r.	Wu (2009, PYMET_050)
		2	0.15	n.r.	n.r.	straw	14 21 28	0.033 0.0097 < 0.001	n.r.	
		1	0.23	n.r.	n.r.	straw	14	0.15	n.r.	

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
							21 28	0.03 0.01		
		2	0.23	n.r.	n.r.	straw	14 21 28	0.056 0.022 0.021	n.r.	
		1	0.23	0.05	n.r.	stalks	0 1 3 5 7 10 14 21	0.097 0.07 0.048 0.031 0.0054 0.0034 0.0016 < 0.001	n.r.	

n.r. = Not reported

DAT = Days after last treatment

Cotton seed hulls

Table 108 Residues of pymetrozine following foliar application in cotton hulls

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Spain, Coria del Rio 1997 (Vulcano)	WG	3	0.2 0.18 0.21	0.2 0.24 0.22	75 76 79	hulls	35 50	< 0.02 < 0.02	9	1162/97 Tribolet (1998, PYMET_164)
Greece, Roumeli 1999 (506 Stoneville)	WP	3	0.2 0.2 0.2	0.04 0.04 0.04	79 81 83	hulls	0 7 14 21 28 35	0.16 0.05 < 0.02 < 0.02 < 0.02 0.02	13	1099/99 Tribolet (2001, PYMET_165)
	WG	3	0.2 0.2 0.2	0.04 0.04 0.04	79 81 83	hulls	0 7 14 21 28 35	0.04 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	13	
Greece, Kato Glines 1999 (453 Stoneville)	WP	3	0.2 0.2 0.2	0.04 0.04 0.04	77 79 83	hulls	0 7 14 21 28 35	0.12 < 0.02 < 0.02 < 0.02 < 0.02 0.02	12	1100/99 Tribolet (2001, PYMET_166)
	WG	3	0.2 0.2 0.2	0.04 0.04 0.04	77 79 83	hulls	0 7 14 21 28 35	0.08 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	12	

Location,	Application					Residues, mg/kg			Storage interval	Trial No.,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	pymetrozine	months	Reference
Greece, Kamaroula 1999 (453 Stoneville)	WP	3	0.2 0.2 0.2	0.04 0.04 0.04	69 73 80	hulls	35	< 0.02	13	1101/99 Tribolet (2001, PYMET_167)
	WG	3	0.2 0.2 0.2	0.04 0.04 0.04	69 73 80	hulls	35	< 0.02	13	
Greece, Mavroglia 1999 (506 Stoneville)	WP	3	0.2 0.2 0.2	0.04 0.04 0.04	69 73 80	hulls	35	< 0.02	15	1102/99 Tribolet (2001, PYMET_168)
	WG	3	0.2 0.2 0.2	0.04 0.04 0.04	69 73 80	hulls	35	< 0.02	15	
Greece, Ipodromos 1999 (453 Stoneville)	WG	3	0.2 0.2 0.2	0.04 0.04 0.04	69 73 80	hulls	35	< 0.02	13	1103/99 Tribolet (2001, PYMET_169)

DAT = Days after last treatment

BBCH 69 = End of flowering

BBCH 71–79 = 10–90% of bolls have attained their final size

BBCH 81–89 = 10–90% of bolls open

FATE OF RESIDUES IN STORAGE AND PROCESSING

Nature of residue during processing

The hydrolysis of pymetrozine under processing conditions was investigated by Morgenroth (1999, PYMET_170). [Pyridine-5-¹⁴C]pymetrozine was incubated in aqueous buffer solutions at a nominal concentration of 5 mg/L under three sets of conditions, each designed to simulate an appropriate process: 90 °C (pH 4, 20 minutes) to simulate pasteurisation, 100 °C (pH 5, 60 minutes), to simulate boiling, baking and brewing, and 120 °C (pH 6, 20 minutes) to simulate sterilisation.

Total recovered radioactivity was measured for each test solution. Radioactive components were characterised by fractionation and co-chromatography with authenticated reference compounds using HPLC, TLC and identities were confirmed by HPLC/MS.

Table 109 Hydrolysis of pymetrozine under simulated processing conditions

Compound	Concentration (% of applied radioactivity)								
	pH 4, 90 °C, 20 min (pasteurisation)			pH 5, 100 °C, 60 min (boiling, baking, brewing)			pH 6, 120 °C, 120 min (sterilisation)		
	1	2	Mean	1	2	Mean	1	2	Mean
Pymetrozine	66.4	66.9	66.6	57.6	57.7	57.6	93.3	93.2	93.2
CGA300407	33.6	32.4	33.0	42.3	41.8	42.1	6.1	6.0	6.0
Total	100	99.3	99.6	99.9	99.5	99.7	99.4	99.2	99.2

Residues after processing

The fate of pymetrozine during processing of raw agricultural commodity (RAC) was investigated in tomatoes and sweet peppers using important processing procedures. As a measure of the transfer of residues into processed products, a processing factor was used, which is defined as:

$$\text{Processing factor (Pf)} = \frac{\text{Residue in processed product (mg/kg)}}{\text{Residue in raw agricultural commodity (mg/kg)}}$$

If residues in the RAC were below the LOQ, no processing factor could be derived. In case of residues below the LOQ, but above the LOD in the processed product, the numeric value of the LOQ was used for the calculation. If residues in the processed product were below the LOD, the numeric value of the LOQ was used for the calculation but the PF was expressed as “less than” (e.g. < 0.5).

All samples were analysed within the maximum storage interval identified for tomatoes (6 months) and peppers (one month).

A summary of all processing factors for pymetrozine relevant for the estimation of maximum residue levels of the dietary intake is given in Table 113.

Tomato

A study on the behaviour of pymetrozine during processing of tomatoes was conducted by Gasser (2002, PYMET_171). Protected tomatoes grown in Switzerland were treated three times with 0.9 kg pymetrozine/ha. Samples were harvested 3 days after the last application. Tomatoes were used for the production of washed tomatoes, tomato juice, tomato puree and canned tomatoes. One full mass balance study was conducted to determine the accountability of the residue, and two follow-up studies were conducted to determine residue transfer into the processed commodities.

- The washed tomatoes were produced by washing for 2 minutes in cold running water.
- Tomato juice was produced by quartering and blanching the washed tomatoes followed by sieving to remove the peel and seeds (wet pomace). The raw juice was pasteurised (20 minutes at 99 °C).
- Tomato puree was produced by concentrating raw juice to approximately 30% dry matter and then pasteurising (20 minutes at 93–95 °C).
- Canned tomatoes were produced by blanching washed tomatoes to remove the peel. The peeled tomatoes and portion of tomato juice from the juicing process were then sterilised in tins.

Table 110 Summary of pymetrozine residues in tomato and processed commodities from a trial conducted in Switzerland

Commodity	Residues (mg/kg)			Processing factors (mean)
	Balance 1	Follow-up 1	Follow-up 2	
Tomato juice production				
Unprocessed fruit	0.58	–	–	–
Washed fruit	0.08	–	–	0.13
Washing water	1.0	–	–	1.7
Blanched fruit	0.03	–	–	0.06
Wet pomace	0.05	–	–	0.08
Raw juice	0.03	–	–	0.05
Pasteurised juice	< 0.02	< 0.02	< 0.02	< 0.03, < 0.03, < 0.03 (< 0.03)
Tomato puree production				
Unprocessed fruit	0.58	–	–	–

Puree pre-pasteurisation	< 0.02	–	–	< 0.03
Puree after pasteurisation	< 0.02	< 0.02	< 0.02	< 0.03, < 0.03, < 0.03 (< 0.03)
Canned tomato production				
Unprocessed fruit	0.58	–	–	–
Peeled fruit	< 0.02	–	–	< 0.03
Peels	0.06	–	–	0.1
Waste water	< 0.02	–	–	< 0.03
Sterilised canned fruit	< 0.02	< 0.02	< 0.02	< 0.03, < 0.03, < 0.03 (< 0.03)

A total of three additional follow-up studies on tomatoes were conducted, two by Joseph (1996, PYMET_172 ,PYMET_173, PYEMT_174 & PYMET_175) in the USA and one by Tribolet (1999, PYMET_176) in Switzerland.

In the US trials tomatoes were processed according to common commercial practice. A flow-chart of all processing steps involved is presented in Figures 6 and 7. Samples of pomace (wet, dry), puree, juice and canned tomatoes were analysed for residues of pymetrozine and GS23199.

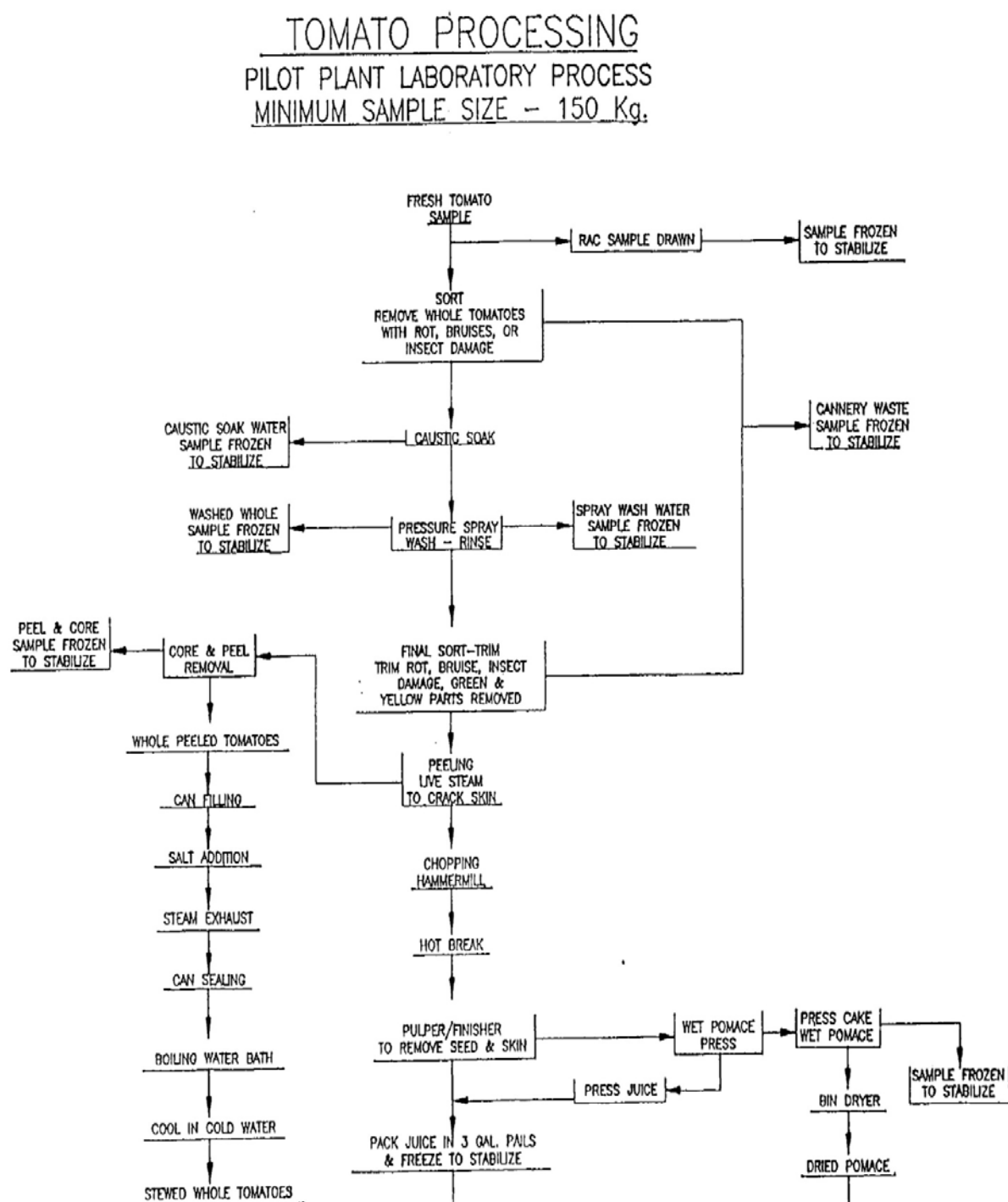


Figure 6 Processing of tomatoes—Part 1

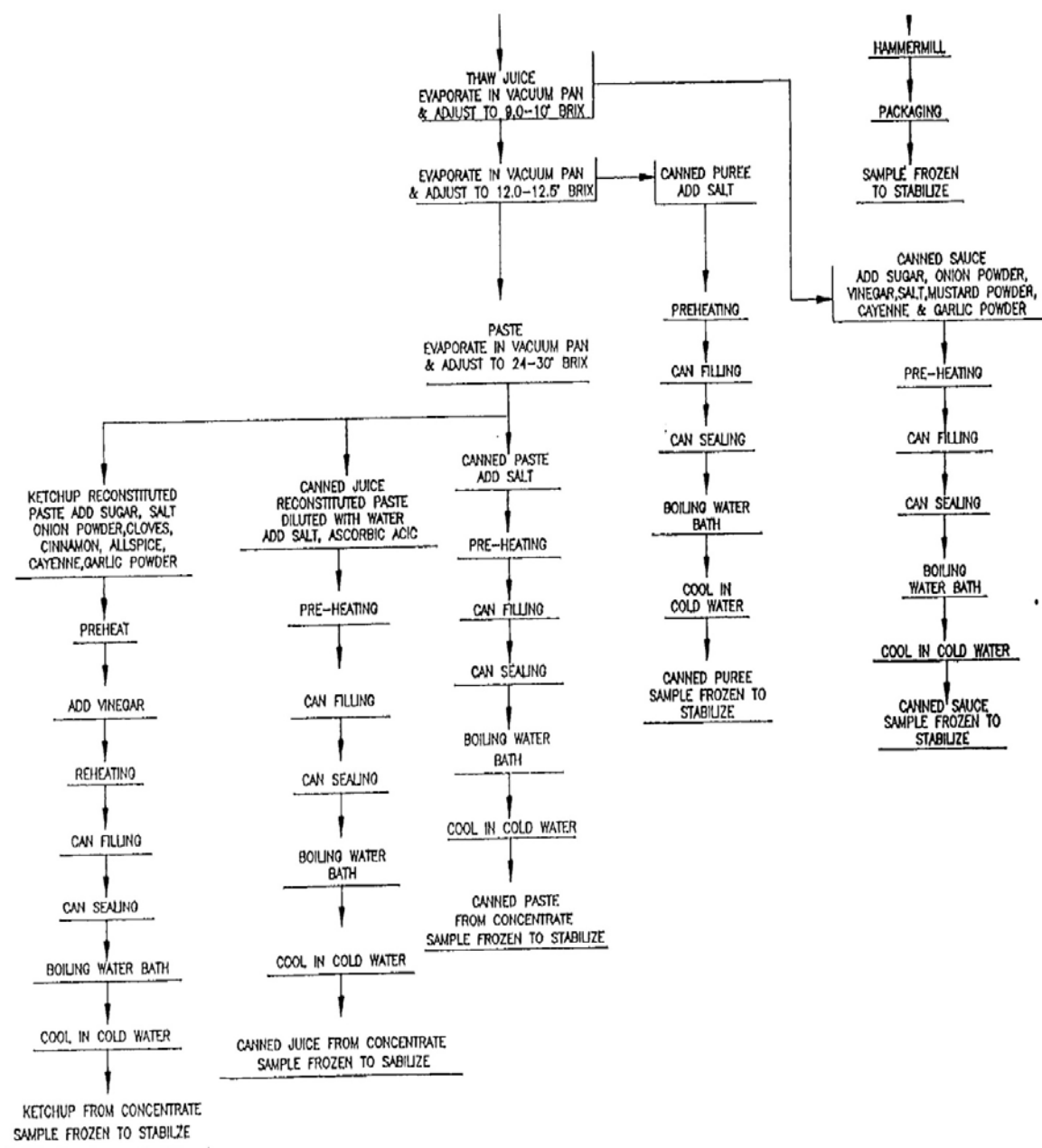


Figure 7 Processing of tomatoes—Part 2

In the follow-up study in Switzerland conducted by Tribolet field tomatoes were sprayed three times with application rates of 0.45 kg ai/ha each. The tomatoes were processed into washed fruits, peeled fruits, preserves, paste and juice on laboratory scale.

For peeled tomatoes the peel was removed with a knife after heating in lukewarm water.

For juicing, 10% water were added and the mixture was boiled for 2–3 minutes. After passing through a strainer the raw juice was collected. The juice was pasteurised before canning.

Tomato paste was also prepared by adding 10% water and boiling for 2–3 min. After passing through a strainer, the juice was concentrated to 27% dry weight, filled into cans and pasteurized.

For tomato preserves peeled fruits (see above) were filled into cans and approximately the same amount of tomato juice was added. The cans were closed and pasteurized at 94 °C.

A summary of all residues and the resulting processing factors for processed tomatoes is presented below:

Table 111 Summary of follow-up studies involving processing of tomatoes

Location, year (variety)	Application	Sample	pymetrozine in mg/kg	processing factor	GS23199 in mg/kg	processing factor	Trial No., Reference
USA, Fayette (OH) 1994 (Heinz 9036)	outdoor foliar spray: 2 × 0.59 kg ai/ha, 7 d interval, DAT: 13	fruit (RAC)	< 0.02	–	< 0.02	–	NE-IR-206-94 Joseph (1996, PYMET_172 & PYMET_173)
		pomace,	< 0.02	–	< 0.02	–	
		wet	< 0.02	–	< 0.02	–	
		pomace,	< 0.02	–	0.02	–	
		dry	< 0.02	–	< 0.02	–	
		puree	< 0.02	–	< 0.02	–	
	outdoor foliar spray: 2 × 0.99 kg ai/ha, 7 d interval, DAT: 13	fruit (RAC)	< 0.02	–	< 0.02	–	
		pomace,	< 0.02	–	< 0.02	–	
		wet	< 0.02	–	0.05	–	
		pomace,	< 0.02	–	0.03	–	
		dry	< 0.02	–	0.04	–	
		puree	< 0.02	–	0.03	–	
USA, Fresno (CA) 1994 (UC 82-L-Petoseed)	outdoor foliar spray: 2 × 0.59 kg ai/ha, 7 d interval, DAT: 14	fruit (RAC)	0.03	< 0.66	< 0.02	–	02-IR-017-94 Joseph (1996, PYMET_174 & PYMET_175)
		pomace,	< 0.02	< 0.66	0.02	–	
		wet	< 0.02	< 0.66	0.07	–	
		pomace,	< 0.02	< 0.66	0.12	–	
		dry	< 0.02	< 0.66	0.1	–	
		puree	< 0.02	< 0.66	0.08	–	
	outdoor foliar spray: 2 × 0.99 kg ai/ha, 7 d interval, DAT: 14	fruit (RAC)	0.05	–	0.04	–	
		pomace,	< 0.02	< 0.4	0.04	1	
		wet	< 0.02	< 0.4	0.19	4.8	
		pomace,	< 0.02	< 0.4	0.2	5	
		dry	< 0.02	< 0.4	0.28	7	
		puree	< 0.02	< 0.4	0.38	9.5	
Switzerland, Fully 1998 (Paola)	outdoor foliar spray: 3 × 0.45 kg ai/ha, 7 d interval, DAT: 3	fruit (RAC)	0.06	–	< 0.02	–	1085/98 Tribolet (1999, PYMET_176)
		fruit, peeled	0.0083	0.14	< 0.02	–	
		preserves	0.0042	0.07	< 0.02	–	
		paste	< 0.0025	< 0.04	< 0.02	–	
		juice	< 0.004	< 0.01	< 0.005	–	

DAT = Days after last treatment

Sweet pepper

The influence of processing to pymetrozine residues in sweet peppers was investigated by Gasser (2003, PYMET_177). Protected peppers grown in Switzerland were treated three times with 0.54 kg pymetrozine/ha. Samples were harvested 3 days after the last application. Peppers were used for the production of washed fruit, cut fruit and cooked fruit.

The washed peppers were produced by washing for 2 minutes in a vessel using cold running water.

Cooked fruit was produced by removing seed and the inner waste portion from the washed peppers, quartering the fruit and cooking in water for 15 minutes at 98–100 °C.

In the following table the residues found and the corresponding processing factors are summarised:

Table 112 Processing of sweet peppers following application of 3×0.45 kg pymetrozine/ha

Commodity	Residues (mg/kg)			
	Balance A	Balance B	Follow-up 1	Follow-up 2
Unprocessed fruit	0.76	0.76		
Washed fruit	0.24	< 0.02		
Washing water	0.46	0.56		
Cut fruit	0.02	< 0.02		
Waste fruit (seeds and inners)	0.03	0.02		
Cooked fruit	< 0.02	< 0.02	< 0.02	< 0.02
Cooking water	< 0.02	< 0.02		

Table 113 Overview of processing factors for pymetrozine-derived residues

Raw commodity	Processed commodity	Pymetrozine		GS23199	
		Individual processing factors	Mean or best estimate processing factor	Individual processing factors	Median or best estimate processing factor
Tomato	Juice, raw	<u>0.05</u>	0.05	–	–
	Juice, pasteurized	< 0.01, < <u>0.03</u> (4), < 0.4	< 0.03	9.5	9.5
	Puree	< <u>0.03</u> (4), < 0.4	< 0.03	5	5
	Paste	< <u>0.04</u> , < <u>0.4</u>	< 0.22	7	7
	Peeled fruits	< <u>0.02</u> , <u>0.14</u>	0.08	–	–
	Canned/preserve	< <u>0.03</u> (4), 0.07	0.03	–	–
	pomace, wet	<u>0.08</u> , < <u>0.4</u>	0.24	1	1
Sweet pepper	Cooked fruit	< 0.02(3)	< 0.02	–	–

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

For the estimation of residues of pymetrozine and its metabolite CGA313124 in animal matrices a lactating cow feeding study was submitted to the Meeting.

Lactating cows

Residues in lactating cows were investigated by Joseph (1997, PYMET_178, PYMET_179 & PYMET_180). Eleven lactating Holstein dairy cows (*Bos taurus*; three cows/treatment group, one control cow) were dosed orally, via capsule, for 28 consecutive days with pymetrozine with either 0 ppm (control, 0×), 1 ppm (1× dose group), 3 ppm (3× dose group), or 10 ppm (10× dose group).

Milk was collected twice daily. On day 29 after the administration of the first dose, the animals were sacrificed and liver, kidney, round and tenderloin muscle and omental and perirenal fat were collected for analysis. For the depuration group (10×), milk for analysis was additionally sampled during the depuration phase. The animals were sacrificed approximately 20–24 h after the final administration.

Tissue and milk samples were analysed for pymetrozine and CGA313124 residue by methods AG-644 (LOQ: 0.01 mg/kg) and AG-658 (LOQ: 0.01 mg/kg) described in the analytical methods section. The samples were stored deep-frozen before analysed for 10–12 months.

In the following table the residues of pymetrozine and CGA313124 in milk and tissues are summarized. In the control animal no detectable residues of pymetrozine or CGA313124 were found:

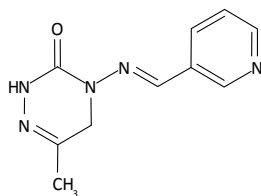
Table 114 Residues of pymetrozine and CGA313124 in cow tissues and milk following administration of pymetrozine at 1, 3 and 10 ppm in the diet

Commodity	Sampling Interval (days)	Maximum Pymetrozine Residues (mg/kg)			Maximum CGA313124 Residues ^a (mg/kg)		
		Group 2 (1.03 ppm)	Group 3 (2.97 ppm)	Group 4 (9.95 ppm)	Group 2 (1.03 ppm)	Group 3 (2.97 ppm)	Group 4 (9.95 ppm)
Milk	0	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
	1	< 0.01	< 0.01	< 0.01	< 0.01	0.01	0.05
	3	< 0.01	< 0.01	< 0.01	< 0.01	0.02	0.05
	7	< 0.01	< 0.01	< 0.01	< 0.01	0.01	0.03
	14	< 0.01	< 0.01	< 0.01	< 0.01	0.01	0.04
	21	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.04
	26	< 0.01	< 0.01	< 0.01	< 0.01	0.01	0.03
Liver	28–30	–	–	< 0.01	–	–	< 0.01
Perirenal fat	28–30	–	–	< 0.01	–	–	< 0.01
Omental fat	28–30	–	–	< 0.01	–	–	< 0.01
Round muscle	28–30	–	–	< 0.01	–	–	< 0.01
Tenderloin muscle	28–30	–	–	< 0.01	–	–	< 0.01
Kidney	28–30	–	–	< 0.01	–	–	< 0.01

^a Including its phosphate conjugate

APPRAISAL

Pymetrozine is a pyridine azomethine insecticide used to control homopteran insects (aphids and whiteflies) as well as pollen beetle selectively. Although it has no knockdown effect, pymetrozine rapidly affects the feeding behaviour of the insect pests. It was considered for the first time by the 2014 JMPR for toxicology and residues.

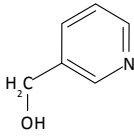
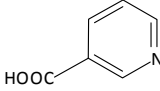
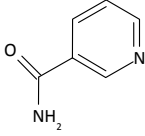
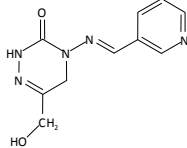
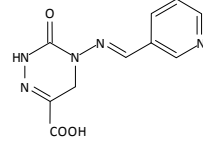
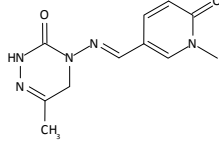
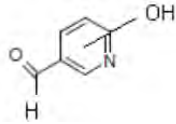
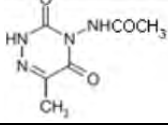
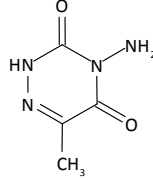
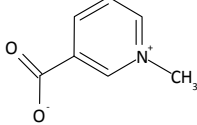


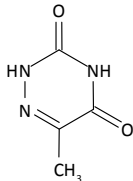
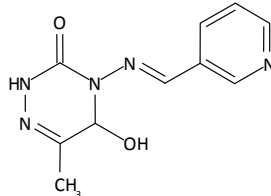
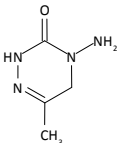
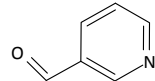
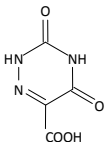
The IUPAC name of pymetrozine is (E)-4,5-dihydro-6-methyl-4-(3-pyridylmethyleneamino)-1,2,4-triazin-3(2H)-one.

The pymetrozine molecule contains a double bond about which E/Z isomerism is possible. However, pymetrozine technical material is manufactured by a process that yields almost exclusively the E isomer.

Pymetrozine labelled either in the pyridine- or triazine-moiety was used in the metabolism and environmental fate studies.

The following abbreviations are used for the metabolites discussed below:

CGA128632	3-pyridinemethanol	
CGA180777 (nicotinic acid)	3-pyridinecarboxylic acid	
CGA180778 (nicotinamide)	3-pyridinecarboxamide	
CGA313124	4,5-dihydro-6-hydroxymethyl-4-[(3-pyridinyl-methylene)amino]-1,2,4-triazine-3(2H)-one	
U5/I _{A2}	4,5-dihydro-6-carboxy-4-[(3-pyridinyl methylene)-amino]-1,2,4-triazine-3(2H)-one	
I _{A7}	4,5-dihydro-6-methyl-4-[(3-(1-methyl-6-oxo-1,6-dihydropyridinyl)methylene)-amino]-1,2,4-triazine-3(2H)-one	
IA17	hydroxylated 3-pyridinecarboxaldehyde	
CGA259168	N-(4,5-dihydro-6-methyl-3,5-dioxo-1,2,4-triazine-4(2H)-yl)-acetamide	
CGA294849	4-amino-6-methyl-1,2,4-triazine-3,5(2H,4H)-dione	
CGA96956 (trigonelline)	1-methyl-3-pyridinecarboxylic acid	

GS23199	6-methyl-1,2,4-triazine-3,5(2H,4H)-dione	
CGA359009	4,5-dihydro-5-hydroxy-6-methyl-4-[(3-pyridinylmethylene)-amino]-1,2,4-triazine-3(2H)-one	
CGA215525	4-amino-6-methyl-1,2,4-triazine-3(2H)-one	
CGA300407 (nicotinealdehyde)	3-pyridinecarboxaldehyde	
CGA266591	2,3,4,5-tetrahydro-3,5-dioxo-1,2,4-triazine-6-carboxylic acid	

Animal metabolism

Information was available on metabolism of pymetrozine in laboratory animals, lactating goats and laying hens.

In the rat the extent of oral absorption is high (> 80%), based on urinary and biliary data. Pymetrozine is widely distributed in the body. High concentrations of both triazine- and pyridine-labelled material were found in the liver and kidney. The labelled material was rapidly excreted via urine (50–75% in 24 hours). Absorbed pymetrozine was extensively metabolized, with unmetabolized parent compound representing approximately 10% of the excreted radiolabel. Compounds containing both ring structures represented over 50% of the identified metabolites. The kinetics, excretion pattern, tissue distribution of radioactivity and metabolite profile were similar for both radiolabelled sites and administered dose levels as well as when the administration of radiolabelled pymetrozine was preceded by 14 days of administration of the unlabelled material (see WHO Monograph).

One study on the metabolism in lactating goats was available for each of the labels. Over four consecutive days the goats received daily doses of radiolabelled pyridine-¹⁴C- or triazine-¹⁴C-pymetrozine at rates equivalent to 7.5 ppm (0.39 mg/kg bw) or 10 ppm (0.54 mg/kg bw) in the diet, respectively. In both studies approximately 5-6% of the total dose was recovered from milk or tissues of the animals. Most of the administered radioactivity was recovered in faeces (15–17%) and urine (47–52%).

Following application of pyridine-¹⁴C-pymetrozine highest TRR levels were found in liver (1.5 mg eq/kg), kidney (0.72 mg eq/kg) and milk (0.33 mg eq/kg). Into muscle (0.068 mg eq/kg) and fat (0.027 mg eq/kg) the overall transfer of radioactivity was minor.

Parent pymetrozine was identified in all tissues and milk, however only in muscle (11% TRR) the contribution to the TRR was more than 10 %. In tissues nicotinamide (CGA180778), which is

formed by cleavage of the parent substance, was the major residue representing 44% TRR in muscle, 24% TRR in fat, 37% TRR in liver and 27% TRR in kidney.

In most tissues CGA313124 (pymetrozine-hydroxy) was also a major metabolite identified with levels of 10% TRR in muscle and 11% TRR in kidney. In milk, CGA313124 (36% TRR) and its phosphate conjugate (39% TRR) were the only major metabolites identified. In addition, I_{A17} was present in liver at 10% of the TRR.

For the triazine-¹⁴C-label again highest TRR levels were found in liver (1.1 mg eq/kg), kidney (0.57 mg eq/kg) and milk (0.45 mg eq/kg). Muscle (0.047 mg eq/kg) and fat (0.098 mg eq/kg) contained lower residues.

Unchanged pymetrozine was again found in all tissues and milk, but only in muscle as a major residue representing 10% of the TRR. In tissues CGA313124 was the dominant residue found in levels of 9.5% TRR in muscle, 25% TRR in fat, 5% TRR in liver and 15% TRR in kidney. In kidney 5U/I_{A2} was another major metabolite present at 12% of the TRR. Several triazine-based cleavage products were identified, however at individual levels below 10% TRR each.

In milk, again CGA313124 (40% TRR) and its phosphate conjugate (41% TRR) were the only major metabolites identified.

For laying hens groups of hens received daily doses of [pyridine-¹⁴C]-pymetrozine or [triazine-¹⁴C]-pymetrozine at rates equivalent to approximately 10 ppm for four consecutive days (0.79 mg/kg bw). The animals were sacrificed ca. 6 hours after the last dose. Approximately 0.4–1.3% of the total dose in both studies was recovered from eggs or tissues of the animals. Most of the radioactivity administered was found in the excreta (76–81% AR). Total radioactive residues were 0.006–0.016 mg eq/kg in eggs, 0.021–0.043 mg eq/kg in muscle, 0.019–0.024 mg eq/kg in fat, 0.16–0.54 mg eq/kg in kidney and 0.11–0.99 mg eq/kg in liver. For both labels parent pymetrozine was found in very low levels, not exceeding 5% of the TRR. In meat, fat and eggs yolk it was below the LOQ of the method or was undetected.

Following administration of the pyridine-¹⁴C-label, nicotinamide (CGA180778) was the major residue in meat (77% TRR), skin + fat (63% TRR), egg white (28% TRR) and liver (70% TRR). In kidney, nicotinic acid (CGA180777) was the major residue, representing 65% of the TRR. Further major metabolites identified were I_{A7}, present at levels of 17% TRR in skin + fat, 15% TRR in egg white and 13% TRR in kidney as well as CGA300407 with 11% TRR in egg white.

For the triazine-¹⁴C-label CGA259168 was the major residue in meat (39% TRR), skin + fat (24% TRR), egg white (45% TRR), egg yolk (13% TRR) and kidney (11% TRR). I_{A7} was mainly found in kidney (49% TRR), followed by liver (27% TRR), skin + fat (22% TRR) and egg white (11% TRR). The only other major metabolite found was CGA294849 present in meat at levels of 11% of the TRR.

In summary pymetrozine is quickly degraded in goats and hens. A large quantity of the parent molecule is cleaved, resulting in formation of nicotinic acid (up to 65% TRR) and nicotinamide (up to 70% TRR) for the pyridine-moiety and several metabolites for the triazine-moiety. Another important metabolic step is the oxidation of the parent to CGA313124, which is found in most tissues and mainly in milk, including its phosphate conjugate (up to 40% and 41% TRR, respectively).

In goats, I_{A17} represented up to 10% TRR in the liver. In hen tissues, pymetrozine is also metabolised into I_{A7} (13–41% TRR in fat, egg white and kidney). Both metabolites were not identified in the rat.

Plant metabolism

The Meeting received plant metabolism studies for pymetrozine following foliar application of either [pyridine-¹⁴C]-pymetrozine or [triazine-¹⁴C]-pymetrozine to tomatoes (protected), potatoes, paddy rice and cotton (protected). For paddy rice, the metabolism following granular application was also investigated.

Following foliar application of [pyridine-¹⁴C]-pymetrozine to tomatoes at rates of 2×0.25 kg ai/ha, total radioactive residues in the fruits declined from 1 mg eq/kg (1h) to 0.23 mg eq/kg (7 days) and finally to 0.016 mg eq/kg 27 days after the last application. In the leaves corresponding TRR levels were 14 mg eq/kg (1 hour), 7.4 mg eq/kg (7 days) and 2.4 mg eq/kg (27 days).

In the samples collected directly after treatment (1 hour), pymetrozine located in the fruit and leaf surface was the dominant residue, representing 78% TRR and 60% TRR, respectively.

After 15 days, the pymetrozine surface residues declined to 5.9% TRR in the fruits and 16% TRR in the leaves. Most of the radioactivity was recovered in the extracts. In the fruits, trigonelline (CGA96956) was the only major residue representing 70% of the TRR. In leaves, trigonelline (26% TRR) and sugar conjugates of CGA128632 (23% TRR) were major residues besides the parent.

[Triazine-¹⁴C]-pymetrozine was applied to tomato plants with three foliar application with 0.47 kg ai/ha each. In the fruits collected (3, 7 and 14 days after treatment) TRR levels were 0.51–0.58 mg eq/kg. In the leaves TRR levels declined from 28 mg eq/kg (3 d) to 22 mg eq/kg (7 days) and finally to 17 mg eq/kg (14 days).

In all samples unchanged pymetrozine was the major residues, representing 32–57% TRR in the fruits and 31–50% TRR in the leaves. The only other major metabolite identified was CGA294849 present at levels of 14% TRR in fruits collected 14 days after harvest.

In the first study investigating the metabolism of pymetrozine in potatoes either [pyridine-¹⁴C]- or [triazine-¹⁴C]-pymetrozine were applied to potato plants with three foliar sprayings at rates of 0.15 kg ai/ha (low-dose) or 1.05 kg ai/ha (high-dose) each 61 days after planting. In the foliage samples collected after 7, 14 and 19 days TRR levels were 6.4–11.7 mg eq/kg following low-dose and 29–46 mg eq/kg following high-dose treatment. In the tubers TRR levels amounted 0.11–0.36 mg eq/kg (low-dose) and 0.34–1.1 mg eq/kg (high-dose).

For the [pyridine-¹⁴C]-label, trigonelline (CGA96956) was the major residue in tubers (7, 14 and 29 days after last treatment) representing 54–75% of the TRR. In addition, nicotinic acid (CGA180777) and its glycoside represented major residues present up to 22% of the TRR. Unchanged parent pymetrozine was detected in all samples, however its levels did not exceed 2.2% of the TRR. No further major metabolites were identified in the tubers.

In the foliage, unchanged pymetrozine was present at higher levels of up to 18% TRR in day 7 samples. The only major metabolite present was CGA128632 and its glycoside, representing 1.1–3.1% TRR and 9.5–18% TRR, respectively. The major part of the radioactivity in the foliage remained unextracted (54–70% TRR).

For the [triazine-¹⁴C]-label, neither parent (0.2–4.9% TRR) nor metabolites (up to 5.7% TRR) represented major residues in tubers. Most of the radioactivity was distributed into multiple minor fractions too low for identification. The foliage showed GS23199 (1.4–2.4% TRR) and its glycoside (7.6–16% TRR) as major residues. Parent pymetrozine was found in lower amounts of 6.3–10% of the TRR. Again, the amount of unextracted radioactivity in the foliage was high, representing 37–45% of the TRR.

In two additional studies either [pyridine-¹⁴C]- or [triazine-¹⁴C]-pymetrozine were applied to potato plants with two foliar sprayings at rates of 0.2 kg ai/ha each. In the leaves, residues declined from 9.5–11 mg eq/kg (1h after treatment) to 1.3–1.8 mg eq/kg (55 days, final harvest). Special investigation of radioactive residues in new leaves above or below the treated plant parts reveals approximately 3–4 times higher residues on the upper leaves than in the lower leaves. In tuber collected 55 days after the final application, TRR levels were 0.051–0.072 mg eq/kg.

The identification of the radioactivity in the tubers showed no pymetrozine above the LOQ of 0.001 mg eq/kg. The only major residues identified were nicotinic acid (CGA180777) and its glycoside for the pyridine-label (22 and 29% TRR, respectively) and GS23199 (11% TRR) and its glycoside (1.7% TRR) for the triazine-label.

In the foliage pymetrozine was the dominant residue directly after treatment (42–58% TRR). In samples collected after longer intervals, the radioactivity was distributed into several minor fractions mostly too low for identification. The only identified major metabolite was conjugated CGA128632 (up to 28% TRR) for the pyridine-label. Unextracted radioactivity in potato foliage averaged at 36% TRR for both labels.

Paddy rice was treated with either [pyridine-¹⁴C]- or [triazine-¹⁴C]-pymetrozine with one foliar treatment of 0.25 kg ai/ha 45 days before harvest. TRR levels found were 1.7–2.1 mg eq/kg in foliage (19 days after treatment), 5.3–6.3 mg eq/kg in straw and 0.14–0.24 mg eq/kg in grain (both samples 45 days after treatment).

The identification of the radioactivity in foliage and straw showed unchanged pymetrozine as the only major residue, representing 86–89 % TRR and 63–74% TRR, respectively.

In the grain no metabolites were identified exceeding 10% TRR. The parent substance was detected in all samples, however its levels were low (0.8–2.3% TRR). In the grain 63–86% of the TRR remained unextracted.

In addition paddy rice was treated with granules of [pyridine-¹⁴C]- or [triazine-¹⁴C]-pymetrozine at rates corresponding to 0.6 kg ai/ha to seedling boxes. Foliage was collected 1, 41 and 69 days later, grain husks and straw at maturity after 116 days.

Total radioactive residues were highest in foliage directly after treatment with 33–42 mg eq/kg and declined to 0.72–0.82 mg eq/kg after 69 days. At harvest dry straw contained TRR levels of 2.6 mg eq/kg. In the grain the radioactivity was much lower, representing 0.21–0.52 mg eq/kg.

In the foliage parent pymetrozine was the major residue directly after treatment (1 day, 38–60% TRR), but quickly declined to < 10% TRR in all other samples collected after a longer interval. In grain, pymetrozine was not found above the LOQ of 0.001 mg eq/kg (0.2% TRR).

Major metabolites identified in foliage and straw after application of pyridine-¹⁴C-pymetrozine were trigonelline (CGA96956) with 9.5–28% TRR and free and conjugated nicotinic acid (CGA180777) with a total of 17–26% TRR. For the triazine-¹⁴C-label no metabolites exceeding 10% of the TRR were identified except for CGA359009 (15% TRR) and GS23199 (17% TRR) in foliage samples one day after treatment.

Grain contained very few identified metabolites. The only major metabolites were trigonelline (CGA96956, 11% TRR) and free and conjugated nicotinic acid (CGA180777, 26% TRR). Most of the radioactivity in grain remained unextracted (56–86% TRR).

The metabolism of pymetrozine in cotton was investigated by application of either [pyridine-¹⁴C]- or [triazine-¹⁴C]-labelled active substance under glasshouse conditions. The plants were treated with two foliar spraying at rates equivalent to 0.2 kg ai/ha each. After 93 days samples of treated leaves (0.6–5.9 mg eq/kg), new grown leaves (0.03–0.2 mg eq/kg), stem (1.6–1.7 mg eq/kg), hulls (2.7–4.8 mg eq/kg) fibres (0.065–0.17 mg eq/kg) and seeds (0.043–0.21 mg eq/kg) were collected.

For the [pyridine-¹⁴C]-label unchanged pymetrozine was the major residue in all green plant parts, hulls and fibres, representing 28–83% of the TRR. The only metabolite exceeding 10% of the TRR in the plant was trigonelline (CGA96956, 0.6–23% TRR). In the seeds pymetrozine was detected at levels of 7.4–9% of the TRR. Most of the parent substance (35–58%) was found in cotton oil extracted from the seeds. The major residues in the seeds was trigonelline (CGA96956), representing 50% of the TRR.

For the [triazine-¹⁴C]-label only pymetrozine was present in major amount in the plant (28–66% TRR). Minor residues identified were CGA294849 and GS23199 (< 4% TRR each). In the seeds pymetrozine was present at TRR levels of 7.4%. On processing of seeds, 35% of the parent substance was recovered in the oil.

In summary pymetrozine is deposited on the plant surface and more or less quickly adsorbed. Unchanged parent represented 7.4–75% of the TRR in treated parts. In the plant tissue, the active substance is quickly degraded by cleavage, forming nicotinic acid, nicotinamide or trigonelline from

the pyridine-moiety. Additional major metabolites found were CGA359009 (rice: 15% TRR), GS23199 including conjugates (potato tuber: 13% TRR; potato foliage: 18% TRR; rice: 17% TRR) and CGA294849 (tomato fruits: 14% TRR).

CGA128632 including its conjugates, which were major metabolites in tomato foliage (23% TRR) and potato foliage (up to 36% TRR) were not identified in the rat.

Environmental fate in soil

The Meeting received information on the fate of pymetrozine under aqueous hydrolysis. In addition, the Meeting received information on the uptake and metabolism of pymetrozine in rotational crops under confined and field conditions.

Hydrolysis in aqueous buffer solutions revealed a moderate to quick decline of the parent under acidic conditions (pH5 or less) while samples at pH 7 and pH 9 were stable (> 90% remaining). The major degradation products identified were CGA300407 for the pyridine-label and CGA215525 for the triazine-label, which are formed by direct cleavage of the parent molecule.

Confined rotational crop studies on mustard greens, radish and wheat were conducted at rates equivalent to a soil application of 0.41 kg ai/ha using either [pyridine-¹⁴C]- or [triazine-¹⁴C]-pymetrozine. Plantback intervals were 30, 60, 90, 120 and 360 days. Total radioactive residues in edible commodities were in the range of 0.018–0.049 mg eq/kg (wheat grain), 0.025–0.13 mg eq/kg (mustard leaves) and 0.026–0.061 mg eq/kg (radish tubers) for [triazine-6-¹⁴C]-pymetrozine and of 0.036–0.11 mg eq/kg (wheat grain), 0.011–0.053 mg eq/kg (mustard leaves) and 0.014–0.042 mg eq/kg (radish tubers) for [pyridine-5-¹⁴C]-pymetrozine. In potential feed items TRR levels were 0.047–0.48 mg eq/kg and 0.021–0.23 mg eq/kg in wheat forage and fodder for [triazine-6-¹⁴C]- and [pyridine-5-¹⁴C]-label, respectively.

In all crop samples unchanged pymetrozine was found in minor amounts not exceeding 10% of the TRR. Highest concentrations were found in wheat forage (0.011 mg/kg) while all other matrices with detected residues gave concentrations between 0.001 mg/kg and 0.01 mg/kg.

In crops planted in soil treated with [pyridine-¹⁴C]-pymetrozine nicotinic acid incl. its glycoside (CGA180777, up to 24% TRR or 0.016 mg eq/kg), trigonelline (CGA96956, up to 32% TRR or 0.021 mg eq/kg), nicotinamide (CGA180778, up to 17% TRR, 0.005 mg eq/kg) and CGA-128632-glycoside (up to 11% TRR, 0.011 mg eq/kg) were found as major metabolites.

After application of [triazine-¹⁴C]-pymetrozine most of the radioactivity was represented by GS23199 and its glycosides (total up to 39% TRR or 0.056 mg eq/kg in wheat forage) or CGA266591 (up to 34% TRR or 0.027 mg eq/kg).

For longer plantback intervals, unextracted radioactivity increased ranging from 19–78% TRR.

Two additional studies investigating the uptake of total radioactive residues from the soil, either [pyridine-¹⁴C]- or [triazine-¹⁴C]-pymetrozine were applied to bare soil at rates equivalent to 0.5 kg ai/ha. Lettuce, wheat, sugar beet and maize were cultivated as succeeding crops after plantback intervals of 63, 91 or 307 days.

TRR levels found in the various matrices were low. In edible commodities TRR levels from all plantback intervals were between 0.002–0.01 mg eq/kg. In feed commodities TRR levels ranged up to 0.061 mg eq/kg in wheat straw.

Field rotational crop studies were conducted on four locations in the USA. Pymetrozine was applied to either tomatoes, peppers, cucumbers or lettuce at rates of 4 × 0.1 kg ai/ha (7 day interval). After 30 days the crop was destroyed and wheat, turnips or lettuce were planted as rotational crops. In all samples collected (mature and immature) no pymetrozine or GS23199 above their LOQ of 0.02 mg/kg were found. However, for GS23199 no hydrolysis step was conducted to release conjugates, which posed the main part of total GS23199 identified in confined rotational crop studies.

In summary the Meeting concluded that the transfer of residues into rotational crops is low. Parent pymetrozine is quickly degraded, not resulting in significant residues at harvest.

The metabolite GS23199, mainly present as sugar-conjugate, was the major residue in most samples (up to 39% TRR). Its concentrations ranged up to a maximum of 0.056 mg eq/kg in wheat forage. In edible commodities highest residues were found in mustard leaves amounting 0.008 mg eq/kg. Approximating the maximum seasonal application rate of 0.45 kg ai/ha based on the submitted GAPs and the involved interception of the treated crop, the Meeting concluded that no residues of GS23199 (including its sugar conjugate) above 0.01 mg/kg are expected in edible commodities obtained from rotational crops.

Other potential metabolites formed are either identical to naturally occurring substances (CGA180777 and CGA180778) or also present at levels below 0.01 mg/kg under confined conditions (e.g., CGA266591).

Methods of residue analysis

The Meeting received analytical methods for the analysis of pymetrozine in plant and animal matrices. The basic principle employs extraction by homogenisation with aqueous borate buffer/methanol or n-hexane with acetonitrile/water partitioning for fatty samples. The extracts were cleaned by C18 solid-phase extraction. Residues are determined by liquid chromatography (LC) in combination with tandem mass spectroscopy (MS/MS) or UV (300 nm). The methods submitted are suitable for measuring residues with a LOQ of 0.01 mg/kg (LC-MS/MS) to 0.02 (HPLC-UV).

In addition specialised methods using LC-MS/MS methods for measuring the metabolites CGA300407 (LOQ: 0.005 mg/kg, high water and acidic matrices) and CGA313124 (LOQ: 0.01 mg/kg, animal matrices) were submitted.

The application of multi-residue methods was not tested.

In-trial validation of the analytical methods submitted was achieved at LOQs of 0.001 mg/kg up to 0.05 mg/kg, depending on the matrix.

Stability of residues in stored analytical samples

The Meeting received information on the storage stability of pymetrozine in plant and animal matrices. In addition the storage stability of GS23911 was investigated in plant matrices and the storage stability of CGA313124 in animal matrices.

Plant matrices

In fortified samples, depending on the matrix, parent pymetrozine decomposes rather quickly. In different studies the following intervals were identified, which showed at least 70% of the initial pymetrozine concentration:

Oranges	at least 24 months
Peaches	up to 1 month
Tomato, fruits	up to 6 months
Tomato, paste	up to 6 months
Cucumbers	up to 2 months
Melons	at least 24 months
Lettuce	up to 1 month
Potatoes	up to 1 month
Cottonseed	at least 25 months
Cotton oil	at least 24 months

The Meeting noted that pymetrozine residues may degrade in stored samples, however the effect is strongly depending on the specific commodity. No reasons for the observed degradation could be identified. Due to the inconsistency within commodity groups (e.g., like for cucurbits), more data on the storage stability for each commodity is required.

One study investigating the storage stability of incurred residues in plant commodities was submitted involving re-analysis for stored samples after 8–13 months. However, since the initial analysis of the samples was performed after 9–14 months, which is longer than the maximum storage interval identified, the data was not considered appropriate to conclude on the overall stability of the residue.

Animal matrices

Definition of the residue

Nicotinic acid (CGA180777) and nicotinamide (CGA180778) were identified as major residues (up to 77% TRR) in livestock animals, however due to their natural occurrence (Vitamin B group) they are not suitable as a marker substance for enforcement purposes in animal commodities. Parent pymetrozine was present in all goat tissues, most hen tissues and eggs (for eggs triazine-label only), however only in low amounts up to 11% TRR. In milk no parent pymetrozine was found. Nearly the entire residue was identified as CGA313124 and its phosphate conjugate. The Meeting recognized that pymetrozine is strongly metabolized in livestock animals. However, being the only representative analyte in most commodities, the Meeting concluded that parent pymetrozine is a suitable marker for the purpose of MRL setting in animal tissues and eggs. For milk, the residue for enforcement purposes is defined as CGA313124. Analytical methods are capable of measuring pymetrozine in animal matrices and CGA313124 in milk at LOQs of 0.01 mg/kg each.

In hens tissues and eggs CGA259168 (up to 45% TRR), I_{A7} (up to 49% TRR) and CGA294849 (up to 11% TRR) were major residues present at concentrations up to 0.079 mg eq/kg at 10 ppm dose. Parent pymetrozine itself was a minor residue in all samples. CGA294849 was identified as a minor metabolite in hens muscle and liver, representing up to 9% of TRR. The Meeting concluded, that parent pymetrozine is a relevant residue for the dietary intake of poultry tissues and eggs.

The significance of the animal metabolites CGA245342, CGA294849, I_{A7} and I_{A17} was assessed with the TTC approach based on exposure levels related to the uses evaluated. Exposure to I_{A17} did not exceed the TTC value for chronic exposure of 0.0025 µg/kg bw per day (EMEA for genotoxic impurities) as well as the single-exposure TTC of 0.2 µg/kg bw. CGA245342 and I_{A7} gave estimated exposure levels below 1.5 µg/kg bw per day (Cramer Class III), respectively. Based on the assessed uses, these metabolites are not considered relevant for the dietary intake.

CGA294849 was also assessed with the TTC approach with the major part of the exposure resulting from plant commodities. CGA294849 has a structural alert for genotoxicity but has not been tested. Since the exposure assessment exceeded the applicable TTC values, no conclusion on the relevance of CGA294849 for dietary intake assessment can be made.

In all samples residue concentrations in fat tissues were in the same order of magnitude as in muscle tissues. The log P_{ow} of pymetrozine is < 0. The Meeting decided that residues of pymetrozine are not fat soluble.

In plants following foliar treatment, pymetrozine was the major residue in plant parts directly contacted, representing 28–89% of the TRR. The major degradation products in all matrices were either trigonelline (CGA96956) present at level between 9–70% of the TRR or nicotinic acid (CGA180777) present up to 26% TRR, depending on the label. The pyridine-cleavage product CGA128632 (including sugar conjugates) was a major metabolite in tomato fruits (11% TRR) and in tomato and potato foliage (up to 26% TRR). The counterpart for the triazine-moiety was identified as GS23199 (including sugar conjugates) representing 11–18% of the TRR in rice straw and potato foliage. The metabolic pattern in rotational crops was comparable to the degradation products identified in primary treated crops.

The Meeting concluded that pymetrozine is present in major amounts in most plant matrices and therefore qualifies as a marker substance for enforcement purposes. The major plant metabolites trigonelline (CGA96956) and nicotinic acid (CGA180777) occur naturally in plants and are unspecific markers for the residue. Analytical methods are capable of measuring pymetrozine in all plant matrices at a LOQ of 0.01 mg/kg.

For dietary intake purposes nicotinic acid (CGA180777) and trigonelline (CGA96956) add insignificantly to natural background levels. The Meeting noted that no metabolism study on leafy crops was submitted. In the foliage of tomatoes and potatoes used as a substitute, the cleavage products CGA128632 (up to 26% TRR) and GS23199 (up to 18% TRR) and their sugar conjugates were major residues. However, CGA128632 was not found in rats. In addition, CGA294849 was found in tomato fruit representing 7.4% TRR after 3 days increasing to 14% TRR after 14 days.

The Meeting also noted that pymetrozine may degrade under acidic food processing condition, resulting in the formation of CGA300407 (up to 42% TRR), based on hydrolysis data for pyridine-labelled active substance. The hydrolysis of triazine-labelled active substance conducted at pH5 (25 °C and 75 °C), showed CGA215525 as the major degradation product (up to 48% TRR). No data on hydrolysis under simulated processing conditions were submitted for the triazine-label.

Besides parent pymetrozine, which is a major residue in plant commodities and should to be taken into account for dietary intake assessment, the relevance of the plant metabolites GS23199, CGA128632 and CGA294849 as well as of the degradation products formed during processing (CGA215525 and CGA300407) was assessed with the TTC approach.

Based on the exposure levels (IEDI and IESTI) estimated for the uses evaluated, GS23199 (Cramer Class III) and CGA215525 (Cramer Class III) were not considered relevant for dietary intake.

CGA128632 has therapeutic uses as a vasodilator with a minimal therapeutic dose of 1 mg/kg bw. In view of the margin compared to the estimated exposure levels (> 1000 to the IEDI and > 50 to the maximum IESTI), CGA128632 is not considered a relevant metabolite of pymetrozine for dietary intake.

CGA294849 was also assessed with the TTC approach with the major part of the exposure resulting from plant commodities. CGA294849 has a structural alert for genotoxicity but has not been tested. Since the exposure assessment exceeded the applicable TTC values, no conclusion on the relevance of CGA294849 for dietary intake assessment can be made.

The processing degradate CGA300407 does not have a structural alert for genotoxicity but the Meeting was made aware that positive genotoxicity results, in vitro and in vivo, exist for this compound. No conclusion on the relevance of CGA300407 can be made.

If future uses for pymetrozine result in changes of the dietary intake, reconsideration on the relevance of metabolites in plant and animal matrices and after processing may become necessary.

Definition of the residue for compliance with MRL for plant commodities, mammalian tissues, poultry tissues and eggs: *pymetrozine*

Definition of the residue for compliance with MRL for milk: *CGA313124 (4,5-dihydro-6-hydroxymethyl-4-[(3-pyridinyl-methylene)amino]-1,2,4-triazine-3(2H)-one)*

Definition of the residue for dietary intake in plant and animal commodities: *a conclusion could not be reached*

The residue is not fat-soluble.

Results of supervised residue trials on crops

The Meeting received supervised trial data for applications of pymetrozine on various fruit and vegetables crops as well as for oilseeds and rice conducted in China, Europe and the USA.

The Meeting recognized that pymetrozine may degrade under freezer storage conditions. All supervised field trials exceeding the maximum storage interval identified for the specific commodities are not taken into account for the evaluation of GAPs and the resulting residues. In addition residues of pymetrozine were considered stable in all plant commodities for maximum storage period of one months.

Citrus fruits

Pymetrozine is registered in Portugal for the use on citrus fruit at rates of 1×0.01 kg ai/hL with a PHI of 21 days. Supervised field trials conducted in the Europe according to this GAP were submitted.

In lemons (whole fruits) residues of pymetrozine were (n=4): 0.02, 0.02, 0.03, 0.07 mg/kg.

In mandarins (whole fruits) residues of pymetrozine were (n=4): < 0.02, 0.02, 0.03, 0.03 mg/kg.

In oranges (whole fruits) residues of pymetrozine were (n=13): 0.006, 0.007, 0.01, 0.012, < 0.02, 0.02(4), 0.03, 0.04, 0.05 and 0.06 mg/kg.

The Meeting noted that pymetrozine is registered in Portugal for the whole citrus group. Supervised field trials on oranges, lemons and mandarins indicated no significant difference in the datasets (Kruskal-Wallis-Testing). Therefore the Meeting decided to extend its estimations on the whole group of citrus fruits based on the combined dataset from all three commodities:

Pymetrozine residues in citrus fruits (n=21): 0.006, 0.007, 0.01, 0.012, < 0.02, < 0.02, 0.02(7), 0.03(4), 0.04, 0.05, 0.06 and 0.07 mg/kg.

In the pulp (flesh) corresponding residues were (n=21): < 0.005(4) and < 0.02(17) mg/kg.

The Meeting estimated a maximum residues level of 0.15 mg/kg for pymetrozine in citrus fruit and a median and highest residue of 0.02 mg/kg in citrus pulp.

Pome fruit

Pymetrozine is registered in Italy for the use on apples and pears at a rate of 1×0.25 kg ai/ha with a PHI of 14 days. Supervised field trials conducted in Italy according to this GAP were submitted.

However, the Meeting noted that for pome fruit no data on the storage stability were provided. In view of the general degradation of pymetrozine in stored commodities, specific data on the storage stability in pome fruits is required to assess the validity of the supervised field trials.

Apricot

Pymetrozine is registered in Belgium for use on apricots at the rate of 2×0.1 kg ai/ha with a PHI of 21 days. Supervised field trials were conducted in Europe at rates of 3×0.25 kg ai/ha.

The supervised field trials submitted for apricots were all conducted as decline studies. Since the active substance almost completely degraded within the 21 days investigated, the Meeting concluded that the additional treatment in comparison to the GAP from Belgium has no influence on the residue concentrations at harvest, allowing the use of the proportionality approach to adjust for the higher application rates involved.

Pymetrozine residues in apricots treated with 3×0.25 kg ai/ha were (n=4): $< 0.01(3)$ and 0.01 mg/kg.

Under consideration of a proportionality factor of 0.4 (0.1 kg ai/ha divided by 0.25 kg ai/ha), scaled residues were (n=4): 0.004 and $< 0.01(3)$ mg/kg.

Based on scaled residues from Europe, the Meeting estimated a maximum residues level of 0.01 mg/kg and a median and highest residue of 0.01 mg/kg for pymetrozine in apricots.

Peach and nectarines

Pymetrozine is registered in Spain for the use on peach and nectarines at the rate of 2×0.25 kg ai/ha with a PHI of 14 days. Supervised field trials were conducted in Europe at rates of 3×0.25 kg ai/ha.

The supervised field trials submitted for peaches were all conducted with one additional application compared to the GAP. However, taking into account low residues in samples analysed directly before the final treatment and the results from decline studies on peaches, the Meeting concluded that the additional treatment does not add significantly to the residue concentrations at harvest.

Pymetrozine residues in peach (n=6): $< 0.01(4)$, 0.04, 0.04 mg/kg.

Based on the dataset on peach the Meeting estimated a maximum residues level of 0.07 mg/kg, a median residue of 0.01 mg/kg and a highest residue of 0.04 mg/kg for pymetrozine in peach. The Meeting decided to extrapolate the estimations also to nectarines.

Strawberries

Pymetrozine is registered in Belgium for the use on field and protected strawberries at the rate of 3×0.2 kg ai/ha with a PHI of 3 days. Supervised field trials conducted in United Kingdom matching the GAP were submitted.

Pymetrozine residues in field strawberries were (n=2): 0.02 and 0.06 mg/kg.

Pymetrozine residues in protected strawberries were (n=2): 0.12 mg/kg.

The Meeting concluded that the data submitted for the use of pymetrozine on strawberries was insufficient for a maximum residue level estimation.

Broccoli

Pymetrozine is registered in Belgium for the use on broccoli at rates of 3×0.2 kg ai/ha with a PHI of 14 days. Supervised field trials conducted in Switzerland and United Kingdom matching the GAP were submitted.

However, the Meeting noted that for broccoli no data on the storage stability were provided. In view of the general degradation of pymetrozine in stored commodities, specific data on the storage stability in broccoli is required to assess the validity of the supervised field trials.

Cauliflower

Pymetrozine is registered in Belgium for the use on cauliflower at rates of 3×0.2 kg ai/ha with a PHI of 14 days. Supervised field trials conducted in France and Switzerland matching the GAP were submitted, however all samples were cut into segments at harvest before storage.

In 2013 the JMPR pointed out that cutting of large commodities in the field is against the recommended Codex sampling procedure and may cause problems due to an enhanced degradation of the residue. Testing strategies to investigate the stability of the residue were outlined in the 2013 JMPR Report. However, for pymetrozine such data was not submitted. In view of the short interval of the storage stability in various matrices, the possible effect of cutting cannot be assessed.

The Meeting decided that the data on cauliflower is invalid for recommendations of maximum residue levels for pymetrozine.

Head cabbage

Pymetrozine is registered in Germany for the use on head cabbage at rates of 3×0.2 kg ai/ha with a PHI of 7 days. Supervised field trials conducted in Europe matching the GAP were submitted. However some of the samples were cut into segments at harvest before storage, making them invalid for the assessment (see cauliflower).

In Portugal the use of pymetrozine is registered on head cabbage at rates of 3×0.2 kg ai/ha with a PHI of 14 days. Supervised field trials conducted in Europe matching the GAP were submitted.

However, the Meeting noted that for cabbages no data on the storage stability were provided. In view of the general degradation of pymetrozine in stored commodities, specific data on the storage stability in head cabbage is required to assess the validity of the supervised field trials.

Cucumbers

Pymetrozine is registered in Greece for the use on protected cucumbers at rates of 2×0.45 kg ai/ha with a PHI of 3 days. Residue data from Europe under protected conditions matching GAP application rates were submitted.

However, the supervised field trials submitted for cucumbers were all conducted with one additional application compared to the GAP. Taking into account the low residues in samples analysed directly before the final treatment and the results from decline studies on cucumbers, the Meeting concluded that the additional treatment does not add significantly to the residue concentrations at harvest.

Pymetrozine residues in cucumbers (n=8): 0.045, 0.07, 0.08, 0.083, 0.088, 0.09, 0.11 and 0.21 mg/kg.

Four additional trials on cucumbers matching the GAP from Greece were reported, but the samples were stored 3-10 months before analysis, which is longer than the maximum storage interval of 2 month for pymetrozine in cucumbers.

The Meeting estimated a maximum residues level of 0.3 mg/kg, a median residue of 0.0855 mg/kg and a highest residue of 0.21 mg/kg for pymetrozine in cucumbers.

Melons

Pymetrozine is registered in Portugal for the use on protected melons at rates of 3×0.3 kg ai/ha with a PHI of 3 days. Supervised field trials conducted in Europe matching the GAP were submitted. However some of the samples were cut into segments at harvest before storage, making them invalid for the assessment (see cauliflower). Residues in the remaining trials were:

Pymetrozine residues in whole melon fruits (n=2): 0.045 and 0.16 mg/kg.

The Meeting concluded that the data submitted for the use of pymetrozine on melons was insufficient for a maximum residue level estimation.

Peppers, sweet

Pymetrozine is registered in Czech Republic for the use on protected sweet peppers at rates of 3×0.36 kg ai/ha with a PHI of 3 days. Supervised field trials conducted in Europe under protected conditions matching the GAP were submitted.

Pymetrozine residues in protected sweet peppers (n=8): 0.16, 0.43, 0.43, 0.54, 0.64, 0.83, 1.1 and 1.4 mg/kg.

The Meeting estimated a maximum residues level of 3 mg/kg, a median residue of 0.59 mg/kg and an highest residue of 1.4 mg/kg for pymetrozine in pepper, sweet.

Tomatoes

Pymetrozine is registered in The Netherlands for the use on protected tomatoes at rates of 3×0.45 kg ai/ha with a PHI of 1 day. Supervised field trials conducted in Europe under protected conditions matching the GAP were submitted.

Pymetrozine residues in protected tomato fruits (n=8): 0.08, 0.14, 0.18, 0.27, 0.3, 0.33, 0.39 and 0.77 mg/kg.

The Meeting estimated a maximum residues level of 1.5 mg/kg, a median residue of 0.285 mg/kg and a highest residue of 0.77 mg/kg for pymetrozine in tomatoes.

Leafy vegetables, except brassica leafy vegetables

Pymetrozine is registered in the USA for the use on leafy vegetables at rates of 2×0.1 kg ai/ha with a PHI of 7 days. Supervised field trials conducted in the USA on head lettuce, leaf lettuce and spinach matching the GAP were submitted. However, most of the trials were stored for more than 1 months, which was identified as the maximum storage interval without a significant degradation of the residues. The results from trials analysed within this interval were:

Pymetrozine residues in head lettuce (n=2): < 0.02 and 0.1 mg/kg.

The Meeting concluded that the data submitted for the use of pymetrozine on leafy vegetables, except brassica leafy vegetables, was insufficient for a maximum residue level estimation.

Potatoes

Pymetrozine is registered in the United Kingdom for the use on potatoes at rates of 2×0.15 kg ai/ha with a PHI of 7 days. Supervised field trials conducted in Northern Europe at a higher rate of 2×0.2 kg ai/ha were submitted.

Pymetrozine residues in potato tubers (n=4): < 0.01(4) mg/kg.

The Meeting concluded that the data submitted for the use of pymetrozine on potatoes was insufficient for a maximum residue level estimation.

Asparagus

Pymetrozine is registered in the USA for the use on asparagus in vegetative state at rates of 6×0.1 kg ai/ha to the mature ferns with a PHI of 170 days. Supervised field trials conducted in the USA on asparagus treated with rates of 3×0.19 kg ai/ha were submitted involving PHIs of 172-267 days. The results were:

Pymetrozine residues in asparagus (n=6): $< 0.02(6)$ mg/kg.

Although samples of asparagus were stored longer than the maximum storage interval of 1 month for this commodity, the Meeting concluded to make estimations for the commodity. Taking into account the insignificant amounts of pymetrozine found in samples from confined rotational crop studies and the long interval between treatment and harvest without a direct application to the harvested commodity, the Meeting estimated a maximum residues level of 0.02^* mg/kg and a median and highest residue of 0 mg/kg for pymetrozine in asparagus.

Celery

Pymetrozine is registered in the USA for the use on celery at rates of 2×0.1 kg ai/ha with a PHI of 7 days. Supervised field trials conducted in the USA matching the GAP were submitted.

However, the Meeting noted that for celery no data on the storage stability were provided. In view of the general degradation of pymetrozine in stored commodities, specific data on the storage stability in celery is required to assess the validity of the supervised field trials.

Artichoke, globe

Pymetrozine is registered in France for the use on globe artichokes at rates of 2×0.2 kg ai/ha with a PHI of 14 days. Supervised field trials conducted in France matching the GAP were submitted.

Pymetrozine residues in artichokes (n=3): $< 0.02(3)$ mg/kg.

The Meeting concluded that the data submitted for the use of pymetrozine on artichoke is insufficient for a maximum residue level estimation.

Rice

Pymetrozine is registered in China for the use on rice at rates of 2×0.15 kg ai/ha with a PHI of 14 days. Supervised field trials conducted in China matching the GAP were submitted.

However, in the supervised field trials no data on the maximum storage interval between sampling and analysis were reported. In addition, storage stability data for cereal commodities was not provided, denying an assessment on the validity of the trials.

The Meeting concluded that the data submitted for the use of pymetrozine on rice is insufficient for a maximum residue level estimation.

Pecan

Pymetrozine is registered in the USA for the use on pecans at rates of 2×0.14 kg ai/ha with a PHI of 14 days. Supervised field trials conducted in the USA matching the GAP were submitted.

However, the Meeting noted that for pecan no data on the storage stability were provided. In view of the general degradation of pymetrozine in stored commodities, specific data on the storage stability in pecan is required to assess the validity of the supervised field trials.

Chestnut, hazelnut and walnut

Pymetrozine is registered in France for the use on chestnut, hazelnut and walnut at rates of 2×0.1 kg ai/ha with a PHI of 14 days. Supervised field trials on walnuts conducted in France matching the GAP were submitted.

However, the Meeting noted that for chestnuts, hazelnuts or walnuts no data on the storage stability were provided. In view of the general degradation of pymetrozine in stored commodities, specific data on the storage stability for respective commodities is required to assess the validity of the supervised field trials.

Rape

Pymetrozine is registered in Belgium, France and Germany for the use on oilseed rape rates of 1×0.075 kg ai/ha at BBCH 59 (first petals visible, flower buds still closed). The PHI is covered by the growth stage. Supervised field trials conducted in Northern and Southern Europe at slightly exaggerated rates of 1×0.1 kg ai/ha were submitted.

However, the Meeting noted that for rape seeds no data on the storage stability were provided. In view of the general degradation of pymetrozine in stored commodities, specific data on the storage stability in rape seeds is required to assess the validity of the supervised field trials.

Cotton

Pymetrozine is registered in Greece for the use on cotton rates of 2×0.2 kg ai/ha with a PHI of 35 days. Supervised field trials conducted in Southern Europe matching the registered application rate but with one additional treatment were submitted.

Pymetrozine residues in delinted seeds (n=6): $< 0.02(6)$ mg/kg.

The Meeting estimated a maximum residues level of 0.02 mg/kg and a median residue of 0.02 mg/kg for pymetrozine in cotton seeds.

Animal feeds

Rice straw

Pymetrozine is registered in China for the use on rice at rates of 2×0.15 kg ai/ha with a PHI of 14 days. Supervised field trials conducted in China matching the GAP were submitted.

However, in the supervised field trials no data on the maximum storage interval between sampling and analysis were reported. In addition, storage stability data for cereal commodities was not provided, denying an assessment on the validity of the trials.

The Meeting concluded that the data submitted for the use of pymetrozine on rice straw is insufficient for an estimation.

Cotton seed hulls

Pymetrozine is registered in Greece for the use on cotton rates of 2×0.2 kg ai/ha with a PHI of 35 days. Supervised field trials conducted in Southern Europe matching the registered application rate but with one additional treatment were submitted.

Pymetrozine residues in cotton seed hulls (n=6): $< 0.02(4)$, 0.02 and 0.02 mg/kg.

The Meeting estimated a maximum residues level of 0.04 mg/kg and a median residue of 0.02 mg/kg for pymetrozine in cotton seed hulls.

Fate of residues during processing

The Meeting received information on the hydrolysis of radio-labelled pymetrozine as well as processing studies using unlabelled material in tomatoes and sweet peppers.

In a hydrolysis study using [pyridine- ^{14}C]-pymetrozine typical processing conditions were simulated (pH 4,5 and 6 with 90°C, 100°C and 120°C for 20, 60 and 20 minutes). While no degradation of the residue was observed under pH6 (120°C for 20 minutes), a significant loss of parent substance occurred at pH 4 and pH5. The cleavage product CGA300407 was identified as the primary degradation product present at 33% of the TRR at pH4 and at 42% of the TRR at pH5.

No hydrolysis study simulating processing conditions was conducted using [triazine-¹⁴C]-pymetrozine. However, the hydrolysis in buffer solutions for the environmental fate was investigated showing an identical degradation of the active substance. The counterpart to CGA300407 was identified as CGA215525, which is expected to pose the remaining part of the residue in processed products.

The fate of pymetrozine residues has been examined simulating household and commercial processing of tomatoes and sweet peppers. Estimated processing factors for the commodities considered at this Meeting are summarised below.

Raw commodity	Processed commodity	Pymetrozine		
		Individual processing factors	Mean or best estimate processing factor	Median residue in mg/kg
Tomato (median: 0.285 mg/kg)	Juice, raw	0.05	0.05	0.014
	Puree	< 0.03(4), < 0.4	< 0.03	0.008
	Paste	< 0.04, < 0.4	< 0.22	0.063
	Canned/preserve	< 0.03(4), 0.07	0.03	0.008
	pomace, wet	0.08, < 0.4	0.24	0.068
Sweet pepper median: 0.59 mg/kg)	Cooked fruit	< 0.02(3)	< 0.02	0.012

The Meeting considered that maximum residue levels for processed commodities are covered by their raw agricultural commodities.

Residues in animal commodities

Farm animal feeding studies

The Meeting received feeding studies involving pymetrozine on lactating cows.

Three groups of lactating cows were dosed daily at levels of 1, 3 and 10 ppm in the diet for 28 consecutive days. Milk was collected throughout the whole study and tissues were collected on day 29 within 24 hrs after the last dose.

In milk and tissues of all dose groups no detectable residues of pymetrozine above the LOQ of 0.01 mg/kg were found.

The metabolite CGA313124 was also not found in tissues. In milk, CGA313124 was not detected for the 1ppm dose group but was present at levels of 0.02 mg/kg for the 3 ppm group and of 0.05 mg/kg for the 10 ppm group.

The Meeting noted that tissue samples were stored up to 10-13 months, which is longer than the maximum storage interval identified for pymetrozine in muscle. However, taking into account that based on goat metabolism studies liver is expected to be the tissue with highest residue concentrations, which was analysed for the parent residue within the interval supported by storage stability data, the general result of pymetrozine residues being <0.01 mg/kg in cow tissues is accepted.

Estimated maximum and mean dietary burdens of livestock and animal commodities maximum residue levels

Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are presented in Annex 6 of the 2014 Report. The calculations were made according to the livestock diets from US-Canada, EU, Australia and Japan in the OECD Table (Annex 6 of the 2006 JMPR Report).

	Livestock dietary burden, pymetrozine, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	max.	mean	max.	mean	max.	mean	max.	mean
Beef cattle	< 0.01	< 0.01	none	none	0.03	0.03	none	none
Dairy cattle	< 0.01	< 0.01	none	none	0.03 ^a	0.03 ^b	none	none
Poultry - broiler	none	none	none	none	none	none	none	none
Poultry - layer	none	none	none	none	none	none	none	none

^a Highest maximum beef or dairy cattle burden suitable for MRL estimates for mammalian meat and milk

^b Highest mean beef or dairy cattle burden suitable for median estimates for mammalian meat and milk
none no relevant feed items

Animal commodity maximum residues levels

For beef and dairy cattle maximum and mean dietary burdens of 0.03 ppm were estimated, respectively. In farm animal feeding studies on lactating cows no detectable residues of pymetrozine in tissues were found for all dose groups up to 10 ppm.

The Meeting estimated median and highest residue values of 0 for mammalian meat, edible offal and fat and corresponding maximum residue levels of 0.01* mg/kg.

For milk, CGA313124 was not detected in the milk samples for the 1 ppm group. Under consideration of the maximum and mean dietary burden for dairy cattle being 33 times lower than this dose level, the Meeting estimated a maximum residue level of 0.01* mg/kg and a median residue of 0 mg/kg, respectively for milks.

For poultry no relevant feed items were identified. The Meeting estimated median and highest residue values of 0 mg/kg for poultry meat, edible offal of and fat as well as for eggs. The Meeting also estimated maximum residue levels of 0.01* mg/kg for poultry meat, edible offal of and fat as well as for eggs.

RECOMMENDATIONS

Definition of the residue for compliance with MRL for plant commodities, mammalian tissues, poultry tissues and eggs: *pymetrozine*

Definition of the residue for compliance with MRL for milk: *CGA313124 (4,5-dihydro-6-hydroxymethyl-4-[(3-pyridinyl-methylene)amino]-1,2,4-triazine-3(2H)-one)*

Definition of the residue for dietary intake in plant and animal commodities: *a conclusion could not be reached*

The residue is not fat-soluble.

FURTHER WORK OR INFORMATION

- storage stability data on more individual commodities
- supervised field trials analysed within the maximum storage periods
- stability data for pymetrozine during homogenization of field samples
- field rotational crop studies including analysis of conjugates
- applicability of multi-residue analytical methods
- a hydrolysis study simulating industrial processing using [6-triazine-¹⁴C]-pymetrozine
- processing data including analysis of CGA300407 and CGA215525

DIETARY RISK ASSESSMENT

Because the Meeting was unable to conclude on the toxicological relevance of the metabolites CGA294849 and CGA300407, the Meeting could not reach a conclusion on a residue definition for the dietary intake.

As a result, long- and short-term dietary intake assessments could not be conducted.

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PYMET_060	Tribolet, R	2001	Residue Stability Study for Pymetrozine (CGA 215944) and its Metabolite GS 23199 in dried Hops under Freezer Storage conditions, Syngenta Crop Protection AG, Basel, Switzerland, Syngenta Crop Protection AG, Basel, Switzerland, 101/00, GLP, not published, Syngenta File No CGA215944/4814
PYMET_061	Schulz, H	2011	Pymetrozine—Determination of the Storage Stability of Pymetrozine in Plant Matrices, Syngenta—Jealott's Hill, Bracknell, United Kingdom, SGS INSTITUT FRESENIUS GmbH, Im Maisel 14, D-65232 Taunusstein, Germany, IF-08/01163953-REG, GLP, not published, Syngenta File No CGA215944_10930
PYMET_062	Joseph, TA	2000	Stability of incurred residues in crop group 4—leafy vegetables and crop group 5—leafy brassica vegetables under freezer storage conditions, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection Inc., Greensboro, USA, 755-99, GLP, not published, Syngenta File No CGA215944/4761
PYMET_063	Boyette, S	1998	Stability of CGA 215944 and CGA 313124 fortified into meat and milk under freezer storage conditions, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection Inc., Greensboro, USA, ABR-98069, GLP, not published, Syngenta File No CGA215944/0741
PYMET_064	Tribolet, R	1995	CGA 215944, CHESS WP 25, Oranges, Spain, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1033/94, GLP, not published, Syngenta File No CGA215944/0283
PYMET_065	Tribolet, R	1995	CGA 215944, CHESS WP 25, Oranges, Spain, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1034/94, GLP, not published, Syngenta File No CGA215944/0284
PYMET_066	Tribolet, R	1995	CGA 215944, CHESS WP 25, Oranges, Spain, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1035/94, GLP, not published, Syngenta File No CGA215944/0285
PYMET_067	Tribolet, R	1995	CGA 215944, CHESS WP 25, Oranges, Spain, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1036/94, GLP, not published, Syngenta File No CGA215944/0286
PYMET_068	Tribolet, R	1995	CGA 215944, CHESS WP 25, Lemons (fruit), Spain, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1037/94, GLP, not published, Syngenta File No CGA215944/0230
PYMET_069	Tribolet, R	1995	CGA 215944, CHESS WP 25, Mandarins, Spain, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1039/94, GLP, not published, Syngenta File No CGA215944/0273
PYMET_070	Tribolet, R	1995	CGA 215944, CHESS WP 25, Mandarins, Spain, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1040/94, GLP, not published, Syngenta File No CGA215944/0274
PYMET_071	Tribolet, R	1996	CGA 215944, Chess 25 WP, A-8811, oranges (whole fruits, pulp, peel, juice), Spain,, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1006/95, GLP, not published, Syngenta File No CGA215944/0480
PYMET_072	Tribolet, R	1996	CGA 215944, Chess 25 WP, A-8811, mandarins, Spain, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland,

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			1010/95, GLP, not published, Syngenta File No CGA215944/0420
PYMET_073	Tribolet, R	1996	CGA 215944, Chess 25 WP, A-8811, oranges (whole fruits, pulp, peel, juice), Spain,, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1011/95, GLP, not published, Syngenta File No CGA215944/0481
PYMET_074	Tribolet, R	1996	CGA 215944, Chess 25 WP, A-8811, oranges (whole fruits, pulp, peel, juice), Spain,, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1012/95, GLP, not published, Syngenta File No CGA215944/0482
PYMET_075	Tribolet, R	1996	CGA 215944, Chess 25 WP, A-8811, oranges (whole fruits, pulp, peel, juice), Spain,, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1013/95, GLP, not published, Syngenta File No CGA215944/0483
PYMET_076	Tribolet, R	1996	CGA 215944, Chess 25 WP, A-8811, oranges (whole fruits, pulp, peel, juice), Spain,, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1014/95, GLP, not published, Syngenta File No CGA215944/0484
PYMET_077	Tribolet, R	1996	CGA 215944, Chess 25 WP, A-8811, oranges (whole fruits, pulp, peel, juice), Spain,, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1015/95, GLP, not published, Syngenta File No CGA215944/0485
PYMET_078	Tribolet, R	1996	CGA 215944, Chess 25 WP, A-8811, mandarins, Spain, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1016/95, GLP, not published, Syngenta File No CGA215944/0421
PYMET_079	Tribolet, R	1996	CGA 215944, Chess 25 WP, A-8811, lemons, Spain, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1025/95, GLP, not published, Syngenta File No CGA215944/0422
PYMET_080	Tribolet, R	1996	CGA 215944, Chess 25 WP, A-8811, lemons, Spain, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1026/95, GLP, not published, Syngenta File No CGA215944/0423
PYMET_081	Tribolet, R	1996	CGA 215944, Chess 25 WP, A-8811, lemons, Spain, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1027/95, GLP, not published, Syngenta File No CGA215944/0424
PYMET_082	Mason, R	2007	Pymetrozine (CGA215944)—Residue Study on Oranges in Spain in 2006, Syngenta Crop Protection AG, Basel, Switzerland, Syngenta—Jealott's Hill International, Bracknell, Berkshire, United Kingdom, T001731-06, GLP, not published, Syngenta File No CGA215944/5013
PYMET_083	Sole, C	2003	Residue Study with Pymetrozine (CGA 215944) in or on Apples in Italy, Syngenta Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Vergeze, France, 02-1132, GLP, not published, Syngenta File No CGA215944/4883
PYMET_084	Sole, C	2003	Residue Study with Pymetrozine (CGA 215944) in or on Apples in Italy, Syngenta Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Vergeze, France, 02-1133, GLP, not published, Syngenta File No CGA215944/4884
PYMET_085	Sole, C	2003	Residue Study with Pymetrozine (CGA 215944) in or on Apples in Italy, Syngenta Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Vergeze, France, 02-1131, GLP, not published, Syngenta File No CGA215944/4882
PYMET_086	Sole, C	2004	Residue study with Pymetrozine (CGA215944) in or on Apples in Italy, Syngenta Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Vergeze, France, 03-5051, GLP, not published, Syngenta File No CGA215944/4925
PYMET_087	Sole, C	2004	Residue study with Pymetrozine (CGA215944) in or on Apples in Italy, Syngenta Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Vergeze, France, 03-5052, GLP, not published, Syngenta File No

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			CGA215944/4926
PYMET_088	Sole, C	2004	Residue study with Pymetrozine (CGA215944) in or on Apples in Italy, Syngenta Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Vergeze, France, 03-5053, GLP, not published, Syngenta File No CGA215944/4924
PYMET_089	Sole, C	2004	Residue study with Pymetrozine (CGA215944) in or on Apples in Italy, Syngenta Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Vergeze, France, 03-5054, GLP, not published, Syngenta File No CGA215944/4922
PYMET_090	Sole, C	2004	Residue study with Pymetrozine (CGA215944) in or on Apples in Italy, Syngenta Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Vergeze, France, 03-5055, GLP, not published, Syngenta File No CGA215944/4923
PYMET_091	Allen, L	2011	Pymetrozine—Residue Study on Peach in Southern France, Italy and Spain in 2010, Syngenta—Jealott's Hill, Bracknell, United Kingdom, CEMAS, North Ascot, United Kingdom, CEMR-4669, GLP, not published, Syngenta File No A9364J_11425
PYMET_092	Betson, S	2012	Pymetrozine—Residue Study on Apricots in Southern France and Spain in 2011, Syngenta, CEMAS, North Ascot, United Kingdom, CEMR-4984-REG, GLP, not published, Syngenta File No A9364J_11483
PYMET_093	Bour, D	2005	Determination of Pymetrozine in or on protected strawberries, Agrisearch UK Ltd., Melbourne, Derbyshire, United Kingdom, ADME—Bioanalyses, Vergeze, France, HDC/PYM/05001, GLP, not published, Syngenta File No A9364A_10809
PYMET_094	Sole, C	2005	Determination of Pymetrozine in or on outdoor and protected strawberries., Agrisearch UK Ltd., Melbourne, Derbyshire, United Kingdom, ADME—Bioanalyses, Vergeze, France, HDC/PYM/8180, GLP, not published, Syngenta File No A9364A_10808
PYMET_095	Bohm	1996	CGA 215944, WP, A-8811B, Cabbage, Germany, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy GmbH, Frankfurt a.Main, Germany, EXP. GR 507 (1128/95), GLP, not published, Syngenta File No CGA215944/0370
PYMET_096	Bohm	1996	CGA 215944, WP, A-8811B, Cabbage, Germany, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy GmbH, Frankfurt a.Main, Germany, EXP. GR 51695 (1109/95), GLP, not published, Syngenta File No CGA215944/0367
PYMET_097	Bohm	1996	CGA 215944, WP, A-8811B, Cabbage, Germany, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy GmbH, Frankfurt a.Main, Germany, EXP. RU-CI-0595 MZ (1126/95), GLP, not published, Syngenta File No CGA215944/0369
PYMET_098	Bohm	1996	CGA 215944, WP, A-8811B, Cabbage, Germany, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy GmbH, Frankfurt a.Main, Germany, EXP. GR 51795 (1110/95), GLP, not published, Syngenta File No CGA215944/0368
PYMET_099	Tribolet, R	1998	Residue study with pymetrozine (CGA215944) in or on cabbage in France (North)., Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, 1135/97, GLP, not published, Syngenta File No CGA215944/0703
PYMET_100	Pointurier, R	2000	Residue Study with Pymetrozine (CGA 215944) in or on Cabbage in France (N), Novartis Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Aigues-Vives, France, 9932001, GLP, not published, Syngenta File No CGA215944/4713
PYMET_101	Pointurier, R	2000	Residue Study with Pymetrozine (CGA 215944) in or on Cabbage in France (N), Novartis Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Aigues-Vives, France, 9932002, GLP, not published, Syngenta File No CGA215944/4714
PYMET_102	Bohm, KH	1996	CGA 215944, WP 25, Cabbage (heads), Portugal, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1024/94, GLP, not published, Syngenta File No CGA215944/0383

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PYMET_103	Bohm, KH	1996	CGA 215944, WP 25, Cabbage (heads), Portugal, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1025/94, GLP, not published, Syngenta File No CGA215944/0384
PYMET_104	Bohm, KH	1996	CGA 215944, WP 25, Cabbage (heads), Portugal, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1026/94, GLP, not published, Syngenta File No CGA215944/0385
PYMET_105	Bohm, KH	1996	CGA 215944, WP 25, Cabbage (heads), Portugal, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1191/94, GLP, not published, Syngenta File No CGA215944/0389
PYMET_106	Bohm, KH	1996	CGA 215944, WP 25, Cabbage (heads), Portugal, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1030/95, GLP, not published, Syngenta File No CGA215944/0386
PYMET_107	Bohm, KH	1996	CGA 215944, WP 25, Cabbage (heads), Portugal, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1031/95, GLP, not published, Syngenta File No CGA215944/0387
PYMET_108	Bohm, KH	1996	CGA 215944, WP 25, Cabbage (heads), Portugal, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1035/95, GLP, not published, Syngenta File No CGA215944/0388
PYMET_109	Mason, R	2005	Pymetrozine (CGA215944): Residue study in or on Head Cabbage in Spain and France (South), Syngenta Crop Protection AG, Basel, Switzerland, Syngenta—Jealott's Hill, Bracknell, United Kingdom, 04-5006, GLP, not published, Syngenta File No CGA215944/4947
PYMET_110	Sole, C	2004	Residue study with Pymetrozine (CGA215944) in or on Broccoli in Switzerland, Syngenta Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Vergeze, France, 03-5006, GLP, not published, Syngenta File No CGA215944/4929
PYMET_111	Mason, R	2009	Pymetrozine (CGA215944): Residue study in or on Broccoli in the United Kingdom, Syngenta—Jealott's Hill, Bracknell, United Kingdom, Syngenta—Jealott's Hill, Bracknell, United Kingdom, 04-5024, GLP, not published, Syngenta File No A9364A_10812
PYMET_112	Pointurier, R	2001	Residue Study with Pymetrozine (CGA 215944) in or on Cauliflower in France (North), Syngenta Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Vergeze, France, 0030602, GLP, not published, Syngenta File No CGA215944/4807
PYMET_113	Pointurier, R	2001	Residue Study with Pymetrozine (CGA 215944) in or on Cauliflower in France (North), Syngenta Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Vergeze, France, 0030702, GLP, not published, Syngenta File No CGA215944/4800
PYMET_114	Pointurier, R	2001	Residue Study with Pymetrozine (CGA 215944) in or on Cauliflower in France (North), Syngenta Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Vergeze, France, 0030601, GLP, not published, Syngenta File No CGA215944/4808
PYMET_115	Pointurier, R	2001	Residue Study with Pymetrozine (CGA 215944) in or on Cauliflower in France (North), Syngenta Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Vergeze, France, 0030701, GLP, not published, Syngenta File No CGA215944/4801
PYMET_116	Gasser, A	2002	Residue Study with Pymetrozine (CGA 219417) in or on Cauliflower in Switzerland, Syngenta Crop Protection AG, Basel, Switzerland, Syngenta Crop Protection AG, Basel, Switzerland, 1011/01, GLP, not published, Syngenta File No CGA215944/4871
PYMET_117	Pointurier, R	2002	Residue Study with Pymetrozine (CGA 215944) in or on Cauliflower in France (North), Syngenta Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Vergeze, France, 0131201, GLP, not published, Syngenta File No CGA215944/4845
PYMET_118	Pointurier, R	2002	Residue Study with Pymetrozine (CGA 215944) in or on Cauliflower in France (North), Syngenta Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Vergeze, France, 0131301, GLP, not published, Syngenta File No

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			CGA215944/4863
PYMET_119	Gasser, A	2002	Residue Study with Pymetrozine (CGA 219417) in or on Cauliflower in Switzerland, Syngenta Crop Protection AG, Basel, Switzerland, Syngenta Crop Protection AG, Basel, Switzerland, 1012/01, GLP, not published, Syngenta File No CGA215944/4872
PYMET_120	Tribolet, R	1996	Determination of Residues of CGA215944 in Cucumbers (Fruit)—Greenhouse trial, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1133/95, GLP, not published, Syngenta File No CGA215944/0433
PYMET_121	Tribolet, R	1996	Determination of residues of CGA215944 in Cucumbers (fruit)—greenhouse trial, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1134/95, GLP, not published, Syngenta File No CGA215944/0434
PYMET_122	Tribolet, R	1996	Determination of residues of CGA215944 in Cucumbers (Fruit)—Greenhouse trail, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1135/95, GLP, not published, Syngenta File No CGA215944/0435
PYMET_123	Tribolet, R	1998	Residue Study with Pymetrozine (CGA215944) on Cucumbers in Switzerland, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, 1111/97, GLP, not published, Syngenta File No CGA215944/0693
PYMET_124	Mason, R	2007	Pymetrozine (CGA215944)—Residue Study on Protected Cucumber in the Netherlands and Northern France in 2006, Syngenta Crop Protection AG, Basel, Switzerland, Syngenta—Jealott's Hill International, Bracknell, Berkshire, United Kingdom, T001736-06, GLP, not published, Syngenta File No CGA215944/5018
PYMET_125	Mason, R	2007	Pymetrozine (CGA215944)—Residue Study on Protected Cucumber in Spain and Southern France in 2006, Syngenta Crop Protection AG, Basel, Switzerland, Syngenta—Jealott's Hill International, Bracknell, Berkshire, United Kingdom, T001737-06, GLP, not published, Syngenta File No CGA215944/5019
PYMET_126	Tribolet, R	2000	Residue Study with Pymetrozine (CGA 215944) in or on Melons in Italy, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, 1072/99, GLP, not published, Syngenta File No CGA215944/4725
PYMET_127	Tribolet, R	2000	Residue Study with Pymetrozine (CGA 215944) in or on Melons in Italy, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, 1071/99, GLP, not published, Syngenta File No CGA215944/4724
PYMET_128	Pointurier, R	1999	Residue Study with CGA 215944 in or on Melon in South of France, Novartis Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Aigues-Vives, France, 9931602, GLP, not published, Syngenta File No CGA215944/4663
PYMET_129	Pointurier, R	1999	Residue Study with CGA 215944 in or on Melon in North of France, Novartis Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Aigues-Vives, France, 9931601, GLP, not published, Syngenta File No CGA215944/4662
PYMET_130	Tribolet, R	1999	Residue study with pymetrozine (CGA215944) in or on Melons in Spain, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, 1075/98, GLP, not published, Syngenta File No CGA215944/0782
PYMET_131	Tribolet, R	1999	Residue Study with pymetrozine (CGA215944) in or on Melons in Spain, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, 1076/98, GLP, not published, Syngenta File No CGA215944/0783
PYMET_132	Tribolet, R	1999	Residue study with pymetrozine (CGA215944) in or Melons in Spain, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, 1077/98, GLP, not published, Syngenta File No CGA215944/0784
PYMET_133	Tribolet, R	1999	Residue study with pymetrozine (CGA215944) in or on Melons in Spain, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, 1078/98, GLP, not published, Syngenta File No CGA215944/0785

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PYMET_134	Tribolet, R	2000	Residue Study with Pymetrozine (CGA 215944) in or on Sweet Peppers in the Netherlands, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, 1091/99, GLP, not published, Syngenta File No CGA215944/4677
PYMET_135	Tribolet, R	2000	Residue Study with Pymetrozine (CGA 215944) in or on Sweet Peppers in Italy, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, 1073/99, GLP, not published, Syngenta File No CGA215944/4726
PYMET_136	Tribolet, R	2000	Residue Study with Pymetrozine (CGA 215944) in or on Sweet Peppers in Italy, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, 1074/99, GLP, not published, Syngenta File No CGA215944/4727
PYMET_137	Mason, R	2007	Pymetrozine (CGA215944)—Residue Study on Protected Sweet Pepper in Spain and Southern France in 2006, Syngenta Crop Protection AG, Basel, Switzerland, Syngenta—Jealott's Hill International, Bracknell, Berkshire, United Kingdom, T001735-06, GLP, not published, Syngenta File No CGA215944/5017
PYMET_138	Mason, R	2007	Pymetrozine (CGA215944)—Residue Study on Protected Sweet Pepper in the Netherlands in 2006, Syngenta Crop Protection AG, Basel, Switzerland, Syngenta—Jealott's Hill International, Bracknell, Berkshire, United Kingdom, T001734-06, GLP, not published, Syngenta File No CGA215944/5016
PYMET_139	Pointurier, R	1996	CGA 215944, WP 25, A-8811 B, Sweet Peppers (greenhouse), France (South), Novartis Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Aigues-Vives, France, 9831702, GLP, not published, Syngenta File No CGA215944/0777
PYMET_140	Tack, T	1998	CGA 215944, WG 50, A-9364 A, 25 WP, A-8811 B, Sweet peppers (glasshouse), United Kingdom, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection UK Ltd., Whittlesford, United Kingdom, IR2297 (NL), GLP, not published, Syngenta File No CGA215944/0683
PYMET_141	Tribolet, R	1999	CGA 215944, WP 25, A-8811 B, Sweet Peppers (greenhouse), Spain, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, 1055/98, GLP, not published, Syngenta File No CGA215944/0793
PYMET_142	Pointurier, R	1999	CGA 215944, WP 25, A-8811 B, Sweet Peppers (greenhouse), France (North), Novartis Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Aigues-Vives, France, 9831701, GLP, not published, Syngenta File No CGA215944/0776
PYMET_143	Tribolet, R	1999	CGA 215944, WP 25, A-8811 B, Sweet Peppers (greenhouse), Spain, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, 1054/98, GLP, not published, Syngenta File No CGA215944/0778
PYMET_144	Tribolet, R	1998	CGA 215944, WG 50, A-9364 A, Sweet peppers, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, 1110/97, GLP, not published, Syngenta File No CGA215944/0716
PYMET_145	Tribolet, R	1995	Determination of residues of CGA215944 in Tomatoes (fruit)—field trial (plastic tunnel), Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1083/94, GLP, not published, Syngenta File No CGA215944/0276
PYMET_146	Tribolet, R	1995	Determination of residues of CGA215944 in Tomatoes (fruit)—field trial (greenhouse), Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1084/94, GLP, not published, Syngenta File No CGA215944/0338
PYMET_147	Tribolet, R	1995	Determination of residues of CGA215944 in Tomatoes (fruit)—greenhouse trial, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1085/94, GLP, not published, Syngenta File No CGA215944/0277
PYMET_148	Tribolet, R	1995	Determination of residues of CGA215944 in Tomatoes (fruit)—greenhouse trial, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Ltd., Basel, Switzerland, 1086/94, GLP, not published, Syngenta File No CGA215944/0253
PYMET_149	Tribolet, R	1998	Residue study with Pymetrozine (CGA216944) in or on Tomatoes in Switzerland, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop

Code	Author	Year	Title, Institute, Report reference
			Protection AG, Basel, Switzerland, 1112/97, GLP, not published, Syngenta File No CGA215944/0710
PYMET_150	Tribolet, R	1999	Residue study with Pymetrozine (CGA215944) in or on Tomatoes in Netherlands., Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, 1093/98, GLP, not published, Syngenta File No CGA215944/0795
PYMET_151	Allen, L	2011	Pymetrozine ? Residue Study on Protected Tomatoes in Northern France and Germany in 2010, Syngenta—Jealott's Hill, Bracknell, United Kingdom, CEMAS, North Ascot, United Kingdom, CEMR-4675-REG, GLP, not published, Syngenta File No A9364J_11430
PYMET_152	Allen, L	2011	Pymetrozine ? Residue Study on Protected Tomatoes in Southern France in 2010, Syngenta—Jealott's Hill, Bracknell, United Kingdom, CEMAS, North Ascot, United Kingdom, CEMR-4674-REG, GLP, not published, Syngenta File No A9364J_11431
PYMET_153	Campbell, DD	1998	CGA 215944 + CGA 293343, FULFILL, ADAGE, PLATINUM, Leafy vegetables, USA, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection Inc., Greensboro, USA, ABR-98051, GLP, not published, Syngenta File No CGA215944/0745
PYMET_154	Allan, L	2011	Pymetrozine—Residue Study on Potatoes in Northern France, Germany and the United Kingdom in 2010, Syngenta—Jealott's Hill, Bracknell, United Kingdom, CEMAS, North Ascot, United Kingdom, CEMR-4670-REG, GLP, not published, Syngenta File No A9364J_11432
PYMET_155	Sole, C	2004	Residue study with Pymetrozine (CGA215944) in or on Artichoke in France (North), Syngenta Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Vergeze, France, 03-5060, GLP, not published, Syngenta File No CGA215944/4933
PYMET_156	Sole, C	2004	Residue study with Pymetrozine (CGA215944) in or on Artichoke in France (North), Syngenta Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Vergeze, France, 03-5061, GLP, not published, Syngenta File No CGA215944/4932
PYMET_157	Sole, C	2003	Residue Study with Pymetrozine (CGA 215944) in or on Artichokes in France (North), Syngenta Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Vergeze, France, 02-1129, GLP, not published, Syngenta File No CGA215944/4880
PYMET_158	Sole, C	2003	Residue Study with Pymetrozine (CGA 215944) in or on Artichokes in France (North), Syngenta Crop Protection AG, Basel, Switzerland, ADME—Bioanalyses, Vergeze, France, 02-1130, GLP, not published, Syngenta File No CGA215944/4881
PYMET_159	Samoil, K	2002	Pymetrozine: Magnitude of the Residue on Asparagus, IR-4 Project, North Brunswick, USA, IR-4 Project, North Brunswick, USA, 07341, GLP, not published, Syngenta File No 412339
PYMET_160	Malet, J and Allard, L	2004	Residues of Pymetrozine after 2 Applications of Plenum 50 WG in Walnut, Syngenta Crop Protection AG, Basel, Switzerland, Ministère de l'agriculture, Paris, France, RANO38302, GLP, not published, Syngenta File No CGA215944/4969
PYMET_161	Malet, J and Allard, L	2004	Residues of Pymetrozine after 2 Applications of Plenum 50 WG in Walnut, Syngenta Crop Protection AG, Basel, Switzerland, Ministère de l'agriculture, Paris, France, RANO20103, GLP, not published, Syngenta File No CGA215944/4968
PYMET_162	Schulz, H	2009	Pymetrozine (CGA215944)—Residue Study on Oilseed Rape in the United Kingdom, France (North) and Germany, Syngenta, SGS Institut Fresenius GmbH, Geneva, Switzerland, T009434-07-REG, GLP, not published, Syngenta File No A9364J_11311
PYMET_163	Schulz, H	2009	Pymetrozine (CGA215944) ? Residue study on Oilseed Rape in France (South) and Italy, Syngenta—Jealott's Hill, Bracknell, United Kingdom, SGS Institut Fresenius GmbH, Geneva, Switzerland, T009435-07-REG, GLP, not published, Syngenta File No CGA215944_10901

Code	Author	Year	Title, Institute, Report reference
PYMET_164	Tribolet, R	1998	CGA 215944, WG 50, A-9364 A, Cottonseed, Spain, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, 1162/97, GLP, not published, Syngenta File No CGA215944/0725
PYMET_165	Tribolet, R	2001	Residue Study with Pymetrozine (CGA 215944) in or on Cotton in Greece, Syngenta Crop Protection AG, Basel, Switzerland, Syngenta AG, Basel, Switzerland, 1099/99, GLP, not published, Syngenta File No CGA215944/4792
PYMET_166	Tribolet, R	2001	Residue Study with Pymetrozine (CGA 215944) in or on Cotton in Greece, Syngenta Crop Protection AG, Basel, Switzerland, Syngenta AG, Basel, Switzerland, 1100/99, GLP, not published, Syngenta File No CGA215944/4793
PYMET_167	Tribolet, R	2001	Residue Study with Pymetrozine (CGA 215944) in or on Cotton in Greece, Syngenta Crop Protection AG, Basel, Switzerland, Syngenta AG, Basel, Switzerland, 1101/99, GLP, not published, Syngenta File No CGA215944/4795
PYMET_168	Tribolet, R	2001	Residue Study with Pymetrozine (CGA 215944) in or on Cotton in Greece, Syngenta Crop Protection AG, Basel, Switzerland, Syngenta AG, Basel, Switzerland, 1102/99, GLP, not published, Syngenta File No CGA215944/4796
PYMET_169	Tribolet, R	2001	Residue Study with Pymetrozine (CGA 215944) in or on Cotton in Greece, Syngenta Crop Protection AG, Basel, Switzerland, Syngenta AG, Basel, Switzerland, 1103/99, GLP, not published, Syngenta File No CGA215944/4797
PYMET_170	Morgenroth, U	1999	Hydrolysis of (Pyridine-5- ¹⁴ C)-labelled CGA 215944 under processing conditions, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, 99MO03, GLP, not published, Syngenta File No CGA215944/4659
PYMET_171	Gasser, A	2002	Residue Study with Pymetrozine (CGA 215944) in or on Tomatoes in Switzerland, including Processing, Syngenta Crop Protection AG, Basel, Switzerland, Syngenta Crop Protection AG, Basel, Switzerland, 1014/01, GLP, not published, Syngenta File No CGA215944/4873
PYMET_172	Joseph, TA	1996	CGA 215944, 50 WP, tomatoes, Ohio, USA,, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Corp., Greensboro, USA, NE-IR-206-94/1, GLP, not published, Syngenta File No CGA215944/0520
PYMET_173	Joseph, TA	1996	CGA 215944, 50 WP, tomatoes, Ohio, USA,, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Corp., Greensboro, USA, NE-IR-206-94/2, GLP, not published, Syngenta File No CGA215944/0521
PYMET_174	Joseph, TA	1996	CGA 215944, 50 WP, tomatoes, California, USA,, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Corp., Greensboro, USA, 02-IR-017-94/1, GLP, not published, Syngenta File No CGA215944/0522
PYMET_175	Joseph, TA	1996	CGA 215944, 50 WP, tomatoes, California, USA,, Novartis Crop Protection AG, Basel, Switzerland, Ciba-Geigy Corp., Greensboro, USA, 02-IR-017-94/2, GLP, not published, Syngenta File No CGA215944/0523
PYMET_176	Tribolet, R	1999	CGA 215944, WP 25, A-8811 B, Tomatoes, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, 1085/98, GLP, not published, Syngenta File No CGA215944/0802
PYMET_177	Gasser, A	2003	Residue Study with Pymetrozine (CGA 215944) in or on Sweet Peppers in Switzerland, including Processing, Syngenta Crop Protection AG, Basel, Switzerland, Novartis Crop Protection AG, Basel, Switzerland, 1013/01, GLP, not published, Syngenta File No CGA215944/4891
PYMET_178	Joseph, TA	1997	CGA 215944 (and metabolites CGA 313124 and CGA 313124), Dairy Cows (Three-level dairy feeding study), Milk, USA, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection Inc., Greensboro, USA, 62-96 / MILK-10X, 3X, 1X, GLP, not published, Syngenta File No CGA215944/0619
PYMET_179	Joseph, TA	1997	CGA 215944 (and metabolites CGA 313124 and CGA 313124), Dairy Cows (Three-level dairy feeding study), Tissues, USA, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection Inc., Greensboro, USA, 62-96 / TISSUES-10X, GLP, not published, Syngenta File No CGA215944/0620
PYMET_180	Joseph, TA	1997	CGA 215944—Magnitude of the residues in meat and milk resulting from the feeding of three levels to dairy cattle, Novartis Crop Protection AG, Basel, Switzerland, Novartis Crop Protection Inc., Greensboro, USA, ABR-97030,

Code	Author	Year	Title, Institute, Report reference
			GLP, not published, Syngenta File No CGA215944/0621
PYMET_181	Dorange, JB	2014	Pymetrozine—Residue Study on Protected Cucumber in the United Kingdom, Poland and Spain in 2013, Syngenta Ltd., Bracknell, United Kingdom, S13-02540, GLP, not published
PYMET_182	Gemrot, F	2014	Pymetrozine—Residue Study on Protected Bell Peppers in the United Kingdom, Northern France and Southern France in 2013, Syngenta Ltd., Bracknell, United Kingdom, S13-02500, GLP, not published
PYMET_183	Gemrot, F	2014	Pymetrozine—Residue Study on Protected Tomatoes in Northern France, the United Kingdom and Spain in 2013, Syngenta Ltd., Bracknell, United Kingdom, S13-02535, GLP, not published

PYRACLOSTROBIN (210)

The first draft was prepared by Dr Ursula Banasiak, Delegate of the Federal Institute for Risk Assessment, Berlin, Germany

EXPLANATION

The fungicide pyraclostrobin belonging to the strobilurin group was first evaluated for toxicology by the JMPR in 2003 where an ADI of 0–0.03 mg/kg bw per day and an ARfD of 0.05 mg/kg bw per day were established. The 2004 JMPR concluded that the residue definition for plant commodities for compliance with MRL values and for dietary risk assessments was parent pyraclostrobin and estimated a number of maximum residue levels. The compound was re-evaluated for residues by the JMPR in 2006, 2011 and 2012.

Pyraclostrobin was listed by the Forty-fifth Session of the CCPR (2013) for the review of an additional MRL for apricots.

The 2011 JMPR withdraw the previous group MRL recommendation of 1 mg/kg for Stone fruits and estimated different maximum residue levels for the individual commodities; cherries, plums, peaches and nectarines, but made no recommendation for apricots. The 2014 JMPR received GAP information and residue trial data for pyraclostrobin in apricots.

USE PATTERN

The information available to the 2014 JMPR on registered uses of pyraclostrobin in stone fruits, apricot, plums, peaches and nectarines is summarized in Table 1. Copies of labels including an English translation, where appropriate, were made available to the Meeting.

Table 1 Registered uses of pyraclostrobin in stone fruits, apricots, plums, peaches and nectarines

Country	Form	Application			Application rate per treatment			PHI, days
		Method	No.	Interval, days	kg ai/hL max	Water L/ha min max	kg ai/ha min max	
Stone fruits								
Canada	WG	Foliar spray	5	7-14	0.013	1000	0.13	10
Canada	WG	Foliar spray	5	7-14	-	-	0.09-0.13	0
Germany	WG	Foliar spray	3	7-14	0.003	max. 500 L/ha or per m crown height	0.02 (per growing metre)	7
USA	WG	Foliar spray	5	7-14	-	-	0.13	0
USA	SC	Foliar spray	3	7-14	-	-	0.06-0.1	0
USA	WG	Foliar spray	5	7-14	-	-	0.09-0.13	0
Apricots								
Belgium	WG	Foliar spray	1-3	10-14	-	-	0.03	7
Czech Republic	WG	Foliar spray	3	-	0.005	1000	0.05	-
France	WG	Foliar spray	3	8-12	0.005	1000	0.04-0.05	3
Greece	WG	Foliar spray	2-3	7-14	0.005	1000	0.04-0.05	7
Hungary	WG	Foliar spray	3	7	0.008	800-1000	0.05-0.07	7
Italy	WG	Foliar spray	3	7-14	0.005	1000	0.04-0.05	3
Japan	WG	Foliar spray	1-2	-	0.012	2000-7000	0.07-0.24	7
Slovakia	WG	Foliar spray	1-3	-	0.005	1000	0.05	7
Plums								
Belgium	WG	Foliar spray	1-3	7-10	-	-	0.03	7
Denmark	WG	Foliar spray	3	-	0.025	200-400	0.05	-
Finland	WG	Foliar spray	2	5-10	0.011	600-1000	0.05-0.07	3
France	WG	Foliar spray	3	8-12	0.005	1000	0.03-0.05	3
Greece	WG	Foliar spray	2-3	7-14	0.005	1000	0.04-0.05	7

Country	Form	Application			Application rate per treatment			PHI, days
		Method	No.	Interval, days	kg ai/hL max	Water L/ha min max	kg ai/ha min max	
Hungary	WG	Foliar spray	3	7	0.008	800-1000	0.05-0.07	7
Italy	WG	Foliar spray	3	7-14	0.005	1000	0.04-0.05	3
Japan	WG	Foliar spray	1-2	-	0.012	2000-7000	0.07-0.24	7
Norway	WG	Foliar spray	2	5-10	0.005	150	0.007	-
Slovakia	WG	Foliar spray	1-3	5-10	0.005	1000	0.05	7
<i>Peaches, nectarines</i>								
Belgium	WG	Foliar spray	1-3	10-14	-	-	0.03	7
France	WG	Foliar spray	3	8-12	0.005	1000	0.04-0.05	3
Greece	WG	Foliar spray	2-3	7-14	0.005	1000	0.04-0.05	7
Hungary	WG	Foliar spray	3	7	0.008	800-1000	0.05-0.07	7
Italy	WG	Foliar spray	3	7-14	0.005	1000	0.04-0.05	3
Japan	WG	Foliar spray	1-2	-	0.012	2000-7000	0.07-0.24	1
Slovakia	WG	Foliar spray	1-3	5-10	0.005	1000	0.05	7

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The current Meeting received information on two field trials of pyraclostrobin residues in apricots in 2013 in Southern Europe. The applications were done by foliar spray three times at rates of 0.05 kg ai/ha at 30–31, 17 and 3 days before harvest. Specimens of apricot fruits were collected at the day of the last application and 3, 6–7 and 13–14 days thereafter. Residues were analysed with method 535/1 (determination via LC/MS, see JMPR 2011) with mean recovery rate of 87.6% for pyraclostrobin at fortification levels from 0.01 to 1.0 mg/kg. Residues were corrected to the whole fruit (stone + flesh) with the pulp/whole fruit correction factor, which was calculated by dividing flesh weight by the weight of the whole fruit. The results are summarized in Table 2.

Table 2 Pyraclostrobin residues in apricots

Country, Year Location (variety) Trial No.	Application				PHI, days	Sample material	Residues, mg/kg	Author, Report Year Study No., DocID
	Form	Method	Rate, kg ai/ha	Spray volume, L/ha				
Italy, 2013 Ravenna (Farbaly) L130390	WG	Foliar	3 × 0.05	1000	0	Fruit	0.11	Martin T. 2013 428270 2013/1349143
					3	Fruit	0.07	
					7	Fruit	<u>0.08</u>	
					14	Fruit	0.04	
Spain, 2013 Sevilla (Lillycot) L130391	WG	Foliar	3 × 0.05	1000	0	Fruit	0.11	Martin T. 2013 428270 2013/1349143
					3	Fruit	<u>0.08</u>	
					6	Fruit	0.06	
					13	Fruit	0.02	

APPRAISAL

Pyraclostrobin was first evaluated for toxicology by the JMPR in 2003 where an ADI of 0–0.03 mg/kg bw per day and an ARfD of 0.05 mg/kg bw per day were established. The 2004 JMPR evaluated the residue behaviour of the fungicide and concluded that the residue definition for both, plant and animal commodities was parent pyraclostrobin for compliance with MRL values as well as for dietary risk assessments. The compound was re-evaluated for residues several times by the JMPR since 2006 and was listed by the 45th Session of the CCPR (2013) for the review of an additional MRL for apricot.

The 2011 JMPR withdraw the formerly group MRL recommendation of 1 mg/kg for stone fruits and estimated different maximum residue levels for the single commodities cherries, plums

(including prunes), peach and nectarine but did no recommendation for apricot. The 2014 JMPR received GAP information and residue data of pyraclostrobin in apricot.

Results of supervised residue trials in crops

Stone fruits

The maximum GAP for the use of pyraclostrobin in stone fruits was from Canada and the USA, consisting of 5×0.13 kg ai/ha and a PHI of 0 days. Labels for alternative use patterns of pyraclostrobin in France and Italy on apricots, plums, peach and nectarine were submitted with application of 3×0.04–0.05 kg ai/ha and a PHI of 3 days.

The 2011 JMPR estimated for pyraclostrobin residues different maximum residue levels, STMRs and HRs for cherries, plums, peach and nectarine. The 2011 Meeting noted that the estimations for peach and nectarine were based on an alternative GAP (France) as the evaluation based on the maximum GAP of the USA exceeded the ARfD. The residue data on which the estimations were based are summarized as follows:

Crop	2011 Estimation, mg/kg			Residue data, mg/kg	Region, application kg ai/ha, PHI
	MRL	STMR	HR		
Cherries	3	0.51	1.57	0.03, 0.27, 0.38, 0.42, 0.47, 0.5, <u>0.51</u> , 0.56, 0.63, 0.82, 1.06, 1.08, 1.57	USA, 5×0.13, 0 days
Plums	0.8	0.09	0.40	0.02, 0.02, 0.04, 0.05, 0.06, 0.07, 0.09, <u>0.09</u> , 0.12, 0.19, 0.22, 0.34, 0.38, 0.40, 0.40	USA, 5 × 0.13, 0 days
Peach, nectarine	0.3	0.07	0.13	< 0.02, 0.03, 0.04, 0.05, <u>0.07</u> , <u>0.07</u> , 0.08, 0.11, 0.12, 0.13	South-EU, 3×0.04–0.05, 3 days

The current Meeting received information on two field trials for pyraclostrobin uses on apricots in 2013 in Italy and Spain. Three foliar sprays at rates of 0.05 kg ai/ha were made matching the French and Italian GAP. The residues were 0.08 (2) mg/kg in fruits at a 3-days PHI.

The Meeting noted that the two residue values on apricots of 0.08 mg/kg are in the same order of magnitude as the residues in peaches matching the alternative European GAP (3×0.04–0.05, PHI 3 days).

The Meeting concluded that the MRL of 0.3 mg/kg, the STMR of 0.51 mg/kg and the HR of 1.57 mg/kg established for cherries should be extrapolated to the whole Stone fruits subgroup 003A Cherries (includes all commodities in this subgroup). The meeting withdrew its previous recommendation of 0.3 mg/kg for the single commodity cherries.

The Meeting decided that the maximum residue level of 0.8 mg/kg, the STMR of 0.09 mg/kg and the HR of 0.40 mg/kg established for plums should be extrapolated to the whole stone fruit subgroup 003B for Plums. The meeting withdrew its previous recommendation of 0.8 mg/kg for the single commodity Plums (including prunes).

The Meeting decided that each maximum residue level of 0.3 mg/kg, the STMR of 0.07 mg/kg and the HR of 0.13 mg/kg established for peaches and nectarines should be extrapolated to the whole stone fruit subgroup 003 C for Peaches (including nectarines and apricots). The meeting withdrew its previous recommendation of 0.3 mg/kg for the individual commodities peaches and nectarines.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for dietary risk assessment.

Definition of the residue (for compliance with the MRL and for estimation of dietary intake) for plant and animal commodities: Pyraclostrobin.

The residue is not fat-soluble.

CCN	Commodity name	MRL, mg/kg		STMR mg/kg	HR mg/kg
		Proposed	Previous		
FS 003C	Peaches (including Nectarine and Apricots) (includes all commodities in this subgroup)	0.3		0.07	0.13
FS 003A	Cherries (includes all commodities in this subgroup)	0.3		0.51	1.57
FS 003B	Plums (includes all commodities in this subgroup)	0.8		0.09	0.40
FS 0013	Cherries	W	0.3		
FS 0014	Plums (including Prunes)	W	0.8		
FS 0245	Nectarine	W	0.3		
FS 0247	Peach	W	0.3		

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of pyraclostrobin were calculated for the 17 GEMS/Food cluster diets using STMRs and STMR-Ps estimated by the JMPR in 2004, 2006, 2011, 2012 and 2014. The results are shown in Annex 3 to the 2014 Report.

The ADI is 0–0.03 mg/kg bw and the calculated IEDIs were 1–6% of the maximum ADI. The Meeting concluded that the long-term intake of residues of pyraclostrobin resulting from the uses considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short Term Intake (IESTI) for pyraclostrobin was calculated by the current Meeting for apricot. The results are shown in Annex 4 to the 2014 Report.

For the commodities considered by the 2014 JMPR, the IESTI represented 0–30% of the ARfD for the general population and 0–40% for children. The Meeting concluded that the short-term intake of residues of pyraclostrobin, when used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

REFERENCES

DocID	Author	Year	Title
2013/1349143	Martin T.	2013	Study on the residue behaviour of pyraclostrobin (BAS 500 F) and boscalid (BAS 510 F) on apricots after the application of BAS 516 07 F under field conditions in Italy and Spain, 2013. Agrologia SLU, Utera, Spain. Unpublished.

SEDAXANE (259)

The first draft was prepared by Dr Michael Doherty, United States Environmental Protection Agency, Washington, DC, USA

EXPLANATION

Sedaxane is a mixture of two *cis* isomers 2'-[(1*RS*,2*RS*)-bicycloprop-2-yl]-3-(difluoromethyl)-1-methylpyrazole-4-carboxanilide, and two *trans* isomers 2'-[(1*RS*,2*SR*)-bicycloprop-2-yl]-3-(difluoromethyl)-1-methylpyrazole-4-carboxanilide (IUPAC). This fungicide was first evaluated by the JMPR in 2012, the Meeting derived an ADI of 0–0.1 mg/kg bw and an ARfD of 0.3 mg/kg bw. The Meeting determined that the residue definitions for MRL compliance and the estimation of dietary intake, for both plant and animal commodities, is parent sedaxane and that the residue is fat-soluble.

Sedaxane was listed by the Forty-fifth Session of the CCPR for the evaluation of additional maximum residue levels. The 2014 Meeting received residue data reflecting use of sedaxane as a seed treatment on corn/maize, pulses, potatoes, and sorghum. In addition to evaluating those crops, the Meeting was asked to consider use on rice, without residue data, on the basis of residue data supplied to the 2012 and 2014 Meetings for other grains.

RESIDUE ANALYSIS

Analytical Methods

The analytical method used in the residue and processing studies is the same as that reviewed and deemed acceptable for data collection and enforcement purposes by the 2012 Meeting (Specht method P-14.141). The reported LOQ is 0.005 mg/kg for the *cis* isomers and the *trans* isomers, each, with an overall LOQ of 0.01 mg/kg in plant and animal matrices.

USE PATTERN

Information on the registered uses of sedaxane for the relevant crops, along with copies of labels, were provided to the 2014 Meeting (Table 1).

Table 1 Registered uses of sedaxane for corn/maize, pulses, potatoes, rice, and sorghum

Crop	Country	Formulation		Application				PHI (days)
		g ai/L or g ai/kg	Type	Method	g ai/100 kg seed	Water L/ha	Number	
Beans (dry)	Canada	500	FS	Seed treatment	5	n/a	1	n/a
Beans (dry)	USA	500	FS	Seed treatment	5	n/a	1	n/a
Lentils	USA	500	FS	Seed treatment	5	n/a	1	n/a
Peas (dry)	Canada	500	FS	Seed treatment	5	n/a	1	n/a
Peas (dry)	USA	500	FS	Seed treatment	5	n/a	1	n/a
Potatoes	Canada	500	FS	Seed treatment	2.5	n/a	1	n/a
Maize	Argentina	500	FS	Seed treatment	10	n/a	1	n/a
Maize (corn)	Canada	500	FS	Seed treatment	12.5	n/a	1	n/a
Maize	Chile	500	FS	Seed treatment	50	n/a	1	n/a
Maize (corn)	USA	500	FS	Seed treatment	40	n/a	1	n/a
Pop corn	USA	500	FS	Seed treatment	40	n/a	1	n/a
Sweet corn	Canada	500	FS	Seed treatment	12.5	n/a	1	n/a
Sweet corn	USA	500	FS	Seed treatment	40	n/a	1	n/a
Rice	Uruguay	50	FS	Seed treatment	10	n/a	1	n/a
Sorghum	Canada	500	FS	Seed treatment	5	n/a	1	n/a
Sorghum	USA	500	FS	Seed treatment	5	n/a	1	n/a

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Residue trials were reported with sufficient details for evaluation, including field conditions, application parameters, planting dates, storage conditions and durations, and procedural recoveries. Separate residue values are reported, as measured, for the *cis* and *trans* isomers of sedaxane. Generally, two samples were collected from treated plots and one sample from control plots. Mean residue values suitable for maximum residue level and STMR estimation are underlined in the tables. For all crops and matrices except potato, residues of each isomer were < 0.005 mg/kg (i.e., < 0.01 mg/kg combined) in all samples.

Storage stability data were not submitted to the 2014 Meeting. Data evaluated by the 2012 Meeting showed that residues of sedaxane are stable under frozen conditions for at least 2 years in wheat grain and straw, spinach, potato, orange, lentils, and soya beans. The 2014 Meeting has translated those storage stability data to the pulse, potato, corn, and sorghum samples in this evaluation.

The 2014 Meeting received data from supervised trials depicting residues in the following crops grown from treated seeds:

Commodity	Group	Table No.
Sweet Corn	VO Fruiting Vegetables, other than Cucurbits	2
Beans, dry	VD Pulses	3
Pea, dry	VD Pulses	4
Potato	VR Root and tuber vegetables	5
Maize, grain	GC Cereal grains	6
Sorghum, grain	GC Cereal grains	7
Bean, forage and hay	AL Legume animal feeds	8
Pea, hay	AL Legume animal feeds	9
Maize, fodder, forage	AS Straw, fodder and forage of cereal grains and grasses	10
Sorghum, fodder, forage	AS Straw, fodder and forage of cereal grains and grasses	11

Fruiting Vegetables, other than Cucurbits

Sweet Corn (Corn-on-the-cob)

Thirteen supervised trials were conducted on corn in Canada and the USA during the 2008 (USA) and 2010 (Canada) growing seasons. Of these, six were conducted with sweet corn varieties (* in table); for the remaining seven trials, ears were harvested prior to maturity to simulate sweet corn. Sedaxane was applied as a seed treatment at a nominal rate of 40 g ai/100 kg seed. Commodities were collected at normal commercial harvest and were stored frozen for up to 10 months (0.8 years). Trials conducted in the same location and with the same variety were not considered to be independent.

Table 2 Results of residue trials on sweet corn

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DAT	Residues (mg/kg)	Mean residue (mg/kg)	Report ID, remarks
cGAP: USA	--	40	--		--	--	--
Canada (2010) Abbotsford, BC (T802)	Peaches Cream*	& 30	19	106	< 0.01, < 0.01	< 0.01	CER 05720/10
Canada (2010) Branchton, ON (T800)	Peaches Cream*	& 30	16	79	< 0.01, < 0.01	< 0.01	CER 05720/10
Canada (2010) Taber, AB (T801)	Peaches Cream*	& 30	15	121	< 0.01, < 0.01	< 0.01	CER 05720/10
USA (2008) Aromas, (W27CA081484)	Jubilee*	50	n.s.	Matu- ri- ty	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008)	NK	47	n.s.	82	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DAT	Residues (mg/kg)	Mean residue (mg/kg)	Report ID, remarks
Athens, GA (E12GA081463)	70-C7- GT/CB/LL						
USA (2008) Fitchburg, WI (C08WI081469)	NK 40T- GT/CB/LL	41	n.s.	85	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Germansville, PA (E04PA081461)	NK 40T- GT/CB/LL	41	n.s.	70	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Highland, KS (C19KS081466)	83F11- GT/CB/LL	51	n.s.	92	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Hillsboro, OR (W21OR081486)	Jubilee*	50	n.s.	Matu- ri- ty	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Oregon, MS (C19MO081465)	83F11- GT/CB/LL	51	n.s.	90	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Richwood, OH (C01OH081468)	H8212- GT/CB/LL	40	n.s.	106	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Rupert, ID (W15ID081485)	Jubilee* C4274MF	50	n.s.	Matu- ri- ty	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Saginaw, MI (C01MI081467)	H8212- GT/CB/LL	40	n.s.	88	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07

* Sweet corn variety

Pulses

Beans, dry

Thirteen independent supervised trials were conducted in dry beans in Canada and the USA during the 2010 growing season. Sedaxane was applied as a seed treatment at a nominal rate of 5 g ai/100 kg seed, except for two trials (Fresno, CA; Parkdale, OR) in which the application rate was ca. 12 g ai/100 kg seed. Commodities were collected at normal commercial harvest and were stored frozen for up to 402 days (1.1 years).

Table 3 Results of residue trials in dry beans

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DA T	Residues (mg/kg)	Mean residue (mg/kg)	Report ID, remarks
cGAP: USA/Canada	--	5	--		--	--	--
Canada (2010) St. George, ON (T811)	Pintoba	4.5	17	107	< 0.01, < 0.01	<u>< 0.01</u>	CER 05722/10
Canada (2010) Branchton, ON (T812)	Navy	4.5	9	101	< 0.01, < 0.01	<u>< 0.01</u>	CER 05722/10
Canada (2010) Elm Creek, MB (T813)	Envoy (Navy)	4.5	85	134	< 0.01, < 0.01	<u>< 0.01</u>	CER 05722/10
Canada (2010) St. Marc-sur-Richelieu, QC (T814)	Envoy (Navy)	4.5	41	128	< 0.01, < 0.01	<u>< 0.01</u>	CER 05722/10
Canada (2010) Taber, AB (T815)	Pintoba	5.4	53	148	< 0.01, < 0.01	<u>< 0.01</u>	CER 05722/10
USA (2010) Carrington, ND (C13- 0665)	Pinto Lapaz	4.4	62	114	< 0.01, < 0.01	<u>< 0.01</u>	TK0024902

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DA T	Residues (mg/kg)	Mean residue (mg/kg)	Report remarks ID,
USA (2010) Eaton, CO (W12-0666)	Pinto Lapaz	4.4	47	105	< 0.01, < 0.01	<u>< 0.01</u>	TK0024902
USA (2010) Wellington, CO (W12-0667)	Pinto Lapaz	4.4	56	106	< 0.01, < 0.01	<u>< 0.01</u>	TK0024902
USA (2010) Fresno, CA (E19-0668)	Foxfire LRK	13	43	106	< 0.01, < 0.01	< 0.01	TK0024902
USA (2010) Parkdale, OR (W20-0669)	Foxfire LRK	13	88	105	< 0.01, < 0.01	< 0.01	TK0024902
USA (2010) Parkdale, OR (W20-0672)	Banner	6.3	83	102	< 0.01, < 0.01	<u>< 0.01</u>	TK0024902
USA (2010) Hermiston, OR (W21-0671)	Banner	6.3	98	75	< 0.01, < 0.01	<u>< 0.01</u>	TK0024902
USA (2010) Hughson, CA (W26-0670)	Banner	6.3	41	104	< 0.01, < 0.01	<u>< 0.01</u>	TK0024902

Pea, dry

Ten independent supervised trials were conducted in dry pea in Canada and the USA during the 2010 growing season. Sedaxane was applied as a seed treatment at a nominal rate of 5 g ai/100 kg seed. Commodities were collected at normal commercial harvest and were stored frozen for up to 401 days (1.1 years).

Table 4 Results of residue trials in dry peas

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DA T	Residues (mg/kg)	Mean residue (mg/kg)	Report remarks ID,
cGAP: USA/Canada	--	5	--		--	--	--
Canada (2010) Portage la Prairie, MB (T803)	CDC Meadow	5	179	91	< 0.01, < 0.01	<u>< 0.01</u>	CER 05721/10
Canada (2010) Elm Creek, MB (T804)	Golden	5	177	92	< 0.01, < 0.01	<u>< 0.01</u>	CER 05721/10
Canada (2010) Minto, MB (T805)	Golden	5	181	88	< 0.01, < 0.01	<u>< 0.01</u>	CER 05721/10
				95	< 0.01, < 0.01	<u>< 0.01</u>	CER 05721/10
				102	< 0.01, < 0.01	<u>< 0.01</u>	CER 05721/10
				110	< 0.01, < 0.01	<u>< 0.01</u>	CER 05721/10
				115	< 0.01, < 0.01	<u>< 0.01</u>	CER 05721/10
Canada (2010) Minto, MB (T806)	CDC Meadow	5	181	102	< 0.01, < 0.01	<u>< 0.01</u>	CER 05721/10
Canada (2010) Boisservain, MB (T807)	CDC Meadow	5	179	98	< 0.01, < 0.01	<u>< 0.01</u>	CER 05721/10
Canada (2010) Rosthern, SK (T808)	Golden	5	116	116	< 0.01, < 0.01	<u>< 0.01</u>	CER 05721/10
Canada (2010) Blaine Lake, SK (T809)	CDC Meadow	5	131	128	< 0.01, < 0.01	<u>< 0.01</u>	CER 05721/10
Canada (2010) Alvena, SK (T810)	Golden	5	225	131	< 0.01, < 0.01	<u>< 0.01</u>	CER 05721/10
USA (2010) Gardner, ND (C12-0674)	Ariel	3.9	132	94	< 0.01, < 0.01	<u>< 0.01</u>	TK0024902
USA (2010) Ephrata, WA (W18-0673)	Ariel	3.9	212	87	< 0.01, < 0.01	<u>< 0.01</u>	TK0024902

*Root and Tuber Vegetables**Potato*

Thirty-one independent supervised trials were conducted in potato in Canada and the USA during the 2010 growing season. Sedaxane was applied as a seed treatment at a nominal rate of 2.5 g ai/100 kg seed, except for trials in Campbell, MN and Rupert, ID in which the application rates were 4.5 and 5.2 g ai/100 kg seed, respectively. Commodities were collected at normal commercial harvest and were stored frozen for up to 292 days (0.8 years).

Table 5 Results of residue trials in potato

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DA T	Residues (mg/kg)	Mean residue (mg/kg)	Report ID, remarks
cGAP: Canada	--	2.5	--		--	--	--
Canada (2011) New Glasgow, PE (T900)	Ranger Russet	2.5	2190	95	< 0.01, < 0.01	< 0.01	CER 05724/11
				97	< 0.01, < 0.01	< 0.01	CER 05724/11
				102	< 0.01, < 0.01	< 0.01	CER 05724/11
				106	< 0.01, < 0.01	< 0.01	CER 05724/11
				111	< 0.01, < 0.01	< 0.01	CER 05724/11
Canada (2011) New Glasgow, PE (T901)	Yukon Gold	2.5	1850	87	< 0.01, < 0.01	< 0.01	CER 05724/11
				93	< 0.01, < 0.01	< 0.01	CER 05724/11
Canada (2011) New Glasgow, PE (T902)	Shepody	2.5	1940	78	< 0.01, < 0.01	< 0.01	CER 05724/11
				84	< 0.01, < 0.01	< 0.01	CER 05724/11
Canada (2011) Brackley Beach, PE (T903)	Russet Burbank	2.5	1910	95	< 0.01, < 0.01	< 0.01	CER 05724/11
				97	< 0.01, < 0.01	< 0.01	CER 05724/11
				102	< 0.01, < 0.01	< 0.01	CER 05724/11
				106	< 0.01, < 0.01	< 0.01	CER 05724/11
				111	< 0.01, < 0.01	< 0.01	CER 05724/11
Canada (2011) Brackley Beach, PE (T904)	Green Mountain	2.5	1910	95	< 0.01, < 0.01	< 0.01	CER 05724/11
				102	< 0.01, < 0.01	< 0.01	CER 05724/11
Canada (2011) Malden, NB (T905)	Yukon Gold	2.5	2000	85	< 0.01, < 0.01	< 0.01	CER 05724/11
				86	< 0.01, < 0.01	< 0.01	CER 05724/11
				92	< 0.01, < 0.01	< 0.01	CER 05724/11
Canada (2011) Malden, NB (T906)	Norland	2.5	1990	86	< 0.01, < 0.01	< 0.01	CER 05724/11
				92	< 0.01, < 0.01	< 0.01	CER 05724/11
Canada (2011) St. Marc-sur-Richelieu, QC (T907)	Chieftan	2.5	2000	88	< 0.01, < 0.01	< 0.01	CER 05724/11
				94	< 0.01, < 0.01	< 0.01	CER 05724/11
Canada (2011) St. Marc-sur-Richelieu, QC (T908)	Kennebec	2.5	2000	93	< 0.01, < 0.01	< 0.01	CER 05724/11
				99	< 0.01, < 0.01	< 0.01	CER 05724/11
Canada (2011) Branchton, ON (T909)	Chieftan	2.5	2675	99	< 0.01, < 0.01	< 0.01	CER 05724/11
				104	< 0.01, < 0.01	< 0.01	CER 05724/11
Canada (2011) Branchton, ON (T910)	Gold Rush	2.5	2675	98	< 0.01, < 0.01	< 0.01	CER 05724/11
				104	< 0.01, < 0.01	< 0.01	CER 05724/11
Canada (2011)	Bintje	2.5	2240	110	< 0.01, < 0.01	< 0.01	CER 05724/11

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DA T	Residues (mg/kg)	Mean residue (mg/kg)	Report ID, remarks
Taber, AB (T912)				117	< 0.01, < 0.01	< 0.01	CER 05724/11
Canada (2011) Taber, AB (T913)	Sangre	2.5	2700	110	< 0.01, < 0.01	< 0.01	CER 05724/11
				117	< 0.01, < 0.01	< 0.01	CER 05724/11
USA (2011) North Rose, NY (TK0049813-01)	Genesee	2.6	2272	98	< 0.0075, < 0.005	< 0.0063	TK0049813
USA (2011) Germansville, PA (TK0049813-02)	Chieftan	2.6	3388	83	< 0.0075, < 0.0075	< 0.0075	TK0049813
USA (2011) Seven Springs, NC (TK0049813-03)	Red LaSoda	1.1	2380	109	< 0.0075, < 0.0075	< 0.0075	TK0049813
USA (2011) Oviedo, FL (TK0049813-04)	Red LaSoda	2.6	3256	82	< 0.01028, < 0.0075	< 0.0089	TK0049813
USA (2011) Rice, MN (TK0049813-05)	Russet Norkotah Sel 8	2.5	1074	129	< 0.0075, < 0.0075	< 0.0075	TK0049813
USA (2011) Gardner, ND (TK0049813-06)	Red LaSoda	2.5	1307	82	< 0.0075, < 0.0075	< 0.0075	TK0049813
USA (2011) Jerome, ID (TK0049813-07)	Yukon Gold	2.6	4249	105	< 0.0075, < 0.0075	< 0.0075	TK0049813
USA (2011) Madera, CA (TK0049813-08)	Dark Red Norland G3	2.2	2559	129	< 0.0082, < 0.0075	< 0.0079	TK0049813
USA (2011) Jerome, ID (TK0049813-09)	Ranger Russet	2.6	4019	130	< 0.0075, < 0.0085	< 0.008	TK0049813
USA (2011) American Falls, ID (TK0049813-10)	Russet Burbank	2.5	2332	103	< 0.0075, < 0.0075	< 0.0075	TK0049813
USA (2011) Ephrata, WA (TK0049813-11)	Russet Burbank	2.6	4032	111	< 0.0075, < 0.0075	< 0.0075	TK0049813
USA (2011) American Falls, ID (TK0049813-12)	Dark Red Norland	2.5	2302	83	< 0.0075, < 0.0075	< 0.0075	TK0049813
USA (2011) Campbell, MN (TK0049813-13)	Red Norland	2.6	2829	66 ^a	< 0.0195, < 0.0155	< 0.018	TK0049813
				71	< 0.016, < 0.014	< 0.015	TK0049813
				76	< 0.0091, < 0.0075	< 0.0083	TK0049813
				81	< 0.0075, < 0.0075	< 0.0075	TK0049813
USA (2011) Ephrata, WA (TK0049813-14)	Umatilla	2.6	4053	98	< 0.0075, < 0.005	< 0.0063	TK0049813
				103	< 0.005, < 0.005	< 0.005	TK0049813
				108	< 0.0075, < 0.0075	< 0.0075	TK0049813
				113	< 0.0075, < 0.01	< 0.0088	TK0049813
USA (2011) Campbell, MN (TK0049813-15(1))	Kennebec	2.6	3025	128	< 0.01, < 0.01	< 0.01	TK0049813

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DA T	Residues (mg/kg)	Mean residue (mg/kg)	Report ID, remarks
USA (2011) Campbell, MN (TK0049813-15(2))	Kennebec	5.2	3102	128	< 0.01, < 0.01	< 0.01	TK0049813
USA (2011) Rupert, ID (TK0049813- 16(1))	Russet Burbank	2.7	2806	115	< 0.01, < 0.01	< 0.01	TK0049813
USA (2011) Rupert, ID (TK0049813- 16(2))	Russet Burbank	4.5	3926	115	< 0.01, < 0.01	< 0.01	TK0049813

^a Immature tubers

Cereal Grains

Maize and popcorn

Twenty supervised trials were conducted in corn in Canada and the USA during the 2008 (USA) and 2010 (Canada) growing seasons. Of these, one trial was conducted with popcorn. Sedaxane was applied as a seed treatment at a nominal rate of 40 g ai/100 kg seed. Two trials were conducted at an exaggerated rate (120 g ai/100 kg seed). Commodities were collected at normal commercial harvest and were stored frozen for up to 10 months (0.8 years). Trials conducted in the same location and with the same variety were not considered to be independent.

Table 6 Results of residue trials in maize

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DAT	Residues (mg/kg)	Mean residue (mg/kg)	Report ID, remarks
cGAP: USA	--	40	--		--	--	--
USA (2008) Athens, GA (E12GA081463)	NK 70-C7- GT/CB/LL	47	n.s.	120	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Bagley, IA (C30IA081479)	NK 70-C7- GT/CB/LL	47	n.s.	167	< 0.01, < 0.01	< 0.01	T006043-07
		120	n.s.	167	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Claude, TX (E13TX081483)	A3035	48	n.s.	210	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Fitchburg, WI (C08WI081469)	NK 40T- GT/CB/LL	41	n.s.	154	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Fitchburg, WI (C08WI081470)	NK 40T- GT/CB/LL	41	n.s.	161	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Fitchburg, WI (C08WI081471)	NK 40T- GT/CB/LL	41	n.s.	161	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Germansville, PA (E04PA081461)	NK 40T- GT/CB/LL	41	n.s.	133	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Highland, KS (C19KS081466)	83F11- GT/CB/LL	51	n.s.	147	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Lime Springs, IA (E19IA081476)	NK 40T- GT/CB/LL	41	n.s.	149	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Lime Springs, IA	NK 40T-	41	n.s.	148	< 0.01, < 0.01	< 0.01	T006043-07

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DAT	Residues (mg/kg)	Mean residue (mg/kg)	Report ID, remarks
(E19IA081477)	GT/CB/LL						
USA (2008) Lime Springs, IA (E19MN081478)	NK 40T- GT/CB/LL	41	n.s.	143	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Northwood, ND (C13ND081472)	N23F- GT/CB/LL	40	n.s.	177	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Northwood, ND (C13ND081473)	N23F- GT/CB/LL	40	n.s.	175	< 0.01, < 0.01	< 0.01	T006043-07
		120	n.s.	175	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Oregon, MS (C19MO081465)	83F11- GT/CB/LL	51	n.s.	153	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Perry, IA (C30IA081480)	NK 70-C7- GT/CB/LL	47	n.s.	128	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
				135	< 0.01, < 0.01	< 0.01	T006043-07
				142	< 0.01, < 0.01	< 0.01	T006043-07
				149	< 0.01, < 0.01	< 0.01	T006043-07
				156	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Richland, IA (C18IA081474)	H8212- GT/CB/LL	40	n.s.	130	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
				137	< 0.01, < 0.01	< 0.01	T006043-07
				144	< 0.01, < 0.01	< 0.01	T006043-07
				151	< 0.01, < 0.01	< 0.01	T006043-07
				158	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Richland, IA (C18IA081475)	H8212- GT/CB/LL	40	n.s.	141	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Richwood, OH (C01OH081468)	H8212- GT/CB/LL	40	n.s.	171	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Saginaw, MI (C01MI081467)	H8212- GT/CB/LL	40	n.s.	152	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Wharton, TX (W05TX081482)	83F11- GT/CB/LL	51	n.s.	113	< 0.01, < 0.01	< 0.01	T006043-07

n.s.: not specified

Sorghum

Twelve supervised trials were conducted in sorghum in the USA during the 2010 growing season. Sedaxane was applied as a seed treatment at a nominal rate of 40 g ai/100 kg seed. Commodities were collected at normal commercial harvest and were stored frozen for up to 9.5 months (0.8 years). Trials conducted in the same location and with the same variety were not considered to be independent.

Table 7 Results of residue trials in sorghum

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DA T	Residues (mg/kg)	Mean residue (mg/kg)	Report ID, remarks
cGAP: USA/Canada	--	5	--		--	--	--
USA (2010) Bagley, IA (C30-0684)	NK6638	40	8	157	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901
USA (2010) Cheneyville, LA (E17-	NK6638	40	7	114	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DA T	Residues (mg/kg)	Mean residue (mg/kg)	Report ID, remarks
0682)							
USA (2010) Eaton, CO (W12-0690)	WGF	39	12	168	< 0.01, < 0.01	<u>≤ 0.01</u>	TK0024901
USA (2010) Elko, SC (E11-0681)	NK7829	42	4	107	< 0.01, < 0.01	<u>≤ 0.01</u>	TK0024901
USA (2010) Jamestown, ND (C12-0689)	NK7829	42	5	144	< 0.01, < 0.01	<u>≤ 0.01</u>	TK0024901
USA (2010) LaPlata, IA (C18-0686)	NK6638	40	9	138	< 0.01, < 0.01	<u>≤ 0.01</u>	TK0024901
USA (2010) Levelland, TX (W39-0692)	NK7829	42	8	124	< 0.01, < 0.01	<u>≤ 0.01</u>	TK0024901
USA (2010) Raymondville, TX (W08-0688)	NK7829	42	8	149	< 0.01, < 0.01	<u>≤ 0.01</u>	TK0024901
USA (2010) Richland, IA (C18-0683)	NK6638	40	9	125	< 0.01, < 0.01	<u>≤ 0.01</u>	TK0024901
USA (2010) Richland, IA (C18-0685)	NK6638	40	9	132	< 0.01, < 0.01	< 0.01	TK0024901
USA (2010) Uvalde, TX (W07-0687)	NK7829	42	4	114	< 0.01, < 0.01	< 0.01	TK0024901
USA (2010) Uvalde, TX (W07-0691)	NK7829	42	8	147	< 0.01, < 0.01	<u>≤ 0.01</u>	TK0024901

Legume animal feeds

Bean (forage and hay)

Seven independent supervised trials were conducted in beans in Canada and the USA during the 2010 growing season. Sedaxane was applied as a seed treatment at a nominal rate of 5 g ai/100 kg seed, except for two trials (Fresno, CA; Parkdale, OR) in which the application rate was ca. 12 g ai/100 kg seed. Commodities were collected at normal commercial harvest and were stored frozen for up to 402 days (1.1 years). Forage was collected from only five of the seven trials.

Table 8 Results of residue trials in bean forage and hay

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DAT	Residues (mg/kg)	Mean residue (mg/kg)	Report ID, remarks
cGAP: USA/Canada	--	5	--		--	--	--
Forage							
Canada (2010) St. George, ON (T811)	Pintoba	4.5	17	44	< 0.01, < 0.01	<u>≤ 0.01</u>	CER 05722/10
Canada (2010) Branchton, ON (T812)	Navy	4.5	9	38	< 0.01, < 0.01	<u>≤ 0.01</u>	CER 05722/10
USA (2010) Carrington, ND (C13-0665)	Pinto Lapaz	4.4	62	56	< 0.01, < 0.01	<u>≤ 0.01</u>	TK0024902
USA (2010) Eaton, CO (W12-0666)	Pinto Lapaz	4.4	47	105	< 0.01, < 0.01	<u>≤ 0.01</u>	TK0024902
USA (2010) Fresno, CA (E19-0668)	Foxfire LRK	13	43	93	< 0.01, < 0.01	< 0.01	TK0024902
Hay							
Canada (2010) St. George, ON (T811)	Pintoba	4.5	17	76	< 0.01, < 0.01	<u>≤ 0.01</u>	CER 05722/10
Canada (2010) Branchton, ON (T812)	Navy	4.5	9	73	< 0.01, < 0.01	<u>≤ 0.01</u>	CER 05722/10

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DAT	Residues (mg/kg)	Mean residue (mg/kg)	Report remarks ID,
USA (2010) Carrington, ND (C13-0665)	Pinto Lapaz	4.4	62	87	< 0.01, < 0.01	<u>≤ 0.01</u>	TK0024902
USA (2010) Eaton, CO (W12-0666)	Pinto Lapaz	4.4	47	105	< 0.01, < 0.01	<u>≤ 0.01</u>	TK0024902
USA (2010) Fresno, CA (E19-0668)	Foxfire LRK	13	43	106	< 0.01, < 0.01	< 0.01	TK0024902
USA (2010) Parkdale, OR (W20-0672)	Banner	6.3	83	102	< 0.01, < 0.01	<u>≤ 0.01</u>	TK0024902
USA (2010) Hughson, CA (W26-0670)	Banner	6.3	41	87	< 0.01, < 0.01	<u>≤ 0.01</u>	TK0024902

Pea (hay)

Nine independent supervised trials were conducted in pea in Canada and the USA during the 2010 growing season. Sedaxane was applied as a seed treatment at a nominal rate of 5 g ai/100 kg seed. Hay was collected at normal commercial harvest and was stored frozen for up to 401 days (1.1 years).

Table 9 Results of residue trials in pea hay

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DA T	Residues (mg/kg)	Mean residue (mg/kg)	Report remarks ID,
cGAP: USA/Canada	--	5	--	--	--	--	--
Canada (2010) Portage la Prairie, MB (T803)	CDC Meadow	5	179	61	< 0.01, < 0.01	<u>≤ 0.01</u>	CER 05721/10
Canada (2010) Elm Creek, MB (T804)	Golden	5	177	61	< 0.01, < 0.01	<u>≤ 0.01</u>	CER 05721/10
Canada (2010) Minto, MB (T805)	Golden	5	181	46	< 0.01, < 0.01	<u>≤ 0.01</u>	CER 05721/10
				53	< 0.01, < 0.01	< 0.01	CER 05721/10
				60	< 0.01, < 0.01	< 0.01	CER 05721/10
				67	< 0.01, < 0.01	< 0.01	CER 05721/10
				74	< 0.01, < 0.01	< 0.01	CER 05721/10
Canada (2010) Minto, MB (T806)	CDC Meadow	5	181	60	< 0.01, < 0.01	<u>≤ 0.01</u>	CER 05721/10
Canada (2010) Boisservain, MB (T807)	CDC Meadow	5	179	60	< 0.01, < 0.01	<u>≤ 0.01</u>	CER 05721/10
Canada (2010) Rosthern, SK (T808)	Golden	5	116	59	< 0.01, < 0.01	<u>≤ 0.01</u>	CER 05721/10
Canada (2010) Blaine Lake, SK (T809)	CDC Meadow	5	131	60	< 0.01, < 0.01	<u>≤ 0.01</u>	CER 05721/10
Canada (2010) Alvena, SK (T810)	Golden	5	225	59	< 0.01, < 0.01	<u>≤ 0.01</u>	CER 05721/10
USA (2010) Gardner, ND (C12-0674)	Ariel	3.9	132	94	< 0.01, < 0.01	<u>≤ 0.01</u>	TK0024902

Straw, fodder, and forage of cereal grains

Maize

Twenty-three supervised trials were conducted in maize, from which animal feeds were harvested, in Canada and the USA during the 2008 (USA) and 2010 (Canada) growing seasons. Sedaxane was applied as a seed treatment at a nominal rate of 40 g ai/100 kg seed. Commodities were collected at

normal commercial harvest and were stored frozen for up to 10 months (0.8 years). Trials conducted in the same location and with the same variety were not considered to be independent.

Table 10 Results of residue trials in maize

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DAT	Residues (mg/kg)	Mean residue (mg/kg)	Report ID, remarks
cGAP: USA	--	40	--		--	--	--
Fodder (stover)							
USA (2008) Aromas, (W27CA081484)	CA Jubilee	50	n.s.	Maturity	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Athens, (E12GA081463)	GA NK 70-C7- GT/CB/LL	47	n.s.	120	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Bagley, (C30IA081479)	IA NK 70-C7- GT/CB/LL	47	n.s.	167	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Claude, (E13TX081483)	TX A3035	48	n.s.	210	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Fitchburg, (C08WI081469)	WI NK 40T- GT/CB/LL	41	n.s.	154	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Fitchburg, (C08WI081470)	WI NK 40T- GT/CB/LL	41	n.s.	161	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Fitchburg, (C08WI081471)	WI NK 40T- GT/CB/LL	41	n.s.	161	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Germansville, (E04PA081461)	PA NK 40T- GT/CB/LL	41	n.s.	133	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Highland, (C19KS081466)	KS 83F11- GT/CB/LL	51	n.s.	147	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Hillsboro, (W21OR081486)	OR Jubilee	50	n.s.	Maturity	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Lime Springs, (E19IA081476)	IA NK 40T- GT/CB/LL	41	n.s.	149	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Lime Springs, (E19IA081477)	IA NK 40T- GT/CB/LL	41	n.s.	148	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Lime Springs, (E19MN081478)	IA NK 40T- GT/CB/LL	41	n.s.	143	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Northwood, (C13ND081472)	ND N23F- GT/CB/LL	40	n.s.	177	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Northwood, (C13ND081473)	ND N23F- GT/CB/LL	40	n.s.	175	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Oregon, (C19MO081465)	MS 83F11- GT/CB/LL	51	n.s.	153	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Perry, (C30IA081480)	IA NK 70-C7- GT/CB/LL	47	n.s.	128	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
				135	< 0.01, < 0.01	< 0.01	T006043-07
				142	< 0.01, < 0.01	< 0.01	T006043-07
				149	< 0.01, < 0.01	< 0.01	T006043-07
				156	< 0.01, < 0.01	< 0.01	T006043-07

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DAT	Residues (mg/kg)	Mean residue (mg/kg)	Report ID, remarks
USA (2008) Richland, (C18IA081474)	IA H8212- GT/CB/LL	40	n.s.	130	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
				137	< 0.01, < 0.01	< 0.01	T006043-07
				144	< 0.01, < 0.01	< 0.01	T006043-07
				151	< 0.01, < 0.01	< 0.01	T006043-07
				158	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Richland, (C18IA081475)	IA H8212- GT/CB/LL	40	n.s.	141	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Richwood, (C01OH081468)	OH H8212- GT/CB/LL	40	n.s.	171	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Rupert, (W15ID081485)	ID Jubilee C4274MF	50	n.s.	Maturi ty	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Saginaw, (C01MI081467)	MI H8212- GT/CB/LL	40	n.s.	152	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Wharton, (W05TX081482)	TX 83F11- GT/CB/LL	51	n.s.	113	< 0.01, < 0.01	< 0.01	T006043-07
Forage							
Canada (2010) Abbotsford, BC (T802)	Peaches & Cream	30	19	106	< 0.01, < 0.01	<u>< 0.01</u>	CER 05720/10
Canada (2010) Branchton, ON (T800)	Peaches & Cream	30	16	79	< 0.01, < 0.01	<u>< 0.01</u>	CER 05720/10
Canada (2010) Taber, AB (T801)	Peaches & Cream	30	15	121	< 0.01, < 0.01	<u>< 0.01</u>	CER 05720/10
USA (2008) Aneta, (C13ND081481)	ND N23F- GT/CB/LL	40	n.s.	60	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Athens, (E12GA081463)	GA NK 70-C7- GT/CB/LL	47	n.s.	60	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Bagley, (C30IA081479)	IA NK 70-C7- GT/CB/LL	47	n.s.	60	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Fitchburg, (C08WI081469)	WI NK 40T- GT/CB/LL	41	n.s.	64	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Fitchburg, (C08WI081470)	WI NK 40T- GT/CB/LL	41	n.s.	60	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Fitchburg, (C08WI081471)	WI NK 40T- GT/CB/LL	41	n.s.	63	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Germansville, (E04PA081461)	PA NK 40T- GT/CB/LL	41	n.s.	60	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Highland, (C19KS081466)	KS 83F11- GT/CB/LL	51	n.s.	60	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Lime Springs, (E19IA081476)	IA NK 40T- GT/CB/LL	41	n.s.	60	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Lime Springs, (E19IA081477)	IA NK 40T- GT/CB/LL	41	n.s.	60	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Lime Springs, (E19MN081478)	IA NK 40T- GT/CB/LL	41	n.s.	66	< 0.01, < 0.01	< 0.01	T006043-07

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DAT	Residues (mg/kg)	Mean residue (mg/kg)	Report ID, remarks
USA (2008) Northwood, (C13ND081472)	ND N23F- GT/CB/LL	40	n.s.	59	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Northwood, (C13ND081473)	ND N23F- GT/CB/LL	40	n.s.	59	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Oregon, (C19MO081465)	MS 83F11- GT/CB/LL	51	n.s.	61	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Perry, (C30IA081480)	IA NK 70-C7- GT/CB/LL	47	n.s.	31	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Perry, (C30IA081480)	IA NK 70-C7- GT/CB/LL	47	n.s.	40	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
				50	< 0.01, < 0.01	< 0.01	T006043-07
				59	< 0.01, < 0.01	< 0.01	T006043-07
				69	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Richland, (C18IA081474)	IA H8212- GT/CB/LL	40	n.s.	31	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
				41	< 0.01, < 0.01	< 0.01	T006043-07
				50	< 0.01, < 0.01	< 0.01	T006043-07
				61	< 0.01, < 0.01	< 0.01	T006043-07
				70	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Richland, (C18IA081475)	IA H8212- GT/CB/LL	40	n.s.	60	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Richwood, (C01OH081468)	OH H8212- GT/CB/LL	40	n.s.	61	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Saginaw, (C01MI081467)	MI H8212- GT/CB/LL	40	n.s.	61	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
Plants without ears							
USA (2008) Aromas, (W27CA081484)	CA Jubilee	50	n.s.	Maturity	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Athens, (E12GA081463)	GA NK 70-C7- GT/CB/LL	47	n.s.	82	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Fitchburg, (C08WI081469)	WI NK 40T- GT/CB/LL	41	n.s.	85	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Germansville, (E04PA081461)	PA NK 40T- GT/CB/LL	41	n.s.	70	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Highland, (C19KS081466)	KS 83F11- GT/CB/LL	51	n.s.	92	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Hillsboro, (W21OR081486)	OR Jubilee	50	n.s.	Maturity	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Oregon, (C19MO081465)	MS 83F11- GT/CB/LL	51	n.s.	90	< 0.01, < 0.01	< 0.01	T006043-07
USA (2008) Richwood, (C01OH081468)	OH H8212- GT/CB/LL	40	n.s.	106	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07
USA (2008) Rupert, (W15ID081485)	ID Jubilee C4274MF	50	n.s.	Maturity	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DAT	Residues (mg/kg)	Mean residue (mg/kg)	Report ID, remarks
USA (2008) Saginaw, (C01MI081467)	MI H8212- GT/CB/LL	40	n.s.	88	< 0.01, < 0.01	<u>< 0.01</u>	T006043-07

Sorghum

Twelve supervised trials were conducted in sorghum in the USA during the 2010 growing season. Sedaxane was applied as a seed treatment at a nominal rate of 40 g ai/100 kg seed. Commodities were collected at normal commercial harvest and were stored frozen for up to 9.5 months (0.8 years). Trials conducted in the same location and with the same variety were not considered to be independent.

Table 11 Results of residue trials in sorghum

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DAT	Residues (mg/kg)	Mean residue (mg/kg)	Report ID, remarks
cGAP: USA/Canada	--	5	--		--	--	--
Forage							
USA (2010) Bagley, IA (C30-0684)	NK6638	40	8	54	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901
USA (2010) Cheneyville, LA (E17-0682)	NK6638	40	7	60	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901
USA (2010) Eaton, CO (W12-0690)	WGF	39	12	62	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901
USA (2010) Elko, SC (E11-0681)	NK7829	42	4	62	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901
USA (2010) Jamestown, ND (C12-0689)	NK7829	42	5	61	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901
USA (2010) LaPlata, IA (C18-0686)	NK6638	40	9	56	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901
USA (2010) Levelland, TX (W39-0692)	NK7829	42	8	60	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901
USA (2010) Raymondville, TX (W08-0688)	NK7829	42	8	61	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901
USA (2010) Richland, IA (C18-0683)	NK6638	40	9	60	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901
USA (2010) Richland, IA (C18-0685)	NK6638	40	9	60	< 0.01, < 0.01	< 0.01	TK0024901
USA (2010) Uvalde, TX (W07-0687)	NK7829	42	4	81	< 0.01, < 0.01	< 0.01	TK0024901
USA (2010) Uvalde, TX (W07-0691)	NK7829	42	8	59	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901
Stover							
USA (2010) Bagley, IA (C30-0684)	NK6638	40	8	157	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901
USA (2010) Cheneyville, LA (E17-0682)	NK6638	40	7	114	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901
USA (2010) Eaton, CO (W12-0690)	WGF	39	12	168	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901
USA (2010) Elko, SC (E11-0681)	NK7829	42	4	107	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901
USA (2010) Jamestown, ND (C12-	NK7829	42	5	144	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901

Country (year) location (trial ID)	Variety	Application rate (g ai/100 kg seed)	Seeding rate (kg seed/ha)	DAT	Residues (mg/kg)	Mean residue (mg/kg)	Report ID, remarks
0689)							
USA (2010) LaPlata, IA (C18-0686)	NK6638	40	9	138	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901
USA (2010) Levelland, TX (W39- 0692)	NK7829	42	8	124	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901
USA (2010) Raymondville, TX (W08-0688)	NK7829	42	8	149	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901
USA (2010) Richland, IA (C18-0683)	NK6638	40	9	125	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901
USA (2010) Richland, IA (C18-0685)	NK6638	40	9	132	< 0.01, < 0.01	< 0.01	TK0024901
USA (2010) Uvalde, TX (W07-0687)	NK7829	42	4	114	< 0.01, < 0.01	< 0.01	TK0024901
USA (2010) Uvalde, TX (W07-0691)	NK7829	42	8	147	< 0.01, < 0.01	<u>< 0.01</u>	TK0024901

FATE OF RESIDUES IN STORAGE AND PROCESSING

A high-temperature hydrolysis study was reviewed by the 2012 Meeting, which concluded that sedaxane is stable under conditions representative of pasteurization, baking, and sterilization. The 2014 Meeting received data showing residues of sedaxane in raw commodities and processed commodities of field corn and potato.

Corn

In the field corn study conducted at 120 g ai/100 kg seed (ca. 2.4× GAP), corn grain was randomly taken from bulk treated and untreated samples. The grain was aspirated and screened to remove impurities and then tempered in preparation for dry milling or steeped in water/sulphurous acid in preparation for wet milling. For dry milling, the kernels were cracked using a disc mill and then dried, screened, and separated into bran, germ, grits, meal, and flour. The product was again aspirated to remove bran, germ with attached hull/endosperm, and large grits. The remaining product underwent additional screening, milling, and sifting. Grits, meal, and flour were separated using a Forsberg gravity separator. An aliquot of germ collected from the dry milling process was heated, flaked, and batch extracted with hexane to produce crude oil, which was additionally processed into refined, neutral oil. For wet milling, steeped corn was passed through a disc mill and centrifuged to remove germ and hulls, which were then dried, aspirated, and screened. The cornstock was milled and screened to produce gluten and starch. Starch was dried to a final moisture content of 15%. Wet-milled germ was moisture conditioned, heated, flaked, and pressed to expel crude oil. The resulting press cake was extracted with hexane to generate more crude oil. Crude oil was refined into neutral oil.

For all samples, including the raw commodity, residues of sedaxane were < 0.01 ppm; therefore, processing factors could not be derived.

Potato

In the potato processing study, tubers from an exaggerated-rate trial (5 g ai/100 kg seed, 2× GAP) were harvested, analysed for residues, and processed into flakes, chips, and wet peel. Samples of tubers were stored for up to 8.3 months and samples of the processed commodities were stored for up to 6.2 months. All samples were stored frozen (ca. -10 °C).

Potatoes were cleaned prior to processing by soaking in 32–37 °C for 5–10 minutes to loosen foreign material. The potatoes were then brushed and rinsed, and then peeled in a mechanical peeler. The removed peel was dewatered in a basket centrifuge. Any green or bruised areas of the peeled

potatoes were removed. For granule/flake production, peeled potatoes were sliced (ca. 1 cm) and washed to remove starch. The potato slices were cooked in hot water (71–74 °C, 20 min.), cooled (32 °C, 20 min.), and then steamed (94–100°C, 35–40 min.) for final cooking. The cooked slices were then flash dried, rolled to a thickness of 0.18–0.25 mm, and adjusted to a moisture content of 8–12% as necessary. The material was then milled and screened to produce flakes and granules. For fried chip production, the peeled potatoes were sliced (1.5 mm) and rinsed to remove starch. After the surface moisture was removed, the slices were fried (177–191 °C, 45–75 sec.) in vegetable oil.

Residues of sedaxane did not concentrate in flakes/granules or in chips, but did concentrate in wet peel.

Table 12 Summary of residues and processing factors for sedaxane in potato

Commodity	Report-Trial	Residue (ppm)	Mean residue (ppm)	Processing factor	Mean processing factor
Tubers (prior to processing)	TK0049813-15	0.0131, 0.0125, 0.0100	0.012	n.a.	n.a.
	TK0049813-16	0.0138, 0.0170, 0.0138	0.015	n.a.	
Flakes/Granules	TK0049813-15	0.0124	0.012	1.0	0.83
	TK0049813-16	0.0090	0.0090	0.61	
Chips	TK0049813-15	0.0075	0.0075	0.63	0.57
	TK0049813-16	0.0075	0.0075	0.50	
Wet Peel	TK0049813-15	0.0360	0.036	3.0	4.3
	TK0049813-16	0.0819	0.082	5.5	

Residues of sedaxane are not expected to concentrate in processed potato commodities except for wet peel, for which residues increased by ca. 4.3-fold relative to the washed tuber.

APPRAISAL

Sedaxane was first evaluated by the JMPR in 2012. The 2012 Meeting concluded that the residue definition for MRL compliance and estimation of dietary intake is sedaxane. An ADI of 0–0.1 mg/kg bw and an ARfD of 0.3 mg/kg bw were established. The residue was determined to be fat-soluble. The 2012 Meeting noted that none of the uses resulted in residues in human foods and that those uses are unlikely to present a public health concern.

The Forty-fifth Session of the CCPR listed sedaxane for the evaluation of additional MRLs. The 2014 Meeting received information on GAP and supervised residue trials reflecting the use of sedaxane as a seed treatment on corn/maize, pulses, potatoes, and sorghum. In addition to evaluating those crops, the Meeting was asked to consider use on rice, without residue data, on the basis of residue data supplied to the 2012 and 2014 Meetings for other grains.

Methods of analysis

Acceptable analytical methods were developed and validated for determination of sedaxane and its metabolites in plant and animal matrices. The methods were evaluated by the 2012 Meeting. The reported LOQ for the sedaxane *cis* and *trans* isomers, each, was 0.005 mg/kg, while the LOQ for all metabolites was 0.01 mg/kg in all matrices (plant and animal).

Results of supervised residue trials on crops

Supervised residue trials submitted for evaluation were as seed treatments and were conducted in Canada and/or the USA. All of the trials submitted to the 2014 Meeting are supported by storage

stability data evaluated by the 2012 Meeting. Residue values listed below reflect the average for each field trial, unless otherwise noted.

Sweet corn

The critical GAP for sweet corn is from the USA at 40 g ai/100 kg seed. In 13 trials, conducted at rates ranging from 29.7 to 51.2 g ai/100 kg seed, residues of sedaxane were < 0.01 mg/kg in all samples.

Based on the results of previously evaluated metabolism data from wheat (2012 Meeting) and on the results of the submitted residue trials, the Meeting agreed that no sedaxane residues are expected in sweet corn (corn-on-the-cob).

The Meeting estimated a maximum residue level of 0.01* mg/kg for sedaxane on sweet corn (corn-on-the-cob), an HR of 0.01 mg/kg and a STMR of 0 mg/kg.

Pulses (Beans, dry and peas, dry)

The critical GAP for beans, dry and peas, dry is from Canada and the USA for use as a seed treatment at a rate of 5 g ai/100 kg seed.

Eleven trials were conducted on dry bean matching GAP in Canada (5) and the USA (6), with an additional two trials in the USA conducted at approximately a 2.5-fold exaggerated rate.

Ten trials were conducted on dry pea matching GAP in Canada (8) and the USA (2).

Sedaxane residues in harvested dry beans and dry peas from all trials (n=21), including the exaggerate-rate trials, were: < 0.01 mg/kg. Based on the results of previously evaluated metabolism data from soya beans (2012 Meeting) and on the results of the submitted residue trials, the Meeting agreed that no sedaxane residues are expected in pulses.

The Meeting estimated a maximum residue level of 0.01* mg/kg for sedaxane on pulses and an STMR of 0 mg/kg. The Meeting recommended that the individual MRL for soya bean, dry be withdrawn.

Potato

The critical GAP for potato is from Canada, as a seed-piece treatment, at a rate of 2.5 g ai/100 kg seed. Twenty-nine trials were conducted matching GAP in Canada (13) and the USA (16). Three additional trials were conducted in the USA; one at approximately a 0.4× rate and two at approximately a 2× exaggerated rate.

Sedaxane residues in harvested tubers from all at-GAP trials (n=29) were: ≤0.01 (28) and 0.018 mg/kg.

The Meeting estimated a maximum residue level of 0.02 mg/kg for sedaxane on potato, an STMR of 0.01 mg/kg, and an HR of 0.02 mg/kg (from a single sample).

Cereal Grains

Maize and popcorn

The critical GAPs are from Chile for maize (50 g ai/100 kg seed) and from the USA for popcorn (40 g ai/100 kg seed). No trials from Chile were provided; however, data from 15 trials with rates ranging from 40–51.2 g ai/100 kg seed were available from the USA depicting residues of sedaxane in maize. In addition, there are two trials conducted at an exaggerated rate of 120 g ai/100 kg seed.

Sedaxane residues in maize from all trials (n=15) were: < 0.01 mg/kg.

Sorghum

The critical GAP for sorghum is from Canada and the USA as a seed treatment at a rate of 5 g ai/100 kg seed. Twelve trials were conducted at an 8-fold exaggerated rate (ca. 40 g ai/100 kg seed) in the USA, of which ten were determined to be independent.

Sedaxane residues in sorghum grain from all trials (n=10) were: < 0.01 mg/kg.

The Meeting noted that while there are no GAP registrations for cereal grains as a group, there are GAPs around the world with GAPs covering the major cereal grain commodities. Based on the results of previously evaluated metabolism data from wheat (2012 Meeting) and on the results of the submitted residue trials for maize and sorghum, the Meeting agreed that no sedaxane residues are expected in cereal grains.

The Meeting estimated a maximum residue level of 0.01* mg/kg for sedaxane on cereal grains and an STMR of 0 mg/kg, and recommends that the individual MRLs for barley, oats, rye, triticale, and wheat be withdrawn.

Legume animal feeds

The critical GAP is for beans, peas, and lentils from Canada and/or the USA as a seed treatment at a rate of 5 g ai/100 kg seed. Fifteen trials were conducted matching GAP in Canada and the USA from which forage and/or hay were harvested. An additional trial was conducted in the USA at approximately a 2.5-fold exaggerated rate.

Sedaxane residues in bean and pea forage (n=5) and hay (n=16) from all trials, including the exaggerate-rate trial, were: < 0.01 mg/kg.

The Meeting estimated a maximum residue level of 0.01* mg/kg and a median residue of 0 mg/kg for sedaxane in bean fodder and pea hay or pea fodder (dry).

The Meeting estimated a highest residue of 0.01 mg/kg and a median residue of 0 mg/kg for sedaxane in bean fodder and pea vines.

Straw, fodder, and forage of cereal grains

The critical GAP for maize is from Chile as a seed treatment at a rate of 50 g ai/100 kg seed. Nineteen trials (15 independent) were conducted at GAP in Canada and the USA from which fodder (stover) and/or forage were harvested.

Sedaxane residues in maize fodder (stover; n=15) and forage (n=15) were: < 0.01 mg/kg.

The critical GAP for sorghum is from the USA and Canada as a seed treatment at a rate of 5 g ai/100 kg seed. Twelve trials were conducted at an 8-fold exaggerated rate (ca. 40 g ai/100 kg seed) in the USA, of which ten were determined to be independent.

Sedaxane residues in sorghum forage and stover (n=10) were: < 0.01 mg/kg.

The 2014 Meeting estimates for sorghum forage (dry), a highest residue of 0.01 mg/kg, and a median residue of 0 mg/kg.

The 2012 Meeting estimated residues of sedaxane in barley, oats, rye, triticale, and wheat straw and fodder, on an as-received basis at 0.075 mg/kg (HR) and 0.01 mg/kg (STMR). In estimating those residues, the 2012 meeting noted that these commodities are not always readily distinguishable in trade and the preference for having a common MRL.

Based on the rationale of the 2012 Meeting and on the results from the maize and sorghum residue trials, the 2014 Meeting agreed to estimate residues of sedaxane in straw, fodder of cereal grains and grasses (including buckwheat fodder) (straws and fodders, dry at a maximum residue level of 0.1 mg/kg).

The Meeting estimates a highest residue of 0.075 mg/kg, and a median of 0.01 mg/kg (as received).

The Meeting withdraws its previous recommendations for straw and fodder (dry) of barley, oat, rye, triticale, and wheat.

Fate of residue during food processing

Residues of sedaxane are stable to hydrolysis (2012 JMPR).

The Meeting received processing studies for corn and potato. In the corn study, residues of sedaxane were < 0.01 mg/kg in all commodities.

Raw agricultural commodity (RAC)	STMR, mg/kg	Processed commodity	Processing factor	STMR-P, mg/kg
Potato tuber	0.01	Flakes/granules	0.83	0.0083
		Chips	0.57	0.0057
		Wet peel	4.27	0.0427

Residues in animal commodities

The 2014 Meeting evaluated sedaxane residues in animal feed items from pulses, cereal grains (corn, rice, and sorghum), and potato in addition to the feed items evaluated by the 2012 Meeting (cereal grains) as listed in the OECD feeding table.

Estimated maximum and mean dietary burdens of livestock

Estimated dietary burdens for Australia, the EU, Japan, and the US/Canada are summarized below. The livestock diets are summarized in Annex 6 to the 2014 Report.

Livestock Dietary Burdens (ppm of dry matter diet) for Sedaxane.

Livestock	Australia		EU		Japan		US/Canada	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Cattle (beef)	0.106	0.057	0.199	0.169	0.006		0.150	0.126
Cattle (dairy)	0.090	0.039	0.168	0.136	0.014		0.080	0.054
Poultry (broiler)			0.010	0.008				
Poultry (layer)			0.023	0.012				

For all livestock, the sedaxane burdens based on the EU animal diets (bold values in the table) reflect the highest burdens for both MRL estimation (maximum diet) and STMR estimation (mean diet).

Animal commodities residue level estimation

In a cattle feeding study evaluated at the 2012 Meeting, residues in all commodities were < 0.01 mg/kg at all dose levels (0.11–2.2 ppm). The 2012 Meeting estimated a maximum residue level of 0.01* mg/kg and STMR and HR values of 0 mg/kg for mammalian commodities.

The 2014 Meeting confirms the previous recommendations.

In a poultry metabolism study evaluated by the 2012 JMPR Meeting, laying hens were dosed at a rate equivalent to 20 ppm (dry matter basis) in their diet. Sedaxane was < 0.01 mg/kg in all tissues at that dose level. Based on the results of that study, the 2012 Meeting estimated a maximum residue level of 0.01* mg/kg and STMR and HR values of 0 mg/kg for poultry commodities.

The 2014 Meeting confirms the previous recommendations.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI assessment.

Definition of the residue (for compliance with the MRL and for estimation of dietary intake) for plant and animal commodities: *sedaxane*.

The residue is fat-soluble.

CCN	Commodity Name	MRL, mg/kg		STMR, mg/kg	HR, mg/kg
		New	Previous		
GC 0640	Barley	W	0.01*		
AS 0640	Barley straw and fodder, dry	W	0.01*		
AL 0061	Bean fodder	0.01*		0	
GC 0080	Cereal grains	0.01*		0	
AS 0647	Oat straw and fodder, dry	W	0.01*		
GC 0647	Oats	W	0.01*		
AL 0072	Pea hay or pea fodder (dry)	0.01*		0	
VR 0589	Potato	0.02		0.01	0.018
VD 0070	Pulses	0.01*		0	
GC 0650	Rye	W	0.01*		
AS 0650	Rye straw and fodder, dry	W	0.01*		
VD 0541	Soya bean (dry)	W	0.01*		
AS 0161	Straw, fodder (dry) and hay of cereal grains and other grass-like plants	0.1		0	
VO 0447	Sweet corn (corn-on-the-cob)	0.01*		0	0.01
GC 0653	Triticale	W	0.01*		
AS 0653	Triticale straw and fodder, dry	W	0.01*		
GC 0654	Wheat	W	0.01*		
AS 0654	Wheat straw and fodder, dry	W	0.01*		

For dietary burden only

				Median, mg/kg	Highest res., mg/kg
AF 0645	Maize forage			0	
AF 0651	Sorghum forage (green)			0	
AF 5249	Corn forage			0	
AL 1030	Bean forage (green)			0	
AL 0528	Pea vines			0	
	Potato, wet peel			0.0427	

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of sedaxane were calculated for the 17 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the current and previous Meetings. The ADI is 0–0.1 mg/kg bw and the calculated IEDIs were 0–0% of the maximum ADI (0.1 mg/kg bw). The Meeting concluded that the long-term intakes of residues of sedaxane, when used in ways that have been considered by the JMPR, are unlikely to present a public health concern.

Short-term intake

The International Estimated Short-Term Intakes (IESTI) of sedaxane were calculated for food commodities and their processed commodities using HRs/HR-Ps or STMRs/STMR-Ps estimated by the current Meeting. The ARfD is 0.3 mg/kg bw and the calculated IESTIs were 0% of the ARfD for all commodities. The Meeting concluded that the short-term intake of residues of sedaxane, when used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

REFERENCES

Code	Author	Year	Title
CER 05720/10	Sagan K.	2012	Sedaxane 500 FS (A16148C) Residue Levels on Sweet Corn (Forage and K + CWHR) from Trials Conducted in Canada During 2010 Syngenta Crop Protection AG, Basel, Switzerland, Syngenta Canada Inc., Guelph, ON, Canada, ALS Laboratory Group, Edmonton, Alberta, Canada, ALS 11SYN281.REP
T006043-07	Hamilton L.	2010	SYN524464 - Magnitude of the Residues in Corn Following Seed Treatment Application Syngenta Crop Protection AG, Basel, Switzerland, Syngenta Crop Protection, Inc., Greensboro, USA, ADPEN Laboratories Inc., Jacksonville FL, USA, 2K9-901-T006043-07
CER 05722/10	Sagan K.	2012	Sedaxane 500 FS (A16148C) Residue Levels on Dry Beans (Seed) from Trials Conducted in Canada During 2010 Syngenta Crop Protection AG, Basel, Switzerland, Syngenta Canada Inc., Guelph, ON, Canada, ALS Laboratory Group, Edmonton, Alberta, Canada, ALS 11SYN277.REP
TK0024902	Hampton M.	2012	Sedaxane FS (A16148C) - Magnitude of the Residues in or on Dried Pea and Dried Bean (except Soybean), Subgroup 6C, and in or on Foliage of Legume Vegetables (except Soybean), Subgroup 7A, Following Seed Treatment Application Syngenta Crop Protection AG, Basel, Switzerland, Syngenta Crop Protection, LLC, Greensboro, NC, USA, SGS North America, Inc., 236 32nd Ave., Brookings, SD, USA, ABC Laboratories Missouri, Columbia, USA, G-1008BK, 66923
CER 05721/10	Sagan K.	2012	Sedaxane 500 FS (A16148C) Residue Levels on Dry Peas from Trials Conducted in Canada During 2010 Syngenta Crop Protection AG, Basel, Switzerland, Syngenta Canada Inc., Guelph, ON, Canada, ALS Laboratory Group, Edmonton, Alberta, Canada, ALS 11SYN282.REP
CER 05724/11	Sagan K.	2012	Sedaxane FS (A16148C) and Diquat SN (A12872A) - Residue Levels on Potatoes from Trials Conducted in Canada During 2011 Syngenta Crop Protection AG, Basel, Switzerland, Syngenta Canada Inc., Guelph, ON, Canada, ALS Laboratory Group, Edmonton, Alberta, Canada, 12SYN319.REP
TK0049813	Willard T.R.	2012	SYN524464 500FS (A16148C) - Magnitude of the Residues in Potato Following Seed Treatment Application USA 2011 Syngenta Crop Protection AG, Basel, Switzerland, Syngenta Crop Protection, LLC, Greensboro, NC, USA, GLP Technologies, Navasota, TX, USA, Golden Pacific Laboratories, LLC (GPL), Fresno, USA, 120420
TK0024901	Hampton M.	2012	Sedaxane FS (A16148C) - Magnitude of the Residues in or on Grain Sorghum Following Seed Treatment Application Syngenta Crop Protection AG, Basel, Switzerland, Syngenta Crop Protection, LLC, Greensboro, NC, USA, SGS North America, Inc., 236 32nd Ave., Brookings, SD, USA, ABC Laboratories Missouri, Columbia, USA, G-1005BK, 66922

SPIRODICLOFEN (237)

The first draft was prepared by Mr David Lunn, New Zealand Food Safety Authority, Wellington, New Zealand

EXPLANATION

Spirodiclofen, a ketoenol (tetronic acid) insecticide was evaluated for the first time by the 2009 JMPR, where an ADI of 0–0.01 mg/kg bw was established and it was agreed that an ARfD was not necessary, residue definitions were proposed and maximum residue levels were recommended for a number of uses where GAP and supporting information were available.

The 2009 JMPR established residue definitions for spirodiclofen:

- For plant products (compliance with MRLs and dietary intake estimation): *spirodiclofen*
- For animal products (compliance with MRLs): *spirodiclofen (fat soluble)*
- For animal products (dietary intake assessment): *sum of spirodiclofen and spirodiclofen-enol, expressed as spirodiclofen.*

Spirodiclofen was listed by the Forty-fifth Session of the CCPR (2013) for the review of additional maximum residue levels for blueberries and avocados and the 2014 JMPR received GAP and residue information for these crops.

METHODS OF RESIDUE ANALYSIS

Analytical methods

The 2009 JMPR reviewed and summarised analytical method descriptions and validation data for spirodiclofen and some of its metabolites in crop, processed commodities and animal commodities, including the HPLC/MS-MS multi-residue method 109351 (validated for the determination of parent spirodiclofen in plant commodities with high acid content, high water content and high fat content) and the HPLC/MS-MS method BA-001-P06-01 (validated for the determination of parent spirodiclofen and metabolites (3) in apples, grapes and their processed fractions).

The analytical method used in the supervised residue trials on blueberry was a modification of Method 109351 (i.e., without the ENVI-Carb or amino-cartridge clean-up steps) and involved extraction with acetonitrile/water/20% cysteine hydrochloride (200:100:1), filtration with Celite, acidification with HCl, partitioning into dichloromethane, evaporation and re-suspension acetonitrile/formic acid prior to analysis by HPLC/MS-MS (triple-quad, positive MRM mode). The reported LOQ of this method was 0.01 mg/kg. [Ref: 109351-1]

Table 1 Recoveries of spirodiclofen with HPLC/MS-MS method 109351

Commodity	LOQ (mg/kg)	Recovery type	spike level (mg/kg)	recoveries % range (mean)	SD (n)	Reference
blueberry	0.01	validation	0.01	88-120 (103)	16 (3)	IR4 PR-09679
			0.2	93-100 (97)	4 (3)	
			5.11	93-103 (97)	5 (3)	
		procedural	0.01	81-109 (93)	9 (14)	
			5.11	85-98 (92)	4 (14)	

Samples from the supervised trials on avocados were analysed using a modification of the HPLC/MS-MS Method BA-001-P06-01 (i.e., with the addition of cysteine hydrochloride to the acetonitrile/water extraction solvent) and involved acetonitrile/water extraction, the addition of isotopic internal standards and filtration prior to analysis for parent spirodiclofen by HPLC/MS-MS (positive MRM mode). The reported LOQ of this method was 0.01 mg/kg. [Ref: BA-001-P06-01]

Table 2 Recoveries of spirodiclofen with HPLC/MS-MS method BA-001-P06-01

Commodity	Reported LOQ (mg/kg)	Recovery type	spike level (mg/kg)	recoveries % range (mean)	SD (n)	Reference
avocado	0.01	validation	0.01	100-116 (109)	5 (7)	RABAP001
		procedural	0.01	93-109 (101)	6 (2)	
			0.5	95-101 (98)	3 (3)	
			1.0	98-105 (101)	4 (3)	

Stability of residues in stored analytical samples

The 2009 JMPR reviewed freezer storage stability studies on a range of representative substrates and concluded that spirodiclofen was stable when stored frozen for at least 13 months in crops with high water content (peach), at least 24 months in crops with high acid content (citrus, grapes), 16 months in crops with oil content (almond nutmeat, dry hop cones), at least 8 months in fruit juice (apple juice, grape juice), and at least 10 months in dried fruit (dried apples, raisins, dried plums).

The Meeting received additional spirodiclofen storage stability studies on blueberry [Ref: IR4 PR-09679] where untreated samples from the field trials were fortified with 0.2 mg/kg spirodiclofen and stored for 47–51 weeks at or below -12 °C.

Samples were taken for extraction and analysis after 47–51 weeks, with the stored control samples being freshly fortified and analysed concurrently to determine the procedural recovery efficiency. Analysis was by LC-MS/MS (Method 109351, with procedural recovery rates of 78–101% in samples fortified at 0.01 mg/kg to 5.1 mg/kg). After 47–51 weeks storage, measured residues of spirodiclofen in stored samples were 85% of the spiked level.

Table 3 Stability of residues in plant matrices fortified with spirodiclofen and stored at ≤ -4 °C

Commodity	Fortification (mg/kg)	Storage interval (weeks)	Spirodiclofen residues (mg/kg)	% Remaining	Procedural recovery (%)
blueberry	0.2	47	0.18, 0.18, 0.17	85	82% at 0.01 mg/kg 81% (at 0.2 mg/kg) 87% at 5.1 mg/kg

USE PATTERNS

Information on GAP in USA was provided to the Meeting for foliar applications to blueberries and avocados and these are summarised in Table 4.

Table 4 Registered uses of spirodiclofen (240 g ai/litre SC formulation)

Crop	Country	Application				Max/season		PHI (days)	Remarks:
		method	kg ai/ha	kg ai/hL	water L/ha	no	kg ai/ha		
Bush berries (004B)									
Blueberries	Canada	spray	0.31		100-1000	1	0.31	7	
Assorted tropical and sub-tropical fruits – inedible peel-large (006B)									
Avocado ^a	USA	spray	0.31-0.35		468	1	0.35	2	

^a GAP is for Avocados and also Black sapote, Canistel, Mamey sapote, Mango, Papaya, Sapodilla, Star apple.

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received new information on supervised field trials involving foliar applications of spirodiclofen to the following crops.

Crop group	Code	Crop	Source	Table
Bush berries	004B	Blueberries	USA, Canada	Table 5
Assorted tropical and sub-tropical fruits – inedible peel (large)	006B	Avocado	USA	Table 6

The supervised trials were well documented with laboratory and field reports. Laboratory reports included method validation including procedural recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables unless residues in control samples exceeded the LOQ. Residue data are recorded unadjusted for recovery.

Intervals of freezer storage between sampling and analysis were recorded for all trials and were covered by the conditions of the freezer storage stability studies.

Results from replicated field plots are presented as individual values. When residues were not detected they are shown as below the LOQ (e.g., < 0.01 mg/kg). Residues and application rates have been rounded to two significant digits (or if close to the LOQ, rounded to one significant digit). Average values and proportionally adjusted residues have been calculated from the residue results prior to rounding and the results from trials used for the estimation of maximum residue levels have been (underlined).

Bush berries

Blueberries

Results from supervised trials from USA and Canada on highbush and lowbush blueberries were provided to the Meeting. In these trials, one foliar application of spiroadiclofen (SC 240 formulation) was applied without spray adjuvant to mature bushes using backpack 1–6 nozzle boom sprayers or tractor-mounted airblast sprayer. Plot sizes in these trials ranged from 33–167 square metres.

Duplicate samples of at least 0.9 kg fruit were taken from at least 12 bushes/plot at maturity or when 70–80% of fruit were ripe, frozen within 24 hours of sampling and stored at -12 °C for up to 327 days before berries were extracted and analysed for spiroadiclofen using LC/MS/MS Method 109351 within 1 day of extraction. The reported LOQ was 0.01 mg/kg and the mean recovery rates in samples spiked with 0.01–0.5 mg/kg spiroadiclofen ranged from 81–109%.

Table 5 Residues on blueberries (highbush and lowbush) from trials in Canada and USA involving one foliar application of spiroadiclofen (240 SC formulation)

BLUEBERRIES Country, year Location (variety)	Application			Matrix	DAT	Residues (mg/kg)		Reference & Comments
	kg ai/ha	water (L/ha)	kg ai/hL			Spirodiclofen	mean	
GAP: Canada	1 × 0.31	100-1000			7			max 0.31 kg ai/ha/season
Canada, 2006 Agassiz, BC (Bluecrop)	0.32	178	0.18	berries	6	1.1, 1.0	<u>1.1</u>	IR4 PR-09679 Trial 06-BC08
USA, 2006 Fennville, MI (Rubel)	0.31	617	0.051	berries	8	0.4, 0.36	<u>0.38</u>	IR4 PR-09679 Trial 06-M131 [airblast]
USA, 2006 Jonesboro, ME (Lowbush-wild)	0.32	374	0.084	berries	7	0.67, 1.0	<u>0.84</u>	IR4 PR-09679 Trial 06-ME03

BLUEBERRIES Country, year Location (variety)	Application			Matrix	DAT	Residues (mg/kg)		Reference & Comments
	kg ai/ha	water (L/ha)	kg ai/hL			Spirodiclofen	mean	
USA, 2006 Castle Hayne, NC (Croatan)	0.31	384	0.081	berries	1 3 7 10 14	0.87, 0.76 0.79, 0.61 0.45, 0.55 0.39, 0.46 0.34, 0.27	0.81 0.7 <u>0.5</u> 0.43 0.31	IR4 PR-09679 Trial 06-NC22
USA, 2006 Castle Hayne, NC (Summit)	0.32	402	0.078	berries	6	0.61, 0.55	<u>0.58</u>	IR4 PR-09679 Trial 06-NC23
USA, 2006 Chatsworth, NJ (Bluecrop)	0.32	421	0.076	berries	6	0.85, 0.55	<u>0.7</u>	IR4 PR-09679 Trial 06-NJ28
Canada, 2006 Kentville, NS (Lowbush-wild)	0.34	440	0.078	berries	1 3 6 9 14	3.1, 2.9 3.3, 3.2 2.3, 2.3 2.1, 2.1 1.4, 1.8	3.0 3.3 <u>2.3</u> 2.1 1.6	IR4 PR-09679 Trial 06-NS06
Canada, 2006 Kentville, NS (Lowbush-wild)	0.34	430	0.078	berries	7	1.5, 1.5	<u>1.5</u>	IR4 PR-09679 Trial 06-NS07
Canada, 2006 Kentville, NS (Lowbush-wild)	0.34	440	0.078	berries	6	1.4, 1.6	<u>1.5</u>	IR4 PR-09679 Trial 06-NS08
USA, 2006 Aurora, OR (Bluecrop)	0.32	290	0.11	berries	6	0.56, 0.46	<u>0.51</u>	IR4 PR-09679 Trial 06-OR24 [Mostly green berries]
Canada, 2006 St-David-de- Falardeau, QC (Lowbush-wild)	0.3	290	0.1	berries	7	1.0, 0.97	<u>0.99</u>	IR4 PR-09679 Trial 06-QC07
Canada, 2006 Labreque, QC (Lowbush-wild)	0.32	299	0.11	berries	7	1.1, 1.2	<u>1.1</u>	IR4 PR-09679 Trial 06-QC08

Assorted tropical and sub-tropical fruits – inedible peel- large (006B)

Avocado

Results from supervised trials from USA on avocado were provided to the Meeting. In these trials, one foliar application of spiroadiclofen (SC 240 formulation) was applied without spray adjuvant, as either a dilute spray (1800–3700 litres/ha) or a concentrate spray (330–650 litres/ha) to mature trees using airblast sprayers. Plot sizes in these trials ranged from 125–372 square metres with a minimum of 4 trees.

Duplicate samples of at least 2 kg mature fruit were frozen within 9 hours of sampling and stored at -15 °C for up to 10 months before whole fruit were extracted and analysed for spiroadiclofen using LC/MS/MS Method BA-001-P06-01 within 4 days of extraction. The reported LOQ was 0.01 mg/kg and the mean recovery rates in samples spiked with 0.01–1.0 mg/kg spiroadiclofen ranged from 98–101%.

Table 6 Residues on avocado from trials in USA involving one foliar application of spirodiclofen (240 SC formulation)

AVOCADO Country, year Location (variety)	Application			Matrix	DAT	Residues (mg/kg)		Reference & Comments
	kg ai/ha	water (L/ha)	kg ai/hL			Spirodiclofen	mean	
GAP: USA	1×0.35				2			max 0.35 kg ai/ha/season
USA, 2007 Canal Point, FL (Tonnage)	0.35	2241	0.016	fruit	2	0.18, 0.2	0.19	RABAP001 BA001-07-HA-A
	0.35	511	0.068	fruit	2	0.33, 0.61	<u>0.47</u>	RABAP001 BA001-07-HA-B
USA, 2007 Carpenteria, CA (Haas)	0.35	2338	0.015	fruit	2	0.02, 0.12	<u>0.07</u>	RABAP001 BA004-07-HA-A 12 yr trees 26 Jul application BBCH 85
	0.35	468	0.075	fruit	2	< 0.01, < 0.01	< 0.01	RABAP001 BA004-07-HA-B
USA, 2007 Carpenteria, CA (Haas)	0.35	2125	0.016	fruit	2	0.03, 0.05	<u>0.04</u>	RABAP001 BA005-07-HA-A 27 yr trees 17 Aug application BBCH 89
	0.35	427	0.082	fruit	2	0.02, 0.01	0.015	RABAP001 BA005-07-HA-B
USA, 2008 San Luis Obispo, CA (Haas)	0.37	3390	0.011	fruit	2	0.11, 0.12	0.12	RABAP001 BA002-07-DA-A
	0.35	652	0.054	fruit	0	0.25, 0.16	0.21	RABAP001 BA002-07-DA-B
					2	0.13, 0.08	0.11	
					5	0.15, 0.15	<u>0.15</u>	
					7	0.15, 0.14	0.15	
					10	0.11, 0.07	0.09	
USA, 2007 Arroyo Grande, CA (Tonnage)	0.36	2041	0.017	fruit	2	0.06, 0.06	0.06	RABAP001 BA003-07-DA-A
	0.36	460	0.077	fruit	0	0.12, 0.11	0.12	RABAP001 BA003-07-DA-B
					2	0.08, 0.05	<u>0.07</u>	
					5	0.02, 0.03	0.03	
					7	0.03, 0.03	0.03	
					10	0.03, 0.01	0.02	

APPRAISAL

Spirodiclofen, a ketoenol (tetronic acid) insecticide was evaluated for the first time by the 2009 JMPR, where an ADI of 0–0.01 mg/kg bw was established and it was agreed that an ARfD was not necessary. Residue definitions were proposed and maximum residue levels were recommended for a number of uses where GAP and supporting information were available.

Residue definitions established by the 2009 JMPR are:

- For plant products (compliance with MRLs and dietary intake estimation): *spirodiclofen*
- For animal products (compliance with MRLs): *spirodiclofen (fat soluble)*
- For animal products (dietary intake assessment): *sum of spirodiclofen and spirodiclofen-enol, expressed as spirodiclofen.*

Spirodiclofen was listed by the Forty-fifth Session of the CCPR (2013) for the review of additional MRLs for blueberries and avocados and the 2014 JMPR received GAP and residue information for these crops.

Methods of analysis

The 2009 JMPR reviewed and summarised analytical method descriptions and validation data for spirodiclofen and some of its metabolites in crop, processed commodities and animal commodities, including the HPLC-MS-MS multi-residue method 109351 (validated for the determination of parent spirodiclofen in plant commodities with high acid content, high water content and high fat content) and the HPLC/MS-MS method BA-001-P06-01 (validated for the determination of parent spirodiclofen and metabolites (3) in apples, grapes and their processed fractions).

These methods, with minor modifications were validated and used in the supervised residue trials on blueberry (Method 109351) and avocado (Method BA-001-P06-01) with an LOQ of 0.01 mg/kg.

Stability of residues in stored analytical samples

The 2009 JMPR reviewed freezer storage stability studies on a range of representative substrates and concluded that in stored frozen analytical samples, spirodiclofen was stable for at least 13 months in crops with high water content (peach), 24 months in crops with high acid content (citrus, grapes), 16 months in crops with oil content (almond nutmeat, dry hop cones), 8 months in fruit juice (apple juice, grape juice) and 10 months in dried fruit (dried apples, raisins, dried plums).

The Meeting received additional spirodiclofen storage stability studies on blueberry, showing that residues were stable in blueberry analytical samples stored frozen for at least 12 months.

Results of supervised residue trials on crops

The Meeting received new information on supervised field trials involving foliar applications of spirodiclofen to blueberries and avocados.

Blueberries

Results from supervised trials from USA and Canada on highbush and lowbush blueberries were provided to the Meeting.

The critical GAP for blueberries is in Canada, one application of 0.31 kg ai/ha with a PHI of 7 days. In trials from North America matching this GAP, spirodiclofen residues were: 0.38, 0.5, 0.51, 0.58, 0.7, 0.84, 0.99, 1.1, 1.1, 1.5, 1.5 and 2.3 mg/kg (n=12).

The Meeting estimated a maximum residue level of 4 mg/kg and an STMR of 0.92 mg/kg for spirodiclofen on blueberries.

Avocado

Results from supervised trials from USA on avocado were provided to the Meeting.

The critical GAP for avocados is in USA, one application of up to 0.35 kg ai/ha with a PHI of 2 days. In trials from USA matching this GAP, spirodiclofen residues were: 0.04, 0.07, 0.07, 0.15 and 0.47 mg/kg.

The Meeting estimated a maximum residue level of 0.9 mg/kg and an STMR of 0.07 mg/kg for spirodiclofen on avocado.

Farm animal dietary burden

As neither of these commodities are livestock feed items, the Meeting agreed that the 2009 JMPR conclusions and maximum residue level recommendations for livestock commodities did not need to be reviewed.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI assessment.

Definition of the residue for compliance with the MRL and for the estimation of dietary intake for plant commodities: *spirodiclofen*

Definition of the residue for compliance with the MRL for animal commodities: *spirodiclofen*

Definition of the residue for the estimation of dietary intake for animal commodities: *sum of spirodiclofen and spirodiclofen-enol, expressed as spirodiclofen.*

The residue is fat soluble

CCN	Commodity	MRL (mg/kg)		STMR
	Name	New	Prev	
FB 0020	Blueberry	4.0		0.92
FI 0326	Avocado	0.9		0.07

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDI) for spirodiclofen was calculated from recommendations for STMRs for raw commodities in combination with consumption data for corresponding food commodities. The results are shown in Annex 3 to the 2014 Report.

The International Estimated Daily Intakes (IEDI) of in the 17 GEMS/Food cluster diets, based on the estimated STMRs were in the range 1–8% of the maximum ADI of 0.01 mg/kg bw. The Meeting concluded that the long-term intake of residues of spirodiclofen from uses considered by the Meeting is unlikely to present a public health concern.

Short-term intake

No ARfD was considered necessary. The Meeting concluded that the short-term intake of residues of spirodiclofen from uses considered by the Meeting is unlikely to present a public health concern.

REFERENCES

Reference	Author(s)	Year	Title
JMPR 2009	Anon	2009	Pesticide residues in food – 2009 Evaluations. Part I. Residues. Spirodiclofen (237), pp 925-1080. FAO Plant Production and Protection Paper 198, 2010. Published.
109351-1	Gould, T.J	2009	An analytical method for the determination of BAJ 2740 residue in various plant matrices by LC-MS/MS. Bayer Corporation, Stilwell, KS, USA. Bayer CropScience, Report No. 109351-1, Amended: 2009-01-12. Unpublished.
BA-001-P06-01	Netzband, D.; Yin, J.	2006	Analytical method for the determination of residues of spirodiclofen and its metabolites BAJ2510, 3-OH-enol spirodiclofen and 4-OH-enol spirodiclofen in apple and grape matrices using LC/MS/MS. Bayer CropScience LP, Stilwell, KS, USA. Bayer CropScience, Report No. BA-001-P06-01. Unpublished.
IR4 PR-09679	Dorschner, K. W.	2009	Spirodiclofen: Magnitude of the residue on blueberry. IR-4 Project HQ, Rutgers, The State University of New Jersey, Princeton, NJ, USA. Report No. IR-4 PR No. 09679, includes Trials: 06-BC08, 06-ME03, 06-MI31, 06-NC22, 06-NC23, 06-NJ28, 06-NS06, 06-NS07, 06-NS08, 06-OR24, 06-QC07 and 06-QC08. Unpublished.

Reference	Author(s)	Year	Title
RABAP001	Dacus, S. C.; Hoag, R. E.	2008	Spirodiclofen - Magnitude of the residue in/on avocado. Bayer CropScience LP, Stilwell, KS, USA. Bayer CropScience, Report No.: RABAP001, includes Trial Nos: BA001-07HA-A, BA001-07HA-B, BA002-07DA-A, BA002-07DA-B, BA003-07DA-A, BA003-07DA-B, BA004-07DA-A, BA004-07DA-B, BA005-07DA-A and BA005-07DA-B, Unpublished

SULFOXAFLOR (252)

First draft was prepared by Dr Paul Humphrey, Australian Pesticides and Veterinary Medicines Authority, Canberra, Australia

EXPLANATION

Sulfoxaflor is the first member of the novel sulfoximine class of insecticides. It controls insects via a unique interaction with the insect nicotinic acetylcholine receptor and has a broad lack of cross-resistance to other insecticides.

Sulfoxaflor was evaluated by JMPR in 2011. An ADI and AfRD of 0–0.05 mg/kg bw and 0.3 mg/kg bw respectively were established by the 2011 Meeting. A residue definition of *sulfoxaflor* was established for both compliance and dietary risk assessment in both plant and animal commodities.

After the subsequent meeting of CCPR, the proposed MRLs for citrus fruits, pome fruits, stone fruits and tree nuts were held at Step 4 because the GAP reviewed by JMPR differed from the registered USA GAP.

The Meeting received information on registered use patterns in pome fruit, stone fruit, citrus fruit and tree nuts in Australia and the USA. The residue trial data submitted to the 2011 Meeting for these crops was reconsidered against the registered GAPs.

USE PATTERN

Information on registered uses made available to this Meeting is shown in Table 1.

Table 1 Registered uses of sulfoxaflor on citrus fruits, pome fruits, stone fruits and tree nuts

Crop	Country	Formulation		Application					PHI [days]
		g ai/L or [g ai/kg]	Type	Method	Timing [Interval - days]	Rate [g ai/ha]	Concentration [g ai/100 L]	Maximum [g ai/ha/year] or (no. per crop)	
Citrus Fruits									
Citrus, including oranges, lemons, grapefruit, limes, mandarins and tangerines	Australia	240	SC	Foliar	–	–	9.6	192 g ai/ha/single application (2)	1
Citrus (Crop Group 10) including citrus citron, grapefruit, kumquat, lemon, lime, orange, tangelo, tangerines and hybrids of these	USA	[500]	WG	Foliar	14	96	–	298 g ai/ha/year (4)	1
Pome Fruits									
Pome fruit, including apples, pears and nashi	Australia	240	SC	Foliar	–	–	9.6	192 g ai/ha/single application (2)	7
Pome fruits (Crop Group 11) including apples, crabapple, loquat, mayhaw, pears, quince	USA	[500]	WG	Foliar	7	96	–	298 g ai/ha/year (4)	7
Stone Fruits									
Stone fruit, including apricots, cherries, nectarines, peaches and plums	Australia	240	SC	Foliar	–	–	7.2	144 g ai/ha/single application (2)	7

Crop	Country	Formulation		Application					PHI [days]
		g ai/L or [g ai/kg]	Type	Method	Timing [Interval - days]	Rate [g ai/ha]	Concentration [g ai/100 L]	Maximum [g ai/ha/year] or (no. per crop)	
Stone fruits (Crop Group 12) including apricot, nectarine, peach, plum, prune, sweet cherry, tart cherry	USA	[500]	WG	Foliar	7	96	–	298 g ai/ha/year (4)	7
Tree Nuts									
Tree nuts (Crop Group 14) including almonds, cashew, chestnut, filbert (hazelnut), macadamia nut, pecan, walnut	USA	[500]	WG	Foliar	7	96	–	298 g ai/ha/year (4)	7

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received no new information on residues resulting from supervised trials.

The citrus fruits, pome fruits, stone fruits and tree nuts data evaluated at JMPR 2011 which are tabulated below, were also evaluated for this appraisal, with reference to the submitted labels showing registered uses for these crops in Australia and the USA. The tables of residues data have been taken essentially unaltered from the 2011 evaluation, but with the addition of a column in the citrus, pome and stone fruits tables expressing the applied active as grams per 100 L, as well as *g ai/ha*, to enable easier comparison with Australian GAP for citrus, pome and stone fruits, which are expressed as concentrations. In addition, when two trials were performed at the same location in the same year and differ only by variety of fruit or nut, they have been separated by a dotted line in the tables and the highest residue from one trial only has been used in the estimation of an MRL and STMR.

Group	Commodity	Country	Table No.
FC Citrus Fruits	Grapefruit	USA	2
	Lemons	USA	3
	Oranges	Australia, Brazil, USA	4
	Mandarins	Australia	5
FP Pome Fruits	Apples	Australia, New Zealand, Europe,	6
	Pears	Australia, Europe, USA	7
FS Stone Fruits	Cherry	Australia, New Zealand, Europe,	8
	Nectarines	Australia, New Zealand	9
	Peach	Australia, New Zealand, Europe,	10
	Apricot	Australia, New Zealand	11
	Plums	Australia, USA	12
	Almonds	USA	13
TN Tree Nuts	Pecans	USA	14
	Almond hulls	USA	15
Animal feeds	Almond hulls	USA	15

*Citrus fruits**Grapefruit*

Table 2 USA residue trials for grapefruit

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No.	g ai/ha	g ai/100L	L/ha				Sulfoxaflor	X11719474	X11721061
De Leon Springs, FL, USA, 2010	Star red	2	202	19.0	1060	7	0 1 7 13	Whole fruit	0.059 0.012 0.010 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01
De Leon Springs, FL, USA, 2010	Ruby red	2	202	19.0	1060	7	0 1 7 13	Whole fruit	0.088 0.013 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01
Fort Pierce, FL, USA, 2010	Star red	2	202	19.0	1060	7	0 1 7 13	Whole fruit	0.076 0.010 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01
Fort Pierce, FL, USA, 2010	Ruby red	2	202	19.0	1060	7	1 1 1	Whole fruit Peel Pulp	< 0.01 0.050 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
Vero Beach, FL, USA 2010	White	2	202	19.0	1060	7	1	Whole fruit	0.016	< 0.01	< 0.01
Edinburg, TX, USA 2010	Rio red	2	202	19.4	1040	7	1 1 1	Whole fruit Peel Pulp	0.13 0.79 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
San Luis Obispo, CA, USA 2010	Not specified	2	202	15.2	1330	7	0 1 7 14 0 1 7 14 0 1 7 14	Whole fruit Peel Pulp	0.029 0.024 < 0.01 < 0.01 0.081 0.065 0.039 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01
Springville, CA, USA 2010	Oro blanco	2	202	16.1	1250	7	1	Whole fruit	0.11	< 0.01	< 0.01

Lemons

Table 3 USA residue trials in lemons

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No.	g ai/ha	g ai/100L	L/ha				Sulfoxaflor	X11719474	X11721061
Ft. Pierce, FL, USA, 2010	Bears	2	202	19.1	1060	7	1	Whole fruit	0.11	< 0.01	< 0.01
San Luis Obispo, CA, USA, 2010	Lisbon	2	202	20.0	1010	7	0 1 7 14	Whole fruit	0.081 0.028 0.040 0.031	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No.	g ai/ha	g ai/100L	L/ha				Sulfoxaflor	X11719474	X11721061
San Luis Obispo, CA, USA, 2010	Lisbon	2	202	21.2	950	7	0 1 7 14	Whole fruit	0.072 0.051 <u>0.083</u> 0.014	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01
Porterville, CA, USA, 2010	Improved meyer	2	202	16.2	1250	8	0 1 8 15	Whole fruit	0.31 <u>0.29</u> 0.20 0.16	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01
Maricopa, CA, USA 2009	Lisbon	2	202	20.6	980	6	1	Whole fruit	<u>< 0.01</u>	< 0.01	< 0.01
Bakersfield, CA, USA 2009	Lisbon	2	202	21.2	1050	6	1	Whole fruit	<u>< 0.01</u>	< 0.01	< 0.01

Oranges

Table 4 Residue trials in Australia, Brazil and the USA for oranges

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No.	g ai/ha	g ai/100L	L/ha				Sulfoxaflor	X11719474	X11721061
Gayndah, Queensland, Australia, 2008	Valencia "Honey Balls"	2	192	20.2	950	7	0 1 7 14	Whole fruit	0.47 <u>0.41</u> 0.35 0.29	< 0.01 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 0.01
		4	96	8.82	1090	7	0 1 7 14		0.41 0.37 0.32 0.30	0.01 < 0.01 < 0.01 < 0.01	0.02 0.01 0.01 0.02
		2	192	9.6	2000	7	1		<u>0.33</u>	< 0.01	< 0.01
		4	94	4.7	2000	7	7		0.18	0.02	< 0.02
		2	192	9.6	2000	7	1		<u>0.09</u>	< 0.01	< 0.01
		4	96	4.8	2000	7	7		0.02	< 0.01	< 0.01
		2	193	6.43	3000	7	1		<u>0.16</u>	< 0.01	< 0.01
		2	191	7.64	2500	7	1		<u>0.15</u>	< 0.01	< 0.01
Ramco, South Australia, Australia	Valencia	2	194	7.76	2500	7	1	Whole fruit	<u>0.43</u>	0.010	< 0.01

		Application							Residues (mg/kg)		
Location Year	Variety	No	g ai/ha	g ai/100L	L/ha	RTI	DAT	Portion	Sulfoxaflor	X11719474	X11721061
Mogi Mirim, SP, Brazil, 2008	Pera Coroa	2	200	10	2000	7	0	Whole fruit	0.11	< 0.01	< 0.01
							1		0.099	< 0.01	< 0.01
							3		0.010	0.010	< 0.01
							7		0.068	< 0.01	< 0.01
							14		0.057	< 0.01	< 0.01
							28		0.062	< 0.01	< 0.01
							0	Juice	0.025	< 0.01	< 0.01
							1		0.013	< 0.01	< 0.01
							3		0.012	< 0.01	< 0.01
							7		0.018	< 0.01	< 0.01
							14		< 0.01	< 0.01	< 0.01
							28		< 0.01	< 0.01	< 0.01
							0	Peel	0.52	0.032	0.015
							1		0.55	0.038	0.019
							3		0.37	0.032	0.014
							7		0.38	0.062	0.036
							14		0.18	0.030	0.026
							28		0.085	0.021	0.015
Piracicaba, SP, Brazil, 2008	Valencia	2	200	9.99	2000	7	1	Whole fruit	0.096	< 0.01	< 0.01
							7		Juice	0.061	< 0.01
							1	Peel	0.026	< 0.01	< 0.01
							7		< 0.01	< 0.01	< 0.01
							1		0.52	0.012	< 0.01
7	0.21	0.010	< 0.01								
Ibiraci, MG, Brazil, 2008	Pera Rio	2	200	10	2000	7	1	Whole fruit	0.28	0.012	< 0.01
							7		Juice	0.21	0.040
							1	Peel	< 0.01	< 0.01	< 0.01
							7		< 0.01	< 0.01	< 0.01
							1		1.3	0.075	0.026
7	0.69	0.10	0.044								
Rolandia, PR, Brazil, 2008	Valencia	2	200	10	2000	7	1	Whole fruit	0.12	< 0.01	< 0.01
							7		Juice	0.11	0.015
							1	Peel	< 0.01	< 0.01	< 0.01
							7		< 0.01	< 0.01	< 0.01
							1		0.58	0.31	0.026
7	0.29	0.051	0.023								
De Leon Springs, FL, USA, 2008	Navel	2	202	18.9	1070	7	0	Whole fruit	0.11	< 0.01	< 0.01
							1		0.074	< 0.01	< 0.01
							7		0.060	< 0.01	< 0.01
							15		0.051	< 0.01	< 0.01
26	0.044	< 0.01	< 0.01								
De Leon Springs, FL, USA, 2010	Hamlin	2 ^a	198	18.7	1060	7	1	Whole fruit	0.11	< 0.01	< 0.01
							7		0.082	< 0.01	< 0.01
		2 ^b	194	18.5	1050	7	7	Whole fruit	0.082	< 0.01	< 0.01
DeLeon Springs, FL, USA, 2010	Valencia	2	202	15.3	1320	7	1	Whole fruit	0.16	< 0.01	< 0.01
							7		< 0.01	< 0.01	< 0.01
DeLeon Springs, FL, USA, 2010	MidSweet	2	202	15.4	1320	7	1	Whole fruit	0.085	< 0.01	< 0.01
							7		0.011	< 0.01	< 0.01
Kathleen, FL, USA, 2008-2009	Hamlin	2 ^a	203	18.7	1090	7	1	Whole fruit	0.12	< 0.01	< 0.01
		2 ^b	202	18.5	1090	7	7	Whole fruit	0.11	< 0.01	< 0.01
Vero Beach, FL, USA, 2008	Hamlin	2 ^c	203	18.9	1080	7	1	Whole fruit	0.12	< 0.01	< 0.01
		2 ^d	203	18.9	1080	7	7	Whole fruit	0.099	< 0.01	< 0.01
Fort Pierce, FL, USA, 2008	Hamlin	2 ^a	206	18.5	1110	7	1	Whole fruit	0.093	< 0.01	< 0.01
		2 ^b	201	18.4	1090	7	7	Whole fruit	0.047	< 0.01	< 0.01
Edinburg, TX, USA, 2009	Valencia	2	203	20.2	1000	7	1	Whole fruit	0.098	< 0.01	< 0.01
							7		0.23	< 0.01	< 0.01
Sanger, CA, USA, 2009	Washington Navel	2	202	6.45	3130	7	1	Whole fruit	0.062	< 0.01	< 0.01
							6		0.036	< 0.01	< 0.01

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No	g ai/ha	g ai/100L	L/ha				Sulfoxaflor	X11719474	X11721061
Sanger, CA, USA, 2008	Valencia	2	202	15.8	1280	7	1 7	Whole fruit	0.050 0.041	< 0.01 < 0.01	< 0.01 < 0.01
Mount Dora, FL, USA, 2010	Hamlin	2	202	15.4	1310	7	1 7	Whole fruit	0.12 0.020	< 0.01 < 0.01	< 0.01 < 0.01
San Louis, Obispo, CA, USA, 2010	Valencia	2	202	18.6	1090	7	1 7	Whole fruit	0.038 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01

^a Both applications used non-ionic surfactant at 0.05% (v/v)

^b Both applications used crop oil concentrate at 1% (v/v)

^c Application #1 used non-ionic surfactant at 0.05% (v/v), while application #2 used citrus oil at 1% (v/v).

^d Application #1 used citrus oil at 1% (v/v), while application #2 used non-ionic surfactant at 0.05% (v/v).

Mandarins

Table 5 Residue trials for sulfoxaflor in mandarins (Australian trials)

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No	g ai/ha	g ai/100L	L/ha				Sulfoxaflor	X11719474	X11721061
Gayndah, Queensland, Australia, 2010	Imperials	2	148	7.59	1950	7	0 1 7 14	Whole fruit	0.45 <u>0.44</u> 0.40 0.32	< 0.01 < 0.01 < 0.01 0.01	< 0.01 0.009 0.01 0.02
Wallaville, Queensland, Australia, 2008	Murcott	2	192	9.6	2000	7	1	Whole fruit	<u>0.15</u>	< 0.01	< 0.01
		4	95	4.8	2000	7	7	Whole fruit	0.18	< 0.01	< 0.01
Wallaville, Queensland, Australia, 2010	Imperials	2	191	7.64	2500	7	1	Whole fruit	<u>0.28</u>	< 0.01	< 0.01
Mundubbera, Queensland, Australia, 2010	Imperials	2	192	9.6	2000	7	1	Whole fruit	<u>0.34</u>	< 0.01	< 0.01

Pome fruits

Apples

Table 6 Residue trials for sulfoxaflor in apples (Australia, New Zealand, Europe, and USA)

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No	g ai/ha	g ai/100L	L/ha				Sulfoxaflor	X11719474	X11721061
Orange, New South Wales, Australia, 2008	Red Delicious	2	195	10.3	1900	7	3 7 14 28	whole fruit	0.22 <u>0.19</u> 0.14 0.08	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01
		4	98	4.9	2000	7	3 7 14 28		0.21 0.15 0.10 0.11	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.010 < 0.01 0.010
Lenswood, Australia, 2008	Fuji	2	164	9.11	1800	8	7	whole fruit	<u>0.14</u>	< 0.01	< 0.01

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No	g ai/ha	g ai/100L	L/ha				Sulfoxaflor	X11719474	X11721061
		4	81	4.5	1800	7	7	whole fruit	0.09	< 0.01	< 0.01
Havelock North, New Zealand, 2008	Braeburn	2	193	10.7	1800	7	3	whole fruit	0.09	< 0.01	< 0.01
							7		<u>0.07</u>	< 0.01	< 0.01
							14		0.06	< 0.01	< 0.01
							28		0.06	< 0.01	< 0.01
		4	96	5.33	1800	7	3		0.08	< 0.01	< 0.01
							7		0.06	< 0.01	< 0.01
							14		0.05	< 0.01	< 0.01
							28		0.06	< 0.01	< 0.01
Havelock North, New Zealand, 2008	Royal Gala	2	198	16.5	1200	7	7	whole fruit	0.10	< 0.01	< 0.01
		4	98	8.17	1200	7	7		0.055	< 0.01	< 0.01
Nelson, New Zealand, 2008	Butte	2	193	16.1	1200	7	7	whole fruit	<u>0.065</u>	< 0.01	< 0.01
		4	98	8.17	1200	7	7		0.09	< 0.01	< 0.01
Motueka, New Zealand, 2008	Monterey	2	193	12.9	1500	7	7	whole fruit	<u>0.02</u>	< 0.01	< 0.01
		4	97	6.47	1500	7	7		0.05	< 0.01	< 0.01
Jork, Germany, EU, 2008	Jonagold	2	203	20.1	1010	6	3	whole fruit	0.24	< 0.01	< 0.01
							7		<u>0.18</u>	< 0.01	< 0.01
							14		0.18	< 0.01	< 0.01
							27		0.10	< 0.01	< 0.01
Wielonek, Poland, EU, 2008	Idared	2	202	10.1	1010	7	7	whole fruit	<u>0.078</u>	< 0.01	< 0.01
Barboles, Spain, EU, 2008	Golden	2	210	20.2	1040	7	3	whole fruit	0.27	< 0.01	< 0.01
							7		<u>0.27</u>	< 0.01	< 0.01
							14		0.20	< 0.01	0.010
							28		0.18	< 0.01	0.012
Korifi, Greece, EU, 2008	Mutsu	2	200	20	1000	7	7	whole fruit	<u>0.074</u>	< 0.01	< 0.01
Hereford, PA, USA, 2008	Star Krimson	2	205	9.07	2260	8	7	whole fruit	<u>0.043</u>	< 0.01	< 0.01
							14		0.041	< 0.01	< 0.01
Hereford, PA, USA, 2010	Star Krimson	2	202	15.4	1310	6	8	whole fruit	<u>0.12</u>	< 0.01	< 0.01
							15		0.12	< 0.01	< 0.01
Blissfield, MI, USA, 2008	Reds	2	202	11.5	1750	7	4	whole fruit	0.059	0.009	< 0.01
							7		<u>0.040</u>	0.010	< 0.01
							14		0.025	0.010	< 0.01
							28		0.021	< 0.01	< 0.01
Sanger, CA, USA, 2008	Pink Lady	2	204	14.3	1430	7	6	whole fruit	<u>0.064</u>	< 0.01	< 0.01
							14		0.050	0.011	< 0.01
Ephrata, WA, USA, 2008	Red Delicious	2	200	10.7	1870	7	7	whole fruit	<u>0.063</u>	< 0.01	< 0.01
							14		0.044	< 0.01	0.011
Royal City, WA, USA, 2008	Red Delicious	2	201	10.7	1880	7	7	whole fruit	<u>0.066</u>	< 0.01	< 0.01
							14		0.065	< 0.01	0.019

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No	g ai/ha	g ai/100L	L/ha				Sulfoxaflor	X11719474	X11721061
Orefield, PA, USA, 2010	Jonomac	2	202	15.4	1310	7	7 14	whole fruit	<u>0.072</u> 0.060	< 0.01 < 0.01	< 0.01 < 0.01
Moultrie, GA, USA, 2008	Red Fuji	2	202	19.8	1020	7	7 14	whole fruit	<u>< 0.01</u> < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Deerfield, MI, USA, 2010	Red Delicious	2	202	10.7	1890	7	7 14	whole fruit	<u>0.039</u> 0.024	< 0.01 < 0.01	< 0.01 < 0.01
Perry, UT, USA, 2010	Gala	2	201	15	1340	6	7 13	whole fruit	<u>0.089</u> 0.10	< 0.01 < 0.01	< 0.01 < 0.01
Caldwell, ID, USA, 2010	Honey Crisp	2	202	8.56	2360	6	6 14	whole fruit	<u>0.056</u> 0.045	< 0.01 < 0.01	< 0.01 < 0.01
Hood River, OR, USA, 2008)	Honey Crisp	2	202	8.86	2280	7	7 14	whole fruit	<u>0.068</u> 0.064	< 0.01 < 0.01	< 0.01 < 0.01

Pears

Table 7 Residue trials for sulfoxaflor in pears (Australia, Europe and USA)

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No	g ai/ha	g ai/100L	L/ha				Sulfoxaflor	X11719474	X11721061
Zeerust, Australia, 2009	Williams	2	194	10.8	1800	7	3 7 14 28	Whole fruit	0.12 <u>0.11</u> 0.060 0.070	< 0.01 < 0.01 < 0.01 < 0.01	0.010 0.010 < 0.01 0.010
							3 7 14 28		0.14 0.13 0.080 0.050	< 0.01 < 0.01 < 0.01 < 0.01	0.010 0.020 0.020 < 0.01
		4	96	5.33	1800	7	3 7 14 28				
							3 7 14 28				
							3 7 14 28				
							3 7 14 28				
							3 7 14 28				
							3 7 14 28				
Paracombe, Australia, 2009	Packham Triumph	2	157	10.5	1500	7	7	Whole fruit	<u>0.22</u>	< 0.01	0.010
		4	80	5.33	1500	7	7	Whole fruit	0.18	0.010	0.020
Heidelberg-Kercheim, Germany, EU, 2008	Williams Christ	2	192	20.0	960	7	3 7 14 28	Whole fruit	0.10 <u>0.058</u> 0.055 0.025	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01
							3 7 14 28				
							3 7 14 28				
							3 7 14 28				
Mor, Hungary, EU, 2008	Pap korte	2	205	20.0	1020	7	7	Whole fruit	<u>0.052</u>	< 0.01	< 0.01
Nisi, Greece, EU, 2008	Williams	2	200	20.0	1000	7	7	Whole fruit	<u>0.18</u>	< 0.01	0.011
Alcarras, Spain, EU, 2008	Flor de invierno	2	213	13.4	1590	7	3 7 14 28	Whole fruit	0.074 0.097 <u>0.099</u> 0.097	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01
							3 7 14 28				
							3 7 14 28				
							3 7 14 28				

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No	g ai/ha	g ai/100L	L/ha				Sulfoxaflor	X11719474	X11721061
Gehrden, Germany, EU, 2010	Condo	2	193	20.0	970	7	0	Whole fruit	0.38	0.010	< 0.01
							3		0.13	< 0.01	< 0.01
							7		<u>0.10</u>	< 0.01	< 0.01
							14		0.10	< 0.01	< 0.01
							21		0.084	< 0.01	< 0.01
Malalbergo, Spain, EU, 2010	Abate	2	206	21.0	980	7	7	Whole fruit	<u>0.18</u>	< 0.01	< 0.01
Orefield, PA, USA, 2009	Bartlett	2	204	7.41	2750	7	3	Whole fruit	0.17	< 0.01	< 0.01
							8		<u>0.16</u>	< 0.01	< 0.01
							15		0.12	< 0.01	< 0.01
							21		0.097	< 0.01	< 0.01
Poplar, CA, USA, 2009	Olympic	2	204	13.6	1500	7	7	Whole fruit	<u>0.13</u>	< 0.01	< 0.01
							14		0.096	< 0.01	< 0.01
Lindsay, CA, USA, 2009	Red Olympic	2	202	9.84	2050	7	7	Whole fruit	<u>0.18</u>	< 0.01	< 0.01
							14		0.14	< 0.01	< 0.01
Ephrata, WA, USA, 2009	Concord	2	202	43.1	470	7	7	Whole fruit	0.26	< 0.01	0.017
							14		0.23	< 0.01	0.019
Ephrata, WA, USA, 2009	Star Bartlett	2	202	10.8	1870	7	7	Whole fruit	<u>0.23</u>	< 0.01	0.015
							14		0.18	< 0.01	0.015
Hood River, OR, USA, 2009	Red D'Anjou	2	202	13.1	1540	7	7	Whole fruit	0.075	< 0.01	< 0.01
							14		<u>0.078</u>	< 0.01	< 0.01

Stone fruits

Cherries

Table 8 Results of residue trials conducted with two (or four) applications of sulfoxaflor 240 g ai/L SC in/on cherry

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No	g ai/ha	g ai/100L	L/ha				Sulfoxaflor	X11719474	X11721061
Orange, N.S. Wales, AU, 2008	Merchant	2	193	9.65	2000	7	3	Pitted fruit	0.33	< 0.01	< 0.01
							7		<u>0.35</u>	< 0.01	< 0.01
							11		0.18	< 0.01	< 0.01
							14		0.17	< 0.01	< 0.01
							25		0.12	< 0.01	< 0.01
		4	98	4.9	2000	7	3		0.30	0.014	< 0.01
							7		0.37	0.013	< 0.01
							11		0.27	0.012	< 0.01
							14		0.24	0.010	< 0.01
							25		0.16	0.015	< 0.01
Havelock, N. NZ, NZ, 2008	Lapin	2	195	16.3	1200	7	7	Pitted fruit	<u>0.38</u>	< 0.01	0.013
									1.4	< 0.01	0.052
		4	97	8.08	1200	7	7	Pitted fruit	1.4	< 0.01	0.053
Frankfurt/Oder, DE, EU, 2008	Schatten- morelle	2	192	20.0	960	7	3	Pitted fruit	1.8	0.031	0.040
							7		<u>1.5</u>	0.029	0.048
							14		1.2	0.029	0.043
							21		0.96	0.031	0.044
Agárd, HU, EU, 2008	Kavics	2	194	20.0	970	7	7	Pitted fruit	<u>0.90</u>	0.011	0.017
Söllingen, DE, EU, 2009	Schatten- morelle	2	190	16.7	1140	7	7	Pitted fruit	<u>0.77</u>	0.011	< 0.01

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No	g ai/ha	g ai/100L	L/ha				Sulfoxaflor	X11719474	X11721061
Epila, ES, EU, 2008	Blanca	2	201	10.1	1990	7	7	Pitted fruit	<u>0.54</u>	0.010	0.026
Panagitsa, GR, EU, 2008	Mpakirtz-eika	2	202	16.7	1210	7	3	Pitted fruit	0.95	0.011	0.012
							7		<u>0.98</u>	0.014	0.016
							14		0.80	0.019	0.021
							21		0.73	0.021	0.022
Sahorre, FR, EU, 2008	Burlat	2	199	20.1	990	7	7	Pitted fruit	<u>0.80</u>	< 0.01	0.044
Orefield, PA, US, 2009	Montmorency	2	205	8.63	2380	7	6	Pitted fruit	<u>1.2</u>	0.016	0.026
Fennville, MI, US, 2009	Sour	2	202	21.0	960	7	7	Pitted fruit	<u>1.2</u>	0.013	0.048
Hart, MI, US, 2009	Sour	2	202	16.3	1240	7	7	Pitted fruit	<u>1.0</u>	0.016	0.042
Fennville, MI, US, 2009	Sweet	2	202	21.0	960	7	3	Pitted fruit	0.62	< 0.01	0.010
							7		<u>0.76</u>	< 0.01	0.018
							14		0.56	< 0.01	0.013
							21		0.35	< 0.01	0.012
Hart, MI, US, 2009	Sweet	2	202	16.3	1240	7	7	Pitted fruit	<u>0.59</u>	< 0.01	0.019
Marysville, CA, US, 2010	Brooks	2	204	16.2	1260	7	6	Pitted fruit	<u>0.55</u>	< 0.01	0.010

Nectarines

Table 9 Residue trials for sulfoxaflor in nectarines

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No	g ai/ha	g ai/100L	L/ha				Sulfoxaflor	X11719474	X11721061
Shepparton, Australia, 2009	Grand Sweet	2	192	16	1200	7	7	Pitted fruit	<u>0.12</u>	< 0.01	< 0.01
Ardmona, Australia, 2008	A38-039 Yellow	2	192	10.7	1800	8	3	Pitted fruit	0.27	< 0.01	< 0.01
							7		<u>0.18</u>	< 0.01	< 0.01
							14		0.13	< 0.01	< 0.01
		4	96	5.33	1800	7	22		0.16	0.010	0.011
							3		0.17	< 0.01	< 0.01
							7		0.14	0.011	< 0.01
							14		0.12	0.010	0.061
							22		0.15	0.013	0.010
Orange, Australia, 2008	Fairlane	2	195	9.75	2000	7	7	Pitted fruit	<u>0.14</u>	< 0.01	0.010
		4	87	4.85	2000	7	7	Pitted fruit	<u>0.16</u>	< 0.01	0.010
Karragullen, Australia, 2008	Red Gold	2	197	19.7	1000	7	7	Pitted fruit	<u>0.10</u>	< 0.01	< 0.01
		4	98	9.8	1000	7	7	Pitted fruit	0.23	0.010	0.015
Havelock, New Zealand, 2008	Red Gold	2	195	13	1500	7	7	Pitted fruit	<u>0.11</u>	< 0.01	0.014
		4	98	6.53	1500	7	7	Pitted fruit	0.19	< 0.01	0.021

Peaches

Table 10 Results of residue trials conducted with two (or four) applications of sulfoxaflor 240 g ai/L SC in/on peach

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No	g ai/ha	g ai/100L	L/ha				Sulfoxaflor	X11719474	X11721061
Shepparton, Victoria, Australia, 2008	Taylor Queen	2	194	10.8	1800	7	3 7 14 28	Pitted fruit	0.34 0.22 0.20 <u>0.24</u>	< 0.01 < 0.01 < 0.01 0.011	< 0.01 < 0.01 < 0.01 0.013
		4	97	5.4	1800	7	3 7 14 28		0.22 0.16 0.23 0.14	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.011 < 0.01
		2	194	9.7	2000	7	7		<u>0.27</u>	< 0.01	0.021
		4	98	4.9	2000	7	7		0.13	< 0.01	0.014
		2	190	19	1000	7	7		<u>0.15</u>	< 0.01	0.010
		4	97	9.7	1000	7	7		0.15	< 0.01	0.018
		2	192	10.7	1800	7	7		<u>0.14</u>	< 0.01	< 0.01
		4	97	5.4	1800	7	7		0.10	< 0.01	< 0.01
Ballandean, Queensland, Australia, 2009	Diamond Princess	2	194	19.4	1000	7	7	Pitted fruit	<u>0.012</u>	< 0.01	< 0.01
Shadforth, New South Wales, Australia, 2010	O'Henry	2	198	19.8	1000	7	3 7 14	Pitted fruit	0.18 0.10 <u>0.11</u>	< 0.01 < 0.01 < 0.01	0.022 0.010 0.014
		2	199	16.6	1200	7	8		<u>0.12</u>	< 0.01	0.010
		2	192	16	1200	7	7		<u>0.11</u>	< 0.01	0.016
Szekefehervar, Hungary, EU, 2008	Padana	2	198	20	990	7	3 8 14 21	Pitted fruit	0.64 0.36 0.22 0.14	< 0.01 < 0.01 < 0.01 < 0.01	0.040 0.029 0.024 0.019
		2	197	22.2	890	7	7		0.20	< 0.01	0.010
		2	203	20.0	1020	7	7		0.54	0.010	0.022
		2	202	9.99	2020	7	3 7 14 21		0.49 0.27 0.25 0.12	< 0.01 < 0.01 < 0.01 < 0.01	0.017 0.017 0.018 0.013
Barbiano, Italy, EU, 2008	Sweet Red	2	198	13.4	1480	7	7	Pitted fruit	0.21	< 0.01	< 0.01
Lakka, Greece, EU, 2009	Andross	2	210	13.4	1570	7	7	Pitted fruit	0.83	< 0.01	0.012
Orefield, PA, USA, 2008	Glen Glow	2	202	7.18	2820	7	7	Pitted fruit	0.90	< 0.01	< 0.01
Montezuma, GA, USA, 2008	Flame Prince	2	202	21.4	940	7	4 7 10 14	Pitted fruit	0.058 0.032 0.032 0.028	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No	g ai/ha	g ai/100L	L/ha				Sulfoxaflor	X11719474	X11721061
Barney, GA, USA, 2008	Suwane	2	202	21.0	960	7	7	Pitted fruit	0.054	< 0.01	< 0.01
Blissfield, MI, USA, 2008	TNR 31	2	202	10.1	1990	7	7	Pitted fruit	0.17	< 0.01	0.017
Vernon, TX, USA, 2008	Loring	2	203	17.1	1180	7	7	Pitted fruit	0.14	< 0.01	< 0.01
Sanger, CA, USA, 2008	Tra-Zee	2	203	14.3	1420	7	6	Pitted fruit	0.12	< 0.01	< 0.01

Apricots

Table 11 Results of residue trials conducted with two (or four) applications of sulfoxaflor 240 g ai/L SC in/on apricots

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No	g ai/ha	g ai/100L	L/ha				Sulfoxaflor	X11719474	X11721061
Shepparton, Victoria, Australia 2008	Trevatt	2	193	14.8	1300	7	0 3 7 10	Pitted fruit	0.70	< 0.01	0.026
									0.46	< 0.01	0.036
									<u>0.42</u>	0.011	0.039
									0.40	0.010	0.043
		4	97	7.5	1300	7	0 3 7 10	Pitted fruit	0.29	< 0.01	0.011
									0.22	< 0.01	0.011
									0.18	< 0.01	0.017
									0.28	< 0.01	0.024
Hastings, Hawkes Bay, New Zealand 2008	Sundrop	2	190	9.5	2000	7	7	Pitted fruit	<u>0.15</u>	< 0.01	< 0.01
		4	95	4.8	2000	7	7	Pitted fruit	0.17	< 0.01	< 0.01

Plums

Table 12 Results of residue trials conducted with two applications of sulfoxaflor 240 g ai/L SC in/on plum

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No	g ai/ha	g ai/100L	L/ha				Sulfoxaflor	X11719474	X11721061
Nashdale, Australia, 2009	Autumn Giant	2	192	19.2	1000	7	3 7 14 28	Fruit, pitted	0.040	< 0.01	< 0.01
									<u>0.020</u>	< 0.01	< 0.01
									< 0.01	< 0.01	< 0.01
									< 0.01	< 0.01	< 0.01
Blissfield, MI, USA, 2009	NS	2	208 [240 SC]	6.93	3000	7	1 3 7 14	Fruit, pitted	0.27	< 0.01	< 0.01
									0.27	< 0.01	< 0.01
									0.21	0.011	< 0.01
									<u>0.26</u>	0.014	< 0.01
		2	202 [500 WDG]	15.7	1280	7	1 3 7 14	Fruit, pitted	0.37	0.012	< 0.01
									0.39	0.014	< 0.01
									0.36	0.015	< 0.01
									0.31	0.017	< 0.01
Fresno, CA, USA, 2009	NE Flavor	2	203 [240 SC]	10.8	1880	7	7	Fruit, pitted	0.062	< 0.01	< 0.01

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No	g ai/ha	g ai/100L	L/ha				Sulfoxaflor	X11719474	X11721061
		2	202 [500 WDG]	10.8	1870	7	7	Fruit, pitted	<u>0.090</u>	< 0.01	< 0.01
Porterville, CA, USA, 2009	NE Fortune	2	203 [240 SC]	12.3 12.3	1650 1660	7	7	Fruit, pitted	0.044	< 0.01	< 0.01
		2	204 [500 WDG]	12.3	1660	7	7	Fruit, pitted	<u>0.066</u>	< 0.01	< 0.01
Monmouth, OR, USA, 2008	Moyer	2	200 [240 SC]	13.8	1450	7	7	Fruit, pitted	0.045	< 0.01 < 0.01	< 0.01 < 0.01
		2	201 [500 WDG]	13.8	1460	7	7	Fruit, pitted	<u>0.054</u>	< 0.01	< 0.01
Dinuba, CA, USA, 2008	Black Fryers	2	202 [240 SC]	10.6	1900	7	7	Fruit, pitted	<u>0.030</u>	< 0.01	< 0.01
		2	205 [500 WDG]	10.6	1940	7	7	Fruit, pitted	0.017	< 0.01	< 0.01
Lindsay, CA, USA 2009	Angelina's	2	202 [240 SC]	20.9	960	7	7	Fruit, pitted	<u>0.11</u>	< 0.01	< 0.01
		2	201 [500 WDG]	20.8	960	7	7	Fruit, pitted	0.11	< 0.01	< 0.01

*Tree nuts**Almonds*

Table 13 Residue trials for sulfoxaflor in almonds

Location Year	Variety	Application				RTI	DAT	Portion	Residues (mg/kg)		
		No	g ai/ha	L/ha					Sulfoxaflor	X11719474	X11721061
Sanger, CA USA 2008	Ne Plus	2	202	1280		7	7	nutmeat	< 0.01	< 0.01	< 0.01
Kerman, CA USA 2008	Carmels	2	205	1870		7	3 7 14 21	nutmeat	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01
Visalia, CA USA 2008	Non-Pareil	2	200	1870		7	7	nutmeat	< 0.01	< 0.01	< 0.01
Madera, CA USA 2008	Non-Pareil	2	200	1880		7	7	nutmeat	0.012	< 0.01	< 0.01
Madera, CA USA 2008	Butte	2	203	1870		7	7	nutmeat	< 0.01	< 0.01	< 0.01
Terra Bella, CA 2008	Monterey	2	202	2980		7	7	nutmeat	< 0.01	< 0.01	< 0.01

Pecans

Table 14 Residue trials for sulfoxaflor in pecans

Location Year	Variety	Application				DAT	Portion	Residues (mg/kg)		
		No	g ai/ha	L/ha	RTI			Sulfoxaflor	X11719474	X11721061
Chula, GA, USA, 2009	Summer	2	202	1280	7	3 7 14 21	nutmeat	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01
Scooterville, GA, USA, 2009	Summer	2	202	1280	7	7	nutmeat	< 0.01	< 0.01	< 0.01
Sycamore, GA, USA, 2009	Summer	2	202	1270	7	7	nutmeat	< 0.01	< 0.01	< 0.01
Lonoke, AR, USA, 2009	Native/Stuart	2	202	960	7	7	nutmeat	< 0.01	< 0.01	< 0.01
Madill, OK, USA, 2009	Desirable	2	204	1140	7	7	nutmeat	< 0.01	< 0.01	< 0.01
Claytonville, TX, USA, 2009	Choctaw	2	204	1140	7	7	nutmeat	< 0.01	< 0.01	< 0.01

Animal Feeds

Table 15 Results of residue trials conducted with two applications of sulfoxaflor 240 g ai/L SC in/on almond hulls in the USA in 2008

Location Year	Variety	Application				DAT	Portion	Residues (mg/kg)		
		No	g ai/ha	L/ha	RTI			Sulfoxaflor	X11719474	X11721061
Sanger, CA USA 2008	Ne Plus	2	202	1280	7	7	hulls	3.1	0.097	0.15
Kerman, CA USA 2008	Carmels	2	205	1870	7	3 7 14 21	hulls	2.1 1.7 2.2 1.6	0.032 0.033 0.043 0.039	0.09 0.093 0.13 0.13
Visalia, CA USA 2008	Non-Pareil	2	200	1870	7	7	hulls	1.5	0.039	0.078
Madera, CA USA 2008	Non-Pareil	2	200	1880	7	7	hulls	1.2	0.028	0.047
Madera, CA USA 2008	Butte	2	203	1870	7	7	hulls	1.5	0.028	0.090
Terra Bella, CA 2008	Monterey	2	202	2980	7	7	hulls	2.2	0.040	0.14

APPRAISAL

Sulfoxaflor was first evaluated for residues and toxicological aspects by the 2011 JMPR. The 2011 Meeting established an ADI of 0–0.05 mg/kg bw and an ARfD of 0.3 mg/kg bw. The 2011 Meeting established a residue definition of sulfoxaflor for both compliance and dietary risk assessment in both plant and animal commodities. The 2011 Meeting estimated a number of maximum residue levels prior to registration of sulfoxaflor in any country as a pilot project.

After the subsequent meeting of CCPR, the proposed MRLs for citrus fruits, pome fruits, stone fruits and tree nuts were held at Step 4 because the provisional GAP reviewed by JMPR differed from the GAP finally approved in the USA.

The 2014 Meeting received information on registered use patterns for citrus fruit, pome fruit, stone fruit and tree nuts from the manufacturer and the residue data for those crops evaluated by the 2011 Meeting are reconsidered here against the submitted GAPs.

Results of supervised residue trials on crops

Citrus Fruit—Grapefruit, Lemon and Oranges

Registered use patterns from Australia and the USA were submitted. Supervised trial data for citrus were available from Australia, Brazil, and the USA.

Australian GAP for citrus fruit is for application at 9.6 g ai/100L (maximum of two applications, maximum 192 g ai/ha/single application, 1 day PHI).

USA GAP for citrus fruit is for application at 96 g ai/ha (14 day retreatment interval, maximum of four applications maximum 298 g ai/ha/ year, 1 day PHI).

None of the citrus fruit trials were conducted in accordance with the USA GAP. The majority of the Australian trials in oranges and mandarins were conducted at Australian GAP, while USA trials in oranges, grapefruit and lemons approximated Australian GAP after scaling.

The Meeting considered that US and Australian citrus fruit growing practices are similar, and noted the 2013 Meeting General Consideration item number 2.8 (Guidance for Use of Residue Trial Data from Different Geographical Locations for Estimation of Pesticide Residue Levels).

The Meeting determined that trials for all fruit will be related to Australian GAP for citrus fruit. The Meeting decided to use the concept of proportionality to estimate residue levels in citrus fruit in comparison to the Australian GAP where required. Scaled results for citrus fruit were within a range of 0.67–2.2× GAP, within the acceptable range for use of proportionality. Scaled results are indicated by an (s).

Grapefruit

Residue trials were conducted in grapefruit in the USA, approximating the critical GAP in Australia after scaling.

The results for grapefruit at a 1-day PHI after 2× 15.1–19.4 g ai/100L applications were: 0.010, 0.013, 0.016, 0.024, 0.11, and 0.13 mg/kg.

Residues in whole grapefruit after scaling to the Australian GAP were ≤ 0.01 (×3, all s), 0.015 (s), 0.064 (s) and 0.066 (s) mg/kg, where (s) indicates a result scaled to account for application rates outside $\pm 25\%$ of GAP.

Residues in pulp of grapefruit, in ranked order after scaling, were ≤ 0.01 (×3, all s) mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg for the subgroup shaddocks or pomelos. The Meeting noted that sulfoxaflor has systemic properties and considered that three edible portion data points was not sufficient for estimation of STMR and HR values. Therefore, the Meeting estimated an STMR of 0.0125 mg/kg and an HR of 0.066 mg/kg for sulfoxaflor in shaddocks or pomelos, based on the whole fruit data.

Lemons and limes

Residue trials were conducted in lemons in the USA, approximating the critical GAP in Australia after scaling.

Results for whole lemons at a 1-day PHI after $2 \times 16.2\text{--}21.2$ g ai/100L were: < 0.01 (2), 0.083, 0.11, and 0.29 mg/kg.

Residues in whole lemons after scaling to the Australian GAP where required were < 0.01 ($\times 2$, both s), 0.038 (s), 0.055 (s) and 0.17 (s) mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg for the subgroup lemons and limes, together with an STMR of 0.038 mg/kg, and an HR of 0.17 mg/kg.

Oranges and mandarins

Residue trials were conducted in oranges, in Australia approximating the critical GAP in Australia.

Residues of sulfoxaflor in whole oranges from the Australian trials at a 1-day PHI after $2 \times 6.4\text{--}20.2$ g ai/100L applications were: 0.09, 0.15, 0.16, 0.33, 0.41, and 0.43 mg/kg.

Scaled residues in whole oranges from the Australian trials with scaling to the Australian GAP where necessary were 0.09, 0.15, 0.19 (s), 0.24 (s), 0.33, and 0.43 mg/kg.

Residues trials were conducted in mandarins in Australia, at GAP.

Residues in whole mandarins from the Australian trials at a 1-day PHI after $2 \times 7.6\text{--}9.6$ g ai/100L applications were: 0.15, 0.28, 0.34, and 0.44 mg/kg.

The Meeting noted that the medians for the Australian datasets for oranges and mandarins differed by less than fivefold (medians differed by a factor of only 1.4 $\times$). The similarity of the datasets was further confirmed by the Mann-Whitney U-test. The Meeting concluded that the orange and mandarin datasets were mutually supportive and agreed to combine them for the purpose of estimation of maximum residue levels for the subgroups oranges, sweet, sour and mandarins.

The combined Australian data set for oranges and mandarins is 0.09, 0.15 (2), 0.19, 0.24, 0.28, 0.33, 0.34, 0.43, and 0.44 mg/kg.

The Meeting estimated maximum residue levels of 0.8 mg/kg for the subgroup oranges, sweet, sour and the subgroup mandarins, together with STMR values of 0.26 mg/kg and HR values of 0.44 mg/kg.

The Meeting withdrew the previous maximum residue level recommendation of 0.9 mg/kg for sulfoxaflor in citrus fruit.

Pome fruit - Apples and Pears

Registered use patterns from Australia and the USA were submitted. Supervised trials data were available for apples and pears from Australia/ New Zealand, Europe, and the USA.

Australian GAP for pome fruit is for application at 9.6 g ai/100L (maximum of two applications, maximum 192 g ai/ha/ single application, 7 day PHI).

USA GAP for pome fruit is for application at 96 g ai/ha (7 day retreatment interval, maximum of four applications maximum 298 g ai/ha/ year, 7 day PHI).

The Meeting noted that none of the trials were conducted in accordance with the USA GAP, while a number of the trials from both Australia/New Zealand and the USA matched the Australian GAP.

The Meeting considered that the US and Australian pome fruit growing practices are similar, and noted the 2013 Meeting General Consideration item number 2.8.

The Meeting determined that trials for both apples and pears will be related to Australian GAP for pome fruit. The Meeting decided to use the concept of proportionality as appropriate to estimate residue levels in pome fruit in comparison to the Australian GAP (trials for which proportionality was used were within 1.3–2.2 $\times$ GAP, within the acceptable range).

Residue trials were conducted in apples and pears in Australia/ New Zealand, approximating the GAP in Australia.

Results in apples and pears from the Australia/New Zealand trials at a 7-day PHI after 2× 9.1–16.1 g ai/100L applications were 0.02, 0.065, 0.07, 0.11, 0.14, 0.19, and 0.22 mg/kg.

Residues in apples and pears from the Australia/New Zealand trials were 0.015 (s), 0.039 (s), 0.07, 0.11, 0.14, 0.19, and 0.22 mg/kg (STMR = 0.11 mg/kg), where (s) indicates a result scaled to account for application rates outside ± 25% of GAP.

Residue trials were conducted in apples and pears in the EU. Results at a 7-day PHI after 2× 10.1–21.0 g ai/100L applications were: 0.052, 0.058, 0.074, 0.078, 0.099, 0.10, 0.18 (3), and 0.27 mg/kg.

Residues in apples and pears from the European trials scaled according to the Australian GAP are: 0.025 (s), 0.028 (s), 0.036 (s), 0.048 (s), 0.071 (s), 0.078, 0.082 (s), 0.082 (s), 0.086 (s), and 0.13 (s) mg/kg (STMR = 0.075 mg/kg).

Residue trials were conducted in apples and pears in the USA.

USA apple and pear results at a 7-day PHI after 2× 7.4–19.8 g ai/100L applications were: < 0.01, 0.039, 0.040, 0.043, 0.056, 0.063, 0.064, 0.066, 0.068, 0.072, 0.075, 0.089, 0.12, 0.13, 0.16, 0.18, and 0.26 mg/kg.

Residues in apples and pears from the USA trials scaled to the Australian GAP where necessary were < 0.01 (s), 0.039, 0.040, 0.043, 0.043 (s), 0.045 (s), 0.055 (s), 0.056, 0.057 (s), 0.063, 0.066, 0.068, 0.075 (s), 0.092 (s), 0.16, 0.18, and 0.23 mg/kg (STMR = 0.057 mg/kg).

The Meeting considered that the seven data points from the Australia/New Zealand apple and pear trials were not sufficient for estimation of a group maximum residue level for pome fruit. The Meeting noted that results from Europe and the USA relevant to the Australian GAP were available and combined the Australian, European and USA data sets:

- < 0.01, 0.015, 0.025, 0.028, 0.036, 0.039 (2), 0.040, 0.043, 0.043, 0.045, 0.048, 0.055, 0.056, 0.057, 0.063, 0.066, 0.068, 0.07, 0.071, 0.075, 0.078, 0.082 (2), 0.086, 0.092, 0.11, 0.13, 0.14, 0.16, 0.18, 0.19, 0.22, and 0.23 mg/kg

The Meeting estimated a maximum residue level of 0.3 mg/kg for sulfoxaflor in pome fruit, together with an STMR of 0.067 mg/kg and an HR of 0.23 mg/kg.

The Meeting withdrew the previous maximum residue level recommendation of 0.4 mg/kg for sulfoxaflor in pome fruit.

Stone Fruit

Registered use patterns from Australia and the USA were submitted. Supervised trial data were available for apricot (Australia and New Zealand), cherries (Australia, EU and USA), nectarine (Australia and New Zealand), peach (Australia, EU and USA) and plums (Australia and USA).

Australian GAP for stone fruit is for application at 7.2 g ai/100L (maximum of two applications, maximum 144 g ai/ha/ single application, 7-day PHI).

USA GAP for stone fruit is for application at 96 g ai/ha (7-day retreatment interval, maximum of four applications maximum 298 g ai/ha/ year, 7-day PHI).

Insufficient trials were conducted in accordance with the USA GAP, while a significantly greater number were conducted in accordance with the Australian GAP, and further trials could be related to the Australian GAP through the use of proportionality (trials for which results were scaled were within 1.3–2.9× of GAP, within the acceptable range).

The Meeting considered that the US and Australian stone fruit growing practices are similar, and noted the 2013 Meeting General Consideration item number 2.8.

Therefore trials for all stone fruit will be related to the Australian GAP for stone fruit.

Cherries subgroup

A total of 14 trials on cherries were available from Australia/ New Zealand (1 each), Europe (6), and USA (6).

Australia/New Zealand cherry results at a 7-day PHI after 2×9.7 – 16.3 g ai/100L applications were 0.35 and 0.38 mg/kg.

Residues of sulfoxaflor in cherries from Australia were: 0.17 (s) and 0.35 mg/kg (STMR = 0.26 mg/kg) where (s) indicates a result scaled to account for application rates outside $\pm 25\%$ of GAP.

European cherry results at a 7-day PHI after 2×10.1 – 20.1 g ai/100L applications were: 0.54, 0.77, 0.80, 0.90, 0.98 and 1.5 mg/kg.

Residues of sulfoxaflor measured in cherries from in Europe in accordance with Australian GAP (scaled where necessary) were: 0.29 (s), 0.32 (s), 0.33 (s), 0.38 (s), 0.42 (s), and 0.54 (s) mg/kg (STMR = 0.355 mg/kg).

USA cherry results at a 7-day PHI after 2×8.6 – 21.0 g ai/100L applications were: 0.55, 0.59, 0.76, 1.0, and 1.2 (2) mg/kg.

Residues of sulfoxaflor in cherries from USA in accordance with the Australian GAP (scaled where necessary) were: 0.24 (s), 0.26 (s), 0.26 (s), 0.42 (s), 0.46 (s) and 1.2 mg/kg (STMR = 0.34 mg/kg).

The Meeting noted that there were insufficient residue trials conducted in Australia/New Zealand in accordance with the Australian GAP, and combined the Australia, American and European datasets for the purpose of estimating a maximum residue level for the cherries subgroup.

Residues in cherries from trials conducted in Australia/New Zealand, EU countries and the USA were:

- 0.17, 0.24, 0.26 (2), 0.29, 0.32, 0.33, 0.35, 0.38, 0.42 (2), 0.46, 0.54, and 1.2 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg for the subgroup cherries, together with an STMR of 0.34 mg/kg, and an HR of 1.24 mg/kg (unrounded result).

Peaches subgroup

A total of five trials on nectarines were available from Australia and New Zealand. The results for nectarines at a 7-day PHI after 2×9.8 – 19.7 g ai/100L applications were: 0.10, 0.11, 0.12, 0.14, and 0.18 mg/kg.

Residues of sulfoxaflor in nectarines from Australia and New Zealand were: 0.037 (s), 0.054 (s), 0.061 (s), 0.10 (s) and 0.12 (s) mg/kg (STMR = 0.061 mg/kg) where (s) indicates a result scaled to account for application rates outside $\pm 25\%$ of GAP.

Two trials on apricots were available from Australia and New Zealand. The results for apricots at a 7-day PHI after 2×9.5 – 14.8 g ai/100L applications were: 0.15 and 0.42 mg/kg.

Residues of sulfoxaflor apricots from Australia/New Zealand in accordance with the Australian GAP (scaled where necessary) were: 0.11 (s) and 0.20 (s) mg/kg (STMR = 0.155 mg/kg).

Eight trials in peaches were conducted in Australia and New Zealand. The results at a 7-day PHI after 2×9.7 – 19.8 g ai/100L applications were: 0.012, 0.11, 0.11, 0.12, 0.14, 0.15, 0.24, and 0.27 mg/kg.

Residues of sulfoxaflor in peaches from Australia and New Zealand (scaled to the Australian GAP where required) were: < 0.01 (s), 0.040 (s), 0.050 (s), 0.052 (s), 0.057 (s), 0.094 (s), 0.16 (s) and 0.20 (s) mg/kg (STMR = 0.0545 mg/kg).

The Meeting noted that the GAP under consideration is for Australia, and that a large regional (Australia/New Zealand) data set for the peach group is available (15 trials in total for peaches, nectarines and apricots), and that the median values of these data sets are within a factor of $5 \times$ each

other (the medians differed by a maximum factor of 2.8×). The Meeting agreed to combine the Australia/New Zealand peach, nectarine and apricot data for the purpose of estimating a maximum residue level for the peach subgroup:

- < 0.01, 0.037, 0.04, 0.05, 0.052, 0.054, 0.057, 0.061, 0.094, 0.10, 0.11, 0.12, 0.16, and 0.20 (2) mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg for the subgroup peaches, together with an STMR of 0.061 mg/kg and an HR of 0.2 mg/kg.

Plums

A total of seven trials on plums were available from Australia (1) and USA (6).

The Australian result for plums at a 7-day PHI after 2× 19.2 g ai/100L applications was: 0.020 mg/kg.

Residues of sulfoxaflor measured in plums from Australia were: < 0.01 (s) mg/kg where (s) indicates a result scaled to account for application rates outside ± 25% of GAP.

The USA results for plums at a 7-day PHI after 2× 6.9–20.9 g ai/100L applications were: 0.030, 0.054, 0.066, 0.090, 0.11, and 0.26 mg/kg.

Residues of sulfoxaflor in plums from the USA in accordance with the Australian GAP (scaled where necessary) were: 0.020 (s), 0.028 (s), 0.038 (s), 0.039 (s), 0.06 (s) and 0.26 mg/kg.

Residues in plums from trials conducted in Australia and the USA were: < 0.01, 0.020, 0.028, 0.038, 0.039, 0.06, and 0.26 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg for the subgroup plums, together with an STMR of 0.038 mg/kg, and an HR of 0.26 mg/kg.

The Meeting withdrew the previous maximum residue level recommendation of 3 mg/kg for sulfoxaflor in stone fruit.

Tree Nuts

USA GAP for tree nuts (Crop Group 14) including almonds, cashew, chestnut, hazelnut, macadamia, pecan and walnut is for application at 96 g ai/ha (7 day retreatment interval, maximum of four applications maximum 298 g ai/ha/ year, 7 day PHI).

Residues data from trials conducted in the USA were available for almonds and pecans. However, the trials do not match the USA GAP, with only two applications being made at a rate of 200–205 g ai/ha with a 7-day PHI. The Meeting therefore did not estimate maximum residue levels, STMRs or HRs for the tree nut group.

The Meeting agreed to withdraw the previous maximum residue level recommendation of 0.015 mg/kg for sulfoxaflor in tree nuts.

Animal feeds

Almond hulls

Residue data for sulfoxaflor in almond hulls were available to the Meeting. However, as the trials were not conducted in accordance with the USA GAP for almonds, the Meeting did not estimate a median residue value.

Fate of residues during processing

The 2011 Meeting received information on the fate of sulfoxaflor residues during the processing of apple to juice, sauce and wet and dry pomace; cherry to dried cherries, jam and juice and oranges to juice, wet and dry pulp, oil and peel.

Calculated processing factors are summarized in the following table based on the JMPR 2014 recommendations for MRLs and STMRs. Factors are indicated with a '<' (less-than) sign when the residue in the processed commodity is below the LOQ of the analytical method. The calculation is then made on the LOQ of the analytical method and the residue concentration of the RAC (raw agricultural commodity).

Processes included in the table are those that lead to STMR-P or HR-P values useful for dietary intake estimations or for livestock dietary burden calculations.

Raw Agricultural Commodity (RAC)	Processed Commodity	Best Estimate Processing Factor (PF)	RAC MRL (mg/kg)	RAC STMR (mg/kg)	RAC HR (mg/kg)	Processed commodity STMR-P (mg/kg)	Processed commodity HR-P (mg/kg)
Apple	Wet pomace	1.1	0.3	0.067	0.23	0.074	–
	Dry pomace	4.2				0.28	–
	Juice	0.4				0.027	–
	Sauce	0.6				0.040	–
Cherry	Juice	0.8	1.5	0.34	1.24	0.27	–
	Jam	1.1				0.37	–
	Dried	5.1				1.73	6.32
Orange	Juice	0.14	0.8	0.26	0.44	0.036	–
	Wet pulp	2.5				0.65	–
	Dried pulp	8.3				2.16	–
	Oil	< 0.2				< 0.052	–
	Peel	5.6				1.46	2.45

Animal commodities

The Meeting recalculated the livestock dietary burden based on the uses considered by the current Meeting and by the 2011 Meeting on the basis of diets listed in the FAO Manual Appendix IX (OECD Feedstuff Table).

The maximum dietary burden is 3.22 ppm for beef and dairy cattle, while the mean dietary burden is 1.26 ppm for beef and dairy cattle. The values calculated by the 2011 Meeting were: maximum dietary burden of 3.04 ppm for beef cattle and 2.68 ppm for dairy cattle, and a mean dietary burden of 0.91 ppm for both dairy and beef cattle. Interpolation of these values between the appropriate feeding levels in the lactating cattle feeding study considered by the 2011 Meeting showed that no changes to the maximum residue levels estimated by the 2011 Meeting for milks, edible offal (mammalian) or meat (from mammals other than marine mammals) were required.

The maximum and mean dietary burdens for poultry (both layers and broilers) are 0.93 and 0.31 ppm respectively. These have changed very little from the values determined by the 2011 Meeting (maximum and mean values of 0.89 and 0.30 ppm respectively).

The Meeting confirmed its previous recommendations for meat (from mammals other than marine mammals), edible offal (mammalian), milks, poultry meat, poultry, edible offal of, and eggs.

The Meeting noted that the 2011 Meeting did not estimate maximum residue levels for mammalian fats (except milk fats) or poultry fats.

The Meeting noted the STMR values of 0.03 mg/kg and HR value of 0.073 mg/kg estimated by the 2011 Meeting for the fat compartment of mammalian meat (from mammals other than marine mammals). Noting that the dietary burden has not significantly increased, the Meeting estimated a maximum residue level of 0.1 mg/kg for mammalian fats (except milk fats), together with an STMR of 0.03 mg/kg and an HR of 0.073 mg/kg.

The Meeting noted the STMR values of 0.005 mg/kg and HR value of 0.021 mg/kg estimated by the 2011 Meeting for the fat compartment of poultry meat. Noting that the dietary burden has not significantly increased, the Meeting estimated a maximum residue level of 0.03 mg/kg for poultry fats, together with an STMR of 0.005 mg/kg and an HR of 0.021 mg/kg.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI assessment.

Definition of the residue (for compliance with the MRL and for estimation of dietary intake) for plant and animal commodities: *sulfoxaflor*

The residue is not fat soluble

The Meeting estimated the maximum residue levels and STMR values shown below.

Commodity		MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
CCN	Name	New	Previous		
FS 0013	Cherries	1.5	–	0.34	1.24
FC 0001	Citrus fruits	W	0.9	–	–
FC 0002	Lemons and limes	0.4	–	0.038	0.17
FC 0003	Mandarins	0.8	–	0.26	0.44
MF 0100	Mammalian fats (except milk fats)	0.1	–	0.03	0.073
FC 0004	Oranges, Sweet, Sour	0.8	–	0.26	0.44
FS 2001	Peaches	0.4	–	0.061	0.20
FS 0014	Plums	0.5	–	0.038	0.26
FP 0009	Pome fruits	0.3	0.4	0.067	0.23
PF 0111	Poultry fats	0.03	–	0.005	0.021
FC 0005	Pomelos and Grapefruit	0.15	–	0.0125	0.066
FS 0012	Stone fruits	W	3	–	–
TN 0085	Tree nuts	W	0.015	–	–
	Apple juice			0.027	–
	Apple sauce			0.040	–
	Cherry juice			0.27	–
	Cherry jam			0.37	–
	Cherry, dried			1.73	6.32
	Orange juice			0.036	–
	Orange oil			< 0.052	–
	Orange peel			1.46	2.46

Commodity		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
CCN	Name		
–	Apple pomace, wet	0.074	–
–	Apple pomace, dry	0.28	–
–	Citrus dried pulp	2.16	–

DIETARY RISK ASSESSMENT

Long-term intake

The evaluation of sulfoxaflor has resulted in recommendations for MRLs and STMRs for raw and processed commodities. The International Estimated Daily Intakes for the 17 GEMS/Food cluster diets, based on estimated STMRs were in the range 1–7% of the maximum ADI of 0.05 mg/kg bw (Annex 3 of the 2014 Report).

The Meeting concluded that the long-term intake of residues of sulfoxaflor, from uses that have been considered by the JMPR, is unlikely to present a public health concern.

Short-term intake

The International Estimated Short Term Intake (IESTI) for sulfoxaflor was calculated for the plant and livestock commodities (and their processing fractions) for which new STMRs and HRs were

estimated and for which consumption data were available. The results are shown in Annex 4 to the 2014 Report.

The IESTI varied from 0–9 % of the ARfD (0.3 mg/kg bw). The Meeting concluded that the short-term intake of residues of sulfoxaflor, from uses that have been considered by the JMPR, is unlikely to present a public health concern.

Author(s)	Year	Title, Source, Report No., Date,
Bacher, R.	2006	Independent laboratory validation of Bayer CropScience method No. 01009 for the determination of residues of JAU 6476-desthio, JAU 6476-3-hydroxy-desthio, JAU 6476-4-hydroxy-desthio, JAU 6476-3,4-dihydroxy-desthio, and JAU 6476-4,5-...PTRL Europe GmbH, Ulm, Germany, BCS, Report No.: P/B 1111 G, Edition Number: M-279818-01-1, Method Report No.: P613060597 Date: 2006-11-02, GLP/GEP: yes, unpublished.
Brumhard, B.; Stuke, S.	2007	Analytical method 01013 for the simultaneous determination of residues of the active Items BYF00587, prothioconazole, tebuconazole, trifloxystrobin and the metabolites BYF00587-desmethyl, JAU6476-desthio (SXX0665) and CGA321113 in/on ...Bayer CropScience, Report No.: 01013, Edition Number: M-283439-03-1 Method Report No.: MR-06/138 Date: 2007-01-25, ...Amended: 2008-02-18, GLP/GEP: yes, unpublished.
Ellis, A.	2012	Determination of residues of prothioconazole in peanut kernels, shells and fodder following three, four or five applications of Proline (JAU 6476) 480 SC at rates of 120 or 180 g a.i./ha at ten day intervals up to 14 days before harvest, Bayer CropScience Residue Laboratory, Brisbane, QLD, Australia, Report No.: BCS-0232, Report includes Trial Nos.: C351, C352, C353, C354, Edition Number: M-437933-01-1, Date: 2012-09-06, GLP/GEP: yes, unpublished.
Gould, T. J.; Timberlake, B. C.; Krolski, M. E.; Nguyen, S.; Moore, S.	2003	Bayer CropScience analytical method RPA JA/03/01: JAU6476: An analytical method for the determination of residues of JAU6476 and desthio-JAU 6476 in plant matrices using LC/MS-MS, Bayer CropScience LP, Stilwell, KS, USA, Bayer CropScience, Report No.: RPA JA/03/01, Edition Number: M-003024-01-1, Method Report No US: 200799, Method Report No US: RPA JA/03/01, GLP/GEP: yes, unpublished.
Gould, T. J.; Timberlake, B. C.	2010	An analytical method for the determination of residues of prothioconazole and desthio-prothioconazole in plant matrices using LC-MS/MS, Bayer CropScience LP, Stilwell, KS, USA, Bayer CropScience, Report No.: JA-001-P04-02, Edition Number: M-362015-01-1, EPA MRID No.: 47948901 Date: 2010-01-08, GLP/GEP: no, unpublished.
Gould, T. J.; Timberlake, B. C.	2008	Storage stability of prothioconazole in canola, wheat, mustard greens, turnip root, and tomato fruit, and processed products. Bayer CropScience LP, Stilwell, KS, USA, Bayer CropScience, Report No.: RAJAY016, Edition Number: M-300712-01-1, EPA MRID No.: 47408201, Date: 2008-04-18, GLP/GEP: yes, unpublished.
Heinemann, O.	2000	Analytical determination of residues of JAU6476 and JAU6476-desthio in/on cereals and canola by HPLC-MS/MS (method modification 00598/M001), Bayer AG, Leverkusen, Germany, Bayer CropScience, Report No.: 00598/M001, Edition Number: M-047681-01-1, Method Report No.: MR-689/99, EPA MRID No.: 46477703, GLP/GEP: yes, unpublished.
Schulte, G.; Oel, D.	2006	Analytical method 01009 for the determination of residues of JAU 6476-desthio, JAU 6476-3-hydroxy-desthio, JAU 6476-4-hydroxy-desthio, JAU 6476-3,4- dihydroxy-desthio, and JAU 6476-4,5-dihydroxy-desthio in/on matrices of animal origin by .Bayer CropScience, Report No.: MR-06/120, Edition Number: M-279725-02-1, Method Report No.: MR-06/120, Date: 2006-10-26, .Amended: 2013-01-25, GLP/GEP: yes, unpublished.
Bomke, S.; Bauer, J.	2011	Determination of the residues of penflufen and prothioconazole in/on potato after seed treatment, general of BYF 14182 & Prothioconazole FS 118 in the field in Germany, France (North), Belgium and the Netherlands, Bayer CropScience, Report No.: 10-2085, Report includes Trial Nos.: 10-2085-01, 10-2085-02, 10-2085-03, 10-2085-04, Edition Number: M-410267-02-1, Date: 2011-06-29, .Amended: 2011-07-25, GLP/GEP: yes, unpublished.
Bomke, S.	2012	Determination of the residues of AE C656948 and prothioconazole in/on maize/corn after spray application of AE C656948 & JAU 6476 SE 250 in southern France, Italy, Spain and Greece, Bayer CropScience, Report No.: 11-2110, Report includes Trial Nos.: 11-2110-02, 11-2110-03, 11-2110-04, 11-2110-05. Edition Number: M-442647-01-1, Date: 2012-11-28, GLP/GEP: yes, unpublished.

Author(s)	Year	Title, Source , Report No., Date,
Duah, F. K.; Harbin, A. M.	2006	JAU6476 480 SC - Magnitude of the residue in/on soybeans, Pyxant Labs, Inc., Colorado Springs, CO, USA, Bayer CropScience, Report No.: RAJAY026, Edition Number: M-270206-01-1, EPA MRID No.: 46841001, Date: 2006-04-28. GLP/GEP: yes, unpublished.
Ellis, A.	2012	Determination of residues of prothioconazole in peanut kernels, shells and fodder following three, four or five applications of Proline (JAU 6476) 480 SC at rates of 120 or 180 g a.i./ha at ten day intervals up to 14 days before harvest, Bayer CropScience Residue Laboratory, Brisbane, QLD, Australia, Report No.: BCS-0232, Report includes Trial Nos.:C351, C352, C353, C354. Edition Number: M-437933-01-1, Date: 2012-09-06. GLP/GEP: yes, unpublished.
Ellis, A.	2012	Determination of residues of prothioconazole in peanut kernels, shells and fodder following a range of applications of Rudis SC 480 at rates of 192 and 288 g a.i./ha at various timings. Bayer CropScience, Eight Mile Plains, QLD, Australia, Bayer CropScience, Report No.: BCS-0383, Report includes Trial Nos.: C668, C680, C682, C697, Edition Number: M-438813-01-1, Date: 2012-09-19, GLP/GEP: yes, unpublished.
Freitag, T.; Wolters, A.	2007	Determination of the residues of JAU 6476 and penycuron in/on potato after seed treatment of JAU 6476 & NTN 19701 (258 FS) in the field in the Netherlands, United Kingdom, Germany and Northern France, Bayer CropScience, Report No.: RA-2569/05, Report includes Trial Nos.: R 2005 0466/7 = 0466 - 05, R 2005 0873/5 = 0873 - 05, R 2005 0874/3 = 0874 - 05, R 2005 0875/1 = 0875 - 05, Edition Number: M-283397-01-1, Date: 2007-02-02, GLP/GEP: yes, unpublished.
Freitag, T.; Wolters, A.	2007	Determination of the residues of JAU 6476 and penycuron in/on potato after seed treatment of JAU 6476 & NTN 19701 (258 FS) in the field in Southern France, Italy and Spain, Bayer CropScience, Report No.: RA-2604/05, Report includes Trial Nos.: R 2005 0881/6 = 0881 - 05, R 2005 0882/4 = 0882 - 05, R 2005 0883/2 = 0883 - 05, R 2005 0884/0 = 0884 - 05. Edition Number: M-283206-01-1. Date: 2007-01-30, GLP/GEP: yes, unpublished.
Freitag, Th.; Wolters, A.	2007	Determination of the residues of JAU 6476 and penycuron in/on potato after spraying of JAU 6476 & NTN 19701 (258 FS) in the field in Southern France, Italy and Spain, Bayer CropScience, Report No.: RA-2570/05, Report includes Trial Nos.: R 2005 0467/5 = 0467 - 05, R 2005 0877/8 = 0877 - 05, R 2005 0878/6 = 0878 - 05, R 2005 0879/4 = 0879 - 05. Edition Number: M-283404-01-1, Date: 2007-02-02, GLP/GEP: yes, unpublished.
Freitag, T.; Wolters, A.	2007	Determination of the residues of JAU 6476 and penycuron in/on potato after spraying of JAU 6476 & NTN 19701 (258 FS) in the field in Germany, Northern France and the Netherlands, Bayer CropScience, Report No.: RA-2603/05, Report includes Trial Nos.: R 2005 0880/8 = 0880 - 05, R 2005 0885/9 = 0885 - 05, R 2005 0886/7 = 0886 - 05, R 2005 0887/5 = 0887 - 05, Edition Number: M-283415-01-1, Date: 2007-02-02, GLP/GEP: yes, unpublished.
Freitag, Th.; Reineke, A.	2009	Determination of the residues of prothioconazole and tebuconazole in/on maize/corn after spraying of HWG 1608 & JAU 6476 in the Field in Germany, the Netherlands and the United Kingdom, Bayer CropScience, Report No.: 08-2035, Report includes Trial Nos.:08-2035-01, 08-2035-02, 08-2035-03, 08-2035-04, Edition Number: M-358275-01-1 Date: 2009-10-19, GLP/GEP: yes, unpublished.
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Krolski, M. E.; Harbin, A. M.	2008	JAU6476 480 SC- Magnitude of the residue in/on corn field corn, pop corn, and sweet corn, Bayer CropScience LP, Stilwell, KS, USA, Bayer CropScience, Report No.: RAJAP004, Edition Number: M-304689-01-1, EPA MRID No.: 47521901, Date: 2008-07-25, GLP/GEP: yes, unpublished.

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Murphy, I.; Dallstream K.	2012	Proline 480 SC - Magnitude of the residue in/on bushberry (crop subgroup 13-07B) Bayer CropScience LP, Stilwell, KS, USA, Bayer CropScience, Report No.: RAJAP022, Report includes Trial Nos.: JA001-10HA, JA002-10HA, JA003-10HA, JA004-10DA, JA005-10HA, JA006-10HA, JA007-10DA, JA008-10HA, JA009-10HA, JA010-10HA, JA011-10HA, Edition Number: M-428253-01-1, EPA MRID No.: 48803301, Date: 2012-03-23, GLP/GEP: yes, unpublished.
Murphy, I.; Dallstream, K. A.	2012	Proline 480 SC - Magnitude of the residue in/on cranberry, Bayer CropScience LP, Stilwell, KS, USA, Bayer CropScience, Report No.: RAJAP023, Report includes Trial Nos.: JA012-10HA, JA013-10HA, JA014-10HA, JA015-10DA, JA016-10HA, JA017-10HA, Edition Number: M-428331-01-1, EPA MRID No.: 48803302, Date: 2012-04-03, GLP/GEP: yes, unpublished.
Murphy, I.; Harbin, A.	2012	Proline 480 SC - Magnitude of the residue in/on cucurbit vegetables (CG 9), Bayer CropScience LP, Environmental Science, Stilwell, KS, USA, Bayer CropScience, Report No.: RAJAP024, Edition Number: M-428931-01-1, EPA MRID No.: 48803303, Date: 2012-04-05 GLP/GEP: yes, unpublished.
Resende, G.	2013	Determinação de resíduos de bixafen, prothioconazole e trifloxystrobin e seus respectivos metabólitos na cultura da soja após a pulverização de bixafen & prothioconazole & trifloxystrobin 450 SC juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil, Bayer S/A, Sao Paulo, Brazil, Bayer CropScience, Report No.: F12-012, Report includes Trial Nos.: F12-012-01, F12-012-02, F12-012-03, F12-012-04, F12-012-05, Edition Number: M-448125-02-1, Method Report No.: VM12-006, Date: 2013-03-01, Amended: 2013-05-06, GLP/GEP: no, unpublished.
Anon.	2013	CGA 279202 & BYF 00587 & JAU 6476; SC 450; soybean; Brazil; BBA, Bayer S/A, Sao Paulo, Brazil, Bayer CropScience, Report No.: M-453756-01-2, Report includes Trial Nos.: F12-012-01, F12-012-02, F12-012-03, F12-012-04, F12-012-05, Edition Number: M-453756-01-2, Date: 2013-04-15, GLP/GEP: no, unpublished.
Resende, G.	2013	Determinação de resíduos de Bixafen, Prothioconazole e Trifloxystrobin e seus respectivos metabólitos na cultura da Soja após aplicação de Bixafen & Prothioconazole & Trifloxystrobin 450 SC, juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil, Bayer CropScience, Sao Paulo, Brazil, Bayer CropScience, , Report No.: F13-008, Report includes Trial Nos.: F13-008-01, F13-008-02, F13-008-03, F13-008-04, F13-008-05, Edition Number: M-465974-01-1, Date: 2013-10-01, GLP/GEP: yes, unpublished.
Anon.	2013	Prothioconazole & trifloxystrobin & bixafen; 450SC (175 + 150 +125); Soybean; Brazil; BBA Bayer CropScience, Bayer CropScience, , Report No.: M-472259-01-1, Report includes Trial Nos.: F13-008-01, F13-008-02, F13-008-03, F13-008-04, F13-008-05, Edition Number: M-472259-01-1, Date: 2013-10-01, GLP/GEP: yes, unpublished
Santiago, L.	2012	Determinação de resíduos de prothioconazole e trifloxystrobin e seus respectivos metabólitos na cultura do milho após a pulverização de Fox (325 SC) juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil, Bayer S.A., Bayer CropScience, Sao Paulo, Brazil, Bayer CropScience, Report No.: F11-034, Report includes Trial Nos.: F11-034-01, F11-034-02, F11-034-03, F11-034-04, Edition Number: M-435783-01-1, Date: 2012-07-31, GLP/GEP: yes, unpublished.
Anon	2012	JAU 6476 & CGA 279202; SC 325; maize; Brazil; BBA, Bayer S.A., Bayer CropScience, Sao Paulo, Brazil, Bayer CropScience, Report No.: M-444409-01-1, Report includes Trial Nos.: F11-034-01, F11-034-02, F11-034-03, F11-034-04, F11-034-05, Edition Number: M-444409-01-1, Date: 2012-07-31, GLP/GEP: no, unpublished.
Santiago, L.	2013	Determinação de resíduos de bixafen, prothioconazole e trifloxystrobin e seus respectivos metabólitos na cultura do milho após a pulverização de bixafen & prothioconazole & trifloxystrobin 450 SC juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil, Bayer S/A, Sao Paulo, Brazil, Bayer CropScience, Report No.: F12-011, Report includes Trial Nos.: F12-011-01, F12-011-02, F12-011-03, F12-011-04, F12-011-05, Edition Number: M-448104-01-1, Date: 2013-03-01, GLP/GEP: yes, unpublished.
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Freitag, T.; Reineke, A.; Krusell, L.	2010	Amendment No. 0002 to Report No. 08-2112 - Determination of the residues of BYF 00587 and prothioconazole in/on winter rape after spraying of bixafen & prothioconazole EC 225 in the field in Belgium, France (North) and Germany, Bayer CropScience, Report No.: 08-2112, Report includes Trial Nos.: 08-2112-01, 08-2112-02, 08-2112-03, 08-2112-04, Edition Number: M-385740-03-1, Date: 2010-07-07, .Amended: 2012-04-10, GLP/GEP: yes, unpublished.
Freitag, Th.; Hoffmann, M.	2010	Determination of the residues of BYF 00587 and prothioconazole in the processed fractions of winter rape (oil, screwpressed; pomace; meal; oil, solvent extracted; oil, crude; crude oil, preclarified; crude oil, neutralized and oil, refined) after spraying of Bixafen & Prothioconazole EC 225 in the field in Germany, Bayer CropScience, , Report No.: 08-3112, Report includes Trial Nos.: 08-3112-01, Edition Number: M-392874-02-1 Date: 2010-10-12, .Amended: 2011-05-02, GLP/GEP: yes, unpublished.
Krolski, M. E.; Harbin, A. M.	2008	JAU6476 480 SC - Magnitude of the residue in/on field corn processed commodities, Bayer CropScience LP, Stilwell, KS, USA, Bayer CropScience, Report No.: RAJAP001, Edition Number: M-304727-01-1, EPA MRID No.: 47521903, Date: 2008-07-25, GLP/GEP: yes, unpublished.

THIAMETHOXAM (245)

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EXPLANATION

Thiamethoxam and clothianidin (including CGA322704 as a metabolite of thiamethoxam) were evaluated for toxicology and residues as a new compound in 2010, resulting in a number of MRL recommendations. Additional residue data for both compounds were evaluated in 2011 and 2012. The residue definition for thiamethoxam in plant commodities for enforcement is thiamethoxam, while the residue definition for dietary risk assessment is thiamethoxam and the metabolite CGA322704 (clothianidin), considered separately. The residue definition for clothianidin in plant commodities for enforcement and dietary risk assessment is clothianidin.

For the current evaluation the Meeting received data on analytical methods, use patterns, and supervised residue trials in support of the use on avocado, hops, legume vegetables and mint (USA IR-4 programme) and persimmon (Republic of Korea, (RoK)). Summaries of the trials have been provided to support MRL recommendations for thiamethoxam and clothianidin on the respective crops.

RESIDUE ANALYSIS

The Meeting received descriptions of analytical methods used for analysis of thiamethoxam and CGA322704 in mango, persimmon, avocado, fresh beans, mint and hop samples from the trials.

Method AG-675 (used for avocado, mint and hop samples) has been evaluated previously for other animal and plant commodities by the JMPR in 2010. Crop specific modifications are described below. Method REM 197.03 was used on the trials in beans. This method has also been evaluated by the JMPR in 2010. The current Meeting received additional validation data, based on historical findings, for the analytical method REM 179.06 of thiamethoxam and clothianidin. The results are summarized below. The two analytical methods LC-MS/MS Method 954/2010 and Method HPLC/DAD to determine thiamethoxam and CGA322704 (clothianidin) in mango and persimmon, respectively, were not previously evaluated by the JMPR and are described below.

Validation results are required for every commodity submitted for MRL-setting: at least one full validation for a commodity within the five defined crop groups (high acid content, high water content, high oil content, high protein content, high starch content) and a reduced validation for every other commodity within a certain crop group. Where validation results do not meet the criteria given below, this is indicated.

When the analytical method is validated according to a full validation scheme, it means that

- at least 5 recovery experiments per level were conducted on at least 2 levels (LOQ and $10\times$ LOQ) and average recovery per level was shown to be between 70–120% and the relative standard deviation (RSDr or CV) per level was shown to be $< 20\%$,
- at least two control samples were analysed and were shown to be below $0.3\times$ LOQ and
- the calibration was conducted with at least 5 single points or at least 3 duplicate points and was shown to be linear (either standards in solvent or matrix matched standards).
- When the analytical method is validated according to a reduced validation scheme, it means that
- a full validation is available for a crop in the same crop group (high acid content, high water content, high oil content, high protein content, high starch content);
- at least 3 recovery experiments per level were conducted on at least 2 levels (LOQ and $10\times$ LOQ) and the average recovery per level was shown to be between 70–120% and the relative standard deviation (RSDr or CV) per level was $< 20\%$;

- at least two control samples were analysed and shown to be below $0.3 \times \text{LOQ}$
- the calibration was conducted with at least 5 single points or at least 3 duplicate points and was shown to be linear (only relevant for matrix matched standards; standards in solvent are already covered by full validation).

LC-MS/MS Method 054/2010

For the trials on mango [Thirion, 2013a-c, A9795B_11446, 11447 and 11448] performed in 2012/2013 the SABS In-house method was used with reference 054/2010.

Dried mango samples (processing study) were first soaked in water. Subsamples of (fresh) mango (10 g) were extracted with water/methanol (1:1) and filtered through a funnel with Celite. The extract was diluted with water and adjusted to pH with a 10% NH_3 solution. Clean-up was performed by elution with water and methanol over a SPE cartridge and evaporated to dryness. The residue was redissolved in methanol/water (1:1 v/v) and filtered for LC-MS/MS analysis using m/z 292 to 211 for thiamethoxam and m/z 250 to 132 for CGA322704.

The LC-MS/MS SABS In-House method 054/2010 was validated for determination of thiamethoxam and CGA322704 (clothianidin) residues in mango and whole fruit (peel and flesh together, peel and dried mango in the range of 0.01 mg/kg-1.0 mg/kg with a limit of quantification of 0.1 mg/kg. Acceptable concurrent recovery data were obtained for thiamethoxam (66–102, 9–10% RSD) and CGA322704 (clothianidin) (60–96, 7–13% RSD) in each matrix at 0.01–1 mg/kg. The specificity of the method was demonstrated by analysing control samples of each matrix. No background interference was experienced at the limit of quantification of 0.01 mg/kg. Linearity was demonstrated by using linear regression analysis of the detector response area of both compounds against the compounds in the calibration standard (calibration curves existed of at least four different concentrations of the two compounds).

HPLC/DAD method (persimmon), further to be referred to as Method MFDS201304

In the residue trials for persimmon [Kim, 2012, MFDS201304] a HPLC/DAD analytical method, not previously described in JMPR 2010, 2011, or 2012, was used. Fresh persimmon (25 g) was extracted with acetone and filtered over filter paper. The flask and filter cake were rinsed again with acetone. The combined filtrate and rinsate was partitioned with a mixture of water, NaCl and dichloromethane (100:50:100 v/v). The organic layer was filtered through an anhydrous Na_2SO_4 layer (20 g). The filter cake was rinsed with dichloromethane. The pooled organic extract was evaporated to dryness at 40 °C by rotary vacuum evaporation. The residue was redissolved with 5 mL of methanol:water (50:50 v/v) solution. Samples were analysed by HPLC with diode array detector (DAD) with a mobile phase of methanol:water of 30:70 (first experiment) or 25:75 (second experiment). The reported LOQ was 0.02 mg/kg for both parent and CGA322704 (clothianidin).

There was no interference with other substances observed at the retention times of thiamethoxam and CGA322704 (clothianidin) above 30% of the LOQ. Acceptable concurrent recovery data were obtained for each analyte (85.9–99.4%) at 0.02, 0.2, and 2 mg/kg spike levels. Linearity was demonstrated by using linear regression analysis of the detector response area of both compounds against the compounds in the calibration standard (calibration curves existed of at least four different concentrations of the two compounds).

Method AG-675

For the trials on avocado [Syngenta, 2008, 464507, T010202-06], hops [Syngenta, 2013a, A9549C_50012, TK0053990 and Starner, 2006, CGA293343/2850, 08451] and mint and mint oil [Syngenta, 2001, 406892, 07362] LC-MS/MS method AG-675 was used. This method has been evaluated by the JMPR in 2010 for various animal and plant matrices (e.g. apple, cotton seed, sorghum forage), but not yet for avocado, mint and hop.

Avocado samples (fruits without stones) were extracted with acetonitrile/water (80:20 v/v), filtered through Celite under vacuum, and the diluted extracts analysed directly by LC-MS/MS using m/z 292 to 211 for thiamethoxam and m/z 250 to 132 for CGA322704.

Mint top samples (leaves and stems) were extracted with acetonitrile/water (80:20 v/v), filtered under vacuum, and the filter cake re-extracted. The combined filtered extracts were cleaned-up by liquid/liquid partition followed by GPC. The resulting extracts were analysed directly by LC-MS/MS using m/z 292 to 211 for thiamethoxam and m/z 250 to 132 for CGA322704.

For mint oil, the extraction procedure was not necessary. The mint oil was diluted with the GPC mobile phase (ethyl acetate/cyclohexane, 50:50 v/v) and cleaned-up by GPC. The resulting extract was analysed directly by LC-MS/MS using m/z 292 to 211 for thiamethoxam and m/z 250 to 132 for CGA322704.

Hop samples (dry cones) were extracted with acetonitrile/water (80:20 v/v), filtered through Celite under vacuum, and the filter cake re-extracted. Aliquots of the combined filtered extracts were concentrated, buffered with 50 mM sodium phosphate, filtered and cleaned-up by solid phase extraction (SPE) using phenyl sorbent cartridges. The resulting extracts were diluted and analysed by LC-MS/MS using m/z 292 to 211 for thiamethoxam and m/z 250 to 132 for CGA322704.

Method REM 179.06

For the trials on fresh beans [Kang, 2005a, CGA293343/2217, CEMR-2185, Kang 2005b, CGA293343/2218, CEMR-2186, Rawle, 2005a, CGA293343/2203, CEMR-2011, Rawle, 2005b, CGA293343/2204, CEMR-2202, Kang, 2005c, CGA293343/2633, CEMR-2322] method REM 179.06 was used to establish the residue levels. This method has been evaluated JMPR evaluation 2010. For the current evaluation, the Meeting received an independent method validation for this method.

Independent method validation REM 179.06

Method REM 179.06 was subject to an independent laboratory validation for residue analysis of several crops. The validation consists of a collation of data retrieved for the commodity crops as specified in the OECD Guidance on pesticide Residue Analytical Methods and an additional dry matrix [Anderson and Crook, 2013, CGA293343_11624, TK0122383]. The recovery data are included in tables 1 (thiamethoxam) and 2 (GA322704 (clothianidin)). Sufficient recovery data are available for the majority of the described crops (high water content crops, high protein content crops, high starch content crops, high acid content crops and dry content crops for both thiamethoxam as well as for CGA322704 (clothianidin)). The method has however been used only on a limited number and range of high oil content crops. At present insufficient recovery data are available to permit extrapolation to all high oil content crops. The LOQ of the method is 0.02 mg/kg for each analyte for all crops tested. Linearity was sufficiently demonstrated.

Table 1 Analytical recoveries for spiked thiamethoxam in various substrates using method REM 179.06 LC-MS/MS

Commodity	Spike con, mg/kg	n	Mean recov%	Range recov%	RSD (%)	Ref
High water content crops						
Pome fruit						
Apple fruit	0.02	5	99.8 (109, 97, 92, 99, 102 ^a)	92-109	6.3	CEMR-3520, CEMR-2324, CEMR-2323, CEMR-3521, T010193-05
	0.20-2.0	6	95.2 (101, 92 98, 97, 96, 87 ^{a,b})	87-101	5.2	
Stone fruit						
Cherry fruit	0.02	13	94.2 (79, 101, 100, 80, 96, 78, 94, 112, 88, 84, 109, 103, 100)	79-112	12.1	T000641-06, T000642-06, CEMR-2330, CEMR-2329, CEMR-2444, CEMR-2331, 05-0416, 05-0506, 05-0417,
	0.2-2.0	15	94.6 (93, 91, 96, 86, 88, 96, 84, 88, 109, 103, 101,	84-109	8.9	

Commodity	Spike con, mg/kg	n	Mean recov%	Range recov%	RSD (%)	Ref
			108, 84, 94, 88)			05-0406
Peach fruit	0.02	4	91.8 (91, 95, 98, 83)	83-98	7.1	T001232-06
	0.20	4	96.8 (95, 103, 99, 90)	90-103	5.7	
Plum fruit	0.02	12	92 (101, 93, 102, 79, 100, 95, 96, 83, 81, 88, 91, 95)	79-102	8.5	05-0522, 03-5067, 03-5068, 03-1029, 03-1028, CEMR-2328, 05-0702
	0.20	12	98.8 (107, 101, 101, 107, 124, 97, 100, 88, 90, 87, 85, 92)	85-124	11.0	
Bulb vegetables						
Bulb onions	0.02	2	83 (81, 84)			FSGD-106
	0.05	2	110 (97, 122)			
Fruiting vegetables and cucurbits						
Tomato fruit	0.02	18	93.7 (101, 95, 99, 86, 91, 81, 106, 76, 90, 108, 97, 100, 72, 86, 105, 98, 98)	72-108	10.8	CEMR-3518, CEMR-3519, 03-1030, 03-1006, 03-1020, CEMR-2321, CEMR2320, T000796-06, T001231-06, T000803-06
	0.04-0.2	16	95.4	77-109	9.2	
Pepper fruit	0.02	3	99.7 (101, 86, 112)	86-112	13.1	03-1008, CEMR-2318, CEMR-2319
	0.10-0.20	3	100.3	90-113	11.6	
Cucumber	0.02	4	104.3 (105, 102, 107, 103)	102-107	2.1	CEMR-2204, CEMR-2317, CEMR-2316, T002058-06
	0.20	4	101.8 (107, 104, 101, 95)	95-107	5.0	
Melon and squash	0.02	5	102.6 (99, 105, 103, 102, 104)	99-105	2.2	CEMR-2206, T000807-06, CEMR-3471
	0.20	5	101.4 (106, 90, 105, 105, 101)	90-106	6.6	
Brassica vegetables						
Broccoli inflorescence	0.02	13	95 (98, 94, 104, 91, 108, 96, 111, 89, 74, 86, 90, 95, 94)	74-111	10.2	CEMR-2199, CEMR-2200, CEMR-2315, 05-0504, S08-00739, T001198-09, S11-00619, FSGD-102
	0.20-1.0	13	98 (99, 98, 101, 112, 97, 95, 109, 111, 87, 70, 100, 97, 96)	70-112	11.1	
Broccoli whole plant	0.02	3	98 (92, 102, 101)	92-102	5.6	05-0504
	0.20	3	101 (111, 96, 95)	95-111	8.9	
Sprout buttons	0.02	7	99 (105, 96, 80, 110, 106, 95, 103)	80-110	10.1	T011024-06, S11-00620, FSGD-103
	0.2	7	86 (99, 100, 76, 84, 79, 83, 82)	76-100	11	
Cabbage head/plant	0.02	13	94 (79, 105, 80, 105, 102, 81, 96, 100, 90, 99, 103, 80)	79-105	11.4	05-0701, 05-0507, S09-01595, FSGD-099, FSDG-105, S10-0214, S11-00621
	0.2-0.4	13	96 (95, 85, 90, 101, 101, 101, 92, 93, 107, 87, 98, 98, 87)	85-107	7.0	
Cauliflower inflorescence	0.02	12	92 (91, 100, 87, 102, 96, 76, 91, 106, 79, 87, 102, 84)	76 - 106	10.5	05-0521, 05-0703, S09-01604, S08 00745, T000558-08, T001200-09, FSGD-104, S10-00965, S11-00622
	0.2-2.0	14	95 (100, 95, 93, 92, 95, 87, 82, 111, 101, 91, 103, 87, 98, 89)	82-111	8.0	
Cauliflower	0.02	3	93	84-106	12.2	05-0521, 05-0703

Commodity	Spike con, mg/kg	n	Mean recov%	Range recov%	RSD (%)	Ref
whole plant			(84, 106, 90)			
	0.2-2.0	3	93 (92, 84, 103)	84-103	10.3	
Kale leaves	0.02	1	106	106		FSGD-141
	0.2-2.0	1	89	89		
Leafy vegetables						
Lettuce	0.02	33	97 (99, 110, 105, 99, 92, 109, 93, 97, 97, 98, 96, 85, 82, 95, 91, 88, 99, 89, 108, 93, 112, 108, 112, 81, 110, 88, 110, 73, 86, 110, 86)	73-112	10.8	03-1018, 03-1016, 05-0428, 05-0902, T000805-06, T000804-06, T001230-06, CEMR-3516, CEMR-3517, CEMR-2191, CEMR-2194, 03-4007, 03-4002, 03-4008, 03, 4001, 03-4005, gel515103
	0.1-5.0	33	96 (99, 104, 100, 96, 106, 100, 104, 103, 92, 92, 95, 94, 99, 92, 99, 96, 84, 106, 98, 105, 77, 97, 88, 97, 105, 88, 103, 88, 103, 67, 82, 103, 89)	67-106	9.4	
Forage crops and seedlings						
Alfalfa	0.02	13	96 (99, 99, 110, 89, 87, 119, 96, 75, 95, 96, 101, 105, 75)	75-119	13.0	03-4011, 03-4012, T011008-06, 05-0612, VEMR-2313, CEMR-2314, S08-00735
	0.2-2.0	13	92 (109, 96, 85, 95, 93, 89, 72, 69, 9, 92, 97, 99, 111)	69-111	12.7	
Seedlings (carrot and lettuce)	0.02	8	100 (103, 94, 107, 96, 98, 99, 100, 99)	94-107	4.1	S09-02707, S09-01605, S09-01607
	0.2-25	10	97 (90, 94, 99, 91, 96, 110, 95, 96, 98, 100)	91-110	5.8	
Stem vegetables						
Artichoke flower heads	0.02-0.2	2	108 (113, 102)	102-113	7.2	S08-00742
Fresh legume vegetables						
Beans and peas with and without pods	0.02	27	92 (95, 86, 69, 100, 105, 95, 91, 87, 107, 104, 92, 91, 99, 92, 103, 92, 99, 94, 100, 95, 78, 7, 86, 95, 72, 73)	69-107	11.1	CEMR-2186, CEMR-2185, CEMR-2202, CEMR-2322, 03-8005, 04-8005, 04-8000, 05-0520, FSGD-095, FSGD-098, FSGD-097
	0.2-0.4	27	91 (93, 87, 95, 94, 86, 99, 93, 93, 95, 92, 91, 90, 90, 88, 90, 97, 100, 88, 91, 88, 91, 86, 78, 95, 83, 83)	78-100	5.5	
Beans and peas, remaining plant	0.02	20	91 (91, 98, 94, 102, 116, 104, 92, 72, 76, 75, 107, 84, 98, 100, 64, 91, 95, 109, 64, 91)	64-116	16	CEMR02322, 05-0520, FSGD-095, FSGD-098, FSGD-097
	0.2-0.4	20	85 (83, 97, 99, 86, 90, 92, 84, 93, 71, 71, 89, 79, 97, 79, 76, 75, 90, 92, 76, 75)	71-99	16.6	
High oil content crops						
OSR seeds and maize kernels	0.02	4	90 (82, 89, 80, 108)	80-108	14.2	05-0418, 03-4016
	0.2	4	98 (91, 72, 124, 105)	72-124	22.4	
High protein content crops						
Dry pea and bean seed	0.02	5	86 (97, 95, 86, 68, 82)	68-97	13.6	FSGD-094-REG
	0.2	5	105 (110, 106, 106, 102, 101)	101-110	3.4	
High starch content crops						
Carrot root	0.02	5	95 (99, 85, 111, 89, 90)	85-111	11.0	CEMR-3470, FSGD-096, S11-00615
	0.05-0.2	5	95 (97, 84, 110, 91, 92)	84-110	10.2	
Potato tuber	0.02	7	98 (102, 96, 98, 92, 101, 87, 108)	87-108	7.1	CEMR-2326, 05-0407, 05-0408, CEMR-2325,

Commodity	Spike con, mg/kg	n	Mean recov%	Range recov%	RSD (%)	Ref
	0.2-0.5	7	98 (104, 99, 97, 92, 105, 91, 99)	91-105	5.5	T014228-05, S08-00750
Wheat grain	0.2	1	105	105	-	S08-00736
	0.5	1	102	102	-	
High acid content crops						
Citrus (mandarin, orange)	0.02	7	99 (96, 96, 105, 104, 100, 96, 97)	96-105	4.0	03-1001, 03-1004
	0.1	7	102 (103, 99, 110, 102, 100, 97, 100)	97-110	4.1	
Kiwi	0.02	8	97 (78, 95, 104, 96, 101, 100, 103, 100)	78-104	8.6	T014177-05, T011091-06
	0.2-0.5	6	102 (93, 101, 110, 117, 94, 96)	93-117	9.5	
Grapes	0.02	8	88 (83, 93, 83, 86, 99, 80, 93, 89)	80-99	7.3	05-0901
	0.2	8	95 (96, 101, 90, 96, 98, 85, 98, 93)	85-101	5.4	
Berries; rasp- and strawberry	0.02	19	94 (104, 93, 80, 94, 87, 85, 92, 109, 90, 88, 100, 90, 102, 103, 83, 80, 100, 100, 100)	80-109	9.2	055-0307, -5-0308, 05-0423, 05-0422, 05-0421, 05-0420, T014214-05, T014215-05, S09-01598, S11-01604
	0.10-2.0	20	99 (105, 97, 103, 96, 100, 102, 107, 110, 104, 110, 86, 105, 104, 89, 81, 92, 98, 95, 98, 102)	81-110	7.8	
Dry crops						
Wheat straw (2), maize plant (1), dry pulse haulm (5)	0.02	8	95 (82, 92, 94, 95, 102, 88, 107, 100)	82-107	8.4	S08-00736, CEMR-2198, FSGD-094-REG
	0.2-0.5	8	91 (90, 77, 90, 96, 96, 95, 94, 93)	77-96	6.9	

^a Wax apple (T010193)^b One value at fortification level 2.0 mg/kg

Table 2 Analytical recoveries for spiked CGA322704 (clothianidin) in various substrates using method REM 179.06 LC-MS/MS

Commodity	Spike con, mg/kg	n	Mean and individual recoveries %	Recovery Range %	RSD (%)	Ref
High water content crops						
Pome fruit						
Apple fruit	0.02	7	97 (108, 105, 87, 87, 94, 93, 105)	87-108	9.2	CEMR-3520, CEMR-2324, CEMR-2323, CEMR-3521, T010193-05
	0.20-2.0	4	92 (102, 89, 92, 86)	86-102	7.5	
Stone fruit						
Cherry fruit	0.02	13	90 (78, 74, 81, 72, 99, 72, 86, 109, 102, 92, 107, 99, 96)	72-109	14.9	T000641-06, T000642-06, CEMR-2330, CEMR-2329, CEMR-2444, CEMR-2331, 05-0416, 05-0506, 05-0417, 05-0406
	0.2-2.0	15	92 (89, 74, 88, 91, 93, 93, 80, 85, 108, 93, 96, 102, 93, 96, 94)	74-108	8.9	
Peach fruit	0.02	4	94 (90, 101, 94, 92)	90-101	5.1	T001232-06
	0.20	4	96 (92, 104, 94, 95)	92-104	5.5	
Plum fruit	0.02	12	91 (99, 80, 99, 98, 92, 85, 92, 73, 94, 92, 89, 97)	73-99	8.8	05-0522, 03-5067, 03-5068, 03-1029, 03-1028, CEMR-2328, 05-0702
	0.20	12	97 (120, 106, 98, 95, 86, 91, 83, 101,	83-120	10.1	

[illegible]

Commodity	Spike con, mg/kg	n	Mean and individual recoveries %	Recovery Range %	RSD (%)	Ref
Citrus (mandarin, orange)	0.02	7	96 (93, 83, 101, 103, 95, 92, 102)	83-103	7.4	03-1001, 03-1004
	0.1	7	101 (100, 103, 99, 109, 99, 101, 99)	99-101	3.6	
Kiwi	0.02	8	91 (71, 79, 92, 101, 105, 96, 92, 90)	71-105	12.3	T014177-05, T011091-06
	0.2-0.5	6	98 (86, 93, 110, 116, 96, 85)	85-116	13.0	
Grapes	0.02	8	90 (80, 91, 78, 89, 98, 82, 105, 99)	78-105	10.9	05-0901
	0.2	8	96 (97, 99, 96, 95, 100, 81, 105, 95)	81-105	7.2	
Berries; rasp- and strawberry	0.02	19	95 (102, 93, 113, 74, 96, 103, 100, 86, 105, 96, 95, 102, 82, 99, 82, 89, 94, 103, 93)	74-113	9.9	055-0307, -5-0308, 05-0423, 05-0422, 05-0421, 05-0420, T014214-05, T014215-05, S09-01598, S11-01604
	0.10-2.0	20	96 (100, 97, 94, 93, 95, 100, 98, 96, 97, 100, 95, 98, 102, 88, 78, 98, 99, 90, 101)	78-102	5.8	
Dry crops						
Wheat straw (2), maize plant (1), dry pulse haulm (5)	0.02	8	90 (81, 104, 75, 82, 92, 90, 103, 93)	75-104	11.5	S08-00736, CEMR-2198, FSGD-094-REG
	0.2-0.5	8	88 (95, 77, 77, 91, 91, 90, 91, 89)	77-95	7.7	

^a Wax apple (T010193)

Stability of pesticide residues in stored analytical samples

The freezer storage stability of thiamethoxam and metabolite CGA322704 (clothianidin) at residue concentrations was already established for by the JMPR 2010 evaluation of thiamethoxam in apples, tomatoes, potato tubers, rape seed, maize grain, cranberries, hops, barley grain, barley hay, barley straw, pearled barley and barley flour. Thiamethoxam, and metabolites CGA322704 (clothianidin) and CGA265307 were apparently stable at residue concentrations in the various substrates tested at the freezer temperatures and test durations. The durations of test were mostly 1–2 years, but some were less. Test temperatures were mostly approximately -18 °C to -20 °C, but other storage temperatures were used in some storage stability tests, e.g., between -26 °C and -4 °C. The JMPR 2010 evaluation of clothianidin has established that parent clothianidin was stable when stored at temperatures of -10 °C or lower for at least 24 months in crops with high water content (apple, Japanese pear, apricot, peach, cauliflower, head cabbage, cucumber, tomato, lettuce, maize and forage), for at least 18 months in crops with high acid content (cranberries and grapes), for at least 24 months in crops with high oil content (dry soya beans, cottonseed, rape and seed), for at least 24 months in crops with high starch content (maize grain, rice grain, sugar beet roots and potatoes), for at least 10 months in dry tea leaves, for at least 24 months in maize straw, for at least 2 months in tomato paste, for at least 4 months in cotton meal, and for at least 4 months in cotton oil. The storage stability data as provided in the different residue trials submitted for the current evaluation have been summarized in Table 3.

Table 3 Storage stability data of thiamethoxam and CGA322704 (clothianidin)

Commodity	Residue	Storage time (days)	% remaining mean range RSD _r	concurrent recovery	reference, method
persimmon	Thiamethoxam Fresh	31-52	95.1%, RSD 2.1 (n=5)	89.2-94.8% (fortification level 0.02, 0.2, 2.0 mg/kg)	Kim 2012, MFDS201304, HPLC/DAD 2011 data
	Thiamethoxam Freezer	31-52	92.7%, RSD 1.8 (n=5)		

Commodity	Residue	Storage time (days)	% remaining mean range RSD _r	concurrent recovery	reference, method
	CGA322704 Fresh	31-52	85.9%, RSD 3.1 (n=5)	85.9-99.4% (fortification level 0.02, 0.2, 2.0 mg/kg)	
	CGA322704 Freezer	31-52	87.1%, RSD 3.4 (n=5)		
persimmon	Thiamethoxam Fresh	14-35	85.3%, RSD 4.0 (n=5)	83.1-93.5% (fortification level 0.032, 0.2, 0.05, 2.0 mg/kg)	Kim 2012, MFDS201304, HPLC/DAD 2012 data
	Thiamethoxam Freezer	14-35	85.0%, RSD 5.6 (n=5)		
	CGA322704 Fresh	14-35	82.0%, RSD 4.2 (n=5)	81.5-85.5% (fortification level 0.02, 0.2, 0.5, 2.0 mg/kg)	
	CGA322704 Freezer	14-35	81.2%, RSD 5.1 (n=5)		
avocado	Thiamethoxam	290	85%, s.d. 2 (n=3)	77.3 ± 3% (n=5, fortification levels 0.01-0.10 mg/kg)	Syngenta 2008, 464507, Method AG-675
	CGA322704	290	98%, s.d. 3 (n=3)	93 ± 5% (n=5, fortification levels 0.01-0.10 mg/kg)	
mint, tops	Thiamethoxam	452	90, 91, and 102% of 0.5 mg/kg	77-85% (means of fortification levels 0.05, 0.5, and 5.0 (n=4, 7, 4))	Syngenta, 2001, 406892, Method AG-675
	CGA322704	452	113, 114, and 121% of 0.05 mg/kg	83.9-96.5 % (means of fortification levels 0.05, 0.5, and 5.0 (n= 4, 7, 4))	
mint, oil	Thiamethoxam	465	98, 103, and 109% of 0.05 mg/kg	92-116% (means of fortification levels 0.05, 0.5, and 5.0 mg/kg (n=5, 6, 4))	
	CGA322704	465	118, 125, and 125% of 0.05 mg/kg	92-111% (means of fortification levels 0.05, 0.5, Ans 5.0 mg/kg (n= 5, 6, 4))	

USE PATTERN

Copies or English translations of thiamethoxam labels from the following countries were made available to the Meeting from USA, Portugal, South Africa and Korea.

Table 4 Registered uses of thiamethoxam

Crop	Country	Form	Application				PHI, days
			Method	Rate kg ai/ha	Spray conc, kg ai/hL	Number (max rate/season)	
avocado	USA	(25%) WG	Foliar	0.070	0.015	3 ^a (max 0.21 kg ai/ha)	0
fresh beans	Portugal	(25%) WG	Foliar ^b	0.100	0.010	1-2 (max 0.200 kg ai/ha/crop season)	3
fresh beans	Portugal	(25%) WG	Drip irrigation ^c	0.100-0.200		1-2 (max 0.200 ai/ha) ^c	3
hops	USA	(75%) SG	Soil	0.140 ^d		1	65
mango	South Africa	(240 g/L) SC	Soil	1.44 g ai/tree (6 ml/tree)	0.144	1	130
mint	USA	(25%) WG	Foliar ^e	0.0263-0.0525	0.028-0.112	4-8 (max 0.210 kg ai/ha)	7
mint	USA	(25%) WG	Foliar ^e	0.0525-	0.056-0.149	3-4	7

Crop	Country	Form	Application				PHI, days
			Method	Rate kg ai/ha	Spray conc, kg ai/hL	Number (max rate/season)	
				0.0700		(max 0.210 kg ai/ha)	
Persimmon (Japanese)	Korea	(10%) WG	Foliar	- ^f	0.005	3 (interval 10 days)	7

^a Do not exceed 0.21 kg ai/ha (equiv. to 0.188 lbs ai/acre) per growing season. Minimum of 7 days interval. Do not use less than 467 L/ha (50 GPA) for ground application. Use sufficient water volume to cover all foliage.

^b Indoor and outdoor use, according to Portuguese label. Interval of 7 days. For outdoor use only apply after flowering.

^c Only indoor use according to Portuguese label.

^d Apply specified dosage in sufficient water volume to ensure uniform application and incorporation into the soil using one of the following methods: 1. Apply a surface band on each side of the row out to the plant canopy drip line or within the vegetation free herbicide strip, followed by sufficient irrigation to incorporate the product into the plants root zone. 2. apply by chemigation into the root zone through low-pressure microsprinkler, trickle or drip type irrigation systems. 3. hill drench in sufficient water to ensure incorporation into the root zone followed by irrigation.

^e 0.11-0.21 and 0.21-0.28 kg product/ha (equiv. to 1.5-3.0 or 3.0-4.0 oz product/A) for peppermint and spearmint. Max of 0.21 kg ai/ha (equiv. to 0.188 lb ai/A) per growing season. Do not use less than 467 L/ha (50 GPA) for ground application. Use sufficient water volume to cover all foliage. Minimum interval 14 days.

^f The spray application is commonly conducted until the fluid is dripping down the leaves.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on supervised residue trials of thiamethoxam for avocado, mango, fresh common beans, hops, mint, and persimmon. The commodities are grouped into Codex commodity groups.

Crop group	Commodities	Table No.
Pome fruit	Persimmon: Korea	Table 5
Assorted tropical fruits – inedible peel	Avocado: USA	Table 6
Assorted tropical fruits – inedible peel	Mango: South Africa	Table 7
Legume vegetables	Fresh beans: Portugal	Table 8
Fresh herbs	Mint: USA	Table 9
Dried herbs	Hops: USA	Table 10

Application rates and spray concentrations have been rounded to three figures; residues have been rounded to two figures. Residue data are recorded unadjusted for percentage recoveries or for residue values in control samples unless otherwise stated. Unquantifiable residues are shown as below the reported LOQ (e.g. < 0.01 mg/kg). Where multiple samples were taken from a single plot or where multiple analyses were conducted on a single sample, the average value is reported. Where results from separate plots with distinguishing characteristics such as different formulations, crop varieties or treatment schedules were reported, results are listed separately for each plot. Residues from the trials conducted according to critical GAP have been used for the estimation of maximum residue levels, STMR and HR values. Those results are underlined.

The residues presented in the tables are given as individual compounds (parent and CGA322704, separately).

Pome fruits

Japanese persimmon

Six supervised residue field trials were conducted on Japanese persimmon in the 2011–2012 growing season in the Korea [Kim, 2012, MFDS201304].

Small plots (80 m²) of 12–32 year old persimmon trees were treated three times with a WG formulation at a nominal rate of 156–438 g ai/ha (all with spray concentrations of 0.005 kg ai/hL) with a 10–11 days interval at growth stage BBCH 77–79. The plots were treated as indicated in Table 5 using a hand sprayer.

Samples of persimmon (6–51 kg) were taken at maturity at six time points after the final application. Growth stages were not reported. Seeds and stalk were removed from the persimmon before preparation. Subsequently, the samples were chopped and thoroughly mixed. All samples were stored frozen for 31–52 days (2011 trials) and 14–35 days (2012 trials) at -20 °C. The stability of thiamethoxam and clothianidin (CGA322704) during storages was tested (recovery was 85.9–95.1% for the 2011 trials and 81–85% for 2012 trials).

Samples were analysed for parent and clothianidin (CGA322704) with a HPLC/DAD method (Method MFDS201304) with an LOQ of 0.02 mg/kg. The method is described in the analytical section of the evaluation.

Concurrent recoveries were 89.2–94.8% and 85.9–99.4% for parent and CGA322704, respectively at fortification levels of 0.02, 0.2 and 2.0 mg/kg in the 2011 trials and 83.1–93.5% and 81.5–85.5% for parent and CGA322704, respectively at fortification levels of 0.02, 0.2, 0.5 and 2.0 mg/kg in the 2012 trials.

Note: The analytical method is considered valid in the range 0.02–2.0 mg/kg for the determination of thiamethoxam and CGA322704 in persimmon.

Table 5 Residues of thiamethoxam and metabolite CGA322704 (clothianidin) after pre-harvest treatment of Japanese persimmon (fruits)

Location, year, (variety) Soil type	Form	No	Interval (days)	g ai/ha	L/ha	method, last application	DAT	parent, mg/kg ^a	CGA322704 mg/kg ^a	Author, Report, Trial
Woodong-ri ^b , Gyeongnam province, Republic of Korea (Fuyu Persimmon), Sandy loam	WG	3	10 10	210 214 211	4200 4275 4225	Foliar spray, 26 Sept, 2011 BBCH 77-79	0 1 3 7 14 21	0.27 0.22 0.20 0.15 0.09 0.08	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Kim, 2012, MFDS201304, Field 1
Mosan-ri ^b , Gyeongnam province, Republic of Korea (Fuyu Persimmon), Loam	WG	3	10 10	300 275 280	6000 5500 5600	Foliar spray, 26 Sept, 2011 BBCH 77-79	0 1 3 7 14 21	0.24 0.21 0.13 0.14 0.13 0.08	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Kim, 2012, MFDS201304, field 2
Hwacheon-ri ^b , Gyeongnam province, Republic of Korea (Fuyu Persimmon), Sandy loam	WG	3	10 10	156 156 158	3125 2110 3150	Foliar spray, 26 Sept, 2011 BBCH 77-79	0 1 3 7 14 21	0.25 0.28 0.19 0.13 0.12 0.08	0.02 0.02 0.02 0.02 0.02 0.03	Kim, 2012, MFDS201304, Field 3
Noyeon-ri ^b , Gyeongnam province, Republic of Korea (Fuyu Persimmon), Sandy clay loam	WG	3	10 11	437 438 438	8740 8750 8765	Foliar spray, 08 Oct, 2012 BBCH 77-79	0 1 3 7 14 21	0.29 0.25 0.25 0.19 0.10 0.04	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Kim, 2012, MFDS201304, Field 4
Masan-ri ^b , Gyeongnam province, Republic of	WG	3	10 11	400 385 388	8000 7700 7750	Foliar spray, 08 Oct, 2012 BBCH 77-	0 1 3 7	0.27 0.25 0.22 0.18	< 0.02 < 0.02 < 0.02 < 0.02	Kim, 2012, MFDS201304, Field 5

Location, year, (variety) Soil type	Form	No	Interval (days)	g ai/ha	L/ha	method, last application	DAT	parent, mg/kg ^a	CGA322704 mg/kg ^a	Author, Report, Trial
Korea (Fuyu Persimmon), Sandy clay loam						79	14 21	0.13 0.07	< 0.02 < 0.02	
Gamgye-ri, Gyeongnam province ^b , Republic of Korea (Fuyu Persimmon), Sandy clay loam	WG	3	10 11	360 350 368	7200 7000 7350	Foliar spray, 08 Oct, 2012 BBCH 77-79	0 1 3 7 14 21	0.28 0.24 0.16 0.14 0.12 0.05	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Kim, 2012, MFDS201304, Field 6

^a Results are mean values of 4 replicate samples.

^b Trials were performed in the same region (<10 km apart)

Assorted (sub) tropical fruits with inedible peel

Avocado

Supervised residue field trials were conducted on avocado in the 2006–2007 growing seasons in the USA and Mexico [Syngenta, 2008, 464507, T010202-06]. Plots (size not reported) of avocado trees were treated thrice with a 25 WG formulation at an application rate of approximately 70 g ai/ha (0.0625 lbs ai/A) using boom sprayers using 93–3738 L/ha (10–400 GPA).

Avocados were harvested at two time points after the final application at maturity (growth stages not reported). Samples were collected from (24 fruits from at least 4 separate trees) reaching at least 2 kg. Stems and pits were removed at time of sampling. All samples were stored frozen for a maximum of 9 months at -20 °C. This storage period is covered by the storage stability studies (24 months).

Samples were analysed for parent and its metabolite CGA322704 using HPLC-MS/MS method Syngenta Analytical Method AG-675, with an LOQ of 0.01 mg/kg for parent and metabolite. Residues of thiamethoxam and CGA322704 from the trials are summarised in Table 6. Residues of each of these analytes from untreated plots were less than the LOQ (0.01 mg/kg). Average concurrent recoveries at 0.01 and 0.14 mg/kg were within 70–120% for each analyte (range 72–99%).

Note: The analytical method is considered valid in the range 0.01–0.14 mg/kg for the determination of thiamethoxam and CGA322704 in avocado.

Table 6 Residues of thiamethoxam and metabolite CGA322704 (clothianidin) after pre-harvest treatment of avocado in RAC and (fruits without pits)

Location, year, (variety) Soil type	Form	No	Interval (days)	g ai/ha	kg ai/hL	method, last application	DAT	parent, mg/kg ^a	CGA322704 mg/kg ^a	Ref, trial
Irvine CA, USA, 2007 (Hass), fine sandy loam	SC	3	7 6	70.1 70.1 70.1	18.7 18.4 18.5	Backpack spraying, 19 March, 2007, Fruit ripening	0 3	0.03 (0.04) <u>0.06</u> (0.08)	< 0.01 <u>< 0.01</u>	Syngenta 464507, 2008, 09607.06-CA116
Porterville, CA, USA, 2006 (Zutano), clay	SC	3	7 7	70.1 70.1 70.1	5.2 5.2 5.1	Airblast spraying, 14 November, 2006, fruiting	0 3	0.03 (0.04) <u>0.03</u> (0.04)	< 0.01 <u>< 0.01</u>	Syngenta 464507, 2008, 09607.06-CA117
Homestead, FL, USA, 2006	SC	3	7 7	70.7 70.7	7.3 7.3	Tractor mounted	0 3	< 0.01 <u>< 0.01</u>	< 0.01 <u>< 0.01</u>	Syngenta 464507,

Location, year, (variety) Soil type	Form	No	Interval (days)	g ai/ha	kg ai/hL	method, last application	DAT	parent, mg/kg ^a	CGA322704 mg/kg ^a	Ref, trial
(Booth 7), loam				70.7	7.3	spraying, 22 September, 2006, fruiting				2008, 09607.06-FL51
Los Reyes, Mexico, 2006 (Hass), loamy sand	SC	3	7 6	64.1 70.2 71.1	75.4 75.5 69.7	Backpack spraying, 03 November, 2006, mature	0 3	<u>0.24</u> (0.30) 0.13 (0.16)	0.01 (0.01) <u>0.02</u> (0.02)	Syngenta 464507, 2008, 01-IR-06-7255
Los Reyes, Mexico, 2006 (Hass), sandy loam	SC	3	6 6	67.6 68.4 68.1	7.2 7.4 7.4	Backpack spraying, 03 November 2006, mature	0 3	<u>0.10</u> (0.12) 0.08 (0.10)	< 0.01 <u>0.02</u>	Syngenta 464507, 2008, 01-IR-06-7256

^a Results are the average of two replicate analytical samples. Original data $\times$ 0.80 conversion factor to represent the RAC whole fruit (original data representing the fruit without the stone).

Mango

The Meeting received information on supervised residue trials on mango conducted in South Africa in the growing seasons 2003/2004 and 2011/2012. The data from 2003/2004 were already evaluated by the JMPR in 2010. The data from 2011/2012 were submitted to supplement the original studies [Thirion, 2013a, Syngenta A9795B_11446, 2418/F787] and the results are presented in Table 7. In addition residue data for thiamethoxam after an exaggerated application (3X) were generated for determining processing factors for thiamethoxam and CGA322704 in mango [Thirion, 2013b, and c, Syngenta A9795B_11447, 2418/F788 and Syngenta A9795B_1148, 2418/G676]. These results are summarized in the section on processing.

Plots (462–1111 trees/ha, 5–8 trees/plot) of mango trees were treated once with a 240 SC formulation at an application rate of 6 or 12 ml diluted in 1 or 2 litre of water and poured around the tree with a jug at a rate of , respectively 1.4 or 2.9 g/tree.

Mangoes were harvested at maturity at one or more time points after the treatment. Samples were collected (8–24 fruits from at least 8 separate trees) ranging from 1.3 to >6 kg. Despite picking more than 12 items (21 to 24) in trial ZA13 RMG 001-2012, the samples sizes were < 2 kg at BBCH 75 and 76 (DAT 61 and 74, respectively). The samples sizes at DAT 118 and more were $\geq$ 3.4 kg. In addition, at DAT 89, 79 and 81 in the same trail only 8 items were picked instead of the required 12. In the other three trials the weight of the sample and the number of picked fruits was sufficient. All samples were stored frozen at <-18 °C and processed and analysed within one year. This storage period is covered by the storage stability studies (24 months).

Samples were analysed for parent and its metabolite CGA322704 using SABS in-house Method No 054/2010 with an LOQ of 0.01 mg/kg for both parent and metabolite. Results were not corrected for control levels (< 0.01 mg/kg for each analyte) nor for average concurrent method recoveries (72–92% for each analyte at 0.01, 0.03, 0.05 and 0.10 mg/kg). Residues of thiamethoxam and CGA322704 from the trials are summarised in Table 7.

Table 7 Residues of thiamethoxam and metabolite CGA322704 (clothianidin) after pre-harvest treatment of mango (whole fruits)

Location, year, (variety)	Form	No	g ai/tree	method, last application	DAT	Portion analyzed ^a	parent, mg/kg	CGA322704 mg/kg	Author, Study number, Trial
Schagen, Nelspruit Mpumalanga, South Africa, 2011-2012	SC	1	1.44 (6 ml/tree)	Jug pouring, 15 Sept,	61 74 89	whole fruit	0.02 0.02 0.02	< 0.01 < 0.01 < 0.01	Thirion, 2013a, 11/787,

Location, year, (variety)	Form	No	g ai/tree	method, last application	DAT	Portion analyzed ^a	parent, mg/kg	CGA322704 mg/kg	Author, Study number, Trial
(Tommy Atkins), soil type not reported				2011 BBCH 67	105		0.02	< 0.01	ZA13 RMG 001 2012
					118		<u>0.02</u>	<u>< 0.01</u>	
					61	peel and flesh	0.03	0.01	
					74		0.03	0.01	
					89		0.03	0.01	
Schagen, Nelspruit Mpumalanga, South Africa, 2011-2012 (Tommy Atkins), soil type not reported	SC	1	2.88 (12 ml/tree)	Jug pouring, 15 Sept, 2011, BBCH 67	105		0.03	0.01	Thirion, 2013a, 11/787, ZA13 RMG 001 2012
					118		0.03	0.02	
					61	whole fruit	0.07	0.01	
					74		0.06	0.01	
					89		0.04	0.01	
Welgelegen, Nelspruit Komatipoort, Mpumalanga Mooketsi, South Africa, 2011-2012 (Kent), sandy loam	SC	1	1.44 (6 ml/tree)	Jug pouring, 16 Sept, 2011, BBCH 67	105		0.04	0.01	Thirion, 2013a, 11/787, ZA13 RMG 002 2012
					118		0.04	0.01	
					61	peel and flesh	0.08	0.01	
					74		0.06	0.01	
					89		0.04	0.01	
Welgelegen, Nelspruit Komatipoort, Mpumalanga Mooketsi, South Africa, 2011-2012 (Kent), sandy loam	SC	1	2.88 (12 ml/tree)	Jug pouring, 16 Sept, 2011, BBCH 67	105		0.05	0.02	Thirion, 2013a, 11/787, ZA13 RMG 002 2012
					118		0.05	0.02	
					61	whole fruit	<u>0.02</u>	<u>0.01</u>	
					118	peel and flesh	<u>0.02</u>	<u>0.01</u>	
Jonkmanspruit, Hoedspruit, Limpopo, 2011-2012, South Africa (Tommy Atkins), soil type not reported	SC	1	1.44 (6 ml/tree)	Jug pouring, 14 Sept, 2011, BBCH 65-67	118	whole fruit	0.03	0.01	Thirion 2013a, 11/787, ZA14 RMG 003 2012
					75		0.03	0.01	
					90		0.03	0.01	
					105		0.02	0.02	
					119		<u>0.02</u>	<u>0.02</u>	
Jonkmanspruit, Hoedspruit, Limpopo, 2011-2012, South Africa (Tommy Atkins), soil type not reported	SC	1	2.88 (12 ml/tree)	Jug pouring, 14 Sept, 2011, BBCH 65-67	61	peel and flesh	0.03	0.01	Thirion 2013a, 11/787, ZA14 RMG 003 2012
					75		0.03	0.01	
					90		0.03	0.01	
					105		0.03	0.02	
					119		0.03	0.02	
Mahuka Block 3A, Tzaneen, Limpopo, South Africa, 2011-2012 (Keitt), soil type not reported	SC	1	1.44 (6 ml/tree)	Jug pouring, 14 Sept, 2011, BBCH 65	61	whole fruit	0.15	0.03	Thirion 2013a, 11/787, ZA14 RMG 003 2012
					75		0.13	0.03	
					90		0.14	0.04	
					105		0.11	0.04	
					119		0.13	0.05	
Mahuka Block 3A, Tzaneen, Limpopo, South Africa, 2011-2012 (Keitt), soil type not reported	SC	1	2.88 (12 ml/tree)	Jug pouring, 14 Sept, 2011, BBCH 65	61	peel and flesh	0.16	0.03	Thirion 2013a, 11/787, ZA14 RMG 004 2004
					75		0.15	0.04	
					90		0.18	0.06	
					105		0.15	0.06	
					119		0.15	0.06	
Mahuka Block 3A, Tzaneen, Limpopo, South Africa, 2011-2012 (Keitt), soil type not reported	SC	1	1.44 (6 ml/tree)	Jug pouring, 14 Sept, 2011, BBCH 65	119	whole fruit	<u>0.01</u>	<u>0.01</u>	Thirion 2013a, 11/787, ZA14 RMG 004 2004
					119	peel and flesh	<u>0.01</u>	<u>0.02</u>	
Mahuka Block 3A, Tzaneen, Limpopo, South Africa, 2011-2012 (Keitt), soil type not reported	SC	1	2.88 (12 ml/tree)	Jug pouring, 14 Sept, 2011, BBCH 65	119	whole fruit	0.03	0.03	Thirion 2013a, 11/787, ZA14 RMG 004 2004
					119	peel and flesh	0.03	0.04	

n.a. = not applicable

^a In accordance with OECD Guideline Test No. 509 (crop field trial) the residues were determined in flesh and skin after removal of the stone and calculated to and expressed as whole fruit.

Legume vegetables

Fresh beans with pods

Eight residue trials were conducted on fresh beans in the 2003 and 2004 in Spain [Kang, 2005a, CGA293343/2217, CEMR-2185, Kang 2005b, CGA293343/2218, CEMR-2186, Rawle, 2005a, CGA293343/2203, CEMR-2201, Rawle, 2005b, CGA293343/2204, CEMR-2202, Kang, 2005c, CGA293343/2633, CEMR- 2322]. Beans were treated with a WG formulation at a nominal rate of 100 g ai/ha at growth stage BBCH 65–89 and again 6–7 days later (BBCH 71–89). Small plots (23–46 m²) were treated as indicated in table 8 using boom sprayers with a spray volume of approximately 1000 L/ha.

Samples of fresh beans with pods (1.0–2.4 kg) were taken at maturity at three time points after the final application within growth stages BBCH 71–78. In the 2004 trials also samples of remaining plant (5.7–14 kg) were taken at the same stages. Samples were collected at random from the whole plot. Plant samples were collected without roots. All samples were stored frozen at -18 °C. No data on storage duration were submitted. As the study was completed within a year, the storage duration is <365 days and sufficiently covered by the storage stability studies performed in a large variety of crops.

Samples were analysed for parent and CGA322704 with LC-MS/MS analytical method REM 179.03 with an LOQ of 0.02 mg/kg. Results were not corrected for control levels (< 0.01 mg/kg for each analyte) nor for average concurrent recoveries (69–95% and 76–117% for parent and CGA322704, respectively at fortification levels of 0.02 and 0.20 mg/kg) in the different studies. Residues of thiamethoxam and CGA322704 from the trials are summarized in Table 8.

Table 8 Residues of thiamethoxam and metabolite CGA322704 (clothianidin) after pre-harvest treatment of fresh (common) beans (pods with seeds)

Location, year, (variety) Soil type	Form	No	Interval (days)	g ai/ha	L/ha	method, last application	DAT	BBCH at sampling ^b	parent, mg/kg	CGA322704 mg/kg	Author, Study number, Trial
El Ejido, Almeria, Spain, 2003 (Oriente), Sandy gravelly	WG	2	6	102 103	1020 1025	Foliar spray, 27 May, 2003 BBCH 75	0 1 3 7 14	75 75 75 77 79	0.32 0.22 <u>0.08</u> < 0.02 < 0.02	0.06 0.08 <u>0.07</u> 0.05 < 0.02	Kang 2005a, CEMR-2185, trial 03-1012
El Matagorda, Almeria, Spain, 2003 (Dona), sandy	WG	2	6	100 102	1004 1023	Foliar spray, 03 December, 2003 BBCH 71	0 1 3 8 14	71 72 72 75 77	0.20 0.12 <u>0.09</u> < 0.02 < 0.02	0.06 0.07 <u>0.10</u> 0.07 < 0.02	Kang, 2005b, CEMR-2186, trial 03-1013
Sanlúcar de Barrameda, Spain, 2003 (Dulce), sandy	WG	2	6	97 98	967 984	Foliar spray, 29 April, 2003 BBCH 73-74	0 1 3 7 14	73-74 73-74 74 75 76	0.14 0.11 <u>0.08</u> < 0.02 < 0.02	0.05 0.07 <u>0.08</u> 0.03 < 0.02	Rawle, 2005a, CEMR-2201, trial 03-1011
Puntalon, Granada, Spain, 2003 (Maite), sandy	WG	2	6	101 101	1007 1006	Foliar spray, 10 March, 2003 BBCH 78	0 1 3 7 14	78 78 78 78 78	0.18 0.12 <u>0.08</u> 0.02 < 0.02	0.04 0.05 <u>0.04</u> 0.04 < 0.02	Rawle, 2005b, CEMR-2202, trial 03-1014
Lora der Rio, Seville,	WG	2	7	96 93	957 926	Foliar spray, 23	0 1	71-75 71-75	0.30 0.23	0.06 0.04	Kang, 2005c,

Location, year, (variety) Soil type	Form	No	Interval (days)	g ai/ha	L/ha	method, last application	DAT	BBCH at sampling ^b	parent, mg/kg	CGA322704 mg/kg	Author, Study number, Trial
Spain, 2004 (Oriente), loam						Nov, 2004 BBCH 71-75	3 7 16	≤77 ≤77 ≤77	<u>0.16</u> 0.03 ^a < 0.02	<u>0.06</u> 0.05 ^a < 0.02	CEMR-2322, trial ES-IR-04-0054
Penaflo, Seville, Spain, 2004 (Oriente), silt clay	WG	2	7	69 93	996 978	Foliar spray, 12 July, 2004 BBCH 73-74	0 1 3 8 14	73-74 73-74 74 75 77-79	0.20 0.20 <u>0.18</u> 0.03 ^a < 0.02	0.04 0.05 <u>0.04</u> 0.04 ^a < 0.02	Kang, 2005c, CEMR-2322, trial ES-IR-04-0055
El Maren de Barraquetes, Sueca, Valencia, Spain, 2004 (Perona), sandy	WG	2	8	113 99	1127 990	Foliar spray, 07 Sept, 2004 BBCH 85-89	0 1 3 7 13	85-89 85-89 89 89 89	0.16 0.14 0.08 ^b 0.02 ^b < 0.02	0.10 0.12 0.09 ^b 0.08 ^b < 0.02	Kang, 2005c, CEMR-2322, trial ES-IR-04-0056
Matagorda, Almeria, Spain, 2004 (Festival), sandy	WG	2	7	104 105	1042 1052	Foliar spray, 05 Oct, 2004 BBCH 74	0 1 3 8 14	74 74 75 77 77	0.21 0.09 ^a <u>0.03^a</u> < 0.02 ^a < 0.02	0.09 0.11 ^a <u>0.09^a</u> 0.03 ^a < 0.02	Kang, 2005c, CEMR-2322, trial ES-IR-04-0057

^a Mean of three values

^b BBCH growth stage is included to demonstrate that the samples were collected at the green stage of the beans. BBCH 79 is before ripening of the fruit (beans get hard). This process starts at BBCH 81 to 89.

Fresh herb

Mint

Five supervised residue field trials were conducted on mint in 2000–2001 in the USA [Syngenta, 2001, 406892, 07362]. The trials were not previously evaluated by the JMPR.

Plots (61–293 m²) of mint received three foliar applications at an interval of 13–15 days and a rate of approximately 70 g ai/ha (0.066 lbs ai/A, with a total of 0.198 lb ai/A) per treatment with a WG formulation, using 245–300 L water/ha (20–50 GPA). At two sites one additional plot received three applications at an intended rate of 370 g ai/ha (5×) each for a total of 1.1 kg ai/ha with an interval of 13–14 days. Six or 7 days after the last treatment at least 1.8 kg of mature mint tops (leaves and stem) were harvested. Mint oil was distilled from additional tops (sample size 0.9 kg) collected from the 5X tops (see section on processing).

All samples were stored frozen for a maximum of 482 days (tops) or 502 days (oil) at < 0 °C. This storage period is covered by the storage stability (452 days for tops and 465 days for oil).

Samples were analysed for parent and its metabolite CGA322704 using LC-MS/MS Method No AG-675 with an LOQ of 0.05 mg/kg for both parent and metabolite (see analytical section). Results were not corrected for control levels (< 0.05 mg/kg for each analyte) nor for average concurrent method recoveries (77–85% (parent) and 92–96% CGA322704) for each analyte at 0.05, 0.50 and 5.0 mg/kg). Residues of thiamethoxam and CGA322704 from the trial are summarised in Table 9.

Table 9 Residues of thiamethoxam and metabolite CGA322704 (clothianidin) in mint after pre-harvest treatment (tops)

Location, year, (variety)	Form	No (interval)	g ai/ha	water L/ha	method, timing	DAT	residues, mg/kg ^a		Reference, Study number, Trial
							Parent	CGA322704	
Sullivan, WI, USA, (Murray mitchem) Sand, silt clay 42, 17, 41 (clay muck)	WG	3 (13-15 d)	73.9 74.2 73.9	287 297 291	Foliar backpack, 26 July, 1999, crop stage: bud	7	<u>0.24</u>	<u>0.06</u>	Syngenta, 2001, 07362, 99-WI25
	WG	3 (13-15 d)	377 371 381	291 297 300		7	1.82	0.20	
Marshall, WI, USA (Scotch spearmint) Sand, silt clay 42, 17, 41 (clay muck)	WG	3 (13-15 d)	72.7 72.7 73.9	289 286 287	Foliar backpack, 29 June, 1999, crop stage: vegetative	6	<u>0.28</u>	<u>0.07</u>	Syngenta, 2001, 07362, 99-WI26
Prosser, WA, USA, 1999 (native spearmint), Sand, silt clay 58.7, 38.1, 3.2 (sandy loam)	WG	3 (13-15 d)	72.9 74.9 72.6	265 247 292	Foliar backpack, 17 June, 1999, crop stage: vegetative	7	<u>0.36</u>	<u>0.12</u>	Syngenta, 2001, 07362, 99-WA*18
	WG	3 (13-15 d)	372 383 361	271 253 290	Foliar backpack, 17 June, 1999, crop stage: vegetative	7	1.54	0.26	
Prosser, WA, USA, 1999 (Native spearmint), Sand, silt clay 58.7, 38.1, 3.2 (sandy loam)	WG	3 (13-15 d)	72.3 74.2 72.9	263 245 292	Foliar backpack, 18 June, 1999, crop stage: vegetative, early bud	6	<u>0.34</u>	<u>0.11</u>	Syngenta, 2001, 07362, 99-WA*51
Paterson, WA, USA, 1999, (Black Mitchem), Sand, silt clay 65, 29.5, 5.5 (sandy loam)	WG	3 (13-15 d)	72.3 74.3 73.1	271 283 277	Foliar backpack, 08 July, 1999, crop stage: bud	6	<u>0.86</u>	<u>0.11</u>	Syngenta, 2001, 07362, 99-WA*52

^a Results are mean of 2 replicates.

Dried herbs

Hops

Supervised residue field trials were conducted on hops in 2006 and 2011 in the USA [Syngenta, 2013a, A9549C_50012, TK0053990]. The trials conducted in 2006 were evaluated by the JMPR 2010 and not further addressed in this evaluation.

In the newly submitted study [Syngenta, 2013a, A9549C_50012, TK0053990] plots (size not reported) of hop were treated once with a SG formulation at an application rate of approximately 140 g ai/ha (0.125 lbs ai/A) by soil surface band application, using 187–467 L/ha (20–50 GPA). Sixty five days after treatment the hop was harvested and dried (confirmed in personal communication with manufacturer, Syngenta 08092014).

Samples of at least 0.45 kg (1 lb) of dry cones were collected. All samples were stored frozen for a maximum of 8.5 months at -10 to -20 °C. This storage period is covered by the storage stability studies (24–40 months) already evaluated by the JMPR in 2010.

Samples were analysed for parent and its metabolite CGA322704 using LC-MS/MS Syngenta Method No AG-675 with an LOQ of 0.01 mg/kg for both parent and metabolite. Results were not corrected for control levels (< 0.01 mg/kg for each analyte) nor for average concurrent method recoveries (79.9–92.4% for each analyte at 0.01 and 1.0 mg/kg). Residues of thiamethoxam and CGA322704 from the trial are summarised in Table 10.

Table 10 Residues of thiamethoxam and metabolite CGA322704 (clothianidin) in hops after pre-harvest treatment (cones, dried)

Location, year, (variety)	Form	No	kg ai/ha	water L/ha	method, timing	DAT	residues, mg/kg		Reference, trial
							Parent	CGA322704	
Ephrata, WA, USA (Cascade), sandy loam	SG	1	0.14	187-467	Soil surface treatment, 02 Aug, 2011, BBCH 61	65	0.029 ^a	0.027 ^a	Syngenta, 2013a, TK0053990-01

^a Results are mean of 2 replicates.

Legume vegetables animal feeds

Several residue trials were conducted on fresh beans in Spain. These have been summarized in the relevant residue section. In four of these residue trials [Kang, 2005c, Syngenta CGA293343/2633, CEMR-2322] residue data in remaining plant material, after removal of beans with pods and roots, was also reported. Details of the trials are provided under legume vegetables: fresh beans with pods. The residue data relevant to bean forage are summarized in Table 11.

Table 11 Residues of thiamethoxam and metabolite CGA322704 (clothianidin) after pre-harvest treatment of fresh (common) beans (remaining plant)

Location, year, (variety) Soil type	Form	No	Inter val (days)	g ai/ha	L/ha	method, last application	DAT	BBCH at sampling ^a	parent, mg/kg	CGA322704 mg/kg	Reference, Study number, Trial
Lora der Rio, Seville, Spain, 2004 (Oriente), loam	WG	2	7	96 93	957 926	Foliar spray, 23 Nov, 2004 BBCH 71-75	16	65-77	0.82	0.06	Kang, 2005c, CEMR-2322, trial ES-IR-04-0054
Penaflor, Seville, Spain, 2004 (Oriente), silt clay	WG	2	7	69 93	996 978	Foliar spray, 12 July, 2004 BBCH 73-74	14	77-79	1.4	0.07	Kang, 2005c, CEMR-2322, trial ES-IR-04-0055
El Mareny de Barraquetes, Sueca, Valencia, Spain, 2004 (Perona) ^b , sandy	WG	2	8	113 99	1127 990	Foliar spray, 07 Sept, 2004 BBCH 85-89	13	89	0.56	0.08	Kang, 2005c, CEMR-2322, trial ES-IR-04-0056

Location, year, (variety) Soil type	Form	No	Interval (days)	g ai/ha	L/ha	method, last application	DAT	BBCH at sampling ^a	parent, mg/kg	CGA322704 mg/kg	Reference, Study number, Trial
Matagorda, Almeria, Spain, 2004 (Festival), sandy	WG	2	7	104 105	1042 1052	Foliar spray, 05 Oct, 2004 BBCH 74	14	77	0.93	0.11	Kang, 2005c, CEMR-2322, trial ES-IR-04-0057

^a BBCH growth stage is included to demonstrate that the samples were collected at the green stage of the beans. BBCH 79 is before ripening of the fruit (beans get hard). This process starts at BBCH 81 to 89.

^b Perona beans is a Spanish variety of flat pod beans (vining variety).

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

The Meeting received processing studies on the magnitude of residues in processing studies with mangoes and mint.

The processing factors (residue processed commodity divided by residue RAC) were calculated separately for parent thiamethoxam and metabolite CGA322704 (clothianidin).

Mango processing

A field trial was conducted in the South Africa in 2001/2012 [Thirion, 2013b and c, Syngenta A9795B_11447, 2418/F788 and Syngenta A9795B_11448, 2418/G676] to measure the magnitude of thiamethoxam residues in mango processed commodities. The mango trees were treated once with the thiamethoxam formulated product, Actara 240 SC (Code No A9795B), at a rate of 18 ml product/tree or 4.32 g ai/tree. The applications were done when the trees were in the flowering state early in the season. The fruit was harvested when they were commercially ready for processing (BBCH 85–87). Mango pulp and dried mangoes were processed from the fruit, analysed and the processing factors were calculated. The residues in the whole fruit as well as in the peel were also determined. SABS In-house Method No. 054/2010 viz *The determination of Residues of Thiamethoxam and CGA 322 704 in Crops by LC-MS/MS*.

Dried fruit: Three samples were taken from each plot, one sample for determination of residues in the whole fruit (> 2.5–5 kg), one (< 2.5–5 kg) sample serving as for a contingency sample, and one sample (34–42 kg) for processing of pulp and drying. From the bulk 5 kg samples were taken for processing of pulp and drying, respectively. For processing the fruit was peeled and the flesh removed from the stones. The stone was discarded. Flesh and peel were kept for further analyses. The flesh of the fruit was preserved with sodium metabisulphite and dried in an automatic electrical drying oven by using the following temperature and humidity program: 60 °C at 40% relative humidity for 360 min; 57 °C at 27% relative humidity for 60 minutes; 55 °C at 18% relative humidity for 360 minutes. The moisture content after processing ranged between 9.1 and 9.9% in both studies.

Table 12 Residues of thiamethoxam and CGA322704 after processing of mangoes

Location, year, (variety)	Treatment	DAT	processed products	parent		CGA322704 mg/kg		Reference, Study number, Trial
				residues (mg/kg)	PF	residues (mg/kg)	PF	
Nelspruit, South Africa, 2011/2012 (Tommy Atkins)	Soil treatment, 4.32 g ai/tree (18 ml/tree), 15 Sept, 2011	131	Peel + flesh	0.09	-	0.03	-	Thirion, 2014b, 2418/G676, ZA RMG 005 2012 (5)
		144	Flesh (pulp)	0.05	0.56	0.02	0.67	
		144	Peel	0.10	n.a.	0.03	n.a.	
		145	Dried fruit	0.36	4.00	0.17	5.67	

Location, year, (variety)	Treatment	DAT	processed products	parent		CGA322704 mg/kg		Reference, Study number, Trial
				residues (mg/kg)	PF	residues (mg/kg)	PF	
Jonkmanspruit, Hoedspruit, Limpopo, South Africa, 2011/2012 (Tommy Atkins)	Soil treatment, 4.32 g ai/tree (18 ml/tree), 14 Sept, 2011	119	Peel + flesh	0.11	-	0.05	-	Thirion, 2014b, 2418/G676, ZA RMG 006 2012 (6)
		130	Flesh (pulp)	0.11	1.00	0.06	1.20	
		130	Peel	0.12	n.a.	0.08	n.a.	
		132	Dried fruit	0.74	6.73	0.42	8.40	
Mpumalanga, South Africa, 2012/2013 (Keitt)	Soil treatment, 4.32 g ai/tree (18 ml/tree), 14 Sept, 2011	122	Peel + flesh	0.03	-	0.06	-	Thirion, 2014b, 2418/G676, ZA RMG 009 2013 (9)
		134	Flesh (pulp)	0.02	0.67	0.05	0.83	
		134	Peel	0.03	n.a.	0.05	n.a.	
		136	Dried fruit	0.22	7.33	0.42	7.00	
Mpumalanga, South Africa, 2012/2013 (Kent)	Soil treatment, 4.32 g ai/tree (18 ml/tree), 14 Sept, 2011	122	Peel + flesh	0.04	-	0.03	-	Thirion, 2014b, 2418/G676, ZA RMG 0010 2013 (10)
		134	Flesh (pulp)	0.02	0.50	0.01	0.33	
		134	Peel	0.03	n.a.	0.02	n.a.	
		136	Dried fruit	0.20	5.00	0.12	4.00	

The effect of processing on the nature of residues was not specifically investigated. The processing factors for both the parent and the metabolite CGA322704 indicate that no significant changes are expected to occur with regard to the nature of the residues during drying process of mangoes.

Mint processing

Two supervised residue trials with spearmint were conducted in the USA in 1999 [Syngenta, 2001, 406892, 07362]. Three foliar applications were made with an exaggerated rate (5×) of 370 g ai/ha, with a spray interval of 13–15 days. The applications were made with a backpack sprayer at a spray volume of 296 L/ha. The last application was made at (early) bud stage (26 July, 1999 and June 17, 1999 for trial WI25 and WA*18, respectively). Mature tops were harvested at normal commercial harvest, 7 days after the last application. Tops were harvested manually. Dead and/or senesced leaves were removed. The tops were processed into oil using standardised procedures designed to simulate relevant industrial processes. All weight fractions have been corrected for subsampling and represent weight fractions for trial 99-WI25 and 99-WA*18, respectively.

Oil processing

Large samples of fresh mint plants were collected. About 1.1 kg from each sample was distilled immediately after harvest, using a mint still and boiler for distillation (at steam pressure of 75 psi, pot pressure of 1–1.5 psi and distillate temperature of -1 to 7 °C for 1.5–2.5 hours. For analysis, the (2 g) oil was diluted with GPC mobile phase (50% ethyl acetate/50% cyclohexane). Clean up was done by injection in GPC, followed by evaporation of the eluate to ~0.5 mL at 35–40 °C on a Turbovap. The sample was dissolved in a total volume of 4.0 mL using acetonitrile.

Mint oil samples were stored at < 0 °C for a maximum of 502 days prior to analyses. Mint top samples were stored at < 0 °C for a maximum of 482 days. This storage period is sufficiently covered by the storage stability (452 days for tops and 465 days for oil). Samples were analysed for parent and CGA322704 using LC-MS/MS Method No AG-675 with an LOQ of 0.05 mg/kg for both parent and metabolite. Samples were analysed for parent and its metabolite CGA322704. Residues of thiamethoxam and CGA322704 and processing factors from the trial are summarised in Tables 13 and 15.

Results were not corrected for control levels (< 0.05 mg/kg for each analyte) nor for average concurrent method recoveries (77–85% (parent) and 92–96% (CGA322704) at 0.05, 0.50 and 5.0 mg/kg in tops and 92–116% (parent) and 92–111% (CGA322704) at 0.05, 0.50 and 5.0 mg/kg in oil).

Note: The analytical method is considered valid in the range 0.05–5.0 mg/kg for the determination of thiamethoxam and CGA322704 in mint.

Table 13 Residues of thiamethoxam and CGA322704 (clothianidin) and processing factors in mint tops and processed mint (oil)

Location, year, (variety)	Treatment	DAT	processed products	parent		CGA322704 mg/kg		reference
				residues (mg/kg)	PF	residues (mg/kg)	PF	
Sullivan, WI, USA, 1999 (Murray Mitchem) peppermint, soil: clay muck	Foliar spray, 3 x 0.370 kg ai/ha, interval 13-15 days	7	Mint tops	2.36	-	0.23	-	Syngenta, 2001, 406892, 99-WI25
			Mint oil	< 0.05	< 0.02	< 0.05	< 0.22	
Prosser, WA, USA, 1999 (native spearmint), soil: sandy loam	Foliar spray, 3 x 0.370 kg ai/ha, interval 13-15 days	7	Mint tops	1.83	-	0.273	-	Syngenta, 2001, 406892, 99-WA*18
			Mint oil	< 0.05	< 0.03	< 0.05	< 0.19	

Processing studies summary

An overview of calculated processing factors for mango and mint is given in Tables 14 and 15 for thiamethoxam and CGA322704 (clothianidin), respectively.

Table 14 Overview of processing factors for thiamethoxam in mango and mint

Compound	Processed fraction	Processing Factor (n=2-4)
Mango	Pulp	0.6 (4)
Mango	Dried	5.9 (4)
Mint	Aspirated oil	< 0.02 (2)

Table 15 Overview of processing factors for metabolite CGA322704 (clothianidin) in mango and mint

Compound	Processed fraction	Processing Factor (n=2-4)
Mango	Pulp	0.8 (4)
Mango	Dried	6.3 (4)
Mint	Aspirated oil	< 0.2 (2)

APPRAISAL

SEE ALSO CLOTHIANIDIN (238)

Thiamethoxam is a neonicotinoid compound with broad-spectrum insecticidal properties. The compound was evaluated by the JMPR in 2010 (T, R), 2011 (R) and 2012 (R). The 2010 Meeting established an ADI for Thiamethoxam of 0–0.08 mg/kg bw and an ARfD of 1 mg/kg bw. It was listed by the Forty-fifth Session of CCPR (2013) for the evaluation of 2014 JMPR for additional MRLs.

The residue definition for enforcement for thiamethoxam in plant and animal commodities is thiamethoxam. The residue for dietary risk assessment for plant and animal commodities (except poultry) is thiamethoxam and clothianidin (considered separately). The residue definition for risk

assessment dietary intake for poultry is the sum of thiamethoxam, CGA 265307 and MU3, expressed as thiamethoxam and clothianidin (clothianidin to be considered separately from thiamethoxam). In the current appraisal CGA322704 is referred to as clothianidin.

At the 2014 JMPR Meeting residue data were submitted to support maximum residue level recommendations for use of thiamethoxam on several crops. Residue trials were conducted to support use avocado, mango, beans, mint and hops. In addition, trials were submitted by Republic of Korea to support the use on persimmon. Summaries of the trials have been provided to support the recommendations for thiamethoxam and clothianidin on the respective crops.

Methods of Analysis

The Meeting received additional validation data for the analytical method REM 179.06 (evaluated by the 2010 Meeting) of thiamethoxam and clothianidin.

Most of the methods used in the supervised residue trials had previously been evaluated (2010 JMPR) and determined parent compound thiamethoxam and the metabolite clothianidin. Samples were extracted with methanol:water. The final residue could then be determined by HPLC-MS/MS. The Meeting considers validation of the method sufficient for the respective crops with an LOQ of 0.01 mg/kg for parent and its metabolite, respectively. The analytical methods not previously evaluated, but used in the trials for the current evaluation (LC-MS/MS Method 954/2010 and Method HPLC/DAD) were sufficiently validated.

Stability of pesticide residues in stored analytical samples

At the 2010 JMPR, thiamethoxam and clothianidin were shown to be stable for 1–2 years when stored frozen at < -18 °C or lower for a large range of commodities. The stability in the crops under evaluation in 2014 was sufficiently demonstrated in the respective residue trials.

Results of supervised residue trials on crops

The Meeting received supervised trials data for persimmon, avocado, mango, beans, mint (herbs) and hops.

Pome fruit

Residue data in support of the use of thiamethoxam and clothianidin on pome fruit (apples and pears) were previously evaluated by the 2010 JMPR. In 2010 insufficient data were submitted for the use of clothianidin on persimmons to lead to a maximum residue level recommendation (not yet categorized under pome fruit at that time). No data were previously submitted to support the use of thiamethoxam on persimmons.

The critical GAP for thiamethoxam on persimmon in Republic of Korea is for 3 foliar applications at 0.005 kg ai/hL (interval of 10 days) and a PHI of 7 days, until “leaves are dripping”. The Meeting received two independent trials that were performed according to the Korean critical GAP using an application rate of 0.005 kg ai/hL.

As the GAPs for pome fruit and persimmon are different, a specific maximum residue level for persimmon would need to be estimated. However, the Meeting agreed that the dataset was insufficient for the estimation of a maximum residue level for persimmon.

The Meeting confirmed the maximum residue level, STMR and HR for thiamethoxam and clothianidin in pome fruit as recommended by the 2010 JMPR Meeting.

Assorted tropical and sub-tropical fruits – inedible peel

Supervised trials were available for avocado and mango. The use of thiamethoxam and/or clothianidin on avocados had not been previously evaluated by the JMPR. The use of thiamethoxam on mango was evaluated by the 2010 JMPR, but there were insufficient data to support a maximum residue

level recommendation. The Meeting had not previously received residue data supporting the use of clothianidin on mangoes.

Avocado

The critical GAP for use on avocado's in the USA is for 3 foliar directed applications at 0.070 kg ai/ha (minimum interval of 7 days) and no PHI.

Five field trials involving the use of thiamethoxam (3 foliar applications at approximately 70 g ai/ha, with a 6–7 day interval and PHI of 3 days) on avocados were performed in the USA (3) and in Mexico (2) in 2006 (4) and 2007 (1). Thiamethoxam residues on whole fruit (RAC) were not reported. Using a conversion factor to correct from flesh and peel to whole fruit ($0.8 \times$ residue level), calculated residue levels in whole fruit were: < 0.01, 0.03, 0.06, 0.10, and 0.24 mg/kg (n=5). Converted clothianidin residues in RAC were: < 0.01, < 0.01, < 0.01, 0.016, and 0.016 mg/kg (n=5).

The Meeting agreed that the dataset for avocados matching the USA critical GAP could be used to support a maximum residue level recommendation for avocados, and estimated a maximum residue level of 0.5 mg/kg for thiamethoxam and a maximum residue level of 0.03 mg/kg for clothianidin on avocado.

The residue data for estimating the STMR and HR (flesh+peel) were: < 0.01, 0.04, 0.08, 0.12, and 0.30 mg/kg (n=5), resulting in an STMR and HR of 0.08 mg/kg and 0.30 mg/kg, respectively for thiamethoxam.

Clothianidin residues (flesh+peel) were: < 0.01, < 0.01, < 0.01, 0.02, and 0.02 mg/kg (n=5), resulting in STMR and HR estimates of 0.01 and 0.02 mg/kg, respectively.

Mango

Field trials involving the use of thiamethoxam on mango were performed in South Africa. Eight trials in 2003–2005 were evaluated by the JMPR in 2010. Trials performed 2011–2012 were submitted to the 2014 Meeting.

The critical GAP for use of thiamethoxam on mangoes in the South Africa is a single soil application at 1.44 g ai/tree poured from a jug with a PHI of 130 days. Seven residue trials (three in 2003/2005 and four in 2011/2012) matched the South African GAP, using a single soil application at a rate of 1.44 g ai/tree and a PHI of 130 days. Thiamethoxam residues (whole fruit) were: 0.01, 0.02, 0.02, 0.02, 0.036[#], 0.08[#], and 0.088[#] mg/kg. Clothianidin residues were: < 0.01, 0.01, 0.01, < 0.02[#], < 0.02[#], < 0.02[#] and 0.02 mg/kg. The values identified with [#] are residue data of flesh and peel corrected for stone using a default conversion factor of 0.8, because the weights of the different fractions of the fruit were not reported in the 2010 evaluation.

The Meeting agreed that the dataset for mangoes matching the South African critical GAP could be used to support a maximum residue level recommendation for mangoes, and estimated a maximum residue level of 0.2 mg/kg for thiamethoxam on mango. For clothianidin the Meeting estimated a maximum residue level of 0.04 mg/kg.

For the STMR and HR estimates the residue data excluding the stone are used. Thiamethoxam residues (flesh+peel) were: 0.01, 0.03, 0.03, 0.03, 0.04, 0.10, and 0.11 mg/kg, resulting in a STMR and HR of 0.03 and 0.11 mg/kg, respectively for thiamethoxam. Clothianidin residues (flesh and peel) were: 0.01, < 0.02, 0.02, 0.02, 0.02, 0.02, and 0.02 mg/kg. The Meeting estimated STMR and HR values of 0.02 and 0.02, respectively for clothianidin.

Legume vegetables

The Meeting received data on the use of thiamethoxam on fresh beans. A Portuguese label for use of thiamethoxam on fresh beans was submitted to the Meeting. The label included indoor use (foliar application and drip irrigation) and outdoor use (foliar application).

Indoor use: No data were submitted to support the indoor use (neither the foliar, nor the drip irrigation uses).

Outdoor use: The use of thiamethoxam on beans and peas had been evaluated by the 2010 Meeting. In 2010 the data reviewed covered seed treatment use; therefore the datasets could not be combined. According to the 2010 evaluation there was no GAP for clothianidin on legume vegetables. The critical GAP for outdoor use of thiamethoxam is a double foliar application of 100 g ai/ha, with an interval of 7 days and a PHI of 3 days.

Eight field trials involving fresh beans (with pods) were conducted in 2003 and 2004 in Spain. The critical GAP for outdoor use was supported with seven trials using two foliar applications of 93–113 g ai/ha (total rate of 200 g ai/ha within $\pm 25\%$ range), 6–8 day interval and 3 day PHI. Thiamethoxam residues were: 0.03, 0.08, 0.08, 0.08, 0.09, 0.16, and 0.18 mg/kg.

The Meeting agreed that the dataset for fresh beans matching the Portuguese critical GAP for outdoor use could be used to support a maximum residue level recommendation for fresh beans, and estimated a maximum residue level of 0.3 mg/kg for thiamethoxam in fresh beans.

The Meeting estimated STMR and HR values of 0.08 and 0.18 mg/kg, respectively for thiamethoxam in fresh beans.

The same trials were used for clothianidin. The residues were: 0.04, 0.04, 0.06, 0.07, 0.08, 0.09, and 0.10 mg/kg.

The Meeting estimated a maximum residue level of 0.2 mg/kg for clothianidin and STMR and HR values of 0.07 mg/kg and 0.10 mg/kg, respectively for clothianidin in fresh beans.

Fresh herbs

The Meeting received data on the use of thiamethoxam on mints. No uses of thiamethoxam or clothianidin on fresh herbs (mints) have previously been evaluated by the JMPR.

Mints

Field trials involving the use of thiamethoxam on mint were performed in the USA in 1999.

The critical GAP for mint in the USA is 3 foliar applications of 0.070 kg ai/ha (minimum interval 14 days, maximum total rate of 0.21 kg ai/ha per season) and PHI of 7 days. Five trials matched this GAP (3×0.072 – 0.074 kg/ai/ha, interval 13–15 days, PHI 6–7 days), although slightly higher applications rates were used ($< 25\%$ difference). Thiamethoxam residues were: 0.24, 0.28, 0.34, 0.36, and 0.86 mg/kg.

The Meeting agreed that the dataset on mints matching the USA GAP could be used to support a maximum residue level recommendation for mints and estimated a maximum residue level of 1.5 mg/kg for thiamethoxam on mint and STMR and HR values of 0.34 and 0.86 mg/kg, respectively for thiamethoxam.

The same trials were used to derive the residue levels for clothianidin. Residues were: 0.06, 0.07, 0.11, 0.11, and 0.12 mg/kg.

The Meeting agreed that the dataset on mints could be used to support a maximum residue level for mints and estimated a maximum residue level of 0.3 mg/kg for clothianidin and STMR and HR values of 0.11 mg/kg and 0.12 mg/kg, respectively for clothianidin.

Dried herbs

The Meeting received data on the use of thiamethoxam on hops. The use of thiamethoxam on hops was evaluated by the 2010 Meeting. No maximum residue level was recommend due to the limited dataset ($n=3$). The JMPR has not evaluated any uses for clothianidin on dried herbs.

Hops

Field trials involving hops were performed in the USA in 2002 (three evaluated by JMPR 2010) and 2011 (one submitted in 2014).

The critical GAP for hops in the USA is a single soil surface treatment (band application with incorporation) at a rate of 0.14 kg ai/ha and a PHI of 65 days. The trials performed in the USA matched this GAP (1×0.13–0.14 and PHI 62–66 days).

Thiamethoxam residues in hops (dried cones) were: < 0.025, 0.027, 0.029, and 0.055 mg/kg. The Meeting agreed that the dataset for hops matching the USA GAP could be used to support a maximum residue level recommendation for hop, and estimated a maximum residue level of 0.09 mg/kg for thiamethoxam on dried hops. The Meeting estimated STMR and HR values of 0.028 and 0.055 mg/kg, respectively for thiamethoxam.

Clothianidin residues were: < 0.025, 0.025, 0.027, and 0.028 mg/kg. The Meeting estimated a maximum residue level of 0.07 mg/kg for clothianidin on hops. The Meeting estimated STMR and HR values of 0.026 and 0.028 mg/kg, respectively for clothianidin, respectively.

Legume animal feeds

The Meeting received data on the outdoor use of thiamethoxam on fresh beans. The use of thiamethoxam on beans and peas has been evaluated by the 2010 Meeting, but only covered seed treatment uses and provided residue data for thiamethoxam and clothianidin in pea vines and pea hay/fodder (dry), but not for bean forage. No residue data for clothianidin in legume animal feeds was available.

Bean forage

The Portuguese outdoor GAP for use of thiamethoxam on beans is a foliar application 2×100 g ai/ha, with a PHI of 3 days and a minimum interval of 7 days.

Outdoor use

Eight field trials involving fresh beans (with pods) were conducted in 2003 and 2004 in Spain. The application rates used were two foliar applications of 69–113 g ai/ha with an interval of 6–8 days and a PHI of 3 days. Matching the Portuguese GAP. Residues in bean forage were determined in four field trials. Thiamethoxam residues in bean forage (rest of plants harvested at BBCH < 80) were: 0.56, 0.82, 0.93, and 1.4 mg/kg. Clothianidin residues in bean forage were: 0.06, 0.07, 0.08, and 0.11 mg/kg.

The Meeting agreed that the dataset for fresh bean forage matching the outdoor Portuguese GAP could be used to estimate median residues of 0.87 and 0.075 mg/kg for thiamethoxam and clothianidin, respectively. The respective highest residues are 1.4 and 0.11 mg/kg.

Fate of residues during processing

Processing studies were undertaken for mango and mint. Processing factors based on the residue for parent and metabolite clothianidin are listed in the table below. Using the STMR_{RAC} obtained from the thiamethoxam use, the Meeting estimated STMR-Ps for processed commodities to be used in dietary intake calculations.

Commodity	PFs	PF (median or best estimate)	STMR-P = STMR _{pulp+peel} × PF (mg/kg)	HR-P = HR _{pulp+peel} × PF (mg/kg)	PFs	PF (median or best estimate)	STMR-P = STMR _{RAC} × PF (mg/kg)	HR-P = HR _{RAC} × PF (mg/kg)
	Parent thiamethoxam (STMR _{pulp+peel} = 0.03 mg/kg HR _{pulp+peel} = 0.11 mg/kg)				clothianidin (STMR _{pulp+peel} = 0.02 mg/kg, HR _{pulp+peel} = 0.02 mg/kg)			
Mango, dried flesh	4.0, 6.7, 7.3, 5.0	5.9	0.18	0.65	5.7, 8.4, 7.00, 4.00	6.3	0.13	0.13
Mint, oil	< 0.02, < 0.03	< 0.02	n.a.	n.a.	< 0.22, < 0.19	< 0.20	n.a.	n.a.

Residues in animal commodities

The Meeting estimated the dietary burden of thiamethoxam residues (thiamethoxam only, for CGA322704 see clothianidin appraisal 2014) on the basis of the livestock diets listed in the FAO manual appendix IX (OECD feedstuff table) using the OECD_Feed_Calculator_V1_4. Calculation from highest residue, STMR (some bulk commodities) and STMR-P values provides the levels in feed suitable for estimating MRLs, while calculation from STMR and STMR-P values from feed is suitable for estimating STMR values for animal commodities.

The Meeting recalculated the livestock dietary burden based on the uses presented by the 2010 JMPR and including the residue values for fresh bean forage from the 2014 JMPR Meeting. The maximum dietary burden for cattle for MRL (tissues) setting changed slightly from 5.2 to 6.1 ppm, and the mean dietary burden for cattle changed only marginally from 2.1 to 2.4 ppm. For residue level estimations in milk the dietary burden raised from 5.2 to 6.1 ppm (maximum) and from 1.6 to 2.4 ppm (mean). The new maximum dietary burden of thiamethoxam for poultry of 1.64 ppm is only marginally higher than the maximum dietary burden of 1.59 ppm as calculated by the Meeting in 2010, rounded both 1.6 ppm. The new mean dietary burden for poultry of 0.59 ppm is not changed since the evaluation in 2010.

The Meeting agreed that no new mean and maximum residue level estimations are needed and confirmed its previous recommendations.

Residue data for clothianidin used for the dietary burden calculation were derived from the 2010 JMPR appraisal for clothianidin, where the data from thiamethoxam and clothianidin use were combined and included the residue data on bean forage from the 2014 JMPR Meeting. For the dietary burden calculations and recommendations for animal commodities see appraisal clothianidin 2014.

RECOMMENDATIONS

THIAMETHOXAM

On the basis of the data from supervised residue trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

The 2011 Meeting recommended the following residue definition for thiamethoxam:

Definition of the residue for animal and plant commodities (for compliance with the MRL): *thiamethoxam*.

Definition of the residue for plants and animals (except poultry), (for estimation of dietary intake): *thiamethoxam and clothianidin* (considered separately).

Definition of the residue for poultry (for estimation of dietary intake): *sum of thiamethoxam, CGA 265307 and MU3, expressed as thiamethoxam and clothianidin* (clothianidin to be considered separately from thiamethoxam).

The residue is not fat-soluble.

Note that thiamethoxam metabolite CGA322704 (N-(2-chlorothiazol-5-ylmethyl)-N'-methyl-N"-nitroguanidine) will appear as clothianidin in the analytical method and residues of CGA322704 occurring in food are included in the clothianidin MRLs.

Metabolite CGA 265307: N-(2-chlorothiazol-5-ylmethyl)-N'-nitroguanidine.

Metabolite MU3: amino-[(2-chlorothiazol-5-ylmethyl)-amino]-methylene)-hydrazide.

	Commodity	THIAMETHOXAM Maximum residue level recommendations (mg/kg)		STMR or STMR-P (mg/kg)		HR or HR-P
CCN	Name	New	previous	new	previous	mg/kg
FI 0326	Avocado	0.5		0.08		0.30
VP 0061	Beans with pods	0.3		0.08		0.18
DH 1100	Hops	0.09		0.028		0.55
FI 0345	Mango	0.2		0.03		0.11
HH 0738a	Mints	1.5		0.34		0.86
-	Mango pulp			0.02		0.07
-	Mango dried			0.18		0.65

^a It is noted that there are several types of mints (native mints HH0764, Vietnamese mint HH0765). The reported CCN code is listed for several types of mints, including spearmint and peppermint.

	Commodity	THIAMETHOXAM Maximum residue level recommendations	Median residue	Highest residue
CCN	Name	mg/kg	mg/kg	mg/kg
AL	Bean forage	n.a.	0.87	1.4

The recommendations for clothianidin resulting from thiamethoxam use are listed in the clothianidin appraisal 2014.

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDI) of thiamethoxam, based on the STMRs estimated for 112 commodities, for the 17 cluster diets were in the range of 1-3% of the maximum ADI (0.08 mg/kg bw) (Annex 3 to the 2014 Report). The Meeting concluded that the long-term intake of residues of thiamethoxam resulting from its uses that have been considered by the 2010, 2011 2012 and the present Meeting is unlikely to present a public health concern.

For the International Estimated Daily Intakes (IEDI) for clothianidin resulting from thiamethoxam and clothianidin use see appraisal clothianidin 2014.

Short-term intake

The International Estimated Short Term Intake (IESTI) for thiamethoxam was calculated for food commodities and their processed fractions for which maximum residue levels were estimated and for which consumption data were available. The results are shown in Annex 4 to the 2014 Report.

The IESTI represented 0–1% of the ARfD (1.0 mg/kg bw). The Meeting concluded that the short-term intake of residues of thiamethoxam, when used in ways that have been considered by the present Meeting, is unlikely to present a public health concern.

For the International Estimated Short Term Intake (IESTI) for clothianidin resulting from thiamethoxam and clothianidin use see appraisal clothianidin (2014).

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TRIADIMENOL (168)

The first draft was prepared by Mr Christian Sieke, Federal Institute for Risk Assessment, Berlin, Germany

EXPLANATION

Triadimenol is a systemic fungicide formed as the primary metabolite of triadimefon but also used as an active substance on its own. Its main mode of action is an inhibition of the ergosterol biosyntheses in fungi. Triadimenol was evaluated by JMPR several times since 1978 and the last time in 2004 for toxicology, when an ADI of 0–0.03 mg/kg bw and an ARfD of 0.08 mg/kg bw was established and in 2007 for residues.

Definition of the residue in plant and animal commodities (for the estimation of dietary intake and for compliance with MRLs): *sum of triadimefon and triadimenol*

In 2007 the Meeting evaluated use patterns of triadimefon and triadimenol in grapes; however, a short-term dietary intake concern was identified without opportunity for an alternative GAP.

The current Meeting received new information on use patterns for triadimenol in grapes supported by new supervised residue trials on grapes from Europe.

ANALYTICAL METHODS

Method 01072/M001 (Bomke, S; 2010)

The basic principle of method 01072/M001 involves extraction of homogenized samples with methanol/water (4/1; v/v) by high-speed blending. After filtration, an aliquot of the extract was evaporated to dryness, re-dissolved under addition of deuterated internal standard, filtered and measured by LC-MS/MS (silica based C18-column using acetonitrile/water eluent).

The characteristic mass transition for triadimenol used for quantification are m/z 296.1 → 70.2 and m/z 298.0 → 70.1 for confirmation.

In the following table the recoveries in selected fortified samples are reported.

Table 1 Recovery data for method 01072/M001 in plant matrices (LC-MS/MS, m/z: 296.1 → 70.2 & m/z: 298.0 → 70.2)

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Analyte, reference, MRM transition
Lettuce	0.01	5	90–110	102	8	Triadimenol
	0.1	5	80–112	93	13	Bomke, S; 2010
Wheat grain	0.01	5	90–110	98	9	m/z: 296.1 → 70.2 (quantification)
	0.1	5	87–122	101	14	
Beans without pods	0.01	5	100–120	110	9	
	0.1	5	89–108	103	8	
Orange fruits	0.01	5	80–100	88	10	
	0.1	5	86–110	100	10	
Avocado fruits	0.01	5	100–110	108	4	
	0.1	5	93–112	104	7	
Lettuce	0.01	5	90–110	98	9	Triadimenol
	0.1	5	80–106	90	12	Bomke, S; 2010
Wheat grain	0.01	5	90–120	102	11	m/z: 298.0 → 70.1 (confirmation)
	0.1	5	88–104	97	8	
Beans without pods	0.01	5	90–110	102	8	
	0.1	5	90–99	93	4	
Orange fruits	0.01	5	80–110	94	14	

	0.1	5	81–94	88	7	
Avocado fruits	0.01	5	90–100	96	6	
	0.1	5	92–107	98	6	

Method 00480/M001 (Bomke, S; 2010a)

The basic principle of method 01072/M001 involves extraction of homogenized samples by refluxing with methanol/water (7/3, v/v). After filtration, the extract was concentrated to the aqueous remainder. The remaining water was enzymatically hydrolysed to release conjugated residues (glucosides). An aliquot was purified on a SPE cartridge. The analytes were eluted with cyclohexane/ethyl acetate (85/15, v/v) and the eluate was evaporated to dryness. The remainder was resolved in internal standard solution and subjected to HPLC-MS/MS. The analytes were chromatographed by reversed-phase HPLC on a silica-based C18-column using a gradient acetonitrile/water eluent containing acetic acid.

The characteristic mass transition for triadimenol used for quantification are m/z 296.1 $\rightarrow$ 70.2 and m/z 298.0 $\rightarrow$ 70.1 for confirmation. For triadimenol-hydroxy, which is the remaining analyte after cleavage of conjugates, mass transition used for quantification are m/z 312.0 $\rightarrow$ 70.1 and m/z 314.0 $\rightarrow$ 70.1 for confirmation.

In the following table the recoveries in selected fortified samples are reported.

Table 2 Recovery data for method 01072/M001 in plant matrices (LC-MS/MS, m/z : 296.1 $\rightarrow$ 70.2 & m/z : 298.0 $\rightarrow$ 70.2)

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Analyte, reference, MRM transition
Apple fruit	0.01	5	88–99	95	4	Triadimenol
	0.1	5	86–98	93	5	Bomke, S; 2010a
Orange fruit	0.01	5	89–109	98	7	m/z : 296.1 $\rightarrow$ 70.2 (quantification)
	0.1	5	91–103	98	5	
Wheat grain	0.01	5	89–99	94	4	
	0.1	5	91–97	93	3	
Beans without pods	0.01	5	83–99	89	7	
	0.1	5	87–99	90	6	
Sunflower seeds	0.01	5	81–91	86	5	
	0.1	5	82–92	88	5	
Apple fruit	0.01	5	94–103	98	4	Triadimenol
	0.1	5	84–94	89	5	Bomke, S; 2010a
Orange fruit	0.01	5	84–101	93	8	m/z : 298.0 $\rightarrow$ 70.1 (confirmation)
	0.1	5	88–98	93	5	
Wheat grain	0.01	5	86–92	88	3	
	0.1	5	87–97	91	4	
Beans without pods	0.01	5	82–90	85	4	
	0.1	5	82–95	87	6	
Sunflower seeds	0.01	5	75–88	83	6	
	0.1	5	79–86	81	4	
Apple fruit	0.01	5	87–98	92	5	Triadimenol-hydroxy
	0.1	5	81–105	91	10	Bomke, S; 2010a
Orange fruit	0.01	5	86–98	92	6	m/z : 312.0 $\rightarrow$ 70.1 (quantification)
	0.1	5	87–94	91	3	
Wheat grain	0.01	5	85–95	92	4	
	0.1	5	91–100	95	4	
Beans without pods	0.01	5	80–88	85	4	
	0.1	5	78–87	83	4	
Sunflower seeds	0.01	5	79–90	86	6	
	0.1	5	87–90	88	2	

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Analyte, reference, MRM transition
Apple fruit	0.01	5	88–106	97	8	Triadimenol-hydroxy
	0.1	5	87–97	90	5	Bomke, S; 2010a
Orange fruit	0.01	5	81–91	88	5	m/z: 314.0 → 70.1 (confirmation)
	0.1	5	86–96	89	5	
Wheat grain	0.01	5	87–103	94	6	
	0.1	5	86–99	92	6	
Beans without pods	0.01	5	73–87	81	7	
	0.1	5	80–88	84	4	
Sunflower seeds	0.01	5	84–93	88	4	
	0.1	5	83–93	89	4	

USE PATTERN

Crop (code)	Country	Form	Application					PHI, days
			Method	Rate kg ai/ha	Spray conc, kg ai/hL	Number	Interval	
Grapes	France	EC 7.5%	foliar spray	0.019	–	3	14 d	21
Table grapes	Spain	SC 31%	foliar spray	0.063	–	4	not stated	15
Wine grapes	Spain	SC 31%	foliar spray	0.063	–	4	not stated	21

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received additional information on supervised residue trials of triadimenol to grapes from Europe. In addition, a large dataset on grapes was reported in the 2007 JMPR Evaluation on triadimenol, which is repeated for consistency.

Application rates were reported as triadimenol. Unquantifiable residues are shown as below reported LOQ (< 0.01 mg/kg). Residue data are recorded unadjusted for percentage recoveries or for residues values in control samples, unless otherwise stated. Where multiple samples were taken from a single plot, individual values are reported. Where multiple analyses were conducted on a single sample, the average value is reported. Where results from separate plots with distinguishing characteristics such as different formulations, varieties or treatment schedules were reported, results are listed for each plot.

Residues from the trials conducted according to critical GAP have been used for the estimation of maximum residue levels. These results are underlined.

Table 3 Residues of triadimenol in grapes following foliar treatment (submitted in 2007)

Location (variety)	Year	Form	Application			Analysis			Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	DAT	Residues	
cGAP France		EC	0.019	–	3		PHI 21		
France Sorgues 1993 (Clairette)		300 EC	0.019	0.023	3	Bunch of grapes	0* 0 7 14 21	< 0.02 0.03 0.02 < 0.02 <u>< 0.02</u>	Bachmann, J, 1994d RA-2084/93
France Sorgues 1993		300 EC	0.014–0.015	0.023	3	Bunch of grapes	0* 0 7 14	< 0.02 < 0.02 < 0.02 < 0.02	Bachmann, J, 1994d RA-2084/93

Location (variety)	Year	Form	Application			Analysis			Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	DAT	Residues	
(Cinsault)							25	< 0.02	
cGAP Spain		SC	0.063	–	4		Table grapes: PHI 15d, Wine grapes: PHI 21d		
France St. Andiol	1996	250 EC	0.038–0.063	0.038– 0.063	4	Bunch of grapes	0* 0 7 14 21	0.02 0.09 0.04 <u>0.04</u> <u>0.02</u>	Heinemann, O; Allmendinger, H, 1998 RA-2025/96
(Muscat de Hambourg)						Berries	7	0.03	
France Laudun	1995	250 EC	0.038–0.063	0.038– 0.063	4	Bunch of grapes	0* 0 7 14 21 28	< 0.02 0.05 0.03 0.02 < 0.02 <u>0.1</u>	Nuesslein, F, 1996a RA-2023/95
(Clairette)						Berries	7	0.03	
Greece Ano Diminio - Korinthias	1995	250 EC	0.038–0.063	0.0048– 0.0078	4	Bunch of grapes	0 3 7 14	0.15 0.17 0.19 <u>0.11</u>	Nuesslein, F, 1996a RA-2023/95
(Sultanina)						Berries	7	0.06	
Italy Capriata d'Orba	1996	250 EC	0.038–0.063	0.0038– 0.0063	4	Bunch of grapes	0 7 14 21	0.06 0.05 <u>0.05</u> <u>0.04</u>	Heinemann, O; Allmendinger, H, 1998 RA-2025/96
(Barbera)						Berries	7	0.04	
Italy Borgo Piave	1995	250 EC	0.038–0.063	0.0038– 0.0063	4	Bunch of grapes	0* 0 3 7 14	0.04 0.18 0.11 0.10 <u>0.06</u>	Nuesslein, F, 1996a RA-2023/95
(Trebbiano Toscana)						Berries	7	0.07	
Spain Lavern	1996	250 EC	0.038–0.063	0.0063– 0.011	4	Bunch of grapes	0 7 14 21	0.04 0.03 <u>0.02</u> <u>0.02</u>	Heinemann, O; Allmendinger, H, 1998 RA-2025/96
(Carinena)									
Spain E-41808 Villanueva	1996	250 EC	0.038–0.063	0.0063– 0.011	4	Bunch of grapes	0* 0	< 0.02 0.09	Heinemann, O; Allmendinger, H, 1998
Garrido fino						Berries	7 14 21	< 0.02 < <u>0.02</u> < <u>0.02</u>	RA-2025/96
Spain Umbrete, Sevilla	1995	250 EC	0.038–0.063	0.0063– 0.011	4	Bunch of grapes	0* 0 7 14 21 28	0.04 0.16 0.03 < <u>0.02</u> < <u>0.02</u> < 0.02	Nuesslein, F, 1996a RA-2023/95
(Garrido fino)						Berries	7	0.02	
Spain Sant Marcal	1993	250 EC	0.038–0.05	0.0083– 0.019	3	Bunch of grapes	0* 0 13 21 27 34	< 0.02 0.10 <u>0.07</u> <u>0.04</u> 0.02 0.03	Nuesslein, F, 1996b RA-2016/93
(Xarelo)									
Spain		250	0.038–0.063	0.011–	3	Bunch of grapes	0*	0.02	Nuesslein, F,

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	DAT	Residues	
Les Gunyoles 1993 (Macabeo)	EC		0.019			0 14 21 28 34	0.12 <u>0.04</u> <u>0.04</u> 0.04 0.04	1996b RA-2016/93
Turkey Manisa, Salihli, Taytan 1988 (Sultana Seedless)	50 EW	0.05	0.005	4	Fruit	0 14 21 56	0.12 <u>0.04</u> < <u>0.02</u> < 0.02	Anon., 1989g 0100-88
Turkey Manisa 1990 (Sultanine)	50 EW	0.05	0.005	4	Fruit	0 20	0.2 <u>0.03</u>	Anon., 1992c 0604-90
Turkey Izmir 1990 (Sultanine)	50 EW	0.05	0.005	4	Fruit	0 20	0.07 < <u>0.02</u>	Anon., 1992f 0608-90
Turkey Izmir 1990 (Sultanine)	50 EW	0.05	0.005	4	Fruit	0 13	0.39 <u>0.08</u>	Anon., 1992g 0609-9

Table 4 Residues of triadimenol in grapes following foliar treatment (submitted in 2014)

Location Year (variety)	Form	Application			Analysis					Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	DAT	triadimenol	triadimenol incl. conj.	triadimenol- hydroxy incl. conj.	
cGAP France	EC	0.019	—	3		PHI 21				
France (North), Athée sur Cher 2010 (Chardonnay)	EC 300	0.019	0.01	3	bunch of grapes	0 0 3 7 14 21	0.02 0.03 0.03 0.02 0.01 <u>0.01</u>	0.02 0.03 0.03 0.02 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	Schöning, R, Ballmann, C., 2011 10-2203
France (North), Chamay les Macon 2010 (Pinot noir)	EC 300	0.019	0.01	3	bunch of grapes	0 0 3 7 14 21	0.03 0.05 0.05 0.05 0.04 <u>0.04</u>	0.03 0.05 0.06 0.05 0.04 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	Schöning, R, Ballmann, C, 2011 10-2203
France (North), Saint Nicolas de Bourgueil 2010 (Cabernet Franc)	EC 300	0.019	0.01	3	bunch of grapes	0 0 3 7 14 21	< 0.01 0.02 0.01 < 0.01 < 0.01 < <u>0.01</u>	< 0.01 0.02 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	Schöning, R, Ballmann, C, 2011 10-2203
Germany, Vennungen 2010 (Huxelrebe)	EC 300	0.019	0.002	3	bunch of grapes	0 0 3 7 14 21	0.07 0.13 0.12 0.14 0.13 <u>0.13</u>	0.09 0.1 0.13 0.14 0.13 0.13	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	Schöning, R, Ballmann, C, 2011 10-2203
Germany, Uhldingen- Mühlendorf	EC 300	0.019	0.002	3	bunch of grapes	0 0 3 7	0.06 0.11 0.1 0.1	0.06 0.11 0.1 0.1	< 0.01 < 0.01 < 0.01 < 0.01	Schöning, R, Ballmann, C, 2011

Location (variety)	Year	Form	Application			Analysis					Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	DAT	triadimenol	triadimenol incl. conj.	triadimenol- hydroxy incl. conj.	
2010 (Müller-Thurgau)							14 21	0.08 <u>0.08</u>	0.08 0.08	< 0.01 < 0.01	10-2203
Belgium, Emines	EC 300	0.019	0.002	3	bunch of grapes		0 0 3 7 14 21	0.04 0.06 0.05 0.04 0.05 <u>0.03</u>	0.04 0.06 0.07 0.05 0.06 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	Schöning, R, Ballmann, C, 2011 10-2203

Triadimenol analysed with method 01072/M001

Triadimenol incl. conjugates and triadimenol-hydroxy incl. conjugates analysed with method 004800/M001

APPRAISAL

Triadimenol is a systemic fungicide formed as the primary metabolite of triadimefon but also used as an active substance on its own. Its main mode of action is an inhibition of the ergosterol biosyntheses in fungi. Triadimenol was evaluated by JMPR several times since 1978 and the last time in 2004 for toxicology, when an ADI of 0–0.03 mg/kg bw and an ARfD of 0.08 mg/kg bw was established and in 2007 and 2009 for residues.

Definition of the residue in plant and animal commodities (for the estimation of dietary intake and for compliance with MRLs): sum of triadimefon and triadimenol

In 2007 the Meeting evaluated use patterns of triadimefon and triadimenol in grapes; however, a short-term dietary intake concern was identified without opportunity for an alternative GAP. In 2009 the 2007 evaluation on the use on grapes was repeated by the JMPR, but again no alternative GAP could be identified.

The current Meeting received new information on use patterns for triadimenol in grapes supported by new supervised residue trials on grapes from Europe.

Methods of analysis

The 2007 Meeting evaluated several methods of analyses for triadimenol in different plant and animal matrices with a LOQ of 0.05 mg/kg (GC-FID or GC-MS), based on the multi-residue method DFG-S19.

To the 2014 Meeting two new analytical methods were provided measuring triadimenol, with a LOQ of 0.01 mg/kg. Both methods involve measurement by HPLC-MS/MS for triadimenol. One of these methods measures also triadimenol including sugar conjugates and triadimenol-hydroxy including sugar conjugates in plant matrices with a LOQ of 0.01 mg/kg. For the determination of conjugates enzymatic treatment may be included in the extraction procedure.

Stability of pesticide residues in stored analytical samples

In 2007 the Meeting concluded that triadimenol is stable in stored samples of plant and animal origin for at least 24 months.

Results of supervised residue trial on crops

Grapes

For grapes, two new GAPs from France and Spain have been submitted to the Meeting.

In France, triadimenol is registered for grapes involving three applications of 0.019 kg ai/ha each with a PHI of 21 days. Supervised field trials from Europe matching the GAP were already reported in 2007 (2 trials) and amended by new trial data submitted to the current Meeting (6 trials).

Residues of triadimenol in grapes matching the French GAP were: < 0.01, 0.01, < 0.02(2), 0.03, 0.04, 0.08 and 0.13 mg/kg.

In Spain triadimenol is registered for grapes involving four applications of 0.063 kg ai/ha each with a PHI of 15 days for table grapes and 21 days for wine grapes. Supervised field trials from Southern Europe and Turkey matching the GAP were already reported in 2007.

Residues of triadimenol in table grapes matching the GAP were: < 0.02, < 0.02, 0.02, 0.04(3), 0.05, 0.06, 0.07, 0.08, 0.1, 0.11 mg/kg.

Residues of triadimenol in wine grapes matching the GAP were: < 0.02(4), 0.02, 0.02, 0.03, 0.04(3), 0.1 mg/kg.

Based on the French GAP, the Meeting estimated a maximum residue level of 0.3 mg/kg and HR and STMR values of 0.13 mg/kg and 0.025 mg/kg for grapes, respectively

Fate of residues during processing

The 2007 Meeting estimated processing factors for triadimenol in grapes of 0.45 for juice, 0.42 for wine, 3.1 for raisins, 3 for wet pomace and 5.7 for dry pomace. No additional data were submitted to the current Meeting.

Based on these factors and the newly estimated STMR value of 0.025 mg/kg the Meeting estimated STMR-P values for juice of 0.011 mg/kg, for dried grapes (= raisins) of 0.078 mg/kg, for wet grape pomace of 0.075 mg/kg and for dry grape pomace of 0.14 mg/kg.

Based on the newly estimated STMR value of 0.025 mg/kg for wine grapes, the Meeting estimated STMR-P value of 0.01 mg/kg for wine.

For dried grapes the Meeting estimated a HR-P of 0.4 mg/kg and a maximum residue level of 1 mg/kg to replace its previous recommendation of 10 mg/kg.

Residues in animal commodities

In the 2007 evaluation for triadimenol grape pomace (wet) was already taken into account for the livestock animal dietary burden calculation based on a residue level of 0.5 mg/kg, which is higher than the estimated residue level of 0.075 mg/kg resulting from the current GAPs.

Therefore, the Meeting concluded that the contribution of triadimenol residues in grapes after treatment according to the evaluated GAPs does not influence the overall dietary burden of livestock animals, making a re-assessment of the residue situation in animal commodities unnecessary. The Meeting confirms its previous recommendations for triadimenol in animal commodities.

RECOMMENDATIONS

Definition of the residue in plant and animal commodities (for the estimation of dietary intake and for compliance with MRLs): sum of triadimefon and triadimenol

On the basis of the additional data from supervised trials on grapes the Meeting recommends the following maximum residue levels for triadimenol.

CCN	Commodity	Recommended MRL mg/kg		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		new	previous		
FB 0269	Grapes	0.3	–	0.025	0.13
DF 0269	Dried grapes	1 ^a	10 ^b	0.155	0.4

^a Based on uses for triadimenol

^b Based on uses on triadimefon and triadimenol

For dietary intake only

CCN	Commodity	STMR or STMR-P mg/kg	HR or HR-P mg/kg
JF 0269	Grapes juice	0.011	–
	Grape pomace, dry	0.14	–
	Grape pomace, wet	0.075	–
	Wine	0.01	–

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDI) of triadimefon and triadimenol was calculated from previously estimated STMRs in 2007 and the new STMRs in 2014 for raw and processed commodities in combination with consumption data for corresponding food commodities. The results are shown in Annex 3.

The IEDI of the 17 GEMS/Food cluster diets, based on the estimated STMRs represented 1–3% of the maximum ADI (0.03 mg/kg bw).

The Meeting concluded that the long-term intake of residues of triadimefon and triadimenol from the uses considered by the Meeting is unlikely to represent a public health concern.

Short-term intake

The International Estimated Short-Term Intake (IESTI) of triadimenol calculated on the basis of the estimations made by the 2014 JMPR represented for children 0–10% and for the general population 0–5% of the ARfD (0.08 mg/kg bw). The Meeting concluded that the short-term intake of triadimenol from the uses considered by the 2014 JMPR is unlikely to represent a public health concern.

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TRIFORINE (116)

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EXPLANATION

Triforine is a systemic fungicide for control of blackspot, powdery mildew and rust. It was first evaluated by JMPR in 1978 (T, R) and the latest toxic review was in 1997 (T). The ADI for Triforine was established as 0–0.02 mg/kg bw and no ARfD was set up in previous JMPR. Triforine was scheduled at the 45th session of the CCPR (2013) for the periodic re-evaluation of toxicity and residues by the 2014 JMPR.

The residue data was submitted by the manufacturer and would support the following commodities: apples, blueberries, Brussels sprouts, cereal grains, cherries, common beans, apricots, currants (black, red white), fruiting vegetables, cucurbits, gooseberries, peaches, plums (including prunes), strawberries and tomatoes.

IDENTITY

Common name	Triforine
Chemical name	
IUPAC:	<i>N,N'</i> -{piperazine-1,4-diylbis[(trichloromethyl)methylene]} diformamide
CAS:	<i>N,N'</i> -[1,4-piperazinediylbis(2,2,2-trichloroethylidene)]bis[formamide]
CAS Registry No:	26644-46-2
CIPAC No:	360
Synonyms:	W 524, CME 102, SAG 102, Cela W524
Structural formula:	
Molecular formula:	C ₁₀ H ₁₄ Cl ₆ N ₄ O ₂
Molecular weight:	434.96

PHYSICAL AND CHEMICAL PROPERTIES*Technical material*

Property	Results	Reference
Appearance (colour/ physical state/ odour)	Cream coloured/ solid, crystalline powder/ slightly like garlic (purity: not specified)	Jungblut, 1989 TF-302-006
Vapour pressure	8.0 × 10 ⁻² Pa at 25 °C (98.1% purity)	Cardinaals, 1988 TF-306-004
Melting point	151.3–154.1 °C (98.1% purity) The colour turned from white into light brown at 148.6 °C.	Van Klooster-Cornelissen, 1988 TF-303-001
Octanol/water partition coefficient	log P _{ow} = 2.2 at 20 °C (98.1% purity)	Van Klooster-Cornelissen, 1988

Property	Results	Reference
		TF-315-002
Solubility in water	11.3×10^{-3} g/L in buffer solution, pH 5.0 9.0×10^{-3} g/L in buffer solution, pH 7.0 8.7×10^{-3} g/L in buffer solution, pH 9.0 at 20 °C (98.1% purity)	Van Helvoirt, 1988 TF-311-007
Solubility in organic solvents (98.1% purity)	Methanol (flask method) 46.9 g/L at 19.5 °C Toluene (flask method) 0.11 g/L at 19.5 °C Tetrahydrofuran (flask method) 168 g/L at 19.5 °C Hexane (flask method) $< 4.7 \times 10^{-3}$ g/L at 19.5 °C Hexane (column method) 2.6×10^{-3} g/L at 19.5 °C	Van Klooster-Cornelissen, 1989 TF-312-004
Relative density	1.55 g/cm ³ at 20.0–20.6 °C (98.1% purity)	Van Klooster-Cornelissen, 1988 TF-308-002
Henry's law constant	$2.5 \text{ Pa} \times \text{m}^3 \times \text{mol}^{-1}$ (calculation) (98.1% purity)	Cardinaals, 1988 TF-306-006
Dissociation constant	pK _a = 10.6 at 20 °C (98.1% purity)	Van Klooster-Cornelissen, 1989 TF-311-008

Radioactive substance

Property	Results	Reference
Hydrolysis	The calculated DT ₅₀ (24–25 °C) 3.5 days at pH 5 3.4 days at pH 7 3.5 days at pH 9 The calculated DT ₅₀ (25 °C) 2.6 days at pH 5, 2.8 days at pH 7, 2.6 days at pH 9 for the ring labelled 2.9 days at pH 5, 3.1 days at pH 7, 3.1 days at pH 9 for the side chain labelled The hydrolysis products counting for > 10% of the initial dose: WOS 2379 and W2379	Obrist, 1989 TF-322-017 Bass, 1993 TF-322-018
Photolysis	Initial DT ₅₀ under simulated sunlight (12 h on/12 h off) = 1.5 days at pH 7 (25 °C) (equivalent to 2.1 days of Florida summer sunlight)	Waring, 1993 TF-324-009

Formulations: Emulsifiable concentrate (EC)

METABOLISM AND ENVIRONMENTAL FATE

The metabolism, distribution of triforine has been investigated in animals and plants. The fate and behaviour of triforine in animals, plants and the environment was investigated using the [^{14}C]-labelled test materials shown in Figures 1.

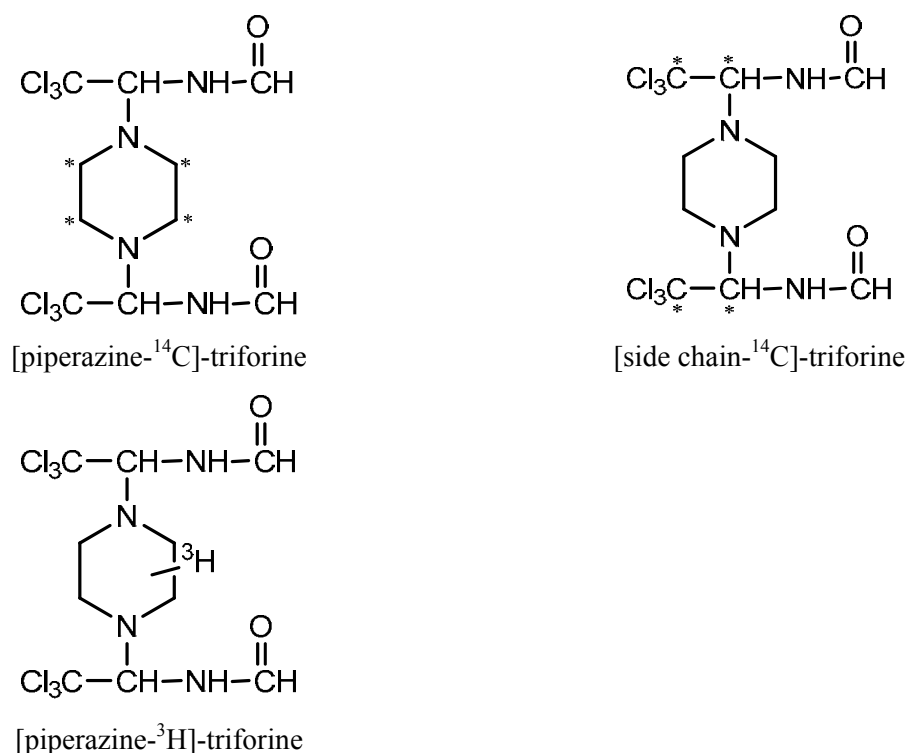
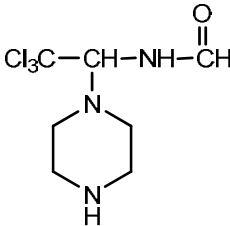
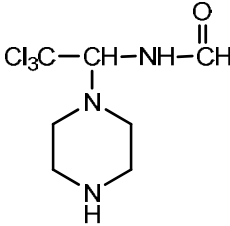
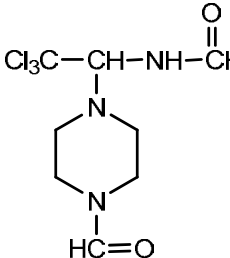
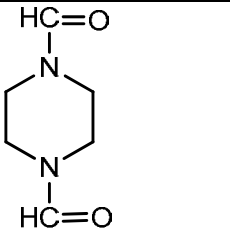
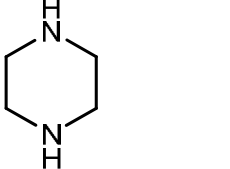
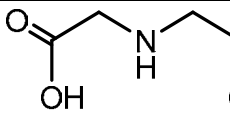
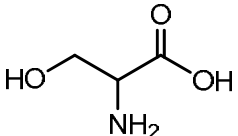


Figure 1 [^{14}C] and [^3H]-Labelled test materials used in animals, plants metabolism studies, and the environmental fate studies

The chemical structures of the major degradation compounds from the metabolism of triforine are provided below.

Compound name		Structure	Found in metabolism studies
WOS 2379	<i>N</i> -{2,2,2-trichloro-1-[4-(2-oxoacetyl)piperazin-1-yl]ethyl} formamide		Livestock, Soil
W 2379	Hydrate of <i>N</i> -(2,2,2-trichloro-1-[4-(2,2-dihydroxyacetyl)piperazin-1-yl]ethyl} formamide		Livestock

Compound name		Structure	Found in metabolism studies
W 1084	<i>N</i> -(2,2,2-trichloro-1-piperazin-1-yl-ethyl)formamide	 <chem>ClC(Cl)(Cl)CN1CCNCC1NC=O</chem>	Rat, Livestock, Plants
W 1069	<i>N</i> -(2,2,2-trichloro-1-piperazin-1-yl-ethyl)formamide hydrochloride	 HCl	Livestock, Plants, Soil
W 625	<i>N</i> -[2,2,2-trichloro-1-(4-formyl piperazin-1-yl)ethyl]formamide	 <chem>ClC(Cl)(Cl)CN1CCN(CC1NC=O)C=O</chem>	Plants, Soil
WOS 613	piperazine-1,4-dicarbaldehyde	 <chem>O=CNC1CCN(CC1)C=O</chem>	Plants
Piperazine		 <chem>C1CCNCC1</chem>	Plants, Soil
Trichloroethanol	2,2,2-trichloroethanol	$\text{Cl}_3\text{C}-\text{CH}_2-\text{OH}$	Rat, Livestock
Trichloroethanol sulphate		$\text{Cl}_3\text{C}-\text{CH}_2\cdot\text{OSO}_3\text{H}$	Livestock
Iminodiacetic acid		 <chem>OC(=O)CNCC(=O)O</chem>	Plants
Glycine		$\text{H}_2\text{N}-\text{CH}_2-\text{COOH}$	Plants

Compound name		Structure	Found in metabolism studies
Serine			Plants

Animal metabolism

The Meeting received animal metabolism studies of triforine in rats, lactating goats and laying hens. The study on rats was evaluated by the WHO Core Assessment Group of the 2014 JMPR. A summary of the rat metabolism is given in this section.

Rats

Triforine is rapidly metabolized and excreted in rats. Highest residues were found in liver followed by kidney. Residues were lower in muscle and fat. The glucuronide of *N*-[2,2,2-trichloro-1-(piperazin-1-yl) ethyl]-formamide (W 1084), which was formed by the cleavage of a side chain, and the side-chain metabolites trichloroethanol including its glucuronide and *N*-acetylcysteine conjugate of 2,2,2-trichloroethylamine was detected in urine. W 1084 and triforine was detected in the faeces (Darda, 1974, Hawkins *et al.*, 1992).

Lactating goat

Study 1

The metabolism study for the lactating goats was performed with [piperazine-¹⁴C]-triforine (Ellgehausen, 1981: TF-440-013). Three lactating goats weighing approximately 20 kg were individually housed in cages and allowed free access to feed and water during the whole study. The test item was given as suspension in 0.5% CMC (Carboxymethyl Cellulose) in distilled water. It was administered by stomach tube to the three goats, one receiving 25 mg/kg bw and the other two receiving 100 mg/kg bw. Non-labeled triforine was given once daily for 7 days, thereafter on Day 8, 9 and 10, [¹⁴C]-labelled triforine was administered once daily.

Samples of milk were taken prior to the first treatment of [¹⁴C]triforine, during the dosing period twice daily, at 8 hours and 24 hours after treatment, and thereafter every 24 hours. Collection of urine and faeces was performed daily. The animals were sacrificed 6 days after the last treatment and the fat, muscle, heart, liver, mammary glands, kidneys, brain and blood removed for analysis. One goat (Goat C) was sacrificed 4 hours after the last treatment.

Goat	Average dose (mg/kg bw/day)	ppm in feed Based on feed consumption of 2 kg/day	Time between last treatment and sacrifice
A	24.41	250	6 days
B	97.98	1000	6 days
C	96.65	1000	4 hours

The radioactivity administered to the goats was rapidly eliminated. A total of 46.6% and 71.5% of the applied radioactivity were eliminated in urine and faeces in 24 hours by Goats A and B, respectively. In the following 5 days, a further 15.9 and 14.0% were excreted by Goats A and B, respectively. The radioactivity found at sacrifice in tissues, blood, and milk of Goats A and B amounted to an average of 1.6%, 0.3% and 0.3% of the applied radioactivity. Goat C sacrificed 4 hours after the last treatment showed higher values in tissues and blood, i.e. 6.1% and 0.8% respectively. Total recoveries of radioactivity from Goats A and B were 65.5% and 89.1% of the applied radioactivity, respectively.

The radioactivity in the milk was determined by combustion of 1 g aliquots. The radioactivity in urine was determined by direct liquid scintillation counting (LSC). The faecal samples were lyophilised, homogenized and removed for combustion followed by LSC of the radioactivity of carbon dioxide collected in scintillation cocktail. The radioactivity in blood was determined by combustion of 1 g samples. Tissues were solubilized overnight at 55 °C and radioassayed by LSC. The tissues, blood and milk samples were stored at -20 °C.

Table 1 Radioactivity in selected tissues from lactating goats (mg triforine equivalent/kg)

Goat	Tissues							
	Blood	Heart	Liver	Kidney	Muscle	Mammary	Brain	Fat
A	2.07	3.54	11.8	7.93	2.35	3.47	1.42	0.03
B	5.23	12.1	32.7	23.1	8.14	12.0	6.65	1.27
C	20.8	35.9	201	101	24.9	39.0	26.1	20.1

Table 2 Radioactivity in milk from lactating goat (mg triforine equivalent/kg)

Goat	Timing of sampling (hours after the first treatment of labelled triforine)											
	0–8	8–24	24–32	32–48	48–52	48–56	56–72	72–96	96–120	120–144	144–168	168–192
	T a		T a		T a							
A	5.23	4.93	8.84	9.65	–	11.6	12.3	4.82	1.99	1.19	0.69	0.59
B	17.9	17.1	38.4	26.6	–	70.9	43.0	9.00	3.98	2.66	2.23	1.54
C	18.8	17.4	32.1	24.9	34.4	–	–	–	–	–	–	–

^a Treatment with [¹⁴C]-labelled triforine

Up to six metabolite fractions were isolated from urine and faeces extracts. The amount of triforine in urine and faeces ranged from 0.4% to 2.6% of the applied radioactivity. The major metabolite in urine was shown by co-chromatography to be W 1084. It amounted to 7.1% (Goat A) and 12.6% (goat B) of the applied radioactivity in urine and less than 1% in faeces.

The study was performed to determine the extractability of residues in liver, kidney and muscles of the goats and to determine the nature of the extractable radioactivity (Ellgehausen, 1982: TF-440-014). Until analysis the tissues were stored at -20 °C. Tissue samples (50 g) were extracted with acetone (100 mL) followed by methanol/water (8:2, 100 mL). Proteins of milk (100 g) were precipitated with acetone (200 mL) by centrifugation and removed. The protein precipitate was then extracted with acetone and methanol/water (8:2) as done with tissues. The organic solvent was evaporated and the remaining aqueous phase was partitioned with chloroform. The amount of extractables and non-extractables were determined by combustion and thin layer chromatography (TLC).

The residual radioactivity in the liver of Goat C, sacrificed 4 hours after the last administration of [¹⁴C]triforine was found to be extractable up to 68.4% TRR. This extractable radioactivity consisted of at least five metabolite fractions. M1 and M2 represented unknown polar metabolite fractions whereas WOS 2379 and W 1084 were characterized. The residual radioactivity in the liver of Goat B, sacrificed 6 days after the last treatment, was extractable up to 14.1% TRR. The metabolite pattern found was similar to that found in the extractables of the liver of Goat C. The predominant metabolite fraction was M1 representing 10.2% TRR. Triforine, WOS 2379 and W 1084 accounted only for 1.3%, 1.6% and 1.0% TRR, respectively.

The residual radioactivity in the kidneys of Goat B and C was extractable up to 17.9% and 78.2% TRR, respectively. These figures corresponded to 4.13 and 78.2 mg equiv/kg. The metabolite pattern of the extractables was similar to that found in the extractable of the liver. The predominant extractable fractions found in Goat C was polar unknown metabolite fraction M1 accounting for 31.1% TRR. Triforine, WOS 2379 and W1084 represented at the same time 19.3%, 8.4% and 18.5% TRR, respectively. Characterization of the extractable radioactivity of the kidneys of Goat B showed a similar metabolite pattern with regard to the relative amounts in the extracts. The

predominant extractable fraction was M1 accounting for 10.7% TRR. Triforine represented 3.6% TRR only.

Due to the low radioactivity concentration in muscle of Goat B and the low specific radioactivity, only muscles of Goat C were analyzed. The residual radioactivity accounted for 19.6 mg equiv/kg (78.7% TRR) was found to be extractable. Analysis by TLC showed that the predominant radioactive fraction was triforine with 40.6% TRR. The other metabolites accounted for 9.5% (W 1084), 13.0% (WOS 2379), 0.70% (M2) and 15.1% TRR (M1).

The radioactivity in the pooled milk fractions of Goat B sampled within 24 hours after the last administration of [^{14}C]triforine accounted for 49.6 mg equiv/kg.

Table 3 Extraction and characterization of residues in liver, kidney and muscle of Goat B sacrificed 6 days after the last treatment at 1000 ppm and Goat C sacrificed 4 hours after the last treatment at 1000 ppm

	Goat B			Goat C		
	% applied	% TRR	TRR(mg/kg) ^a	% applied	% TRR	TRR(mg/kg) ^a
Liver						
Extract	0.043	14.1	4.61	1.25	68.4	137
Triforine	0.004	1.3	0.43	0.278	15.2	30.6
M1-polar unknowns	0.031	10.2	3.33	0.331	18.1	36.4
M2-unknown (Rf=0.32)	< 0.001	< 0.03	< 0.1	0.144	7.9	15.8
WOS 2379	0.005	1.6	0.54	0.268	14.7	29.5
W 1084	0.003	1.0	0.32	0.229	12.5	25.2
Unextracted	0.262	85.9	28.1	0.578	31.6	63.5
Total in liver	0.305	100	32.7	1.83	100	201
kidney						
Extract	0.005	17.9	4.13	0.093	78.2	79.3
Triforine	0.001	3.6	0.83	0.023	19.3	19.6
M1-polar unknowns	0.003	10.7	2.48	0.037	31.1	31.6
M2-unknown (Rf=0.32)	< 0.001	< 0.1	< 0.01	0.001	0.8	0.85
WOS 2379	0.001	3.6	0.83	0.010	8.4	8.53
W1084	< 0.001	< 0.1	< 0.01	0.022	18.5	18.8
Unextracted	0.023	82.1	19.0	0.026	21.8	22.2
Total in kidney	0.028	100	23.1	0.119	100	101
muscle						
Extract	NA ^b	NA	NA	2.63	78.7	19.6
Triforine	NA	NA	NA	1.35	40.6	10.1
M1-polar unknowns	NA	NA	NA	0.505	15.1	3.76
M2-unknown (Rf=0.32)	NA	NA	NA	0.023	0.70	0.17
WOS 2379	NA	NA	NA	0.435	13.0	3.24
W1084	NA	NA	NA	0.318	9.5	2.37
Unextracted	NA	NA	NA	0.713	21.3	5.31
Total in kidney	NA	NA	NA	3.35	100	24.9
milk						
Extract		62.3	30.9	NA	NA	NA
Triforine	< 0.001	–	< 0.1	NA	NA	NA
M1-polar unknowns	0.146	62.3	30.9	NA	NA	NA
M2-unknown (Rf=0.32)	< 0.001	–	< 0.1	NA	NA	NA
WOS 2379	< 0.001	–	< 0.1	NA	NA	NA
W1084	< 0.001	–	< 0.1	NA	NA	NA
Unextracted	0.088	37.7	18.7	NA	NA	NA
Total in milk	0.234	100	49.6	NA	NA	NA

^a mg triforine equivalent/kg tissue

^b Not analyzed

The study was further performed for the investigation of the polar metabolite fractions and unextracted fraction (Schlüter, 1984: TF-440-015). The polar metabolite fraction M1 from Liver B, Liver C and milk was investigated. In all cases, thin layer chromatography showed that M1 consisted of a number of components that could not be well separated. Incubation with β -glucuronidase/aryl

sulphatase did not produce any difference in chromatographic profile indicating that M1 was not an easily hydrolyzed sulphate or glucuronide conjugate. When M1 was heated with strong acid piperazine was the only cleavage product. All the components in fraction M1 disappeared from the TLC plate and piperazine was formed.

Study 2

The adsorption, distribution, metabolism and excretion of radioactivity have been studied following repeated oral administration of [piperazine-¹⁴C]triforine to the dairy goat at a mean dose level of 67 mg/day (Richardson, 1994: TF-440-019). This was equivalent to a dietary inclusion of approximately 49 ppm (based on the food consumption of the goat during the study). One goat received five daily oral doses of [¹⁴C]triforine. The goat was given the dose in the morning before feeding but after milk and excreta collections. At study termination (*ca.* 6 h after the final dose) selected tissues were removed or sampled. The radiolabelled triforine derived material excreted was characterized using chromatographic procedures.

The overall recovery of the administered radioactivity was 72.3%, the majority of which was present in the urine (39.8%) and faeces (19.2%). Most of the remainder was associated with the contents of the gastro-intestinal tract (9.54%). Negligible radioactivity was recovered in cage washings/ debris (1.21%), milk (0.87%), tissues (0.82%) and residual carcass (0.88%).

From day 1, concentrations of radioactivity in whole milk were in the range 0.314 to 0.501 mg equiv/kg. Fractionation of 72–96 h whole milk showed that 32% of the radioactivity was found in cream and 76% was found in skim. Further fractionation showed that 7% of the radioactivity in the skimmed milk was found in the curds and 64% in whey. At *ca.* 6 h after the final dose, highest tissue residue levels were found in the liver (2.60 mg equiv/kg) and kidney (1.46 mg equiv/kg), with lower concentrations of 0.271 and 0.016 mg equiv/kg present in muscle and fat respectively.

Table 4 Extraction of radioactive residues from tissues and milk

	Liver		Kidney		Muscle		Fat		Milk (96–102 h)	
	%TR R	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TR R	mg/kg	%TRR	mg/kg
Extracted	53	1.39	55	0.787	60	0.179	53	0.012	72	0.366
Organic	5	0.131	7	0.100	5	0.013	–	–	4.5	0.023
Aqueous	37	0.973	38	0.543	26	0.078	–	–	48	0.240
Others							17	0.004	15	0.075
Unextracted	46	1.21	47	0.672	31	0.092	17	0.004	34	0.170
Total	99	2.60	102	1.46	91	0.271	70	0.016	106	0.536

Chromatography of the extracted radioactivity revealed metabolites with a wide range of polarity. TLC analysis proved to be the most efficient in terms of resolution of components. However given the very polar nature of some of the metabolites and low relative concentrations, the quality and hence the reproducibility of the chromatography in some solvent systems was poor. Comparison of the extracted radioactivity indicated that there were some similarities in the metabolite profiles for liver, kidney, muscle and urine; however, the metabolites in milk were very polar and more difficult to compare. The radioactivity which partitioned into organic solvent from liver extracts, kidney extracts, muscle extracts and urine all contained the same metabolites. The radioactivity which partitioned into the aqueous phase was more difficult to compare due to the polarity of the metabolites however there were some similarities in profiles for liver, kidney and muscle.

Piperazine was identified in liver extracts (5% TRR), in kidney extracts (6% TRR) and in urine (14% of the 96–102 h urine). W 1069 was identified as a minor metabolite in urine (8%).

The proportion of piperazine in tissue extracts, milk and urine was increased by acid treatment of the samples. These results when compared with the result of acid treatment of triforine hydrolysis products (which showed that the ring system was stable to this process) indicated that the increased proportion of piperazine arose from the cleavage of metabolites which featured remnants of one or both of the triforine side chains but without modification of the piperazine ring.

Unextracted residues in all tissues and milk were solubilized by 6 M acid treatment. Piperazine was identified in samples derived from the acid treatment of unextracted residues from liver but not in other samples (kidney and milk residues).

Laying hens

Study 1

The metabolism study for the laying hens was performed with [piperazine-¹⁴C]-triforine (Ellgehausen, 1981: TF-440-012). Six-month old laying hens (white leghorn hybrids) were housed individually in metabolism cages. They were allowed free access to food and water during the study. The test item was given as suspension in 0.5% CMC (Carboxymethyl Cellulose) in distilled water. It was administered once daily into the crop by intubation to three groups of hens. One group (Group A) received 25 mg/kg bw/day; the other two groups (Groups B and C) received 100 mg/kg bw/day. Non-labelled triforine was given daily for 7 days at the same rates thereafter on day 8, 9 and day 10, [¹⁴C]triforine was administered. Based on an assumed food consumption of 100 g diet per day and a hen body weight of 2 kg the 100 and 25 mg/kg dose were equivalent to 2000 ppm and 500 ppm dietary concentration, respectively.

Hen group	Average dose (mg/kg bw/day)	ppm in feed	Time between last treatment and sacrifice
A	23.40	500	7 days
B	96.79	2000	7 days
C	96.94	2000	4 hours

During the dosing period eggs were collected twice daily 8 hours and 24 hours after the treatment, thereafter every 24 hours. Collection of excreta was performed accordingly. The animals were sacrificed either 4 hours (Group C) or 7 days (Groups A and B) after the last treatment and the liver, heart, muscle, fat, and blood were sampled for analysis.

The eggs were divided into egg yolk/white and shells per group for each interval and pooled samples were homogenized. The subsamples were removed for combustion and radioactivity determination by liquid scintillation counting (LSC). The excreta samples per group for each interval were pooled, lyophilized, homogenized and subsamples were removed for combustion and radioactivity determination by LSC. Tissues and blood were solubilized overnight at 55 °C and direct radio-assayed by LSC. The tissues, blood and egg samples were stored until analysis at -20 °C.

The excreta samples during the experiment were pooled for intervals from day 8 to 8 hours after the last treatment and then thereafter to the end of experiment. The pools were homogenized and an aliquot of 50 g was taken for extraction. Each aliquot was extracted with acetone (3 × 200 mL) by strong agitation for 30 and finally 60 min. Thereafter the excreta samples were continuously extracted overnight in a Soxhlet apparatus. The radioactivity in the extracts was measured by LSC. TLC was used pre-coated silica gel 60 F 254 plates with various solvent systems. Three different solvent systems gave good separation of triforine and the available reference compound (W 1084).

The radioactivity administered to the hens was rapidly eliminated by the animals of all groups (53.7–83.6% in 56 hours after the first dose). In the following 7 days a further 10.2–14.8% of the administered radioactivity was excreted by the remaining groups giving 75.7% excreted from Group A and 93.8% from Group B.

At the lower dose level (Group A) the TRR ranged from < 0.05 mg/kg in fat to 1.80 mg/kg for liver. The corresponding figures at the higher dose level (Group B) were four times higher, clearly showing the direct correlation between dose level and tissue/organ residue. The animals of Group C, which received the same amount of triforine as Group B but which were killed 4 hours after the last treatment showed higher residual radioactivity in tissues and organs.

Table 5 Total radioactive residues in tissues of laying hens

TRR (mg triforine equivalent/kg)

	Blood	Heart	Liver	Muscle	Muscle forestomach	Fat
Group A	1.25	1.22	1.80	0.58	0.74	<0.05
Group B	4.30	4.37	5.89	1.86	3.10	0.25
Group C	23.5	28.7	140	14.1	21.4	2.26

The egg residues in Group A were on the average three times lower than in Group B or C. The highest values in eggs were found about 4 to 5 days after the first treatment with [^{14}C]triforine. No influence of the compound during the whole study on egg production was observed although the hens were dosed up to 100 mg/kg over a period of 10 days.

Table 6 Total radioactive residues in selected eggs of laying hens

Hours after the first dose	TRR (mg triforine equivalent/kg)		
	7 days after the last treatment (500 ppm)	7 days after the last treatment (2000 ppm)	4 hours after the last treatment (2000 ppm)
0–8 T	0.04	0.17	0.02
8–24	–	1.02	–
24–32 T	0.54	1.52	2.71
32–48	0.91	–	–
48–52 T	NS	NS	6.39
48–56	1.88	5.87	–
56–72	–	–	–
72–96	3.11	4.28	–
96–120	3.41	8.53	–
120–144	2.07	6.09	–
144–168	–	–	–
168–192	2.47	4.34	–
192–216	0.84	3.99	–

NS: no egg sampling

T: Treatment with [^{14}C]triforine

Metabolites were characterized by extraction of excreta followed by TLC. Analysis of the pooled 0–56 and 56–216 hours excreta from high and low dose application showed in general the same pattern of metabolites in the extractables. The major metabolite fraction characterized by TLC was W 1084.

Study 2

The metabolism of triforine in laying hens was investigated after administration of [side chain- ^{14}C]triforine for 10 consecutive days at a dose of 32 ppm in the feed (Mayo, 1994: TF-440-020). The dose was administered orally via a gelatine capsule at a rate of 3.5 mg/hen/day. Combined excreta from five treated hens and from two control hens were collected at 24-hour intervals from 24 hours prior to the first treatment up to the time of sacrifice. Any eggs laid were collected prior to administration of the next treatment, labelled and stored at 4 °C prior to further treatment. Hens were sacrificed *ca.* 6 hours after the last treatment and the tissues taken for analysis. There was no visible fat on the skin. All samples were stored at < -15 °C prior to analysis.

The combined extracts of the excreta collected at day 10 were analyzed directly by radio-TLC. Metabolites were scraped from TLC plates and extracted from the silica with methanol and further purified by HPLC. Liver, muscle and egg white and yolk samples were extracted sequentially with acetone (twice), methanol/water 8:2 (twice) and finally with water (twice). Samples of egg white and liver were homogenized and pre-incubated with protease in buffer solution for 12–18 hours at 37 °C before the same extraction process. The residue from the yolk extraction was re-extracted with the same solvent mixtures once more followed by a final water extraction. Skin was extracted by dissolving the sample in hexane and extracting the residue three times with methanol/acetone (9:1). The hexane phase was back extracted five times with acetonitrile. The extracts were radio-assayed separately and combined as appropriate for chromatographic analysis.

Radioactivity recovered in excreta during the 10 days accounted for about 85% of the total cumulative dose with 1.5% remaining in the gastrointestinal tract after sacrifice. Recovery of radioactivity in the faeces had increased from 76% on Day 1 to 90% between Day 6 and Day 9. A slight decrease to 85% observed at Day 10 may be attributed to sacrifice at 6 hours after the last treatment. Concentration of radioactivity in eggs increased steadily during the 10 days to a peak value of 1.6 mg equiv/kg (yolk) and 0.19 mg equiv/kg (white).

Table 7 The concentrations of radioactive components in tissues and eggs

	Fat		Liver		Liver (protease) ^a		Muscle	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Extract	0.09	94.6	0.84	48.9	1.6	94.6	0.21	87.7
Triforine	0.02	17.5	0.05	2.9	< 0.01	< 1.0	0.02	8.4
Fraction A	0.008	8.9	0.52	31.3	1.1	66.7	0.03	11.3
Fraction B	0.003	3.1	–	–	–	–	0.008	3.3
Component C/D (Trichloroethanol sulphate)	0.03	35.9	0.15	9.3	0.25	15.0	0.05	22.0
Fraction E	0.003	3.2	0.02	1.3	0.05	3.1	0.005	2.2
W 1069	–	–	0.04	2.1	–	–	0.05	21.5
Fraction H	0.008	8.9	–	–	–	–	–	–
Trichloroethanol	< 0.001	<1.0	< 0.01	<1.0	< 0.01	<1.0	< 0.01	<1.0
Fraction K	0.01	10.8	–	–	–	–	–	–
Others	0.003	2.8	–	–	0.04	2.3	0.02	8.4
Polar extracted	0.003	3.7	0.03	2.0	0.13	7.6	0.02	7.8
Unextracted	0.005	5.4	0.86	51.2	0.09	5.3	0.03	12.4
TRR	0.09	100	1.7	100	1.7	99.9	0.24	100
	Skin		Egg white ^{a,b}		Egg yolk ^{a,b}			
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR		
Extract	0.18	75.6	0.12	72.6	0.86	85.5		
Triforine	0.01	5.4	0.02	13.2	0.02	2.1		
Fraction A	0.009	3.8	0.08	47.9	0.15	14.2		
Fraction B	0.004	1.6	–	–	0.07	6.3		
Component C/D (Trichloroethanol sulphate)	0.13	55.7	0.01	6.1	0.25	24.6		
Fraction E	0.004	1.5	–	–	–	–		
W 1069	0.004	1.7	–	–	0.10	10.2		
Fraction H	0.006	2.6	–	–	0.19	18.0		
Trichloroethanol	< 0.01	<1.0	–	–	–	–		
Fraction K	–	–	–	–	–	–		
Others	0.008	3.4	–	–	0.04	3.70		
Polar extracted	–	–	0.009	5.4	0.07	6.4		
Unextracted	0.06	24.4	0.04	27.4	0.15	14.6		
TRR	0.24	100	0.16	100	1.0	100		

^a Extraction after protease treatment

^b Representative pool from Day 7–9

Fraction A-K: Unidentified

Treatment of liver with protease reduced the unextracted radioactivity from 0.86 to 0.09 mg equiv/kg (51.2 to 5.3% TRR). Further quantities of the same radioactive components (mainly fraction A and component C/D) were extracted after the protease treatment. Identity of component C/D was confirmed by mass spectrometry as the sulphate conjugate of trichloroethanol. Protease treatment increased the amount of fraction A from 31.3% to 66.7% TRR. The fraction A in the protease-treated extract was separated into 5 separate components each of which accounted for 0.07–0.40 mg equiv/kg (4–24% TRR). The sulphate conjugate of trichloroethanol was present in liver accounting for 9% TRR. There appeared to be a small amount of triforine in liver (2.9% TRR, 0.05 mg/kg) before protease treatment but chromatography was poor. Treatment with β -glucuronidase/sulphatase or mild chemical hydrolytic treatment did not release any identifiable components. Strong acid/base hydrolysis generated some discrete less polar components.

The breast and thigh muscle was extracted separately and sub-samples were combined for chromatography. The main components were the trichloroethanol sulphate conjugate and W 1069 each accounting for about 22% TRR (0.05 mg equiv/kg). Triforine was present at 8.4% TRR (0.02 mg/kg).

Triforine accounted for 17.5% TRR (0.02 mg/kg) and the sulphate conjugate of trichloroethanol accounted for 35.9% TRR (0.03 mg equiv/kg) in fat. There were at least five other components but each was less than 0.01 mg equiv/kg.

Triforine was also found in skin at low levels (0.01 mg/kg). The main fraction corresponded to the retention time of the trichloroethanol sulphate (55.7% TRR, 0.13 mg/kg). There were at least five other components but each was less than 0.01 mg equiv/kg.

Treatment of egg white with protease reduced unextracted radioactivity from 0.09 to 0.04 mg equiv/kg (58.7 to 27.4% TRR). The major component was the Fraction A which accounted for 19% (0.03 mg/kg). It was further separated by TLC into two polar components. Triforine and the trichloroethanol sulphate conjugate were found in small amounts (0.01 and 0.02 mg/kg respectively). All other components were less than 0.01 mg/kg.

Treatment of egg yolk with protease reduced unextracted radioactivity from 0.30 to 0.15 mg equiv/kg (29.1 to 14.6% TRR). Hydrolytic treatment of the residue of protease treated egg yolk, after extraction, with acid separated this unextracted radioactivity into an acid treated extract and residue each accounting for either < 0.05 mg equiv/kg or < 10% egg yolk radioactivity. Increased amounts of the polar fraction A were extracted after the protease treatment. Fraction A was separated into two components each accounting for < 0.05 mg equiv/kg in egg yolk. The major component in egg yolk was the sulphate conjugate of trichloroethanol which accounted for 25% TRR. W 1069 was observed and accounted for a further 10% TRR. Component H (less polar than triforine) in egg yolk was not identified.

Summary of animal metabolism

The metabolism of ^{14}C labelled triforine has been studied in lactating goats and laying hens. Triforine is very rapidly degraded in the goat even at the high dose levels used in the metabolism studies. Oxidation and hydrolysis leading to removal of the side chains from the piperazine ring was the main route of degradation. Free piperazine was not a metabolite but piperazine was identified following the solubilization of the matrix components followed by strong acid hydrolysis. The main extracted product was triforine although there were higher amounts of a mixture of highly polar components which were not completely separated. They were not identified but appeared to yield mainly piperazine on reflux with hydrochloric acid.

As in the goat, one of the side chains was lost to form W 1069 and the carbon from the side chain was found mainly as trichloroethanol and its sulphate conjugate. The major portion of the residue even with a relatively short pre-slaughter interval was unextracted with a very thorough extraction sequence involving a range of solvent polarities and overnight contact with the matrix. It is possible that trichloroethanol was further metabolized into small fragments that were incorporated into the tissue and egg matrix components. It is assumed that W 1069 was further metabolized to piperazine containing compounds which would also be bound to matrix components.

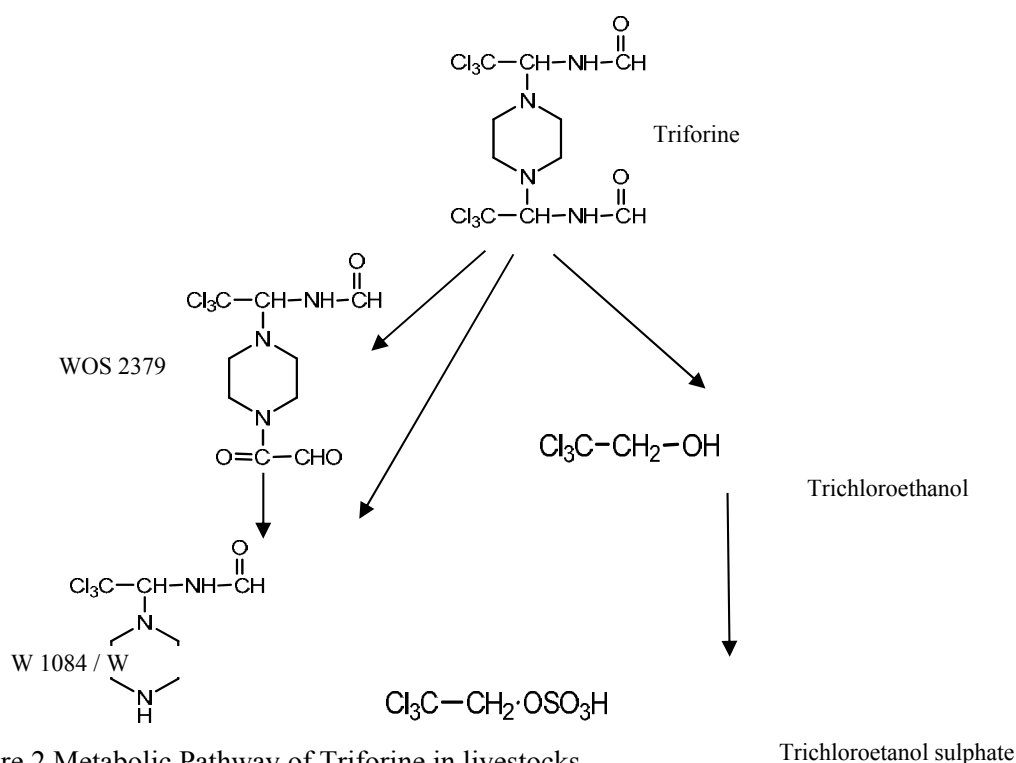


Figure 2 Metabolic Pathway of Triforine in livestock

Plant metabolism

Plant metabolism studies were performed on apples, tomatoes and cucumber with triforine [^{14}C]-labelled in two carbons at the side chain, and on barley with triforine [^3H]-labeled at piperazine ring to track metabolites. Metabolites were identified using multiple chromatographic systems and authentic standards.

Apple

The metabolism of [^{14}C]triforine has been studied in apple fruits and leaves after five successive applications of a commercial formulation of 0.1% [side chain- ^{14}C]-triforine at 8-day intervals (Hawkins *et al*, 1993: TF-640-037). Three 2–3 year old container grown apple trees (variety Lord Lambourne) on semi-dwarf root stock were placed outdoors in a netted enclosure. Samples of [^{14}C]triforine (*ca.* 6 mg) were mixed with 25 μL blank formulation (EC 190 g/L) giving *ca.* 31 mg of formulation. This formulation was diluted with water to give a final concentration of 1.2 g/L. Specific amounts of the formulated [^{14}C]triforine were applied to either apples (100 μL) or leaves (100 μL) using a syringe. The treatment was applied as a series of small droplets at random over the surface of the apple/leaf. Treated apple fruits were harvested 2 weeks after the last of five successive applications. Translocation from treated leaves into untreated apple fruits was also investigated. Some additional apple fruits and leaves were sampled about 2 hours after one application.

The recovery of radioactivity from selected apple fruits and leaves was measured about 2 hours after one application. The remaining apple fruits were taken, along with the leaves and untreated apples, 14 days after the fifth application of [^{14}C]triforine. Surface washes on all treated samples were initiated on the day of sample collection and samples were stored at $< -15^\circ\text{C}$ overnight. The surface of each treated apple or leaf was washed by immersion in acetonitrile for 5 minutes during which time the container was placed in a sonic bath. The surface wash was then separated by decanting and the process repeated with two further acetonitrile washes. The volumes of the surface washes were measured and aliquots (approximately 100 μL) were removed for estimation of radioactivity by LSC. After the surface washes were completed the apple fruits were peeled and the

peel, flesh and leaves were separately homogenized with acetonitrile, sonicated and the samples were centrifuged. A further extraction was performed using acetonitrile: water (70:30).

Table 8 The distribution of radioactive residues in apple fruit and leaves

	2 hours after one application				2 weeks after five applications			
	Fruit		Leaf		Fruit		Leaf	
	%AR	%TRR	%AR	%TRR	%AR	%TRR	%AR	%TRR
Extract	95.4	99.4	57.8	96.6	27.4	84.7	20.4	92.1
Surface wash	94.8	98.8	54.9	91.8	23.8	73.2	14.7	66.7
Peel or leaf	0.4	0.4	2.9	4.8	1.4	4.5	5.7	25.4
Flesh	0.2	0.2	–	–	2.2	7.0	–	–
Unextracted	0.6	0.6	2.1	3.4	4.7	15.3	2.0	7.8
Total	95.8	100	59.9	100	32.2	100	22.3	99.9

After one application of [^{14}C]triforine three fruits and three leaves were taken for analysis. The radioactivity in the surface washes was 98.8% (fruit) and 91.8% (leaf) of TRR. Only small amount of radioactivity were in the remaining extracts of homogenized peel and flesh, 0.4 and 0.2% TRR respectively. Extracts of homogenized leaves contained 4.8% TRR. The mean concentrations in the treated fruits and leaves were 1.59 (fruit) and 92.4 (leaf) mg equiv/kg, respectively.

At harvest, after 5 successive applications of [^{14}C]triforine, 32.2% (fruit) and 22.3% (leaf) of the applied radioactivity was recovered. Acetonitrile surface washes of treated fruits at harvest contained 73.2% TRR. Extracts of peel and flesh homogenized with acetonitrile accounted for 4.5 and 7.0% TRR respectively. The mean concentration of 1.36 mg equiv/kg was recovered in the treated fruits.

At the time of harvest untreated fruits were taken from branches with treated leaves. The radioactivity in untreated fruits accounted for 0.0009 mg equiv/kg. Analysis of untreated fruits showed a very low degree of translocation during this period.

Samples of surface washes and extracts were pooled to give representative samples for both fruits and leaves, and analyzed directly by TLC or HPLC. Radiolabelled metabolites formed in the study were characterized by co-chromatographic comparison using two different systems.

Table 9 Proportion of radioactive components in apple fruits at harvest

	Surface wash		Peel		Flesh		Total	
	Pool 1	Pool 2	Pool 1	Pool 2	Pool 1	Pool 2	Pool 1	Pool 2
	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)
Extract								
Triforine	68 (0.82)	74 (1.14)	2.3 (0.03)	2.5 (0.04)	3.0 (0.04)	2.8 (0.04)	73 (0.88)	79 (1.22)
W 1069	< 0.7 (< 0.008)	< 0.8 (< 0.01)	0.9 (0.01)	0.3 (0.005)	0.9 (0.01)	0.7 (0.01)	1.8 (0.02)	1.0 (0.02)
Unidentified A	< 0.7 (< 0.008)	< 0.8 (< 0.01)	0.3 (0.004)	0.1 (0.002)	0.3 (0.004)	0.2 (0.003)	0.6 (0.007)	0.3 (0.005)
Unidentified B	< 0.7 (< 0.008)	< 0.8 (< 0.01)	0.3 (0.004)	0.2 (0.003)	0.4 (0.005)	0.2 (0.003)	0.7 (0.008)	0.4 (0.006)
Unidentified C	< 0.7 (< 0.008)	< 0.8 (< 0.01)	0.5 (0.006)	0.3 (0.005)	1.3 (0.02)	0.8 (0.01)	1.8 (0.02)	1.1 (0.02)
WOS 613	< 0.7 (< 0.008)	< 0.8 (< 0.01)	0.5 (0.006)	0.2 (0.003)	1.1 (0.01)	0.6 (0.009)	1.6 (0.02)	0.8 (0.01)
W 625	< 0.7 (< 0.008)	< 0.8 (< 0.01)	0.5 (0.006)	0.3 (0.005)	0.9 (0.01)	0.6 (0.009)	1.4 (0.02)	0.9 (0.01)
Other	2 (0.02)	2 (0.03)	0.008 (0.0001)	< 0.04 (< 0.001)	< 0.08 (< 0.001)	< 0.06 (< 0.001)	2 (0.02)	2 (0.03)
Unextracted	–	–	14 (0.17)	12 (0.18)	2.4 (0.03)	1.8 (0.03)	16 (0.19)	14 (0.22)

Note: In order to provide material for duplicate chromatographic runs, surface washes and extracts containing most radioactive components were combined to obtain two pooled samples from different fruits and leaves.

The major component in surface washes and extracts of fruits was identified as triforine by co-chromatography with reference test substances using both normal phase TLC and reverse phase HPLC. Triforine accounted for 73–79% TRR at harvest. The final concentrations of triforine in the fruits at 2 weeks after the final application of triforine were 0.88–1.22 mg/kg.

Several minor components were observed in the extracts and each of these accounted for 1–2% TRR. Two of the identified metabolites, formed by degradation of the piperazine side chain, were the mono and di-formyl compounds W 625 and WOS 613. The third identified metabolite, formed by de-alkylation of one of the piperazine nitrogen, was the amine W 1069. A further three unidentified components A, B and C were also observed that did not co-chromatograph with the available reference compounds. All of these metabolites each accounted for 0.01–0.02 mg equiv/kg.

The nature of the radioactivity unextracted in the peel residue was investigated using some hydrolytic treatments. Apple peel was treated with pectinase enzyme, 0.6 M HCl at 37 °C for 18 hours and 6.0 M HCl at > 90 °C for 1 hour. After adjustment to pH 7 the aqueous layers were extracted with ethyl acetate. The treatments with pectinase and mild acid extracted only small amounts of radioactivity from peel (< 2%) but in these samples the unextracted residue amounted to > 10% recovered radioactivity. On hydrolysis with concentrated acid approximately 7% of the sample radioactivity was extractable. Attempts to chromatograph these extracts were unsuccessful due to the low levels of radioactivity. The radioactivity in the acid peel extract accounted for about 0.1 mg equiv/kg and in the unextracted residue accounted for about 0.09 mg equiv/kg.

Tomato

The metabolism of [¹⁴C]triforine has been studied in tomatoes and leaves after four successive applications of a commercial formulation of 0.1% [side chain-¹⁴C]-triforine at 8–10 days intervals (Hawkins *et al*, 1993: TF-640-038). Tomato plants (variety Moneymaker) were transplanted to 25 cm diameter plastic pots containing potting compost. The study was conducted in a controlled environment room with a daylength of 15 hours and day and night temperatures of 20.9 ± 1.1 °C. Samples of [¹⁴C]triforine (approximately 12 mg) were mixed with 50 µL blank formulation (EC 190 g/L) giving *ca.* 62 mg of formulation. This formulation was diluted with water to give a final concentration of 1.2 g/L. Specific amounts of the formulated [¹⁴C]triforine were applied to either tomatoes (100 µL) or leaves (200 µL) using a syringe. The treatment was applied as a series of small droplets at random over the surface of the tomato/leaf. The treated tomatoes were harvested at 2 hours and 3 days after the last of four successive applications. For comparison additional samples were taken 2 hours after the first application. Translocation from treated leaves into untreated tomatoes was also investigated.

The recovery of radioactivity from each treated tomato leaf was measured separately. Surface washes on all treated samples were collected on the day of sample collection and samples were stored at < –15 °C overnight. The surface of each treated tomato leaf was washed by immersion in acetonitrile for 5 minutes during which time the container was placed in a sonic bath. The surface wash was then separated by decanting, and the process repeated with two further acetonitrile washes. After the surface washes were completed the tomato fruit or leaf was homogenized with acetonitrile, sonicated and the sample centrifuged. The supernatant extract was removed and the process repeated (leaves at harvest were extracted a third time with acetonitrile: water (70:30 v/v)). The volumes of the surface washes and extracts were measured and aliquots (approximately 100 µL) were removed for estimation of radioactive content by LSC. The extracted residues were allowed to air-dry and the radioactive content determined by combustion of sub-samples. A similar extraction procedure was employed for the untreated tomatoes removed at harvest.

Table 10 The distribution of radioactive residues in tomatoes and leaves

	Tomatoes					
	2 hours after one application (n = 3)		2 hours after 4th application (n = 7)		3 days after 4th application (n = 8)	
	%AR	%TRR	%AR	%TRR	%AR	%TRR
Surface wash	93.6	95.6	97.2	91.9	85.1	90.9

	Tomatoes					
	2 hours after one application (n = 3)		2 hours after 4th application (n = 7)		3 days after 4th application (n = 8)	
	%AR	%TRR	%AR	%TRR	%AR	%TRR
Tomato extract	3.6	3.7	5.8	5.8	5.8	6.2
Unextracted	0.7	0.7	2.3	2.3	2.6	2.8
Total	97.9	100	100.2	100	93.5	99.9
	Leaves					
	2 hours after one application (n = 2)			3 days after 4th application (n = 8)		
	%AR	%TRR	%AR	%TRR	%AR	%TRR
Surface wash	93.0	95.1	79.2	86.1		
Leaf extract	1.9	1.9	9.4	10.2		
Unextracted	3.0	3.1	3.5	3.8		
Total	97.8	100.1	92.1	100.1		

Three tomatoes and two leaves were sampled for analysis 2 hours after the first application of [^{14}C]triforine. The radioactivity recovered in the surface washes was 95.6% (tomato) and 95.1% (leaf) TRR. Extracts of the homogenised tomato or leaf contained small amounts of radioactivity (3.7% TRR (tomato) and 1.9% TRR (leaf)). Radioactivity in the unextracted residues accounted for 0.7% TRR (tomato) and 3.1% TRR (leaf). The mean concentrations in whole tomato and leaf were 6.2 and 16.9 mg equiv/kg, respectively.

The initial surface washes of treated tomatoes at harvest contained 91.9% (harvest at 2 hours after the final application) and 90.9% (harvest at 3 days after the final application) of TRR. Acetonitrile extracts of homogenised tomatoes accounted for 5.8% TRR (2 hour harvest) and 6.2% TRR (3 day harvest). After this extraction, 2.3% TRR (2 hour harvest) and 2.8% TRR (3 day harvest) remained unextracted in the tomato residue. The TRR from the treated tomatoes accounted for mean concentrations of 15.6 (2 hour harvest) and 9.7 (3 day harvest) mg equiv/kg.

Leaves were only removed for analysis 3 days after the final application of [^{14}C]triforine. The TRR in these leaves accounted for a mean concentration of 89 mg equiv/kg.

At the time of harvest untreated tomatoes were taken from plants with treated leaves. The initial surface washes of the untreated tomatoes contained 18% TRR with 82% TRR in the remaining tomato flesh extract plus residue. The TRR in untreated tomatoes accounted for a mean concentration of 0.004 mg equiv/kg indicating a very low degree of translocation.

Samples of surface washes and extracts were pooled namely Pool 1 to 2 as in table below to give representative samples for both tomatoes and leaves and analyzed directly by TLC or HPLC. Radiolabelled metabolites formed in the study were characterized by co-chromatographic comparison using two different systems.

Table 11 Proportion of radioactive components in tomatoes at harvest

	2 hours after successive applications					
	Surface wash		Tomato extract		Total	
	Pool 1	Pool 2	Pool 1	Pool 2	Pool 1	Pool 2
	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)
Extract						
Triforine	87.9 (11.6)	89.4 (18.2)	4.56 (0.60)	3.30 (0.67)	92.5 (12.2)	92.7 (18.9)
W 1069	< 0.9 (< 0.12)	< 0.9 (< 0.18)	0.23 (0.030)	0.90 (0.18)	0.23 (0.030)	0.90 (0.18)
Unidentified A	< 0.9 (< 0.12)	< 0.9 (< 0.18)	< 0.06 (< 0.008)	0.24 (0.049)	< 0.09 (< 0.12)	0.24 (0.049)
Unidentified B	< 0.9 (< 0.12)	< 0.9 (< 0.18)	0.17 (0.022)	0.24 (0.049)	0.17 (0.022)	0.24 (0.049)
Unidentified C	< 0.9 (< 0.12)	< 0.9 (< 0.18)	0.29 (0.038)	0.48 (0.098)	0.29 (0.038)	0.48 (0.098)
WOS 613	< 0.9	< 0.9	0.23	0.36	0.23	0.36

	2 hours after successive applications					
	Surface wash		Tomato extract		Total	
	Pool 1	Pool 2	Pool 1	Pool 2	Pool 1	Pool 2
	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)
	(< 0.12)	(< 0.18)	(0.030)	(0.073)	(0.030)	(0.073)
W 625	< 0.9 (< 0.12)	< 0.9 (< 0.18)	0.17 (0.022)	0.24 (0.049)	0.17 (0.022)	0.24 (0.049)
Other	3.7 (0.49)	2.8 (0.57)	0.06 (0.008)	0.30 (0.061)	3.76 (0.50)	3.10 (0.63)
Unextracted	–	–	2.7 (0.36)	1.8 (0.37)	2.7 (0.36)	1.8 (0.37)
	3 days after a successive applications					
	Surface wash		Tomato extract		Total	
	Pool 1	Pool 2	Pool 1	Pool 2	Pool 1	Pool 2
	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)
Extract						
Triforine	86.9 (10.3)	89.5 (7.3)	4.50 (0.53)	2.89 (0.24)	91.4 (10.8)	92.4 (7.58)
W 1069	< 0.9 (< 0.11)	< 0.9 (< 0.07)	1.05 (0.12)	0.78 (0.064)	1.05 (0.12)	0.78 (0.064)
Unidentified A	< 0.9 (< 0.11)	< 0.9 (< 0.07)	0.30 (0.035)	0.20 (0.016)	0.30 (0.035)	0.20 (0.016)
Unidentified B	< 0.9 (< 0.11)	< 0.9 (< 0.07)	0.38 (0.045)	0.20 (0.016)	0.38 (0.045)	0.20 (0.016)
Unidentified C	< 0.9 (< 0.11)	< 0.9 (< 0.07)	0.45 (0.053)	0.29 (0.024)	0.45 (0.053)	0.29 (0.024)
WOS 613	< 0.9 (< 0.11)	< 0.9 (< 0.07)	0.30 (0.035)	0.25 (0.021)	0.30 (0.035)	0.25 (0.021)
W 625	< 0.9 (< 0.11)	< 0.9 (< 0.07)	0.45 (0.053)	0.25 (0.021)	0.45 (0.053)	0.25 (0.021)
Other	2.7 (0.32)	2.8 (0.23)	0.15 (0.018)	0.05 (0.004)	2.85 (0.34)	2.85 (0.23)
Unextracted	–	–	2.8 (0.33)	2.8 (0.23)	2.8 (0.33)	2.8 (0.23)

Note: In order to provide material for duplicate chromatographic runs, surface washes and extracts containing most radioactivity were combined to obtain two pooled samples from different fruits and leaves.

At 2 hours and 3 days after the final application only one major discrete component was observed in surface washes which accounted for 86.9–89.5% TRR. The major component in tomato extracts was also shown to co-chromatograph with triforine and accounted for 2.89–4.56% TRR. This major radioactive component was identified as triforine by co-chromatography with authentic test substance using both normal phase TLC and reverse phase HPLC. Triforine accounted for 91.4–92.7% TRR in in surface washes and extracts of tomatoes taken at 2 hours and 3 days after the final application of ^{14}C -triforine. In addition, W 1069, WOS 613 and W 625 were identified by co-chromatography with reference compounds in both normal phase TLC and reverse phase HPLC. Three further unidentified components A, B and C were observed that did not co-chromatograph with the available reference compounds. The major component present in the tomato at harvest was identified as triforine and accounted for means of 15.6 and 9.19 mg/kg in tomatoes taken 2 hours and 3 days after the final application of triforine respectively. The minor components W 1069, WOS 613, W 625 and each unidentified component (A-C) generally accounted for 0.02–0.1 mg equiv/kg. Radioactivity unextracted from the tomato accounted for 0.2–0.4 mg equiv/kg.

The unidentified radioactive components in the tomato extracts taken 3 days after the final application of [^{14}C]triforine were in the range of 0.02–0.05 mg equiv/kg. In order to provide further information to characterise these unidentified components, their partitioning behaviour was investigated (Hawkins *et al*, 1994: TF-640-040). Samples of the tomato extracts were diluted with water and extracted with ethyl acetate at both pH 2 and pH 9. In addition, further samples were incubated separately with β -glucuronidase/sulphatase and β -glucosidase. These incubates at pH 5

were also extracted with ethyl acetate. The radioactive components in these organic and aqueous layers were quantified by TLC and the partitioning behaviour of each component was calculated.

The unidentified metabolites A and B were shown to be polar water soluble components at pH 2 and 9 and were not hydrolysed with either β -glucuronidase/sulphatase or β -glucosidase. The metabolite C was partitioned between both the organic (ca 80%) and aqueous (ca 20%) layers at pH 2 and 9 but was entirely organo-soluble at pH 5 (after enzyme treatment). Component C was also not hydrolysed with the enzyme treatments.

Cucumber

The metabolism of [^{14}C]triforine has been studied in cucumbers and leaves after four successive applications of a commercial formulation of 0.1% [side chain- ^{14}C]-triforine at 7-day intervals (Hawkins *et al*, 1993: TF-640-039). Cucumber plants (variety Brunex F1) were transplanted to 25 cm diameter plastic plant pots containing potting compost. The study was conducted in a controlled environment room with a daylength of 15 hours and day and night temperatures of $21.8 \pm 0.5^\circ\text{C}$. Samples of [^{14}C]triforine (ca. 6 mg) were mixed with 25 μL blank formulation (EC 190 g/L) giving ca. 31 mg of formulation. This formulation was diluted with water to give a final concentration of 1.2 g/L. Specific amounts of the formulated [^{14}C]triforine were applied to either cucumbers (200 μL first application, 300 μL remaining applications) or leaves (200 μL) using a syringe. The treatment was applied as a series of small droplets at random over the surface of the cucumber/leaf. The treated cucumbers were harvested 3 days after the last of four successive applications. For comparison additional samples were taken 2 hours after the first application.

The recovery of radioactivity from selected sample cucumbers and leaves was measured about 2 hours after the first application. The remaining cucumbers were taken, together with the leaves and untreated cucumbers, 3 days after the fourth application of [^{14}C]triforine. Surface washes on all treated samples were collected on the day of sample collection and samples were stored at $< -15^\circ\text{C}$ overnight. Each treated cucumber or leaf was surface washed by immersion in acetonitrile for 5 minutes during which time the container was placed in a sonic bath. The surface wash was then separated by decanting and the process repeated twice, once with acetonitrile and once with acetonitrile: water (70:30 v/v). After the surface washes were completed the cucumber was peeled and the peel or flesh was homogenized with acetonitrile, sonicated and the sample centrifuged. Leaves removed at harvest were also extracted using this procedure. The supernatant extract was removed and the process repeated once with acetonitrile and once with acetonitrile: water (70:30 v/v). The volumes of the surface washes and extracts were measured and aliquots (ca. 100 μL) removed for estimation of radioactive content by LSC. The extracted residues were allowed to air-dry and the radioactive content determined by combustion of sub-samples. A similar extraction procedure was employed for the untreated cucumbers removed at harvest.

Table 12 The distribution of radioactive residues in cucumbers and leaves

	2 hours after one application (n = 3)				3 days after 4 applications (n = 12)			
	Cucumber		Leaf		Cucumber		Leaf	
	%AR	%TRR	%AR	%TRR	%AR	%TRR	%AR	%TRR
Extract	91.1	98.9	96.6	99.7	76.1	93.4	90.7	99.0
Surface wash	88.2	95.8	95.9	99.0	68.9	84.5	85.5	93.4
Peel or leaf	1.6	1.7	0.7	0.7	6.1	7.5	5.2	5.6
Flesh	1.3	1.4	—	—	1.1	1.4	—	—
Unextracted	1.0	1.1	0.3	0.4	5.5	6.7	1.0	1.1
Total	92.0	100	96.8	100.1	81.6	100.1	91.6	100.1

Three cucumbers and three leaves were sampled for analysis 2 hours after the first application of [^{14}C]triforine. The mean amounts of radioactivity recovered in the surface washes was 95.8% TRR (cucumber) and 99.0% (leaf) TRR. Extracts of the homogenized cucumber peel and flesh contained small amounts of radioactivity (1.7% and 1.4% TRR) and radioactivity extracted from the leaf contained 0.7% TRR. Radioactivity in the unextracted residues accounted for 1.1% TRR

(cucumber) and 0.4% TRR (leaf). The mean concentrations in whole cucumber and leaf were 0.57 and 81.5 mg equiv/kg, respectively.

The initial surface washes of treated cucumbers at harvest accounted for 84.5% TRR. Extracts of homogenised cucumber peel and flesh accounted for 7.5% TRR (peel) and 1.4% TRR (flesh). After these extractions, 6.7% TRR remained unextracted in the residues. The TRR from the treated cucumbers accounted for a mean concentration of 2.17 mg equiv/kg.

The surface washes of treated leaves at harvest contained 93.4% TRR. Acetonitrile extracts of the homogenised leaves contained 5.6% TRR whilst 1.1% TRR remained unextracted in the leaf residue. The TRR in the leaf accounted for a mean concentration of 114 mg equiv/kg.

At the time of harvest untreated cucumbers were taken from plants with treated leaves. The initial surface washes of the untreated cucumbers contained 25% of TRR with 75% in the remaining cucumber flesh extract plus residue. The TRR in untreated cucumbers accounted for a mean concentration of 0.0044 mg equiv/kg indicating a very low degree of translocation.

Table 13 Proportion of radioactive components in cucumbers at harvest

	Surface wash		Peel		Flesh		Total	
	Pool 1	Pool 2	Pool 1	Pool 2	Pool 1	Pool 2	Pool 1	Pool 2
	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)	%TRR (mg/kg)
Extract								
Triforine	84 (1.81)	81 (1.77)	3.3 (0.07)	5.6 (0.12)	0.24 (0.005)	0.34 (0.007)	87.5 (1.9)	86.9 (1.9)
W 1069	< 0.9 (< 0.02)	< 0.8 (< 0.02)	0.72 (0.02)	0.68 (0.01)	0.11 (0.002)	0.11 (0.002)	0.83 (0.02)	0.79 (0.02)
Unidentified A	< 0.9 (< 0.02)	< 0.8 (< 0.02)	1.6 (0.03)	1.6 (0.04)	0.13 (0.003)	0.08 (0.002)	1.73 (0.04)	1.68 (0.04)
Unidentified B	< 0.9 (< 0.02)	< 0.8 (< 0.02)	0.13 (0.003)	0.09 (0.002)	0.15 (0.003)	0.15 (0.003)	0.28 (0.006)	0.24 (0.005)
Unidentified C	< 0.9 (< 0.02)	< 0.8 (< 0.02)	0.20 (0.004)	0.26 (0.006)	0.15 (0.003)	0.10 (0.002)	0.35 (0.008)	0.36 (0.008)
WOS 613	< 0.9 (< 0.02)	< 0.8 (< 0.02)	0.13 (0.003)	< 0.09 (< 0.002)	0.21 (0.005)	0.27 (0.006)	0.34 (0.007)	0.27 (0.006)
W 625	< 0.9 (< 0.02)	< 0.8 (< 0.02)	0.26 (0.006)	0.26 (0.006)	0.20 (0.004)	0.18 (0.004)	0.46 (0.010)	0.44 (0.010)
Other	2.6 (0.06)	1.7 (0.04)	0.20 (0.004)	< 0.09 (< 0.002)	0.20 (0.004)	0.18 (0.004)	3.0 (0.06)	1.88 (0.04)
Unextracted	–	–	5.4 (0.12)	6.6 (0.14)	0.6 (0.01)	0.8 (0.02)	6.0 (0.13)	7.4 (0.16)

Note: In order to provide material for duplicate chromatographic runs, surface washes and extracts containing most radioactive components were combined to obtain two pooled samples from different fruits and leaves.

Three days after the final application only one major discrete component was observed in surface washes which accounted for 81–84% TRR. The major component in cucumber extracts was also shown to co-chromatograph with triforine and accounted for 3.3–5.6% TRR (peel) and 0.24–0.34% TRR (flesh). The major radioactive component was identified as triforine by co-chromatography with authentic test substance using both normal phase TLC and reverse phase HPLC. The total radioactivity associated with triforine in surface washes and extracts accounted for 86.9–87.5% TRR in cucumber taken 3 days after the final application of [^{14}C]triforine.

The extracts also contained several minor components each accounting for 0.3–2% TRR. Three of these were identified as the compounds W 1069, WOS 613 and W 625 by co-chromatography with reference compounds in both normal phase TLC and reverse phase HPLC. Three further unidentified components A, B and C were observed that did not co-chromatograph with the available reference compounds.

The major component present in the cucumber at harvest was identified as triforine and accounted for 1.9 mg/kg. The minor components W 1069, WOS 613, W 625 and unidentified

components A-C each accounted for 0.005–0.04 mg equiv/kg. Radioactivity unextracted from the cucumber accounted for 0.13–0.16 mg equiv/kg.

The unidentified radioactive components in the cucumber extracts taken 3 days after the final application of [^{14}C]triforine were in the range of 0.002–0.04 mg equiv/kg. In order to provide further information to characterise these unidentified components, their partitioning behaviour was investigated (Hawkins *et al*, 1994: TF-640-041). Samples of the cucumber extracts were diluted with water and extracted with ethyl acetate at both pH 2 and pH 9. In addition, further samples were incubated separately with β -glucuronidase/sulphatase and β -glucosidase. These incubates at pH 5 were also extracted with ethyl acetate. The radioactive components in these organic and aqueous layers were quantified by TLC and the partitioning behaviour of each component calculated.

The unidentified A and B were shown to be polar water-soluble components at pH 2 and 9 and were not hydrolysed with either β -glucuronidase/sulphatase or β -glucosidase. The unidentified C was partitioned between both the organic (40–80%) and aqueous (60–20%) layers at pH 2 and 9 but was entirely organo-soluble at pH 5 (after enzyme treatment).

Barley

Study 1

The metabolism of [^3H]triforine in barley plants has been studied (Rouchaud *et al*, 1977: TF-905-013). Barley plants (variety Hebe) were grown (33 plants/pot) in plastic pots (13 cm diameter), containing a mixture of sand and expanded perlite (1/1, v/v). Unlabelled triforine (30 mg/pot) and an equal weight of adjuvant powder were emulsified in water (30 mL/pot) and [^3H]triforine dissolved in methanol was added to the emulsion to give the preparation used for soil drenching. Three leaves were harvested at 15 and 30 days after treatment, immediately weighed and then homogenised (6 min, 4 °C) with ethanol. Each homogenate (3 mL) was filtered on glass filters, the solid residue washed on the filter with ethanol to remove any unbound label, dried under an infrared lamp and ^3H determined with a LSC.

The harvested leaves were frozen in plastic bags with an ethanol-solid CO_2 mixture and stored (-20 °C). The leaves (50 g), warmed up to room temperature, were homogenised with chloroform (300 mL). The supernatant was centrifuged and filtered, giving the primary chloroform extract and a solid residue. The primary chloroform extract was transferred to a separatory funnel and extracted with aqueous HCl, giving the secondary chloroform extract. The secondary chloroform extract was filtered through potassium carbonate and concentrated in a rotary vacuum evaporator. The hydrochloric acid extract was brought to pH 7 with aqueous 2.0 M and 0.1 M NaOH, and freeze-dried. The powder was put on a chromatographic column and eluted with the chloroform which had been used to rinse the dishes used in the freeze-drying. In some experiments, the hydrochloric acid extract was brought to pH 4 with aqueous 2.0 M and 0.1 M NaOH and freeze-dried. The identification of the radioactive TLC spots was made by comparison of their R_f values with those of standard compounds, using four TLC systems for each metabolite.

Each concentrate of the secondary chloroform and acid extracts was analysed by TLC using all four eluting solvents. The compositions of the two extracts, as shown by TLC, were different and no labelled compound was common to both. The secondary chloroform extract contained mostly triforine. In the hydrochloric acid extract, a metabolite was observed for which the chemical structure W 1084 was suggested. Another labelled compound in this extract was probably piperazine, the concentration of which increased with time after treatment. The amounts of piperazine observed were similar in freeze-dried pH 4 or pH 7 extracts. Triforine was found only in the secondary chloroform extract, and W 1084 and piperazine were present only in the 0.1 M HCl extract. None of these compounds was transformed into the others during the analytical process.

Table 14 Total percentage distribution of radioactivity in the leaves of barley plants treated with [^3H]triforine

	%TRR	
	15 days after treatment	30 days after treatment
Extract	76.9	61.8
Secondary chloroform extract	61.6	46.4
Triforine	57.5	43.2
Unidentified	4.1	3.2
0.1 M HCl extract	15.3	15.4
W 1084	12.9	8.4
Piperazine	0.3	4.0
Unidentified	2.1	3.0
Unextracted	23.1	38.2

Study 2

The leaves of barley plants root-treated with [^3H]-triforine (uniformly labelled in the piperazine ring) were analysed 30 days after treatment (Rouchaud *et al*, 1978: TF-640-008). The distribution of the methanol-soluble ^3H -constituents was similar to that of the chloroform soluble (Study 1): 45% triforine, 10% W 1084, 5% piperazine. Methanol extraction left a solid plant residue which contained 33% of the total ^3H which had been incorporated into the leaves. Methanol acidified with hydrochloric acid extracted a further 18% of the triforine-derived bound residues as W 1084 (8%) and piperazine (10%). In the plants, these compounds had thus been complexed to plant constituents.

Hot dimethyl sulphoxide (DMSO) extracted a further 13% of the total ^3H , leaving a solid residue (mainly cellulose) which contained 2% of ^3H , perhaps incorporated into the cellulose. Evaporation of the solvent from the DMSO extract gave a solid substance with the radioactivity (13%), which could not be extracted by methanol. A part (7%) of this radioactivity could be released by successive hydrolysis with aminoglucosidase and β -glucosidase, which generated a complex mixture of polar and water soluble unknown radioactive compounds not including piperazine. These latter compounds would be the products of extensive metabolism of triforine (and its metabolite piperazine) bound to, or incorporated into starch. Most (11%) of the radioactivity of this solid could be released by hydrochloric acid hydrolysis, which also generated a complex mixture of polar and water soluble unknown radioactive compounds not including piperazine; a part (4%) of them could have been associated with lignin in the plant.

Study 3

The metabolism of the [^3H]-triforine, uniformly labelled in the piperazine ring, has been studied in barley (Rouchaud *et al*, 1978: TF-640-010). Barley (variety Hebe) was sown and grown normally in an experimental field. At growth stage J (during the stem extension stage when the second node of the stem was formed and the next-to-last leaf was just visible), the aerial part of the plants was sprayed with an aqueous emulsion of a mixture of the commercial formulation of triforine and [^3H]-triforine. The total dose of 0.25 kg ai/ha was that of agronomic practice. Barley was harvested when ripe, and straw and grain were analysed separately.

The straw and grains were washed rapidly and successively with water and methanol, the washing liquids being discarded as they did not contain significant amount of radioactivity. Extraction with methanol was followed by centrifugation. The first extract was macerated for 72 hours at 20 °C (300 mL to 100 g straw/grains). Three sequential extractions (300 mL methanol) were performed and the methanol extracts were combined. The straw was chopped and the grain was ground for extraction. This process was repeated with a further four samples of grain (100 g each) to provide sufficient material for metabolite identification. The extracts were partitioned with chloroform against dilute HCl. The metabolites in both phases were separated by column chromatography and characterized by TLC. Metabolites were identified by cleaning up by a preparative TLC followed by derivatisation and confirmation by co-chromatography with derivatized reference compounds.

The total radioactivity concentration was 20 times higher in straw than in grain. In straw and grain respectively, 12 and 25% of the total incorporated radioactivity in each of these tissues were methanol soluble; this corresponded to the higher content of cellulosic and lignified tissues in straw than in grain, and the correspondingly higher solubilisation of the radioactive residues. The amount of methanol soluble radioactivity was 14 times higher in straw than in grain.

The methanol soluble radioactive residue contained the triforine and its metabolites which were free and unbound in barley straw and grain. No radioactive piperazine was observed, in spite of the high detection sensitivity for radioactivity. Triforine was identified and accounted for 0.034 mg/kg (18% TRR) in straw and 0.0018 mg/kg (13% TRR) in grain. W 1084 was identified at 0.009 mg/kg (7% TRR) in straw and 0.0006 mg/kg (7% TRR) in grain. Two other radiolabelled components were identified: Glycine was found at 0.043 mg/kg (33% TRR) in straw and 0.0033 mg/kg (34% TRR) in grain. Iminodiacetic acid was also identified at 0.021 mg/kg (17% TRR) in straw and 0.001 mg/kg (11% TRR) in grain.

Extraction of the grain with methanol left methanol-insoluble solids containing an amount of radioactivity (the bound residue) which represented 75% of the total radioactivity incorporated into the grain. Methanol acidified with hydrochloric acid extracted a further 7% TRR of the triforine-derived bound residues in the form of radioactive iminodiacetic acid (1.1% TRR), glycine (3.3% TRR), serine (0.9% TRR), ethanolamine (0.2% TRR) and unidentified compounds (1.5% TRR). Aqueous 0.03 M NaOH extracted a further 27% of the total tritium which had been incorporated by means of chemical bonds into the protein fraction; acid hydrolysis of the proteins yielded radioactive glycine (9.2% TRR), serine (3.9% TRR) and unidentified compounds (13.9% TRR) which could have been a mixture of a large number of other amino acids. The plant solids (which contained 41% of the total tritium) left after the alkaline aqueous extractions were processed and separated into tritiated cellulose (4% TRR) and starch (37% TRR) fractions. The starch was hydrolysed and the resulting glucose was converted into the osazone (34% TRR). After being recrystallized several times, the osazone contained a constant specific radioactivity, indicating that [^3H]-glucose was present. No piperazine was observed in the bound residues in the grain. (Rouchaud *et al*, 1979: TF-905-020)

Extraction of the straw with methanol left methanol-insoluble solids containing an amount of radioactivity (the unextracted residue) which represented 88% of ^3H (as with all the subsequently referred % of ^3H relative to the total of ^3H incorporated into the straw). Methanol acidified with hydrochloric acid extracted a further 8% TRR of the triforine-derived bound residues as radioactive iminodiacetic acid (0.4% TRR), glycine (3.2% TRR), serine (2.0% TRR), ethanolamine (0.2% TRR) and unidentified compounds (2.2% TRR). A neutral detergent solution extracted a further 3% of the radioactive triforine-derived bound residues as unidentified compounds; three radioactive compounds, which were dissolved by the acidified methanol and by the neutral detergent solution, were thus in the straw complexed to straw constituents. An acid detergent solution extracted a further 58% of the total ^3H , which was really incorporated by means of chemical bonds into the hemicelluloses fraction; the acid hydrolysis of the hemicelluloses yielded a mixture of monosaccharides which were derivatised into a mixture of osazones (50% TRR). The plant solids (which contained 19% of the total ^3H), remained after the acid detergent extraction, were processed and separated into the ^3H containing cellulose (13% TRR) and lignin (6% TRR) fractions. No piperazine was observed in the bound residues in the grain. (Rouchaud *et al*, 1979: TF-640-014)

Summary of plant metabolism

Triforine was the major component present in the edible portion of the harvested crops. Only small amounts of metabolites were observed and those identified contained the intact piperazine ring. In barley metabolism was much more extensive with more than 90% of the terminal residue in grain and straw being present as natural products either extractable with neutral solvent (monomers) or incorporated into plant polymers and only extractable after acid hydrolysis or digestion of the plant constituents. Triforine, glycine and iminodiacetic acid were present in small amounts in the methanol extracts of grain and straw with triforine predominating in both grain and straw. Triforine and extractable metabolites were all less than 0.01 mg/kg in harvested grain following application at approximately 0.25 kg ai/ha.

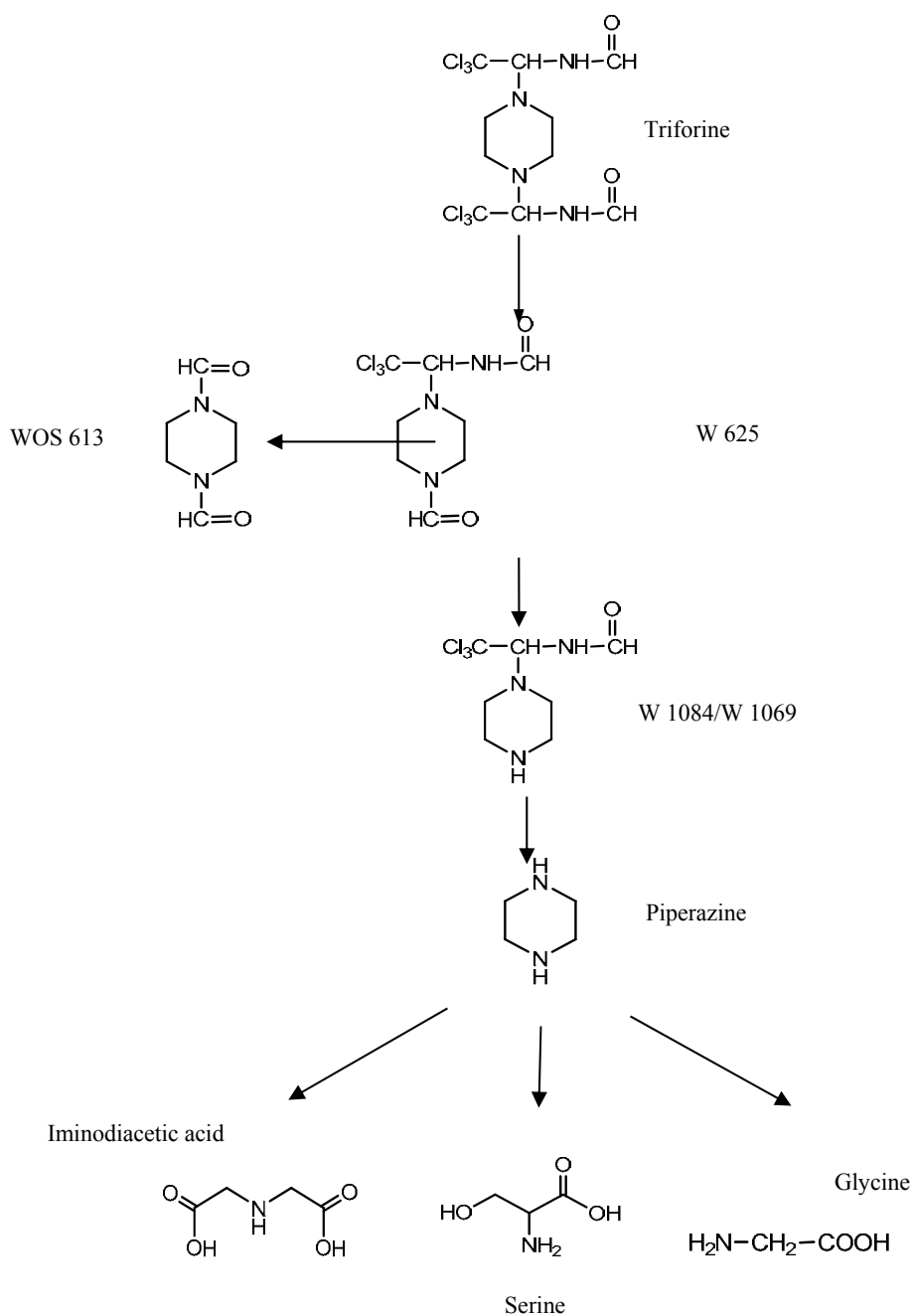


Figure 3 Metabolic pathway of triforine in plants

Environmental fate in soil

The Meeting received information on aerobic degradation in soil, photolysis on soil surface and hydrolytic degradation study. Because triforine is intended for use as foliar treatment, aerobic degradation, soil photolysis and hydrolytic degradation study relevant to the current evaluations are reported below (FAO Manual 2009).

The fate and behaviour of triforine in soils were investigated using [piperazine-¹⁴C] and [side chain-¹⁴C] labelled compounds.

Aerobic degradation

Study 1

The degradation of [piperazine- ^{14}C]-triforine in a silty loam soil and a sandy loam soil when incubated at 20 °C under aerobic and anaerobic conditions has been studied over a period of 184 and 60 days respectively (Rainford, 1990: TF-620-005). Throughout the study evolved CO_2 and organic volatiles were collected from the flasks of soil and aliquots removed for analysis. The soil was extracted using acetone and other solvents, including caustic extracts. The extracts were analyzed for [^{14}C]triforine and its degradation products by TLC. The mean overall recoveries of applied radioactivity for the aerobic and anaerobic incubates containing the sandy loam soil were $88.2 \pm 3.8\%$ and $79.9 \pm 5.7\%$ respectively and for the silty loam soil were $93.7 \pm 4.6\%$ and $92.6 \pm 5.9\%$ respectively. No significant production of $^{14}\text{CO}_2$ occurred before 3 weeks. After 184 days approximately 24% of the applied radioactivity had evolved as $^{14}\text{CO}_2$ from both soils under aerobic conditions. The distribution of radioactivity recovered from the two soil types under aerobic varied slightly. The applied radioactivity in the acetone extracts declined more quickly from the silty loam soil (80% at 0 day, 13% at 28 days and 1% at 184 days) than the sandy loam soil (85% at 0 day, 54% at 28 days and 18% at 184 days). In both soil types an increase in the proportion of applied radioactivity in the caustic extracts occurred reaching a maximum of 36% at 28 days for the silty loam soil and 23% at 42 days for the sandy loam soil. The percentage of applied radioactivity present as bound residues were higher in the silty loam soil (47% at 60 days) compared to that in the sandy loam soil (12% at 42 days).

Degradation half-lives of 35.8 and 69.6 days were determined for triforine in the silty and sandy loam soils respectively under aerobic conditions. After 184 days, approximately 0.35% and 6% of the applied radioactivity remained as triforine in the two soils respectively. The decline in triforine concentrations was accompanied by an increase in bound radioactivity and a large number of very minor degradation products. No one component at any time represented more than 3% of applied radioactivity. Very tentative identification of four degradates was made: W 625, WOS 2379, piperazine and W 1069. Due to the very low levels present there was no possibility of further identification of any of these degradates from samples obtained from this study.

Under aerobic conditions, [^{14}C]triforine is degraded to at least 14 degradates in silty loam and eight degradates in sandy loam when incubated for up to 184 days at 20 °C. No major degradates were produced. The rate of degradation in the silty loam exceeded that in the sandy loam. In both soil types, degradation was accompanied by the evolution of $^{14}\text{CO}_2$, indicating that degradation involved cleavage of the ring structure and bound residues.

Study 2

The aerobic degradation and metabolism of [side chain- ^{14}C]-triforine was investigated in a US soil (sandy loam) under laboratory conditions (20 ± 2 °C, in the dark) for 365 days (Wyss-Benz, 1993: TF-620-033). The test article was applied at a maximum recommended field rate of 0.38 kg ai/ha corresponding to 0.512 mg ai/kg dry soil in the top 5 cm or 0.051 mg ai/100 g soil sample. Duplicate soil samples were applied for 0, 7, 14, 28, 56, 84, 112, 147, 189, 238 and 365 days of incubation. Samples were submitted to several extractions with first acetonitrile, and then acetonitrile/water (1:1). Extracts of acetonitrile were combined, concentrated and submitted to TLC and HPLC analysis.

The mean recoveries from duplicate samples ranged from 91.0% to 106% of the radioactivity applied, with an average mean recovery from all samples of $97.1 \pm 5.6\%$. The radioactivity extracted from the soil decreased from 101% of the radioactivity applied (0.519 mg equiv/kg dry soil) on day 0 to 13.2% (0.067 mg equiv/kg) on day 365. The non-extracted radioactivity increased from 4.6% (0.024 mg equiv/kg) on day 0 to 38.2% (0.196 mg equiv/kg) on day 238, and then decreased slightly until day 365 to 35.0% (0.179 mg equiv/kg). The non-extracted radioactivity bound to the organic matter fractions was compared between day 147 and day 365. On day 147, 58.0% of the non-extracted radioactivity was found to be associated with fulvic acid, 9.9% with humic acids and 24.7% with

humic fraction. On day 365, 10% of the radioactivity proved to be more strongly bound as 32.5% were found in the humic fraction and only 49.8% in the fulvic acid fraction.

Mineralization of triforine took place from the beginning of the study, i.e. on day 7 with 1.3% (0.006 mg equiv/kg) and increased to 44.7% (0.229 mg equiv/kg) on day 365. No volatile compounds other than $^{14}\text{CO}_2$ could be detected. The mineralization was still detectable at the end of the study. The biomass was low at the beginning of the study but increased during the study from 6.0 mg C/100 g dry soils to 18.6 mg C/100 g dry soil on day 365. Most of the radioactivity extracted from the soil was identified as parent. The rate of disappearance was calculated using sampling intervals from day 0 to day 84 only. Half-life (DT_{50}) was determined by the first order reaction kinetics model as 23.9 days with a DT_{90} value of 79.3 days

Many minor degradation products were detected in the extracts during the study. Attempts were made to isolate degradates, however it was very difficult to identify them. One fraction was characterized as reference compound—the hydrochloride salt (W 1069), however the levels were small at a maximum of 0.08 mg/kg on day 56, decreasing until the end of the study to 0.026 mg/kg.

It can be concluded from this study that triforine was rapidly degraded and readily mineralized in the sandy loam soil under the chosen experimental conditions and that many minor degradation products representing < 0.01 mg equiv/kg were formed.

Study 3

The decomposition of triforine in five soils (Speyer 2.2, Riverside, Sion Hill, Middlefield and Silt loam) was studied in laboratory tests (Jones, 1989: TF-620-011). Treated soils were incubated aerobically at $20 \pm 3^\circ\text{C}$ in the dark. The concentration of triforine in each soil was determined at intervals of 0, 2, 8, 16, 32, 64, 100, 147 and 164 days after treatment.

Soil samples were extracted into acetone/chloroform (3:7). The extract was cleaned up by solid phase extraction chromatography using C_{18} bond elute columns. The final extract was then quantified by reverse phase HPLC using a variable wavelength UV absorbance detector. The limit of detection of the HPLC system was 0.1 mg/L of triforine. The microbial biomass was determined by monitoring the $^{14}\text{CO}_2$ evolution from duplicate samples of known masses of test soils fortified with [^{14}C]glucose.

The mean percent nominal recoveries of triforine from the fortified control soil samples ranged between 77 and 85% respectively. There was a significant reduction in the measured concentration of triforine. The DT_{50} values for the five soils ranged between 1 and 7 days.

Table 15 The DT_{50} and DT_{90} values for the five soils

Soil type	50% Degradation time	90% Degradation time
Speyer 2.2 (loamy sand)	7 days	116 days
Riverside (clay loam)	7 days	69 days
Sion Hill (sandy loam)	7 days	78 days
Middlefield (silty clay)	1 day	3 days
Silt loam (silt loam)	4 days	55 days

Soil photolysis

The artificial sunlight photodegradation of [piperazine- ^{14}C]triforine was studied on sandy loam soil. The artificial sunlight source was a xenon lamp that had a special energy distribution similar to that of natural sunlight (Saxena, 1990: TF-620-008). Soil samples in petri dishes were prepared and fortified with [^{14}C]triforine at concentration of 10 mg ai/kg. Soils samples were placed in a xenon lamp chamber and irradiated continuously (24 hours/day) at a temperature of 22.4 to 27.2 $^\circ\text{C}$. Dark control samples were placed in a glass chamber and maintained in the dark at 25 $^\circ\text{C}$. Traps for volatile components were connected to the irradiation and dark control chambers. In addition, a charcoal trap was connected to the irradiation chamber. Two soil samples were analyzed at hour 0, and then duplicated irradiated soil samples were removed for analysis after 1, 2, 4, 8, 10, 24, 30 and 48 hours

of irradiation. Samples were extracted with methanol: water (9:1). The extracts as well as the extracted soil (oxidized by combustion) were analyzed by liquid scintillation counting. The distribution of radioactivity in the methanol: water extract was determined by thin layer chromatography (TLC). The TLC plates were analyzed using a linear analyzer.

The recovery of applied radioactivity for the irradiated and dark control soil samples ranged from 92.2% to 104% and from 91.0% to 104%, respectively. The applied radioactivity remaining in the methanol: water extracts ranged from 97.9% to 72.1% for the irradiated samples and from 101.2% to 83.4% for the dark control samples. The applied radioactivity remaining in the methanol: water extracted soil ranged from a mean of 25.2% to 1.9% at all study intervals for the irradiated samples and from a mean of 1.9% to 18% for the dark control samples. The cumulative radioactivity in the traps was 0.5% or less at all study intervals for both test conditions.

Analysis of the methanol: water extracts of the study samples using TLC system showed that the [^{14}C]triforine degraded rapidly on soil when irradiated with a xenon lamp. The degradation was biphasic. The calculated degradation half-life of [^{14}C]triforine was 11.4 hours of artificial sunlight, equivalent to 0.5 natural sunlight days for phase 1 (hours 0 to 8). For phase 2 (hours 8 to 48), the half-life was 70.9 hours of artificial sunlight equivalent to 3.16 natural sunlight days. Radiolabelled triforine degraded under dark conditions, with a half-life of 76.5 hours. For the irradiated samples, [^{14}C]triforine was observed at all intervals by TLC analysis. Several peaks were observed: one had similar R_f value to that of non-radiolabelled standard W 1069. The maximum was 9.9% at hour 48. For the dark control samples, [^{14}C]triforine was observed at all intervals by TLC analysis. Several peaks were observed, the maximum was 10.3% at hour 24.

RESIDUE ANALYSIS

Analytical methods

Descriptions of analytical methods together with validation data for residues of triforine in plant and animal matrices were submitted to the Meeting. The methods rely on an initial extraction, usually with acetone. After solvent partition, the triforine and metabolites residues are degraded by heating with sulphuric acid. The residues of triforine and metabolites containing piperazine ring can be measured by the analysis of degradate (chloral hydrate) with GC-ECD, typically to an LOQ of 0.01 mg/kg.

Detailed descriptions of all these analytical methods are presented below.

Plant matrices

Cereals (green plants, straw, grain), fruit, vegetables, meat, milk, soil, water (102FX-522-009, TF-240-002)

Analyte: Triforine GC-ECD RU 3,26/12/10

LOD: 0.005–0.01 mg/kg

Description The samples are blended with acetone. After removal of the acetone by distillation the triforine in the remaining aqueous phase is partitioned into toluene. The toluene is evaporated and the active substance was degraded by heating with dilute sulphuric acid. Chloral hydrate thus formed is distilled, extracted with ethyl formate and determined by gas chromatography with an electron capture detector.

Plant and animal products (102AA-522-008, TF-244-006)

Analyte: W 1084 GC-ECD

LOD: 0.01 mg/kg

Description The samples are blended with acetone. After removal of the acetone by distillation the triforine is partitioned into toluene. The remaining aqueous phase contains W 1084, which is the main metabolite of triforine especially in animals. The aqueous solution is heated with dilute sulphuric acid. Chloral hydrate formed is distilled, extracted with ethyl formate and determined by gas chromatography with an electron capture detector.

Chicory, carrot, red beet, Brussels sprout, white cabbage (102AX-522-015, TF-244-010)

Analyte: Triforine GC-ECD
 LOQ: 0.01 mg/kg
 Description: The samples are extracted with acetone. After filtration and evaporation of acetone, triforine is partitioned from the remaining aqueous solution into dichloromethane. The solvent is evaporated and the active substance degraded by heating with dilute sulphuric acid. Chloroform that is formed is determined by head-space gas chromatography with an electron capture detector.

Cherry, peach, plum, prune (TF-244-011)

Analyte: Triforine, W 1084, 2,2,2-trichloroethanol, WOS 2379, W 625 GC-ECD FAMS 041-01
 LOQ: 0.01 mg/kg
 Description: The homogenised samples are diluted with water and sulfuric acid is added. Triforine, W 1084, WOS 2379 and W 625 are degraded by heating. Chloral hydrate and 2,2,2-trichloroethanol are separated by distillation, extracted with ethyl formate and determined by capillary gas chromatography with an electron capture detector.

Green peppers, tomatoes, eggplant, Japanese persimmon (SAI No.135)

Analyte: Triforine LC-MS
 LOQ: 0.01 mg/kg
 Description: The samples are homogenised with acetone. The sample solution is purified with a graphite carbon mini-column, a C₁₈ mini-column and a silica gel mini-column. The elution is evaporated and dissolved with methanol. The residue is determined by LC-MS.

Blueberry and tomato (2040W)

Analyte: Triforine LC-MS/MS
 (m/z 435→390 for quantification, 435→215 for confirmation)
 LOQ: 0.01 mg/kg for blueberries and tomatoes, 0.05 mg/kg for tomato paste
 Description: The samples are extracted with acetone. The extract is subjected to further clean-up involving three SPE cartridge (ENVI-Carb, C₁₈ and Silica gel) clean-ups. The final samples are analysed by LC-MS/MS for quantitation of triforine.

The recoveries from plant matrices obtained during method validation are summarized in Table 16.

Table 16 Summary of recovery data for triforine and its metabolites fortified into plant matrices

Commodity		Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference Method
Cucumber	Triforine	0.20	3	83–119	100	18.3	102AX-522-014, TF-244-008
Apple	Triforine	0.02 0.20	3 8	77–91 71–117	82 89	9.5 17.0	102AX-522-013, TF-244-009
Cherry	Triforine	0.01–0.1	4		91	6	FAMS 041-01, TF-244-011
	W 1084	0.02–0.1	3		76	2	
	Trichloroethanol	0.01–0.1	8		73	10	
	WOS 2379	0.01–0.1	4		92	12	
	W 625	0.01–0.1	4		103	13	

Commodity		Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference Method
Peach	Triforine	0.01–0.1	4		82	6	
	W 1084	0.01–0.1	4		93	13	
	Trichloroethanol	0.01–0.1	8		66	19	
	WOS 2379	0.01–0.1	4		86	21	
	W 625	0.01–0.1	4		89	15	
Plum	Triforine	0.01–0.1	4		82	16	
	W 1084	0.01–0.1	5		92	11	
	Trichloroethanol	0.01–0.1	5		73	11	
	WOS 2379	0.01–0.1	4		108	7	
	W 625	0.01–0.1	4		92	7	
prune	Triforine	0.01–0.1	4		87	9	
	W 1084	0.01–0.1	4		92	12	
	Trichloroethanol	0.01–0.1	4		78	11	
	WOS 2379	0.01–0.1	4		85	21	
	W 625	0.01–0.1	4		81	9	
Cucumber	Triforine	0.01	2	78, 98	88		CFS 1994-105, TF-244-012
		0.10	2	80, 88	84		
		1.0	2	86, 95	91		
	W 1084	0.01	3	70–98	83	17.0	
		0.10	2	91, 93	92		
		1.0	2	76, 103	90		
	Trichloroethanol	0.01	2	65, 72	69		
		0.10	2	79, 88	84		
		1.0	2	97, 97	97		
	WOS 2379	0.01	2	103, 105	104		
		0.10	2	77, 78	78		
		1.0	4	64–71	68	4.5	
	W 625	0.01	2	87, 97	92		
		0.10	2	85, 89	87		
		1.0	2	71, 75	73		
Green pepper	Monitoring ion 391.7	0.01	3	101–113	107	5.6	SAI No.135
		1	3	92–97	95	2.8	
Japanese persimmon	Monitoring ion 391.7	0.01	3	96–105	101	4.5	
		1	3	91–96	94	2.8	
Eggplant	Monitoring ion 391.7	0.01	3	73–85	78	8.2	
		1	3	82–91	87	5.2	
Tomato	Monitoring ion 391.7	0.01	3	101–116	109	1.6	2040W
		1	3	96–99	97	6.9	
Blueberry	Mass transition 435s tr	0.01	5	73–75	74	1	
		0.10	5	71–88	76	9	
	Mass transition 435→215	0.01	5	68–74	71	3	
		0.10	5	68–85	75	9	
Tomato	Mass transition 435s tr	0.01	5	88–103	95	6	
		0.10	5	81–99	89	8	
	Mass transition 435→215	0.01	5	58–92	74	19	
		0.10	5	82–97	90	7	
Tomato paste	Mass transition 435s tr	0.05	5	71–94	79	11	
		0.10	5	69–77	74	4	
	Mass transition 435→215	0.05	5	72–91	79	9	
		0.10	5	72–83	77	5	

Animal matrices

Animal product (102FX-523-001, TF-245-001)

Analyte: Triforine, W 1084, W 2379

GC-ECD

LOD: 0.001 mg/kg for milk and 0.003 mg/kg for the other materials (muscle, liver, kidney and fat)

Description The samples are blended with acetone. The organic solvent is removed by distillation. Triforine and possible metabolites containing the $\text{Cl}_3\text{C-CH}$ group (W 1084, W 2379) are degraded by heating with dilute sulphuric acid. Chloral hydrate formed is distilled, extracted with ethyl formate and determined by gas chromatography with an ECD.

Milk (TF-245-002)

Analyte: Triforine, W 1084, Trichloroethanol GC-ECD FAMS 037-01

LOQ: 0.001 mg/kg for triforine and trichloroethanol, 0.002 mg/kg for W 1084

Description The sample is diluted with water and sulphuric acid is added. Triforine and W 1084 are degraded by heating. Chloral hydrate formed and 2,2,2-trichloroethanol are separated by distillation, extracted with ethyl formate and determined by capillary gas chromatography with an ECD.

Bovine tissue (kidney, liver, muscle), fat and cream (TF-245-003)

Analyte: Triforine, W 1084, Trichloroethanol GC-ECD FAMS 038-01

LOQ: 0.01 mg/kg

Description The homogenised samples are diluted with water and sulphuric acid is added. Triforine and W 1084 are degraded by heating. Chloral hydrate formed and 2,2,2-trichloroethanol are separated by distillation, extracted with ethyl formate and determined by capillary gas chromatography with an ECD.

Egg (TF-245-005)

Analyte: Triforine, W 1084, Trichloroethanol GC-ECD FAMS 084-01

LOQ: 0.01 mg/kg

Description The homogenised samples are diluted with water and sulphuric acid is added. Triforine and W 1084 are cleaved upon heating. Chloral hydrate formed and 2,2,2-trichloroethanol are separated by distillation, extracted with ethyl formate and determined by capillary gas chromatography with an ECD.

The recoveries from animal matrices obtained during method validation are summarized in Table 17.

Table 17 Summary of recovery data for triforine and its metabolites fortified into animal matrices

Commodity		Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference
Milk	Triforine	0.001–0.1	5		75	15	FAMS 037-01 TF-245-002
	W 1084	0.002–0.1	4		63	4	
	Trichloroethanol	0.001–0.1	10		86	8	
Bovine kidney	Triforine	0.01–0.1	4		68	4	FAMS 038-01 TF-245-003
	W 1084	0.01–0.1	4		70	5	
	Trichloroethanol	0.01–0.1	4		89	6	
Bovine liver	Triforine	0.01–0.1	4		68	8	
	W 1084	0.01–0.1	4		68	5	
	Trichloroethanol	0.01–0.1	4		95	10	
Bovine muscle	Triforine	0.01–0.1	4		70	3	
	W 1084	0.01–0.1	5		81	10	
	Trichloroethanol	0.01–0.1	4		92	4	
Peritoneal fat	Triforine	0.01–0.1	4		78	3	
	W 1084	0.01–0.1	4		91	9	
	Trichloroethanol	0.01–0.1	4		90	3	
Subcutaneous fat	Triforine	0.01–0.1	4		76	4	
	W 1084	0.01–0.1	4		75	78	
	Trichloroethanol	0.01–0.1	4		88	7	

Commodity		Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference
Cream	Triforine	0.01–0.1	4		73	12	
	W 1084	0.01–0.1	4		88	7	
	Trichloroethanol	0.01–0.1	4		88	4	
Egg	Triforine	0.01–0.1	8		90	6	FAMS 084-01 TF-245-005
	W 1084	0.01–0.1	8		93	8	
	Trichloroethanol	0.01–0.1	8		88	6	
Egg	Triforine	0.01	3	71–76	73	4.0	FAMS 084-01 TF-245-004
		0.10	3	75–78	76	2.0	
	W 1084	0.01	3	83–89	86	3.5	
		0.10	3	95–101	98	3.1	
	Trichloroethanol	0.01	3	90–95	92	2.9	
		0.10	3	94–98	95	2.4	
Milk	Triforine	0.001	3	92–101	96	4.9	FAMS 037-01 TF-245-006
		0.01	3	97–103	100	3.0	
	W 1084	0.002	3	102–116	111	6.8	
		0.02	3	106–115	111	4.1	
	Trichloroethanol	0.001	3	97–107	103	5.0	
		0.01	3	89–92	91	1.7	
Liver	Triforine	0.01	3	100–116	107	7.8	FAMS 038-01 TF-245-006
		0.1	3	93–114	106	10.9	
	W 1084	0.01	3	66–77	70	9.1	
		0.1	3	97–117	107	9.3	
	Trichloroethanol	0.01	3	69–77	74	5.9	
		0.1	3	65–66	66	0.88	
Kidney	Triforine	0.01	3	77–82	80	3.2	
		0.1	3	78–94	84	10.4	
	W 1084	0.01	3	88–96	93	4.9	
		0.1	3	117–127	121	4.6	
	Trichloroethanol	0.01	3	86–97	90	6.8	
		0.1	3	77–83	80	3.8	
Muscle	Triforine	0.01	3	80–85	83	3.2	
		0.1	3	72–107	88	20.3	
	W 1084	0.01	3	108–135	123	11.1	
		0.1	3	107–111	109	1.8	
	Trichloroethanol	0.01	3	82–88	85	3.6	
		0.1	3	74–78	76	2.8	
Fat	Triforine	0.01	3	118–123	121	2.1	
		0.1	3	85–101	93	8.6	
	W 1084	0.01	3	113–134	121	9.4	
		0.1	3	100–104	102	2.0	
	Trichloroethanol	0.01	3	97–102	100	2.5	
		0.1	3	96–100	98	2.0	

Stability of pesticide residues in stored analytical samples

The Meeting received data on the stability of triforine residues in apples, cherries, plums, peaches, blueberries and hops (cones and processed fractions) samples stored frozen.

The freezer stability study of triforine was conducted on apples, cherries, peaches and blueberries (Eich, 1979: TF-326-010). Untreated samples of blueberries, cherries, apples and peaches were chopped and fortified with triforine at 0.1 or 1 mg/kg respectively. The homogenised samples were stored deep frozen (-20 °C) for different time periods up to 12 months. Samples were analyzed after storage using GC-ECD method RU 3, 26/12/10.

Table 18 Recovery of triforine from stored fortified samples

Storage interval	Residue (mg/kg)	
	0.1 mg/kg fortification	1.0 mg/kg fortification
Apples		
0 month	0.10	0.92

Storage interval	Residue (mg/kg)	
	0.1 mg/kg fortification	1.0 mg/kg fortification
2 months	0.09	0.88
4 months	0.09	0.88
12 months	0.10	0.83
Cherries		
0 month	0.10	0.93
2 months	0.11	0.97
4 months	0.11	0.95
12 months	0.11	0.95
Peaches		
0 month	0.09	1.04
2 months	0.09	0.91
4 months	0.10	0.99
12 months	0.12	1.06
Blueberries		
0 month	0.09	0.84
2 months	0.08	0.83
4 months	0.10	0.78
12 months	0.11	0.92

Means values of two replicates, corrected by procedural recovery.

The storage stability of triforine was investigated under frozen conditions (Weeren, 1994: TF-326-025). Samples of hops and the processed fractions were fortified with triforine at a level of up to 100 mg/kg. Samples were stored in a freezer at -18 °C and analyzed using analytical method RU 3, 26/12/10 after 1, 15, 30, 59, 96, and 180 days of storage.

Table 19 Recovery (%) of triforine from stored fortified samples of hops and processed fractions

Storage interval	Green cones						
	0.5 mg/kg fortification		5 mg/kg fortification			50 mg/kg fortification	
	% remaining	Mean	Procedural	% remaining	Mean	% remaining	Mean
1 day	79, 82	81	90, 93	81, 84	83	96, 97	97
93 days	72, 77	75	86, 89	70, 71	71	76, 80	78
175 days	67, 74	71	73, 88	66, 67	67	67, 75	71
Storage interval	Dried cones						
	10 mg/kg fortification				100 mg/kg fortification		
	Procedural	% remaining		Mean	% remaining		Mean
1 day	90, 91	82, 95		89	76, 85		81
96 days	70, 82	68, 73		71	66, 74		70
181 days	78, 82	68, 70		69	68, 71		70
Storage interval	Spent hops [0.5 mg/kg fortification]						
	Procedural		% remaining			Mean	
1 day	85, 88		79, 79			79	
15 days	77, 83		80, 83			82	
30 days	82, 86		75, 78			77	
59 days	92, 93		73, 74			74	
Storage interval	Beer [0.5 mg/kg fortification]						
	Procedural		% remaining			Mean	
1 day	97, 99		81, 86			84	
15 days	79, 88		75, 77			76	
28 days	81, 95		83, 92			88	
Storage interval	Yeast [0.5 mg/kg fortification]						
	Procedural		% remaining			Mean	
1 day	84, 93		77, 88			83	
15 days	93, 102		64, 66			65	
28 days	77, 80		62, 65			64	

Table 20 Recovery of triforine from stored fortified samples of plums

Table 20 Recovery of triforine from stored fortified samples of plums

Storage interval	Pitted fruits [1.0 mg/kg fortification]		
	Procedural	% remaining	Mean
8 months	82	75, 81, 87	81

Triforine is registered in many countries for the control of various fungal diseases on fruits, fruiting vegetables, legumes, tree nuts, etc. The Meeting received labels in the countries of North and South America, Africa, Asia and Oceania. The information available to Meeting on registered uses of triforine is summarized in Table 21 below.

Table 21 Registered uses of triforine on crops

[illegible]

Crop	Country	F or G	Formulation		Application				Application rate per treatment			PHI, days
			Type	Conc. of ai g/L	Method	Max number	Timing	Interval	Max spray conc g ai/hL	Water volume L/ha	Max rate g ai/ha	
Stone fruits excluding plums (Brownrot)	New Zealand	F	EC	190	Foliar spray	3	1st: Pink fall 3 rd : shuck fall	7–10 days	19	2000 (at least)	–	1
	New Zealand	–	EC	190	Dip fruit for 30 seconds.	NS	Post-harvest	NS	19	–	–	1
Stone fruits excluding plums (Rust)	New Zealand	F	EC	190	Foliar spray	3	1 st : First sign of infection	10–14 day	19	–	–	1
Peach, nectarine, apricot, plum, prune	Australia	F	EC	190	Foliar spray	3	3 rd : 1 week before harvest	14 days	19	–	–	7
	Australia	–	EC	190	Dip for 30 seconds. Renew the dip after 48 hours.	NS	Post-harvest	NS	19	–	–	–
Peach, Cherry, Plum, Prune	Canada	F	EC	190	Foliar spray	3	1 st : Early bloom stage 3 rd : full bloom stage	NS	14 –	– –	– 475	–
Cherry, Nectarine, Peach, Plum	Chile	F	EC	190	Foliar spray	3	1 st : Balloon 3 rd : 100% bloom	NS	19	–	285	14
Apricot	Israel	F	EC	190	Foliar spray	NS	NS	NS	19	1000–2000	380	14
Cherry	Australia	–	EC	190	Dip for 30 seconds. Renew the dip after 48 hours.	4	Post-harvest	NS	14.3	–	–	–
Nectarine	South Africa	F	EC	190	Foliar spray	NS	NS	NS	19	2500–3500	665	3
Peach	Argentina	F	EC	190	Foliar spray	2	1 st : Appearance of the flowers 2 nd : fruits with 1 cm in diameter	NS	23.7	–	–	14
Peach	Brazil	F	EC	190	Foliar spray	3	1 st : First sign of infection	7–10 days	23.7	(400–1000) ^e	(237)	3
Peach	Japan	F	EC	180	Foliar spray	5	NS	NS	18	2000–7000	–	1
Peach	Mexico	F	EC	190	Foliar spray	NS	NS	7 days	28.5	100	–	14
Peach	South Africa	F	EC	190	Foliar spray	2	NS	13–15 days	19	2500–3500	665	3
Plum	Mexico	F	EC	190	Foliar spray	NS	NS	7 days	28.5	100	–	14
Plum and Prune	South Africa	F	EC	190	Foliar spray	2	3 days before harvest	7 days	24.7	2500–3500	865	3
Berries												
Blueberry	Canada (except Eastern Canada)	F	EC	190	Foliar spray	4	1 st : Bud break 4 th : 10–14 days after early bloom	10–14 days	–	1000	570	60

Crop	Country	F or G	Formulation		Application				Application rate per treatment			PHI, days
			Type	Conc. of ai g/L	Method	Max number	Timing	Interval	Max spray conc g ai/hL	Water volume L/ha	Max rate g ai/ha	
	Canada (Eastern Canada only)	F	EC	190	Foliar spray	3	1 st : leaf-bud break 3 rd : pink-bud bloom	10–14 days	–	1000	570	60
Cranberry	Canada (B.C. only)	F	EC	190	Foliar spray	3	1 st : Bud break	10–14 days	–	1000–1500	570	60
Currants (Red and black)	New Zealand	F	EC	190	Foliar spray	3	1 st : Green tip 3 rd : 10% flowering	10–14 days	38	2000–4000	–	14
Grape	Argentina	F	EC	190	Foliar spray	3	1 st : 15 to 20 cm long, 2 nd : in bloom 3 rd : 30 days after the second application	NS	23.8	–	–	14
Grape	Chile	F	EC	190	Foliar spray	3	1 st : First sign of infection	NS	11.4	–	–	14
Grape	Kazakhstan	F	EC	190	Foliar spray	NS	1 st : First sign of infection	NS	–	200–400	285	30
Grape	Kenya	F	EC	190	Foliar spray	NS	NS	NS	–	400–600	285	–
Grape	New Zealand	F	EC	190	Foliar spray	4	1 st : Bud burst	2–3 weeks	19	2000	380 (at least)	14
Grape	Tunisia	F	EC	190	Foliar spray	NS	NS	NS	23.8	–	–	–
Raspberry	Chile	F	EC	190	Foliar spray	3	1 st : Bud break 3 rd : flowering	14 days	–	–	285	14
Strawberry	Brazil	F	EC	190	Foliar spray	4	NS	7 days	28.5	800	–	2
Strawberry	Chile	F	EC	190	Foliar spray	3	1 st : First sign of infection	NS	–	–	285	1
Strawberry	Japan	F	EC	180	Foliar spray	5	NS	NS	9	1000–3000	–	1
Strawberry	Mexico	F	EC	190	Foliar spray	4	NS	4, 7 or 10 days	–	–	190	14
Strawberry	New Zealand	F	EC	190	Foliar spray	NS	NS	7–10 days	38	2000 (at least)	–	14
Strawberry	South Korea	F	EC	190	Foliar spray	3	1 st : First sign of infection	7 days	19	–	–	5
Fruiting vegetable, cucurbits												
Cucurbits (cucumber, marrow, melons etc.)	Kenya	F	EC	190	Foliar spray	NS	1 st : First sign of infection	7–10 days	–	400–600	285	7
Cucurbits	New Zealand	F	EC	190	Foliar spray	2	1 st : First sign of infection	10 days	28.5	1500 (at least)	–	3
Cucurbits	South Africa	F	EC	190	Foliar spray	NS	1 st : First sign of infection	7–10 day	19–28.5	–	190 (at least)	1
Cucumber Melon Squash	Israel	F	EC	190	Foliar spray	NS	NS	NS	–	250–500	152	3

Crop	Country	F or G	Formulation		Application				Application rate per treatment			PHI, days
			Type	Conc. of ai g/L	Method	Max number	Timing	Interval	Max spray conc g ai/hL	Water volume L/ha	Max rate g ai/ha	
Cucumber/melons/watermelons	Mexico	F	EC	190	Foliar spray	3	NS	7 days	—	—	285	7
Cucumber	Algeria	F	EC	190	Foliar spray	NS	NS	NS	28.5	—	—	7
Cucumber	Chile	F	EC	190	Foliar spray	4	1 st . First sign of infection	NS	—	—	285	5
Cucumber	Japan	F	EC	180	Foliar spray	5	NS	NS	18	1000–3000	—	1
Cucumber	Kasakhstan	F	EC	190	Foliar spray	NS	1 st . First sign of infection	NS	—	200–400	190	20
Cucumber	South Korea	F	EC	190	Foliar spray	2	1 st . First sign of infection	10 days	19	—	—	2
Melon	Brazil	F	EC	190	Foliar spray	3	1 st . Early symptoms appearance	7–10 days	23.7	400–1000	—	5
Melon	Chile	F	EC	190	Foliar spray	4	1 st . First sign of infection	NS	—	—	285	2
Melon	Japan	F	EC	180	Foliar spray	6	NS	NS	9	1000–3000	—	1
Pumpkin	Argentina	F	EC	190	Foliar spray	NS	1 st . First sign of infection	1–2 weeks	28.5	—	—	14
Pumpkin	Mexico	F	EC	190	Foliar spray	3	NS	7 days	—	—	285	7
Squash	Chile	F	EC	190	Foliar spray	4	1 st . First sign of infection		—	—	285	8
Watermelon	Chile	F	EC	190	Foliar spray	4	1 st . First sign of infection		—	—	285	—
Fruiting vegetable other than cucurbits												
Eggplant	Israel	F	EC	190	Foliar spray	NS	NS	NS	—	250–500	190	3
Eggplant	Japan	F	EC	180	Foliar spray	5	NS	NS	18	1000–3000	—	1
Eggplant	Kenya	F	EC	190	Foliar spray	NS	NS	NS	—	400–600	285	14
Eggplant	Mexico	F	EC	190	Foliar spray	3	NS	5 or 8 days	—	—	285	15
Pepper	Israel	F	EC	190	Foliar spray	NS	NS	NS	—	250–500	190	3
Pepper	Japan	F	EC	180	Foliar spray	3	NS	NS	18	1000–3000	—	14
Pepper	Kenya	F	EC	190	Foliar spray	NS	NS	NS	—	400–600	285	14
Pepper	Mexico	F	EC	190	Foliar spray	3	NS	7 days	—	—	285	14
Red Pepper (including bell pepper)	South Korea	F	EC	190	Foliar spray	2	1 st . First sign of infection	10 days	19	—	—	7
Tomato	Chile	F	EC	190	Foliar spray	4	1 st . First sign of infection	NS	—	—	285	3
		F	EC	190	Foliar spray (under plastic)	4	1 st . First sign of infection	15 days	28.5	—	—	3
Tomato	Israel	F	EC	190	Foliar spray	NS	NS	NS	—	250–500	190	3
Tomato	Japan	F	EC	180	Foliar spray	3	NS	NS	18	1000–3000	—	1

Crop	Country	F or G	Formulation		Application				Application rate per treatment			PHI, days
			Type	Conc. of ai g/L	Method	Max number	Timing	Interval	Max spray conc g ai/hL	Water volume L/ha	Max rate g ai/ha	
Tomato	Kenya	F	EC	190	Foliar spray	NS	NS	NS	–	400– 600	285	2
Tomato	Mexico	F	EC	190	Foliar spray	4	1 st . First sign of infection	7 days	–	–	380	3
Tomato	New Zealand	F	EC	190	Foliar spray	NS	NS	10–14 days	28.5	at least 1500	–	3
Legume vegetables												
Beans	Argentina	F	EC	190	Foliar spray	2	2 nd . 60 days after planting	15–30 days	38	–	–	8
Beans French bean	Brazil	F	EC	190	Foliar spray	3	1 st . Early symptoms	7–10 days	28.7	400– 1000	290	10
Beans	Chile	F	EC	190	Foliar spray	4	1 st . First sign of infection	NS	–	–	285	10
Beans, green	South Africa	F	EC	190	Foliar spray	NS	1 st . First sign of infection	7–10 day	28.5	1000	–	3
Lupin	South Africa	F	EC	190	Foliar spray	NS	1 st . First sign of infection	2–3 weeks	28.5	500	143	7
		F	EC	190	Aerial spray	NS	NS	NS	–	–	143	7
Peas, shelled	Japan	F	EC	180	Foliar spray	3	NS	NS	12.7	1000– 3000	–	1
Peas	South Africa	F	EC	190	Foliar spray	NS	NS	7–10 day	28.5	1000	285	4
		F	EC	190	Aerial spray	NS	NS	NS	–	40 (at least)	285	4
Cereals												
Cereals	Tunisia	F	–	190	Foliar spray	NS	NS	NS	19	–	–	–

^a NS: not shown

^b May be applied 2 weeks after petal fall (mid-summer) if necessary.

^c Apply maximum six times in seasons of extreme disease pressure.

^d Use the indicated doses in mL/100 L of water to dilute applications (Python). In concentrated sprays, doses are per hectare should be used. Using lower doses under normal pressure conditions. In case of severe attacks or extreme conditions, using a high dose and smaller range.

^e Apply the sufficient to wet the plant with the mixture until it drops.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on triforine supervised field trials for the following crops.

Group	Commodity	Table
Pome fruits	Apple	Table 22–25
	Pear	Table 26
Stone fruits	Cherry	Table 27, 28
	Plum (including Prune)	Table 29–31
	Apricot	Table 32, 33
	Nectarine	Table 34
	Peach	Table 35–39
Berries and other small fruits	Raspberry	Table 40
	Blueberry	Table 41
	Black currant	Table 42
	Grape	Table 43–45

Group	Commodity	Table
Fruiting vegetables, Cucurbits	Cranberry	Table 46
	Strawberry	Table 47–49
	Cucumber	Table 50, 51
	Squash	Table 52, 53
	Melon	Table 54–56
Fruiting vegetables, other than cucurbits	Peppers	Table 57–59
	Egg plant	Table 60, 61
	Tomato	Table 62–64
	Common Bean	Table 65, 66
Legume vegetables		
Cereal grains	Barley	Table 67
	Wheat	Table 68, 69
Straw, fodder and forage of cereals	Barley straw and forage	Table 70
	Wheat straw and forage	Table 71

Triforine formulation was applied for foliar treatment. Each of the field trial sites generally consisted of untreated control plot and treated plot. Application rates and spray concentrations have generally been rounded to two significant figures.

Residue values from the trials, which have been used for the estimation of maximum residue levels, STMRs and HRs, are underlined.

Laboratory reports included method validation with procedural recoveries from spiking at residue levels similar to those occurring in samples from the supervised trials. Date of analyses and duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables except when residues were found in samples from control plots. Residue data are not corrected for percent recovery.

Conditions of the supervised residue trials were generally well reported in detailed field reports. Most field reports provided data on the sprayers used, plot size, field sample size and sampling date.

Pome fruits

Apple

Three residue trials in apples were conducted in the USA. Triforine was quantified by GC-ECD with a LOQ of 0.004 mg/kg.

Three residue trials in apples were conducted in the USA. Method RU3, 26/12/10 was used to analyze apple fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.006 mg/kg.

One residue trial in apples was conducted in Canada. Triforine was quantified by GC-ECD with a LOQ of 0.005 mg/kg.

Three residue trials in apples were conducted in Australia. Method RU3, 26/12/10 was used to analyze apple fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.05 mg/kg.

One residue trial in apples was conducted in Germany. Method RU3, 26/12/10 was used to analyze apple fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.01 mg/kg.

Seven residue trials in apples were conducted in France. Method RU3, 26/12/10 was used to analyze apple fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.01 mg/kg.

Two residue trials in apples were conducted in Brazil. Method RU3, 26/12/10 was used to analyze apple fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.02 mg/kg.

Table 22 Triforine residues on apples from supervised trials in the USA and Canada

Apple, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
GAP, Canada	EC 190	–	–	0.475	5	–		Timing: 1 st :tight cluster 5 th : petal fall stages
USA, 1978 Highland/NY (Rome, Cortland, Golden Delicious, McIntosh, Delicious)	EC 200	0.009	–	–	11	0 ^c	0.37 0.47 0.63 0.39 0.59	102FX-532-2010 ^{a, b} TF-711-010 Sampling to analysis: 202 days
USA, 1978 Kingston/RI (Red Delicious, McIntosh, Golden Delicious)	EC 200	0.009	–	–	7	0 ^c	0.016 0.047 0.083	102FX-532-2011 ^{a, b} TF-711-011 Sampling to analysis: 333 days
USA, 1980 Fabius/NY (McIntosh, IDA Red, Golden Delicious)	EC 200	0.009	–	–	4	21 ^d	0.030, 0.009 0.010, 0.013 0.035, 0.016	102FX-532-2012 ^{a, b} TF-711-012 Sampling to analysis: 145–161 days
						37	0.008	
					10	105 ^d	< 0.005, < 0.006 < 0.005, < 0.006 < 0.005, < 0.006	
						121	< 0.006	
Canada, 1984 Trenton/Ontario (McIntosh)	EC 190	0.015	–	–	5	87	0.019	102FX-532-2018 ^{a, b} TF-711-016 Only information on the date of the last application (7 th June) is available. Sampling to analysis: 38 days
		–	–	0.40	4	103	0.041	
USA, 1977 Kingston/RI (Mixed Varieties)	EC 200	0.015	–	0.56	6	0 7 14 21	0.09 0.09 0.03 0.04	102FX-532-2019 ^a TF-711-017 Sampling to analysis: 272–368 days
USA, 1977 Jackson Springs/NC (Golden Delicious)	EC 200	0.015	–	0.56	5	0 7 14 21	0.41 0.34 0.19 0.14	
USA, 1977 Franklin/MI (Red Delicious)	EC 200	0.015	–	0.56	5	14 28	0.41 0.10	

Analytical portion: fruit

^a The detail of in-field study was not shown in the study report.

^b The detail of analytical method was not shown in the study report.

^c Each residue data for each variety was provided.

^d Duplicate residue data for each variety were provided.

Table 23 Triforine residues on apples from supervised trials in Australia

Apple, country, year (variety)	Application						DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS	no.			
GAP, Australia	EC 190	0.023	—	—		4	1		Interval: 10–14 days
Australia, 1975 Gladysdale (Granny Smith)	EC 190	—	—	—		control		0.06	TF-711-039 ^a Sampling to analysis: 285–399 days
		—	—	0.188	Semi mature	10	107	0.08	
		—	—	0.250	Semi mature	10	107	0.10, 0.13 mean 0.12	
Australia, 1975 Moorooduc (Jonathan)	EC 190	—	—	—		control		0.05	
		—	—	0.125	Semi mature	8	80	< 0.05, < 0.05, 0.05 mean 0.05	
		—	—	0.188	Semi mature	8	80	0.13	
		—	—	0.250	Semi mature	8	80	0.06, 0.10, 0.16 mean 0.11	
		—	—	—	—	—	—	—	
Australia, 1975 Merrindale (Jonathan)	EC 190	—	—	0.125	Semi mature	1	0	0.18, 0.30 mean 0.24	
							3	0.16, 0.19 mean 0.18	
							7	0.38, 0.53 mean 0.46	
							14	c: 0.17 0.12, 0.23 mean 0.18 c: 0.06	
		—	—	0.250	Semi mature	1	0	0.33, 0.40 mean 0.37	
							3	0.32, 0.33 mean 0.33	
							7	< 0.05, 0.22 mean 0.14	
							14	c: 0.17 < 0.05, 0.11 mean 0.08 c: 0.06	
		—	—	0.500	Semi mature	1	0	0.90, 1.0 mean 0.95	
							3	0.76, 0.79 mean 0.78	
							7	< 0.05, 0.22 mean 0.14	
							14	c: 0.17 0.25, 0.36 mean 0.31 c: 0.06	

Analytical portion: fruit

^a The detail of the in-field study was not shown in the study report.

Table 24 Triforine residues on apples from supervised trials in Germany and France

Apple, country, year (variety)	Application						DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS	no.			
Germany, 1987 — (Golden Delicious)	EC 200	0.025	2500	0.625	—	4	105	0.02	102FX-532-2037 ^a TF-711-035 Sampling to analysis: - days Mean recovery 66.6%

Apple, country, year (variety)	Application						DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS	no .			
France, 1990 Rhône (Golden Delicious)	EC 190	0.040	950	0.380	From stage F to J (3.5 to 6 cm)	9	0	0.27	BETR.91.012 ^b TF-711-040 Sampling to analysis: 387–465 days
							1	0.19	
							2	0.10	
							4	< 0.01	
							6	< 0.01	
							8	< 0.01	
							10	< 0.01	
							11	< 0.01	
France, 1990 Les Valentous (Golden Delicious)	EC 190	0.100	380	0.380	From first stage in the formulation of the fruit to 6.5 cm	12	0	0.26	
							1	0.17	
							2	0.08	
							4	< 0.01	
							6	< 0.01	
							8	< 0.01	
							10	< 0.01	
France, 1992 Saint Epain (Smoothee)	EC 190	0.086	330	0.285	–	1	2	0.12	BETR.93.009 ²⁾ TF-711-041 Sampling to analysis: 82–93 days
	EC 190	0.086	330	0.285	–	2	2	0.10	
France, 1995 Fargues st Hilaire (Golden smoothee)	EC 190	–	500	0.333	Maturity	1	3	0.09	CFS 1995-039 TF-711-042 Sampling to analysis: 7–119 days
							7	0.07	
							15	0.09	
							29	0.06	
							59	0.04	
							122	0.04	
							175	0.06	
France, 1995 Brissac-Quince, (Jonagold)	EC 190	–	300	0.333	Maturity	1	3	0.02	
							10	< 0.01	
							17	0.03	
							34	0.02	
							63	0.02	
							125	0.01	
							182	0.02	
France, 1995 Brissac-Quince, (Golden Delicious)	EC 190	–	333	0.333	Maturity	1	3	0.14	
							10	0.07	
							18	0.06	
							34	0.03	
							63	0.03	
							129	0.08	
							186	0.06	
France, 1995 LesChiclets, Molleges (Granny Smith)	EC 190	–	1200	0.333	Maturity	1	3	0.25	
							10	0.27	
							18	0.17	
							32	0.19	
							63	0.12	
							116	0.11	
							183	0.14	

Analytical portion: fruit

^a The detail of in-field study was not shown in the study report.^b The detail of analytical method was not shown in the study report.

Table 25 Triforine residues on apples from supervised trials in Brazil

Apple, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
GAP, Brazil	EC 190	0.024	—	—	3	5		Timing: 1 st First sign infection Interval: 7–10 days
Brazil 1995 SanJose (Fuji)	EC 190	0.024	—	—	3	5	< 0.02	BASF 1995/306189 Sampling to analysis—days
		0.048	—	—	3	5	0.02	
Brazil 1995 Vermelha (Fuji)	EC 190	0.024	—	—	3	5	< 0.02	BASF 1995/306190 Sampling to analysis—days
		0.048	—	—	3	5	< 0.02	

Analytical portion: fruit

Pear

One residue trial in pears was conducted in Australia. Method RU3, 26/12/10 was used to analyze pear fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.05 mg/kg.

Table 26 Triforine residues on pears from supervised trials in Australia

Pear, country, year (variety)	Application						DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS	no.			
Australia, 1975 Merrindale (William bon chretien)	EC 190	—	—	—		control		0.05	TF-711-039 a Sampling to analysis: 285–399 days
		—	—	0.125	Semi mature	1	0	0.75	
							3	0.42	
							7	0.47	
							14	0.40	
		—	—	0.250	Semi mature	1	0	0.13	
							3	0.78	
							7	0.73	
							14	0.31	
		—	—	0.500	Semi mature	1	0	0.68	
							3	1.85	
							7	1.63	
							14	0.70	

Analytical portion: fruit

^a The details of in-field study were not shown in the study report.*Stone fruits**Cherry*

Three residue trials in cherries were conducted in Canada. Method RU3, 26/12/10 was used to analyze cherry fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.007 mg/kg.

Seven residue trials in cherries were conducted in the USA. Method RU3, 26/12/10 was used to analyze cherry fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.001–0.01 mg/kg.

Ten residue trials in cherries were conducted in Germany. Method RU3, 26/12/10 was used to analyze cherry fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.01 mg/kg.

Table 27 Triforine residues on cherries from supervised trials in USA and Canada

Cherry, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
GAP, Canada	EC 190	0.014 —	— —	— 0.475	3 3	— —		Timing: 1 st : Early bloom stage 3 rd : full bloom stage
USA, 1980 Alton/NY (Montmorency)	EC 200	0.013	500– 1000	—	5	0 7	0.99 1.1	102FX-532-2210 ^{a, b} TF-712-010
		0.018	500– 1000	—	5	0 7 9	1.0 0.38 0.09	Sampling to analysis: 138–145 days Recovery = 60 % (n = 1; 1.0 ppm)
USA, 1978 Corvallis/OR (Montmorency, Black Rep., Corum, Royal Ann)	EC 200	0.018	—	—	3 5	83 6 8	0.007 1.2, 1.2 mean 1.2 1.8	102FX-532-2211 ^a TF-712-011
	WP 500	0.030	—	—	3 5	83 6 8	0.017 1.9, 1.9 mean 1.9 2.4	Sampling to analysis: 269–290 days
	F 800	0.030	—	—	3 5	83 6 8	0.005 5.5, 3.5 mean 4.5 3.4	
USA, 1980 Linden/CA (Sweet)	EC 182	0.018	—	—	2	4	1.5, 1.5 mean 1.5	102FX-532-2212 ^b TF-712-012
	EC 182 + WP 500	0.018 + 0.030 Post-harvest	—	—	1 + 1	(4)+0	1.8, 1.7 mean 1.8	Sampling to analysis: 147 days
Canada, 1981 Osoyoos/British Columbia (Lambert)	EC 190	—	1000	0.475	4	1 3 7	1.1, 1.5 mean 1.3 1.8, 1.4 mean 1.6 1.2, 0.95 mean 1.1	102FX-532-2213 ^{a, b} TF-712-013 Sampling to analysis: 307–313 days
Canada, 1981 Cedar Springs/ Ontario (Hedelfingen, Windsor, Van, Stella)	EC 190	—	1000	0.475	5	3	0.71	
USA, 1977 Davis, CA (Bing)	EC 190	0.018	—	—	3	0 3 7 14	1.9 1.1 1.1 0.88	102FX-532-2215 TF-712-015
USA, 1977 Corvallis/OR (Montmorency, Royal Ann)						0 3 7 14 0 3	1.6 0.59 0.41, 1.5 mean 0.96 0.15, 0.29 mean 0.22 1.3 0.85	Sampling to analysis: 307–343 days
		USA, 1977 Alton, Sodus, Wayne, NY (Montmorency)	6 7 7 7	2.1 1.5 1.2 0.78				
			USA, 1977 East Lansing/MI	5	0 3 7	0.94 0.66 0.54		

Cherry, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
(Montmorency)						14	0.15	
Canada, 1983 Osoyoos, British Columbia (Van)	EC 190	—	—	0.235	3	1 3 7	1.3 1.2 1.0	10212-532-2217 ^a TF-712-017 Sampling to analysis: 307–343 days
		—	—	0.475	3	1 3 7	3.3 2.6 1.3	
		—	—	0.950	3	1 3 7	3.9 4.0 2.4	

Analytical portion: fruit Analytical portion: fruit

^a The detail of the in-field study was not shown in the study report

^b The detail of analytical method was not shown in the study report

Table 28 Triforine residues on cherries from supervised trials in Germany

Cherry, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
Germany, 1989 Ingelheim (Schattenmorelle)	EC 190	—	1500	0.428	5	0	0.79	10249-532-2222 ^a TF-712-070 Sampling to analysis: 143–157 days
						3	0.36	
						7	0.18	
						10	0.084	
						14	0.12	
Germany, 1989 Öhringen (Schattenmorelle)	EC 190	—	1500	0.428	5	0	1.7, 2.2 mean 2.0	10249-532-2223, 2226 ^a TF-712-071, 074 Sampling to analysis: 325–345 days
						3	0.56, 0.57 mean 0.57	
						7	0.23, 0.26 mean 0.25	
						10	0.053, 0.079 mean 0.066	
						14	0.046, 0.058 mean 0.052 c: 0.01–0.113	
Germany, 1989 Altenhain/Taunus (Schattenmorelle)	EC 190	—	1500	0.428	5	0	0.787	10249-532-2224 ^a TF-712-072 Sampling to analysis: 318–332 days
						3	1.230	
						7	0.746	
						10	0.913	
						14	0.135	
Germany, 1989 Ingelheim (Schattenmorelle)	EC 190	—	1500	0.428	5	0	0.636	10249-532-2225 ^a TF-712-073 Sampling to analysis: 318–339 days
						3	0.357	
						7	0.165	
						10	0.072	
						14	0.060	
Germany, 1990 Ffm (Schattenmorelle)	EC 190	—	1500	0.428	7	0	0.92	10249-532-2228 ^a TF-712-075 Sampling to analysis: 21–35 days
						3	0.88	
						7	0.42	
						10	0.43	
						14	0.11	
Germany, 1990 Maintal (Schattenmorelle)	EC 190	—	1500	0.428	7	0	0.90	10249-532-2229 ^a TF-712-076 Sampling to analysis: 216–230 days
						3	0.53	
						7	0.19	
						10	0.10	
						14	0.09	

Cherry, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no .			
					7	0 3 7 10 14	1.2 1.2 0.42 0.39 0.24 c: 0.07–0.44	10249-532-2230 ^a TF-712-077 Sampling to analysis: 216–230 days
Germany, 1989 Altenhain (Schattenmorelle)	EC 190	–	1500	0.428	7	0 3 7 10 14	1.2 0.47 0.39 0.49 < 0.005	10249-532-2231 ^a TF-712-078 Sampling to analysis: 17–30 days
Germany, 1990 Ingelheim (Schattenmorelle)	EC 190	–	1500	0.428	7	0 3 7 10 14	0.68 0.60 0.30 0.17 0.14	10249-532-2232 ^a TF-712-079 Sampling to analysis: 25–39 days
Germany, 1990 Öhringen (Schattenmorelle)	EC 190	–	1500	0.428	5	0 3 7 10 14	0.54 0.42 0.13 0.27 0.07 c: 0.39–1.16	10249-532-2233 ^a TF-712-080 Sampling to analysis: 223–237 days
Germany, 1992 – (Schattenmorelle)	EC 190	0.015	1500	0.225	5	0 3 7 10 14 0 3 7 10 14	0.63, 0.68, 0.53 mean 0.61 0.47, 0.60, 0.42 mean 0.50 0.39, 0.30, 0.13 mean 0.27 0.49, 0.17, 0.26 mean 0.31 < 0.01, 0.14, 0.07 mean 0.07 0.75, 0.81, 0.64 mean 0.73 0.55, 0.71, 0.50 mean 0.59 0.46, 0.36, 0.15 mean 0.32 0.58, 0.21, 0.31 mean 0.37 < 0.01, 0.17, 0.08 mean 0.09	10249-532-2234 ^a TF-712-090 Sampling to analysis: 33–47 days

Analytical portion: fruit

^a The details of the in-field study were not shown in the study report.

Plum

Three residue trials in plums were conducted in Canada. Method RU3, 26/12/10 was used to analyze plum fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.07 mg/kg.

Six residue trials in plums were conducted in USA. Method RU3, 26/12/10 was used to analyze plum fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.01 mg/kg.

Six residue trials in plums were conducted in Germany. Methods RCC Prj. No275793 and RU3, 26/12/10 were used to analyze plum fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.01 mg/kg.

One residue trial was conducted in France. Triforine was quantified by GC-ECD with a LOQ of 0.005 mg/kg.

One residue trial was conducted in South Africa. Triforine was quantified by GC-ECD with a LOQ of 0.1 mg/kg.

Table 29 Triforine residues on plums from supervised trials in USA and Canada

Plum, country, year (variety)	Application						Commodity	DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS	no.				
GAP, Canada	EC 190	0.014 –	– –	– 0.475	– –	3 3	Fruit	–		Timing: 1 st : Early bloom stage 3 rd : full bloom stage
USA, 1977 Parlier/CA (Casselman)	EC 200	0.018	–	–	–	3	Fruit	0 5 11	0.43 0.29 < 0.01	TF-712-061 ^a Sampling to analysis: 282–307 days
USA, 1977 Weatherford/TX (Ozark Premier)		0.018	–	–	–	2	Fruit	0 0 0	0.58 0.46 0.49 mean 0.51	
USA, 1977 Davis/CA (French)	EC 200	0.018	–	–	–	3	Fruit Dried (Prune)	0 10	2.0 0.91	102FX-538-002 ^a TF-712-066 Sampling to analysis: 239–262 days
USA, 1977 Corvallis/OR (Early Italian)	EC 200	0.018	–	–	–	1	Fruit	0 3 7 14	0.34 0.39 0.17 0.15	
USA, 1977 Alton, sodus, Wayne/NY (Fehlenburg)	EC 200	0.018	–	–	–	10	Fruit	0 3 8 14	1.6 1.3 1.2 1.2	
USA, 1980 Parlier /CA (Casselman)	EC 200	0.018	–	–	–	2	Fruit	4	0.75, 1.7 mean 1.2	
	EC 200 + WP 500	0.018 + 0.033 Post-harvest	–	–	–	2 + 1	Fruit	(4)+0	1.2, 1.3 mean 1.3	102FX-532-2212 ^{a, b} TF-712-012 Sampling to analysis: 77 days
Canada, 1981 Osoyoos/British Columbia (Santa Rosa)	EC 190	–	1000	0.475	–	4	Fruit	1 3 7	1.3, 1.0 mean 1.2 0.63, 0.64 mean 0.64 0.71, 0.42 mean 0.57	
Canada, 1981 Cedar Springs/ Ontario (Early Italian)	EC 190	–	1000	0.475	–	4	Prune	1 3 7	1.1, 0.93 mean 1.0 0.99, 1.1 mean 1.0 0.50, 0.57 mean 0.54	102FX-532-2213 ^{a, b} TF-712-013 Sampling to analysis: 307–313 days
Canada, 1983 Osoyoos, British Columbia (–)	EC 190	–	–	0.235	–	3	Fruit	1 3 7	0.45 0.40 0.43	
		–	–	0.475	–	3	Fruit	1 3 7	0.71 0.60 0.39	102FX-532-2217 ^a TF-712-017 Sampling to analysis: 307–343 days
		–	–	0.235	–	1–2	Prune	1 3 7	0.53 0.40 0.45	
		–	–	0.475	–	1–2	Prune	1 3 7	0.26 0.96 0.61	

Analytical portion: fruit

^a The details of the in-field study were not shown in the study report.

^b The details of the analytical method were not shown in the study report.

Table 30 Triforine residues on plums from supervised trials in Germany and France

Plum, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no .			
France, 1981 Toulouse (-)	Vereor Multi Triforine +Carbdenazime 100	0.021	1000	0.210	3	1	0.38	10288-532-2301 ^{a, b} TF-712-060 Sampling to analysis: 223 days
Germany, 1990 Kriftzen (Ortenauer)	EC 190	0.028	1500	0.427	4	0	0.41	TF-712-091 ^{a, b} TF -712-081 to 84 (4 reports) Sampling to analysis: 24–54 days
						3	0.35	
						7	0.22	
						10	0.27	
Germany, 1990 Ingelheim (Chridimer)	EC 190	0.028	1500	0.427	3	14	0.13	
						0	0.57	
						3	0.32	
						7	0.18	
Germany, 1990 Veitshöchheim (Chridimer)	EC 190	0.028	1500	0.427	3	10	0.21	
						14	0.17	
						0	0.47	
						3	0.29,	
Germany, 1990 Öhringen (Hauszwetsche)	EC 190	0.028	1500	0.427	3	7	0.11,	
						10	0.26	
						14	0.10	
						0	4.09	
Germany, 1992 – (-)	EC 200	–	–	0.428	3	3	0.47 c: 0.02	SHTR.93.004 TF-712-088 Sampling to analysis: 225–255 days
						7	0.64 c: 0.03	
						0	0.2	
						7	0.15	
Germany, 1991 – (-)	–	–	–	–	–	0	0.63	
						3	0.50	
						7	0.54	
						10	0.35	
						14	0.19	
						0	0.83	
						4	0.39	
						7	0.41	
						11	0.56	
						0	0.058	
						3	0.056	
						7	0.048	
						10	0.031	
						14	0.013	
						0	0.14	
						3	0.090	
						7	0.083	
						11	0.045	
						14	0.027	

Analytical portion: fruit

^a The details of the in-field study were not shown in the study report.

^b The details of the analytical method were not shown in the study report.

Table 31 Triforine residues on plums from supervised trials in South Africa

Plum, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
GAP, South Africa	EC 190	0.025	2500–3500	0.865	2	3		Interval: 7 days
South Africa, 1979 Stellenbosch (Santa Rosa, Beauty)	Denarin 200	0.030	1000–1100	0.300	2	0 4 17	0.75 0.43 0.38	311/8978/S39 a, b TF -712-065
		0.030	900	0.300	2	0 4	0.75 0.35	LOQ: < 0.1

Analytical portion: fruit

^a The detail of the in-field study were not shown in the study report.

^b The detail of analytical method was not shown in the study report.

Apricot

Two residue trials in apricots were conducted in the USA. Triforine was quantified by GC-ECD with a LOQ of 0.007–0.01 mg/kg.

A total of five residue trials in apricots were conducted in France, Greece and Italy. Methods FAMS 041-01 and AGR010 were used to analyze apricot fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.01 mg/kg.

Table 32 Triforine residues on apricots from supervised trials in the USA

Apricot, country, year (variety)	Application						DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS	no.			
USA, 1977 Brentwood/CA (Blenheim)	EC 190	0.018	–	–	Blossom	3	3 7 13	2.0 1.6 0.81	102FX -532 –2502 TF-712-068 Sampling to analysis: 289– 298days
USA, 1980 Brentwood /CA (–)	EC 200	0.018	–	–	–	1	4	0.49	102FX-532-2212 ^a TF-712-012 Recovery = 58.8% (n = 1; 0.51 ppm) Sampling to analysis: 125–126 days
	2					2	4	0.76	
	EC 200 + WP 500	0.018 + 0.030 Post- harvest				1 + 1	(4)+0	2.5, 2.6	

Analytical portion: fruit

^a The details of the analytical method were not shown in the study report.

Table 33 Triforine residues on apricots from supervised trials in France, Greece and Italy

Plum, country, year (variety)	Application						Analytical portion	DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS (BBCH)	no.				
France, 1998 Quartier La Garenne/Mezoar gues (Hargrand /Myroboland)	DC 190	0.038	981– 1020	0.373– 0.388	77–81	3	Whole fruit	–0	0.37	CFS 1999-065 TF-712-093 Sampling to analysis: 234 days
								+0	0.78	
								3	1.1	
								7	0.74	
								10	0.44	
								13	0.40	
							Flesh	–0	0.39	
								+0	0.83	
								3	1.1	
								7	0.78	
								10	0.44	
								13	0.42	

Plum, country, year (variety)	Application						Analytical portion	DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS (BBCH)	no.				
France, 1998 Rognonas/Beauc aire (Precoce de Thyrinthe /Myroboland)	DC 190	0.038	1025– 1054	0.390– 0.401	81	3	Whole fruit	3 7 13	0.53 0.41 0.29	CFS 1999-064 TF-712-095 Sampling to analysis: 247 days
							Flesh	3 7 13	0.56 0.44 0.30	
Greece, 1999 Portaria- Halkidiki (Tirinthos)	DC 190	0.038	1490	0.566	85	3	Whole fruit	7	0.18	TF-HE-99-21 TF-712-096 Sampling to analysis: 70 days
Italy, 1999 Bentivoglio (Caldese)	DC 190	0.038	597– 643	0.227– 0.244	77	3	Whole fruit	–0 +0 3 7 10 14	0.13 0.52 0.31 0.14 0.069 0.070	TF-IT-1999-1 TF-712-097 Sampling to analysis: 153 days
Italy, 1999 Imola (Portici)	DC 190	0.038	670– 789	0.254– 0.300	81	3	Whole fruit	7	0.066	TF-IT-1999-2 TF-712-098 Sampling to analysis: 146 days

Nectarine

Four residue trials in nectarine were conducted in USA. Triforine was quantified by GC-ECD with a LOQ of 0.001–0.007 mg/kg.

Table 34 Triforine residues on nectarine from supervised trials in the USA

Nectarine, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
USA, 1978 Parlier/CA (Le Grand)	EC 190	0.018	–	–	4	0 6	0.59 0.19	102FX-532-2211 TF-712-011 Sampling to analysis: 259–265 days
	WP 500	0.03	–	–	4	0 6	2.8 1.8	
	F 800	0.015	–	–	4	0 6	3.6 2.4	
USA, 1980 Parlier/CA (Le Grand)	EC 190	0.018	–	–	1 2	10	0.32 0.32	102FX-532-2212 ²⁾ TF-712-012 Sampling to analysis: 88 days Recovery 55.13% (0.48 mg/kg)
	EC 190 + WP 500	0.018 + 0.03 Post- harvest	–	–	2 + 1	(10) 0	0.11 0.93	
USA, 1981 Sultana (–)	EC 182	0.018	–	–	3	2	0.036, 0.043 mean 0.040	102FX-532-2418 ²⁾ TF- 712-037 Sampling to analysis: 175 days
USA, 1977 Parlier/CA (–, Le Grand, Red Bud)	EC 190	0.018	–	–	3	0 1 3 7 0 5 11	0.54 0.21 0.11 0.08 0.50 0.47 0.28	102FX-532-2419 ¹⁾ TF-712-038 Sampling to analysis: 245–322 days

Nectarine, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
						0	0.73	
						1	0.64	
						3	0.53	
						7	0.34	

Analytical portion: fruit

^a The details of the in-field study were not shown in the study report.

^b The details of the analytical method were not shown in the study report.

Peach

Nineteen residue trials in peaches were conducted in USA. Method RU3, 26/12/10 was used to analyze peach fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.001–0.01 mg/kg.

One residue trials in peaches was conducted in Canada. Triforine was quantified by GC-ECD with a LOQ of 0.01 mg/kg.

Total three residue trials in peaches were conducted in France and Greece. Triforine was quantified by GC-ECD with a LOQ of 0.01 mg/kg.

Two residue trials in peaches were conducted in Japan. Triforine was quantified by LC-MS with a LOQ of 0.01 mg/kg.

Two residue trials in peaches were conducted in Brazil. Triforine was quantified by GC-ECD with a LOQ of 0.01 mg/kg.

Two residue trials in peaches were conducted in South Africa. Triforine was quantified by GC-ECD with a LOQ of 0.1 mg/kg.

Table 35 Triforine residues on peaches from supervised trials in the USA and Canada

Peach, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
GAP, Canada	EC 190	0.014 –	– –	– 0.475	3	–		Timing: 1 st : Early bloom stage 3 rd : full bloom stage
					3	–		
USA, 1978 Lockford/CA (Loadel)	EC 190	0.018	–	–	2	133	0.006	102FX-532-2211 TF -712-011
					4	0	3.5	
					5	5	2.0	
USA, 1978 Parlier/CA (Fay Elberta)	EC 190	0.018	–	–	4	0 7	4.3 3.3	Sampling to analysis: 189–235 days
USA, 1978 Corvallis /OR (Elberta)	EC 190	0.018	–	–	3	160	0.015	
					5	7	0.12	
USA, 1978 Lockford/CA (Loadel)	WP 500	0.030	–	–	2	133	0.012	
					4	0	4.6	
					5	5	5.2	
USA, 1978 Davis/CA (Halloween)	WP 500	0.030	–	–	1 Po	0	8.8 5.1	
USA, 1978 Parlier/CA (Fay Elberta)	WP 500	0.030	–	–	4	0 7	11 4.3	
USA, 1978 Corvallis /OR (Elberta)	WP 500	0.030	–	–	3	160	0.004	
					5	7	3.5	
USA, 1978	F	0.015	–	–	2	133	0.022	

Peach, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
Lockford/CA (Loadel)	800				4	0 5	6.8 7.4	
USA, 1978 Parlier/CA (Fay Elberta)	F 800	0.015	—	—	4	0 7	5.7 4.5	
USA, 1978 Corvallis /OR (Elberta)	F 800	0.015	—	—	3 5	160 7	0.02 0.07	
USA, 1980 Parlier/CA (Fay Elberta)	EC 190	0.018	—	—	2	10	0.34 0.96	102FX-532-2212 ^b TF-712-012 Recovery 56.22% (0.48 mg/kg) Sampling to analysis: 88 days
	EC 190 + WP 500 (Post- harvest)	0.03	—	—	2 + 1	(10) 0	1.0 1.8	
USA, 1983 /CA (—)	EC 182 Aerial	—	—	0.504	4	0 1 3 7	0.32 0.36 0.70 0.62 0.21, 0.084 0.031 0.043	102FX-532-2414 ^{a, b} TF-712-033 Sampling to analysis: 176–183 days
		—	—	0.672	4	0 1 3 7	0.80, 0.71 0.50, 0.23, 0.15, 0.14, 0.21, 0.35	
Canada, 1981 British Colombia (Red Fairhaven)	EC 190	—	—	0.235	3	1 3 7	0.60 0.61 0.37	10212-532-2217 ^{a, b} TF-712-017
		—	—	0.475	3	1 3 7	1.5 1.2 0.67	
USA, 1981 Sultana	EC 190	0.018	—	—	3	0	0.58, 0.014	102FX-532-2418 ^{a, b} TF-712-037 Sampling to analysis: 175 days
USA, 1977 Kingston/RI (mixed)	EC 190	—	—	0.672	4	0 1 3	1.9 0.6 0.41	102FX-532-2420 TF-712-039 Sampling to analysis: 218–253 days
USA, 1977 Linden /CA (Dixon)		—	—	0.672	3	0 7 15	4.3 3.5 2.4	
		—	—	0.448	3	0 7 15	3.6 1.5 0.66	
USA, 1977 Parlier/CA (Fay Elberta)		—	—	0.672	3	0 5 11	1.2 0.97 0.49	
USA, 1977 Corvallis /OR (Elberta)		—	—	0.672	3	0 3 7 14	1.1, 0.99 0.9 0.34 0.16	
USA, 1977 Blacksburg /VA (Elberta)		—	—	0.448	2	0 3 7 14	1.0 0.26 0.23 0.98	
USA, 1977 Weatherford /TX (Red Globe)		—	—	0.448	2	0	0.55 0.72	

Analytical portion: fruit

^a The details of the in-field study were not shown in the study report.

^b The details of the analytical method were not shown in the study report.

Table 36 Triforine residues on peach from supervised trials in France and Greece

Peach, country, year (variety)	Application						Analytical portion	DAT Days	Residues, mg/kg	Ref	
	For m, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS	no .					
France, 1990 Cabannes (Elegant Lady)	EC 190	0.054 – 0.076	500– 700	0.380	G to J stage	8	Whole fruit	0 3 7 14 28 42	0.14 0.07 0.05 0.03 < 0.01 < 0.01	BETR.91.016 TF-712-087 Sampling to analysis: 434– 463 days	
Greece, 1999 Skidra Pelis (Andross / GF 677)	DC 190	0.038	996– 999	0.379	BBC H 85	3	Flesh	3 7 14	1.1 0.82 0.68	CFS 1999-041 TF-712-92 Sampling to analysis: 189– 207 days	
							Whole fruit	3 7 14	0.99 0.75 0.62		
		0.024	1504	0.361		1	Flesh	3 7 14	0.59 0.61 0.27		
							Whole fruit	3 7 14	0.54 0.56 0.25		
France, 1999 Barbentane (Gracia)	EC 190	0.038	1000	0.380	BBC H 77– 85	3	Whole fruit	3 7 14	0.52 0.42 0.23	TF-FR-99-F01 TF-712-100 Sampling to analysis: 113– 123 days	

Table 37 Triforine residues on peaches from supervised trials in Japan

Peach, country, year (variety)	Application					Analytical portion	DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.				
GAP, Japan	EC 180	0.015 – 0.018	2000–7000	–	5	Fruit	1		
Japan, 2009– 2010 Ezohara Yamanashi (Hikawa- Hakuhou)	EC 180	0.023	3548	0.798	5	Pulp	1 3 7 14	0.03 0.07 0.04 < 0.01	Sampling to analysis: 60–91 days
						Whole fruit	1 3 7 14	0.77 0.84 0.69 0.15	
Japan, 2009– 2010 Kishigawacho /Wakayama (Hakuhou)	EC 180	0.023	4000	0.900	5	Pulp	1 3 7 14	0.08 0.05 0.04 < 0.01	
						Whole fruit	1 3 7 14	1.4 0.62 0.32 0.10	

Table 38 Triforine residues on peaches from supervised trials in Brazil

Peach, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
GAP, Brazil	EC 190	0.024	(400–1000)	0.23 7	3	3		Timing: 1st : First sign of infection Interval: 7–10 days
Brazil, 1985/86 Cascata (Magno)	Saprol EC	0.015	–	–	3	3 7	1.5 0.95	TF-712-036 a, b
		0.030	–	–	3	3 7	9.4 4.2	
Brazil, 1996 Charqueadas (Sinuelo)	Saprol EC	0.024	–	–	3	4	< 0.01	BASF 1986/306182
		0.048	–	–	3	4	< 0.01	

Analytical portion: fruit

^a The details of the in-field study were not shown in the study report.^b The details of the analytical method were not shown in the study report.

Table 39 Triforine residues on peaches from supervised trials in South Africa

Peach, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
GAP, South Africa	EC 190	0.019	2500–3500	0.665	2	3		Interval: 13–15 days
South Africa, 1977 Ganskraal (Woltemade)	Denarin 200	0.025	3000	0.750	2	0	1.1,1.4	0311/8994/P131 TF-712-049 a, b)
						4	mean 1.2	
						8	1.0,1.0 mean 1.0 0.69,0.70 mean 0.70	
South Africa, 1977 Lourensford (Woltemade)	Denarin 200	0.025	3000	0.750	2	0	1.3,1.7	
						2	mean 1.5	
						7	1.6,1.6 mean 1.6 1.0,1.0 mean 1.0	

Analytical portion: fruit

^a The details of the in-field study were not shown in the study report.^b The details of the analytical method were not shown in the study report.*Berries and other small fruits**Cane berries**Raspberries, Red, black*

One residue trial in raspberries was conducted in France. The 190 g/L DC formulation was applied five times at a rate equivalent to 0.33–3.8 kg ai/ha. Method FAMS 041-01 was used to analyze raspberry fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.01 mg/kg.

Table 40 Triforine residues on raspberries from supervised trials in France

Raspberries, country, year (variety)	Application						DAT Days	Residues, mg/kg	Ref
	For m, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS	no			
France, 1993 Machy/Chasselay (Meeker)	DC 190	—	—	0.332	5,10,30 and 40 cm shoot length, end of flowering	5	16	0.24	CFS1994-024 TF-713-046 Sampling to analysis: 128–141 days
				0.760		5	16	0.56	
				3.8		5	16	2.4	

Analytical portion: fruit

*Bush berries**Blueberries*

Five residue trials in blueberries were conducted in Canada. Triforine was quantified by LC-MS/MS with a LOQ of 0.01 mg/kg.

Table 41 Triforine residues on blueberries from supervised trials in Canada

Blueberries, country, year (variety)	Application						DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS	no.			
GAP, Canada (except Eastern Canada)	EC 190	—	1000	0.570	1 st : bud-break 4 th : 10–14 days after early bloom*	4	60		Interval: 10–14 days
GAP, Eastern Canada only	EC 190	—	1000	0.570	1 st : leaf-bud break 3 rd : pink-bud stage	3	60		Interval: 10–14 days
Canada, 2010 Mount Stewart/ Prince Edward Island (Wild Low Bush) [Eastern Canada]	Saprol 190	0.059	964–1022	0.564– 0.598	1 st : leaf-bud break 3 rd : pink bud stage	3	78	< 0.01	875.2100-10- 543-24A- 13~20 Sampling to analysis: 63– 109 days
	DC	0.059	991–1015	0.579– 0.594			78	< 0.01	
Canada, 2010 Mount Vernon/ Prince Edward Island (Wild Low Bush) [Eastern Canada]	Saprol 190 DC	0.058	981–999	0.574– 0.584	1 st : leaf-bud break 3 rd : pink bud stage	3	85	< 0.01	
Canada, 2010 Burford/Ontario (Patriot/Blue Crop) [Eastern Canada]	Saprol 190 DC	0.058	1059– 1079	0.577– 0.586	1 st : leaf-bud break 3 rd : pink bud stage	3	45	< 0.01	
		0.060	1050– 1071	0.583– 0.602			49	< 0.01	
							56	< 0.01	
							60	< 0.01	
	0.059	1054– 1068	0.559– 0.593			60	< 0.01		
Canada, 2010 Abbotsford/British Columbia (Reka/Patriot) [Canada except Eastern Canada]	Saprol 190 DC	0.058	998–1010	0.582– 0.589	1 st : leaf-bud break 4 th : 10–14 days after early bloom	4	60	0.018 0.011 Mean: <u>0.015</u>	875.2100-10- 543-24A- 13~20 Sampling to analysis: 63– 109 days
Canada, 2010 Delta/British Columbia (Reka/Patriot) [Canada except	Saprol 190 DC	0.059	985–1016	0.574– 0.592	1 st : leaf-bud break 4 th : 10–14 days after early bloom	4	61	< 0.01	109 days

Blueberries, country, year (variety)	Application						DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS	no.			
Eastern Canada]									

Analytical portion: fruit

Currant, Black

Total four residue trials in black currants were conducted in Germany and UK. Triforine was quantified by GC-ECD with a LOQ of 0.01 mg/kg.

Table 42 Triforine residues on black currants from supervised trials in Germany and the UK

Berries, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	For m, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
Germany, 1975 Hamburg	DC 190	0.015	–	–	5	0 7 14 21	5.6 3.2 1.1 0.89	102FX-532-3205 TF-713-025 Sampling to analysis: 181–202 days
UK, 1976 Charlsfield (Baldwin/Wellington)	EC 190	–	–	0.492	4	7 14 7	1.0 0.61 c: 0.015 0.75 c: 0.009	102FX-532-3212 TF-713-032 Sampling to analysis: 82–92 days Recovery 66–72%
UK, 1976 Matley (Baldwin)		–	–	0.280	5 6	16 7	0.17 0.71 c: 0.038	
UK, 1976 Risby (Wellington)		–	–	0.560	4	14	1.2 c: 0.014	

Analytical portion: fruit

Small fruit vine climbing

Grapes

Seven residue trials in grapes were conducted in Germany. Method RU 3, 26/12/10 was used to analyze grape fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.01–0.05 mg/kg.

Two residue trials in grapes were conducted in Mexico. Triforine was quantified by GC-ECD with a LOQ of 0.002–0.003 mg/kg.

One residue trial in grapes was conducted in New Zealand. Triforine was quantified by GC-ECD with a LOQ of 0.1 mg/kg.

Table 43 Triforine residues on grapes from supervised trials in Germany

Grapes, country, year (variety)	Application					DAT Days	Residue s, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
Germany, 1983 Schwabenheim (Müller-Thurgau)	EC 190	0.029	200	0.570	3	0 21 28 35	5.5 1.9 1.6 1.4	10238-532-3003 ^a TF -713-003 Sampling to analysis: 67–112 days

Grapes, country, year (variety)	Application					DAT Days	Residue s, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no .			
Germany, 1983 Bockenheim (Müller-Thurgau)	EC 190	0.029	200	0.570	3	0 21 28 35 44	1.7 0.55 0.39 0.38 0.25	10238-532-3004 TF-713-004 Sampling to analysis: 51–103 days
Germany, 1983 Kippenhausen (Müller-Thurgau)	EC 190	0.029	150	0.427	3	0 21 28 35 42 49	1.6 0.25 0.13 0.11 0.08 0.08	10238-532-3005 TF-713-005 Sampling to analysis: 64–119 days
Germany, 1978 Impflingen (Reichensteiner)	EC 190	0.014	2000	0.285	3	0 14 21 28 35	0.64 0.72 0.55 0.50 0.51	102FX-532-3009 TF-713-009 Sampling to analysis: 107– 142 days
Germany, 1977 Schwabenheim (Müller-Thurgau)	EC 190	0.029	1160	0.331	3	0 14 21 28 35	1.93 0.38 0.22 0.17 0.18	10238-532-3010 TF -713-010 Sampling to analysis: 89–124 days
Germany, 1977 Haugnau/Bodensee (Müller-Thurgau)	EC 190	0.019	1200	0.228	3	0 21 28 35 42	2.76 0.88 0.52 0.38 0.40	10238-532-3011 TF -713-011 Sampling to analysis: 64–106 days
Germany, 1984 Schwabenheim (Kerner)	EC 190	0.057	887	0.505	1	0 3 7 10 14 21	1.1 0.96 0.95 0.79 0.6 0.65	10238-532-3012 TF -713-012 Sampling to analysis: 73–101 days

Analytical portion: fruit

^a The details of the in-field study were not shown in the study report.

Table 44 Triforine residues on grapes from supervised trials in Mexico

Grapes, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no .			
Mexico, 1983 Caborca (Thompson seedless)	EC 190	—	—	0.273	1	0 3 7 14 21	0.21, 0.22, 0.26, 0.25, 0.31 0.46, 0.69, 0.02, 0.21, 0.12 0.79, 0.37, 0.37, 0.74, 0.12 < 0.002(2), 0.40, 0.19, 0.34 0.22, 0.33, 0.26, 0.46, 0.16	102FX-532-3007 a, b TF -713-007 Sampling to analysis: 204–224 days
Mexico, 1983 San Juan Del Rio, Torreon (Carignan)	EC 190	—	—	0.285	1	0 3 7 14 21	0.93, 0.61, 1.17, 3.38, 1.88 1.55, 0.30, 0.041, 3.93, 0.72 0.79, 1.53, 0.076, 2.91, 0.36 2.43, 0.19, 0.026, 4.81, 1.12 0.18, 0.24, 0.18, 0.39, 1.89	102FX-532-3008 b TF -713-008 Sampling to analysis: 814–834 days

Analytical portion: fruit

^a The details of the in-field study were not shown in the study report.

^b The details of the analytical method were not shown in the study report.

Table 45 Triforine residues on grapes from supervised trials in New Zealand

Grapes, country, year (variety)	Application						DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS	no			
GAP, New Zealand	EC 190	0.019	–	0.380 (at least)	1st: bud burst	4	14		Interval: 2–3 weeks
New Zealand, 1978 Blenheim (Cabernet Sauvignon)	EC 200	0.02	8970	0.18	Bud burst, blossom.	8	1 7 14 21	< 0.1 < 0.1 < 0.1 < 0.1	102FX-532-3013 TF -713-013 Sampling to analysis: 108–129 days

Analytical portion: fruit

Low growing berries

Cranberry

Two residue trials in cranberry were conducted in the USA. Method RU 3, 26/12/10 was used to analyze cranberry fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.004–0.01 mg/kg.

Table 46 Triforine residues on cranberries from supervised trials in the USA

Cranberry, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
GAP, Canada	EC 190	–	1000–1500	0.570	3	60		Timing: 1 st : bud break Interval: 10–14 days
USA, 1977 Long Beach, Grayland Bog/WA (–)	EC 190	–	–	0.336	1	84 91 114 125 143	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	102FX-532-3402 ^a TF-713-038 Sampling to analysis: 196–237 days
USA, 1983 Plymouth/MA (–)	EC 190	–	–	1.05	1	64	0.022, 0.020, 0.016, 0.016 mean 0.019	102FX-532-3401 TF-713-041 Sampling to analysis: 248 days
		–	–	2.10	1	64	0.022, 0.038, 0.029, 0.026 mean 0.029	

Analytical portion: fruit

^a The details of the in-field study were not shown in the study report.

Strawberry

Two residue trials in strawberries were conducted in Mexico. Triforine was quantified by GC-ECD.

Two residue trials in strawberries were conducted in Brazil. Triforine was quantified by GC with a LOQ of 0.02 mg/kg.

Four residue trials in strawberries were conducted in Japan. Triforine was quantified by GC with a LOQ of 0.005 mg/kg.

Table 47 Triforine residues on strawberries from supervised trials in Mexico

Strawberry, country, year (variety)	Application						DAT Days	Residue s, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS	no			
GAP, Mexico	EC 190	–	–	0.190	–	4	14		Interval: 4, 7 or 10 days
Mexico, 1978 Irapuato/Gto (Tioga)	EC 190	0.019	1010	0.190	–	4	0 3 7 14 22	1.4 1.3 1.2 0.57 0.14	102FX-532-3108 TF-701-004 Sampling to analysis: 344–366 days
Mexico, 1979 Irapuato/Gto (Fresno)	EC 190	–	1010	0.190	Flowering and production	4	0 1 3	1.4 1.0 0.78	102FX-532-3109 TF-713-019 Sampling to analysis: 49–52 days

Analytical portion: fruit

Table 48 Triforine residues on strawberries from supervised trials in Brazil

Strawberry, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
GAP, Brazil	EC 190	0.029	800	0.228	4	2		Interval: 7 days
Brazil, 1985 Botucatu (Campinas)	EC 190	–	–	0.370	4	2	0.9	102FX-532-3101 ^{a,b} TF-713-014
		–	–	0.741		4	0.3	
		–	–	0.741		7	0.2	
		–	–	0.741		2	2.0	
Brazil, 1985 Botucatu (Campinas)	EC 190	–	–	0.428	4	4	1.2	102FX-532-3102 ^{a,b} TF-713-015
		–	–	0.428		4	0.4	
		–	–	0.428		7	0.2	
		–	–	0.855		2	3.2	
		–	–	0.855		4	1.0	
		–	–	0.855		7	0.7	

Analytical portion: fruit

^a The details of the in-field study were not shown in the study report.^b The details of the analytical method were not shown in the study report.

Table 49 Triforine residues on strawberries from supervised trials in Japan

Strawberry, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
GAP, Japan	EC 180	0.009	1000–3000	–	5	1		
Japan, 1982 Greenhouse Hanyu-shi/Saitama (Reikou)	EC 150	0.007– 0.010	2000	0.142–0.190	5	1	0.78	Sampling to analysis: 57– 184 days
						3	0.41	
						7	0.26	
Japan, 1982 Greenhouse Toriya-cho/Nara (Houkou-Wase)	EC 150	0.010– 0.013	1500	0.142–0.190	5	1	0.48	
						3	0.45	
						7	0.23	
Japan, 1985 Greenhouse Honjo/Tochigi (Reikou)	EC 150	0.008	1500	0.113	5	1	0.20	Sampling to analysis: 58– 153 days
						3	0.12	

Strawberry, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
Japan, 1985 Greenhouse Toyota- machi/Shizouka (Houkou-Wase)	EC 150	0.008	1500	0.113	5	1 3	0.39 0.25	

Analytical portion: fruit

Fruiting vegetables, Cucurbits

Cucumber

Four residue trials in cucumber were conducted in the USA. Triforine was quantified by GC-ECD with a LOQ of 0.004 mg/kg.

One residue trial in cucumber was conducted in Canada. Method RU 3, 26/12/10 was used to analyze cucumber fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD.

One residue trial in cucumber was conducted in Mexico. Method RU 3, 26/12/10 was used to analyze cucumber fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD.

Total three residue trials in cucumber were conducted in Hungary, France and Germany. Triforine was quantified by GC with a LOQ of 0.02 mg/kg.

Table 50 Triforine residues on cucumbers from supervised trials in USA, Canada and Mexico

Cucumber, country, year (variety)	Application						DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS	no.			
GAP, Mexico	EC 190	—	—	0.285		3	7		Interval: 7 days
Mexico, 1978 Culiacan, Sin (Poinsett)	EC 190	—	320	0.285	Mature	3	0 3 7 14 22	0.065 0.17 0.066 0.008 0.009	102FX-532-3108 TF-701-004 Sampling to analysis: 359–379 days
Canada, 1984 — (—)	EC 190	—	—	0.285	—	1	1	0.13 0.14 0.14 mean: 0.14	102FX-532-4503 TF-723-012 Sampling to analysis: 352 days
USA, 1984 Homestead/ FL (—)	EC 190	—	—	0.285	—	4	3	0.014, 0.041 0.047, 0.024 mean: 0.032	102FX-532-4504 TF-723-013 Sampling to analysis: 98 days
				0.570			3	0.050, 0.053 0.056, 0.037 mean: 0.049	
USA, 1986 Lafayette/IN (—)	EC 190	—	—	0.285	—	4	2	0.031, 0.013 0.033, 0.030 mean: 0.027	10238-532-4516 TF-723-024 Sampling to analysis: 224 days (Control < 0.004–0.015)
				0.570			2	0.013, 0.035 0.064, 0.027 mean: 0.035	
USA, 1986 Weslaco/TX (—)	EC 190	—	—	0.285	—	4	2	0.14, 0.11, 0.20, 0.22 mean: 0.17	10238-532-4522 TF-723-030 Sampling to analysis: 285 days
				0.570			2	0.39, 0.30, 0.19, 0.36 mean: 0.31	
USA, 1987 Columbus/OH (—)	EC 190	—	—	0.285	—	5	1	0.14, 0.16, 0.098, 0.16 mean: 0.14	10238-532-4523 TF-723-031

Cucumber, country, year (variety)	Application						DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS	no.			
				0.570			1	0.052, 0.099 0.034, 0.079 mean: 0.066	Sampling to analysis: 105 days

Analytical portion: fruit

Table 51 Triforine residues on cucumber from supervised trials in Hungary, France and Germany

Cucumber, country, year (variety)	Application						DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS	no.			
Hungary, 1985 Nograd cucumber (-)	EC 190	0.057	500	0.285	—	1	0 1 5 10 14 21	2.0, 2.6, 3.0 Mean: 2.5 1.2, 2.9, 3.2 Mean: 2.4 0.29, 0.55, 0.62 Mean: 0.49 0.065, 0.10, 0.13 Mean: 0.10 0.010, 0.040 0.097 Mean: 0.049 < 0.001(3)	10238-532-4515 TF-723-023 ^a Sampling to analysis: 37–65 days Recovery 61%
Germany, 1989 Bonn Cucumber (Sporu)	EC 190	0.029– 0.228	1200– 1500	0.342	—	8	0 1 2 3 4	0.22 0.025 0.097 0.11 0.022	10249-532-4526 TF-723-048 ^a Sampling to analysis: 257– 274 days
France, 1993 Tassin Cucumber (Girola)	EC 190	0.024	1201	0.285	Flowering –mature	3	3	0.17	CFS1994-095 TF-723-057 Sampling to analysis: 235– 239 days

Analytical portion: fruit

^a The details of the in-field study were not shown in the study report.

Squash

Total three residue trials in courgettes were conducted in France and Germany. Method RU 3, 26/12/10 was used to analyze courgette fruits samples for the residues of Triforine. Triforine was quantified by GC with a LOQ of 0.02 mg/kg.

Five residue trials in winter and summer squash were conducted in USA. Triforine was quantified by GC-ECD with a LOQ of 0.003–0.005 mg/kg.

Table 52 Triforine residues on summer squash from supervised trials in France and Germany

Squash, country, year (variety)	Application						DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS	no.			
France, 1979 – Courgettes (-)	EC 100 + Carbend azim	0.020	2000	0.400	—	5	7	0.05	10238-532- 4701 TF-723-040 ^a Sampling to analysis: 123 days

Squash, country, year (variety)	Application						DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS	no			
France, 1979 – Courgettes (–)	EC 190	0.015	2000	0.570	–	5	7	0.02	10238-532- 4702 TF-723-041 ^a Sampling to analysis: 123 days
Germany, 1989 Frankfurt Courgettes (Chinese Schlange)	EC 190	0.029	600	0.171	–	9	0 1 2 3 4	0.097 c: 0.014 0.10 0.023 0.090 c: 0.013 0.084	10249-532- 4525 TF-723-047 ^a Sampling to analysis: 268– 285 days

Analytical portion: fruit

^a The details of the in-field study were not shown in the study report.

Table 53 Triforine residues on summer and winter squash from supervised trials in the USA

Squash, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	For m, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
USA, 1987 Lafayette/IN Squash (–)	EC 190	–	–	0.285	5	1	0.097, 0.097, 0.111, 0.005 mean: 0.078	TF-723-044 ^{a, b} Sampling to analysis: 222 days
				0.456		1	0.18, 0.22 0.23, 0.26 mean: 0.22	
USA, 1987 Brulington/VT Summer Squash (–)	EC 190	–	–	0.228	5	1	0.026, 0.036 0.044, 0.044 mean: 0.038	102FX-532-4706 ^{a, b} TF-723-045 Sampling to analysis: 329 days
				0.456		1	0.054, 0.059 mean: 0.057	
USA, 1987 Salisbury/MD Pumpkins (–)	EC 190	–	–	0.228	1	1	0.031, 0.032 0.061, 0.074 mean: 0.038	102FX-532-4707 ^{a, b} TF-723-046 Sampling to analysis: 361 days Control: < 0.004–0.085
				0.456		1	0.12, 0.13, 0.14, 0.21 mean: 0.22	
USA, 1987 Davis/CA Squash (–)	EC 190	–	–	0.228	5	1	0.076, 0.13, 0.21, 0.32 mean: 0.17	TF-723-053 ^{a, b} Sampling to analysis: 317 days Control: < 0.003–0.007
				0.456		1	0.23, 0.23, 0.24, 0.28 mean: 0.25	
USA, 1987 Lafayette/IN Pumpkins (–)	EC 190	–	–	0.228	5	1	0.018, 0.028, 0.028, 0.029 mean: 0.026	102FX-532-4704 ^b TF-723-043 Sampling to analysis: 142 days
				0.456		1	0.027, 0.029, 0.030, 0.031 mean: 0.029	

Analytical portion: fruit

^a The details of the in-field study were not shown in the study report.

^b The details of the analytical method were not shown in the study report.

Melon

Three residue trials in melon were conducted in USA. Triforine was quantified by GC-ECD with a LOQ of 0.003–0.004 mg/kg.

One residue trial in melon was conducted in Mexico. Triforine was quantified by GC-ECD with a LOQ of 0.004 mg/kg.

Two trials were conducted in Italy and France on melon. Triforine was quantified by GC-ECD.

Two trials in melon were conducted in Japan. Triforine was quantified by GC-ECD with a LOD of 0.005 mg/kg.

Table 54 Triforine residues on melons from supervised trials in USA and Mexico

Melon, country, year (variety)	Application						DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	GS	no.			
GAP, Mexico	EC 190	–	–	0.285		3	7		Interval: 7 days
Mexico, 1979 Apatzingan/Mich Cantaloupe (Top Mark)	EC 190	–	400	0.285	Fully grown 45 days after sowing	3	0 1 3 7 14 21	0.35 0.18 0.090 0.039 0.017 0.006	102FX-532-4604 ^a TF-723-036 Sampling to analysis: 76–97 days
USA, 1985 Weslaco/TX Cantaloupes	EC 190	–	–	0.228	–	1	3	0.023, 0.027, 0.032, 0.039 mean: 0.030	102FX-532-4603 TF-723-035
				0.456		1	3	0.048, 0.081 0.089, 0.108 mean: 0.082	Sampling to analysis: 96 days
USA, 1986 Lafayette/IN Muskmelons	EC 190	–	–	0.228	–	5	1	0.036, 0.078, 0.091 mean: 0.068	102FX-532-4607 TF-723-039
				0.456		5	1	0.22, 0.23 0.34 mean: 0.26	Sampling to analysis: 317 days
USA, 1987 Davis/CA Cantaloupes	EC 190	–	–	0.228	–	5	1	0.009, 0.015 0.018, 0.029 mean: 0.018	TF-723-053 Sampling to analysis: 433 days
				0.456		5	1	< 0.004, 0.011 0.024, 0.028 mean: 0.017	(Control < 0.004–0.015)

Analytical portion: whole fruit

^a The details of the analytical method were not shown in the study report.

Table 55 Triforine residues on melons from supervised trials in France and Italy

Melons, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
France, 1981 Toulouse Melons (Orlinabel)	Vereor Multi (100 g ai/L) + Carbendazim	0.02	1000	0.20	4	1	0.059	10288-532-4602 ^a TF-723-034
Italy, 1977 Melons	EC 190	0.029	1000	0.285	1	0 3 7 10	0.72 0.43 0.11 0.15	102FX-532-4606 ^a TF-723-038

Analytical portion: whole fruit

^a The details of the analytical method were not shown in the study report.

Table 56 Triforine residues on melons from supervised trials in Japan

Melons, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	For m, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
GAP, Japan	EC 180	0.009	1000–3000	–	6	1		
Japan, 1978 Greenhouse Hamamatsu, Shizuoka, Muskmelon (Earl's-Favorite)	EC 150	0.015 – 0.019	3000	0.450– 0.563	6	1 3 5 7	< 0.005 < 0.005 < 0.005 < 0.005	Sampling to analysis: 61–212 days
Japan, 1978 Greenhouse Tateyama, Chiba Muskmelon (Earl's-Favorite)	EC 150	0.015	2500	0.375	6	1 3 5 7	0.006 0.007 < 0.005 < 0.005	

Analytical portion: whole fruit

*Fruiting vegetables, other than Cucurbits**Peppers*

Two residue trials in peppers were conducted in Mexico. Method RU 3, 26/12/10 was used to analyze pepper fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.003–0.004 mg/kg.

Four residue trials in peppers were conducted in Japan. Triforine was quantified by GC-ECD with a LOD of 0.005 mg/kg or LC-MS with a LOQ of 0.01 mg/kg.

One trial in chilli peppers was conducted in Korea. Triforine was quantified by GC-ECD with a LOQ of 0.05 mg/kg.

Table 57 Triforine residues on peppers from supervised trials in Mexico

Peppers, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
GAP, Mexico	EC 190	–	–	0.285	3	14		Interval: 7 days
Mexico, 1978 Culiacan, Sin Pepper (chilli) (Wonder 300)	EC 190	–	160	0.285	3	7 16 23	0.24 0.12 0.14	102FX-532-3108 TF-701-004 Sampling to analysis: 352–368 days
Mexico, 1979 Culiacan, Sin Pepper (Yolo Wonder)	EC 190	–	160	0.285	3	0 1 3	2.6 0.95 0.45	102FX-532-4401 TF-723-007 Sampling to analysis: 96– 99 days

Analytical portion: fruit

Table 58 Triforine residues on peppers from supervised trials in Japan

Peppers, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
GAP, Japan	EC 180	0.018	1000– 3000	–	3	14		
Japan, 1983 Ushiku, Ibaraki Pepper (Green) (Ace) Greenhouse	EC 150	0.015	1500	0.23	3	1 3	0.79 0.54	Sampling to analysis: 60 days
		0.008	1500	0.11		1	0.41	
Japan, 1983 Miyazaki, Miyazaki, Pepper (Green) (Tosa-Kotobuki) Greenhouse	EC 150	0.015	2000	0.30	3	1 3	1.2 1.2	
		0.008	2000	0.15		1	0.63	
Japan, 1983 Konan, Kochi Pepper (Green) (Tosahime R) Greenhouse	EC 150	0.015	2500	0.38	3	1 3 14	1.2 1.0 0.22	
		0.010	2500	0.25	1	1 3 14	0.52 0.44 0.08	
Japan, 1983 Miyazaki, Miyazaki, Pepper (Green) (Kyosuzu) Greenhouse	EC 150	0.015	3000	0.45	3	1 3 14	0.59 0.56 0.06	
		0.010	3000	0.30	1	1 3 14	0.36 0.28 0.06	

Analytical portion: fruit

Table 59 Triforine residues on pepper from supervised trials in South Korea

Peppers, country, year (variety)	Application					DA T Day s	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
GAP, South Korea	EC 190	0.019	–	–	2	7		Timing: First sign of infection Interval: 10 days
S.Korea, 2009 Chungcheongnam -do Peppers (chilli) (Nokkwang) Greenhouse	DC 190	0.019 –	2000	0.380	1	1	0.62, 0.61, 0.64, mean 0.62	Sampling to analysis: 22–28 days
						3	0.51, 0.53, 0.53 mean 0.52	
						5	0.42, 0.47, 0.52 mean 0.47	
						7	0.33, 0.35, 0.37 mean 0.35	
					2	1	1.04, 1.08, 1.02 mean 1.05	
						3	0.83, 0.80, 0.78 mean 0.80	
						5	0.76, 0.91, 0.84 mean 0.84	
						7	0.33, 0.35, 0.37 mean 0.35	

Analytical portion: fruit

Eggplant

Two residue trials in eggplants were conducted in Mexico. Method RU 3, 26/12/10 was used to analyze eggplant fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD.

Five residue trials in eggplants were conducted in Japan. Triforine was quantified by GC-ECD with a LOD of 0.005 mg/kg or LC-MS with a LOQ of 0.01 mg/kg.

Table 60 Triforine residues on eggplants from supervised trials in Mexico

Eggplant, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
GAP, Mexico	EC 190	—	—	0.285	3	15		Interval: 5 or 8 days
Mexico, 1976 Sinaloa	EC 190	—	—	0.285	3	0 2 4 6	0.37 0.30 0.69 0.28	102FX-532-4403 ^{a, b} TF-723-009
Mexico, 1979 Culiacan, Sin (Black Beauty)	EC 190	—	160	0.285	3	0 3 7 14 21	0.19 0.058 0.032 0.066 0.017	102FX-532-3108 TF-701-004 Sampling to analysis: 355–376 days

Analytical portion: whole fruit

^a The details of the in-field study were not shown in the study report.

^b The details of the analytical method were not shown in the study report.

Table 61 Triforine residues on eggplants from supervised trials in Japan

Eggplant, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
GAP, Japan	EC 180	0.018	1000–3000	—	5	1		
Japan, 2008 Kohnan, Kochi (Ryoma)	EC 150	0.015	3000	0.450	5	1 3 7 14	0.28 0.24 0.06 < 0.01	Sampling to analysis: 100 days
Japan, 2008 Miyazaki, Miyazaki (Kokuyou)	EC 150	0.015	3000	0.450	5	1 3 7 14	0.38 0.25 0.12 0.02	
Japan, 1986 Sousa-shi, Chiba (Senryo)	EC 150	0.015	3000	0.450	5	1	0.39	Sampling to analysis: 30– 92days
Japan, 1986 Shakudo, Osaka (Senryo-2)	EC 150	0.015	2500	0.375	5	1	0.29	
Japan, 1986 Noichi-cho, Kochi (Hayabusa)	EC 150	0.015	2600	0.390	5	1	0.25	

Analytical portion: fruit

Tomato

Twelve residue trials in tomatoes were conducted in USA. Triforine was quantified by GC-ECD with a LOQ of 0.003 mg/kg and LC-MS/MS with a LOQ of 0.01 mg/kg.

Six residue trials in tomatoes were conducted in Mexico. Triforine was quantified by LC-MS/MS with a LOQ of 0.01 mg/kg and GC-ECD with a LOD of 0.005 mg/kg.

One trial in tomatoes was conducted in Denmark Method RU 3, 26/12/10 was used to analyze eggplant fruits samples for the residues of Triforine. Triforine was quantified by GC-ECD.

Five trials in tomatoes were conducted in Japan. Triforine was quantified by GC-ECD with a LOD of 0.005 mg/kg and LC-MS with a LOQ of 0.01 mg/kg.

Table 62 Triforine residues on tomatoes from supervised trials in the USA and Mexico

Tomato, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	For m, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no			
GAP, Mexico	EC 190	–	–	0.380	4	3		Timing: 1 st : first sign of infection Interval: 7 days
USA, 1985 Orlando/FL (–)	EC 190	–	–	0.192	5	0 (12h)	0.032, 0.032, 0.143 mean: 0.069	102FX-532-4303 ¹⁾ TF-723-003 Sampling to analysis: 31–155 days
USA, 1985 Winterville/GA (–)		–	–	0.192	5	0	0.68, 0.54, 0.27 mean: 0.49 c: 0.006, 0.028	
USA, 1985 Elko/SC (–)		–	–	0.192	5	0	0.067, 0.079, 0.065 mean: 0.070	
USA, 1985 Geneseo/IL (–)		–	–	0.179	5	0	0.052, 0.11, 0.091 mean: 0.085	
USA, 1985 Fremont/OH (–)		–	–	0.192	5	0 (4h)	0.025, 0.063, 0.093 mean: 0.060	
USA, 1985 Tudor/CA (–)		–	–	0.192	5	0	0.26, 0.18, 0.25 mean: 0.23	
USA, 1985 Uvalde/TX (–)		–	–	0.192	5	0	0.19, 0.063, 0.098 mean: 0.12	
USA, 1985 Fresno/CA (–)	EC 190	–	–	0.183	5	0 3 7 14 21	0.039, 0.027, 0.013 mean: 0.026 0.022, 0.023 mean: 0.023 0.021, 0.022 mean: 0.022 0.010, 0.011 mean: 0.011 0.011, 0.013 mean: 0.012	102FX-532-4303 ¹⁾ TF-723-003 Sampling to analysis: 174–218 days
USA, 1985 Phelps/NY (–)		–	–	0.192	5	0 3 7 14 21	0.085, 0.038, 0.079 mean: 0.067 0.038, 0.030 mean: 0.034 0.029, 0.021 mean: 0.025 0.011, 0.009 mean: 0.010 0.008, 0.009 mean: 0.009	
Mexico, 2010 Chaves Talamanda /Sinaloa (Toro/Brigade/ UG194-06)	DC 190	–	494–510	0.386–0.398	4	3	0.085, 0.081 mean 0.083	810.1500-10-479- 15B-01-22 Sampling to analysis: 30–68 days
Mexico, 2010 Costencia /Sinaloa (Toro/Brigade/ UG194-06)	DC 190	–	495–518	0.386–0.404	4	3	0.076, 0.12 mean <u>0.096</u>	
Mexico, 2010 Guasave/Sinalo a (Toro/Brigade/	DC 190	–	496–518	0.386–0.405	4	3	0.11, 0.15 mean <u>0.13</u>	

Tomato, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	For m, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no			
UG194-06)								
Mexico, 2010 Meneadero /Baja California (Alto May 0142/Torres)	DC 190	–	504–517	0.394–0.403	4	3	0.28, 0.26 mean <u>0.27</u>	
Mexico, 2010 Colnett/Baja California (Alto May 0142/Torres)	DC 190		500–515	0.390–0.401	4	3	0.39, 0.40 mean <u>0.40</u>	
			490–505	0.383–0.394	4	3	0.28, 0.21 mean <u>0.25</u>	
			482–509	0.375–0.397	4	3	0.21, 0.21 mean <u>0.21</u>	
USA, 2010 Yuma/AZ (Camelia/Yaqui/ Giante Verde)	DC 190	–	455–480	0.381–0.402	4	3	0.087, 0.11 mean <u>0.096</u>	
			465–477	0.390–0.398	4	3	0.059, 0.060 mean <u>0.059</u>	
			410–476	0.343–0.400	4	3	0.21, 0.36 mean <u>0.28</u>	
USA, 2010 Thermal/CA (Yaqui)	DC 190		467–488	0.386–0.410	4	3	0.16, 0.18 mean <u>0.17</u>	
USA, 2010 Yuma/AZ (Mountain Fresh)	DC 190	–	453– 480	0.379–0.403	4	3 5 7 8 12	0.091, 0.052 mean <u>0.071</u> 0.019, 0.038 mean <u>0.028</u> 0.039, 0.049 mean <u>0.042</u> 0.039, 0.045 mean <u>0.042</u> 0.014, 0.025 mean <u>0.020</u>	
Mexico, 1978 Culiacan, Sin (Walter)	DC 190	–	160	0.285	3	0 3 7 14	0.13 0.066 0.030 0.034	102FX-532-3108 TF-701-004 Sampling to analysis: 352–373 days

Analytical portion: whole fruit

^a The details of the analytical method were not shown in the study report.

Table 63 Triforine residues on tomatoes from supervised trials in Denmark

Tomato, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no			
Denmark, 1988 – (–)	EC 190	–	5000	0.950	1	1 3 5 7	0.39 0.48 0.30 0.23	102FX-532-4306 TF-723-006 Sampling to analysis: 435–442 days

Analytical portion: fruit

Table 64 Triforine residues on tomatoes from supervised trials in Japan

Tomato, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
GAP, Japan	EC 180	0.018	1000–3000	–	3	1		
Japan, 2008 Ushiku/Ibaraki (Momotaro T93) Greenhouse	EC 150	0.015	3000	0.450	1	1 3 7 14	0.09 0.09 0.11 0.10	Sampling to analysis: 30–68 days
					3	1 3 7 14	0.26 0.30 0.30 0.17	
					1	1 3 7 14	0.16 0.16 0.18 0.14	
					3	1 3 7 14	0.56 0.67 0.42 0.29	
Japan, 2008 Miyazaki/Miyazaki (House Taro) Greenhouse	EC 150	0.015	3000	0.450	1	1 3 7 14	0.16 0.16 0.18 0.14	
Japan, 1986 Mito/Ibaraki (First memory) Greenhouse	EC 150	0.015	3000	0.450	3	1	0.17	Sampling to analysis: 62–123 days
					3	1	0.28	
					3	1	0.14 c: 0.007	
Japan, 1986 Yatsushiro/Kumamoto (First) Greenhouse	EC 150	0.015	3000	0.450	3	1	0.14 c: 0.007	

Analytical portion: fruit

*Legume vegetables**Common bean*

Four residue trials in beans were conducted in Brazil. Triforine in pods and seeds was quantified by GC-ECD with a LOQ of 0.01–0.02 mg/kg.

One trial in beans was conducted in South Africa. Triforine in pods was quantified by GC with a LOQ of 0.01 mg/kg.

Table 65 Triforine residues on beans from supervised trials in Brazil

Bean, country, year (variety)	Application					Analytical portion	DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.				
GAP, Brazil	EC 190	–	400–1000	0.285	3	–	10		Timing: 1 st : early symptoms Interval: 7–10 days
Brazil, 1985 Cambui String beans (–)	EC 190	–	–	0.285	5	Pods	1 3 4 5 6	1.9 0.4 0.6 0.2 0.2	TF-720-001 ^b Sampling to analysis: 18–23 days
				0.570			1 3 4 5	4.0 0.9 0.9 0.7	

Bean, country, year (variety)	Application					Analytical portion	DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.				
							6	0.4	
Brazil, 1984 Prudente (-)	EC 190	—	—	0.285	3	Seeds	21	< 0.01	102FX-532-4003 ^a TF-720-003 Sampling to analysis: 213 days
Brazil, 1985 Guaira (Carioca)	EC 190	—	—	0.285	4	Seeds	28	< 0.01	TF-720-004 Sampling to analysis: 134 days
				0.570			28	< 0.01	
Brazil, 1996 Tupaciguar (-)	EC 190	—	—	0.285	3	Seeds	10	< 0.01	BASF 1996/306188
				0.570			10	< 0.01	

^a The details of the in-field study were not shown in the study report.

^b The details of the analytical method were not shown in the study report.

Table 66 Triforine residues on beans from supervised trials in South Africa

Bean, country, year (variety)	Application					Commodity	DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.				
GAP, South Africa	EC 190	28.5	1000	0.285	NS	—	3		Timing: 1 st : first sign of infection Interval: 7–10 days
South Africa, 1976 Transvaal (Gelatin)	EC 190	—	—	0.285	4	Pods	0	1.2	TF-720-005 ^{a, b} Sampling to analysis: 121–131 days
							3	0.44	
							5	0.44	
							7	0.34	
							10	0.01	

^a The details of the in-field study were not shown in the study report.

^b The details of the analytical method were not shown in the study report.

Cereal grains

Barley

Two residue trials in barley were conducted in France. Method RU 3, 26/12/10 was used to analyze barley grain samples for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.01 mg/kg.

Table 67 Triforine residues on barley from supervised trials in France

Barley, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
France, 1979 - (Lely)	Vereor Multi (100 g ai/L)	0.05	500	0.250	1	64	< 0.01	10283-532-0102 ^a TF-730-024 Sampling to analysis: 146 days
France, 1979 (Astrix) (Six-row)	Vereor Multi (100 g ai/L)	0.05	500	0.250	1	50	< 0.01	10283-532-0103 TF-730-025 Sampling to analysis: 174 days

Analytical portion: grains

^a The details of the in-field study were not shown in the study report.

Wheat

Total three residue trials in wheat were conducted in Austria, France and UK. Method RU 3, 26/12/10 was used to analyze wheat grain samples for the residues of Triforine. Triforine in grains was quantified by GC-ECD with a LOQ of 0.01–0.02 mg/kg.

Two residue trials in wheat were conducted in Brazil. Triforine in grains was quantified by GC-ECD with a LOQ of 0.01 mg/kg.

Table 68 Triforine residues on wheat from supervised trials in Austria, France and UK

Wheat, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
Austria, 1980 – wheat (–)	Vereor Multi (100 g ai/L)	–	–	0.200	1	30	< 0.01	10288-532-0003 ^a TF-730-003 Sampling to analysis: 102 days
France, 1980 – (Lutin)	Vereor Multi (100 g ai/L)	0.05	500	0.250	2	61	< 0.01	10283-532-0004 ^a TF-730-004 Sampling to analysis: 150 days Analytical method: RU 3, 26/12/10
UK, 1977 Abbenes (Lely)	Funginex (200 g ai/L)	0.06	500	0.300	1	72	< 0.01 (4)	102FX-532-0014 ^a TF-730-013 Sampling to analysis: 150 days Analytical method: RU 3, 26/12/10

Analytical portion: grains

^a The details of the in-field study were not shown in the study report.

Table 69 Triforine residues on wheat from supervised trials in Brazil

Wheat, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
Brazil, 1978 Município de Paicandu-Pr. (BH1146)	EC 190	–	200	0.285	3	–	< 0.01	102FX-532-0015 ^a TF-730-014
Brazil, 1984 Guaira/SP (–)	EC 190	–	400	0.285–0.380	4	30	< 0.01	102FX-532-0016 TF-730-015 Sampling to analysis: 134 days Analytical method: RU 3, 26/12/10

Analytical portion: grains

^a The details of the analytical method were not shown in the study report.

*Straw, fodder and forage of cereals**Barley straw and forage*

Two residue trials in barley straw and forage were conducted in France. Method RU 3, 26/12/10 was used to analyze barley straw and forage for the residues of Triforine. Triforine was quantified by GC-ECD with a LOQ of 0.04–0.07 mg/kg.

Table 70 Triforine residues on barley straw from supervised trials in France

Barley,	Application	DAT	Residues,	Ref

country, year (variety)	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.	Days	mg/kg	
France, 1979 – (Lely)	Vereor Multi (100 g ai/L)	0.05	500	0.250	1	64	0.07	10283-532-0102 ^a TF-730-024 Sampling to analysis: 146 days Analytical method: RU 3, 26/12/10
France, 1979 Astrix (Six- row)	Vereor Multi (100 g ai/L)	0.05	500	0.250	1	50	0.08	10283-532-0103 ^a TF-730-025 Sampling to analysis: 174 days Analytical method: RU 3, 26/12/10

Analytical portion: straw

^a The details of the in-field study were not shown in the study report.

Wheat straw and forage

Total two residue trials in wheat straw and forage were conducted in France and UK. Method RU 3, 26/12/10 was used to analyze wheat straw and forage for the residues of Triforine. Triforine in grains was quantified by GC-ECD with a LOQ of 0.01–0.04 mg/kg.

Table 71 Triforine residues on wheat straw from supervised trials in France and UK

Wheat, country, year (variety)	Application					DAT Days	Residues, mg/kg	Ref
	Form, g ai/L	kg ai/hL	water, L/ha	kg ai/ha	no.			
France, 1980 – (Lutin)	Vereor Multi (100 g ai/L)	0.05	500	0.250	2	61	0.04	10283-532-0004 ^a TF-730-004 Sampling to analysis: 150 days
UK, 1977 Abbenes (Lely)	Funginex (200 g ai/L)	0.06	500	0.300	1	72	0.05,0.07, 0.06,0.07 Mean:0.06	102FX-532-0014 ^a TF-730-013 Sampling to analysis: 150 days

Analytical portion: straw

^a The details of the in-field study were not shown in the study report.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In Processing

The Meeting received information on the fate of triforine residues during the processing of plums, grapes, tomatoes and hops.

The Meeting did not receive information on supervised field trials with hops. Therefore, the study on processing of hops is not summarized in this report.

Plum

The plum samples were treated with formulation containing triforine as active ingredient. The analytical material was shipped under deep-frozen conditions. The samples were stored in a deep-freeze compartment in the dark. The plum samples were heated to make processed commodities (compote, stewed and jam).

The samples were blended with acetone. After removal of the acetone by distillation, triforine in the remaining aqueous phase was partitioned into toluene. The toluene was evaporated and the

active substance degraded by heating with diluted sulfuric acid. Chloral hydrate thus formed was distilled and determined by GC-ECD (RU 3, 26/12/10).

Table 72 Triforine residues in processed commodities of plums

Country, year	Application				DAT Days	Commodity	Residues		Ref
	kg ai/hL	water, L/ha	kg ai/ha	no.			mg/kg	PF	
Germany, 1990	0.028	1500	0.427	4	7	Fruit Compote	0.22 0.06	0.27	TF-712-091
				3	7	Fruit Compote Jam	0.11 0.10 0.10	0.91 0.91	
Germany, 1992	–	–	0.428	2	7	Fruit Dried (prunes) Jam	0.15 0.12 0.02	0.80 0.13	SHTR.93.004 TF-712-088
Germany, 1991	–	–	0.428	3–4	7	Fruit Stewed Dried plums	0.048 < 0.01 0.023	< 0.21 0.48	10249-532-2312 TF -712-089

Grapes

The samples were blended with acetone. After removal of the acetone by distillation, triforine in the remaining aqueous phase was partitioned into toluene. The toluene was evaporated and the active substance degraded by heating with diluted sulfuric acid. Chloral hydrate thus formed was distilled and determined by GC-ECD (RU 3, 26/12/10). The LOQ was 0.01 mg/kg. Procedural recoveries were 90% for fruits, 111% for juice and 95% for wine, respectively.

Table 73 Triforine residues in processed commodities of grapes

Country, year	Application				DAT Days	Commodity	Residues		Ref
	kg ai/hL	water, L/ha	kg ai/ha	no.			mg/kg	PF	
Germany, 1984	0.057	887	0.505	1	21	Fruit	0.65		10238-532-3012 TF -713-012
						Juice	0.20	0.31	
						Wine	0.09	0.14	

Tomato

Study 1

Fresh tomatoes were dipped in a spray wash of EC formulation solution diluted to 1.3% or 4%. After treatment the fruits were dried under room temperature for one day. Five kg of treated tomatoes were homogenized and the juice was separated by a suction filter. 300 g juice was concentrated by freeze drying to water content of 16% for purée, 28% for ketchup and 73% for dehydrated pulp.

Fifty grams of chopped tomatoes, 50 g juice or the whole quantity of each material were extracted with acetone. The solvent was removed and the residues partitioned into toluene. The organic phase was separated and evaporated. After addition of sulphuric acid, triforine was decomposed in the heat. Chloral hydrate formed was distilled and determined by GC-ECD.

Table 74 Triforine residues in processed commodities of tomatoes

Country, year	Application	Commodity	Residues		Ref.
			mg/kg	PF	
Germany, 1982	Dipped in 1.3% solution	tomatoes	3.4		TF-790-019 Eichler, 1982
		juice	2.6	0.76	
		puree	8.9	2.6	
		ketchup	15.6	4.6	
		dehydrated pulp	36.3	11	

Country, year	Application	Commodity	Residues		Ref.
			mg/kg	PF	
	Dipped in 4% solution	tomatoes	8.8		
		juice	6.5	0.74	
		puree	20.6	2.3	
		ketchup	36.9	4.2	
		dehydrated pulp	96.4	11	

Study 2

At two locations (California and New York) processing tomatoes were treated at the normal application rate (0.183 kg ai/ha) and five times rate (0.964 kg ai/ha) and processed into tomato juice, wet pomace, dry pomace, paste or puree, and ketchup in order to determine possible residues in these commodities. Field samples were received in good condition frozen in dry ice and transferred to freezer at -20 °C.

The samples were washed and chopped in mechanical slicer. The tomato juice was preheated at 82.2 °C, sterilized at 126.6 °C and filled at 87.7 °C with 60 g salt tablet in can. Tomato juice was boiled in a vacuum kettle for 2.5 hours with constant stirring to prepare paste.

The residues were extracted using standard method via Chloral hydrate formation and analysis determined by GC-ECD.

Table 75 Triforine residues in processed commodities of tomatoes

country, year	Application		DALA Days	Commodity	Residues, mg/kg		Ref
	kg ai/ha	no.			mg/kg	PF	
USA, 1986 Fresno/CA	0.183	5	0	Tomatoes	0.039, 0.027, 0.013 mean:		102FX-532- 4303 TF-723-003, TF-790-020 Beevers and McLellan, 1986
				Juice	0.026	< 0.12	
				Ketchup	< 0.003, < 0.003 mean < 0.003	< 0.12	
				Paste	< 0.003, < 0.003 mean < 0.003	< 0.12	
				Puree	< 0.003, < 0.003 mean < 0.003	< 0.12	
				Wet Pomace	< 0.003, < 0.003 mean < 0.003	< 0.12	
				Dry Pomace	< 0.003, < 0.003 mean < 0.003 0.033, 0.048 mean 0.041	1.6	
USA, 1985 Geneva/NY	0.964	5		Juice	0.047, 0.051, 0.053, 0.057		
				Ketchup	0.039, 0.040, 0.054, 0.068		
				Paste	0.15, 0.12, 0.095, 0.082		
				Puree	0.096, 0.092, 0.10, 0.095		
				Wet Pomace	6.2, 2.9		
				Dry Pomace	7.3, 7.5, 4.8, 3.7		

Study 3

The test substance, formulated as a dispersible concentrate (190 g triforine/L), was applied four times as a foliar treatment at an exaggerated (5×) application rate. The target application rate for each event was 1.95 kg ai/ha. Bulk tomato samples were shipped ambient to the processing facility, where tomato puree and paste were produced. Those samples were shipped frozen to the analytical laboratory and maintained in frozen storage until analysis. Following extraction and clean-up, triforine residues were separated and measured using HPLC-MS/MS. The LOQ was defined as 0.01 mg/kg. The overall mean procedural recovery was 77%, with a RSD of 8.8%.

Table 76 Triforine residues in processed commodities of tomatoes from supervised trials

Country, year (variety)	Application			DALA Days	Commodity	Residues		Ref.
	kg ai/ha	water, L/ha	no .			mg/kg	PF	
USA, 2010 Yuma/AZ (6366)	1.96	564	4	3	Tomato RAC Puree Paste	1.0, 1.3 mean 1.2	0.14 < 0.008	47915A003 Hummel, 2011 Sampling to analysis: 71-125 days
	1.94	557				0.15, 0.19 mean		
	1.92	550				0.17		
	1.93	555				< 0.01, < 0.01		

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

The Meeting received a lactating goat feeding study.

Lactating goat

The study was designed to determine the residues of triforine found in milk and tissues following oral administration to lactating goats (Eichler, 1974: TF-440-016). Three groups of goats, each group containing three animals, were orally dosed with triforine for 30 days at dosages of approximately 5, 15 and 50 mg triforine per animal per day. This would be equivalent to total dietary residues of approximately 5, 15 and 50 ppm feed. Dose levels were based on a nominal feed intake of 1 kg (dry matter equivalent) per day for a goat. The concentrate mixture with flour containing triforine was used for administration.

Milking was done daily at morning and evening. The pooled samples of day 3, 5, 8, 11, 15, 22, 29 and of day 31 were stored deep frozen (-20 °C) until analysis. At the morning of day 31, all goats were sacrificed and the edible tissues (liver, kidney, muscle and fat) from each animal were collected. All samples were stored at -20 °C until they were analyzed. The storage period was between 1 day and 4 weeks.

The analytical procedures consider both triforine and those metabolites containing Cl₃C-CH group. The results were expressed as triforine equivalents and corrected with recoveries. The chopped samples were extracted with acetone and the extracts were concentrated by rotary evaporation. The remaining aqueous phase was mixed with sulphuric acid and distilled under nitrogen. To the aqueous solution of chloral hydrate formed, sodium chloride was added. The chloral hydrate was extracted with ethyl formate and quantified as chloral hydrate using gas chromatography with an electron capture detector. The LOQ of this method in milk and tissues was 0.001 mg/L and 0.003 mg/kg, respectively. Concurrent recoveries obtained from control samples spiked with the test item were reported to be 80 ± 10%.

No residues were detected in milk from the lowest dose level with the exception of two animals on day 29 (traces only considered as outliers). Residues in milk from group of 15 ppm feed were at the level of 0.001–0.003 mg/L on sampling days 8, 22 and 29. The milk collected on the other days did not contain detectable residues. At the high dose group (50 ppm feed), residue levels reached a plateau at the level of 0.002–0.010 mg/L after 3 days. One day after the last administration, no residues were detected in milk. The results indicate no increase of the residues during the treatment phase and a rapid decline in levels detected in milk during the last part of the withdrawal period.

Table 77 Residues of triforine and those metabolites containing Cl₃C-CH group in whole milk

Day	Residues, mg/L			
	Control	5 ppm feed	15 ppm feed	50 ppm feed
3	< 0.001 (3)	< 0.001 (3)	< 0.001 (3)	0.006, 0.008, 0.010 mean 0.008
5	< 0.001 (3)	< 0.001 (3)	< 0.001 (3)	0.002, 0.003, 0.005 mean 0.003
8	< 0.001 (3)	< 0.001 (3)	0.001, 0.002, 0.003	0.004, 0.004, 0.006

Day	Residues, mg/L			
	Control	5 ppm feed	15 ppm feed	50 ppm feed
			mean 0.002	mean 0.005
11	< 0.001 (3)	< 0.001 (3)	< 0.001 (3)	0.004, 0.004, 0.004 mean 0.004
15	< 0.001 (3)	< 0.001 (3)	< 0.001 (3)	0.003, 0.003, 0.003 mean 0.003
22	< 0.001 (3)	< 0.001 (3)	0.001, 0.001, 0.003 mean 0.002	0.005, 0.005, 0.007 mean 0.006
29	< 0.001 (3)	< 0.001, 0.002, 0.003 mean 0.002	0.001, 0.002, 0.002 mean 0.002	0.005, 0.005, 0.007 mean 0.006
31	< 0.001 (3)	< 0.001 (3)	< 0.001 (3)	< 0.001 (3)

In tissues, no significant residues were detected in the fat, liver, kidney and muscle of 5 ppm and 15 ppm feed groups except several samples from 15 ppm feed animals exceeding slightly the LOQ. In the 50 ppm feed group, residues were detected in all of the analyzed tissues with the exception of muscle in an animal and fat of all animals. The maximum values in kidney and liver were 0.009 and 0.012 ppm, respectively.

Table 78 Residues of triforine and those metabolites containing Cl₃C-CH group in tissues

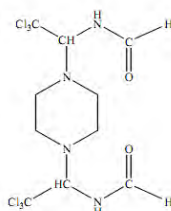
Group	Residues, mg/kg			
	Liver	Kidney	Muscle	Fat
Control	< 0.003 (3)	< 0.003, 0.007, 0.009 mean 0.006	< 0.003 (3)	< 0.003 (2), 0.011 mean 0.006
5 ppm feed	< 0.003 (3)	< 0.003 (3)	< 0.003 (3)	< 0.003 (3)
15 ppm feed	< 0.003 (2), 0.004 mean 0.003	< 0.003 (2), 0.006 mean 0.004	< 0.003, 0.003, 0.005 mean 0.004	< 0.003 (3)
50 ppm feed	0.004 (2), 0.012 mean 0.007	0.003, 0.005, 0.009 mean 0.006	< 0.003 (2), 0.005 mean 0.005	< 0.003 (3)

APPRAISAL

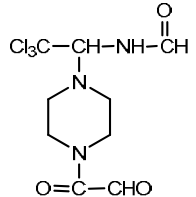
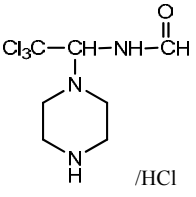
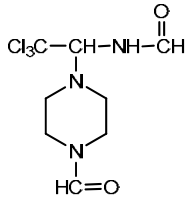
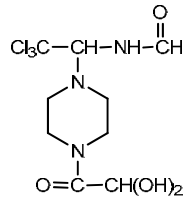
Triforine is a systemic fungicide for control of powdery mildews, rusts, scabs and rots. It was first evaluated by JMPR in 1977 (T), 1978 (T, R) and lastly in 1997 (T). The ADI for Triforine was 0–0.02 mg/kg bw and no ARfD was recommended by the previous JMPR. Triforine was scheduled at the Forty-fifth Session of the CCPR (2013) for the periodic re-evaluation of toxicity and residues by the 2014 JMPR.

The Meeting received information on identity, physical and chemical properties, animal and plant metabolism, environmental fate in soil, analytical methods, storage stability, use patterns, supervised trials, farm animal feeding studies and fates of residues in processing.

N,N'-{piperazine-1,4-diylbis[(trichloromethyl)methylene]} diformamide



In this appraisal, the following abbreviated names were used for metabolites.

WOS 2379	W 1084/W 1069	W 625	W 2379
			
<i>N</i> -[2,2,2-trichloro-1-[4-(2-oxoacetyl)piperazin-1-yl]ethyl]formamide	<i>N</i> -(2,2,2-trichloro-1-piperazin-1-yl-ethyl)formamide/hydrochloride	<i>N</i> -[2,2,2-trichloro-1-(4-formylpiperazin-1-yl)ethyl]formamide	Hydrate of <i>N</i> -[2,2,2-trichloro-1-[4-(2-oxoacetyl)piperazin-1-yl]ethyl]formamide

Animal metabolism

The Meeting received animal metabolism studies with triforine in rats, lactating goat and laying hens. The metabolism and distribution of triforine in animals were investigated using the [^{14}C -piperazine] and [^{14}C -side chain]-labelled triforine.

Metabolism in rats was summarized and evaluated by the WHO panel of the JMPR in 2014.

Triforine is rapidly metabolized and excreted in rats. Highest residues were found in liver followed by kidney. Residues were lower in muscle and fat. Radioactive residues were identified in the excreta only. *N*-[2,2,2-trichloro-1-(piperazin-1-yl)ethyl]-formamide (W 1084), which is formed by the cleavage of a side chain, was the major component in urine of rats. It was excreted in urine as the glucuronide. The side-chain metabolites trichloroethanol including its glucuronide and *N*-acetylcysteine conjugate of 2,2,2-trichloroethylamine was detected in urine. W 1084 and triforine were detected in the faeces of rats.

Lactating goats were administered with [piperazine- ^{14}C]-triforine as an oral dosage equivalent to a dietary level of 250 or 1000 ppm once daily for 3 consecutive days. The goats were sacrificed 4 hours or 6 days after the last treatment.

Radioactivity administered to the goats was rapidly eliminated in urine and faeces. A total of 47% and 72% of the applied radioactivity were eliminated in 24 hours, and a further 16 and 14% were excreted in the following 5 days by the 250 ppm and the 1000 ppm dose group goats, respectively.

Up to 68% of the residual radioactivity in the liver of goat sacrificed 4 hours after the last treatment was extracted. The extracted radioactivity consisted of at least five metabolite fractions. M1 and M2 represented unknown polar metabolite fractions whereas WOS 2379 and W 1084 were characterized. The fraction M1 represented 18% TRR. Triforine, WOS 2379 and W 1084 accounted for 15%, 15% and 13% TRR, respectively. Up to 14% of the residual radioactivity in the liver of goat sacrificed 6 days after the last treatment was extracted. This metabolite pattern was similar to that in the extracted radioactivity of the liver of goat sacrificed 4 hours after the last treatment.

Seventy-eight percent of the residual radioactivity in the kidneys of goats sacrificed 4 hours after the last treatment was extracted. The metabolite pattern of the extracted radioactivity was similar to that in the extracted radioactivity of the liver. The predominant fraction found in the extracted radioactivity of goat sacrificed 4 hours after the last treatment was metabolite fraction M1 (31% TRR). Triforine, WOS 2379 and W1084 represented 19%, 8.4% and 19% TRR, respectively. Analysis of the extracted radioactivity of the kidneys of a goat sacrificed 6 days after the last treatment showed a similar metabolite pattern in terms of their relative amounts in the extracts. The predominant fraction was M1 accounting for 11% TRR. Triforine represented 3.6% TRR.

Seventy-nine percent of the residual radioactivity in the muscle of goat sacrificed 4 hours after the last treatment was extracted. The predominant radioactive fraction was triforine at 41% TRR. The other metabolites accounted for 9.5% (W 1084), 13% (WOS 2379), 0.70% (M2) and 15% TRR (M1).

In one study, laying hens were orally administered with [piperazine-¹⁴C]-triforine at the dietary dose equivalent to 500 or 2000 ppm in the feed once daily for 3 consecutive days. The hens were sacrificed 4 hours or 7 days after the last treatment.

The radioactivity administered to the hens was rapidly eliminated (54–84% in 56 hours after the first dose). In the following 7 days a further 10–15% of the administered radioactivity was excreted (76% excreted from 500 ppm dosed hens and 94% from 2000 ppm dosed hens).

The highest values in eggs were found about 4 to 5 days after the first treatment with [¹⁴C] triforine.

In the other study, laying hens were administered with [side chain-¹⁴C]-triforine for 10 consecutive days at a dose of 32 ppm in the feed. Radioactivity recovered in excreta during the 10 days accounted for about 85% of the total cumulative dose.

Total radioactivity in eggs increased steadily during the 10 days to a peak value of 1.6 mg equiv/kg (yolk) and 0.19 mg equiv/kg (white). The major components in egg white and egg yolk were the fraction A which accounted for 48% TRR (0.08 mg/kg) and the sulphate conjugate of trichloroethanol which accounted for 25% TRR (0.25 mg/kg), respectively. W 1069 was observed and accounted for 10% TRR (0.10 mg/kg) in egg yolk. Triforine accounted for 13% TRR (0.02 mg/kg) and 2.1% TRR (0.02 mg/kg) in egg white and egg yolk, respectively.

The fraction A in the protease-treated extract of liver was separated into five components each of which accounted for 0.07–0.40 mg equiv/kg (4–24% TRR). The sulphate conjugate of trichloroethanol was present in liver accounting for 9% TRR. There appeared to be a small amount of triforine in liver (2.9% TRR, 0.05 mg/kg).

The main components in muscle were the trichloroethanol sulphate conjugate and W 1069 each accounting for about 22% TRR (0.05 mg equiv/kg). Triforine was present at 8.4% TRR (0.02 mg/kg).

Triforine accounted for 18% TRR (0.02 mg/kg) and the sulphate conjugate of trichloroethanol accounted for 36% TRR (0.03 mg equiv/kg) in fat.

Triforine was also found in skin at low levels (0.01 mg/kg). The retention time of the main fraction corresponded to that of trichloroethanol sulphate (56% TRR, 0.13 mg/kg).

In animal metabolism studies, triforine, W 1084/W 1069, WOS 2379 and trichloroethanol sulphate were predominantly found in tissues of lactating goats and laying hens. The major component in milk and egg white was the polar fraction consisting of several components but they were not identified. Triforine was identified in egg white and egg yolk. Excretion, distribution and triforine and its metabolites found in excreta of lactating goats and laying hens were similar to those in rats.

Plant metabolism

The Meeting received plant metabolism studies performed on apples, tomatoes and cucumber with triforine ¹⁴C-labelled in two carbons at the side chain, and on barley with triforine ³H-labelled at piperazine ring ([side chain-¹⁴C] and [piperazine-³H]).

In an outdoor apple metabolism study, a number of fruits or leaves of apple were treated at random on the surface with [side chain-¹⁴C]-triforine at a rate of 1.2 g ai/L in a series of small droplets. Treated apple fruits were harvested 2 weeks after the last of five successive applications with 8-day intervals. After five successive applications of [¹⁴C] triforine, 32% (fruit) and 22% (leaf) of the applied radioactivity were recovered. On an average, 1.36 mg equiv/kg was recovered in the treated fruits.

The major component in the surface washes and extracts of fruits was identified as triforine accounted for 73–79% TRR (0.88–1.2 mg/kg) two weeks after the last application. Several minor components were observed in the extracts and each of them accounted for 1–2% TRR.

In an indoor tomato metabolism study, a number of fruits or leaves of tomato were treated at random on the surface with [side chain- ^{14}C]-triforine at a rate of 1.2 g ai/L in a series of small droplets. The treated tomatoes were harvested at 2 hours and 3 days after the last of four successive applications with 8–10 days intervals. The initial surface washes of treated tomatoes at harvest contained, on an average, 92% (2 hours after the last application) and 91% (3 days after the last application) of TRR. Acetonitrile extracts of washed and homogenised tomatoes accounted for, on an average, 5.8% TRR (2 hours after the last application) and 6.2% TRR (3 days after the last application). The TRR from the treated tomatoes accounted for, on an average, 16 (2 hours after the last application) and 9.7 (3 day after the last application) mg equiv/kg.

Triforine in the surface washes and extracts accounted for 91–93% TRR (7.6–19 mg/kg) in tomatoes taken at 2 hours and 3 days after the final application of [^{14}C] triforine. The extracts also contained several minor components each accounting for, on an average, 0.05–1.1% TRR.

In an indoor cucumber metabolism study, a number of fruits or leaves of cucumber were treated at random on the surface with [side chain- ^{14}C]-triforine at a rate of 1.2 g ai/L in a series of small droplets. The treated cucumbers were harvested 3 days after the last of four successive applications at 7-day intervals. The surface washes of treated cucumbers at harvest contained, on an average, 85% of TRR. Extracts of washed and homogenised cucumber peel and flesh accounted for, on an average, 7.5% TRR (peel) and 1.4% TRR (flesh). The TRR from the treated cucumbers accounted for, on an average, 2.2 mg equiv/kg.

The major radioactive component was identified as triforine in the surface washes and extracts accounted for 87–88% TRR (1.9 mg/kg) in cucumber taken 3 days after the final application of [^{14}C] triforine. The extracts also contained several minor components each accounting for means of 0.3–2% TRR.

In the first indoor barley metabolism study, [piperazine- ^3H]-triforine was applied to barley plants grown in plastic pots as soil drenching. The leaves were harvested at 15 and 30 days after treatment.

Triforine was identified as a major component in the barley leaves, amounting to 58% TRR at 15 days after the treatment and 43% TRR at 30 days after the treatment. W 1084 was also observed in the 0.1M HCl extract (8.4–13% TRR).

In the second indoor barley metabolism study, the leaves of barley plants root-treated with [piperazine- ^3H]-triforine were collected 30 days after treatment. The major component was identified as triforine, accounted for 45% TRR, and W 1084 and piperazine were also observed.

In the third outdoor barley metabolism study, the plants (during the stem extension stage when the second node of the stem was formed and the next-to-last leaf was just visible) were sprayed with an aqueous emulsion of a mixture of the commercial formulation of triforine and [piperazine- ^3H]-triforine at a rate of 0.25 kg ai/ha. Barley was harvested when ripe, and straw and grain were analysed separately.

The methanol soluble radioactive residue contained triforine and its metabolites which were free in barley straw and grain. Triforine accounted for 0.034 mg/kg (18% TRR) in straw and 0.0018 mg/kg in grain (13% TRR). W 1084 was identified as a minor component. Two other radiolabelled components were identified in straw: glycine at 0.043 mg/kg (33% TRR) and iminodiacetic acid at 0.021 mg/kg (17% TRR), respectively.

In the plant metabolism studies, triforine was the major component of the residues found in all plants studied.

Environmental fate

The Meeting received information on aerobic degradation in soil, photolysis on soil surface and hydrolytic degradation study.

In soil under the aerobic conditions, the DT_{50} ranged from 1–70 days at 20 °C. Many minor degradation products were detected in the extracts during the study. Most of the radioactivity was

recovered from natural components. Mineralization was up to 45%. Minor degradates were identified as W 625, WOS 2379, piperazine and W 1069, but all of them were less than 3% TAR.

In soil photolysis study, the degradation was biphasic. The photodegradation half-life of triforine was 11 hours of artificial sunlight or 0.5 natural sunlight days for phase 1 (hours 0 to 8). For phase 2 (hours 8 to 48), the half-life was 71 hours of artificial sunlight equivalent to 3.2 natural sunlight days.

In summary, triforine was rapidly and completely degraded in soil and is unlikely to be taken up by crops from the soil after soil treatment.

Methods of analysis

The Meeting received description of validation data on analytical methods for residues of triforine in plant and animal commodities.

In most of the methods for the determination of triforine in plant, homogenized samples were extracted with acetone, and the extract was partitioned into organic solvent and the active substance was degraded by heating with dilute sulphuric acid. Chloral hydrate thus formed was determined by GC-ECD. The methods of analysis with GC-ECD for a range of matrices were validated with acceptable recoveries with the LOQs of 0.01 mg/kg for triforine.

New methods using LC-MS or LC-MS/MS were developed for analysing triforine directly. In the methods, homogenized samples were extracted with acetone, and the extract was purified with SPE cartridge clean-ups. The methods of analysis with LC-MS or LC-MS/MS for a range of matrices were validated with acceptable recoveries with the LOQs of 0.01 mg/kg except for tomato paste for which the LOQ was 0.05 mg/kg.

In the methods for animal commodities, homogenized samples were extracted with acetone, or were diluted with water. Triforine and possible metabolites containing the $\text{Cl}_3\text{C-CH}$ group in the acetone extract or diluted homogenate were degraded by heating with dilute sulphuric acid. Chloral hydrate thus formed was determined by GC-ECD. These methods were validated with acceptable recoveries with the LOQ of 0.001–0.003 mg/kg for milk and animal tissues, and 0.01 mg/kg for egg.

The Meeting was aware that the QuEChRS-multi residue method was validated for most plant matrices with LOQs of 0.01–0.02 mg/kg for triforine.

Stability of residues in stored analytical samples

The Meeting received information on the freezer storage stability of triforine in plant (apples, cherries, plums, peaches, blueberries and hops) and their processed (beer) commodities. Analysis was done by the common moiety method.

Using the common moiety method, storage stability results indicated that residues with common moiety including triforine were stable for at least 1 month in processed hops (beer), at least 6 months in plums and hops (dried cones), at least 12 months in apples, cherries, peaches and blueberries. However, according to the result of plant metabolism study on tomato, triforine seems stable for at least 5 months.

Definition of the residue

In the lactating goat metabolism studies, TRRs in liver and kidney were higher than those in milk, muscle and fat. Triforine, WOS 2379 and W 1084 accounted for 15%, 15% and 13% TRR in liver, and 19%, 8.4% and 19% TRR in kidney. The polar unknown fraction M1 represented 18% TRR in liver and 31% TRR in kidney. In the laying hen metabolism studies, TRR in liver was also higher than those in other tissues. In the study using [side chain- ^{14}C]-triforine, the trichloroethanol sulphate conjugate and W 1069 were the main components in muscle (22% TRR) and egg yolk (10–25% TRR).

The analytical methods for animal commodities provided determine only the residues of triforine and metabolites containing the $\text{Cl}_3\text{C-CH}$ group converted to chloral hydrate which is formed

by heating in acidic condition. No method of analysis was available for quantification of triforine alone.

The Meeting decided that triforine and its metabolites containing the $\text{Cl}_3\text{C-CH}$ group were suitable analytes for enforcement purposes and dietary risk assessment for animal commodities.

The octanol/water coefficient ($\log P_{ow}$) of triforine was 2.2 at 20 °C. In the lactating goat and the laying hen metabolism studies, triforine and its metabolites found in muscle were 2–100 times higher than those in fat. Fractionation of whole milk showed that 32% of the radioactivity was found in cream and 76% was found in skim milk. In the lactating goat feeding study, triforine and its metabolites were detected at 0.003–0.007 mg/kg in liver, kidney and muscle but less than LOQ (< 0.003 mg/kg) in fat. The Meeting considered the residue of triforine is not fat soluble.

In plant metabolism studies, parent triforine was the major component (43–93% TRR) in apple, tomato, cucumber and barley. Several metabolites identified accounted for $< 10\%$ TRR.

In most of the analytical methods for triforine in plant, since chloral hydrate formed by heating with dilute sulphuric acid was quantified with GC-ECD, triforine and its metabolites containing piperazine ring were simultaneously measured. As the predominant residue was the parent compound in the plant metabolism study, using the common moiety method would result in only slight over-estimation of residues if PHI was short. Recently LC-MS and LC-MS/MS methods were available for determining triforine only.

The Meeting decided that parent triforine was a suitable analyte for enforcement purposes and dietary risk assessment in plant commodities.

The Meeting recommended the following residue definition:

Definition of the residue (for compliance with the MRL and for estimation of dietary intake) for plant commodities: *Triforine*

Definition of the residue (for compliance with the MRL and for estimation of dietary intake) for animal commodities: *Triforine and its metabolites determined as chloral hydrate expressed as triforine*

The residue is not fat soluble

Residue of supervised residue trials on crops

The Meeting received supervised trial data for the foliar application of triforine on apple, pear, cherry, plum, apricot, nectarine, peach, raspberry, blueberry, black currant, grape, cranberry, strawberry, cucumber, squash, melon, peppers, eggplant, tomato, common bean, barley and wheat. Residue trials were conducted in Australia, Brazil, Canada, Denmark, France, Germany, Greece, Hungary, Italy, Japan, Mexico, New Zealand, South Africa, South Korea, the United Kingdom (UK) and the USA. Most of the supervised residue trials employed the common moiety method, while the results of plant metabolism studies showed that triforine was the main component of residues in crops.

Labels were available from a number of countries in North and South America, Africa, Asia and Oceania describing the registered uses of triforine.

Pome fruits

Apple

Data were available from supervised trials conducted on apples in the USA, Canada, Australia, Germany, France and Brazil.

The GAP on apples in Canada was five foliar applications at a maximum rate of 0.475 kg ai/ha between tight cluster and petal fall stage. Trials in the USA and Canada on apples were conducted with foliar applications of EC formulation. Triforine residues in apple from the trials in Canada matching GAP of Canada were (n=1): 0.041 mg/kg.

Trials in Australia on apples were conducted with one to ten foliar applications of EC formulation (GAP: four foliar applications at a maximum spray concentration of 0.023 kg ai/hL with a PHI of 1 day). Triforine residue trials on apples in the Australia did not match the GAP of Australia.

Trials in Germany and France on apples were conducted with foliar applications of EC formulation. No GAP from European countries was available for apple.

The GAP on apples from Brazil was three foliar applications at a maximum concentration of 0.024 kg ai/hL with a PHI of 5 days. Triforine residues in apple from the trials in Brazil matching Brazilian GAP were (n=2): < 0.02 mg/kg (2).

As the data were insufficient for estimating a maximum residue level, the Meeting agreed to withdraw its previous recommendation for apple.

Pear

Data were available from a supervised trial on pears in Australia. No GAP from Australia was available for pear.

The Meeting agreed that estimation of maximum residue level was not possible for pear.

Stone fruits

Cherry

Data were available from supervised trials on cherries conducted in the USA, Canada and Germany.

The GAP on cherries in Canada was for three foliar applications at a maximum spray concentration of 0.014 kg/hL or a rate of 0.475 kg ai/ha between early and full bloom stages. Triforine residue in cherries from the trials in Canada and the USA matching GAP of Canada was (n=1): 0.007 mg/kg.

Trials in Germany on cherries were conducted with foliar applications of an EC formulation. No GAP from European countries was available for cherries.

The Meeting agreed to withdraw its previous recommendation for cherries.

Plum

Data were available from supervised trials on plums conducted in the USA, Canada, Germany, France and South Africa.

The GAP on plums in Canada is for three foliar applications at a maximum spray concentration of 0.014 kg/hL or a rate of 0.475 kg ai/ha between early and full bloom stages. No trials on plums in Canada and the USA matched the GAP of Canada.

Trials in Germany and France were conducted with foliar applications of an EC formulation. No GAP from European countries was available for plums.

The GAP on plums in South Africa was two foliar applications at a rate of 0.87 kg ai/ha with a PHI of 3 days. No trials for plums in South Africa matched the GAP of South Africa.

The Meeting agreed to withdraw its previous recommendation for plums.

Apricot

Data were available from supervised trials on apricots from the USA, France, Greece and Italy.

Trials in the USA on apricots were conducted with one to three foliar applications of an EC formulation at a spray concentration of 0.018 kg ai/hL. No GAP of the USA was available.

Trials in France, Greece and Italy on apricots were conducted with three foliar applications of DC formulation at a spray concentration of 0.038 kg ai/hL. No GAP from European countries was available for apricots.

The Meeting agreed that estimation of maximum residue level was not possible for apricots.

Nectarine

Data were available from supervised trials on nectarines from the USA.

Trials in the USA on nectarines were conducted with one to four foliar applications of an EC formulation at a spray concentration of 0.015–0.018 kg ai/hL. No GAP from the USA was available.

The Meeting agreed that the estimation of maximum residue level was not possible for nectarines.

Peach

Data were available from supervised trials on peaches from the USA, Canada, France, Greece, Japan, Brazil and South Africa.

The GAP on peaches in Canada is three foliar applications at a maximum spray concentration of 0.014 kg/hL or a rate of 0.475 kg ai/ha between early and full bloom stages. Triforine residue in peaches from trials in Canada and the USA matching GAP of Canada was (n=1): 0.02 mg/kg.

The GAP on peaches in Brazil is for three foliar applications at a maximum spray concentration of 0.024 kg ai/hL with a PHI of 3 days. Triforine residues in peaches from trials in Brazil matching GAP were (n=2): < 0.01 and 9.4 mg/kg.

Trials in France and Greece on peaches were conducted with one to eight foliar applications of a DC formulation at a rate of 0.36–0.38 kg ai/ha. No GAP from European countries was available for peaches.

The GAP on peaches in South Africa is two foliar applications at a maximum rate of 0.67 kg ai/ha with a PHI of 3 days. No trials from South Africa on peaches matched the GAP of South Africa.

The GAP on peaches in Japan is five foliar applications at a maximum spray concentration of 0.018 kg ai/hL with a PHI of 1 day. Triforine residues in peaches from the trials in Japan matching GAP of Japan were (n=2): 0.77 and 1.4 mg/kg.

The Meeting considered the data insufficient for the estimation of a maximum residue level, and agreed to withdraw its previous recommendation for peaches.

Berries and other small fruits

Raspberries, Red, black

Data were available from supervised trials on raspberries in France. No GAP from European countries was available for raspberry.

The Meeting agreed that estimation of maximum residue level was not possible for raspberry.

Blueberries

Data were available from supervised trials on blueberries in Canada.

The GAP on blueberries in Canada (except for Eastern Canada) is four foliar applications at a maximum rate of 0.570 kg ai/ha from bud break to 10–14 days after early bloom with a PHI of 60 days. Triforine residues in blueberries from the trials conducted in Canada (except for Eastern Canada) matching this GAP were (n=2): < 0.01 and 0.015 mg/kg.

The GAP for Eastern Canada (only) is three foliar applications at a maximum rate of 0.570 kg ai/ha from leaf-bud break to pink-bud stage with a PHI of 60 days. Triforine residues in blueberries from the trials conducted in Eastern Canada matching this GAP were (n=3): < 0.01(3) mg/kg.

Based on the trials on blueberries in Canada, the Meeting estimated a maximum residue level of 0.03 mg/kg to replace its previous recommendation (1 mg/kg) for blueberry. The Meeting also estimated an STMR and an HR for triforine in blueberry of 0.01 and 0.018 mg/kg, respectively. The highest residue concentration in an individual sample was selected for HR.

Currant, Black

Data were available from supervised trials on black currants in Germany and UK. No GAP from European countries was available for black currants.

The Meeting agreed to withdraw its previous recommendation for black currants.

Grapes

Data were available from supervised trials on grapes from Germany, Mexico and New Zealand.

Trials in Germany on grapes were conducted with one to three foliar applications of an EC formulation at a rate equivalent to 0.28–0.57 kg ai/ha. No GAP from European countries was available for grapes.

Trials in Mexico on grapes were conducted with one foliar application of EC formulation at a rate equivalent to 0.28 kg ai/ha. No GAP from Mexico was available for grapes.

The GAP on grapes in New Zealand is four foliar applications at a rate of at least 0.38 kg ai/ha with a PHI 14 days. No trials in New Zealand on grapes matched the GAP of New Zealand.

The Meeting agreed that estimation of maximum residue level was not possible for grapes.

Cranberry

Data were available from supervised trials on cranberries from the USA.

The GAP on cranberries in Canada was three foliar applications at a maximum rate of 0.570 kg ai/ha with a PHI of 60 days. No trials for cranberries from the USA matching the GAP of Canada were available.

The Meeting agreed that estimation of maximum residue level was not possible for cranberry.

Strawberry

Data were available from supervised trials on strawberries from Mexico, Brazil and Japan.

The GAP on strawberries in Mexico was four foliar applications at a maximum rate of 0.19 kg ai/ha with a PHI of 14 days. Triforine residue in strawberries from the trials in Mexico matching GAP of Mexico was (n=1): 0.57 mg/kg.

The GAP on strawberries in Brazil was four foliar applications at a maximum rate of 0.23 kg ai/ha with a PHI of 2 days. No trials in Brazil on strawberries matched the GAP of Brazil.

The GAP on strawberries in Japan is five foliar applications at a spray concentration equivalent to 0.009 kg ai/hL with a PHI of 1 day. Triforine residues in strawberries from the trials in Japan matching GAP of Japan were (n=4): 0.20, 0.39, 0.48 and 0.78 mg/kg.

The Meeting considered that the data was insufficient for estimating a maximum residue level, the Meeting agreed to withdraw its previous recommendation for strawberry.

Gooseberry

No supervised trials on gooseberry were available.

The Meeting agreed to withdraw its previous recommendation for gooseberry.

*Brassica vegetables**Brussels sprouts*

No supervised trials on Brussels sprouts were available.

The Meeting agreed to withdraw its previous recommendation for Brussels sprouts.

*Fruiting vegetables, Cucurbits**Cucumber*

Data were available from supervised trials on cucumbers from the USA, Canada, Mexico, Hungary, France and Germany.

Trials from the USA on cucumbers were conducted with four or five foliar applications of an EC formulation at a rate equivalent to 0.29–0.57 kg ai/ha. No GAP from the USA was available for cucumber.

One trial in Canada on cucumbers was conducted with one foliar application of an EC formulation at a rate of 0.29 kg ai/ha. No GAP from Canada was available for cucumber.

The GAP on cucumbers in Mexico is three foliar applications at a maximum rate of 0.29 kg ai/ha with a PHI of 7 days. Triforine residue in cucumber from a trial in Mexico matching the GAP of Mexico was (n=1): 0.066 mg/kg.

Trials in Hungary, France and Germany on cucumbers were conducted but no GAP from European countries were available.

The Meeting considered that the data was insufficient for estimating a maximum residue level for cucumbers.

Squash

Data were available from supervised trials on summer squash and winter squash from the USA, France and Germany. No GAP from the USA or European countries was available for squash.

The Meeting agreed that estimation of maximum residue level was not possible for squash.

Melon

Data were available from supervised trials on melon from the USA, Mexico, France, Italy and Japan.

Trials in the USA on melons were conducted with one or five foliar applications of an EC formulation at a rate of 0.23–0.46 kg ai/ha. No GAP from the USA was available for melons.

The GAP on melons in Mexico is three foliar applications at a maximum rate of 0.29 kg ai/ha with a PHI of 7 days. Triforine residue in melon from one trial in Mexico matching GAP of Mexico was (n=1): 0.039 mg/kg.

Trials from Italy and France on melon were conducted with one or four foliar applications of an EC formulation at a rate of 0.20–0.29 kg ai/ha. No GAP from European countries was available for melons.

The GAP on melon in Japan is six foliar applications at a spray concentration of 0.009 mg/hL with a PHI of 1 day. Triforine residues in melon from the trials in Japan were (n=2): < 0.005 and 0.006 mg/kg.

The Meeting considered the data was insufficient for estimating a maximum residue level for melons.

The Meeting agreed to withdraw its previous recommendation for fruiting vegetables, cucurbits.

*Fruiting vegetables, other than Cucurbits**Peppers*

Data were available from supervised trials on peppers from Mexico, Japan and South Korea.

The GAP on peppers in Mexico is three foliar applications at a maximum rate of 0.29 kg ai/ha with a PHI of 14 days. Triforine residue in peppers from the trials in Mexico matching GAP of Mexico was (n=1): 0.12 mg/kg.

The GAP on peppers in Japan is three foliar applications at a spray concentration of 0.018 kg ai/hL with a PHI of 14 days. Triforine residues in peppers from trials in Japan matching GAP of Japan were (n=2): 0.06 and 0.22 mg/kg.

The GAP on chili peppers in South Korea is two foliar applications at a spray concentration equivalent to 0.019 kg ai/ha with a PHI of 7 days. Triforine residue in peppers from the trial in South Korea matching GAP of South Korea was (n=1): 0.35 mg/kg.

The Meeting considered that the data was insufficient for estimating a maximum residue level for peppers.

Egg plant

Data were available from supervised trials on egg plants in Mexico and Japan.

The GAP on egg plants in Mexico is three foliar applications at a maximum rate of 0.29 kg ai/ha with a PHI of 15 days. Triforine residue in egg plants from a trial in Mexico matching GAP of Mexico was (n=1): 0.066 mg/kg.

The GAP on eggplants in Japan is five foliar applications at a spray concentration of 0.018 kg ai/hL with a PHI of 1 day. Triforine residues in egg plants from the trials in Japan matching GAP of Japan were (n=5): 0.25, 0.28, 0.29, 0.38 and 0.39 mg/kg.

Based on the trials on egg plants in Japan, the Meeting estimated a maximum residue level, an STMR and an HR for triforine in egg plants of 1, 0.29 and 0.39 mg/kg, respectively.

Tomato

Data were available from supervised trials on tomatoes in the USA, Mexico, Denmark and Japan.

One trial from Denmark on tomatoes was conducted with one foliar application of EC formulation at a rate equivalent to 0.95 kg ai/ha. No GAP of European countries were available for tomatoes.

The GAP on tomatoes in Japan was three foliar applications at a spray concentration of 0.018 kg ai/hL with a PHI of 1 day. Triforine residues in tomatoes from trials in Japan matching GAP of Japan were (n=5): 0.14, 0.17, 0.26, 0.28 and 0.56 mg/kg.

The GAP on tomatoes in Mexico is four foliar applications at a maximum rate of 0.38 kg ai/ha with a PHI of 3 days. Triforine residues in tomatoes from the trials in Mexico matching GAP of Mexico were (n=5): 0.083, 0.096, 0.13, 0.27 and 0.40 mg/kg.

Trials from the USA on tomatoes were conducted with four or five foliar applications of an EC formulation at a rate of 0.18–0.41 kg ai/ha. Triforine residues in tomatoes from the trials in the USA matching GAP of Mexico were (n=3): 0.072, 0.17 and 0.28 mg/kg.

The Meeting decided to use the triforine residue data from the trials in Mexico and the USA. Triforine residues in tomatoes from the trials in Mexico and the USA matching GAP of Mexico were (n=8): 0.072, 0.083, 0.096, 0.13, 0.17, 0.27, 0.28, 0.40 mg/kg.

Based on the data, the Meeting estimated a maximum residue level of 0.7 mg/kg to replace its previous recommendation (0.5 mg/kg). The Meeting also estimated an STMR and an HR for triforine in tomato of 0.15 and 0.40 mg/kg, respectively.

*Legume vegetables**Common bean*

Data were available from supervised trials on common beans from Brazil and South Africa.

The GAP on beans in Brazil is three foliar applications at a maximum rate of 0.29 kg ai/ha with a PHI of 10 days. Triforine residue in bean seeds from the trials in Brazil matching GAP of Brazil was (n=1): < 0.01 mg/kg.

The GAP on beans in South Africa is for foliar application(s) with spray at a rate of 0.29 kg ai/ha with a PHI of 3 days (the maximum numbers of applications not specified). Triforine residue in beans from one trial in South Africa matching GAP of South Africa was (n=1): 0.44 mg/kg.

As the available data was insufficient for estimating a maximum residue level, the Meeting agreed to withdraw its previous recommendation for common bean (pods and immature seeds).

Cereal grains

Data were available from supervised trials on barley in France. No GAP from European countries was available for barley.

Data were available from supervised trials on wheat in Austria, France, UK and Brazil. No GAP from European countries and Brazil was available for wheat.

No other information was available for any other cereal grains.

The Meeting agreed that the estimation of maximum residue level was not possible for cereal grains.

The Meeting agreed to withdraw its previous recommendation for cereal grains.

*Animal feedstuffs**Barley straw and forage*

Data were available from supervised trials on barley straw and forage in France. No GAP from European countries was available for barley straw and forage.

The Meeting agreed that the estimation of a maximum residue level was not possible for barley straw and forage.

Wheat straw and forage

Data were available from supervised trials on wheat straw and forage in France and UK. No GAP from European countries was available for wheat straw and forage.

The Meeting agreed that the estimation of a maximum residue level was not possible for wheat straw and forage.

Fate of residues during processing*Residues in processed commodities*

The fate of triforine residues following the processing of plums, grapes and tomatoes was made available to the Meeting. Estimated processing factors and the derived STMR-Ps are summarized in the Table below.

Processing factors, STMR-P for food

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors ^a	PF (Mean or best estimate)	RAC STMR (mg/kg)	STMR-P (mg/kg)
Tomato	Juice	0.74 ^b , 0.76 ^b	0.75	0.15	0.11
	Paste	< 0.008	< 0.008		< 0.001

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors ^a	PF (Mean or best estimate)	RAC STMR (mg/kg)	STMR-P (mg/kg)
	Puree	0.14, 2.3 ^b , 2.6 ^b	2.3		0.35
	Wet pomace	< 0.12	< 0.12		< 0.018
	Dry pomace	1.6	1.6		0.24

^a Each value represents a separate study.

^b RAC was dipped in EC formulation solution.

Residues in animal commodities

Estimated maximum and mean dietary burdens of farm animals

The maximum and mean dietary burdens were calculated using the median residue of triforine in dry tomato pomace estimated at the current Meeting on a basis of the OECD Animal Feeding Table.

Summary of livestock dietary burdens (ppm of dry matter diet)

Livestock dietary burden, triforine, ppm of dry matter diet								
	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0	0	0	0	0.027 ^a	0.027 ^b	0	0
Dairy cattle	0	0	0	0	0.027	0.027 ^c	0	0
Broilers	0	0	0	0	0	0	0	0
Layers	0	0	0	0	0	0	0	0

^a Highest maximum beef cattle dietary burden suitable for MRL estimates for mammalian meat, fat, edible offal and milk

^b Highest mean beef cattle dietary burden suitable for STMR estimates for mammalian meat, fat and edible offal

^c Highest mean dairy cattle dietary burden suitable for STMR estimates for milk

Farm animal feeding studies

The Meeting received a lactating dairy goat feeding studies using triforine, which provided information on likely residues resulting in animal commodities and milk from triforine residues in the animal diet.

Lactating dairy goats

Lactating dairy goats were dosed with triforine for 30 days at doses equivalent to 5, 15 and 50 ppm in the diet. Residues of triforine were at or less than the LOQ (0.001 mg/L) in whole milk at the 5 ppm of feeding level except on sampling day 29 (< 0.001–0.003 mg/L). In the highest dose group (50 ppm feed), triforine residues in milk reached a plateau at the level of 0.002–0.010 mg/L after 3 days. In tissues, no measurable residues were found in fat, liver, kidney and muscle of the 5 ppm feed group. In the 15 ppm feed group, triforine concentration slightly exceeded the LOQ in several samples. In the 50 ppm feed group, residues were detected in all of the analysed tissues with exception of muscle in one animal and fat of all animals. The maximum values in kidney and liver were 0.009 and 0.012 ppm, respectively.

Animal commodities maximum residue levels

For MRL estimation, the residue in the animal commodities is triforine.

The maximum dietary burden for beef and dairy cattle was 0.027 ppm. The maximum dietary burden for beef and dairy cattle was 0.54% of the lowest dose of 5 ppm in feed of the lactating goat feeding study. In the lactating goat feeding study at 5 ppm, triforine was at < 0.01 mg/kg in milk and < 0.01 mg/kg in liver.

The Meeting estimated a maximum residue level of 0.01* mg/kg and an STMR of 0 mg/kg in milk.

The Meeting estimated a maximum residue level of 0.01* mg/kg, an STMR of 0 mg/kg and an HR of 0 mg/kg in mammalian meat and fat.

The Meeting estimated a maximum residue level of 0.01* mg/kg, an STMR of 0 mg/kg and an HR of 0 mg/kg in mammalian edible offal.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed below are suitable for estimating maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue (for compliance with the MRL and for estimation of dietary intake) for plant commodities: *Triforine*.

Definition of the residue (for compliance with the MRL and for estimation of dietary intake) for animal commodities: *Triforine and its metabolites determined as chloral hydrate expressed as triforine*.

The residue is not fat soluble.

Commodity		Recommended Maximum residue level, mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
CCN	Name	New	Previous		
FP 0226	Apple	W	2		
FB 0020	Blueberries	0.03	1	0.01	0.018
VB 0402	Brussels sprouts	W	0.2		
GC 0080	Cereal grains	W	0.1		
FS 0013	Cherries	W	2		
VP 0526	Common bean (pods and immature seeds)	W	1		
FB 0021	Currants, Black, Red, White	W	1		
VO 0440	Egg plant	1	–	0.29	0.39
VC 0045	Fruiting vegetables, Cucurbits	W	0.5		
FB 0268	Gooseberry	W	1		
FS 0247	Peach	W	5 Po		
FS 0014	Plums (including Prunes)	W	2		
FB 0275	Strawberry	W	1		
VO 0448	Tomato	0.7	0.5	0.15	0.40
MO 0105	Edible offal (Mammalian)	0.01 ^a	–	0	0
MF 0100	Mammalian fats (except milk fat)	0.01 ^a	–	0	0
MM 0095	Meat (from mammals except marine)	0.01 ^a	–	0	0
ML 0106	Milks	0.01 ^a	–	0	0

^a At or about the LOQ.

Commodity	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
Name		
Tomato juice	0.11	
Tomato paste	< 0.001	
Tomato puree	0.35	
Tomato wet pomace	< 0.018	
Tomato dry pomace	0.24	

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of triforine were calculated for the 17 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the current Meeting (Annex 3). The ADI is 0–0.03 mg/kg bw and the calculated IEDIs were 0–2% of the maximum ADI. The Meeting concluded

that the long-term intakes of residues of triforine, resulting from the uses considered by current JMPR, are unlikely to present a public health concern.

Short-term intake

The ARfD for triforine is 0.3 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for triforine were calculated for the food commodities for which STMRS or HRs were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2014 JMPR Report. The IESTIs were 0–5% of the ARfD for children and the general population. The Meeting concluded that the short-term intake of residues of triforine from other uses that have been considered by the present Meeting is unlikely to present a public health concern.

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TF-712-012	Ogawa, JM	1979	Analysis of Triforine Residue: cherry, apricot, nectarine, peach and plum. Department of Plant Pathology, University of California; Non-GLP, unpublished report, Document no. 102FX-532-2212, TF-712-012
TF-712-013	Adair, J	1982	Analysis of triforine residues: cherry, peach, plum and prune. Ciba-Geigy Canada Ltd., Canada; Non-GLP, unpublished report, Document no. 102FX-532-2213, TF-712-013
TF-712-015	Goldenthal, EI	1978	Triforine residues in cherries, International Research and Development Corporation; Non-GLP, unpublished report, Document no. 102FX-532-2215, TF-712-015
TF-712-017	Thorstenson, JH	1985	Triforine analysis in stone fruit. International Research and Development Corporation; Non-GLP, unpublished report, Document no. 10212-532-2217, TF-712-017
TF-712-070	Anonymous	1990	Triforine analysis in cherries. Shell Agrar Germany; Non-GLP, unpublished report, Document no. 10249-532-2222, TF-712-070
TF-712-071 to TF-712-080	Anonymous	1990	Triforine analysis in cherries. Shell Agrar Germany; Non-GLP, unpublished reports, Document no. 10249/10238-532-2223 to 10249/10238-532-2233, 10 reports TF-712-071 to TF-712-080
TF-712-090	Schulz, H	1993	Determination of the Total Residues of Triforine in Cherries (FRG-0056). RCC Umweltchemie AG, Switzerland; GLP, unpublished report no. 275815, TF-712-090
TF-712-061	Anonymous	1980	Triforine residue analysis in plums. International Research and Development Corporation; Non-GLP, unpublished report, Document no. TF-712-061
TF-712-066	Goldenthal, EI	1978	Triforine Residue in Prunes. International Research and Development Corporation; Non-GLP, unpublished report, Document no. 102FX-538-002, TF-712 066
TF-712-060	Anonymous	1981	Analysis of Residues in Prunes after treatment Vereor Multi (100g/l Triforine plus 100g/L Carbendazime), Sovilo. Toulouse, France; Non-GLP, unpublished report, Document no. 10288-532-2301, TF-712-060
TF-712-081 to TF-712-084	Anonymous	1991	Triforine analysis in plums. Shell Agrar Germany; Non-GLP, unpublished reports, Document no. 10249-532-2307 to 10249-532-2310, 4 reports TF-712-081 to TF-712-084
TF-712-091	Schulz, H	1992	Determination of the Total Residues of Triforine in Plums (FRG-0054). RCC Umweltchemie AG, Switzerland; GLP, unpublished report no. 275793, TF-712-091
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TF-712-065	Anonymous	1979	Determination of Triforine residue Analysis Plums. SABS, South Africa; unpublished report no. 311/8978/S39, TF-712-065

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TF-712-093	Scharm, M and Weitzel, R	1999	Triforine (CL 902194) 190 g ai/L DC (CF07738): Decline curve residue study on triforine (CL 902194) in apricots (France-South, 1998). Cyanamid Forschung GmbH, Germany; GLP, unpublished report no. CFS 1999-065, TF-712-093
TF-712-095	Scharm, M and Weitzel, R	1999	Triforine (CL 902194) 190 g ai/L DC (CF07738): At harvest residue study on triforine (CL 902194) in apricots (France-South, 1998). Cyanamid Forschung GmbH, Germany; GLP, unpublished report no. CFS 1999-064, TF-712-095
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TF-712-097	Fagnani, A	1999	Triforine (CL 902194) 190 g as/L DC (CF07738): Decline curve residue study on triforine (CL 902194) in apricot (Italy, 1999). Cyanamid Agricoltura S.p.A., Italy; GLP, unpublished report no. TF-IT-1999-1, TF-712-097
TF-712-098	Fagnani, A	1999	Triforine (CL 902194) 190 g as/L DC (CF07738): At harvest residue study on triforine (CL 902194) in apricot (Italy, 1999). Cyanamid Agricoltura S.p.A., Italy; GLP, unpublished report no. TF-IT-1999-2, TF-712-098
TF-712-037	Bros, G	1982	Analysis of Triforine Residues: peach and nectarine. FMC Corp., Fresno Ca. 93717, USA; Non-GLP, unpublished report, Document no. 102FX-532-2418, TF-712-037
TF-712-038	Goldenthal, EI	1978	Triforine Residue in nectarines. International Research and Development Corporation; Non-GLP, unpublished report, Document no. 102FX-532-2419, TF-712-038
TF-712-033	Betts, A	1982	Analysis of triforine residues: Peach. Agricultural Advisors, Inc., Yuba City, Ca, USA; Non-GLP, unpublished report, Document no. 102FX-532-2414, TF-712-033
TF-712-037	Bros, G	1982	Analysis of Triforine Residues: peach and nectarine. FMC CORP., Fresno Ca. 93717, USA; Non-GLP, unpublished report, Document no. 102FX-532-2418, TF-712-037
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TF-712-087	Carlou, R	1990	Residues of Triforine in Peaches from France Treated with Sapro—1990 Trials. Shell Chimie, Complexe Chimique de Berre, France; GLP, unpublished report no. BETR.91.016, TF-712-087
TF-712-092	Rohde, H, Klitsinaris, A and Weitzel, R	1999	Triforine (CL 902194) 190 g ai/L DC (CF07738): At harvest residue study on triforine (CL 902194) in peaches (Hellas, 1998). Cyanamid Forschung GmbH, Germany; GLP, unpublished report no. CFS 1999-041, TF-712-092
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BASF 1986/30618	Galhiane, MS	1996	Residue of Triforine in Peach, Cyanamid Quimica do Brasil Ltda., Brazil; Non-GLP, unpublished report, Document no. BASF

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TF-713-025	Anonymous	1976	Triforine in Johannisbeeren. Celamerck, Germany; Non-GLP, unpublished report, Document no. 102FX-532-3205, TF-713-025
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TF-713-038	Goldenthal, EI	1978	Triforine Residues in Cranberries; International Research and Development Corporation; Non-GLP, unpublished report, Document no. 102FX-532-3402, TF-713-038
TF-713-041	Anonymous	1984	Analysis of Triforine Residue: Cranberries; Ocean Spray Cranberries, Inc.; Non-GLP, unpublished report no. 102FX-532-3401, TF-713-041
TF-713-003	Anonymous	1983	Residues in Grapes (Translation). Celamerck, Ingeheim, Germany; Non-GLP unpublished report, Document no. 10238-532-3003, TF-713-003
TF-713-004	Anonymous	1983	Residues in Grapes (Translation). Celamerck, Ingeheim, Germany; Non-GLP, unpublished report, Document no. 10238-532-3004, TF-713-004
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TF-713-009	Anonymous	1978	Residues in Grapes (Translation). Celamerck, Ingeheim, Germany; Non-GLP unpublished report, Document no. 102FX-532-3009, TF-713-009
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TF-713-008	Anonymous	1985	Analysis of Triforine Residue: Grapes; Ciba-Geigy Mexicana S.A. de C.V., Mexico; Non-GLP, unpublished report, Document no. 102FX-532-3008, TF-713-008
TF-713-013	Anonymous	1978	Residues in Grapes. Cawthron Technical Group, New Zealand; Non-GLP unpublished report, Document no. 102FX-532-3013, TF-713-013
TF-701-004	Thorstenson, JH	1979	Triforine Residues in Various Crops in Mexico. International Research and Development Corporation, USA; Non-GLP, unpublished report, Document no. 102FX-532-3108, TF-701-004

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TF-713-015	Anonymous	1985	Triforine Residues in Strawberries (Translation). Celamerck, Brazil; Non-GLP, unpublished report Document no. 102FX-532-3102, TF-713-015
	Kuroda, M	1985	Determination of Residues of Triforine in/on Strawberry. Japan Analytical Chemistry Consultants Co., Ltd.; Non-GLP, unpublished report. (2 trials)
	Aratani, K	1982	Determination of Residues of Triforine in/on Strawberry. Japan Analytical Chemistry Consultants Co., Ltd.; Non-GLP, unpublished report. (2 trials)
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TF-723-013	Anonymous	1984	Analysis of Triforine Residue (Cucumber). Dade County Cooperative Extension, Florida, USA; Non-GLP, unpublished report, Document no. 102FX-532-4504, TF -723-013
TF-723-024	Latin, RX	1987	Analysis of Triforine Residue in Cucumber. Purdue University, Dept. of Botany and Plant Pathology, Indiana, USA; Non-GLP, unpublished report, Document no. 10238-532-4516, TF -723-024
TF-723-030	Chandler, LD	1987	Analysis of Triforine Residues in Cucumber. USDA—ARS, Texas, USA; Non-GLP, unpublished report, Document no. 10238-532-4522, TF -723-030
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TF-723-023	Anonymous	1985	Report on Supervised Trials for Residue Analysis (Hungary). Celamerck; Non-GLP, unpublished report, Document no. 10238-532-4515, TF-723-023
TF-723-040	Anonymous	1980	Analysis of residues—courgettes (France) Celamerck; Non-GLP, unpublished report, Document no. 10238-532-4701, TF -723-040
TF-723-041	Anonymous	1980	Analysis of residues—courgettes (France) Celamerck; Non-GLP, unpublished report, Document no. 10238-532-4702, TF -723-041
TF-723-047	Anonymous	1989	Analysis of residues in cucumbers (Translation) Shell Agrar., Germany; Non-GLP, unpublished report, Document no. 10249-532-4525, TF-723-047
TF-723-048	Anonymous	1989	Analysis of residues in cucumbers (Translation) Shell Agrar., Germany; Non-GLP, unpublished report, Document no. 10249-532-4526, TF-723-048
TF-723-057	Bitz, K	1995	Triforine: determination of the residues in cucumbers at harvest from field trials in France in 1993. Cyanamid Forschung GmbH (CFS), Germany; GLP, unpublished report no. CFS 1994-095, TF -723-057
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TF-723-045	Gotlieb, A	1988	Analysis of Triforine Residues in Summer Squash. Plant and Soil Department, University of Vermont, USA; Non-GLP, unpublished

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TF-723-053	Davis, M	1988	Analysis of Triforine Residue in Squash and Cantaloupe. Plant Pathology Dept. University of California, USA; Non-GLP, unpublished report, Document no. TF-723-053
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TF-723-035	Chandler, LD	1985	Analysis of Triforine Residue: Cantaloupes. USDA ARS, Texas, USA; Non-GLP, unpublished report, Document no.102FX-532-4603, TF-723-035
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TF-723-038	Anonymous	1977	Analysis of Triforine Residues in Melons (Translation). Celamerck; Non-GLP unpublished report, Document no. 102FX-532-4606, TF-723-038
	Kuroda, M	1978	Determination of residues of triforine in Muskmelon. Japan Analytical Chemistry Consultants Co., Ltd.; Non-GLP, unpublished report. (2 trials)
TF-701-004	Thorstenson, JH	1979	Triforine Residues in Various Crops in Mexico. International Research and Development Corporation, USA; Non-GLP, unpublished report, Document no. 102FX-532-3108, TF-701-004
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	Asari, T	2008	Determination of residues of triforine in/on green peppers. Japan Analytical Chemistry Consultants Co., Ltd., Tokyo, Japan; Non-GLP, unpublished report (2 trials)
TF-765-001	Anonymous	2009	Determination of residue of Triforine DC in chilli pepper (South Korea). Technical Institute, Dongbang Agro Corporation, Korea; Non-GLP, unpublished report
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	Higuchi, T	2008	Determination of residues of triforine in/on eggplant. Japan Analytical Chemistry Consultants Co., Ltd., Tokyo, Japan; Non-GLP, unpublished report (2 trials)
	Kuroda, M	1987	Determination of residues of triforine in/on eggplant. Japan Analytical Chemistry Consultants Co., Ltd., Tokyo, Japan; Non-GLP, unpublished report (3 trials)
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TF-723-006	Anonymous	2002	Report on residue trials with Triforine (Denmark). Shell Forschung GmbH, Germany; Non-GLP, unpublished report, Document no. 102FX-532-4306, TF-723-006
	Higuchi, T	2009	Determination of residues of triforine in/on tomato. Japan Analytical Chemistry Consultants Co., Ltd., Tokyo, Japan; Non-GLP, unpublished report (2 trials)
	Kuroda, M	1987	Determination of residues of triforine in/on tomato. Japan Analytical Chemistry Consultants Co., Ltd., Tokyo, Japan; Non-GLP, unpublished report (3 trials)
TF-720-001	Nakahara, K	1985	Residues Analysis of Saprol in String Bean from Brazil. Shell Agrar; Non-GLP, unpublished report, Document no. TF-720-001
TF-720-003	Anonymous	1985	Residues in bean (Translation) (Brazil). Celamerck; Non-GLP, unpublished report, Document no. 102FX-532-4003, TF -720-003
TF-720-004	Nakahara, K	1984	Residues Analysis of Saprol in Bean from Brazil. Shell Agrar; Non-GLP, unpublished report, Document no. TF-720-004
TF-767-004	Galhiane, MS	1996	Residue of Triforine in Bean Grain. Cyanamid Quimica do Brasil Ltda, Brazil; Non-GLP, unpublished report, Document no. BASF 1996/306188
TF-720-005	Anonymous	1976	Saprol residues on beans (South Africa) Bayer/Celamerck; Non-GLP, unpublished report, Document no. TF -720-005
TF-730-024	Anonymous	1979	Residue in barley (Translation) (France). Celamerck; Non-GLP, unpublished report, Document no. 10283-532-0102, TF-730-024
TF-730-025	Anonymous	1979	Residue in barley (Translation) (France). Celamerck; Non-GLP, unpublished report, Document no. 10283-532-0103, TF -730-025
TF-730-003	Anonymous	1980	Residue in wheat (Translation) (Austria). Celamerck; Non-GLP, unpublished report, Document no. 10288-532-0003, TF -730-003
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TF-790-019	Eichler, D	1982	Triforine Residues in the processed fractions of Tomatoes. Celamerck, Non-GLP, unpublished report, Document no. 10211-538-001, TF-790-019
TF-790-020	Beevers, M	1986	Analysis of Triforine residues in Processed Tomatoes. California Agricultural Research, USA, Non-GLP unpublished report, Document no. 102FX-538-006 (Part of Tomato Residue trials conducted during 1985 season in the USA. unpublished report 102FX 532-4303 /102FX-532-4303), TF-790-020
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47	The sago palm, 1983 (E F)		Pesticide residues in food 1985 – Evaluations –
48	Guidelines for integrated control of cotton pests, 1983 (Ar E F S)	73	Part II: Toxicology, 1986 (E)
49	Pesticide residues in food 1982 – Evaluations, 1983 (E)	74	Early agrometeorological crop yield assessment, 1986 (E F S)
50	International plant quarantine treatment manual, 1983 (C E)	75	Ecology and control of perennial weeds in Latin America, 1986 (E S)
51	Handbook on jute, 1983 (E)	76	Technical guidelines for field variety trials, 1993 (E F S)
52	The palmyrah palm: potential and perspectives, 1983 (E)	77	Guidelines for seed exchange and plant introduction in tropical crops, 1986 (E)
53/1	Selected medicinal plants, 1983 (E)		Pesticide residues in food 1986 – Report, 1986 (E F S)
54	Manual of fumigation for insect control, 1984 (C E F S)	78	Pesticide residues in food 1986 – Evaluations –
55	Breeding for durable disease and pest resistance, 1984 (C E)	78/2	Part I: Residues, 1986 (E)
56	Pesticide residues in food 1983 – Report, 1984 (E F S)	79	Pesticide residues in food 1986 – Evaluations –
57	Coconut, tree of life, 1984 (E S)		Part II: Toxicology, 1987 (E)
58	Economic guidelines for crop pest control, 1984 (E F S)	80	Tissue culture of selected tropical fruit plants, 1987 (E)
59	Micropropagation of selected rootcrops, palms, citrus and ornamental species, 1984 (E)	81	Improved weed management in the Near East, 1987 (E)
60	Minimum requirements for receiving and maintaining tissue culture propagating material, 1985 (E F S)	82	Weed science and weed control in Southeast Asia, 1987 (E)
61	Pesticide residues in food 1983 – Evaluations, 1985 (E)	83	Hybrid seed production of selected cereal, oil and vegetable crops, 1987 (E)
62	Pesticide residues in food 1984 – Report, 1985 (E F S)	84	Litchi cultivation, 1989 (E S)
63	Manual of pest control for food security reserve grain stocks, 1985 (C E)	85	Pesticide residues in food 1987 – Report, 1987 (E F S)
64	Contribution à l'écologie des aphides africains, 1985 (F)	86/1	Manual on the development and use of FAO specifications for plant protection products, 1987 (E** F S)
65	Amélioration de la culture irriguée du riz des petits fermiers, 1985 (F)	86/2	Pesticide residues in food 1987 – Evaluations –
66	Sesame and safflower: status and potentials, 1985 (E)		Part I: Residues, 1988 (E)
67	Pesticide residues in food 1984 – Evaluations, 1985 (E)	87	Pesticide residues in food 1987 – Evaluations –
68	Pesticide residues in food 1985 – Report, 1986 (E F S)		Part II: Toxicology, 1988 (E)
69	Breeding for horizontal resistance to wheat diseases, 1986 (E)	88	Root and tuber crops, plantains and bananas in developing countries – challenges and opportunities, 1988 (E)
70	Breeding for durable resistance in perennial crops, 1986 (E)	89	Jessenia and Oenocarpus: neotropical oil palms worthy of domestication, 1988 (E S)
71	Technical guideline on seed potato	90	Vegetable production under arid and semi-arid conditions in tropical Africa, 1988 (E F)
		91	Protected cultivation in the Mediterranean climate, 1990 (E F S)
		92	Pastures and cattle under coconuts, 1988 (E S)
		93/1	Pesticide residues in food 1988 – Report, 1988 (E F S)
			Pesticide residues in food 1988 – Evaluations –

	Part I: Residues, 1988 (E)		I: Residues, 1993 (E)
93/2	Pesticide residues in food 1988 – Evaluations – Part II: Toxicology, 1989 (E)	119	Quarantine for seed, 1993 (E)
94	Utilization of genetic resources: suitable approaches, agronomical evaluation and use, 1989 (E)	120	Weed management for developing countries, 1993 (E S)
		120/1	Weed management for developing countries, Addendum 1, 2004 (E F S)
95	Rodent pests and their control in the Near East, 1989 (E)	121	Rambutan cultivation, 1993 (E)
96	Striga – Improved management in Africa, 1989 (E)	122	Pesticide residues in food 1993 – Report, 1993 (E F S)
97/1	Fodders for the Near East: alfalfa, 1989 (Ar E)	123	Rodent pest management in eastern Africa, 1994 (E)
97/2	Fodders for the Near East: annual medic pastures, 1989 (Ar E F)	124	Pesticide residues in food 1993 – Evaluations – Part I: Residues, 1994 (E)
98	An annotated bibliography on rodent research in Latin America 1960-1985, 1989 (E)	125	Plant quarantine: theory and practice, 1994 (Ar)
99	Pesticide residues in food 1989 – Report, 1989 (E F S)	126	Tropical root and tuber crops – Production, perspectives and future prospects, 1994 (E)
100	Pesticide residues in food 1989 – Evaluations – Part I: Residues, 1990 (E)	127	Pesticide residues in food 1994 – Report, 1994 (E)
100/2	Pesticide residues in food 1989 – Evaluations – Part II: Toxicology, 1990 (E)	128	Manual on the development and use of FAO specifications for plant protection products – Fourth edition, 1995 (E F S)
101	Soilless culture for horticultural crop production, 1990 (E)	129	Mangosteen cultivation, 1995 (E)
102	Pesticide residues in food 1990 – Report, 1990 (E F S)	130	Post-harvest deterioration of cassava – A biotechnology perspective, 1995 (E)
103/1	Pesticide residues in food 1990 – Evaluations – Part I: Residues, 1990 (E)	131/1	Pesticide residues in food 1994 – Evaluations – Part I: Residues, Volume 1, 1995 (E)
104	Major weeds of the Near East, 1991 (E)	131/2	Pesticide residues in food 1994 – Evaluations – Part I: Residues, Volume 2, 1995 (E)
105	Fundamentos teórico-prácticos del cultivo de tejidos vegetales, 1990 (S)	132	Agro-ecology, cultivation and uses of cactus pear, 1995 (E)
106	Technical guidelines for mushroom growing in the tropics, 1990 (E)	133	Pesticide residues in food 1995 – Report, 1996 (E)
107	Gynandropsis gynandra (L.) Briq. – a tropical leafy vegetable – its cultivation and utilization, 1991 (E)	134	(Number not assigned)
108	Carambola cultivation, 1993 (E S)	135	Citrus pest problems and their control in the Near East, 1996 (E)
109	Soil solarization, 1991 (E)	136	El pepino dulce y su cultivo, 1996 (S)
110	Potato production and consumption in developing countries, 1991 (E)	137	Pesticide residues in food 1995 – Evaluations – Part I: Residues, 1996 (E)
111	Pesticide residues in food 1991 – Report, 1991 (E)	138	Sunn pests and their control in the Near East, 1996 (E)
112	Cocoa pest and disease management in Southeast Asia and Australasia, 1992 (E)	139	Weed management in rice, 1996 (E)
113/1	Pesticide residues in food 1991 – Evaluations – Part I: Residues, 1991 (E)	140	Pesticide residues in food 1996 – Report, 1997 (E)
114	Integrated pest management for protected vegetable cultivation in the Near East, 1992 (E)	141	Cotton pests and their control in the Near East, 1997 (E)
115	Olive pests and their control in the Near East, 1992 (E)	142	Pesticide residues in food 1996 – Evaluations – Part I Residues, 1997 (E)
116	Pesticide residues in food 1992 – Report, 1993 (E F S)	143	Management of the whitefly-virus complex, 1997 (E)
117	Quality declared seed, 1993 (E F S)	144	Plant nematode problems and their control in the Near East region, 1997 (E)
118	Pesticide residues in food 1992 – Evaluations – Part	145	Pesticide residues in food 1997 – Report, 1998 (E)
		146	Pesticide residues in food 1997 – Evaluations – Part I: Residues, 1998 (E)

147	Soil solarization and integrated management of soilborne pests, 1998 (E)	172	Pesticide residues in food, 2002 – Report, 2002 (E)
148	Pesticide residues in food 1998 – Report, 1999 (E)	173	Manual on development and use of FAO and WHO specifications for pesticides, 2002 (E S)
149	Manual on the development and use of FAO specifications for plant protection products – Fifth edition, including the new procedure, 1999 (E)	174	Genotype x environment interaction – Challenges and opportunities for plant breeding and cultivar recommendations, 2002 (E)
150	Restoring farmers' seed systems in disaster situations, 1999 (E)	175/1	Pesticide residues in food 2002 – Evaluations – Part 1: Residues – Volume 1 (E)
151	Seed policy and programmes for sub-Saharan Africa, 1999 (E F)	175/2	Pesticide residues in food 2002 – Evaluations – Part 1: Residues – Volume 2 (E)
152/1	Pesticide residues in food 1998 – Evaluations – Part I: Residues, Volume 1, 1999 (E)	176	Pesticide residues in food 2003 – Report, 2004 (E)
152/2	Pesticide residues in food 1998 – Evaluations – Part I: Residues, Volume 2, 1999 (E)	177	Pesticide residues in food 2003 – Evaluations – Part 1: Residues, 2004 (E)
153	Pesticide residues in food 1999 – Report, 1999 (E)	178	Pesticide residues in food 2004 – Report, 2004 (E)
154	Greenhouses and shelter structures for tropical regions, 1999 (E)	179	Triticale improvement and production, 2004 (E)
155	Vegetable seedling production manual, 1999 (E)	180	Seed multiplication by resource-limited farmers - Proceedings of the Latin American workshop, 2004 (E)
156	Date palm cultivation, 1999 (E)	181	Towards effective and sustainable seed-relief activities, 2004 (E)
156 Rev.1	Date palm cultivation, 2002 (E)	182/1	Pesticide residues in food 2004 – Evaluations – Part 1: Residues, Volume 1 (E)
157	Pesticide residues in food 1999 – Evaluations – Part I: Residues, 2000 (E)	182/2	Pesticide residues in food 2004 – Evaluations – Part 1: Residues, Volume 2 (E)
158	Ornamental plant propagation in the tropics, 2000 (E)	183	Pesticide residues in food 2005 – Report, 2005 (E)
159	Seed policy and programmes in the Near East and North Africa, 2000	184/1	Pesticide residues in food 2005 – Evaluations – Part 1: Residues, Volume 1 (E)
160	Seed policy and programmes for Asia and the Pacific, 2000 (E)	184/2	Pesticide residues in food 2005 – Evaluations – Part 1: Residues, Volume 2 (E)
161	Silage making in the tropics with particular emphasis on smallholders, 2000 (E S)	185	Quality declared seed system, 2006 (E F S)
162	Grassland resource assessment for pastoral systems, 2001, (E)	186	Calendario de cultivos – América Latina y el Caribe, 2006 (S)
163	Pesticide residues in food 2000 – Report, 2001 (E)	187	Pesticide residues in food 2006 – Report, 2006 (E)
164	Seed policy and programmes in Latin America and the Caribbean, 2001 (E S)	188	Weedy rices – origin, biology, ecology and control, 2006 (E S)
165	Pesticide residues in food 2000 – Evaluations – Part I, 2001 (E)	189/1	Pesticide residues in food 2006 – Evaluations – Part 1: Residues, Volume 1 (E)
166	Global report on validated alternatives to the use of methyl bromide for soil fumigation, 2001 (E)	189/2	Pesticide residues in food 2006 – Evaluations – Part 1: Residues, Volume 2 (E)
167	Pesticide residues in food 2001 – Report, 2001 (E)	190	Guidance for packing, shipping, holding and release of sterile flies in area-wide fruit fly control programmes, 2007 (E)
168	Seed policy and programmes for the Central and Eastern European countries, Commonwealth of Independent States and other countries in transition, 2001 (E)	191	Pesticide residues in food 2007 – Report, 2007 (E)
169	Cactus (<i>Opuntia</i> spp.) as forage, 2003 (E S)	192	Pesticide residues in food 2007 – Evaluations – Part 1: Residues, 2008 (E)
170	Submission and evaluation of pesticide residues data for the estimation of maximum residue levels in food and feed, 2002 (E)	193	Pesticide residues in food 2008 – Report, 2008 (E)
171	Pesticide residues in food 2001 – Evaluations – Part I, 2002 (E)	194	Pesticide residues in food 2008 – Evaluations, 2008 (E)
		195	Quality declared planting material – Protocols and

	standards for vegetatively propagated crops, 2010 (E)	219	Pesticide residues in food 2013 – Report, 2011 (E)
196	Pesticide residues in food 2009 – Report, 2009 (E)	220	Pesticide Residues in food 2013 – Evaluations – Part 1
197	Submission and evaluation of pesticide residues data for the estimation of maximum residue levels in food and feed, 2009 (E)	221	Pesticide residues in food 2014 – Report, 2011 (E)
198	Pesticide residues in food 2009 – Evaluations – Part 1: Residues, 2010 (E)	222	Pesticide Residues in food 2015 – Evaluations
199	Rearing codling moth for the sterile insect technique, 2010 (E)	Availability: December 2014	
200	Pesticide residues in food 2010 – Report, 2011 (E)		
201	Promoting the Growth and Development of Smallholder Seed Enterprises for Food Security Crops Case Studies from Brazil, Côte d'Ivoire and India (E) 2010	Ar – Arabic	Multil – Multilingual
		C – Chinese	* Out of print
		E – English	** In preparation
		F – French	
202	Seeds in Emergencies: a technical handbook (E) 2011	P – Portuguese	
		S – Spanish	
203	Sustainable wheat rust resistance – Learning from history		
204	State of knowledge on breeding for durable resistance to soybean rust disease in the developing world	The FAO Technical Papers are available through the authorized FAO Sales Agents or directly from Sales and Marketing Group, FAO, Viale delle Terme di Caracalla, 00153 Rome, Italy.	
205	The FAO/IAEA Spreadsheet for Designing and Operation of Insect Mass Rearing Facilities		
206	Pesticide Residues in food 2010 – Evaluations – Part 1		
207	Plant breeding and seed systems for rice, vegetables, maize and pulses in Bangladesh		
208	The dynamic tension between public and private plant breeding in Thailand		
209	The strategic role of plant breeding in Uruguay: analysis through an agricultural innovation system framework		
210	Evolving a plant breeding and seed system in sub-Saharan Africa in an era of donor dependence		
211	Pesticide residues in food 2011 – Report, 2011 (E)		
212	Pesticide Residues in food 2011 – Evaluations – Part 1		
213	Evaluation of pesticide residues - Training Manual		
214	Agricultural handtools; Guidelines for Field Officers and Procurement		
215	Pesticide residues in food 2012 – Report, 2011 (E)		
216	Pesticide residues in Food 2011 – Evaluations – Part 1 (E)		
217	Good Agricultural Practices for greenhouse vegetable crops: Principles for Mediterranean climate areas (E)		
218	Cassava Farmer Field Schools – Resource material for facilitators in sub-Saharan Africa		

The annual Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Core Assessment Group on Pesticide Residues was held in Rome, Italy, from 16 to 25 September 2014. The FAO Panel of Experts had met in preparatory sessions from 11 to 15 September 2014. The Meeting was held in pursuance of recommendations made by previous Meetings and accepted by the governing bodies of FAO and WHO that studies should be undertaken jointly by experts to evaluate possible hazards to humans arising from the occurrence of pesticide residues in foods. During the meeting the FAO Panel of Experts was responsible for reviewing pesticide use patterns (use of good agricultural practices), data on the chemistry and composition of the pesticides and methods of analysis for pesticide residues and for estimating the maximum residue levels that might occur as a result of the use of the pesticides according to good agricultural use practices. The WHO Core Assessment Group was responsible for reviewing toxicological and related data and for estimating, where possible and appropriate, acceptable daily intakes (ADIs) and acute reference doses (ARfDs) of the pesticides for humans. This report contains information on ADIs, ARfDs, maximum residue levels, and general principles for the evaluation of pesticides. The recommendations of the Joint Meeting, including further research and information, are proposed for use by Member governments of the respective agencies and other interested parties.

