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Food and Agriculture
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Pesticide residues in food 2015

Joint FAO/WHO Meeting on Pesticide Residues

EVALUATIONS

2015

PART I - RESIDUES

Pesticide residues in food 2015

Evaluations
Part I - Residues

FAO
PLANT
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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
Geneva, Switzerland 15-24 September 2015

WORLD HEALTH ORGANIZATION
FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS
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GENEVA, 15–24 SEPTEMBER 2015

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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 **CH**emische 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
*c*GAP	Critical GAP
CXL	Codex MRL
DALA	days after last application
DAP	days after planting
DALT	days after last treatment
DAT	days after treatment
DM	dry matter
DT_{50}	time required for 50% dissipation of the initial concentration
dw	dry weight
ECD	electron capture detector
EFSA	European Food Safety Authority
EPO	early post-emergence
equiv	equivalent
EU	European Union
FAO	Food and Agriculture Organization of the United Nations
fw	fresh weight
GA	glufosinate-ammonium
GAP	good agricultural practice
GC	gas chromatography
GC-ECD	gas chromatography with electron capture detection
GC-FID	gas chromatography with flame ionization detection

GC-FPD	gas chromatography with flame photometric 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
GLC	gas liquid chromatography
GLP	good laboratory practice
GPC	gel permeation chromatography
HEPA	2-hydroxyethyl phosphonic acid; 2-hydroxyethephon
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
ISO	International Organization for Standardization
IUPAC	International Union of Pure and Applied Chemistry
JMPR	Joint FAO/WHO Meeting on Pesticide Residues
JP	Japan
LC	liquid chromatography
LOD	limit of detection
log P_{ow}	octanol-water partition coefficient
LOQ	limit of quantification
MOA	mode of action
MRL	maximum residue limit
MS	mass spectrometry
MS/MS	tandem mass spectrometry
ND	non-detect - below limit of detection
OECD	Organisation for Economic Co-operation and Development
OP	organophosphorus compound
PBI	plant back interval
Pf	processing factor
PH	pre-harvest
PHI	pre-harvest interval
ppm	parts per million
PRE	pre-emergence

QuEChERS	Quick, Easy, Cheap, Effective, Rugged, and Safe–Multiresidue pesticide analysis
RAC	raw agricultural commodity
RSD	relative standard deviation
RTI	re-treatment interval
SC	suspension concentrate
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
TAR	total administered radioactivity
TF	transfer factor
TFNA	4-trifluoromethylnicotinic acid
TFNA-AM	4-trifluoromethylnicotinamide
TFNA-OH	6-hydroxy-4-trifluoromethylnicotinic acid
TFNG	*N*-(4-trifluoromethylnicotinoyl) glycine
TFNG-AM	*N*-(4-trifluoromethylnicotinoyl) glycinamide
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
WG	wettable granule
WHO	World Health Organization
WP	wettable powder

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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 WHO Headquarters, Geneva (Switzerland), from 15 to 24 September 2015. The FAO Panel Members met in preparatory sessions on 10–14 September.

The meeting was opened by Dr Angelika Tritscher, Coordinator, Risk Assessment and Management, Department of Food Safety and Zoonoses, WHO. On behalf of WHO and FAO, Dr Tritscher welcomed and thanked the participants for providing their expertise and for devoting significant time and effort to the work of JMPR. She noted that the work of JMPR is of great importance, as it provides the scientific basis for international food safety standards as recommended by the Codex Alimentarius Commission. She emphasized that the programme is also important for other programmes within the Organizations; for example, the WHO Guidelines for Drinking-water Quality use the scientific advice provided by JMPR as the basis for the derivation of drinking-water guidelines for pesticides.

Dr Tritscher noted that further important considerations at the meeting related to methodological aspects, such as discussing the outcome of the recent workshop to review the international estimate of short-term dietary intake (IESTI) equations, in an effort to further improve and harmonize risk assessment methodology for pesticide residues. The Meeting was also asked to consider the outcome of the WHO Expert Task Force on Carcinogenicity of Diazinon, Glyphosate and Malathion, to provide recommendations to the Organizations on necessary actions in light of recent International Agency for Research on Cancer (IARC) hazard classifications. Dr Tritscher reminded the Meeting of the importance of food safety in public health; in order to raise awareness of this issue, WHO dedicated the 2015 World Health Day to food safety, with important advocacy and information material being available from the WHO website. Lastly, she reminded participants that they were invited as independent experts and not as representatives of their countries or organizations. She also reminded them of the confidential nature of the meeting, in order to allow experts to freely express their opinions.

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, supervised trials median residue (STMR) levels and highest residue (HR) levels were estimated for commodities of plant and 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 29 pesticides, including eight new compounds and four 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 original schedule of compounds to be evaluated was amended, with dicamba and methoxyfenozide not considered for residues and fluazifop-*p*-butyl not considered for toxicity or residues owing to the submission of incomplete data sets.

The Meeting established ADIs and ARfDs, estimated maximum residue levels and recommended them for use by CCPR, and estimated STMR and 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 residues 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.

ABAMECTIN (177)

First draft prepared by Professor Eloisa Dutra Caldas, University of Brasilia, Brazil

BACKGROUND INFORMATION

Abamectin belongs to the family of avermectins, which are macrocyclic lactones produced by a soil actinomycete, *Streptomyces avermitilis*. It is a broad-spectrum acaricide with additional insecticidal action on a limited number of insects. The compound acts on insects by increasing the membrane permeability to chloride ions, and it mainly stimulates the release of γ-aminobutyric acid (GABA). The affected arthropod becomes paralysed, stops feeding, and dies after a few days. It exerts contact and stomach action, with limited plant systemic activity, but exhibits translaminar movement into treated leaves. Abamectin is also used as an anthelmintic drug in veterinary medicine.

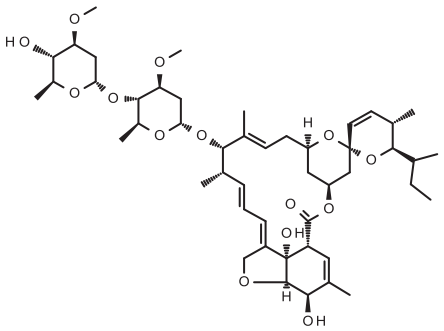
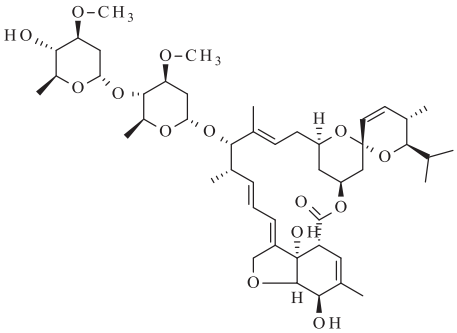
Abamectin was firstly evaluated by JMPR in 1992 (T,R). The latest review of toxicology data was conducted in 1997 and of residue data in 2000. Abamectin was scheduled at the 46th Session of the CCPR (2014) for the periodic re-evaluation of toxicology and residues by the 2015 JMPR.

For the residue evaluation, data were submitted on physical chemical properties, environmental fate, metabolism on plants and lactating goats, analytical methods, GAP, supervised trials on fruits, vegetables, nuts, beans, coffee, cotton and cereals, processing studies and a cow feeding study.

IDENTITY

Abamectin is a mixture containing ≥ 80% avermectin B_{1a} and ≤ 20% avermectin B_{1b}. The absolute stereochemistry of both avermectin homologues is known and defined at each chiral centre and stereogenic carbon-carbon double bond by their IUPAC nomenclature.

ISO Common Name:	Abamectin
Composition:	a mixture containing ≥ 80% avermectin B_{1a} and ≤ 20% avermectin B_{1b}
IUPAC nomenclature:	
Avermectin B_{1a}:	(10*E*,14*E*,16*E*)-(1*R*,4*S*,5′*S*,6*S*,6′*R*,8*R*,12*S*,13*S*,20*R*,21*R*,24*S*)-6′-[(*S*)-*sec*-butyl]-21,24-dihydroxy-5′,11,13,22-tetramethyl-2-oxo-(3,7,19-trioxatetracyclo [15.6.1.1^{4,8}.0^{20,24}]pentacosa-10,14,16,22-tetraene)-6-spiro-2′-(5′,6′-dihydro-2′*H*-pyran)-12-yl 2,6-dideoxy-4-*O*-(2,6-dideoxy-3-*O*-methyl-α-L-*arabino*-hexopyranosyl)-3-*O*-methyl-α-L-*arabino*-hexopyranoside
Avermectin B_{1b}:	(10*E*,14*E*,16*E*)-(1*R*,4*S*,5′*S*,6*S*,6′*R*,8*R*,12*S*,13*S*,20*R*,21*R*,24*S*)-21,24-dihydroxy-6′-isopropyl-5′,11,13,22-tetramethyl-2-oxo-(3,7,19-trioxatetracyclo[15.6.1.1^{4,8}.0^{20,24}] pentacosa-10,14,16,22-tetraene)-6-spiro-2′-(5′,6′-dihydro-2′*H*-pyran)-12-yl 2,6-dideoxy-4-*O*-(2,6-dideoxy-3-*O*-methyl-α-L-*arabino*-hexopyranosyl)-3-*O*-methyl-α-L-*arabino*-hexopyranoside
CA nomenclature:	
Abamectin:	Avermectin B_1
Avermectin B_{1a}:	5-O-demethyl-avermectin A_{1a}
Avermectin B_{1b}:	5-O-demethyl-25-de(1-methylpropyl)-25-(1-methylethyl)-avermectin A_{1a}
CAS registry no:	
Abamectin:	71751-41-2
Avermectin B_{1a}:	65195-55-3
Avermectin B_{1b}:	65195-56-4

CIPAC no:	495
Chemical structures	
:	 Avermectin B_{1a} : $C_{48}H_{72}O_{14}$; mm: 873.1
	 Avermectin B_{1b}: $C_{47}H_{70}O_{14}$; mm= 859.1

Physical and chemical properties

Abamectin technical material was of high purity (> 98%) and was used for the determination of the physical and chemical properties of the pure active substance.

Properties of abamectin (> 98% purity) and degradation in water (avermectin B_{1a})

Property	Results	Reference; Report
Appearance(physical state, colour, odour)	White powder, odour was not determined	Das, R 1999
Vapour pressure	< 3.7×10^{-6} Pa at 25 °C was calculated using the LOQ of the test substance	Widmer, H 1999;1999a
Melting point	Melting range: 161.8 °C–169.4 °C, with thermal decomposition during melting	Das, R 1999;
Partition coefficient n-octanol/water	Average log K_{OW} was 4.4 ± 0.3	McCauley, JA 1996
Solubility in water	1.21 ± 0.15 mg/L (pH = 7.57 ± 0.23) at 25 ºC	McCauley, JA 1997
Solubility in organic solvents	At 25 ºC: acetone: 72 g/L dichloromethane: 470 g/L ethyl acetate: 160 g/L hexane: 0.11 g/L methanol: 13 g/L octanol: 83 g/L toluene: 23 g/L	Stulz, J 1999
Density	Density 1.18×10^3 kg/m^3, corresponding to a relative density of 1.18. At 22 ºC.	Füldner, HH 1999
Hydrolysis in water	No hydrolysis at pH 4–9, 25 °C	Maynard, S, Ku, CC 1982;

Property	Results	Reference; Report
[^{3}H] avermectin B_{1a}	No hydrolysis at pH 4–7, 50 °C pH 9, 60 °C: 4.9 d pH 9, 50 °C: 9.9 d pH 9, 25 °C: 213 d (extrapolated) pH 9, 20 °C: 380 d (calculated with Arrhenius equation) Metabolites: 2-epi-avermectin B_{1a}: 25% of AR at 50 and 60 °C 1,18 hydrolysed avermectin B_{1a}: 17.5% of AR at 60 °C unknown: 15.6% of AR at 60 °C	Ellgehausen, H 2001
Photochemical stability in water [23–^{14}C] avermectin B_{1a}	Xenon lamp. DT_{50}: 2 d (equivalent to 1.5 sunlight days at 30–50 °N, pH 7) Metabolites: 8α-oxo-avermectin B_{1a}: 5.6% of AR [8,9-Z]-avermectin B_{1a}: 8.2% of AR, DT_{50},photo 5.8 sunlight days at 30–50 °N	Adam, D 2001
Dissociation constant	No dissociation or spectral changes were observed in the 1–12 pH range at 20 ºC	Hörmann, A 1999

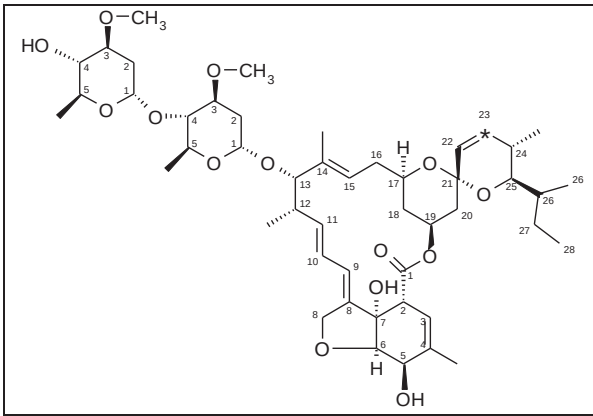
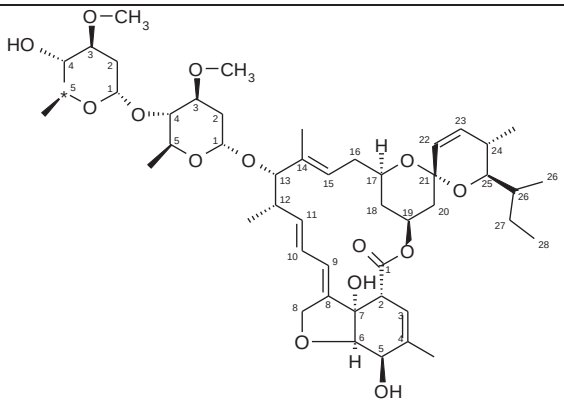
The abamectin technical material of a purity of 96.7% was used for colour, physical state, vapour pressure, melting point, octanol/water partition coefficient, solubility in organic solvents, density, dissociation constant and thermal stability studies. The radio-labelled avermectin B_{1a} used for hydrolysis in water and photochemical stability in water had a radiochemical purity of ≥ 95.6%. The abamectin technical material used for aqueous solubility determination was of unknown purity.

Technical grade material.

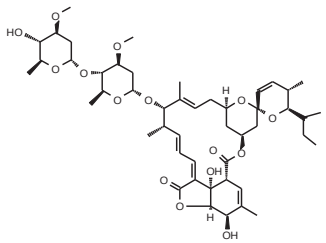
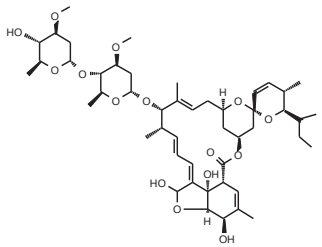
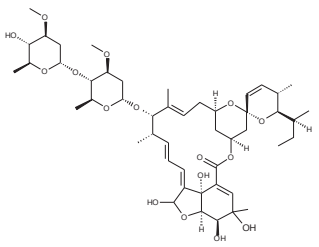
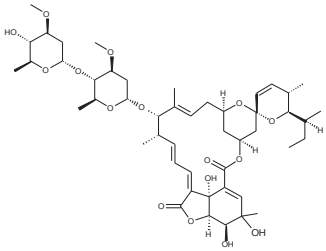
Property	Results	Reference
Minimum purity	Min. 850 g/kg	EC COMMISSION DIRECTIVE 2008/107/EC
Melting Range	Melting range: 161.8 °C–169.4 °C, with thermal decomposition during melting	Das, R 1999; 1999a
Stability (thermal)	Decomposition starts at about 162 °C (see also ‘melting range’)	Das, R 1999; 1999a

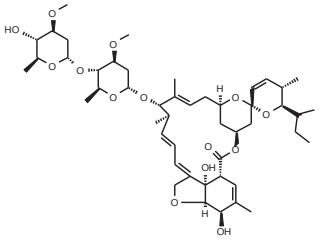
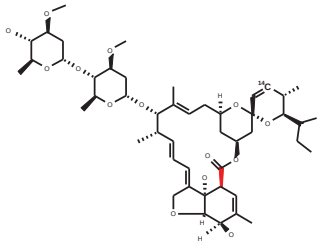
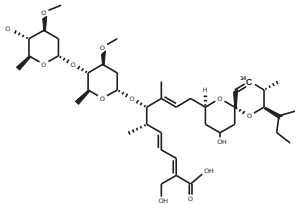
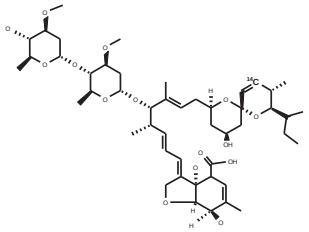
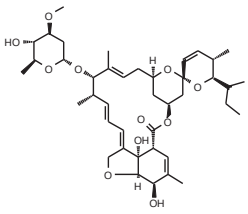
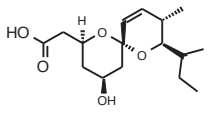
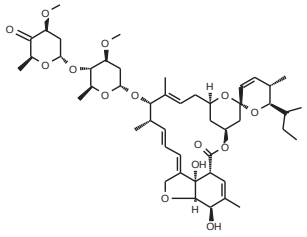
ENVIRONMENTAL FATE AND METABOLISM

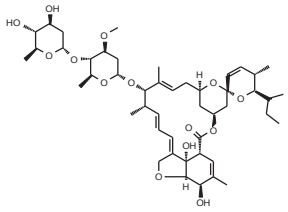
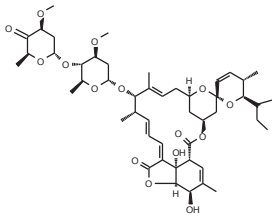
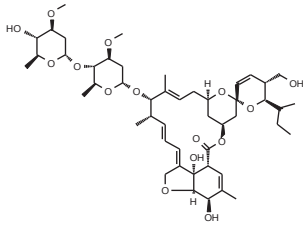
The fate and behaviour of abamectin in soils, water, plants and animals were investigated using [^{14}C] and/or [^{3}H] labelled avermectin $B_{1a.}$

	
[^{14}C] avermectin B_{1a}: mixture of five single ^{14}C-labelled compounds at C3, C7, C11, C13, and C23 of the main complex. A radioactive label only at the C23 position was also used in some studies ([23-^{14}C])	[^{3}H] avermectin B_{1a}: labelled at C5 of the main complex
Used on studies with soil, citrus, cotton and celery, tomato	Used on studies with soil, celery and lactating goat

The chemical structures of the major degradation compounds arising from the environmental fate and metabolism studies are shown below.

Name	Structure	Compound found in
8α-oxo-avermectin B_{1a}		Aerobic soil Tomato Rat
8α-hydroxy-avermectin B_{1a}		Aerobic soil Celery Tomato Rat
4,8α-dihydroxy-avermectin $_{B1a}$ (also 4,8α-dihydroxy-$\Delta^{2,3}$-avermectin $_{B1a}$)		Aerobic soil
8α-oxo-4-hydroxy-avermectin $_{B1a}$ (also 8α-oxo-4-hydroxy-$\Delta^{2,3}$-avermectin B_{1a})		Aerobic soil

Name	Structure	Compound found in
8,9-Z isomer of avermectin B_{1a}		Soil photolysis Citrus Cotton Celery Tomato
2-Epi-avermectin B_{1a}		Hydrolysis product at pH 9
DT3		Hydrolysis product at pH 9
1,18-hydrolysed avermectin B_{1a}		Hydrolysis product at pH 9
Monosaccharide of avermectin B_{1a} or 4'-O-de(2,6-dideoxy-3-O-methyl-α-L-arabino-hexopyranosyl)-5-O-demethyl-avermectin A_{1a} (Unknown 1)		High temperature hydrolysis
((2S,4S,6S,8R,9S)-8-sec-Butyl-4-hydroxy-9-methyl-1,7-dioxa-spiro[5.5]undec-10-en-2-yl)-acetic acid (I_4)		Tomato
4"-oxo-avermectin B_{1a}		Tomato

Name	Structure	Compound found in
3"-O-desmethyl-avermectin B_{1a}		Tomato Goat, Rat
4"-,8α-di-oxo-avermectin B_{1a} (I_{37})		Tomato
(24-hydroxymethyl) avermectin B_{1a}		Goat, Rat

ENVIRONMENTAL FATE

Aerobic degradation in soil

The degradation of [^{14}C]avermectin B_{1a} was investigated in the laboratory under aerobic conditions in one soil (Gartenacker loam) incubated at 20 °C (Nicollier, 2001). The test substance was applied to the soil at a rate of 0.22 mg/kg, equivalent to a field rate of 0.28 kg ai/ha assuming a soil density of 1.3 g/cm^3 and uniform distribution in the upper 10 cm soil layer. Aerobic samples were incubated over 365 days with a soil moisture content of 40% of the maximum water holding capacity. Sampling intervals were immediately after application (0 days) up to 365 days. Samples were submitted to exhaustive extraction and the extracts were analysed by two dimensional TLC and by HPLC. The identity of the soil metabolites was determined by liquid chromatography/mass spectrometry (LC/MS) and nuclear magnetic resonance spectroscopy (NMR). The extracted radioactivity declined from 97.9% at day 0 to 30.6% of the applied radioactivity (AR) at the end of the study (Table 1). Non-extracted residues increased during the study and reached 33.9% AR at Day 365. Non-extracted residues from Day 168 sample were submitted to reflux under neutral and acidic conditions, releasing 5.7% AR. Fractionation of non-extracted residues showed 6–10% AR associated with the fulvic, humin and humic acid fractions. Organic volatiles were ≤ 0.1% AR. The amount of avermectin B_{1a} declined from 97.9% at Day 0 to 1.4% AR at Day 365. 8α-oxo-avermectin $B_{1a}$and 8α-hydroxy avermectin B_{1a} reached a maximum at Day 28. Two minor metabolites were identified as 4,8α-dihydroxy-avermectin B_{1a} and 8α-oxo-4-hydroxy-avermectin B_{1a} amounting at maximum to 9.3% AR. All other metabolites individually represented ≤ 4.1% AR.

Table 1 Distribution of degradation products of avermectin B_{1a} under aerobic conditions (%AR)

Incubation Time	Extracted residues	$^{14}CO_2$	Non-extracted residues	Avermectin B_{1a}	8α-oxo-avermectin B_{1a}	8α-hydroxy avermectin B_{1a}	4,8α-dihydroxy-avermectin B_{1a}	8α-oxo-4-hydroxyavermectin B_{1a}	Recovery
0	97.9	n.d.	0.7	97.9	n.d.	n.d.	n.d.	n.d.	98.6
3	98.6	0.1	2.5	86.8	3.1	5.5	0.2	n.d.	101.2
7	94.9	0.3	5.2	68.2	6.4	9.0	0.9	0.5	100.4
14	90.5	0.8	8.5	51.9	7.5	13.2	2.6	1.3	99.8
28	84.0	1.8	13.6	33.2	10.3	15.7	5.5	3.1	99.5
56	71.0	4.9	21.0	16.7	9.1	13.9	8.9	5.1	96.8
90	63.4	7.8	25.3	9.2	8.0	8.8	9.3	7.8	96.4
120	55.2	11.8	29.0	5.7	4.8	5.2	9.0	8.2	96.0
168	49.8	14.8	29.7	4.5	3.4	3.4	8.2	8.5	94.4
240	39.4	23.6	33.6	3.5	4.1	1.1	5.2	8.3	96.6
294	34.7	23.5	32.3	2.3	1.3	0.9	4.5	7.1	90.6
365	30.6	27.6	33.9	1.4	0.9	0.7	3.8	6.5	92.1

n.d. = Not detected

Avermectin B_{1a} was rapidly degraded under aerobic conditions with a half-life of 18 days. Avermectin B_{1a} was either hydroxylated to 8α-hydroxy avermectin B_{1a} or oxidised to 8α-oxo avermectin B_{1a}. Both of these major metabolites were further hydroxylated with half-lives of 35.4 and 32.5 days, respectively. The endpoint of the metabolic pathway under aerobic conditions was mineralisation to carbon dioxide accounting for up to 27.6% AR, accompanied by the formation of unextracted residues. Table 2 summarizes the half-lives and DT_{90} values for avermectin $B_{1a}$and metabolites.

Table 2 Half-lives and DT_{90} values for avermectin B_{1a} and soil metabolites under aerobic conditions (Nicollier, 2001)

Compound	DT_{50}(days)	DT_{90}(days)
avermectin B_{1a}	18.0	59.6
8α-oxo-avermectin B_{1a}	32.5	108.0
8α-hydroxy-avermectin B_{1a}	35.4	117.8
4,8α-dihydroxy-avermectin B_{1a}	105.2	349.4
8α-oxo-4-hydroxy-avermectin B_{1a}	83.3	276.8

The degradation of [23-^{14}C]-labelled avermectin B_{1a} was investigated in Gartenacker soil (loam/silt loam) under various conditions (Adam, 2001a). Soil samples were treated with avermectin B_{1a} at 0.1 mg/kg dry soil, corresponding to a field rate of 100 g ai/ha. Samples were incubated under aerobic conditions in the dark at a temperature of 30, 20 and 10 °C with a soil moisture content of 40% water holding capacity (WHC; Series 1, Series 2 and Series 3, respectively). In addition, one experiment was performed at 30 °C and 25% WHC (Series 4). Duplicate samples were taken for analysis at each sampling time and submitted to exhaustive extractions before analysis by TLC and HPLC.

The distribution of radioactivity and metabolites at different sampling dates are summarized in Table 3. The extracted radioactivity declined from the beginning to the end of the study, followed by an increase in the non-extracted residues. When non-extracted residues of Day 120 samples were submitted to reflux under neutral and acidic conditions, 4 to 6% AR were released for series 1, 2, 3 and 4, respectively. Subsequent fractionation of the unextracted residues showed that 3 to 12.6% AR associated with the fulvic acid, humic acid and humin fraction.

The amount of avermectin B_{1a} declined from over 90% AR on Day 0 to up to 22.6% on Day 120 (Table 3). 8α-hydroxy-avermectin B_{1a}, formed as major metabolite under all four conditions, reached its highest level on Day 28; 8α-oxo-avermectin B_{1a} was formed above 10%

AR only in series 1, 2 and 3. Two other metabolites, 4,8α-dihydroxy-avermectin B_{1a} and 8α-oxo-4-hydroxy-avermectin B_{1a}, were found in amounts up to 9.9% depending on the incubation conditions (Table 5). Up to 19 minor metabolites were formed during the course of the study, each representing ≤ 5% AR.

Table 3 Recovery of radioactivity in % of applied radioactivity and distribution of metabolites after application of avermectin B_{1a} to soil

DAT, days	$^{14}CO_2$ and Volatiles	Avermectin B_{1a}	8α- oxo-	8α-hydroxy-	4,8α-dihydroxy	8α-oxo-4-hydroxy-	Unknown [a]	Unextracted residues	Total
Series 1 (40% WHC, 30 °C)									
0	–	93.4	2.3	n.d.	n.d.	n.d.	3.9	1.0	100.1
3	0.3	82.4	1.5	4.9	0.4	n.d.	6.5	3.3	98.5
7	0.4	65.6	7.1	7.7	1.3	0.7	9.7	4.2	97.3
14	1.0	49.7	8.1	11.5	2.5	2.2	12.8	7.7	95.7
28	2.8	29.3	13.8	13.0	4.1	2.4	16.9	17.9	99.3
56	7.7	8.9	8.1	7.6	6.3	6.2	21.8	27.3	96.5
90	6.6	8.6	7.7	8.0	4.7	4.3	22.5	26.4	94.6
120	17.0	3.7	4.3	3.5	3.2	6.0	22.0	34.9	97.5
Series 2 (40% WHC, 20 °C)									
0	–	92.6	1.5	n.d.	n.d.	n.d.	2.9	1.3	98.4
3	0.1	81.0	2.9	3.4	0.3	n.d.	4.7	2.3	97.8
7	0.2	72.3	5.2	6.4	1.0	0.3	7.3	2.9	99.6
14	0.7	58.5	10.6	10.4	1.8	1.1	8.2	5.0	99.4
28	1.5	39.4	9.0	13.0	3.9	1.8	16.7	8.9	96.8
56	3.9	16.0	10.2	11.3	7.2	4.8	22.0	19.1	97.4
90	6.5	8.1	8.5	7.2	9.9	8.2	22.3	24.0	98.0
120	8.1	6.7	7.3	6.0	8.4	7.0	24.7	26.9	98.1
Series 3 (40% WHC, 10 °C)									
0	–	90.0	1.8	n.d.	n.d.	n.d.	2.8	1.2	96.1
3	< 0.1	85.3	2.4	2.3	n.d.	n.d.	3.4	1.8	97.8
7	0.1	86.1	3.7	4.7	0.6	n.d.	5.1	1.7	102.3
14	0.2	78.0	4.6	8.1	0.9	n.d.	7.0	2.7	101.5
28	0.4	64.9	5.6	11.2	1.6	0.7	9.5	5.9	101.8
56	1.0	46.0	7.0	13.2	3.1	1.6	16.0	9.2	99.6
90	1.4	32.0	10.8	15.0	4.7	2.3	21.8	11.7	103.5
120	1.5	22.6	10.8	12.7	7.1	4.4	22.2	13.8	97.8
Series 4 (25% WHC, 30 °C)									
0	–	93.0	2.2	n.d.	n.d.	n.d.	2.7	1.2	99.3
3	0.1	85.7	4.1	3.9	0.2	n.d.	4.3	3.2	101.6
7	0.2	73.3	5.5	7.5	0.7	n.d.	7.3	4.5	99.8
14	0.6	58.6	7.0	10.9	2.0	1.6	10.8	7.5	99.0
28	1.9	41.5	7.1	12.3	3.1	2.7	13.4	14.9	99.1
56	3.8	18.6	9.3	12.9	7.3	6.6	19.5	20.6	100.5
90	6.0	10.2	8.9	9.9	8.8	8.2	25.2	23.4	102.7
120	8.2	5.6	7.5	7.6	9.0	9.2	25.6	26.6	101.2

n.d. = Not detected

[a] Unknown = Sum of all other metabolites (up to 19; each single metabolite < 4.9%)

Table 4 summarizes the half-lives and DT_{90} values for avermectin B_{1a} and metabolites under various conditions.

Table 4 Degradation kinetics for [^{14}C]avermectin $B1_a$ under various conditions (Adam 2001)

	Series 1; 30 °C 40% WHC	Series 2; 20 °C 40% WHC	Series 3; 10 °C 40% WHC	Series 4; 30 °C 25% WHC
avermectin B_{1a}				
DT_{50}, days	16.0	21.3	52.7	22.7
DT_{90}, days	53.1	70.6	175.0	75.3
8α-oxo-avermectin B_{1a}				

DT_{50}, days	32.6	42.4	n.a.	49.1
DT_{90}, days	108.2	140.9	n.a.	163.0
8α-hydroxy-avermectin B_{1a}				
DT_{50}, days	22.7	35.6	n.a.	41.3
DT_{90}, days	75.3	118.2	n.a.	137.1

n.a. = Not applicable (metabolite concentration still increasing at the end of the study)

The degradation of [23-^{14}C]-labelled avermectin B_{1a} was investigated in Pappelacker soil (loamy sand), 18 Acres soil (sandy clay loam), and in Marsillargues soil (silty clay loam) under aerobic conditions at 20 ± 2 °C in the dark (Phaff, 2012). Soils were treated with avermectin B_{1a} at 0.125 mg/kg dry soil, incubated over 196 days under aerobic conditions in the dark with a soil moisture content of 40% water holding capacity (WHC). Samples were taken for analysis at 0 up to 196 days after treatment and submitted to exhaustive extraction procedures. The extracts were concentrated and analysed by TLC and HPLC.

The distribution of radioactivity and the metabolites at different sampling dates are summarized in Table 5. Non-extracted residues reached at least 30% AR. Day 126 samples submitted to reflux under neutral and acidic conditions released from 5.6 to 13.6% AR. Subsequent fractionation of the unextracted residues showed the up to 13.7% AR associated with fulvic acid, humic acid and humin. Avermectin B_{1a}residues declined from over 95% AR at the start of the experiment to < 7% AR at Day 196; 8α-oxo-avermectin $B_{1a}$and 8α-hydroxy-avermectin B_{1a} were the major metabolites found, in addition to 4,8α-dihydroxy-avermectin B_{1a} and 8α-oxo-4-hydroxy-avermectin B_{1a}.

Table 5 Recovery of radioactivity in % of applied radioactivity and distribution of metabolites after application of avermectin B_{1a} to various soils

Days after appl.	$^{14}CO_2$ and Volatiles	Avermectin B_{1a}	8α-oxo-avermectin B_{1a}	8α-hydroxy-avermectin B_{1a}	4,8α-di-hydroxy-avermectin B_{1a}	8α-oxo-4-hydroxy-avermectin B_{1a}	Unextracted residues	Total
Pappelacker soil								
0 [a]	–	98.0	n.d.	0.6	0.5	n.d.	0.1	100.9
3	n.d.	95.2	1.2	3.1	n.d.	n.d.	1.0	103.1
7 [a]	0.1	84.0	1.8	4.3	0.3	0.3	2.0	98.8
14	0.3	71.8	4.3	7.7	0.7	0.8	4.1	100.6
28 [a]	1.2	40.3	9.1	13.4	3.6	3.0	10.4	96.9
57 [a]	4.3	16.7	8.7	10.6	6.4	5.7	18.3	95.0
91	5.1	8.1	5.7	6.9	7.6	6.1	23.3	85.5
126 [a]	9.7	4.9	4.4	3.9	7.1	9.9	28.4	93.8
161	15.5	5.7	3.2	1.2	5.1	8.9	30.9	91.1
196 [a]	18.7	4.0	1.6	1.0	5.4	8.9	33.0	92.1
18 Acres Soil								
0 [a]	–	95.8	0.5	n.d.	n.d.	0.2	0.0	99.9
3	0.1	90.1	1.8	n.d.	n.d.	1.9	1.0	102.9
7 [a]	0.1	59.9	3.5	n.d.	0.4	3.9	5.4	99.8
14	0.7	40.9	3.8	0.6	0.1	3.3	14.0	101.1
28 [a]	2.3	15.4	2.6	0.7	0.3	2.2	26.2	95.4
57 [a]	6.4	9.9	1.8	0.9	0.2	0.6	34.8	91.7
91	12.4	8.3	1.4	0.9	0.1	0.3	39.1	93.4
126 [a]	12.5	6.9	1.1	0.7	0.5	0.2	39.6	91.3
161	12.9	5.1	0.6	0.2	n.d.	0.1	43.3	91.9
196 [a]	12.5	5.1	1.0	0.5	n.d.	0.2	44.1	90.9
Marsillargues Soil								
0 [a]	–	98.2	0.2	0.1	n.d.	n.d.	0.1	99.6
3	n.d.	91.3	0.5	1.5	0.1	n.d.	0.7	96.6
7 [a]	n.d.	93.2	1.1	2.9	0.2	n.d.	1.2	103.9
14	0.2	81.4	3.0	4.8	0.3	n.d.	3.2	100.5
28 [a]	0.5	61.8	4.2	7.1	0.6	0.4	6.2	96.7
57 [a]	1.2	44.2	5.1	8.1	1.8	2.0	11.2	93.7
91	4.1	26.8	4.7	8.8	3.1	2.3	18.4	95.8

Days after appl.	$^{14}CO_2$ and Volatiles	Avermectin B_{1a}	8α-oxo-avermectin B_{1a}	8α-hydroxy-avermectin B_{1a}	4,8α-di-hydroxy-avermectin B_{1a}	8α-oxo-4-hydroxy-avermectin B_{1a}	Unextracted residues	Total
126 [a]	4.1	18.2	6.0	7.6	3.1	2.5	22.9	92.3
161	6.9	12.4	5.3	6.0	5.5	5.2	27.2	90.4
196 [a]	13.4	6.6	3.5	4.0	2.2	2.6	30.0	91.5

n.d. = Not detected

[a] Mean of two duplicates

Table 6 summarizes the half-lives and DT_{90} values for avermectin B_{1a} and metabolites in various soils.

Table 6 Degradation kinetics for [^{14}C]avermectin B_{1a} and metabolites in various soils (Phaff, 2012)

	Pappelacker	18 Acres	Marsillargues
r^2 (first order kinetics)	0.99126	0.97373	0.9924
Avermectin B_{1a}			
DT_{50}, days	25.4	11.6 (10.7 [a])	52.2
DT_{90}, days	84.4	38.6 (53.9 [a])	173.3
8α-oxo-avermectin B_{1a}			
DT_{50}, days	20.9	–	49.5
DT_{90}, days	69.3	–	164.4
8α-hydroxy-avermectin B_{1a}			
DT_{50}, days	27.7	–	50.3
DT_{90}, days	92.1	–	167.1
4,8α-dihydroxy-avermectin B_{1a}			
DT_{50}, days	99.7	–	41.5
DT_{90}, days	331.2 [b]	–	137.8
8α-oxo-4-hydroxy--avermectin B_{1a}			
DT_{50}, days	192.2	–	22.2
DT_{90}, days	638.4 [b]	–	73.7

[a] Two compartment model

[b] Extrapolated values

The degradation of ^{3}H-labelled avermectin B_{1a} and ^{14}C-labelled avermectin B_{1a} was investigated in the laboratory under aerobic conditions in three different soils (Lufkin fine sandy loam, Houston clay and a coarse “construction grade” sand) incubated at 25 °C, at a soil moisture level of 75% of Field Capacity (Ku & Jacob, 1983). The test substance was applied to the soil at 0.1, 1.0 and 50 mg/kg. Samples were submitted to exhaustive extraction and the extracts analysed by TLC and HPLC. In order to account for the loss of radioactivity in all the aerobic soil studies a study was carried out with a biometer flask containing Lufkin fine sandy loam treated with ^{14}C-labelled avermectin B_{1a} (10 mg/kg) to determine the amount of $^{14}CO_2$ produced during the course of the study.

Avermectin B_{1a} degraded at a fairly rapid rate to at least 13 radioactive products, the major fraction being an equilibrium mixture (ratio of 1:2.5) of the 8-α hemiacetal derivative and the corresponding ring-opened hydroxy aldehyde derivative of avermectin B_{1a}, identified by NMR, MS and FTIR. Minor products, which individually never exceeded 2–3% AR, were found in addition to the metabolites listed in Table 7. The mineralisation of ^{14}C-labelled avermectin B_{1a} to carbon dioxide reached a maximum of 3.2% during a 21 week study.

Table 7 Soil degradation of [^{3}H]avermectin B_{1a} and [^{14}C]avermectin B_{1a} under aerobic conditions, in %AR [a]

Days after application	Volatiles [a]	Avermectin B_{1a}	8α-hydroxy avermectin B_{1a}	Non-extracted	Days after application	Avermectin B_{1a}	8α-hydroxy avermectin B_{1a}	Non-extracted
50 mg/kg [^{3}H]avermectin B_{1a}; Lufkin fine sandy loam					0.1 mg/kg [^{3}H]avermectin B_{1a}; Lufkin fine sandy loam,			
0	0	96	0.4	3.0	0	95.1	0	4.9
14	0.3	81	8.3	2.4	7	93.2	0	4.9
28	1.9	62.9	13.1	3.0	14	67.3	7.3	6.8
56	7.8	36.8	16.1	6.2	28	44.4	16.7	15.5
112	16.6	16.8	15.5	8.5	56	21.6	18.5	21.4
168	27.6	5.8	5.9	12.2	84	15.4	17.0	30.1
					168	5.3	13.3	35.0
1 mg/kg [^{3}H]avermectin B_{1a}; sand					1 mg/kg [^{3}H]avermectin B_{1a}; Lufkin fine sandy loam			
0	0	99.2	0	0.8	0	94.7	0	5.3
14	0.7	65.8	6.4	2.5	7	83.1	5.1	6.0
28	2.9	64.9	9.7	3.8	14	60.6	12.3	7.3
56	8.2	47.4	13.2	7.2	28	35.5	17.4	9.3
84	11.7	40.1	18.2	7.1	56	18.0	20.1	17.6
112	16.5	22.9	15.1	11.8	84	9.1	14.8	23.7
168	22.5	21.9	20.1	12.5	112	7.1	13.5	27.5
252	31.7	9.8	15.8	17.3	168	3.6	0.0	19.8
1 mg/kg [^{3}H]avermectin B_{1a}; Houston clay loam					0.1 mg/kg [^{3}H]avermectin B_{1a}; Houston clay loam			
0	0	94.4	0	5.6	0	94.9	0	5.1
28	2.6	60.4	4.9	10.1	21	54.6	11.2	9.1
56	6.6	51.6	6.0	11.5	28	47.8	13.4	13.1
84	12.6	22.4	13.0	17.0	56	29.6	18.4	17.2
112	17.9	22.7	14.8	15.8	84	19.4	18.7	20.2
168	25.6	11.3	8.5	18.8	112	12.5	14.4	21.2
252	33.4	11.2	11.4	18.1	168	12.0	14.3	26.3
448	45.5	8.1	5.2	16.8	252	7.5	13.7	21.2
1 mg/kg [^{14}C]avermectin B_{1a}; Lufkin fine sandy loam					1 mg/kg [^{14}C]avermectin B_{1a}; Lufkin fine sandy loam			
0	n.m.	97.9	0.0	2.1	0	99.0	0	1.0
28	n.m.	59.6	10.5	5.2	14	50.3	12.0	6.9
56	n.m.	45.8	15.0	7.7	28	25.2	16.1	10.9
84	n.m.	27.7	17.6	11.6	56	11.0	8.9	15.8
112	n.m.	18.4	11.8	27.4	84	8.1	8.4	18.8

[a] Average of duplicates

n.d. = Not detected

n.m. = Not measured

In experiments with [^{3}H]avermectin B_{1a} there were substantial quantities of volatile radioactive material (approximately 27.6–45.5% of the dose through the experiments) condensed in the water which was used to maintain the level of relative humidity. Since none of this radioactive material partitioned into dichloromethane it is concluded that it represents tritiated water rather than volatile organic materials. As the specific activity of ^{3}H-labelled avermectin B_{1a} was unchanged after 28 days of exposure it can be concluded that there was no apparent tritium exchange upon ageing of [^{3}H]avermectin B_{1a} in treated soil. The apparent release of tritium resulted from metabolic oxidation at the C5 position of the parent molecule or a degradate.

Unextracted residues increased with time, reaching a maximum of 12.2 to 35.0% AR. In most cases, there was a progressive increase in % AR which could not be accounted for in the radio-balance assessment, reaching values below 52% AR at the end of incubation. Since this loss was also observed among samples held in containers in which condensed volatile radioactive material was measured, it was assumed that the trapping of these volatiles was inefficient. Table 8 shows the half-lives estimated for avermectin B_{1a} in the various soils.

Table 8 Estimated DT_{50} values for degradation of [^{3}H] and [^{14}C]avermectin B_{1a} in various soils under aerobic conditions (Ku and Jacob 1983a)

Application rate [mg/kg soil]	Lufkin fine sandy loam	Construction grade sand	Houston clay
0.1	20 [a]	–	28
1.0	20 [b]	47 [a]	36 [a]
50	40 [a]	–	–

[a] [^{3}H] label

[b] [^{3}H] and [^{14}C] label

Soil photolysis

[^{14}C]avermectin B_{1a} was applied at a rate of 0.09 kg/ha onto the surface of a moist (75% FC) 2 mm soil layer and irradiated with a xenon arc light source in a wavelength range of 300–400 nm and at a light intensity of 84.7 ± 3.8 Wm^{-2}(Phaff, 2001). The mean temperature of the soil layers was kept at 24.5 ± 0.1 °C. The total irradiation time was 336 hours of xenon light (28 days incubation) equivalent to 47 days of natural summer sunlight (NSS) at latitudes 30 to 50 °N. Irradiation was performed in cycles of 12 hours xenon light and 12 hours darkness. Dark control samples were incubated for 28 days. Replicate samples were taken at 0 to 28 days, extracted and analysed by TLC and HPLC.

The overall recovery of radioactivity ranged between 96.9 and 102.8% AR for the irradiated samples (Table 9) and between 101.8 and 104.8% AR for the dark controls. At the end of the irradiation period, avermectin B_{1a} accounted for 19.5%AR in the irradiated soil (Table 9) and 86% AR in the control. In addition to the parent compound, six minor photoproducts were formed in the irradiated samples, two identified as 8α-oxo-avermectin B_{1a} and 8α-hydroxy-avermectin B_{1a} (Table 9). All other degradation products were below 5.3%. In the dark control samples four degradation products were observed, two of them were identified as 8α-oxo-avermectin B_{1a} and 8α-hydroxy-avermectin B_{1a} (≤ 5%). Under irradiation, non-extracted radioactivity increased from 0.3% at Day 0 up to 25.9% at the end of the study, and volatiles in the form of $^{14}CO_2$ amounted to 7.6%.

Table 9 Recovery of radioactivity in % of applied radioactivity and distribution of metabolites after application of avermectin B_{1a} to soil and irradiation

Incub. Time [d].	Irrd. Time [hours]	Irrd. Time Summer sunlight 30–50 °N [d]	Avermectin B_{1a}	8α-oxo-avermectin B_{1a}	8α-hydroxy-avermectin B_{1a}	Unknown [a]	Volatiles [a]	Unextracted residues	Total
0	0	0	100.3	1.0	n.d..	1.2	n.d..	0.3	102.8
2	24	3	67.7	4.1	2.6	10.6	0.4	15.6	101.0
4	48	6	77.3	3.6	2.9	8.1	0.7	9.1	101.7
6	72	10	66.7	4.1	2.8	11.1	1.6	13.6	100.1
10	120	17	52.4	3.7	4.0	22.8	2.5	16.2	101.5
15	180	25	42.4	3.4	3.5	27.1	3.1	18.8	98.3
21	252	35	28.6	5.7	3.3	31.2	4.5	22.6	97.2
28	336	47	19.5	4.5	3.1	36.2	7.6	25.9	96.9

n.d. = Not detected

[a] Sum of unidentified zones [TLC), ≤ 5.1% each

In the irradiated samples, avermectin B_{1a} degraded with a net photolysis DT_{50} of 21.7 days assuming first order kinetics (Table 10).

Table 10 Half-lives and DT_{90} values for avermectin B_{1a} on soil in the dark, under irradiation and converted to summer sunlight days

Incubation conditions	DT_{50}, days		DT_{90}, days	
	Sun test	30–50 °N	Sun test	30–50 °N
Dark controls; k_1 = 0.0058 (pseudo 1st order kinetics)	119.5		397.0	–
Irradiated; k_2 = 0.0597 (pseudo 1st order kinetics)	11.6	19.5	38.6	65.1
Irradiated, corrected for dark controls; k_3 = 0.0539 (k_2–k_1))	12.9	21.7	42.7	72.0

A soil photolysis study was conducted using [^{3}H]avermectin B_{1a} applied to a clay loam soil kept outdoors at latitude 40.5 ºN during the summer (Ku & Jacob, 1983a). Soil TLC plates (20 cm × 20 cm) were prepared by spreading a slurry of air dried soil (40 g) and methanol (30 mL) and air dried at room temperature before use. Approximately 50 µL of a solution of [^{3}H]avermectin B_{1a} (0.85 mg/mL methanol) was applied to several pre-scored soil thin layer plates (6.5 cm^2). The treated plates were exposed to sunlight and sampled at 0 to 31 hours. At each sampling time, a square of the soil thin film was carefully scrapped off the plates, transferred to a glass column, eluted with ethyl acetate followed by methanol and the eluents analysed by HPLC. Soil residues were air-dried and combusted for radio assay. Total recovery [%] of [^{3}H]avermectin B_{1a} from the soil thin layer extracts is presented in Table 11.

Table 11 Photodegradation of [^{3}H]avermectin B_{1a} in soil thin layer plates exposed to sunlight

Exposure Time [hr]	% [^{3}H]avermectin B_{1a} remaining		
	Ethyl acetate extract	Methanol extract	Total
0	93.1	5.7	98.8
1	84.7	6.4	91.1
2	82.7	6.2	88.9
4	78.0	6.9	84.9
8	70.5	8.6	79.1
16	56.8	6.4	63.2
31	27.3	5.3	32.6

A plot of the logarithm of the remaining [^{3}H]avermectin B_{1a} against time gives a straight line, indicating first order kinetics. The calculated half-life (DT_{50}) from this plot is approximately 21 hours. The metabolic pathway of avermectin $B1_a$ in soil is proposed in Figure 1.

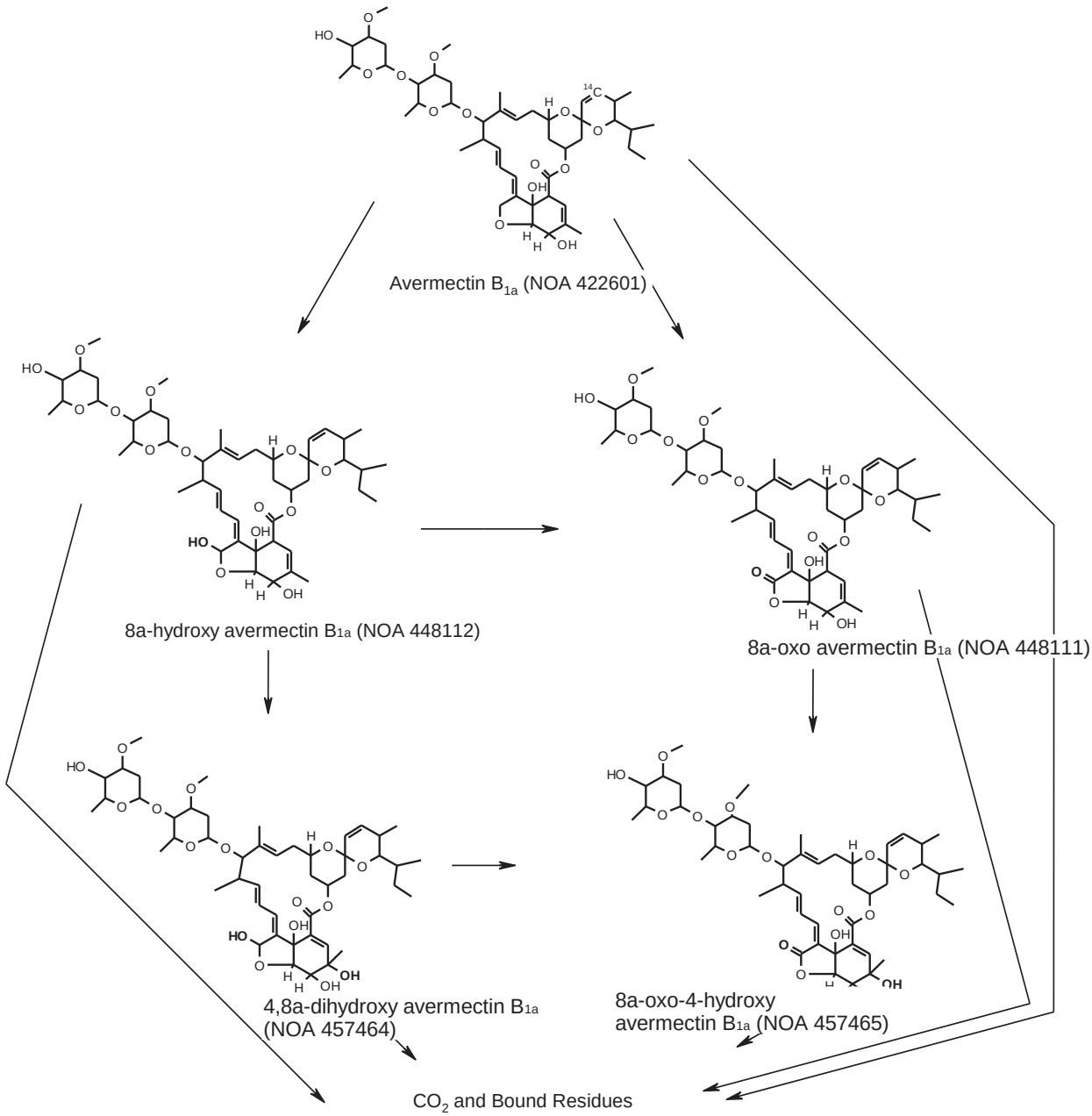


Figure 1 Metabolism of avermectin B_{1a} in soil

Plant metabolism

Citrus

The metabolism of [^{14}C]avermectin B_{1a} was investigated in citrus plants (oranges, lemons and grapefruit) (Maynard *et al.*, 1989). An open wooden frame with a fibreglass roof was constructed over each tree to minimize the reduction in residues by atmospheric precipitation. Solutions of [^{14}C]avermectin B_{1a}were prepared in an EC formulation blank (8 and 80 mg ai/L), and 0.5 mL solution was painted on each fruit using a small brush. Twenty one oranges, lemons and grapefruit were each treated with the 8 mg ai/L solution (4 µg), resulting in an initial concentration of 18 to 36 µg ai/kg on a whole fruit basis. Seventy eight oranges on two adjacent trees were treated with the 80 mg ai/L solution, resulting in initial deposits of 40 µg ai per whole fruit. Samples (three fruits) were collected on the day of application up to 12 weeks post application. For the 80 mg ai/L treatment, 15 additional fruits were sampled at weeks 2 to 12.

Each fruit was rinsed twice with methanol, the fruits peeled, the pulp rinsed with tap water, dried with a paper towel, combusted and the radioactivity, trapped as CO_2, measured. The skin was blended with dry ice, a portion taken for combustion analysis, the remainder extracted with acetone, the extracted dried, the residue partitioned between dichloromethane and water. The radioactivity remaining in the peel solids after acetone extraction was exhaustively extracted with methanol and tetrahydrofuran, followed by six additional methanol extractions or subjected to five successive Bligh-Dyer extractions (mixture of chloroform and methanol, dilution with chloroform and water, the chloroform layer containing all the lipids), methanol extraction, Soxhlet extraction, acid and enzyme hydrolysis procedures. Based on preliminary evidence that the degradation of avermectin B_{1a} was primarily photochemical in nature, the degradation of avermectin B_{1a} was investigated in thin film and aqueous photolysis. All extracts were analysed by reversed-phase HPLC.

The decline of the total radioactivity from the treated fruit over a 12-week period is shown in Table 12. At the end of the experiment, the residues ranged from 33.3% (grapefruit) to 49.8% (lemons) of the applied radioactivity (AR).

Table 12 Decline of radio-labelled residues in citrus following application of a [^{14}C]avermectin B_{1a} solution at 8 mg ai/L (4 μg/fruit) or 80 mg ai/L (40 μg/fruit)

Time (weeks)	Total Radioactive Residue, as % of the applied radioactivity [a] (in mg/kg)			
	Orange (8 mg ai/L)	Lemon (8 mg ai/L)	Grapefruit(8 mg ai/L)	Orange (80 mg ai/L)
0	100 (0.050)	100 (0.028)	100 (0.027)	100.0 (0.229)
1	61.3	72.5	60.5	90.0
2	58.7	72.2	52.9	79.0
4	51.6	59.2	48.2	66.3
8	38.4	45.2	41.5	45.1
12	43.9	49.8	33.3	41.6

[a] TRR is the sum of the radioactivity in all the fruit fractions.

In general, most of the residues were rinsed from the surface with methanol (Table 13). No residues were detected in the pulp portion without the peel/pulp interface at both rates for all fruits. When the interface was included, residues reached a maximum of 12–13%TRR after 8 weeks of application.

Table 13 Extracted residues (%TRR) in citrus following application of a [^{14}C]avermectin B1a solution with at 8 mg ai/L (4 μg/fruit) or 80 mg ai/L (40 μg/fruit)

	Orange (8 mg ai/L)			Lemon (8 mg ai/L)			Grapefruit (8 mg ai/L)			Orange (80 mg ai/L)		
Time (weeks)	Methanol rinse	Acetone Peel Extract	Total Extracted	Methanol rinse	Acetone Peel Extract	Total Extracted	Methanol rinse	Acetone Peel Extract	Total Extracted	Methanol rinse	Acetone Peel Extract	Total Extracted
0	98.6	1.1	99.7	100.0	0.0	100.0	98.4	1.7	100.1	98.6	1.2	99.8
1	74.8	16.5	91.3	59.9	20.6	80.5	68.4	20.0	88.4	87.2	8.5	95.7
2	64.1	15.5	79.6	45.0	26.2	71.2	59.3	16.7	76.0	84.0	8.7	92.7
4	52.3	21.0	73.3	28.8	24.9	53.7	43.7	22.3	66.0	73.9	13.3	87.2
8	32.2	31.0	63.2	13.4	29.8	43.2	34.2	22.6	56.8	41.7	28.1	69.8
12	36.3	21.4	57.7	6.7	30.8	37.5	32.7	18.9	51.6	40.9	19.2	60.1

Table 14 shows the characterization of the extracted residues from the fruits treated at the lowest rate. At least 90% TRR was found to be avermectin B_{1a} at Day 0, a level that decreased rapidly at Day 1 (maximum of 17.4% TRR in orange). After 1 day, most of the extracted residues were of a polar nature, accounting for at least 46% TRR at Day 12 in oranges. The moderately polar fraction (up to 12.5% TRR in 1 day orange samples) included 5 to 10 moieties. The 8,9-Z isomer of avermectin B_{1a}, also identified in the photolysis experiment on orange peel sections, accounted for < 5% TRR in all samples.

The acetone-extracted peel from the 2-week 8 mg/kg fruits was extracted three times with methanol followed by three extractions with THF, releasing 51, 40 and 54% of the matrix radioactivity (or 21, 11 and 25% of TRR) for the oranges, lemons, and grapefruits, respectively. The methanol and THF extracts were combined and partitioned between dichloromethane and water; approximately 60% of the radioactivity partitioned into the dichloromethane phase. The spent peel was extracted six times with methanol, and released an additional 7.0, 6.0 and 5.3% of the matrix radioactivity for the oranges, lemons and grapefruits, respectively. Characterization of the extracted radioactivity from the methanol and THF extractions produced polar, moderately polar, avermectin B_{1a} and the 8,9-Z isomer of avermectin B_{1a} fractions. Avermectin B_{1a} represented 2, 7 and 1% of the radioactivity for the oranges, lemons and grapefruits, respectively. The degradate characterization was qualitatively similar to that observed with the acetone extraction for the same samples.

Table 14 Characterization of the Total Extracted Residue (methanol rinse plus acetone peel extract) from fruits treated (4 µg/fruit) of [^{14}C]avermectin B_{1a}

Time (weeks)	Percent of Total Extracted Residue (%) [a]						Recovery as % of TRR
	Polar	Moderately Polar	Avermectin B_{1a}	8,9-Z isomer	Non-Polar	Column Wash	
Orange							
0	3.9	7.8	85.0	2.3	0.1	1.0	91.1
1	56.4	12.5	17.4	3.9	0.8	9.1	90.4
2	66.0	9.8	9.6	2.8	1.5	10.3	78.8
4	67.3	9.1	10.1	3.3	0.8	9.3	72.2
8	53.0	10.9	13.5	4.7	2.6	15.4	61.7
12	46.4	8.4	7.7	3.2	3.4	31.0	56.2
Lemons							
0	2.4	4.6	88.7	1.7	0.3	2.3	89.6
1	79.3	7.3	5.0	1.3	0.3	8.8	79.4
2	76.9	5.2	3.9	1.3	1.0	11.7	69.2
4	82.0	3.9	3.1	1.0	0.6	9.5	51.6
8	79.6	2.5	2.0	0.7	0.5	14.7	40.2
12	79.9	2.0	2.0	0.9	0.3	14.9	34.3
Grapefruit							
0	2.4	3.7	90.0	1.6	0.4	2.0	91.8
1	82.6	6.1	4.4	1.3	0.5	5.2	86.8
2	81.0	4.7	2.9	1.3	0.8	9.2	74.7
4	85.0	2.3	1.7	0.9	0.5	9.5	64.1
8	85.0	2.2	1.6	0.7	0.7	9.7	54.8
12	84.5	2.2	1.2	0.8	0.8	10.4	49.8

[a] Data are presented as percent of the normalized recovered radioactivity

Table 15 shows the work-up of non-extracted residues of the 12 week oranges using an 80 mg ai/L solution treatment. The acetone-extracted peel was extracted by five successive Bligh-Dyer procedures, which recovered 23.8% TRR. A fraction of this extract was tentatively identified by NMR and mass spectrometry as a mixture of linoleic fatty esters. Reverse-phase HPLC showed the major fraction of the radioactivity was polar degradates and avermectin B_{1a} represented between 9 and 12% TRR. The non-extracted residues after Bligh-Dyer (11.8% TRR) were subjected to Soxhlet extraction with methanol and the remaining peel subjected to acid hydrolysis (pH 1.3 for 24 hours at room temperature), leaving 8.8% TRR as non-extracted (Experiment 1). In another experiment, the peel solids remaining from the Bligh-Dyer were subjected to sequential enzymatic hydrolysis (cellulase, pectinase, and ß-glucosidase), that reduced the non-extracted residues to 7% TRR (Table 15).

Table 15 Removal of radioactivity from the orange peel non-extracted from fruit treated with an 80 mg ai/L solution (40 µg/fruit) of [^{14}C]avermectin B_{1a}

Fraction	% Radioactivity in Fraction [a]	% Whole Fruit TRR
12 week DAT 80 mg/kg		100.0
Methanol wash		40.9
Peel residue after methanol wash		54.7
Acetone Extraction		19.2
Bligh-Dyer Extraction		23.8
Experiment 1		
Bligh-Dyer Peel Solid	100	11.8
Methanol Soxhlet	10	1.2
Peel Solid after Soxhlet Extraction	90	10.6
Filtrate after Acid Hydrolysis	16	1.8
Peel Solid after Acid Hydrolysis	75	8.8
Experiment 2		
Bligh-Dyer Peel Solid	100	11.8
Filtrate after Cellulase, Pectinase, ß-glucosidase Hydrolysis	7	0.8
Peel Solid after Enzyme Hydrolysis	93	11.0

Values for solid samples were determined by subtraction of extracted residues from TRR. Combustion of the solid samples was not possible due to the condition of the solid with associated filter paper.

[a] Values are expressed as a percentage of the Bligh-Dyer Peel Solid

Celery

The metabolism of [^{3}H] and [^{14}C]avermectin B_{1a} was investigated in field-grown celery in two experiments (Moye, 1988). In the first, potted celery plants grown under field conditions were treated 10 times at weekly intervals and harvested at maturity. In the second experiment, potted celery plants were treated four times at weekly intervals and harvested as immature plants. [^{14}C]avermectin B_{1a} was applied at 16.8 g ai/ha and [^{3}H]avermectin B_{1a} was applied at 11.2 g ai/ha or 112 g ai/ha. The test material was applied to the foliar portion of the plants as EC formulated solutions at a rate equivalent to 460 L/ha. Two groups of three plants were harvested at each experiment. Immature celery plants were harvested from the [^{3}H]avermectin B_{1a} treatments at 0 day to 6 weeks after the fourth application and mature plants were harvested 0 days to 22 days after the tenth application of [^{3}H]avermectin B_{1a}. Immature celery plants were harvested from the [^{14}C]avermectin B_{1a} treatments at 0 days and 2 weeks after the fourth application and mature plants were harvested 0 day and 1 week after the tenth application. Samples were blended with acetone, an aliquot extracted three to six times with acetone, the residual solid dried and reconstituted with methanol/water (85:15) for chromatography, and further extracted with several solvents, including methanol/water (40:60 v/v). Hot DMSO was used to solubilise lignin and hot sulphuric acid to convert cellulose to glucose.

Residues in immature and mature celery from plants receiving 4 and 10 applications of [^{3}H]avermectin B_{1a} are shown in Table 16. In average, residues in immature leaves and stalks samples at 43 days after the 4th application accounted for < 1% of the residues at Day 0. In mature plants from the 11.2 g ai/ha treatment, residues after 22 days of the 10th application accounted for 23 and 15% of the residues at Day 0 in leaves and stalks, respectively. Similar results were found in plants treated at the higher rate.

Table 16 Radio-labelled residues in celery following application of [^{3}H]avermectin B_{1a} in µg/kg avermectin B_{1a} equivalents. Three plants per group.

	11.2 g/ha				112 g/ha	
DAT, days	Percent of Applied radioactivity (%)	Group 1	Group 2	Mean	Percent of Applied Dose (%)	Group 1
	Immature Plants(leaves/stalks)—4 applications					
0	1.33/0.31	2360/467	3110/632	2740/550	1.36/0.29	26800/6440
7	0.46/0.10	631/125	457/145	544/135	0.41/0.08	7830/2260
14	0.35/0.09	162/55.0	238/66.2	200/60.6	0.31/0.06	2690/851

29	0.21/0.07	25.4/6.20	26.1/7.64	25.7/6.90	0.19/0.04	286/57.1
43	0.20/0.14	13.1/4.82	9.81/3.36	11.5/4.10	0.21/0.08	96.7/21.6
Mature Plants(leaves/stalks)—10 applications						
0	1.86/0.56	207/30.8	186/27.1	196/28.9	2.56/0.56	2140/400
1	1.55/0.42	164/14.7	107/17.7	135/16.2	2.29/0.42	2170/331
3	1.85/0.52	140/14.9	114/11.6	127/13.3	1.84/0.52	1650/204
7	1.58/0.34	96.2/8.70	95.0/7.95	95.6/8.30	1.38/0.34	1134/238
15	1.18/0.28	60.2/6.41	62.5 /4.07	61.4/5.24	0.75/0.28	554/43.8
22	0.79/0.24	49.6/3.68	41.1/5.31	45.4/4.50	0.74/0.24	458/50.9

On average, residues in immature plants harvested at 14 days after the 4th application of [^{14}C]avermectin B_{1a} at 16.8 g/ha accounted for 5,4 and 12% of the 0 day residues for leaves and stalks, respectively (Table 17). In mature plants harvested after 7 days of the 10th application, these values were 38 and 54%, respectively.

Table 17 Radio-labelled residues in celery following application of [^{14}C]avermectin B_{1a} at 16.8 g/ha. Three plants per group.

DAT, days	Percent of Applied Dose (%)	Residue Found (in µg/kg avermectin B_{1a} equivalents)		
		Group 1	Group 2	Mean
Immature Plants (leaves/stalks)–4 applications				
0	1.67/0.19	4890/648	14300/1670	9570/1160
14	0.52/0.08	651/169	387/115	519/142
Mature Plants (leaves/stalks)—10 applications				
0	3.66/0.55	549/41.2	479/32.0	514/36.6
7	1.50/0.30	198/24.9	196/15.0	197/20.0

Most of the residues in immature and mature plants receiving treated with [^{3}H]avermectin B_{1a} and [^{14}C]avermectin B_{1a} were extracted with acetone at all sampling dates (Table 18).

Table 18 Acetone-extracted residues in celery following application of [^{3}H]avermectin B_{1a} at 11.2 and 112 g/ha and [^{14}C]avermectin B_{1a} at 16.8 g/ha, expressed as %TRR

DAT, days	Leaves			Stalks		
	[^{3}H] 11.2 g/ha	[^{3}H] 112 g/ha	[^{14}C] 16.8 g/ha	[^{3}H] 11.2 g/ha	[^{3}H] 112 g/ha	[^{14}C] 16.8 g/ha
	Immature plants					
0	95.8	96.6	97.1	97.0	95.2	96.0
7	80.6	78.3	–	83.3	78.9	–
14	71.4	68.2	69.9	82.1	74.0	74.
29	73.1	63.6	–	75.4	73.6	–
43	68.9	65.6	–	83.5	83.1	–
	Mature plants					
0	70.9	75.3	73.7	79.8	85.1	75.5
1	69.6	77.0	–	78.7	92.0	–
3	66.9	76.4	–	79.0	78.0	–
7	66.4	64.2	57.8	70.9	81.3	67.0
15	62.7	68.6	–	71.8	83.7	–
22	57.9	66.4	–	69.1	77.5	–

HPLC profiling of the acetone extracts from mature and immature celery plants are shown in Tables 19 and 20. Polar metabolites (more polar than parent) accounted for most of the residues in both leaves and stalks. In leaves, polar metabolite residues increased with the DAT, moderately polar metabolites remained relatively constant, while avermectin B_{1a} and its 8,9-Z isomer decreased during the sampling period. Residues in immature stalks showed a different profile, with polar metabolites decreasing and avermectin B_{1a} increasing after 7 days DAT. Further profiling indicated also the presence of 8-hydroxy avermectin B_{1a} (not quantified) and at least ten other unidentified minor components.

Table 19 Metabolic profile of acetone-extracted residues in immature celery following application of [^{3}H]avermectin B_{1a} and [^{14}C]avermectin B_{1a}, % the extracted residues

DAT, days [a]	[^{3}H]avermectin B_{1a} (11.2 g ai/ha)				[^{3}H]avermectin B_{1a} (112 g ai/ha)				[^{14}C]avermectin B_{1a} (16.8 g ai/ha)			
	Polar metabolites	Mod. polar metabolites	B_{1a}	8,9-Z isomer	Polar metabolites	Mod. polar metabolites	B_{1a}	8,9-Z isomer	Polar metabolites	Mod. polar metabolites	B_{1a}	8,9-Z isomer
Leaves												
0 (19)	4.3	16.5	73.4	5.3	3.3	14.1	74.9	7.7	4.7	19.2	65.3	10.8
7 (26)	54.5	19.9	21.2	4.4	50.3	22.3	22.8	4.5	–	–	–	–
14 (33)	53.1	22.8	18.7	5.3	50.0	19.8	25.6	4.6	62.0	17.0	15.8	5.2
29 (48)	66.2	18.2	14.3	1.4	69.8	13.0	14.5	2.6	–	–	–	–
43 (62)	68.4	14.8	15.8	1.1	61.3	12.2	20.5	5.9	–	–	–	–
Stalks												
0 (19)	4.8	22.8	67.7	4.6	3.3	15.3	80.7	0.7	5.6	28.5	54.8	11.2
7 (26)	42.3	27.2	27.0	3.6	36.0	32.1	28.2	3.6	–	–	–	–
14 (33)	33.4	22.3	37.1	4.6	43.4	19.7	30.7	6.2	50.9	14.9	29.2	5.0
29 (48)	34.6	19.6	43.3	2.6	33.4	21.0	37.5	8.1	–	–	–	–
43 (62)	22.7	20.3	56.1	1.0	30.4	24.9	38.6	6.1	–	–	–	–

[a] Numbers in parenthesis are days after 1st application (Four applications made to immature plants)

Table 20 Metabolic profile of acetone-extracted residues in mature celery following application of [^{3}H]avermectin B_{1a} and [^{14}C]avermectin B_{1a}, % the extracted residues

DAT, days [a]	[^{3}H]avermectin B_{1a} (11.2 g ai/ha)				[^{3}H]avermectin B_{1a} (112 g ai/ha)				[^{14}C]avermectin B_{1a} (16.8 g ai/ha)			
	Polar metabolites	Mod. polar metabolites	B_{1a}	8,9-Z isomer	Polar metabolites	Mod. polar metabolites	B_{1a}	8,9-Z isomer	Polar metabolites	Mod. polar metabolites	B_{1a}	8,9-Z isomer
Leaves												
0 (63)	61.0	19.7	15.2	4.0	42.2	19.8	33.0	5.0	33.8	22.5	38.6	5.2
1 (64)	63.4	19.0	14.5	3.1	46.2	23.7	23.9	6.2	–	–	–	–
3 (66)	67.3	17.4	12.7	2.6	65.1	19.4	11.5	4.0	–	–	–	–
7 (70)	68.3	16.7	11.4	2.7	63.7	18.8	14.8	2.7	71.6	16.2	9.8	2.1
15 (78)	72.3	14.5	10.6	1.9	66.7	19.5	9.9	3.9	–	–	–	–
22 (85)	80.1	11.5	7.5	1.0	71.7	17.7	8.3	2.1	–	–	–	–
Stalks												
0 (63)	36.2	17.9	36.3	4.7	22.3	18.5	56.6	2.7	43.0	18.3	31.6	7.1
1 (64)	41.3	25.2	30.3	3.3	26.0	17.0	55.6	1.3	–	–	–	–
3 (66)	35.3	24.6	36.4	3.3	34.2	18.9	43.7	3.3	–	–	–	–
7 (70)	42.5	20.7	32.4	4.1	31.4	19.2	44.0	5.4	66.7	12.2	17.2	3.5
15	48.1	20.6	26.	4.2	39.9	21.8	31.	6.9	–	–	–	–

DAT, days [a]	[^{3}H]avermectin B_{1a} (11.2 g ai/ha)				[^{3}H]avermectin B_{1a} (112 g ai/ha)				[^{14}C]avermectin B_{1a} (16.8 g ai/ha)			
	Polar metabolites	Mod. polar metabolites	B_{1a}	8,9-Z isomer	Polar metabolites	Mod. polar metabolites	B_{1a}	8,9-Z isomer	Polar metabolites	Mod. polar metabolites	B_{1a}	8,9-Z isomer
(78)			4				4					
22 (85)	51.5	15.4	28.3	4.8	48.3	14.1	29.6	6.1	–	–	–	–

[a] Numbers in parenthesis are days after 1st application (Four applications made to mature plants)

Table 21 shows the radioactivity released from the acetone non-extracted residues. In a preliminary experiment, residual solids following acetone extraction, which contain ^{3}H residues, were serially extracted with methanol/water (40:60), chloroform, dichloromethane, toluene and cyclohexane. Almost all (83%) of the radioactivity removed was associated with the methanol/water fraction, which was further treated with hot DMSO. Characterization of residues showed them to be mostly polar degradates of avermectin B_{1a} and < 1% TRR was released as parent compound. Further experiments with celery leaves using hot sulphuric acid indicated that 15% of the acetone non-extracted residues were incorporated into glucose. Residues in ^{3}H- and ^{14}C-leaves remaining after all treatments represented 10.6% and 4.1% of the TRR, respectively.

Table 21 Release of non-extracted residues from celery following application of [^{3}H] or [^{14}C]avermectin B_{1a}

Treatment/Product	Celery Leaves		Celery Stalks	
	Percent TRR	µg/kg eq.	Percent TRR	µg/kg eq.
[^{3}H]avermectin B_{1a} (112 g/ha 7 day DAT)				
Acetone	64.2	728	81.3	193
Remaining	35.8	485	18.7	53
Methanol/water	13.7	186	4.9	14
DMSO	6.9	94	4.0	11
Remaining	15.2	206	9.8	28
Sulphuric acid (glucose)	4.6	65		
Remaining	10.6	150		
[^{14}C]avermectin B_{1a} (16.8 g/ha 7 day DAT)				
Acetone	57.8	114	67.0	13.4
Remaining	42.2	83	33.0	7
Methanol/water	14.6	29	14.3	3
DMSO	9.0	18	9.9	2
Remaining	18.6	37	8.8	2
Sulphuric acid (glucose)	14.5	29		
Remaining	4.1	8		

Cotton

The metabolism of [^{14}C]avermectin B_{1a} was investigated in cotton in four experiments conducted in Texas and Florida (Wislock, 1986).

Experiment 1

Individual leaves were treated *in situ* by spreading 100 µg of [^{14}C]avermectin B_{1a} in an aqueous emulsion prepared from an EC formulation. Leaves were sampled in triplicate up to 8 days post-treatment, rinsed with alcohol and homogenized with acetone/water (9:1 v/v). Solids were separated by centrifugation and re-extracted twice with acetone.

Experiment 2

Small field plot of cotton plants was treated twice by foliar spray at 20 g ai/ha in a volume equivalent to 100 L/ha. Leaves were manually removed from plants when bolls reached maturity. Cotton bolls were de-linted with acid and the seeds extracted by Soxhlet with hexane for about 17 hours. The resultant solid fraction was extracted sequentially by reflux with methanol, acidic methanol, and basic

methanol. The hexane extract was evaporated, the resulting oil fractionated using a silica gel column, and the major radioactive fraction hydrolysed under alkaline conditions.

Experiments 3 and 4

In Florida, cotton plants were grown in buckets under normal field conditions and treated three times by foliar spray using an EC formulation at 22.4 g ai/ha (Experiment 3) or at 224 g ai/ha (Experiment 4), both using 467 L/ha. The bolls were harvested approximately 20 days after the last treatment (DAT), delinted, and leaves, stems, branches, roots and bract/calyx from each treatment were sampled. The cottonseeds were extracted as described before.

The incorporation of the radioactivity into the cotton leaves in Experiment 1 is summarized in Table 22. The total surface residues decreased by first order kinetics, with residues decreasing from 99.7% of the applied dose at Day 0 to 19.3% at Day 8. The parent compound degraded at a much faster rate, with an apparent half-life of approximately 12 hours, accounting for 1.7% of the applied dose after 8 days.

Table 22 Fate of [^{14}C]avermectin B_{1a}, in % AR,after foliar application to individual cotton leaves at 100 µg/leaf (Experiment 1)

	External rinse with methanol			Internal extract (acetone and water 9:1)				
		Avermectin B_{1a}			Avermectin B_{1a}			
DAT	Total	TLC	HPLC	Total	TLC	HPLC	Non-extracted	Lost
0	99.7	99.2	99.4	0.6	0.4	0.6	0.1	0.0
1/4	84.7	57.1	40.3	3.7	2.6	2.0	2.9	8.7
1	82.7	41.0	36.4	8.6	5.7	4.6	6.3	2.4
2	60.1	13.9	9.7	8.2	4.4	3.2	12.6	19.1
4	43.7	4.2	2.4	9.5	3.2	2.5	26.1	20.7
8	19.3	1.7	1.0	15.9	2.6	3.0	23.1	41.7

Table 23 shows the results of Experiments 2 to 4. In Experiment 2, the highest residues were in the leaves (396 µg/kg), and the lowest in the lint (37 µg/kg) and seeds (50 µg/kg). In Experiment 3, the highest residues were in the leaves (46 µg/kg) and the lint (44 µg/kg), and the lowest in the seeds (10 µg/kg) and roots (6 µg/kg). In Experiment 4, the last treatment was made when approximately 50% of the bolls were open, which may explain the high residues found in the lint (750 µg/kg).

Table 23 Combustion analysis of cotton plants treated with [^{14}C]avermectin B_{1a} under field conditions, TRR, in µg/kg

	Experiment 2	Experiment 3	Experiment 4
Sample	2× 22.4 g/ha, 8 DAT	3× 22.4 g/ha, 20 DAT	3× 224 g/ha, 20 DAT
Roots	25 ± 3	5.5 ± 0.4	107 ± 7.5
Stems	70 ± 5	12.5 ± 1.2	169 ± 5.0
Leaves	396 ± 27	46.4 ± 1.2	404 ± 1.0
Bract/Calyx	228 ± 15	11.9 ± 0.6	97 ± 9.0
Whole seeds	50 ± 3	10.0 ± 0.8	85 ± 6.3
Lint	37 ± 3	43.5 ± 1.2	750 ± 7.3

The metabolic profiles based on HPLC/radiochemical analyses for both the methanol rinse and the acetone/water extracts of the leaves from Experiment 1 are shown in Table 24. The amount of the 8,9-Z isomer of avermectin B_{1a} ranged from 0.1 to 7.0% AR in both the methanol rinse and the acetone/water extract.

Table 24 Extracted radioactivity (% AR) from leaves of cotton plants treated with [^{14}C]avermectin B_{1a} (Experiment 1)

	0 day	0.25 day	1 day	2 day	4 day	8 day
External rinse with methanol						
Polar	–	24.2	27.8	41.2	37.2	17.0
Moderate Polar	–	13.0	12.3	7.4	3.4	1.2
Avermectin B_{1a}	99.4	40.2	36.4	9.7	2.4	1.0
8,9-Z isomer	–	7.0	6.2	1.8	0.7	0.1
Internal extract (acetone/water 9:1)						
Polar	–	1.0	2.4	3.4	5.7	11.4
Moderate Polar	–	0.4	0.9	0.8	0.7	0.7
Avermectin B_{1a}	–	2.0	4.6	3.2	2.5	3.0
8,9-Z isomer	–	0.3	0.7	0.7	0.6	0.8

The radioactive residues extracted from cotton seed at harvest are shown in Table 25. A major fraction of the residues was extracted with hexane, mainly from cottonseed oil. When the oil was chromatographed on silica gel, the residues were found to co-elute with triglycerides. The hydrolysis of this fraction under basic conditions released linoleic acid and palmitic acid. Non-extracted material amounted to 25% of the TRR after sequential extraction with five solvents in Experiment 2.

Table 25 Extracted radioactivity (%TRR) from cottonseed treated with [^{14}C]avermectin B_{1a} in the field

	Experiment 2	Experiment 3	Experiment 3
Fractions	2 × 22.4 g ai/ha	3 × 22.4 g ai/ha	3 × 224 g ai/ha
Hexane	26	35	30
Ethanol	0	–	–
Methanol	13	32	24
Methanol/HCl	9	5	3
Methanol/NaOH	28	28	34
Non-extracted	25	0	19
Total Recovery	101	100	110

The metabolism of [^{14}C]avermectin B_{1a} in citrus fruit, cotton leaves and celery leaves (also [^{3}H]avermectin B_{1a}) was compared with thin film photolysis on glass plates (Crouch, 1988). Nearly mature oranges were treated with [^{14}C]avermectin B_{1a} by application of an aqueous suspension of an EC formulation with a small brush, and oranges harvested at 1 and 2 weeks post-application. Individual leaves of cotton plants were treated with [^{14}C]avermectin B_{1a} and leaves harvested after 2, 4 and 8 days. Orange and cotton leaves were rinsed with methanol. Mature celery plants were treated with [^{3}H]avermectin B_{1a} at 112 g/ha or [^{14}C]avermectin B_{1a} at 16.8 g/ha, harvested at 0 or 7 days after the last application and leaves and stalks homogenized with acetone. In the separate photolysis experiment, a methanol solution of [^{14}C]avermectin B_{1a} was applied to the bottoms of two glass petri dishes and allowed to dry at room temperature. The dishes were placed under two racks of 275 W Suntanner bulbs located 66 cm from the dishes. After 19 hours, the avermectin film was solubilized in methanol, an aliquot removed, and the remaining methanol allowed to dry. The dish was replaced under the lights. The process was repeated at 30, 60 and 137 hours. The temperature under the bulbs was approximately 50 °C.

Reverse-phase HPLC profile of [^{3}H] or [^{14}C]avermectin B_{1a} and its degradates from citrus, cotton, celery and photolysis extracts showed the same profile (Table 26). Re-chromatography of the moderately polar fraction indicated the presence of 2–6 components, one co-chromatographed with 8α-hydroxy avermectin B_{1a}. Re-chromatography of the polar residues from the three treated crops and in the photolysis experiment showed four broad peaks. Spectrometric methods have indicated the presence of numerous multiple-oxygenated, hydrated or dehydrated and de-methylated species, which retain little of the macrocyclic characteristics of the avermectins.

Table 26 Profile of total solvent-extracted residues following application of avermectin B_{1a} to cotton leaves, citrus fruit, celery leaves and stalks and to glass plates using C_{18} HPLC

Sample	Time	% TRR in the fraction			% of Applied Dose
		Polar Fraction	Moderately Polar Fraction	Avermectin B_{1a} Fraction	
Cotton [a]					
Leaf surface wash	2 days	68.6	12.3	16.1	60.1
Leaf surface wash	4 days	85.1	7.8	5.5	43.7
Leaf surface wash	8 days	88.1	6.2	5.2	19.3
Leaf extract	8 days	71.7	4.4	18.9	15.9
Citrus Fruit					
Fruit surface (1×) wash	7 days	88.5	3.9	3.3	15.2
Fruit surface (30×) wash	7 days	74.2	7.2	11.1	17.9
Fruit surface (30×) wash	14 days	82.3	6.0	6.8	12.4
Celery [b]					
Stalk Extract (^{3}H, 5×)	0 days	22.3	18.5	56.6	1.03
Stalk Extract (^{14}C, 0.75×)	0 days	43.0	18.3	31.6	0.55
Stalk Extract (^{14}C, 0.75×)	7 days	66.7	12.2	17.2	0.30
Leaf Extract (^{3}H, 5×)	0 days	42.2	19.8	33.0	2.56
Leaf Extract (^{14}C, 0.75×)	0 days	33.8	22.5	38.6	3.66
Leaf Extract (^{3}H, 5×)	7 days	63.7	18.8	14.8	1.38
Leaf Extract (^{14}C, 0.75×)	7 days	71.6	16.2	9.8	1.50
In Vitro					
petri dish	19 hours	33.3	14.2	36.7	
petri dish	30 hours	81.0	9.5	7.3	
petri dish	60 hours			0.0	
petri dish	137 hours			0.0	

[a] Data from Wislocki *et al.,* 1986

[b] Data from Wislocki *et al.,* 1988

Tomato

Metabolism of avermectin B_{1a} was studied in greenhouse-grown tomato plants transplanted at growth stage BBCH 19 and placed in the greenhouse (Stingelin, 2003). Five spray applications (7 days interval) were made with formulated [23-^{14}C] avermectin B_{1a} at an average rate of 26.4 g/ha (2.2 g/hL) for the normal rate (Sub-Study 1) and three times (14 days interval) at an average rate of 280.8 g/ha (23.4 g/hL) for the exaggerated rate experiment (Sub-Study 2). The first treatment took place at growth stage BBCH 63 and the last at BBCH 71. For the Sub-Study 1, tomato fruits and leaves were collected one hour after the third and fifth application, and 3 to 28 days after the last treatment (final harvest). Sampling for the Sub-Study 2was performed one hour to 28 days after the last application. A cell tomato cells (variety Money Marker) grown as a cell suspension (Sub-Study 3) on medium AM1 under illumination at 27 °C were used for this study. Following sub-culturing, the cells were allowed to reach the log phase of growth prior to the addition of radio-labelled material, dissolved in dimethyl sulfoxide. The cell cultures were incubated for 41 days, separated from the medium by filtration under low vacuum, and washed three times with distilled water. This Sub-Study provided metabolites for identification purposes.

Tomato samples were washed with acetonitrile/water(50/50), washed tomatoes and leaves were homogenized in liquid nitrogen, extracted for at least six hours with acetonitrile/water (80/20 v/v), and the extraction procedure repeated five times or until the radioactivity in the last extract was less than 5% of the first extraction. The solid residues were extracted by microwave with 1-propanol/water (80/20) (10 min. at 100 °C, 20 min. at 120 °C, and 20 min. at 150 °C). Samples of the residual solid and after microwave extraction were air-dried, homogenized and taken for combustion to determine the non-extracted radioactivity.

Before partitioning the soluble radioactivity, samples were concentrated, the aqueous phase partitioned three times with n-hexane, dichloromethane or ethyl acetate. For storage

stability purposes the surface radioactivity washes and the crude extract from the tomato fruit were re-analysed by 2-D TLC after storage at ≤ 8 °C. Additionally, tomato fruit free of surface radioactivity were re-extracted at the end of the experimental phase and the corresponding crude extract was re-analysed by 2-D TLC. Harvested cells (Sub-Study 3) were homogenized in acetonitrile:water (80/20), the homogenate centrifuged, re-extracted and analysed by TLC, reversed-phase HPLC and LC-MS.

Table 30 shows the distribution of radioactivity from the sub-studies. The non-extracted radioactivity (NE) in tomato fruit did not exceed 2% of TRR.

Table 30 Distribution of radioactivity and residual [^{14}C]avermectin B_{1a} in treated tomato samples

Sampling time	Crop Part	TRR [mg/kg] [a]	Avermectin B_{1a} [mg/kg] [a]	Surface Rad.[%] [b]	Extraction		NE [%] [b]	Total [%] [b]
					cold [%] [b]	MW [%] [b]		
Sub-Study 1 (5 × 26 g ai/ha)								
1 h after 3rd application	Tomato	0.314	0.282	95.3	5.4	0.1	0.2	101.0
	Leaves	3.869	3.706	–	112.8	n.a.	3.5	116.3
1 h after 5th application	Tomato	0.205	0.141	84.5	12.6	1.0	0.9	98.9
	Leaves	3.504	2.635	–	105.9	n.a.	4.7	110.5
3 d after 5th application	Tomato	0.098	0.062 [c]	69.1	30.3	2.1	1.8	103.3
	Leaves	4.418	3.205	–	115.5	n.a.	9.0	124.5
7 d after 5th application	Tomato	0.195	0.129 [c]	81.0	16.3	0.8	1.3	99.4
	Leaves	6.590	2.701	–	85.4	3.0	3.8	92.2
14 d after 5th application	Tomato	0.156	0.089 [c]	78.3	17.3	1.1	0.9	97.6
	Leaves	5.908	2.265	–	82.5	5.4	2.9	90.8
28 d after 5th application	Tomato	0.127	0.060 [c]	76.6	17.9	1.9	1.3	97.8
	Leaves	6.421	2.158	–	95.9	8.6	3.6	108.0
Sub-Study 2 (3 × 281 g ai/ha)								
1 h after 3rd application	Tomato	1.555	1.293	90.8	8.6	0.2	0.4	100.0
	Leaves	30.96	26.134	–	96.8	n.a.	3.2	100.0
3 d after 3rd application	Tomato	1.667	1.303	85.2	14.0	0.5	0.3	100.0
	Leaves	38.66	26.952	–	96.0	n.a.	4.0	100.0
7 d after 3rd application	Tomato	1.715	1.376	93.7	5.9	0.1	0.3	100.0
	Leaves	23.84	16.011	–	94.7	n.a.	5.3	100.0
14 d after 3rd application	Tomato	0.880	0.674	82.4	15.4	0.8	0.9	100.0
	Leaves	33.98	20.724	–	93.0	n.a.	7.0	100.0
28 d after 3rd application	Tomato	0.572	0.416	85.8	13.1	< 0.1	1.1	100.0
	Leaves	74.23	37.512	–	93.1	4.2	2.8	100.0

n.a. = Not analysed

MW = Microwave extraction

NE = Non-extracted [a] in avermectin B_{1a} equivalents; [b] in %TRR determined by the sum of surface + extracted + non-extracted radioactivity; [c] corrected for 8,9-Z isomer of avermectin B_{1a} content

Tables 28 and 29 show the metabolite fractions from the two sub-studies. Avermectin B_{1a} and its 8,9-Z isomer was the major fraction in all samples, accounting for at least 38.3%TRR (14 days leaves Sub-Study 1), in a ratio of approximately 9:1. Other identified metabolites are 8α-oxo-avermectin B_{1a}, 8α-hydroxy-avermectin B_{1a}, and 3''-O-desmethyl-avermectin B_{1a}, present at levels < 8%TRR in tomato and leaves at any sampling time in both experiments.

Table 28 Quantification of metabolite fractions in tomato fruit and leaves at various sampling times after the 5th application (in % of TRR), Sub-Study 1

Sampling (after last application)	0 days		3 days		7 days		14 days		28 days	
Plant Part	Fruits	Leaves	Fruits	Leaves	Fruits	Leaves	Fruits	Leaves	Fruits	Leaves
TRR [mg/kg] [a]	0.205	3.5	0.098	4.4	0.195	6.6	0.156	5.9	0.127	6.4
Metabolite Fraction	%TRR [b]	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR
Avermectin B_{1a}+ 8,9-Z isomer	68.7	75.2	70.2	72.5	72.0	41.0	63.9	38.3	51.4	33.6
8α-oxo-avermectin B_{1a}	3.1	6.3	3.4	7.3	5.2	6.0	4.3	4.7	5.5	4.9

Sampling (after last application)	0 days		3 days		7 days		14 days		28 days	
Plant Part	Fruits	Leaves	Fruits	Leaves	Fruits	Leaves	Fruits	Leaves	Fruits	Leaves
TRR [mg/kg] [a]	0.205	3.5	0.098	4.4	0.195	6.6	0.156	5.9	0.127	6.4
Metabolite Fraction	%TRR [b]	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR
8α-hydroxy- avermectin B_{1a}	2.9	1.7	2.9	2.8	1.9	2.1	2.2	2.1	2.0	2.8
3”-O-Desmethyl-avermectin B_{1a}	0.7	0.7	0.5	1.0	0.4	1.3	0.6	1.1	0.7	1.2
I_1	5.6	4.8	4.2	7.8	2.9	8.7	5.9	11.4	8.4	20.5
I_2									0.9	1.7
I_3										0.7
I_4 [c]	0.8	0.5	1.4	0.7	0.5	1.0	1.0	1.0	1.0	2.1
$I_{5–8}$	2.2	4.8	3.2	6.7	3.7	7.1	4.0	7.4	6.8	14.8
I_{14}			0.4		0.3		0.7		0.9	1.4
I_{15}									0.3	
I_{18}										1.1
I_{27}	0.5	0.8	0.4	1.8	0.4	0.7	0.5	0.4	0.4	0.8
I_{29}	1.0	1.2	1.2	1.1	0.8	0.7	1.1	1.0	1.5	
I_{31}	0.2	1.9	1.5	2.6	1.0	3.7	1.1	3.5	1.0	1.2
I_{34}	0.4	0.7	0.6	0.9	0.5	0.9	0.6	0.8	0.7	0.4
$I_{35–37}$	1.2	0.8	1.0	1.3	1.2	1.0	1.2	0.8	2.2	1.0
Unresolved Rad.	9.7	6.5	8.4	8.9	6.5	11.2	8.4	10.1	11.0	7.3
Sub. Total	97.1	5.9	99.4	115.5	97.3	85.4	95.6	82.5	94.5	95.9
Micro Wave Extract	1.0	–	2.1	–	0.8	3.0	1.1	5.4	1.9	8.6
Non-Extr. Rad.	0.9	4.7	1.8	9.0	1.3	3.8	0.9	2.9	1.3	3.6
Total	99.0	110.6	103.3	124.5	99.4	92.2	97.6	90.8	97.7	108.1
Accountability [d]	76.2	84.4	78.4	84.3	80.0	51.4	72.0	47.2	60.6	44.6

[a] In avermectin B_{1a} equivalents

[b] In % of the total radioactivity found in the plant part, surface + penetrated radioactivity (determined by combustion)

[c] I_4 was identified as ((2S,4S,6S,8R,9S)-8-sec-Butyl-4-hydroxy-9-methyl-1,7-dioxa-spiro[5.5]undec-10-en-2-yl)-acetic acid

[d] Sum of I_4 and all identified metabolites

Table 29 Quantification of metabolite fractions in tomato fruit and leaves at various sampling times after the 3rd application (in % of TRR), Sub-Study 2 (exaggerated application rate)

Sampling (after last application)	0 days		3 days		7 days		14 days		28 days	
Plant Part	Fruits	Leaves	Fruits	Leaves	Fruits	Leaves	Fruits	Leaves	Fruits	Leaves
TRR [mg/kg] [a]	1.55	30.9	1.66	38.6	1.71	23.8	0.88	33.9	0.57	74.2
Metabolite Fraction	%TRR [b]	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR
Avermectin B_{1a}+ 8,9-Z isomer	83.2	84.4	78.1	69.7	80.5	67.2	78.6	61.0	75.2	50.5
8α-oxo-avermectin B_{1a}	2.2	1.8	3.0	2.5	4.0	3.0	4.3	3.1	3.8	3.6
8α-hydroxy- avermectin B_{1a}	1.1	1.3	1.7	1.5	1.9	1.9	1.4	2.6	1.5	3.6
3”-O-Desmethyl-avermectin B_{1a}	0.7	0.5	0.4	0.6	0.4	1.4	0.4	1.3	0.5	1.1
I_1	1.1	1.6	1.0	3.6	0.4	4.7	0.9	5.5	4.1	8.6
I_4 [c]	0.2	0.1	0.6	0.2	0.4	0.5	0.4	0.5	0.7	0.8
$I_{5–12}$	4.7	1.7	5.8	4.3	6.0	5.4	6.1	5.9	3.0	10.3
I_{14}	0.2		1.6				0.5		0.6	
I_{15}	0.9		0.5		0.2		0.9			0.6
I_{18}		0.4	0.3	1.1	0.2		0.6	1.1		0.9
I_{27}		0.5	0.5	0.4	0.3	1.0	0.3	0.8	< 0.1	0.8
I_{29}	1.4		1.2	0.6	1.2	0.4	0.8	0.6	0.7	
I_{31}	0.9	0.8	1.4	2.4	0.9	1.2	0.9	1.2	0.9	1.6
I_{34}	0.4	0.3	0.5	0.6	0.7	0.6	0.8	0.7	0.5	0.8
$I_{35–37}$	0.4	0.6	0.5	1.0	0.6	0.8	0.4	0.9	1.3	0.9

Sampling (after last application)	0 days		3 days		7 days		14 days		28 days	
Plant Part	Fruits	Leaves	Fruits	Leaves	Fruits	Leaves	Fruits	Leaves	Fruits	Leaves
TRR [mg/kg] [a]	1.55	30.9	1.66	38.6	1.71	23.8	0.88	33.9	0.57	74.2
Metabolite Fraction	%TRR [b]	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR
Unresolved Rad.	2.0	2.2	2.1	8.0	2.3	6.7	2.7	7.8	7.1	8.6
Sub. Total	99.4	96.8	99.2	96.0	100	94.7	99.9	93.0	99.9	93.1
Micro Wave Extract	0.2	–	0.5	–	–	–	–	–	–	4.2
Non-Extr. Rad.	0.4	3.2	0.3	4.0	-	5.3	0.1	7.0	0.1	2.8
Total	100	100	100	100	100	100	100	100	100	100
Accountability [d]	87.4	88.1	83.8	74.5	87.2	74.0	85.1	68.5	81.7	59.6

[a] In avermectin B_{1a} equivalents

[b] In% of the total radioactivity found in the plant part, surface + penetrated radioactivity (determined by combustion)

[c] I_4 was identified as ((2S,4S,6S,8R,9S)-8-sec-Butyl-4-hydroxy-9-methyl-1,7-dioxa-spiro[5.5]undec-10-en-2-yl)-acetic acid

[d] Sum of I_4 and all identified metabolites

Metabolism of avermectin B_{1a} was studied in field-grown tomato plants under similar conditions as the greenhouse study (Stingelin, 2003a).Five spray applications were made using formulated [23-^{14}C] avermectin B_{1a} at an average rate of 26.4 g/ha (Sub-Study 1) and five times at an average application rate of 245.9 g/ha (Sub-Study 2). The tomato plants were kept unprotected and exposed to all weather conditions over the whole of the growing period. Sample analysis was similar to the greenhouse study.

Table 30 shows the distribution of radioactivity from the sub-studies. Total residues in tomato and leaves from Sub-Study 1 (normal rate) reached 0.017 and 0.716 mg/kg eq at the end of the experiment, respectively. The non-extracted radioactivity in tomato fruit did not exceed 10% of TRR.

Table 30 Distribution of radioactivity and residual [^{14}C]avermectin B_{1a} from the field study (Stingelin, 2003a)

Sampling time	Crop Part	TRR [mg/kg] [a]	Avermectin B_{1a} [mg/kg] [a]	Surface Rad. [%] [b]	Extraction		NE [%] [b]	Total [%] [b]
					cold [%] [b]	MW [%] [b]		
Sub-Study 1 (5 × 26.4 g/ha)								
1 h after 1st application	Tomato	0.019	0.015	88.3	n.a.	n.a.	11.7	100.0
	Leaves	0.982	0.937	n.a.	99.5	n.a.	0.9	100.4
1 h after 3rd application	Tomato	0.027	0.016	59.8	36.6	3.0	2.0	101.4
	Leaves	2.343	1.160	n.a.	79.0	4.5	7.8	91.4
1 h after 5th application	Tomato	0.026	0.016	64.1	30.3	4.5	2.2	101.1
	Leaves	1.424	0.683	n.a.	76.3	11.3	3.9	91.5
3 d after 5th application	Tomato	0.034	0.005	62.6	27.7	4.7	2.8	97.8
	Leaves	1.649	0.239	n.a.	73.1	11.2	8.3	92.6
7 d after 5th application	Tomato	0.020	0.005	30.8	51.5	6.8	6.3	95.4
	Leaves	0.840	0.044	n.a.	67.1	15.8	8.8	91.6
14 d after 5th application	Tomato	0.022	0.005	19.8	60.8	10.2	6.9	97.6
	Leaves	1.161	0.027	n.a.	65.4	17.5	9.6	92.5
28 d after 5th application	Tomato	0.017	0.001	19.3	62.7	9.6	8.0	99.6
	Leaves	0.716	0.015	n.a.	67.9	18.2	9.5	95.6
Sub-Study 2 (5 × 246 g/ha)								
7 d after 3rd application	Tomato	0.131	0.055	46.6	44.3	4.8	4.3	100.0
	Leaves	6.862	1.162	n.a.	78.0	14.2	6.2	98.4
28 d after 3rd application	Tomato	0.108	0.015	22.0	60.8	11.1	6.1	100.0
	Leaves	7.768	0.499	n.a.	70.6	13.8	6.1	90.5

n.a. = Not analysed

MW = Microwave extraction

NE = Non-extracted

[a] In avermectin B_{1a} equivalents

[b] In TRR found in the plant

Tables 31 and 32 show the metabolite fractions from the two sub-studies. The major metabolite fraction in all of the analysed samples was fraction avermectin B_{1a} and the 8,9-Z isomer of avermectin B_{1a} in a ratio of approximately 9:1, accounting for about 70–80%TRR at 0 days and decreasing over time. Other identified metabolites are 8α-oxo-avermectin B_{1a}, 8α-hydroxy-avermectin B_{1a}, and 3''-O-desmethyl-avermectin B_{1a}, present at levels < 7% TRR in tomato and leaves at any sampling time in both experiments.

Table 31 Quantification of metabolite fractions in/on tomato fruit at various sampling times (in % of TRR**),** from the field study (Stingelin, 2003a)

Sampling after appl.	0 days after 1st	0 days after 3rd	0 days after 5th	3 days after 5th	7 days after 5th		14 days after 5th	28 days after 5th	
Sub-Study No.	1	1	1	1	1	2	1	1	2
TRR [mg/kg] [a]	0.019	0.027	0.026	0.034	0.020	0.131	0.022	0.017	0.108
Metabolite Fraction	%TRR [b]	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR
Avermectin B_{1a} and 8,9-Z isomer	80.8	60.8	62.3	14.3	25.3	38.1	23.5	7.1	25.4
8α-oxo-avermectin B_{1a}	2.1	1.3	1.8	3.7	2.5	2.9	0.4	2.7	0.8
8α-hydroxy- avermectin B_{1a}	0.4	0.6	0.4	2.3	0.6	2.0	0.5	1.6	2.2
I_1 [c]	1.2	14.9	11.9	22.6	36.7	11.8	29.2	19.0	28.8
I_2		3.6		7.1	4.2	1.3	3.3	9.0	2.5
I_4 [d]		3.7	2.6	5.9	1.7	1.4	1.9	4.4	0.9
I_5		1.6	0.6	8.5	3.4	6.9	1.6	15.1	3.3
I_{12}	0.3								
I_{14}		0.4		1.8	0.5	1.1	0.5		0.7
I_{15}						1.3			
I_{21}						1.0			1.4
I_{29}	0.3	0.4		0.8	0.4	1.8	0.2	1.4	1.8
I_{30}		0.3		1.3	0.3	0.7	0.5	0.2	0.3
I_{31}	0.9	0.7	0.6	2.2	0.4	1.4		0.1	0.4
I_{34}				1.2		2.5	0.3		3.7
unresolved Rad.	2.4	8.0	14.2	18.5	6.4	16.9	18.8	21.4	10.6
Sub. Total	88.3	96.4	94.4	90.3	82.3	90.9	80.6	82.0	82.8
MW-Extract	–	3.0	4.5	4.7	6.8	4.8	10.2	9.6	11.1
Non-Extr. Rad.	11.7	2.0	2.2	2.8	6.3	4.3	6.9	8.0	6.1
Total	100.0	101.4	101.1	97.8	95.4	100.0	97.6	99.6	100.0

[a] In avermectin B_{1a} equivalents

[b] In% TRR found in the plant part, surface + penetrated radioactivity (determined by combustion)

[c] For the surface radioactivity of tomato fruits it was demonstrated that the origin spot I_1 could be separated into two to three distinct peaks and unresolved radioactivity

[d] I_4 was identified as ((2S,4S,6S,8R,9S)-8-sec-Butyl-4-hydroxy-9-methyl-1,7-dioxa-spiro[5.5]undec-10-en-2-yl)-acetic acid

Table 32 Quantification of metabolite fractions in tomato leaves at various sampling times (%TRR)

Sampling after appl.	0 days after 1st	0 days after 3rd	0 days after 5th	3 days after 5th	7 days after 5th		14 days after 5th	28 days after 5th	
Sub-Study No.	1	1	1	1	1	2	1	1	2
TRR [mg/kg] [a]	0.982	2.34	1.42	1.65	0.84	6.86	1.16	0.71	7.76
Metabolite Fraction	%TRR [b]	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR	%TRR
Avermectin B_{1a} and 8,9-Z isomer	95.4	49.5	48.0	14.5	5.3	16.9	2.3	2.2	6.4
8α-oxo-avermectin B_{1a}	0.5	1.5	0.8	2.7	1.0	1.8	0.7	0.7	1.1
I_{34}					1.1	1.4	1.1		1.2
8α-hydroxy- avermectin B_{1a}	0.3	0.9	0.6	0.9	0.9	1.6	0.7	0.5	1.1
I_1 [c]	0.3	2.2	2.3	5.0	20.7	20.9	25.1	29.4	29.8
I_2		3.9	3.7	8.2	12.7	9.5	13.3	11.7	8.1

Sampling after appl.	0 days after 1st	0 days after 3rd	0 days after 5th	3 days after 5th	7 days after 5th		14 days after 5th	28 days after 5th	
Sub-Study No.	1	1	1	1	1	2	1	1	2
I_4 [d]		3.8	3.3	4.8	3.4	2.4		3.3	2.4
I_5		11.3	10.3	24.5	8.9	9.3	7.8	7.7	9.8
I_{14}						1.4	3.7		0.6
I_{16}			0.8	2.6				3.3	
I_{18}						1.8			3.6
I_{21}			0.3		0.9	1.3	0.7	0.6	1.3
I_{27}						0.2			
I_{29}	0.6	0.9	1.0	1.2	1.2	1.2	0.7	0.6	0.8
I_{30}		0.4	0.5	0.5	0.7	0.8	0.6	0.5	0.5
I_{31}	0.2	0.9	0.5	2.5	0.7	1.0	0.4		0.6
I_{35}		0.5							
unresolved Rad.	2.2	3.8	4.2	5.7	9.6	6.4	8.4	7.4	3.3
Sub. Total	99.5	79.0	76.2	73.1	67.1	78.0	65.4	67.9	70.6
MW-Extract	-	4.5	11.3	11.2	15.8	14.2	17.5	18.2	13.8
Non-Extr. Rad.	0.9	7.8	3.9	8.3	8.8	6.2	9.6	9.5	6.1
Total	100.4	91.4	91.5	92.6	91.6	98.4	92.5	95.6	90.5

[a] In avermectin B_{1a} equivalents

[b] In% of the total radioactivity found in the plant part, surface + penetrated radioactivity (determined by combustion)

[c] For the surface radioactivity of tomato fruits it was demonstrated that the origin spot I_1 could be separated into two to three distinct peaks and unresolved radioactivity

[d] I_4 was identified as ((2S,4S,6S,8R,9S)-8-sec-Butyl-4-hydroxy-9-methyl-1,7-dioxa-spiro[5.5]undec-10-en-2-yl)-acetic acid

The proposed metabolic pathway for avermectin B_{1a} in plants is shown in Figure 2.

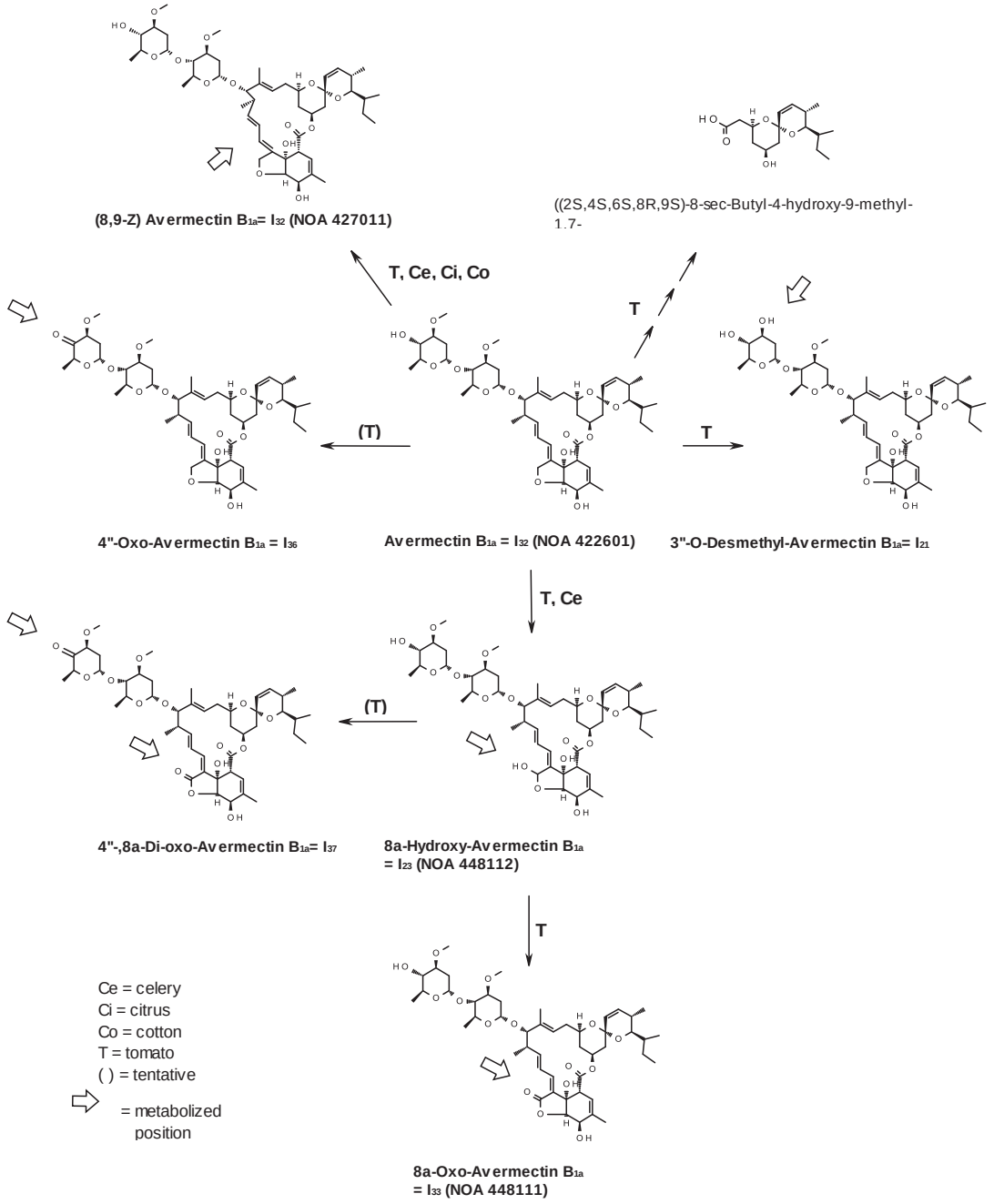


Figure 2 Proposed metabolic pathway of avermectin B_{1a} in plants

Confined rotational crop studies

The uptake, distribution and degradation of [^{14}C]avermectin B_{1a} were investigated in succeeding crops (Moye *et al.*, 1987). Sorghum, lettuce and carrot or turnip were planted in three soil types; a sandy soil, a sandy loam soil and a "muck" soil (high-organic drained swampland), typical US soils for cotton-growing in Georgia, vegetable-growing in California and vegetable-growing in Florida, respectively. The soils were filled into large tubes (three per soil type) and treated at 135 to 155% of the maximum label rate of 21.3 g ai/ha (for non-permanent crops). The sandy soil received three applications at 29.1 g ai/ha and sandy loam and muck soils received 12 applications at 33.6 g ai/ha. After the last application, each tube was divided into thirds and one rotational crop was planted in each third. Three plant-back intervals were used for each soil type. Sorghum and lettuce were planted in all soil types, turnip was planted in the muck soil and carrot planted in the sand and sandy loam soils. The plant-back intervals were 14, 123 and 365 days for the muck soil, 31, 120 and 365 days for the sandy soil and 29, 123 and 365 days for the sandy loam soil. All crops were seeded directly onto the plots. All rotational crops were harvested at 25, 50 and 100% (full) maturity. Soil cores (top 3 inches, middle 3 inches and bottom 3–6 inch layer) were also collected. Samples were combusted to measure radioactivity and lettuce (25% maturity) from a muck soil treatment was extracted with acetone.

The total radioactive residues in rotational crops following the treatment regimes are show in Table 33. The highest TRR was found in the 1/4 maturity lettuce sample from the muck soil (6.94 μg/kg), from which extraction with acetone released only 4.38% of the TRR. The resulting concentrations of radioactivity in succeeding crops were too low to characterize. Total radioactive residues in soil were also low (consistent with the low use rate). Residue levels in soil were proportional to the amount applied and decreased with the depth of sampling and the length of time between application and sampling (data not shown).

Table 33 Uptake and distribution of metabolites in rotational crops (3 plant-back intervals) after bare ground application of [^{14}C]avermectin B_{1a}

	Residue (μg/kg) in avermectin B_{1a} equivalents, mean of two groups														
	Sorghum						Lettuce			Carrots				Turnips	
	Leaf-Stem			Grain			Heads			Tops		Tubers		Tops	Tubers
	Muck	Sand	Sandy loam	Muck	Sand	Sandy loam	Muck	Sand	Sandy loam	Sand	Sandy loam	Sand	Sandy loam	Muck	
Plant-Back Interval (PBI)															
DAT	14	31	29	14	31	29	14	31	29	31	29	31	29	14	14
¼ Mature	4.78 [0.90]	< 0.85	2.54 [2.08]	–	–	–	6.94	0.92	2.40	1.08	2.21	1.49	0.87	0.83	3.45
½ Mature	1.74 [< 0.83]	< 6.03	11.6 [1.82]	–	–	–	2.52	0.77	0.45	0.37	0.62	0.58 [c]	0.42	0.37	0.80
Mature	7.4 [1.70 [a]]	< 2.23	Frost [1.74]	Frost [< 4.71]	< 4.13	Frost [< 3.95]	0.44	0.18	0.67	< 0.66	1.66	< 0.37	0.95	< 0.96	0.14
Plant-Back Interval (PBI)															
DAT	123	120	123	123	120	123	123	120	123	120	123	120	123	123	123
¼ Mature	2.73	3.54 [c]	2.19	–	–	–	0.24	0.48	1.49	0.47 [c]	1.29	1.05 [c]	1.86	< 0.66	1.12
½ Mature	6.56 [c]	< 0.62	1.60 [c]	–	–	–	0.27	0.33	0.50	< 0.68	0.99	< 1.05	1.01	< 1.05	0.18 [c]
Mature	0.60 [c]	< 0.84	1.19	< 5.69	< 0.99	< 1.39	0.15	< 0.15	0.16	< 1.07	2.62	0.91 [c]	1.93	< 0.61	< 0.71
Plant-Back Interval (PBI)															
DAT	365	365	365	365	365	365	365	365	365	365	365	365	365	365	365
¼ Mature	< 0.59	< 0.69	0.90 [c]	–	–	–	0.76	< 0.43	0.47	< 1.00	1.38	< 0.60	1.14	< 0.43	< 0.44
½ Mature	< 1.19	< 1.86	< 1.16	–	–	–	0.72	< 0.35	0.50 [c]	< 1.18	1.53	< 0.80	1.90	< 0.69	< 0.45
Mature	< 2.52	< 2.68	1.85 [c]	< 3.88	< 3.60	< 4.13	1.39	< 0.52	0.67	< 1.02	< 1.07	< 1.01	0.83 [c]	< 0.55	0.37

Values with < reflect the average of the limits of quantification calculated for each of the samples in each group

Values with [] are from repeats caused by frost damage

[a] Value for one group only. Second group had a value below the LOQ

Animal metabolism

Metabolism in rats

The metabolism of abamectin in rats was evaluated the WHO group of the JMPR at the present Meeting. In summary, orally administered [^{3}H] and [^{14}C] abamectin B_{1a} was rapidly and almost completely absorbed, and maximum concentrations in blood were achieved within 4–8 hours after administration. Radio-label was distributed to all major tissues and organs. Elimination of radio-label occurred predominantly by non-biliary excretion into the gastrointestinal tract and excretion with the faeces, while urinary excretion accounted for only 0.5 to 1.4 of the dose. Elimination was moderately fast, with 80 to 101% of the dose excreted within 96 hours. Rate of oral absorption, tissue distribution and excretion were independent of the dose level, treatment regime and/or sex; however, the depletion of tissue residues in males was approximately 2-fold more rapid than in females. There was no evidence for tissue accumulation on repeated administration. Metabolism of avermectin B_{1a} in the rat was moderate to extensive and proceeded predominantly via demethylation, hydroxylation, cleavage of the oleandrosyl ring, and oxidation reactions. The metabolite pattern in urine, faeces and bile was complex but qualitatively independent of the sex and the dose level with some quantitative variations. Eleven metabolites were isolated. Unchanged avermectin B_{1a} and the metabolites 3"-O-desmethyl abamectin B_{1a}, 24-hydroxymethyl abamectin B_{1a}, 27-hydroxymethyl abamectin B_{1a}, 3"-O-desmethyl-24-hydroxymethyl abamectin B_{1a} and 3"-O-desmethyl-27-hydroxymethyl abamectin B_{1a} represented the majority of the faecal radioactivity.

Metabolism in lactating goats

One study was conducted in lactating goats using [^{3}H]avermectin B_{1a} (Merricks, 1983, 1983a, 1983b; Maynard *et al.,* 1986; 1989). Six lactating Nubian goats were dosed daily by gelatine capsule for ten consecutive days with [^{3}H]avermectin B_{1a} at 0.005, 0.05 and 1.0 mg/day (two animals at each dose level), corresponding to 0.00125, 0.0125 and 0.25 ppm, respectively, in the diet. Urine and faeces were collected daily and each goat was milked twice daily. The animals were sacrificed on Day 11 approximately 24 hours after the last dose, and tissue samples collected.

Radioactivity in milk samples were counted directly, and tissue, urine and faeces samples were combusted prior to liquid scintillation counting (LSC). Edible tissues and milk were homogenized, extracted with dichloromethane, and the extract cleaned-up in a silica gel SPE for reverse-phase HPLC analysis. Avermectin B_{1a} residues were determined by reverse isotope dilution assay (RIDA). Profiling of the ethyl acetate eluate from the SPE column produced metabolite regions that were defined by retention times relative to avermectin B_{1a}. A column wash was used to investigate the non-polar fraction; a high dose fat sample was subjected to acid hydrolysis. Avermectin B_{1a} and a metabolite standard were also subjected to the acid hydrolysis conditions to determine reaction products. Since the radioactivity in goat tissue was low, a rat liver microsomal incubation of [^{14}C]avermectin B_{1a} was conducted to generate metabolite standards that could be co-chromatographed with in-vivo goat metabolites. Following incubation, the metabolites were purified by various reversed-phase HPLC and the structures identified by NMR and Fast Atom Bombardment (FAB)-Mass Spectrometry.

The majority (79 to 98%) of the administered dose was found in the faeces, with urine accounting for 0.1 to 0.6% of the daily dose in the highest dosed animals. Milk residues reached plateau (steady state) by Day 4 and were dose dependent (Table 34).

Table 34 Residue levels in milk from goats dosed with [^{3}H]avermectin B_{1a} (Maynard *et al.,* 1989)

Dose	Residue (µg/kg avermectin B_{1a} equivalents)											
Day	0.00125 ppm				0.0125 ppm				0.25 ppm			
	Goat 1		Goat 2		Goat 3		Goat 4		Goat 5 [a]		Goat 6	
	AM	PM	AM	PM	AM	PM	AM	PM	AM	PM	AM	PM
1	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	0.08	< 0.02	0.1	< 0.02	0.45	< 0.02	0.84
2	< 0.02	< 0.02	< 0.02	0.02	0.17	0.26	0.13	0.36	1.11	1.80	0.70	1.33
3	< 0.02	< 0.02	< 0.02	0.02	0.23	0.33	0.29	0.45	2.03	3.00	1.10	1.87
4	< 0.02	< 0.02	< 0.02	0.02	0.34	0.35	0.28	0.40	3.40	4.26	1.31	1.64

Dose Day	Residue (µg/kg avermectin B_{1a} equivalents)											
	0.00125 ppm				0.0125 ppm				0.25 ppm			
	Goat 1		Goat 2		Goat 3		Goat 4		Goat 5 [a]		Goat 6	
	AM	PM	AM	PM	AM	PM	AM	PM	AM	PM	AM	PM
5	< 0.02	< 0.02	< 0.02	0.03	0.26	0.30	0.31	0.38	3.40	4.48	1.38	1.87
6	0.02	0.02	< 0.02	0.03	0.23	0.36	0.32	0.48	3.29	4.48	1.18	2.16
7	< 0.02	0.02	< 0.02	0.03	0.23	0.38	0.28	0.47	3.11	4.71	1.31	2.33
8	< 0.02	0.02	0.02	0.03	0.20	0.29	0.31	0.44	3.19	4.25	1.31	2.06
9	< 0.02	< 0.02	< 0.02	0.03	0.21	0.29	0.29	0.41	3.60	3.71	1.30	1.93
10	< 0.02	< 0.02	< 0.02	< 0.02	0.22	0.34	0.34	0.41	3.05	4.70	1.36	2.26
11	< 0.02	S	0.02	S	0.25	S	0.29	S	5.05	S	1.62	S

[a] Animal off feed days 9–11, low water consumption. All other clinical observations were normal

S = Sacrifice after AM milking

The results of the tissue and organ assays for total radioactive residue (TRR) are shown in Table 35. Highest residues were found in liver, fat and kidney. Residues were not detected in muscle from the lower dose group (< 0.2 µg/kg eq.) and reached approximately 1.5 µg/kg eq. at the highest dose. Goat 5 at the highest dose level, had atypical consumption behaviour (off feed days 9–11, low water consumption).

Table 35 Residue levels in tissues from goats dosed with [^{3}H]avermectin B_{1a} for ten consecutive days(Maynard *et al.*, 1989)

Matrix	Residue (µg/kg avermectin B_{1a} equivalents)					
	0.00125 ppm		0.0125 ppm		0.25 ppm	
	Goat 1	Goat 2	Goat 3	Goat 4	Goat 5 [a]	Goat 6
Liver	0.2	0.6	2.1	3.5	98.0	16.4
Kidney	0.3	0.3	0.9	1.2	22.7	4.8
Lung	< 0.2	< 0.2	0.3	0.7	11.9	2.5
Peripheral fat	< 0.2	< 0.2	1.3	2.2	50.0	7.6
Omental fat	< 0.2	< 0.2	1.4	2.2	49.3	6.8
Leg muscle	< 0.2	< 0.2	0.3	0.4	7.6	1.7
Loin muscle	< 0.2	< 0.2	0.3	0.3	9.9	1.2
Mammary gland	< 0.2	< 0.2	0.4	0.6	13.3	3.6
Brain	< 0.2	< 0.2	< 0.2	< 0.2	1.0	0.3
Heart	< 0.2	< 0.2	0.4	0.8	20.6	2.6

[a] Animal off feed days 9–11, low water consumption. All other clinical observations were normal.

Avermectin B_{1a} was the major residue in all tissues, comprising to up to over 90% TRR (Table 36).

Table 36 Percent unchanged avermectin B_{1a} in tissues from goats dosed with [^{3}H]avermectin B_{1a} determined by reverse isotope dilution assay (RIDA), as % TRR (Maynard *et al.*, 1989)

Animal	Liver	Kidney	Leg Muscle	Loin Muscle	Fat	Milk
0.00125 ppm						
Goat 1	76 [a]	–	–	–	–	
Goat 2	77 [a]	–	–	–	–	
0.0125 ppm						
Goat 3	95 (92)	97	–	96 [a]	97	
Goat 4	87	92	–	–	99	
0.25 ppm						
Goat 5	95	94 (89)	91 (88) (91)	84	99	95 (98)
Goat 6	41 (40)	40 (37)	68	73	86	70 (79)

[a] Tissue residue levels were very low (0.2 µg/kg–0.6 µg/kg), so results should be considered estimates.

Results in parenthesis are repeat determinations

Tables 37 and 38 show the HPLC profile of the residues in tissues, assigned according to retention time relative to that of avermectin B_{1a}. Metabolite 24-hydroxymethyl-avermectin B_{1a},

was a major residue in liver and kidney of the lower dosing goats and was present at 2–11% TRR in milk from D3.

Table 37 Characterization of residue in goat liver extracts, in % of TRR, by reverse-phase chromatography

	0.00125 ppm		0.0125 ppm		0.25 ppm	
Fractions [a]	Goat 1	Goat 2	Goat 3	Goat 4	Goat 5	Goat 6
0.88–1.13, Avermectin B_{1a} [b]	50	40	91	88	90	63
0.11–0.30, 24-hydroxymethyl-avermectin B_{1a} [c]	37	54	1	3	3	26
0.30–0.71	5	3	1	2	2	5
0.71–0.88	5	2	2	4	3	2
1.13–1.55	3	1	1	2	1	1
Column Wash	[b]	[b]	3	1	1	3

[a] Average retention times relative to avermectin B_{1a}

[b] Sample radioactivity was low for these samples

[c] Identified from in-vitro rat liver microsomes

Table 38 Characterization of goat kidney, fat and muscle residues, in % of TRR, by reverse-phase chromatography

	0.00125 ppm						0.25 ppm					
	Kidney		Fat		Muscle (leg/loin)		Kidney		Fat		Muscle (leg/loin)	
Fraction [a]	G3	G4	G3	G4	G3	G 4	G5	G6	G 5	G6	G5	G6
Avermectin B_{1a}	83	83	99	93	–/88	–	84	42	93	85	86/89	77/79
24-hydroxymethyl-avermectin B_{1a}	5	6	< 0.5	< 0.5	–/2	–	6	43	< 0.5	3	1/1	10/10
0.30–0.71	2	2	< 0.5	< 0.5	–/2	–	3	9	1	3	2/2	5/4
0.71–0.88	2	4	< 0.5	1	–/5	–	4	2	1	1	8/5	3/4
1.13–1.55	2	1	< 0.5	1	–/5	–	2	1	1	1	2/1	2/2
Column Wash	5	3	0	5	0	–	1	3	5	8	1/2	4/3

[a] Retention times relative to avermectin B_{1a}

A second metabolite, isolated from the rat liver microsome incubations, and identified as 3"-desmethyl-avermectin B_{1a}, was isolated from Goat 5 liver, and was estimated to comprise < 1 to 5% TRR. This metabolite was identified in urine and faeces, but was not significant in tissues.

Fat tissue contained non-polar material (0–8%), which was captured in a methanol column wash. This fraction from Goat 6 (8%) was hydrolysed with sulphuric acid and analysed by HPLC. Avermectin B_{1a} was hydrolysed under these conditions to the monosaccharide-B_{1a} and further to the aglycone-B_{1a}; 24-hydroxymethyl avermectin B_{1a} was hydrolysed to the aglycone-24-hydroxymethyl avermectin B_{1a}. The reaction product produced from the fat corresponds to the aglycone-24-hydroxymethyl avermectin B_{1a} indicating that the fat must have contained 24-hydroxymethyl avermectin B_{1a} in a conjugated form. In summary Goat 6 fat tissue was shown to contain 85% avermectin B_{1a}, 3% unconjugated 24-hydroxymethyl avermectin B_{1a} and at least 3% conjugated 24-hydroxymethyl avermectin B_{1a} (acid hydrolysis released 40% of the 8% non-polar column-wash fraction).

Based on the structures identified, the metabolism of avermectin B_{1a} in the goat proceeds via oxidation of the methyl group (to a hydroxymethyl group) at the 24 carbon position and to a lesser extent demethylation at the 3" position. The proposed pathway is shown in Figure 3.

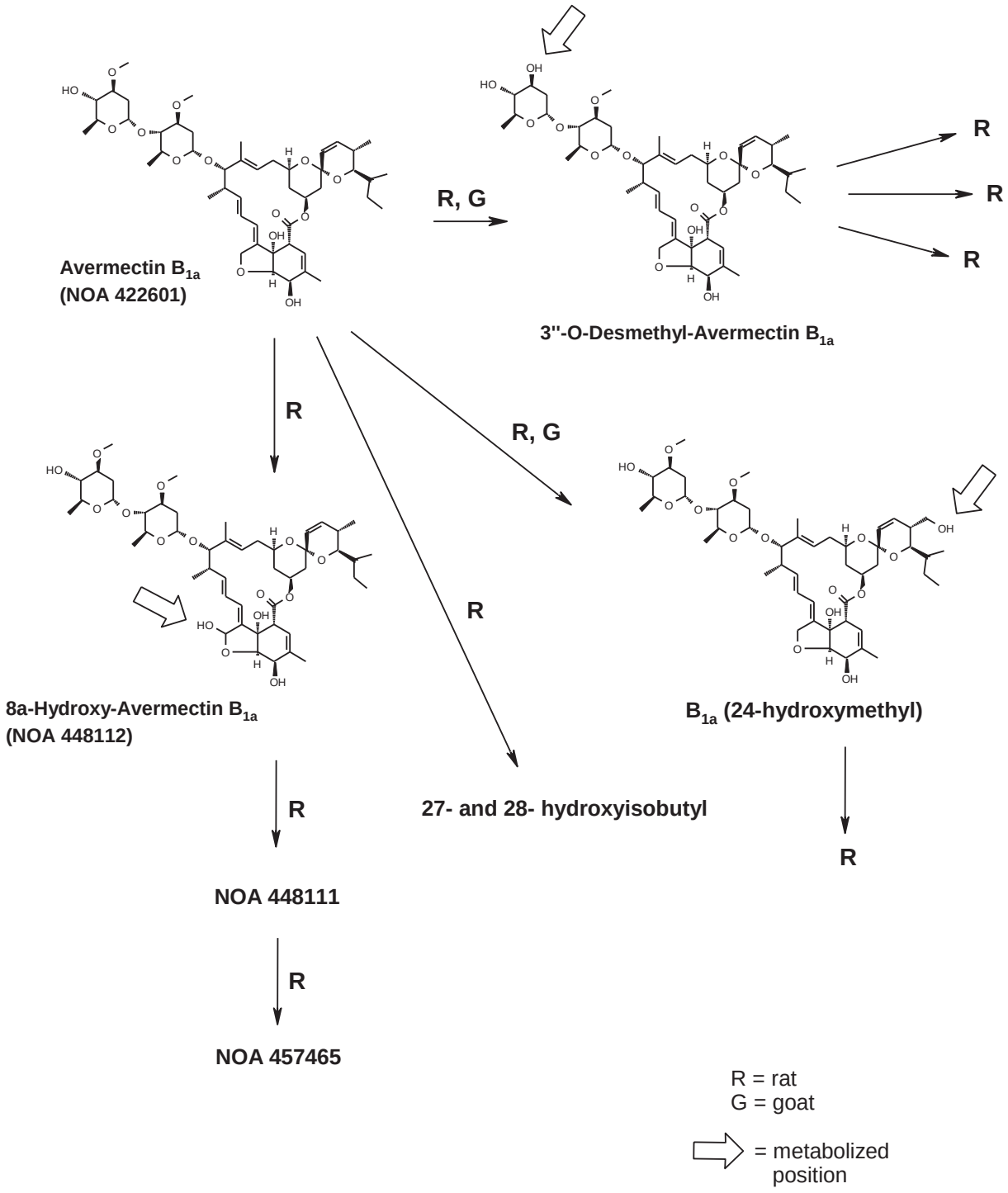


Figure 3 Metabolic pathway of avermectin B_{1a} in the goat and the rat

Residue analytical methods

Methods by HPLC-FL: avermectin B_{1a} is determined as the sum of avermectin B_{1a} and its 8,9-Z isomer and avermectin B_{1b} as the sum of avermectin B_{1b} and its 8,9-Z isomer

Method M-073 was developed to determine avermectin B_{1a}, avermectin B_{1b} and their 8,9-Z isomers in plant material (Arenas, 1996; 1998; Norton, 1997; Giles, 1996; Richard & Mackenzie, 2005).

Residues are extracted with acetonitrile/0.1% phosphoric acid and from the aqueous solution by partitioning into hexane. After adding sodium sulphate to the hexane phase, the organic extract is clean-up in an aminopropyl cartridge, and residues eluted with ethyl acetate/methanol. Fluorescent derivatives are formed by reaction with a mixture of triethylamine, trifluoroacetic anhydride and 1-methylimidazole, and determined by reversed-phase HPLC with fluorescence detection (HPLC-FL; Ex.: 365 nm, Em: 470 nm). HPLC analysis of avermectin B_{1a} and its 8,9-Z isomer results in a single peak, and avermectin B_{1a} is determined as the sum of avermectin B_{1a} and its 8,9-Z isomer and avermectin B_{1b} as the sum of avermectin B_{1b} and its 8,9-Z isomer. Validation data are summarized in Table 39. The limit of quantification for avermectin B_1 residues in crop matrices using Method M-073 was established at 0.002 mg/kg for each component analyte.

Table 39 Recovery data for method M-073 (HPLC-FL)

Commodity	Fortification level (mg/kg)	Range of recovery (%)	n	Mean (%)	RSD (%)	Report
Avermectin B_{1a}						
Fresh prunes	0.002	91–94	3	92	2	M-073 and M-073.1
	0.010	87–94	3	91	4	
	0.050	97–98	3	98	1	
	0.100	89–91	3	90	1	
Dried prunes	0.002	99–104	3	101	3	M-073 and M-073.1
	0.010	86–98	3	91	6	
	0.050	86–95	3	90	4	
	0.100	72–79	3	75	4	
Strawberries	0.001	71–98	2	85	-	E-97-MK-936-SB
	0.002	75–80	3	77	3	
	0.010	70–80	3	75	5	
	0.050	70	3	70	0	
Lettuce	0.002	79–95	5	88	7	RJ3670B
	0.020	88–100	5	92	5	
Radish, whole plant	0.002	96, 93, 100	3	96		MSD 430/961248
	0.010	94, 92, 98	3	95		
	0.031	101, 102, 96	3	100		
	1.027	93, 93, 92	3	93		
Radish, tubers	0.002	90, 92, 82	3	88		
	0.010	96, 93, 102	3	97		
	0.031	95, 100, 101	3	99		
Avermectin B_{1b}						
Fresh prunes	0.002	88–94	3	91	3	M-073 and M-073.1
Dried prunes	0.002	78–82	3	80	2	M-073 and M-073.1
Strawberries	0.002	70–75	3	73	3	E-97-MK-936-SB
Lettuce	0.002	72–92	5	86	7	RJ3670B
	0.020	84–96	5	88	5	
Avermectin B_{1a} 8,9-Z isomer						
Fresh prunes	0.002	100–101	3	101	1	M-073 and M-073.1
	0.010	96	3	96	0	
	0.050	103–105	3	104	1	
Dried prunes	0.002	87–109	4	99	11	M-073 and M-073.1
	0.010	90–113	4	99	10	
	0.050	98–104	3	100	3	
Strawberries	0.002	70–75	3	73	3	E-97-MK-936-SB
	0.010	70–73	3	72	2	
	0.050	70	3	70	0	
Lettuce	0.002	62–75	5	70	8	Richard, 2005; RJ3670B
	0.020	74–81	5	78	4	

The extractability of abamectin residues in citrus fruit (with acetone), celery (with acetone), cotton (with 90/10 v/v acetone/water) and tomatoes (with 80/20 v/v acetonitrile/water)

was demonstrated in radio-labelled metabolism studies. The polarity of the extraction solvent used in analytical method M-073 is comparable to those used in the metabolism studies.

Methods M-007.1 (Cobin, 1995, 1995a; MSD 329/942555), 91-1 (Prabhu, 1991; Kvatemick, 1993, 1996; Richards & Mackenzie, 2005) and MSD 328/942104 (White, 1995) were developed to determine and quantify avermectin B_{1a}, avermectin B_{1b} and their 8,9-Z isomers in different crops, using similar procedures. Homogenized samples are extracted with a hexane/water/acetonitrile, hexane extracts are cleaned up in an aminopropyl SPE, residues derivatized with trifluoroacetic anhydride (reagent) and 1-methylimidazole (catalyst) and determined by reversed-phase HPLC-FL. Validation data for apple, tomato and grapes are summarized in Table 40.

Table 40 Validation recovery data for Methods M-007.1, 91.1 and MSD 328/942104 by HPLC/FL

Analyte	Fortification level (mg/kg)	Range of recovery (%)	n	Mean (%)	RSD (%)	Report
Apple						
Avermectin B_{1a}	0.01	71–100	12	82	12	Cobin, 1995a
	0.01	66–94	15	86	9	
	0.01	71–92	17	81	7	
	0.09	80–85	2	83	–	
Avermectin B_{1b}	0.005	78–84	2	81	–	
Tomato						
Avermectin B_{1a}	0.005	88–90	3	89	1	Kvatemick, 1993, 1996
	0.028	93–114	3	104	11	
	0.070	84–96	3	90	6	
Avermectin B_{1b}	0.002	92–102	3	96	5	
Avermectin B_{1a} 8,9-Z isomer	0.002	87	3	87	0	
	0.027	79–87	3	84	4	
	0.068	78–79	3	79	1	
Avermectin B_{1a}	0.002	95–106	5	102	4	Richards & Mackenzie, 2005a
	0.020	93–119	5	108	9	
Avermectin B_{1a} 8,9-Z isomer	0.002	79–94	5	91	7	
	0.020	97–99	5	97	1	
Avermectin B_{1b}	0.002	97–107	5	104	4	
	0.020	91–117	5	106	9	
Grape						
Avermectin B_{1a}	0.002	70–87	8	82	5	Prabhu, 1991
	0.050	76–91	9	83	5	
Avermectin B_{1b}	0.002	73–93	9	80	7	
Avermectin B_{1a} 8,9-Z isomer	0.002	71–88	8	78	7	
	0.050	70–93	8	77	9	
Avermectin B_{1a}	0.002	85–90	3	87	3	White, 1995
	0.100	92–110	3	99	10	
Avermectin B_{1a} 8,9-Z isomer	0.002	90–100	3	97	6	
Avermectin B_{1b}	0.002	80–90	3	85	6	
	0.100	94–103	3	98	5	

Methods M-044 and M-036.2 were developed to determine and quantify avermectin B_{1a}, avermectin B_{1b} and avermectin B_{1a} 8,9-Z isomer in fresh and immature hops and in dried hops, respectively (Norton, 1997; Report No. MER/AVE/96091). The methods involve rehydration and extraction with a methanol/deionised water mixture, partition into hexane and extract purified on aminopropyl SPE cartridges. The purified extract is derivatised using trifluoroacetic anhydride and residues analysed by HPLC-FL. Validation data are summarized in Table 41. The LOQ was 0.0025 mg/kg for avermectin B_{1a} and 0.005 mg/kg for avermectin B_{1b} and the 8,9-Z isomer of avermectin B_{1a}.

Table 41 Validation Recovery Data for Method M-044 and M-036.2 in hops by HPLC/FL (Norton, 1997)

Commodity	Fortification level (mg/kg)	Range of recovery (%)	n	Mean (%)	RSD (%)
Fresh hops					
Avermectin B_{1a}	0.0025	84–92	3	87	5
	0.005	86–102	3	92	10
	0.100	73–93	3	82	12
Avermectin B_{1b}	0.005	80–84	3	82	2
Avermectin B_{1a}8,9-Z isomer	0.005	84–92	3	88	5
	0.100	86–91	3	89	3
Immature hops					
Avermectin B_{1a}	0.0025	80–96	3	91	10
	0.005	94–100	3	97	3
	0.100	72–81	3	77	6
Avermectin B_{1b}	0.005	70–78	3	73	6
Avermectin B_{1a}8,9-Z isomer	0.005	102–104	3	103	1
	0.100	83–87	3	85	3
Dried hops					
Avermectin B_{1a}	0.0025	96–108	3	103	6
	0.005	98–106	3	101	4
	0.100	83–88	3	85	3
Avermectin B_{1b}	0.005	70–82	3	77	8
Avermectin B_{1a}8,9-Z isomer	0.005	98–106	3	102	4
	0.100	88–91	3	89	2

Methods by LC-MS/MS: determination of individual analytes

Method Meth-192, rev.2 was developed to determine and quantify avermectin B_{1a}, avermectin B_{1b} and their 8,9-Z isomers in plant material by LC-MS/MS. Transition ions for avermectin B_{1a} and its isomer ($[M+Na]^+$) were m/z = 895.5 → 751.5 for quantification and m/z = 895.5 → 449.2 for confirmation. Transitions for avermectin B_{1b} ($[M+Na]^+$) were m/z = 881.2 → 737.0 for quantification and m/z = 881.2 → 449.2 for confirmation. Residues are extracted with acetonitrile: 0.1% H_3PO_4 (25:75), partitioned into toluene and clean-up using aminopropyl solid phase extraction (SPE). The purified extract is evaporated, dissolved in acetonitrile, and then submitted to LC-MS/MS (reverse-phase column). The LOQ for all three analytes, in all matrices, is 0.002 ppm. Validation data are summarized in Table 42.

Table 42 Recovery data for Method Meth-192, rev.2, using LC-MS/MS

Commodity	Fortification level (mg/kg)	Range of recovery (%)	n	Mean (%)	RSD (%)	Report
Cherries						
Avermectin B_{1a}	0.002	93, 97	2	95	–	T005601-07
	0.02	91, 91	2	91	–	
Avermectin B_{1b}	0.002	85, 100	2	93	–	
	0.02	73, 94	2	84	–	
Avermectin B_{1a} 8,9-Z isomer	0.002	69, 84	2	77	–	
	0.02	77, 87	2	82	–	
Peach						
Avermectin B_{1a}	0.002	70, 78	2	74	–	T005601-07
	0.02	78, 98	2	88	–	
Avermectin B_{1b}	0.002	64, 93	2	79	–	
	0.02	79, 106	2	93	–	
Avermectin B_{1a} 8,9-Z isomer	0.002	66, 76	2	71	–	
	0.02	71, 86	2	79	–	
Plum						
Avermectin B_{1a}	0.002	75–99	5	84	11	T005601-07
	0.02	80–103	5	87	11	
	0.10	74, 77	2	76	–	

Commodity	Fortification level (mg/kg)	Range of recovery (%)	n	Mean (%)	RSD (%)	Report
Avermectin B_{1b}	0.002	104–111	3	108	3	
	0.02	64–128	3	100	33	
Avermectin B_{1a} 8,9-Z isomer	0.002	73–102	3	83	20	
	0.02	76–100	3	87	14	
Strawberries						
Avermectin B_{1a}	0.002	74–112	6	88	16	T001870-07
	0.0333	95	1	95	–	
	0.0336	92–111	3	100	10	
	0.05	95, 105	2	100	–	
	0.3333	101	1	101	–	
	0.50	82	1	82	–	
	0.838	90, 91	2	91	–	
Avermectin B_{1b}	0.002	84–133	4	108	20	
	0.022	83	1	83	–	
	0.0298	78	1	78	–	
	0.05	97, 118	2	108	–	
Avermectin B_{1a} 8,9-Z isomer	0.002	78, 98	2	88	–	
Grapes						
Avermectin B_{1a}	0.002	82–101	8	94	6.8	T005598-07
	0.02	85–101	6	94	7.0	
	0.20	93–105	4	99	5.5	
Avermectin B_{1b}	0.002	79–107	6	95	12	
	0.02	82–96	4	90	7.2	
	0.20	88–111	4	97	10	
Avermectin B_{1a} 8,9-Z isomer	0.002	88–100	6	94	4.8	
	0.02	82–92	4	86	5.7	
	0.20	91–103	4	97	5.1	
Celery						
Avermectin B_{1a}	0.002	68–97	4	83	15	T005593-07
	0.033	87–95	5	92	3.4	
	0.50	96	1	96	–	
Avermectin B_{1b}	0.002	72–91	4	81	12	
	0.50	74	1	74	–	
Cotton Seed						
Avermectin B_{1a}	0.002	110–120	5	116	3.6	T005597-07
	0.02	101–119	5	110	6.2	
Avermectin B_{1b}	0.002	72–86	5	76	8.0	
	0.02	70–81	5	77	5.3	
Avermectin B_{1a} 8,9-Z isomer	0.002	75–92	5	83	8.2	
	0.02	73–91	5	83	7.9	
Cotton Gin-Trash						
Avermectin B_{1a}	0.002	72–100	3	85	16	T005597-07
	0.02	65–80	3	74	11	
	1.2	66, 82	2	74	–	
Avermectin B_{1b}	0.002	55–125	3	87	40	
	0.02	67–86	3	79	13	
Avermectin B_{1a} 8,9-Z isomer	0.002	69–88	3	77	13	
	0.02	75–81	3	77	4.2	
Cottonseed Hulls						
Avermectin B_{1a}	0.002	70–90	3	78	13	T005597-07
	0.02	86–98	3	91	7.1	
Avermectin B_{1b}	0.002	73–84	3	79	7.2	
	0.02	70–93	3	85	15	
Avermectin B_{1a} 8,9-Z isomer	0.002	71–84	3	77	8.4	
	0.02	77–87	3	83	6.4	
Cotton Meal						
Avermectin B_{1a}	0.002	107	1	107	–	T005597-07
	0.02	82	1	82	–	
Avermectin B_{1b}	0.002	115	1	115	–	
	0.02	104	1	104	–	

Commodity	Fortification level (mg/kg)	Range of recovery (%)	n	Mean (%)	RSD (%)	Report
Avermectin B_{1a} 8,9-Z isomer	0.002	56	1	56	–	
	0.02	87	1	87	–	
Cotton Refined Oil						
Avermectin B_{1a}	0.002	82	1	82	–	T005597-07
	0.02	85	1	85	–	
Avermectin B_{1b}	0.002	87	1	87	–	
	0.02	89	1	89	–	
Avermectin B_{1a} 8,9-Z isomer	0.002	75	1	75	–	
	0.02	71	1	71	–	

Method 1002 Agri was developed to determine and quantify avermectin B_{1a} in raspberries (Baravelli, 2005). Homogenized samples were extracted with dichloromethane and filtered through sodium sulphate. Quantification was by reverse phase LC-MS/MS operating in Multiple Reaction Monitoring (MRM) mode. Transitions ($[M+H]^+$): m/z = 890.4 → 305.3 for quantification and m/z = 890.4 → 145.3 for confirmation. LOQ for avermectin B_{1a} was established at 0.02 mg/kg. Validation data for method 1002 on grapes are provided in Table 43.

Table 43 Recovery data for avermectin B_{1a} in raspberries by LC-MS/MS (Method 1002)

Commodity	Fortification level (mg/kg)	Range of Recovery (%)	n	Mean (%)	RSD (%)
Avermectin B_{1a}	0.02	92–103	6	100	4
	0.05	101, 106	2	104	–
	0.1	102, 108	2	105	–
	0.15	70, 83	2	74	–
	0.40	75, 85	2	80	–

Method REM 198.02 was developed for individual determination of avermectin B_{1a}, avermectin B_{1b} and the 8,9-Z isomer of avermectin B_{1a} in plant material and foodstuffs of animal origin (Satter, 2002; 2002a). Sample preparation and clean-up vary depending on the type of substrate. For high-water substrates, samples were extracted with methanol and cleaned up by C8-SPE. For fatty/oily substrates, the methanol extract was cleaned up by amino SPE, washed by partitioning with n-hexane and cleaned up by a C8-SPE tube. Hops samples were extracted with water and methanol, and after addition of a 5% calcium chloride solution partitioned with n-hexane and the organic phase was cleaned up by amino-SPE. Avermectin B_{1a}, avermectin B_{1b} and the 8,9-Z isomer of avermectin B_{1a} were eluted with a mixture of ethyl acetate/methanol. Residues were determined with a column-switching LC-MS/MS system. Validation data are summarized in Table 44. The LOQ was 0.002 mg/kg for all analytes in all crops, except for hops where the LOQ was 0.01 mg/kg.

Table 44 Recovery data for Method REM 198.02 in crop matrices by LC-MS/MS (n = 5)

		Avermectin B_{1a}			Avermectin B_{1b}			Avermectin B_{1a} 8,9-Z isomer		
	Fortification Level (mg/kg)	Range of recovery (%)	Mean (%)	RSD (%)	Range of recovery (%)	Mean (%)	RSD (%)	Range of recovery (%)	Mean (%)	RSD (%)
Tomato	0.002	75–86	80	5	77–90	85	7	77–90	85	6
	0.02	84–86	85	1	89–96	91	3	80–85	82	2
Orange	0.002	98–112	106	7	99–106	102	3	81–93	87	6
	0.02	89–98	91	4	92–100	96	3	82–94	86	6
Cotton seed	0.002	88–96	92	4	94–110	101	7	84–93	90	5
	0.02	90–97	94	3	97–102	100	2	87–96	92	4
Dried hops	0.01	53–71	62	11	61–80	70	12	52–70	59	13
	0.1	57–62	60	4	60–66	64	4	54–62	57	6
Fresh hops	0.01	99–106	103	3	100–110	107	4	91–97	95	3
	0.1	95–100	97	2	96–98	97	1	88–92	89	2

Validation data for Method REM 198.02 in foodstuffs of animal origin are shown in Table 45 (Satter, 2002; 2002a). LOQ for avermectin B_{1a}, avermectin B_{1b} and the 8,9-Z isomer of avermectin B_{1a} is 0.002 mg/kg in meat, milk and egg.

Table 45 Recovery data for Method REM 198.02 in animal matrices (LC-MS/MS)

		Avermectin B_{1a}			Avermectin B_{1b}			Avermectin B_{1a} 8,9-Z isomer		
Matrix	Fortification Level (mg/kg)	Range of recovery (%)	Mean (%)	RSD (%)	Range of recovery (%)	Mean (%)	RSD (%)	Range of recovery (%)	Mean (%)	RSD (%)
Meat	0.002 [a]	84–112	97	12	100–124	107	11	77–111	95	16
	0.02 [b]	93–119	101	11	98–116	105	7	90–115	100	11
Milk	0.002 [b]	79–94	87	6	82–104	95	9	79–96	89	7
	0.02 [b]	92–98	95	3	99–102	100	1	85–93	89	4
Eggs	0.002 [b]	86–103	93	7	98–111	104	5	79–97	87	10
	0.02 [a]	71–89	82	10	82–104	96	10	67–77	73	7

[a] n=4

[b] n=5

Storage stability under frozen conditions

The frozen storage stability of residues of avermectin $B_{1a}$was tested in homogenised orange, lemon and grapefruit peel samples (Cobin, 1987). Samples were stored at or below –10 °C up to 52 months. Avermectin B_{1a} was extracted from citrus peel and derivatized to yield a residue that was determined by HPLC-FL. The results are presented in Table 46.

Table 46 Storage stability of avermectin B_{1a} in citrus

		Orange Peel				Lemon peel		Grapefruit peel	
Interval, months	Fortification level, mg/kg	Residue remaining		Interval, months	Fortification level, mg/kg	Residue remaining		Residue remaining	
		mg/kg	%			mg/kg	%	mg/kg	%
0	0.025	0.018	73	0	0.005	0.005	106	0.0049	97
1	0.025	0.018	72		0.025	0.0235	94	0.0218	87
1.5	0.025	0.016	65	5.5	0.005	0.0024	48	0.0032	65
2.4	0.025	0.020	78		0.025	0.0128	51	0.0135	54
3.5	0.025	0.020	80	8.5	0.005	0.0049	98	0.0049	98
4	0.025	0.019	76		0.025	0.019	76	0.019	76
10.5	0.025	0.013	51	48	0.005	0.0047	93	0.0042	85
13.5	0.025	0.018	73		0.025	0.0198	79	0.0175	70
52	0.025	0.017	67						

Studies to investigate the storage stability of residues of avermectin B_{1a}, avermectin B_{1b} and the 8,9-Z isomer of avermectin B_{1a} were conducted in tomatoes (Wertz, 1987), celery (Hughes, 1989), strawberries (Siirila, 1997) and pears (Hicks, 1995). Homogenised tomatoes were fortified, stored at frozen conditions (–20 °C to –10 °C) for 15 up to 35 months and analysed by HPLC-FL against an avermectin B_{1a} standard curve. The results are shown in Table 47.

Table 47 Storage stability of avermectin B_1 in tomatoes, celery, strawberries and pears

	Fortification level, mg/kg	Residues remaining		Fortification level, mg/kg	Residues remaining		Fortification level, mg/kg	Residues remaining	
Interval, Months	Avermectin B_{1a}	mg/kg	%	Avermectin B_{1b}	mg/kg	%	Avermectin B_{1a} 8,9-Z-isomer	mg/kg	%
Tomatoes, –10 °C (Wertz, 1987)									
1 day	0.0101	0.0050	49	0.0038	0.0028	74	0.0092	0.0059	64
	0.0507	0.0385	76						
1	0.0101	0.0075	74	0.0038	0.0025	66	0.0092	0.0046	50

	Fortification level, mg/kg	Residues remaining		Fortification level, mg/kg	Residues remaining		Fortification level, mg/kg	Residues remaining	
Interval, Months	Avermectin B_{1a}	mg/kg	%	Avermectin B_{1b}	mg/kg	%	Avermectin B_{1a} 8,9-Z-isomer	mg/kg	%
	0.0507	0.032	63						
3	0.0101	0.0066	65	0.0038	0.0022	58	0.0092	0.0039	42
	0.0507	0.031	61						
6	0.0101	0.0062	61	0.0038	0.0025	66	0.0092	0.0046	50
	0.0507	0.0335	66						
15	0.0101	0.0083	82	0.0038	0.0039	103	0.0092	0.0084	91
	0.0507	0.0527	104						
Celery,–20 °C (Hughes, 1989)									
0	0.0104	0.0097	93	0.0152	0.0139	91	0.0095	0.0072	76
	0.206	0.184	89						
1	0.0104	0.0087	84	0.0152	0.0151	99	0.0095	0.0075	79
	0.206	0.174	84						
3	0.0104	0.0083	80	0.0152	0.0156	103	0.0095	0.0069	73
	0.206	0.176	85						
6	0.0104	0.0084	81	0.0152	0.0156	103	0.0095	0.008	84
	0.206	0.189	92						
12	0.0104	0.0088	85	0.0152	0.014	92	0.0095	0.0075	79
	0.206	0.187	91						
18	0.0104	0.0071	68	0.0152	0.0122	80	0.0095	0.0065	68
	0.206	0.160	78						
24	0.0104	0.0082	79	0.0152	0.0133	87	0.0095	0.0087	70
	0.206	0.146	71						
Strawberries, -20 °C (Siirila, 1997)									
0	0.0099	0.0096	97	0.0053	0.0049	92	0.01	0.0100	100
	0.071	0.0712	100						
1	0.0099	0.0095	96	0.0053	0.0047	89	0.01	0.0089	89
	0.071	0.0684	96						
3	0.0099	0.0082	83	0.0053	0.0046	87	0.01	0.0078	78
	0.071	0.0577	81						
6	0.0099	0.0098	99	0.0053	0.0050	94	0.01	0.0094	94
	0.071	0.0677	95						
12	0.0099	0.0090	91	0.0053	0.0051	96	0.01	0.0078	78
	0.071	0.0594	84						
18	0.0099	0.0092	93	0.0053	0.0053	100	0.01	0.0096	96
	0.071	0.0671	95						
24	0.0099	0.0097	98	0.0053	0.0058	109	0.01	0.0095	95
	0.071	0.0728	103						
Pears, –10 to –20 °C (Hicks, 1995)									
0	0.0102	0.0091	89	0.0053	0.0046	87	0.01	0.0087	87
	0.071	0.0640	90						
1.5	0.0102	0.0094	92	0.0053	0.0051	96	0.01	0.0095	95
	0.071	0.0605	85						
3	0.0102	0.0092	90	0.0053	0.0055	103	0.01	0.0099	99
	0.071	0.0630	89						
6	0.0102	0.0080	79	0.0053	0.0038	72	0.01	0.0087	87
	0.071	0.0510	72						
12	0.0102	0.0088	86	0.0053	0.0060	113	0.01	0.0097	97
	0.071	0.0595	84						
22	0.0102	0.0091	89	0.0053	0.0049	92	0.01	0.0097	97
	0.071	0.0640	91						
35	0.0102	0.0087	85	0.0053	0.0038	72	0.01	0.0095	95
	0.071	0.0610	86						

The frozen storage stability of residues of avermectin B_{1a} or its 8,9-Z isomer at –20 °C was tested separately in grapes and grape products over approximately 1 year (Cobin, 1998). Samples were analysed by HPLC- FL. The results are presented in Table 48.

Table 48 Storage stability of avermectin B_{1a} in grape and processed fractions

Matrix	Interval, months	Fortification level, mg/kg	Residues remaining of avermectin B_{1a}		Residues remaining of avermectin B_{1a} 8,9-Z isomer	
			mg/kg	%	mg/kg	%
Raisins	12.5	0.02	0.0056	28	0.0131	66
Raisin waste	12	0.02	0.0138	73	0.0123	62
Unwashed grapes	14.5	0.02	0.0149	75	0.0138	69
Washed grapes	14.5	0.02	0.0163	81	0.0146	73
Stems	12	0.02	0.0162	81	0.0150	75
Wet pomace	12 [a]	0.02	0.0160	80	0.0146	73
Dry pomace	12	0.02	0.0177	89	0.0177	89
Fresh juice	14	0.02	0.0133	67	0.0128	64
Processed juice	14	0.02	0.0148	74	0.0119	59

[a] Interval not given in the report but report reflected that all matrices were stored for about one year

Samples of tomatoes, runner beans (beans, green with pods), sunflower seeds, potatoes and orange peel were fortified with avermectin B_{1a}, avermectin B_{1b} and avermectin B_{1a} 8,9-Z-isomer, and stored for up to two years in a deep freezer at ≤ –18 °C (Kwiatkowski & Hill, 2007). Six replicate samples were analysed at zero time and triplicate samples were removed afterwards by LC-MS/MS (REM 198.02).The results presented are an average of multiple samples and are not corrected for freshly fortified recoveries.

Table 49 Storage stability of abamectin in crop commodities fortified at 0.05 mg/kg

	Interval, months	Residues remaining Avermectin B_{1a}		Residues remaining Avermectin B_{1a}		Residues remaining Avermectin B_{1a},9-Z-isomer	
Matrix		mg/kg	%	mg/kg	%	mg/kg	%
Tomatoes	0	0.05	100	0.05	100	0.04	100
	2.8	0.05	91	0.05	95	0.04	94
	5.3	0.04	86	0.04	85	0.04	97
	12.4	0.04	80	0.04	85	0.04	97
	17.7	0.04	85	0.04	84	0.04	101
	23.9	0.05	101	0.04	83	0.05	118
Beans (green with pod)	0	0.04	100	0.04	100	0.04	100
	3.0	0.04	97	0.04	94	0.03	90
	5.1	0.04	102	0.04	98	0.03	97
	12.6	0.03	94	0.04	97	0.03	92
	18.0	0.03	95	0.04	92	0.03	89
	24.2	0.04	103	0.04	94	0.04	103
Sunflower seeds	0	0.04	100	0.04	100	0.04	100
	2.8	0.04	116	0.04	102	0.05	109
	5.1	0.04	101	0.04	94	0.05	117
	11.8	0.04	98	0.04	96	0.04	97
	17.3	0.04	115	0.04	97	0.04	98
	24.2	0.05	121	0.04	103	0.04	106
Potatoes	0	0.04	100	0.04	100	0.04	100
	2.8	0.04	94	0.04	102	0.03	85
	5.1	0.04	102	0.04	106	0.04	94
	12.0	0.04	95	0.04	99	0.04	100
	17.5	0.04	96	0.04	93	0.04	91
	23.9	0.04	98	0.04	91	0.04	104
Orange peel	0	0.04	100	0.04	100	0.04	100
	3.0	0.04	86	0.04	91	0.03	87
	5.9	0.04	90	0.04	94	0.04	102
	13.3	0.04	93	0.04	100	0.04	98

USE PATTERNS

Abamectin is registered in many countries using high or low volume sprayers or, in some countries, by very-low volume or ultra-low volume equipment for aerial application. Table 50 shows the registered uses in countries where supervised trials have been conducted or in countries with GAPs similar to those where the supervised trials were carried out.

Table 50 Selected registered uses for abamectin as foliar spray (EC formulation 18 g ai/L)

Crop	Country	Application			DAT (days)
		Rate g ai/ha	Water L/ha	No or/ Season max kg ai/ ha	
Avocado	USA	26	> 935	2	14
Bean (green with pods)	Spain	18	500–1000	3	3
Bean (dry)	USA	21	> 94	2	7
Raspberry	Italy	22	not specified	1	7
Celeriac	USA	21	> 187	2	7
Celery	Greece	9	500	4	14
	USA	21	> 187	2	7
Citrus	USA	26	> 94	3 [a]	7
Coffee	Brazil	27	400	1	14
Cotton	Spain	18	1000	3	3
	USA	21	> 45.5	2	20
Cucumber/gherkin	Denmark	22	250–1500 [b]	4	3
Eggplant	Greece	22	500–1200	4	3
Endive	Slovenia	18	not specified	1	7
Fruiting vegetables, except curcubits. Include pepper, chilli pepper	USA	21	> 468	2	7
Grape	USA	21	> 468	2	28
Hops	Slovenia	22	300–400	2	28
	USA	21	> 374	2	28
Leek	Belgium	9	1000	3	7
Lettuce	Greece	9	500	4	14
	Italy	18	not specified	3	7
Mango	Brazil	14	800	4	7
Melon/Watermelon	Denmark	22	250–1500 [b]	3	3
Onion/shallot	USA	21	> 187	2	30
Papaya	Brazil	22	1000	3	14
Peach	Italy	22	not specified	2	14
Peanut	Argentina	1.8	not specified	1	30
Pepper	Denmark	22	500–1500 [b]	5	3
Pome Fruit	Italy	22	not specified	2	28
Radish	Belgium	9	> 1000	2	14
Rice	China	14	682	2	21
Spinach	USA	21	> 187	2	7
Stone Fruit	USA	26	> 374	2	21
Strawberries	Denmark	22	250–1500 [b]	3	3
	USA	22	> 468	4	3
Tomato	Denmark	22	250–1500 [b]	5	3
	Greece	22	500–1200	4	3
Tree Nuts	USA	26	> 374	2	21
Tuberous and corm vegetables, include potato, sweet potato and yam	USA	21	> 187	2	14

[a] Subject to a maximum seasonal application of 53 g ai/ha

[b] Greenhouse application only

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Supervised residue trials conducted with abamectin on a variety of crops in China, Brazil, European countries, and USA from 1986 to 2012 were submitted to the Meeting. All trials were conducted using foliar spray of EC formulation. Studies were conducted according to GLP, except those

conducted before the 1990's. Concurrent determination of residues in untreated crops gave residues < LOQ. Residues of abamectin arising from independent trials that used patterns where rate or days after treatment (DAT) ± 25% of GAP are underlined and considered for estimation of maximum residue levels and STMRs. Trials which were not exactly within that range but, with the support of additional information were also considered for the estimations were also underlined.

When residues in samples harvested at a later stage were higher than those found at the critical DAT, they were used for the estimations. When multiple field samples from one plots were taken for analysis, the mean was selected for the estimations. When two field trials were conducted in the same location in the same period/season, only the highest result was considered. For protected trials, the location was considered not relevant.

The data submitted are summarized in Table 51. In total, 601 supervised trials were submitted and food commodities analysed for residues; in some trials, feed commodities were also analysed.

Table 51 Summary of supervised residue trials conducted with abamectin

Commodity	Location	Number of trials	Table	Commodity	Location	Number of trials	Table
Citrus	USA	21	52	Lettuce	Europe	34	70
Pome fruit	Europe	42	53	Spinach	USA	11	71
Cherry	USA	18	54	Bean (green with pods)	Europe	16	72
Peach	Europe/USA	12/17	55	Bean (dry)	USA	12	73
Plums	USA	17	56	Celeriac	USA	2	74
Raspberry	Italy	4	57	Potato	USA	18	75
Strawberries	Europe/USA	8/28	58	Radish	Netherlands	3	76
Grape	USA	24	59	Celery	Europe/USA	7/6	77
Avocado	USA	5	60	Rice	China	24	78
Mango	Brazil	5	61	Tree nuts	USA	32	79
Papaya	Brazil	12	62	Cotton	Europe/USA	8/14	80
Onion/shallot	USA	8	63	Peanut	Brazil	4	81
Leek	Europe	12	64	Coffee	Brazil	5	82
Cucumber/gherkin	Europe	29	65	Hops	Europe/USA	8/4	83
Melon	Europe	13	66	Rice husk	China	25	84
Pepper	Europe/USA	18/4	67	Green bean, vines	Europe	8	85
Tomato	Europe	43	68	Almond hulls	USA	10	86
Eggplant	France	2	69	Cotton hulls	Europe	8	87

Citrus fruits

Twenty one residue trials on citrus were carried out in the USA in 1986. Samples were stored deep-frozen for a maximum of 6.5 months (198 days) and analysed by HPLC-FL. In this study, LOQ was 0.005 mg/kg and LOD was 0.002 mg/kg. The results are shown in Table 55.

Table 52 Supervised trials conducted in the USA in 1986 with abamectin on citrus (whole fruit) (6012-172B and MK 936/0165)

Location	Crop (Variety)	Application rate, g ai/ha	DAT (days)	Residue (mg/kg)		Report; Trial
				Avermectin B_{1a} + 8,9-Z-isomer	Avermectin B_{1b} + 8,9-Z-isomer	
Clemont, FL	Grapefruit (White)	3× 28	0 7	< 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2)	6012-172B; 001-86-002R
Texas	Grapefruit (Ruby Red)	4× 28	0 1 3 7	0.006, < 0.005 (3), 0.009 < 0.005 (4) < 0.005 (4)	not analysed	001-86-620R
		4× 56	0 1	0.008, 0.018, 0.005 (2), 0.012, 0.015 0.008, 0.010, < 0.005	not analysed	

Location	Crop (Variety)	Application rate, g ai/ha	DAT (days)	Residue (mg/kg)		Report; Trial
				Avermectin B_{1a} + 8,9-Z-isomer	Avermectin B_{1b} + 8,9-Z-isomer	
			3 7	(3) < 0.005 (4) < 0.005 (7)		
Corona, CA	Lemon	28, 28, 33	0 1 3 7	0.008, 0.006, 0.007, < 0.005 < 0.005 (4) < 0.005 (4) < 0.005 (4)	not analysed	6012-172B; 001-86-114R
		3× 56	0 1 3 7	0.014, 0.011, 0.012 (2) < 0.005 (4) < 0.005 (4) < 0.005 (4)	not analysed	
Clemont, FL	Orange (Hamilin)	3× 28	0 1 3 7 14	< 0.005 (3), 0.008, < 0.005 (4) < 0.005 (4) <u>< 0.005</u> (4) < 0.005 (4)	not analysed	6012-172B; 001-86-003R
		3× 56	0 1 3 7 14	0.006, 0.007 (2), 0.010 < 0.005 (4) < 0.005 (4) < 0.005 (4) < 0.005 (4)	not analysed	
Lake County, FL	Orange (Navel)	3× 28	0 7	< 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2)	6012-172B; 001-86-061R
Arizona	Orange (Navel)	3× 28	0 7	0.005, 0.006 <u>< 0.005</u> (2)	< 0.005 (2) < 0.005 (2)	6012-172B; 001-86-169R
St. Paula, CA	Orange (Valencia)	30, 35, 28	0 7	0.015, 0.016 <u>0.008</u> (2)	< 0.005 (2) < 0.005 (2)	6012-172B; 001-86-196R
		61, 56, 56	0 7	0.016 (2) 0.012 (2)	< 0.005 (2) < 0.005 (2)	
		24, 26, 37	0 7	< 0.005 (2) <u>< 0.005</u> (2)	< 0.005 (2) < 0.005 (2)	
		66, 56, 47	0 7	< 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2)	
Tulare, CA	Orange (Navel)	3× 28	0 7	0.011, 0.010 <u>< 0.005</u> (2)	< 0.005 (2) < 0.005 (2)	6012-172B; 001-86-515R
Tulare, CA	Orange (Navel)	28, 28, 39	0 7	0.026 (2) <u>0.014</u> (0.012, 0.015)	< 0.005 (2) < 0.005 (2)	6012-172B; 001-86-596R
		3× 56	0 7	< 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2)	
		3× 28	0 7	< 0.005 (2) <u>0.010</u> (0.010, 0.011)	< 0.005 (2) < 0.005 (2)	
		3× 56	0 7	< 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2)	
Texas	Orange (Navel)	3× 28	0 7	0.006, 0.008 <u>< 0.005</u> (2)	< 0.005 (2) < 0.005 (2)	6012-172B; 001-86-698R
Lake County, FL	Tangelo	3× 28	0 7	0.007 (2) < 0.<u>005</u> (2)	< 0.005 (2) < 0.005 (2)	6012-172B; 001-86-062R
Lake County, FL	Tangelo	3× 28	0 7	< 0.005, 0.006 < 0.005 (2)	< 0.005 (2) < 0.005 (2)	6012-172B; 001-86-001R

Pome fruit

Forty two supervised residue trials were conducted on <u>pome fruit</u> (33 × apples, 7 × pears) in Europe from 1986 to 2012. Apple and pear samples were stored deep-frozen for a maximum of 24 months with exception of Study 4161, where samples were analysed after 26–37 months. Residues in pome fruit samples were analysed by HPLC-FL or LC-MS/MS. Residue data from supervised trials on pome fruits are summarized in Table 53.

Table 53 Supervised trials conducted in Europe with abamectin in pome fruits

Country year	Crop (Variety)	Application rate, g ai/ha	Growth stage	DAT, days	Residues, mg/kg			Study; trial
					Avermectin B_{1a}	8,9-Z-isomer	Avermectin B_{1b}+ 8,9-Z-isomer	
France 1991 (October)	Apple (Jonagold)	2× 27	28 days before harvest	0 7 14 21 28	0.025, 0.018 (2), 0.013 0.007, 0.011, 0.008 0.009 (2), 0.004, 0.005 0.006, < 0.002, 0.012, 0.008 <u>0.004</u> (0.006, 0.002, 0.004, 0.003)	included	0.003, < 0.002 (3) < 0.002 (3) < 0.002 (4) < 0.002 (4) < 0.002 (4)	MSD 329/942555 ; 066-91-0016R
France 1991 (August)	Apple (Golden Delicious)	2× 27	28 days before harvest	0 28	0.006, 0.015, 0.003, 0.004 <u>0.003</u> (0.003 (2), < 0.002)	included	< 0.002 (4) < 0.002 (3)	MSD 329/942555 ; 066-91-0017R
France 1993	Apple (Idared 106)	13, 16 (with oil)	28 days before harvest	–0 0 7 15 21 28	< 0.002 (2) 0.017, 0.013 < 0.002 (4) < 0.002 (2) < 0.002 (2) < 0.002 (2)	included	< 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2)	MSD 329/942555 ; 066-93-0015R
France 1993	Apple (Golden Delicious)	23, 28 (with oil)	28 days before harvest	0 28	0.010, 0.014 < 0.002 (2)	included	< 0.002 (2) < 0.002 (2)	MSD 329/942555 ; 066-93-0017R
France 1993	Apple (Golden Delicious)	2× 27	28 days before harvest	0 28	0.030, 0.029 <u>0.004</u> (0.003, 0.005)	included	0.004 (2) < 0.002 (2)	MSD 329/942555 ; 066-93-0016R
France 2007	Apple (Golden)	2× 19	BBCH 79–85	–0 0 7 14 21 28	< 0.002 0.008 < 0.002 < 0.002 < 0.002 <u>< 0.002</u>	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	T011028-06; AF/11538/SY/2
France 2007	Apple (Fuji)	22, 20	BBCH 81–85	–0 0 7 14 21 28	< 0.002 0.010 < 0.002 < 0.002 < 0.002 <u>< 0.002</u>	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	T011027-06; AF/11539/SY/1
France 2009	Apple (Fuji)	21, 20	BBCH 85	–0 0 7 14 21 28	< 0.002 0.011 < 0.002 < 0.002 < 0.002 <u>< 0.002</u>	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	CEMS-4442; S09-01570-01
		2× 21	BBCH 85	–0 0 7 14 21 28	< 0.002 0.006 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	
France 2009	Apple (Golden)	20, 21	BBCH 76–85	–0 0 7 14 21 28	< 0.002 0.014 < 0.002 < 0.002 < 0.002 <u>< 0.002</u>	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	CEMS-4443; S09-01569-01
		20, 21	BBCH 76–85	–0 0 7 14 21 28	< 0.002 0.005 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	

Country year	Crop (Variety)	Application rate, g ai/ha	Growth stage	DAT, days	Residues, mg/kg			Study; trial
					Avermectin B_{1a}	8,9-Z-isomer	Avermectin B_{1b}+ 8,9-Z-isomer	
France, Louret 2012	Apple (Golden)	2× 21	BBCH 78–81	–0 0 7 14 21 28	< 0.002 0.006 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	S12-03308; S12-03308-01
France, Torraine 2012	Apple (Braeburn)	2× 20	BBCH 79–85	–0 0 7 14 22 28	< 0.002 0.004 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	S12-03308; S12-03308-02
Italy 1993	Apple (Red Chief)	2× 27	28 days before harvest	0 28	0.006, 0.007 < 0.002 (2)	included	< 0.002 (2) < 0.002 (2)	MSD 329/942555; 067-93-0007R
Italy 1993	Apple (Red Chief)	25, 27	28 days before harvest	0 28	0.015, 0.008 < 0.002 (2)	included	< 0.002 (2) < 0.002 (2)	MSD 329/942555; 067-93-0006R
Italy 2007	Apple (Imperatore)	2× 20	BBCH 81–85	–0 0 7 14 21 28	< 0.002 0.008 0.003 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	T011027-06; AF/11539/SY/2
Italy 2009	Apple (Pink Lady)	21, 20	BBCH 81–83	–0 0 7 14 21 28	< 0.002 0.012 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	CEMS-4442; S09-01570-02
		2× 21	BBCH 81–83	–0 0 7 14 21 28	< 0.002 0.017 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	
Italy, Bologne 2012	Apple (Nero red Rome)	2× 21	BBCH 78–79	–0 0 7 14 20 28	< 0.002 0.005 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	S12-03309; S12-03309-02
Italy Ferrara 2012	Apple (Golden)	21, 22	BBCH 75–77	–0 0 7 14 20 28	< 0.002 0.006 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	S12-03309; S12-03309-01
Germany 1991	Apple (Golden Delicious)	2× 27 (with oil)	28 days before harvest	0 28	0.030, 0.023, 0.021, 0.014 0.008, 0.007 (2), 0.005	included	0.003, 0.002 (2), < 0.002 < 0.002 (3), 0.002	4161; 072-91-0004R
Germany 1991	Apple (Golden Delicious Smoothee M9)	2× 27 (with oil)	28 days before harvest	0 7 14 21 28	0.026, 0.022 (2), 0.020 0.008, 0.006, 0.005, 0.009 0.007 (3), 0.003 0.007, 0.006, 0.004, 0.005 0.004 (0.005, 0.004 (3))	included	0.003 (2), 0.002 (2) < 0.002 (4) < 0.002 (4) < 0.002 (4) < 0.002 (4)	4161; 072-91-0005R
Germany 1991	Apple (Golden	2× 27 (with oil)	28 days before	0 7	0.026, 0.031 (2), 0.027 0.009, 0.018,	included	0.002 (2), 0.003 (2)	4161; 072-91-0006R

Country year	Crop (Variety)	Application rate, g ai/ha	Growth stage	DAT, days	Residues, mg/kg			Study; trial
					Avermectin B_{1a}	8,9-Z-isomer	Avermectin B_{1b}+ 8,9-Z-isomer	
	Delicious)		harvest		0.013, 0.014		< 0.002 (4)	
				14	0.013 (2), 0.010, 0.007			
				22	0.008, 0.009		< 0.002 (4)	
				29	0.007 (0.010, 0.006 (2))		< 0.002 (4)	
							< 0.002	
Germany 2007	Apple (Gloster)	2× 19	BBCH 81–85	–0	< 0.002	< 0.002	< 0.002	T011028-06; AF/11538/SY/1
				0	0.011	< 0.002	< 0.002	
				7	< 0.002	< 0.002	< 0.002	
				14	< 0.002	< 0.002	< 0.002	
				21	< 0.002	< 0.002	< 0.002	
				28	< 0.002	< 0.002	< 0.002	
Germany 2009	Apple (Elstar)	20, 21	BBCH 78–85	–0	< 0.002	< 0.002	< 0.002	CEMS-4442; S09-01569-02
				0	0.014	< 0.002	< 0.002	
				7	0.003	< 0.002	< 0.002	
				14	< 0.002	< 0.002	< 0.002	
				21	< 0.002	< 0.002	< 0.002	
				28	< 0.002	< 0.002	< 0.002	
		2× 21	BBCH 78–85	–0	< 0.002	< 0.002	< 0.002	
				0	0.014	< 0.002	< 0.002	
				7	< 0.002	< 0.002	< 0.002	
				14	< 0.002	< 0.002	< 0.002	
				21	< 0.002	< 0.002	< 0.002	
				28	< 0.002	< 0.002	< 0.002	
Greece, Megas Alexxandros 2012	Apple (Granny Smith)	2× 20	BBCH 77–81	–0	< 0.002	< 0.002	< 0.002	S12-03309; S12-03309-03
				0	0.005	< 0.002	< 0.002	
				7	< 0.002	< 0.002	< 0.002	
				14	< 0.002	< 0.002	< 0.002	
				20	< 0.002	< 0.002	< 0.002	
				28	< 0.002	< 0.002	< 0.002	
Greece, Giannitsa 2012	Apple (Granny Smith)	2× 20	BBCH 77–81	–0	< 0.002	< 0.002	< 0.002	S12-03309; S12-03309-04
				0	0.003	< 0.002	< 0.002	
				7	< 0.002	< 0.002	< 0.002	
				14	< 0.002	< 0.002	< 0.002	
				20	< 0.002	< 0.002	< 0.002	
				28	< 0.002	< 0.002	< 0.002	
Spain 1991	Apple (Red Delicious)	2× 27	28 days before harvest	0	0.013, 0.014, 0.021, 0.017	included	< 0.002 (3), 0.002	4161; 065-91-0007R
				28	< 0.002 (4)		< 0.002 (4)	
Spain 1991	Apple (Golden Delicious)	2× 27 oil	28 days before harvest	0	0.011, 0.012, 0.019, 0.013	included	< 0.002 (4)	4161; 065-91-0008R
				28	0.002 (0.004, < 0.002 (3))		< 0.002 (4)	
Spain 1991	Apple (Red Delicious, Red Chief)	2× 27 oil	28 days before harvest	0	0.009, 0.016, 0.014, 0.011	included	< 0.002 (3), 0.002	4161; 065-91-0009R
				7	0.002, 0.005, < 0.002, 0.003		< 0.002 (3), 0.002	
				14	< 0.002, 0.004, 0.003 (2)		< 0.002 (3), 0.002	
				21	< 0.002 (3), 0.003		< 0.002 (3), 0.002	
				28	0.003 (< 0.002 (2), 0.004, 0.003)		< 0.002 (3), 0.002	
Spain 1993	Apple (Golden Delicious)	26, 28	28 days before harvest	0	0.018, 0.012	included	< 0.002 (2)	329/942555; 065-93-0006R
				28	< 0.002 (2)		< 0.002 (2)	
Spain 1993	Apple (Golden Delicious)	2× 26	28 days before harvest	0	0.017, 0.014	included	0.002, < 0.002	329/942555; 065-93-0007R
				28	< 0.002 (2)		< 0.002 (2)	
UK 1991	Apple (Cox's Orange Pippin)	2× 27 (with oil)	28 days before harvest	0	0.026, 0.019, 0.027, 0.020	included	0.003 (2), 0.002 (2)	4161; 074-91-0003R
				28	0.007 (0.005, 0.005, 0.010, 0.007)		< 0.002 (4)	
UK	Apple	2× 27	28 days	0	0.035, 0.033,	included	0.003 (2), 0.004	4161; 074-

Country year	Crop (Variety)	Application rate, g ai/ha	Growth stage	DAT, days	Residues, mg/kg			Study; trial
					Avermectin B_{1a}	8,9-Z-isomer	Avermectin B_{1b}+ 8,9-Z-isomer	
1991	(Cox's Orange Pippin)	(with oil)	before harvest	7 14 21 28	0.044, 0.043 0.009 (2), 0.010, 0.011 0.007, 0.008 (3) 0.006 (2), 0.004, 0.009 0.005 (3), 0.006		(2) < 0.002 (4) < 0.002 (4) < 0.002 (4) < 0.002 (4)	91-0004R
UK 2012	Apple (Cox)	20, 21	BBCH 75–76	–0 0 7 14 21 27	< 0.002 0.007 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	S12-03308; S12-03308-04
UK 2012	Apple (Cox)	18, 22	BBCH 75–77	–0 0 7 14 20 28	< 0.002 0.007 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	S12-03308; S12-03308-05
France 1986	Pear (Beurre Hardy)	3× 27		0 1 3 7	0.009 < 0.005 < 0.005 (2) < 0.005 (2)	included	< 0.005 < 0.005 < 0.005 (2) < 0.005 (2)	066-86-004R
		3× 54		0 1 3 7	0.017 0.011 0.007, < 0.005 < 0.005 (2)	included	< 0.005 < 0.005 < 0.005 (2) < 0.005 (2)	
France 1986	Pear (Beurre Hardy)	3× 27		0 1 3 7	0.008 < 0.005 < 0.005 (2) < 0.005 (2)	included	< 0.005 < 0.005 < 0.005 (2) < 0.005 (2)	066-86-005R
		3× 54		0 1 3 7	0.026 0.008 0.006, < 0.005 < 0.005 (2)	included	< 0.005 < 0.005 < 0.005 (2) < 0.005 (2)	
France 1986	Pear (Doyenne du Comice)	3× 27	28 days before harvest	0 1 3 7 14 21 28	0.014 0.005 < 0.005 (2) < 0.005 (2) < 0.005 (2) < 0.005 < 0.005	included	< 0.005 < 0.005 < 0.005 (2) < 0.005 (2) < 0.005 (2) < 0.005 < 0.005	066-86-047R
Italy 1988	Pear (Guyot)	3× 27	28 days before harvest	0 1 3 7 14 21 28	0.019 0.010 < 0.005 < 0.005 < 0.005 < 0.005 < 0.005	included	< 0.005 < 0.005 < 0.005 < 0.005 < 0.005 < 0.005 < 0.005	AB-P1; 067-88-0042R
Italy 1988	Pear (Decana)	3× 27	28 days before harvest	0 3 8 10 14 21 28	0.019 (2), 0.020, 0.021 < 0.005 (2), 0.008, 0.006 < 0.005 (3), 0.006 < 0.005 (3), 0.005 < 0.005 (4) < 0.005 (4) < 0.005 (4)	included	< 0.005 (4) < 0.005 (4) < 0.005 (4) < 0.005 (4) < 0.005 (4) < 0.005 (4) < 0.005 (4)	AB-P1; 067-88-0043R
Spain 1995	Pear (Flor de Invierno)	28, 27	28 days before harvest	0 28	0.006, 0.004 < 0.002 (2)	included	< 0.002 (2) < 0.002 (2)	4586; 065-95-0006R
UK 1995	Pear (–)	2× 27	28 days before harvest	0 30	0.015, 0.021 < 0.002 (2)	included	< 0.002 (2) < 0.002 (2)	4586; 074-95-0006R

Cherries

Eighteen supervised residue trials were conducted on cherries in the USA during 1998, 1999 and 2008. Samples were analysed by HPLC/FL or LC-MS/MS (2008 trials). Cherry samples were stored deep-frozen for a maximum of 15.2 months. Residue data from supervised trials on cherry are summarized in Table 54.

Table 54 Results from supervised trials conducted in the USA with abamectin in cherries at 2× 26 g ai/ha

Location, year	Variety	Growth stage	DAT, days	Residues, mg/kg		Study; trial
				Abamectin B_{1a} + 8,9-Z-isomer	Abamectin B_{1b} + 8,9-Z-isomer	
Washington, 1998	Sweet, Bing	green fruit	21	0.008 (0.007, 0.009)	< 0.002 (2)	161-98; OW-IR-604-8/WA
Oregon, 1998	Sweet, Lambert	05 in. diam.	21	0.009 (0.007, 0.011)	< 0.002 (2)	161-98;OW-IR-605-98/OR
Fresno, CA 1998	Sweet, Bing	immature fruit	0 2 6 9 14 18 21 28	0.018, 0.022 0.019, 0.025 0.010, 0.010 0.017, 0.013 0.008, 0.006 0.004, 0.005 0.005 (0.006, 0.004) 0.002, 0.003	< 0.002, 0.002 < 0.002, 0.002 < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2)	161-98;02-IR-024-98/CA
Stanislaus, CA, 1998	Sweet, Black Tartarian	fruit set green fruit	21	0.004 (0.003, 0.004)	< 0.002 (2)	161-98;OW-IR-433-98/CA
Utah, 1998	Tart, Montmorency	green, salmon	21	0.047(0.058, 0.036)	0.007, 0.004	161-98; OW-IR-701-98/UT
			21	0.025, 0.029	0.003 (2)	
Ottawa, MI 1998	Sweet, Ulster	immature fruit	21	0.018, 0.015	< 0.002 (2)	161-98; NE-IR-706-98/MI
Ottawa, MI 1998	Tart, Montmorency	immature fruit	0 2 6 10 14 18 21 28	0.078, 0.094 0.075, 0.060 0.107, 0.044 0.044, 0.045 0.050, 0.037 0.033, 0.020 0.013, 0.028 0.018, 0.016	0.007, 0.009 0.007, 0.005 0.010, 0.005 0.005 (2) 0.005, 0.004 0.003 (2), 0.002 (2) < 0.002, 0.003 < 0.002 (2)	161-98;NE-IR-708-98/MI
Ottawa, MI 1998	Tart, Montmo-rency	immature fruit	21	0.024 (0.023, 0.024)	0.002 (2)	161-98;NE-IR-709-98/MI
Michingan, Oceana 1998	Cherry sweet (Gold)	immature fruit	21	0.016 (0.014, 0.013, 0.007, 0.007)	< 0.002 (2)	161-98; NE-IR-707-98/MI
			21	0.020, 0.014 (2)	< 0.002 (2)	
Winconsin, 1998	Tart, Galaxy	pea size red-orange	21	0.010 (2)	< 0.002 (2)	161-98;MW-IR-703-98/WI
New York, 1998	Tart, Montmo-rency	1–2.1 cm	21	0.011 (0.007 (2), 0.015)	< 0.002 (2)	161-98;NE-IR-803-98/NY
			21	0.005, 0.004, 0.007 (2)	< 0.002 (2)	
Oregon, 1999	Sweet, Bing	¾ in. diameter	21	0.003 (0.003 (2), 0.004)	< 0.002 (4)	172-99; OW-IR-610-99/OR
New York, 2008	Tart, Montmo-rency	BBCH 71–81	21	0.007 (2)	< 0.002 (2)	T005601-07; E03NY081081
Wisconsin 2008	Tart, Montmo-	not reported	21	0.015 (0.020, 0.010)	< 0.002 (2)	T005601-07; E19WI081082

Location, year	Variety	Growth stage	DAT, days	Residues, mg/kg		Study; trial
				Abamectin B_{1a} + 8,9-Z-isomer	Abamectin B_{1b} + 8,9-Z-isomer	
	rency					
Kernan, CA 2008	Sweet, Brooks	BBCH 75–85	7 14 21 28 35	0.005 0.005 <u>0.006</u> (0.007, 0.004) 0.006 0.003	< 0.002 < 0.002 < 0.002 (2) < 0.002 < 0.002	T005601-07; W30CA081083
Hollister, CA 2008	Sweet, Bing	BBCH 75–85	21	<u>0.003</u> (0.002, 0.004)	< 0.002 (2)	T005601-07; W27CA081084
Ephrata, WA 2008	Sweet, Bing	BBCH 69–75	21	0.005	< 0.002	T005601-07; W18WA081085
Ephrata, WA 2008	Sweet, Bing	BBCH 69–75	21	<u>0.009</u> (0.008, 0.010)	< 0.002 (2)	T005601-07; W18WA081086

Peaches

Twelve supervised residue trials were conducted on <u>peaches</u> in Europe during 2002 and 2003. Samples were analysed by LC-MS/MS. Peach samples were stored deep-frozen for a maximum of 13 months (407 days). Seventeen supervised residue trials were conducted on peaches in the USA during 1998 and 2008. Samples were analysed either by HPLC/FL (1998 trials) or LC-MS/MS (2008 trials). Peach whole fruit samples were stored deep-frozen for a maximum of 15.2 months. Residue data from supervised trials on peaches are summarized in Table 55.

Table 55 Results from supervised trials conducted in Europe with abamectin in peaches

Country	Peach variety	Application rate, g ai/ha	Growth stage	DAT, days	Crop Part	Residues, mg/kg			Study, trial
						Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
France 2001	Dolores/GF 677	14, 13	79–81	0 0 14 14	pulp w/ fruit pulp w/ fruit	0.033 0.031 0.006 (2) 0.006 (2)	< 0.002 < 0.002 < 0.002 (2) < 0.002 (2)	0.0022 0.0021 < 0.002 (2) < 0.002 (2)	1077/01; Roquecourbe
France 2001	July lady	2× 14	79–85	0 0 14 14	pulp w/ fruit pulp w/ fruit	0.043 0.041 0.003, 0.006 0.002, 0.006	< 0.002 < 0.002 < 0.002 (2) < 0.002(2)	0.003 0.003 < 0.002 (2) < 0.002 (2)	1078/01
France 2001	Fidelia/GF 677	2× 13	78–81	0 0 3 3 7 7 10 10 14 14	pulp w/ fruit pulp w/ fruit pulp w/ fruit pulp w/ fruit pulp w/ fruit	0.036 0.031 0.018 0.016 0.006 0.005 0.003 0.003 0.003 (2) 0.003 (2)	< 0.002 < 0.002 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (2) < 0.002 (2)	0.003 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (2) < 0.002 (2)	1079/01; Roquecourbe

Country	Peach variety	Application rate, g ai/ha	Growth stage	DAT, days	Crop Part	Residues, mg/kg			Study, trial
						Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
France 2001	Pavie: Andross	2× 14	70–76	0 0 3 3 7 7 10 10 14 14	w/ fruit pulp pulp w/ fruit pulp w/ fruit pulp w/ fruit pulp w/ fruit	0.013 0.014 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (3)	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (3)	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (3)	1080/01 Trial: 1–Vauvert
France 2002	Symphonie	2× 20	77–78	0 0 14 14	pulp w/ fruit pulp w/ fruit	0.024 0.021 0.004 0.004	< 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002	02-1145; Twissac
France 2002	Bienvenue	20, 21	75–78	0 0 14 14	pulp w/ fruit pulp w/ fruit	0.031 0.028 0.006 0.006	< 0.002 < 0.002 < 0.002 < 0.002	0.002 < 0.002 < 0.002 < 0.002	02-1146; St. Sardos
France 2002	Royal Glori	20, 22	75–85	0 0 3 3 7 7 14 14	pulp w/ fruit pulp w/ fruit pulp w/ fruit pulp w/ fruit	0.040 0.035 0.021 0.019 0.018 0.016 0.007 0.006	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	0.003 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	02-1147; Meauzac
Italy 2002	Elegant lady	2× 21	75–77	0 0 3 3 7 7 14 14	pulp w/ fruit pulp w/ fruit pulp w/ fruit pulp w/ fruit	0.014 0.012 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	02-1148 Trial: 1–Tintoria
Italy 2003	Maria Bianca	2× 20	77–81	0 14 0 14	pulp pulp w/ fruit w/ fruit	0.010 < 0.002 0.009 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002	03-5075

Country	Peach variety	Application rate, g ai/ha	Growth stage	DAT, days	Crop Part	Residues, mg/kg			Study, trial
						Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
Italy 2003	Elegant Lady	2× 20	75–77	0 3 7 10 14 0 3 7 10 14	pulp pulp pulp pulp pulp w/ fruit w/ fruit w/ fruit w/ fruit w/ fruit	0.039 0.004 < 0.002 < 0.002 < 0.002 0.036 0.004 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	03-5076
Spain 2003	Calanda	2× 20	77–81	0 14 0 14	pulp pulp w/ fruit w/ fruit	0.019 0.006 0.018 0.006	< 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002	03-5073
Spain 2003	Carson	21, 20	74–81	0 3 7 10 14 0 3 7 10 14	pulp pulp pulp pulp pulp w/ fruit w/ fruit w/ fruit w/ fruit w/ fruit	0.032 0.015 0.010 0.006 0.005 0.029 0.014 0.009 0.006 0.005	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	03-5074
USA, GA 1998	Summer Gold	2× 26	maturing	14	w/ fruit	0.005, 0.006	included	< 0.002 (2) [a]	161-98; OW-IR-836-98/GA
USA Fresno, CA 1998	Fay Elberta	2× 26	immature	0 2 6 9 15 19 22 29	w/ fruit	0.010 (2) 0.007, 0.004 0.004, 0.006 0.006, 0.004 < 0.002, 0.006 0.003 (2), 0.002 (0.002 (3), 0.003) < 0.002 (4)	included	< 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (4) < 0.002 (4) [a]	161-98; 02-IR-023-98/CA
USA Madera, CA 1998	Camival	2× 26	small green	21	w/ fruit	< 0.002 (2)	included	< 0.002 (2) [a]	161-98; OW-IR-106-98/CA
USA Butte, CA, 1998	Loadels	2× 26	develop	21	w/ fruit	0.006 (< 0.002, 0.009)	included	< 0.002 (2) [a]	161-98; OW-IR-432-98/CA
		2× 26	develop	21	w/ fruit	0.007, < 0.002	included	< 0.002 (2) [a]	

Country	Peach variety	Application rate, g ai/ha	Growth stage	DAT, days	Crop Part	Residues, mg/kg			Study, trial
						Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
USA SC, 1998	Contender	2× 26	1.5–2 in. diam. .	21	w/ fruit	0.002 (< 0.002 (2), 0.003, 0.002)	included	< 0.002 (4) [a]	161-98; OS-IR-607-98/SC
		2× 26		21	w/ fruit	< 0.002 (4)	included	< 0.002(2) [a]	
USA NC, 1998	Bell of Georgia	2× 26	2.3 in. diam.	21	w/ fruit	< 0.002 (2)	included	< 0.002(2) [a]	161-98; OS-IR-608-98/NC
USA Michigan 1998	Elberta	2× 26	immature	21	w/ fruit	< 0.002 (2)	included	< 0.002(2) [a]	161-98; NE-IR-705-98/MI
		2× 26	immature	21	w/ fruit	0.004 (0.005, < 0.002)	included	< 0.002(2) [a]	
USA Pensilvania 1998	Redskin	2× 26	1.5–3 in. diam.	22	w/ fruit	0.005 (2)	included	< 0.002(2) [a]	161-98; NE-IR-602-98/PA
		2× 26	1.5–3 in. diam.	22	w/ fruit	0.002, 0.003	included	< 0.002(2) [a]	
USA Texas, 1998	Florida King	2× 26	ripening	14	w/ fruit	0.08, 0.020	included	< 0.002(2) [a]	161-98; OS-IR-204-98/TX
		2× 26	ripening	14 21	w/ fruit	0.038, 0.033 0.024	included	0.004, 0.003 0.002 [a]	
USA Pennsylvania 1998	Glen Glow	2× 26	3–5 cm diam.	21	w/ fruit	0.002 (< 0.002, 0.002)	included	< 0.002 (2)	T005601-07; E04PA081087
USA Montezuma, GA 2008	Flame Prince	2× 26	69–76	21	w/ fruit	0.002 (< 0.002, 0.002)	included	< 0.002(2)	T005601-07; E19GA081088
USA Montezuma, GA 2008	MarQueen	2× 26	69–76	21	w/ fruit	0.003 (< 0.002, 0.004)	included	< 0.002 (2)	T005601-07; E19GA081089
USA Montezuma, GA 2008	Faye Elberta	2× 26	69–76	21	w/ fruit	0.003, 0.002	included	< 0.002 (2)	T005601-07; E19GA081090
USA Wisconsin, 2008	Redskin	2× 26	–	21	w/ fruit	0.006 (0.006, 0.005)	included	< 0.002 (2)	T005601-07; E19WI081091
USA Madera, CA 2008	Springcrest	2× 26	73	7 14 21 28 35	w/ fruit	0.009 0.005 0.002 (2) < 0.002 < 0.002	included	< 0.002 < 0.002 < 0.002 (2) < 0.002 < 0.002	T005601-07; W29CA081093
USA, Fresno, CA 2008	Autumn Red	2× 26	75–81	21	w/ fruit	0.004 (0.004, 0.003)	included	< 0.002 (2)	T005601-07; E19CA081094
USA Sanger, CA 2008	September Sun	2× 26	75–82	21	w/ fruit	0.008 (0.009, 0.007)	included	< 0.002 (2)	T005601-07; E19CA081095

[a] Include the 8,9-Z-isomer of avermectin B_{1b}

Plums

Seventeen supervised residue trials were conducted on plums in the USA during 1996, 1997 and 2008. Samples were analysed either by HPLC-FL (1996/97 trials) or LC-MS/MS (2008 trials). Plum samples were stored deep-frozen for a maximum of 6.5 months (198 days). Residue data from supervised trials on plum are summarized in Table 56.

Table 56 Results of supervised residue trials conducted with abamectin in USA on plums

Location year	Variety	Application rate, g ai/ha	Growth Stage	DAT, days	Residue Found (mg/kg)		Study; trial
					Avermectin B_{1a}+ 8,9-Z-isomer	Avermectin B_{1b}	
Fresno, CA 1996	French	2× 27	colouring harvest	0 14 21	0.015 (2) 0.003, 0.004 0.004 (< 0.002 (2), 0.006, 0.004)	< 0.002 (2) < 0.002 (2) < 0.002 (4) [a]	ABR-98073; 001-96-4011R
Tulare, CA 1996	French Myro-29 Rootstock	2× 27	colouring mature	0 14 21	0.009, 0.012 < 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2) < 0.002 (2) [a]	ABR-98073; 001-96-4012R
Yolo, CA 1996	French Moraslin Rootstock	2× 27	immature 60% mature	0 14 21	0.015, 0.018 < 0.002 (2) 0.002 (< 0.002, 0.003)	< 0.002 (2) < 0.002 (2) < 0.00 (2) [a]	ABR-98073; 001-96-4013R
Stanislaus, CA 1996	Plum (French)	2× 27	immature near mature	0 14 21	0.011, 0.017 < 0.002, 0.005 < 0.002 (2)	< 0.002 (2) < 0.002 (2) [a]	ABR-98073; 001-96-4014R
Michigan 1997	Stanley	2× 27	immature	0 14 21	0.025, 0.018 0.005 (2) 0.004 (0.003, 0.005)	< 0.002 (2) < 0.002 (2) < 0.002 (2) [a]	ABR-98073; 01-IR-001-97
Fresno, CA 1997	Angelano	2× 27	immature–mature	0 14 21	0.010, 0.010 0.003, 0.008 0.004 (0.003, 0.005)	< 0.002 (2) < 0.002 (2) < 0.002 (2) [a]	ABR-98073; 01-IR-002-97
Fresno, CA 1997	Friar	2× 27	near maturity mature	0 14 21	0.002, < 0.002 0.003, < 0.002 < 0.002 (2)	< 0.002 (2) < 0.002 (2) < 0.002 (2) [a]	ABR-98073; 01-IR-005-97
Washington 1997	Friar	2× 27	green fruit	0 14 21	0.008, 0.012 0.009, 0.003 0.004 (< 0.002, 0.005)	< 0.002 (2) < 0.002 (2) < 0.002 (2) [a]	ABR-98073; 01-IR-003-97
Oregon 1997	Italian	2× 27	colouring to sweeten	0 14 21	0.008 (2) < 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2) < 0.002 (2) [a]	ABR-98073; 01-IR-004-97
Wisconsin, 2008	Early Golden	2× 26	-	21	0.003 (0.004, 0.002)	< 0.002 (2)	T005601-07; E19WI081096
Hughson, CA, 2008	French Plum	2× 26	77, 81	21	0.004 (0.005, 0.003)	< 0.002 (2)	T005601-07; W26CA081097
		129, 131	77, 81	21	0.010, 0.030	< 0.002 (2)	
Hickman, CA, 2008	Grand Rosa	2× 26	77 81	21	< 0.002 (2)	< 0.002 (2)	T005601-07; W26CA081098
Fresno, CA, 2008	Flavor Rich	26, 25	77 81	7 14 21 28 35	< 0.002 < 0.002 0.004 (0.005, < 0.002) < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 (2) < 0.002 < 0.002	T005601-07; W30CA081099
Kerman, CA, 2008	French Prune	2× 26	73, 77	21	< 0.002 (2)	< 0.002, < 0.002	T005601-07; W29CA081100
		2× 131	73, 77	21	0.003 (2)	< 0.002, < 0.002	
Oregon 2008	Italian	26, 27	76, 81	21	< 0.002 (2)	< 0.002, < 0.002	T005601-07; W21OR081101

[a] Includes 8,9-z isomer of avermectin B_{1b}

Raspberries

Four supervised residue trials were conducted on raspberries in Italy in 2004, two open field trials and two trials in open tunnels. Samples of raspberries were stored deep-frozen for a maximum of 7.2 months (218 days). Samples were analysed by LC-MS/MS detection, with only abamectin B_{1a}being analysed. Residue data from supervised trials on raspberries are summarized in Table 57.

Table 57 Results of supervised residue trials conducted with abamectin in on raspberry in Italy in 2004

Location, method	Raspberry variety	Application rate, g ai/ha	DAT, days	Residues, mg/kg Avermectin B_{1a}	Study, trial
Pergine Valsugana, field	Eritage	20.25	7	< 0.02	AGRI 023/04 GLP HAR, GLP 011-04-sm
Frassilongo, field	Eritage	20.25	7	0.02	AGRI 023/04 GLP HAR, GLP 012-04-sm
Balsega di Pine, oppen tunnel	K Polka	20.25	0 3 7 10 14	0.10 0.02 <u>< 0.02</u> < 0.02 < 0.02	AGRI 024/04 DEC, GLP 009-04-sm
Pergine Valsugana, open tunnel	Eritage	20.25	0 3 7 10 14	0.12 0.04 <u>0.03</u> < 0.02 < 0.02	AGRI 024/04 DEC, AGRI 010-04-sm

Strawberries

Eight supervised residue trials were conducted on protected strawberries in Europe during 1999, 2003 and 2004. Samples of strawberries were stored deep-frozen for a maximum of 12 months. Samples were analysed by HPLC-FL or LC-MS/MS. Twenty-eight supervised residue trials were conducted on strawberries in the USA during 1988, 1989, 2007/08 and 2010, protected strawberries or on open-field strawberries. Samples of strawberries were stored deep-frozen for a maximum of 8 months. Samples of the 1988/1989 trials were analysed by HPLC-FL and samples of the 2007–2010 trials were analysed by LC-MS/MS. Residue data from supervised trials on strawberries are summarized in Table 58.

Table 58 Results of supervised residue trials conducted with abamectin on strawberries in Europe and the USA under protected or field conditions

Country, year	Strawberry variety	Application rate, g ai/ha	DAT, days	Residues, mg/kg			Study; trial
				Avermectin B_{1a}	Avermectin B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
France, protected 1999	Selva	22, 23, 22	3	<u>0.071</u> (0.069, 0.073)	included	0.003, 0.003 [a]	0030501; Fontaines de Sologne
France protected 1999	Selva	3× 22	3	<u>0.020</u> (0.022, 0.018)	included	< 0.002 (2) [a]	0030502 Cheverny
France, protected 1999	Selva	23, 23, 24	0 1 2 3	0.072 0.057 0.041 <u>0.045</u>	included included included included	0.003 0.002 0.002 0.002 [a]	0030401 Courmemin

Country, year	Strawberry variety	Application rate, g ai/ha	DAT, days	Residues, mg/kg			Study; trial
				Avermectin B_{1a}	Avermectin B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
France, protected 2003	Diamante	23, 24, 23	0 1 3 7 9	0.029 0.020 <u>0.014</u> 0.010 0.008	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	0.002 < 0.002 0.002 < 0.002 < 0.002	03-5066
France protected 2004	Guariguette	24, 22, 23	0 1 3 8 10	0.054 0.045 <u>0.034</u> 0.023 0.017	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	0.003 0.002 < 0.002 < 0.002 < 0.002	03-5085
France, protected 2004	Campsas	3× 23	0 1 3 7 10	0.068 0.048 <u>0.042</u> 0.024 0.019	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	0.004 0.002 0.002 < 0.002 < 0.002	03-5086
Spain, protected 1999	Camarosa	4× 22	0 0 3	0.040 0.036 <u>0.006</u> (0.005, 0.006)	< 0.002 < 0.002 < 0.002 (2)	0.002 0.002 < 0.002 (2)	1112/99 Bonares
Spain, protected 1999	Camarosa	4× 22	0 3	0.038, 0.039 <u>0.004</u> (2)	< 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	1113/99 Palos de la Frontera
USA Protected 1988	Chandler	4× 22	0 1 2 3 7	0.010 (2), 0.012, 0.018 0.014, 0.011 (2), 0.015 0.008, 0.009, 0.010, 0.011 <u>0.007</u> (0.006, 0.008 (2), 0.005) < 0.005 (3), < 0.002	included	< 0.002 (4) < 0.002 (4) < 0.002 (4) < 0.002 (4) < 0.002 (4) [a]	618.936 FSS; 001-88-1027R
		4× 45	0 1 2 3 7	0.045, 0.049 0.036, 0.039 0.046, 0.045 (2), 0.033 0.033 (2), 0.042, 0.027 0.024, 0.021 (2), 0.019 0.015, 0.010, 0.007, 0.009	included	< 0.005 (4) < 0.005 (4) < 0.005 (4) < 0.005 (4) < 0.002 (4) [a]	
USA, Protected 1988	Pajaro	4× 22	0 1 2 3 7	0.024, 0.022 (2), 0.025 0.016, 0.015, 0.013, 0.012 0.008 (2), 0.012, 0.010 <u>0.006</u> (< 0.002, 0.008 (3)) < 0.005 (4)	included	< 0.005 (4) < 0.002 (4) < 0.002 (4) < 0.002 (4) < 0.002 (4) [a]	618.936 FSS; 001-88-6020R
		4× 45	0 1 2 3 7	0.045, 0.053 0.047, 0.041 0.040, 0.029 0.037, 0.034 0.022, 0.020 (2), 0.019 0.020, 0.025, 0.026, 0.023 0.006, 0.007 (2), < 0.005	included	0.0050, 0.0051 < 0.005 (4) < 0.005 (4) < 0.005 (4) < 0.002 (4) [a]	
USA, protected 1988	Selva	4× 22	0 3	0.013, 0.015 0.012, 0.010 <u>0.008</u> (0.005, 0.012	included	< 0.002 (4) < 0.002 (4)	618.936 FSS; 001-88-6021R

Country, year	Strawberry variety	Application rate, g ai/ha	DAT, days	Residues, mg/kg			Study; trial
				Avermectin B_{1a}	Avermectin B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
				0.009, 0.006)			
USA, protected 1989	Chandler	4× 21	0 2 3	0.018 (2), 0.022 0.017, 0.019, 0.020, 0.014, 0.015 0.008 (3), 0.007 (2), 0.006 (2), 0.010 <u>0.005</u> (< 0.005 (2), 0.006 (3), 0.005 (2), 0.008)	included	< 0.005, < 0.002 (7) < 0.002 (8) < 0.002 (8) [a]	618.936 FSS; 001-89-1007R
USA, protected 1989	Selva	4× 22	0 2 3	0.008, 0.009 0.006 (2) < 0.005 (4) <u>0.005</u> (< 0.005 (3), 0.0052)	included	< 0.002 (4) < 0.002 (4) < 0.002 (4) [a]	618.936 FSS; 001-89-6003R
USA, San Diego, CA field 1988	Douglas	4× 22	0 1 2 3 7	0.020, 0.015 0.016, 0.018 0.018, 0.012 0.008 (2) 0.009 (2), 0.006 (2) <u>0.006</u> (< 0.005 (2), 0.006, 0.009) < 0.005(2), < 0.002 (2)	included	< 0.002 (4) < 0.005, < 0.002 (3) < 0.002 (4) < 0.002 (4) < 0.002 (4) [a]	618.936 FSS; 001-88-1026R
		4× 45	0 1 2 3 7	0.049 (2), 0.048 0.038 0.024, 0.044 0.040, 0.039 0.035, 0.025 0.020, 0.027 0.015 (2), 0.018, 0.022 0.006, 0.007 (2), 0.009	included	0.005 (2), < 0.005 (2) < 0.005 (4) < 0.005 (2), < 0.002 (2) < 0.002 (3), < 0.005 < 0.002 (4) [*]	
USA, Hillsborough, FL field 1989	Pajaro	4× 22	0 3	0.031 (2), 0.024 0.026 <u>0.006</u> (0.006 (3), 0.007)	included	< 0.005 (4) < 0.002 (4) [a]	618.936 FSS; 001-89-0004R
		4× 45	0 3	0.057, 0.079 0.076, 0.068 0.021, 0.017 0.008, 0.020	included	0.007, 0.010, 0.009, 0.008 < 0.005 (2), < 0.002 (2) [a]	
USA, Hillsborough, FL field 1989	Selva (large)	4× 22	0 3	0.032, 0.024 0.030, 0.036 <u>0.006</u> (0.006, 0.005 0.008, 0.006)	included	< 0.005 (4) < 0.002 (4) [a]	618.936 FSS; 001-89-0005R
		4× 45	0 3	0.063, 0.052 0.057, 0.071 0.017, 0.010 0.021, 0.018	included	0.009, 0.007 0.008, 0.010 < 0.002 (2), < 0.005 (2) [a]	
USA, Hillsborough, FL field 1989	Selva	4× 22	0 2 3	0.014 (2), 0.025, 0.015 < 0.005 (8) <u>< 0.005</u> (8)	included	< 0.005 (2) < 0.005 (2) < 0.005 (8), < 0.005 (8) [a]	618.936 FSS; 001-89-0024R
USA, Berrien, MI field 1989	All Star	5× 22	0 2 3	0.0050, < 0.005 (3) < 0.005 (4) <u>< 0.005</u> (4)	included	< 0.005 (4) < 0.005 (4) < 0.005 (4) [a]	618.936 FSS; 001-89-1018R
USA, Berrien,	Jewell	5× 22	0	< 0.005 (2)	included	< 0.005 (4)	618.936 FSS;

Country, year	Strawberry variety	Application rate, g ai/ha	DAT, days	Residues, mg/kg			Study; trial
				Avermectin B_{1a}	Avermectin B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
MI field 1989			 2 3	0.006, 0.012 < 0.005 (4) <u>< 0.005</u> (4)		 < 0.005 (4) < 0.005 (4) [a]	001-89-1019R
USA, Washington, OR field 1989	Benton	4× 22	0 2 3	0.024, 0.025 0.028, 0.029 0.014, 0.012 0.008, 0.014 <u>0.009</u> (0.011, 0.008 (3))	included	< 0.005 (4) < 0.005 (4), < 0.005 (4) [a]	618.936 FSS; 001-89-1020R
USA, Marion, OR field 1989	Benton	2× 22	0 2 3	0.006 (3), 0.011, < 0.005 (4) <u>< 0.005</u> (2)	included	< 0.005 (4) < 0.005 (4) < 0.005 (4) [a]	618.936 FSS; 001-89-1021R
USA, Lehigh, PA field 1989	Earliglow	4× 22	0 2 3	0.007, 0.010 0.014, 0.013 < 0.005 (4) < 0.005 (4)	included	< 0.005 (4) < 0.005 (4) < 0.005 (4) [a]	618.936 FSS; 001-89-3004R
USA, Lehigh, PA field 1989	Guardian	4× 22	0 2 3	0.008 (2), 0.015, 0.013 < 0.005 (4) < 0.005 (4)	included	< 0.005 (4) < 0.005 (4) < 0.005 (4) [a]	618.936 FSS; 001-89-3005R
USA, PA field 2008	Allstar	4× 21	3	<u>0.009</u> (0.010, 0.008)	included	< 0.002 (2)	T001870-07; E04PA078370
USA, FL field 2008	Camerosa	4× 21	3	<u>0.010</u> (0.013, 0.008)	included	< 0.002 (2)	T001870-07; E14FL078371
USA, MI field 2008	Annapolis	4× 22	3	<u>0.016</u> (0.009, 0.011, 0.015, 0.031)	included	< 0.002 (3), 0.003	T001870-07; C01MI078372
USA, Sta Maria, CA field 2007/08	Albion	2× 21 2× 22	0 1 3 5	0.16 0.046 <u>0.026</u> (0.023, 0.034, 0.032, 0.020, 0.024, 0.025) 0.020	included	0.012 0.004 0.002 (2), 0.003 (3), < 0.002 < 0.002	T001870-07; W27CA078373
USA, Aromas, CA field 2007	Raritan	4× 21	3	<u>0.028</u> (0.020, 0.030, 0.036, 0.026, 0.028, 0.027)	included	< 0.002 (2), 0.003 (3), 0.004	T001870-07; W27CA078374
USA, OR field2008	Selva	4× 21	3	<u>0.006</u> (0.004, 0.009)	included	< 0.002 (2)	T001870-07; W21OR078375
USA, NC field 2010	Camino Real	4× 21	3	0.020 (2)	included	< 0.002 (2)	T001870-07; E10-0001
USA, CA field 2010	Albion	4× 21	3	0.010 (2)	included	< 0.002 (2)	T001870-07; W33-0002

[a] Includes the 8,9-z isomer of avermectin B_{1b}

Grapes

Twenty-four supervised residue trials were conducted on <u>grapes</u> in the USA during 1994, 1995 and 2008. Samples of grapes were stored deep-frozen for a maximum of ≤ 28 months. Samples were analysed using method 936-94-4, method M-073.1 and/or Meth-192, rev.2. Residue data from supervised trials on grapes are summarized in Table 62.

Table 59 Results of supervised residue trials conducted with abamectin in USA on grapes

Region Year	Grape variety	Application rate, g ai/ha	DAT, days	Residues, mg/kg		Study; trial
				Avermectin B_{1a} + 8,9-Z-isomer	Avermectin B_{1b}	
Coachela, CA 1994	T	2× 21	0 7 14 28 42	0.043, 0.030 0.007, 0.010 0.003, 0.010 0.004 (0.005, 0.004) 0.003, 0.004	0.005, 0.003 < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) [a]	618-244-94036; 001-94-1009R
Granger, WA 1994	White Reisling	22, 21	0 7 14 28 42	0.022, 0.039 0.004, 0.003 0.003, 0.002 0.002 (0.002, < 0.002) 0.002, < 0.002	0.002, 0.004 < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) [a]	618-244-94036; 001-94-1010R
Phelps, NY 1994	Catawba	21, 22	0 7 14 28 42	0.041, 0.047 0.003 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2)	0.005 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) [a]	618-244-94036; 001-94-2002R
Comstock Park, MI 1994	Concord	2× 21	0 7 14 28 42	0.038, 0.036 < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2)	0.004 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) [a]	618-244-94036; 001-94-2003R
Ceres, CA 1994	French Columbard	2× 21	0 7 14 28 42	0.018, 0.024 0.004 0.004, 0.006 0.006 (0.005, 0.007) 0.006, 0.005	0.002, 0.003 < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) [a]	618-244-94036; 001-94-5004R
Biola, CA 1994	T	2× 21	0 7 14 25 28 42	0.020, 0.023 0.005, 0.007 0.004 (2) 0.010 0.002 (0.003, < 0.002) < 0.002 (2)	0.002 (2) < 0.002 (2)) < 0.002 (2) < 0.002 < 0.002 (2) < 0.002 (2)	618-244-94036; 001-94-5006R
Georg, WA 1995	Reisling	2× 21	0 28	0.021 (2) < 0.002 (2)	0.002 (2) < 0.002 (2) [a]	618-244-94036; 001-95-1005R
Orefield, PA 1995	Niagara	2× 21	0 28	0.016, 0.029 < 0.002 (2)	0.002, 0.003 < 0.002 (2) [a]	618-244-94036; 001-95-2008R
Lodi, CA 1995	Flame Tokay	21, 20	0 28	0.029, 0.015 < 0.002 (2)	0.003, < 0.002 < 0.002 (2) [a]	618-244-94036; 001-95-5003R
Calistoga, CA 1995	Cabenet Sauvignon	2× 21	0 28	0.016, 0.014 < 0.002 (4)	< 0.002 (2) < 0.002 (2) [a]	618-244-94036; 001-95-5009R
Gonzales, CA 1995	Chardonnay	2× 21	0 28	0.043, 0.057 0.002 (< 0.002, 0.003)	0.006, 0.004 < 0.002 (2) [a]	618-244-94036; 001-95-5010R
Biola, CA 1995	Thompson Seedless	2× 21	0 28	0.034, 0.025 < 0.002 (2)	0.004, 0.003 < 0.002 (2) [a]	618-244-94036; 001-95-5011R
Escalon, CA 1995	Carignane	2× 21	0 28	0.008, 0.009 < 0.002 (2)	< 0.002 (2) < 0.002 (2) [a]	618-244-94036; 001-95-5025R
Dundee, NY 2008	Concord	2× 22	28	< 0.002 (2)	< 0.002 (2)	T005598-07; E03NY081041
		2× 107	28 28	0.006, 0.010 0.005, 0.007, 0.004	< 0.002 (2) < 0.002 (3)	
Dundee, NY	Concord	21, 22	28	< 0.002 (2)	< 0.002 (2)	T005598-07;

Region Year	Grape variety	Application rate, g ai/ha	DAT, days	Residues, mg/kg		Study; trial
				Avermectin B_{1a} + 8,9-Z-isomer	Avermectin B_{1b}	
2008						E03NY081042
Hugson, CA 2008	Thompson Seedless	21, 22	28	0.004 (0.003, 0.004)	< 0.002 (2)	T005598-07; W26CA081043
		106, 108	28 28	0.044, 0.069 0.043, 0.052, 0.043	< 0.002 (2) < 0.002 (2), 0.002	
Madera, CA 2008	Thompson Seedless	22, 21	14 21 28 32 35	0.007 0.004 0.003 (3) 0.004 < 0.002	< 0.002 < 0.002 < 0.002 (2) < 0.002 < 0.002	T005598-07; W29CA081044
Fresno, CA 2008	Merlot	2× 21	28	< 0.002 (2)	< 0.002 (2)	T005598-07; E19CA081045
Fresno, CA 2008	Cabernet Sauvignon	22, 21	28	0.002 (0.002, < 0.002)	< 0.002 (2)	T005598-07; E19CA081046
Selma, CA 2008	Ruby Reds	2× 21	28	< 0.002 (2)	< 0.002 (2)	T005598-07; E19CA081047
Ephrata, WA 2008	Riesling	2× 21	28	0.003, < 0.002	< 0.002 (2)	T005598-07; W18WA081048
Ephrata, WA 2008	Chardonnay	2× 21	28	0.006 (0.003, 0.010)	< 0.002 (2)	T005598-07; W18WA081049

[a] Includes the 8,9-z isomer of avermectin B_{1b}

Avocadoes

Five supervised residue trials were conducted on avocadoes in the USA during 1999. Avocado samples were stored deep-frozen for a maximum of 3.8 months (116 days) and analysed by HPLC-FL. Residue data from supervised trials on avocado are summarized in Table 60.

Table 60 Results from supervised trials conducted with abamectin on avocadoes in USA (Study 871-99)

Location	Avocado variety	Application rate, g ai/ha	DAT, days	Residues, mg/kg		Trial
				Avermectin B_{1a} + 8,9-Z-isomer	Avermectin B_{1b} + 8,9-Z-isomer	
Santa Paula, CA	Hass	27, 28	14	0.004 (0.003, 0.006)	< 0.002 (2)	07198.99-CA120
Fallbrook, CA	Hass	26, 28	14	0.004 (< 0.002, 0.005)	< 0.002 (2)	07198.99-CA121
Valley Center, CA	Hass	26, 28	14	0.003 (2)	< 0.002 (2)	07198.99-CA122
Via Vaquero, CA	Hass	27, 25	14	0.007 (0.009, 0.005)	< 0.002 (2)	07198.99-CA135
Florida	Peterson	26, 27	14	< 0.002 (2)	< 0.002 (2)	07198.99-FL50

Mangoes

Five supervised residue trials were conducted on mangoes in Brazil during 2008/09 and 2009/10. Samples were stored deep-frozen for a maximum of 21 months and analysed by either HPLC-FL or LC-MS/MS. Residue data from supervised trials on mango are summarized in Table 61.

Table 61 Results from supervised trials conducted with abamectin on mangoes in Brazil 2008–2010

Location year	Mango variety	Application rate, g ai/ha	Growth stage BBCH	DAT, days	Residues, mg/kg			Study; trial
					Avermectin B_{1a}	Avermectin B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
RN, Mossoro 2008/2009	Tommy	4× 14	73–81	3 7 10	< 0.004 ≤ 0.004 < 0.004	included	< 0.0003 < 0.0003 < 0.00023	M09026; LZF
Minas Gerais 2009/2010	Palmer	4× 14	77- 87	3 7	0.003 0.003	< 0.002 < 0.002	< 0.001 < 0.001	M10046; LZF1

Location year	Mango variety	Application rate, g ai/ha	Growth stage BBCH	DAT, days	Residues, mg/kg			Study; trial
					Avermectin B_{1a}	Avermectin B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
				10	0.004	< 0.002	< 0.001	
RN, Mossoro 2009/2010	Tommy	4× 14	73–81	3 7 10	0.003 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002	< 0.001 < 0.001 < 0.001	M10046; -LZF2
RN, Barauna 2009/2010	Tommy Atkins	4× 14	73–81	3 7 10	0.003 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002	< 0.001 < 0.001 < 0.001	M10046; -LZF3
Sao Paulo 2009/2010	Palmer	4× 14	79–81	3 7 10	0.005 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002	< 0.001 < 0.001 < 0.001	M10046 -AMA

Papaya

Twelve supervised residue trials were conducted on papaya in Brazil during the growing seasons 2002, 2009/10 and 2011/12. Papaya (fruit) samples were stored deep-frozen for a maximum of 23 months and analysed by LC-MS/MS. Residue data from supervised trials on papaya are summarized in Table 62.

Table 62 Results from supervised trials conducted with abamectin on papaya in Brazil 2008/2009

Location, year	Papaya variety	Application rate, g ai/ha	Growth stage BBCH	DAT, days	Crop Part	Residues, mg/kg			Study; trial
						Avermectin B_{1a}	Avermectin B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
Linhares, ES 2002	Golden	2x23, 22, 24	61–89	0 3 3 3 7 7 7 10 10 10 14 14 14	Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit	0.028 0.031, 0.024 < 0.002 (2) 0.009, 0.011 0.016, 0.021 < 0.002 (2) 0.006, 0.007 0.011 < 0.002 0.004 0.009 < 0.002 0.004	< 0.002 0.004 (2) < 0.002 (2) 0.002 (2) < 0.002, 0.004 < 0.002 (2) < 0.002 (2) 0.002 < 0.002 < 0.002 0.002 < 0.002 < 0.002	0.002 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002, 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	02-1057
		46, 43, 44, 47	61–89	0 3 3 3 7 7 7 10 10 10 14 14 14	Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit	0.041 0.060, 0.065 0.002, < 0.002 0.020, 0.022 0.038, 0.039 < 0.002 (2) 0.014 (2) 0.029 < 0.002 0.010 0.024 < 0.002 0.009	0.002 0.006, 0.008 < 0.002 (2) 0.003 (2) 0.006 (2) < 0.002 (2) 0.003 (2) 0.005 < 0.002 0.0024 0.0061 < 0.002 0.0027	0.003 0.004 (2) < 0.002 (2) 0.002 (2) 0.003 (2) < 0.002 (2) < 0.002 (2) 0.0020 < 0.002 < 0.002 0.0020 < 0.002 < 0.002	
Itamara-ju, BA 2002	Golden	23, 22, 22, 22	61–89	0 3 3 3 7 7 7 10	Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit *Peel* *Pulp*	0.014 0.013, 0.011 < 0.002 (2) 0.005, 0.004 0.009 (2) < 0.002 (2) 0.004 (2) 0.005	< 0.002 0.002 (2) < 0.002 (2) < 0.002 (2) 0.002 (2) < 0.002 (2) < 0.002 (2) 0.002	< 0.002 < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002	02-1058

Location, year	Papaya variety	Application rate, g ai/ha	Growth stage BBCH	DAT, days	Crop Part	Residues, mg/kg			Study; trial
						Avermectin B_{1a}	Avermectin B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
				10 10 14 14 14	Fruit *Peel* *Pulp* Fruit	< 0.002 0.002 0.006 <u>< 0.002</u> <u>0.003</u>	< 0.002 < 0.002 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	
		46, 45, 47, 44	61–89	0 3 3 3 7 7 7 10 10 10 14 14 14	Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit	0.038 0.019, 0.017 < 0.002 (2) 0.007, 0.006 0.023, 0.017 < 0.002 (2) 0.008, 0.006 0.014 < 0.002 0.005 0.011 < 0.002 0.004	0.002 0.004, 0.003 < 0.002 (2) 0.002, < 0.002 0.005, 0.004 < 0.002 (2) 0.002, < 0.002 0.005 < 0.002 0.002 0.004 < 0.002 < 0.002	0.003 0.002, < 0.002 < 0.002 (2) < 0.002 (2) 0.002, < 0.002 < 0.002, 0.002 < 0.002 (2) < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	
Pinheiros, ES2002	Taiwan	22, 24, 21, 23	61–89	0 3 3 3 7 7 7 10 10 10 14 14 14	Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit	0.011 0.014, 0.016 < 0.002 (2) 0.005 (2) 0.007, 0.006 < 0.002 (2) 0.003 (2) 0.005 < 0.002 0.002 0.005 <u>< 0.002</u> <u>0.002</u>	< 0.002 0.003 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	02-1059
		44, 46, 44, 46	61–89	0 3 3 3 7 7 7 10 10 10 14 14 14	Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit	0.030 0.043, 0.036 < 0.002 (2) 0.013, 0.011 0.029, 0.033 < 0.002 (2) 0.008, 0.009 0.014 < 0.002 0.005 0.009 < 0.002 0.003	0.003 0.008, 0.007 < 0.002 (2) 0.003 (2) 0.006 (2) < 0.002 (2) 0.002 (2) 0.003 < 0.002 < 0.002 0.003 < 0.002 < 0.002	0.002 0.003 (2) < 0.002 (2) < 0.002 (2) 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	
Aracruz, ES 2002	Golden	21, 22, 22, 24	61–89	0 3 3 3 7 7 7 10 10 10 14 14 14	Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit	0.008 0.005, 0.006 < 0.002 (2) 0.002, 0.003 0.003 (2) < 0.002 (2) < 0.002 (2) 0.003 < 0.002 < 0.002 0.0024 <u>< 0.002</u> <u>< 0.002</u>	< 0.002 < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2)	< 0.002 < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2)	02-1060

Location, year	Papaya variety	Application rate, g ai/ha	Growth stage BBCH	DAT, days	Crop Part	Residues, mg/kg			Study; trial
						Avermectin B_{1a}	Avermectin B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
		44, 41, 44, 45	61–89	0 3 3 3 7 7 7 10 10 10 14 14 14	Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit *Peel* *Pulp* Fruit	0.018 0.015, 0.017 < 0.002 (2) 0.006 (2) 0.009 (2) < 0.002 (2) 0.004 (2) 0.009 < 0.002 0.004 0.007 < 0.002 0.003	0.002 0.004 (2) < 0.002 (2) 0.002 (2) 0.003 (2) < 0.002 (2) < 0.002 (2) 0.004 < 0.002 < 0.002 0.004 < 0.002 < 0.002	< 0.002 < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2)	
Sooretama, ES 2010	Golden	3× 22	51–84	0 0 3 3 5 5 7 7 10 10 14 14	*Pulp* Fruit *Pulp* Fruit *Pulp* Fruit *Pulp* Fruit *Pulp* Fruit *Pulp* Fruit	< 0.002 0.043 < 0.002 0.020 < 0.002 0.014 < 0.002 0.010 < 0.002 0.010 <u>< 0.002</u> <u>0.008</u>	< 0.002 0.006 < 0.002 0.006 < 0.002 0.005 < 0.002 0.004 < 0.002 0.005 < 0.002 0.004	< 0.001 0.005 < 0.001 0.003 < 0.001 0.002 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001	M10044; LZF1
Linhares, ES 2009/10	Golden	3× 22	51–84	0 0 3 3 5 5 7 7 10 10 14 14	*Pulp* Fruit *Pulp* Fruit *Pulp* Fruit *Pulp* Fruit *Pulp* Fruit *Pulp* Fruit	< 0.002 0.020 < 0.002 0.011 < 0.002 0.008 < 0.002 0.007 < 0.002 0.008 <u>< 0.002</u> <u>0.005</u>	< 0.002 0.004 < 0.002 0.004 < 0.002 0.003 < 0.002 0.003 < 0.002 0.003 < 0.002 0.003	< 0.001 0.002 < 0.001 0.002 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001	M10044; LZF2
Linhares, ES 2011/12	Golden	3× 22	71–81	0 3 5 7 10 14	Fruit	0.011 0.005 0.003 0.003 0.003 <u>0.003</u>	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.001 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001	M12047; MFG1
Jaguaré, ES 2011/12	Golden	3× 22	71–81	0 3 5 7 10 14	Fruit	0.027 0.009 0.009 0.007 0.006 <u>0.005</u>	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	0.003 < 0.001 < 0.001 < 0.001 < 0.001 < 0.001	M12047; MFG2

Bulb vegetables

Onions

Eight supervised residue trials were conducted on <u>onions</u> in the USA during 2000 to 2001. Onion bulb samples were stored deep-frozen for a maximum of 7 months and analysed by HPLC-FL. Summaries of the trial results are given in Table 63.

Table 63 Results from supervised trials conducted abamectin on onion bulbs in the USA in 2000/2001 (Study 07237)

Region	Onion variety	Application rate, g ai/ha	Growth Stage	DAT, days	Residue Found (mg/kg)			Trial
					Avermectin B_{1a}+ 8,9-Z-isomer	Avermectin B_{1b}+ 8,9-Z-isomer	Total residue	
California	Texas Grano Dry	22, 22, 21, 21	vegetative	30	< 0.002 (2)	< 0.002 (2)	< 0.004	00-CA69
Colorado	Teton	3× 21	vegetative	31	< 0.002 (2)	< 0.002 (2)	< 0.004	: 00-CO08
New Mexico	Starlite	22, 21, 21	Pre-bloom 8–10 leaves	29	< 0.002 (2)	< 0.002 (2)	< 0.004	00-NM12
New York	Quantum	22, 22, 23	6–8 leaves vegetative	29	0.02 (0.003, < 0.002)	< 0.002 (2)	0.004	00-NY02
Ohio	Burgos	21, 22, 22	vegetative	29	< 0.002 (2)	< 0.002 (2)	< 0.004	00-OH*03
Oregon	Santos Fl	3× 21	early maturity	29	< 0.002 (2)	< 0.002 (2)	< 0.004	00-OR14
Texas	Texas Early White	3× 22	1–3 in. diameter	31	< 0.002 (2)	< 0.002 (2)	< 0.004	00-TX07
Washington	Salem	21, 22, 22	vegetative—bulbing	29	< 0.002 (2)	< 0.002 (2)	< 0.004	00-WA*02

Leeks

Twelve supervised residue trials were conducted on leeks in Europe during 2000 to 2002. In all the trials, whole plant samples were analysed by LC-MS/MS) Leek samples were stored deep-frozen for a maximum of 11 months. Summaries of the trial results are given in Table 64.

Table 64 Results from supervised trials conducted with abamectin on leeks in Europe from 2000–2002

Country (year)	Leek variety	Application rate, g ai/ha	Growth Stage	DAT, days	Residue Found (mg/kg)			Study; trial
					Avermectin B_{1a}	Avermectin B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
France 2000	Porwitt	4× 9	BBCH 43–47	0 7	0.013 < 0.002 (2)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	0032201 Darvoy
France 2000	Albana	4× 9	BBCH 43–47	0 3 5 7 10	0.033 < 0.002 < 0.002 < 0.002 (2) < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 (2) < 0.002	0.002 < 0.002 < 0.002 < 0.002 (2) < 0.002	0032301 St Benoit sur Loire
France 2000	Azur	4× 9	BBCH 43–49	0 7	0.085 < 0.002 (2)	< 0.002 < 0.002 (2)	0.004 < 0.002 (2)	0032202 Marsillargues
France 2000	Amoundo	4× 9	BBCH 19–45	0 3 5 7 10	0.019 < 0.002 < 0.002 < 0.002 (2) < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 (2) < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 (2) < 0.002	0032302; St. Alban
France 2001	Schelton	4× 9	BBCH 401–408	0 7	0.024 < 0.002 (2)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	1069/01; Maslives
France 2001	Géant d'hiver	4× 9	BBCH 41–47	0 7	0.155 0.002 (0.003, < 0.002)	0.002 < 0.002 (2)	0.010 < 0.002 (2)	1070/01 Crest

Country (year)	Leek variety	Application rate, g ai/ha	Growth Stage	DAT, days	Residue Found (mg/kg)			Study; trial
					Avermectin B_{1a}	Avermectin B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
France 2001	Ginka	8 + 3× 9	BBCH 41–47	0 3 5 7 10	0.049 0.002 < 0.002 < 0.002 (2) < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 (2) < 0.002	0.005 < 0.002 < 0.002 < 0.002 (2) < 0.002	1071/01; Labergement les Auxonne
France 2001	Merido r	2× 10 2× 10	BBCH 42–46	0 3 5 7 10	0.073 0.002 < 0.002 < 0.002 (2) < 0.002	0.002 < 0.002 < 0.002 < 0.002 (2) < 0.002	0.005 < 0.002 < 0.002 < 0.002 (2) < 0.002	1072/01; Mauguio
Netherlands 2000	Alesia	4× 10	BBCH 43 - 48	0 7	0.016 < 0.002 (2)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	1119/00 Limburg
Netherlands 2000	Davina	4× 10	BBCH 43- 48	0 3 7 10 14	0.014 0.006 0.002 (2) < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 (2) < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 (2) < 0.002 < 0.002	1120/00 Elst
Netherlands 2001	Schelton	4× 9	50 cm	0 7	0.017 < 0.002 (2)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	1022/01; Etten Leur
Netherlands 2001	Roxton	10, 10, 9, 9	40 -60 cm	0 3 5 7 10	0.024 0.002 < 0.002 < 0.002 (2) < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 (2) < 0.002	0.002 < 0.002 < 0.002 < 0.002 (2) < 0.002	1021/01; TM Oud Gastel

Cucumber

Twenty nine supervised trials were carried out on protected cucumbers and gherkins in 1989–2002 and 2012 in Europe. Samples were stored deep-frozen for a maximum of 21 months and analysed by either by LC-MS/MS or HPLC-FL. Summaries of the trial results are given in Table 65.

Table 65 Results from protected supervised trials conducted with abamectin on cucumber and gherkins (two trials) in Europe

Country (year)	Cucumber variety	Application rate, g ai/ha	Growth stage BBCH	DAT, days	Residue Found (mg/kg)			Study; trial
					Avermectin B_{1a}	Avermectin B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
France 1991	Girola	4× 22	–	0 3 7	< 0.005 (2), 0.007, 0.005 < 0.005 (4) < 0.005 (4)	included	< 0.005 (4) < 0.005 (4) < 0.005 (4) [a]	HWI 6012/378; 066-91-0008R
France 1991	Vitalis	4× 22	–	0 3 7	< 0.009, 0.013, 0.008 (2) < 0.005 (4) < 0.005 (4)	included	< 0.005 (4) < 0.005 (4) < 0.005 (4) [a]	HWI 6012/378; 066-91-0009R

Country (year)	Cucumber variety	Application rate, g ai/ha	Growth stage BBCH	DAT, days	Residue Found (mg/kg)			Study; trial
					Avermectin B_{1a}	Avermectin B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
France 1991	Corona	4× 22	–	0 3 7	0.041, 0.035, 0.027, 0.036 0.025 (0.025, 0.026, 0.021, 0.029) 0.021, 0.014, 0.012 (2)	included	0.005, < 0.005 (3) < 0.005 (4) < 0.005 (4) [a]	HWI 6012/378; 066-91-0010R
Greece 2001	Aris	4× 21	61–89	0 3	0.012 0.004 (0.005, 0.002)	0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	1053/01; Kenourigio Locridos
Greece 2001	Deltastar	4× 21	61–89	0 3	0.006 < 0.005 (2)	< 0.002 < 0.002 (2)	0.002 < 0.002 (2)	1054/01; Kenourigio Locridos
Italy 1991	Darina	5× 22	–	0 3 7	< 0.005 (2) < 0.005 (2) < 0.005 (2)	included	< 0.005 (2) < 0.005 (2) < 0.005 (2) [a]	HWI-6012-374; 067-91-0001R
Italy 1991	Sprint F	5× 22	–	0 3 7	< 0.005 (2) < 0.005 (2) < 0.005 (2)	included	< 0.005 (2) < 0.005 (2) < 0.005 (2) [a]	HWI 6012-358; 067-91-0017R
Italy 2002	Akito	4× 22	64–71	–0 0 1 3 7	< 0.005 0.008 0.003 0.002 < 0.005	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	02-1144; Cerasolo ausa
Netherlands 1989	Corona	4× 22	–	0 1 3 7	0.013, 0.012, 0.011, 0.016 0.010, 0.008, 0.007, 0.011 0.007 (0.007 (2), 0.008, 0.006) 0.005, < 0.005 (3)	included	< 0.005 (4) < 0.005 (4) < 0.005 (4) < 0.005 (4) [a]	HLA-6012-322; 070-89-011R
Netherlands 1989	Ventura	4× 22	–	0 1 3 7	0.012, 0.009 (2), 0.008 0.010 (2), 0.008, 0.006 0.006 (0.007, < 0.005 (2), 0.006) < 0.005 (4)	included	< 0.005 (4) < 0.005 (4) < 0.005 (4) < 0.005 (4) [a]	HLA-6012-322; 070-89-012R
Netherlands 1990	Gherkin (Osiris)	5× 22	NR	0 1 3 7	< 0.005 (4) < 0.002 (4) < 0.002 (4) < 0.002 (4)	included	< 0.005 (4) < 0.005 (4) < 0.005 (4) < 0.005 (4) [a]	HLA-6012-322; 070-90-0010R
				0 1 3 5	< 0.005 (3), < 0.002, < 0.005, < 0.002 (3), < 0.005 (4) < 0.005 (4)	included	< 0.005 (4) < 0.005 (4) < 0.005 (4) < 0.005 (4) [a]	
Netherlands, 1998	Korinda	16, 18, 20, 20	fruiting	0 3	0.007, 0.003 0.002 (0.002, < 0.002)	< 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	1119/98; KN Pijnacker

Country (year)	Cucumber variety	Application rate, g ai/ha	Growth stage BBCH	DAT, days	Residue Found (mg/kg)			Study; trial
					Avermectin B_{1a}	Avermectin B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
Netherlands, 1998	Korinda	17, 18, 20, 20	fruiting	0 3	0.004, 0.004 0.003 (0.004, 0.002)	< 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	1120/98; AX Delfgauw
Netherlands, 1998	Korinda	4× 22	fruiting	0 3	0.004, 0.003 0.002 (0.003, 0.002)	< 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	1121/98; AX Delfgauw
Netherlands, 1998	Korinda	21 + 3× 22	fruiting	0 3	0.003, 0.002 < 0.002 (2)	< 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	1122/98; BE Delfgauw
Netherlands 2013	Venice	4× 22	60–79	–0 0 3 7	< 0.002 0.006 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002	S12-04361-01
Netherlands 2013	Euforia	2× 21 2× 22	60–79	–0 0 3 7	0.006 0.007 0.007 0.003	< 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002	S12-04361-02
Netherlands 2013	Carambole	2× 21 2x 22	60–79	–0 0 3 7	0.002 0.005 0.005 0.002	< 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002	S12-04361-03
Netherlands 2013	Hyjack	4× 21	60–79	–0 0 3 7	0.004 0.007 0.004 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002	S12-04361-04
Spain 1999	Darina	21, 2x 22	87–89	0 3	0.007 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	1106/99
Spain 1999	Darina	2× 21, 22	83–89	0 3	0.004, 0.005 < 0.002 (2)	< 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	1107/99
Spain 2000	Edona	3× 18, 20	87–89	0 3	0.004 0.002 (2)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	1040/00
Spain 2000	Edona	2× 18 2x 19	85–89	0 3	0.012 < 0.002 (2)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	1041/00
Spain 2001	Marumba	4× 22	85–87	0 3	0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	1048/01; Carchuna
Spain 2002	Borja	20, 3× 22	75–715	0 4	0.004 0.002 (0.003, < 0.002)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	02-1036; El Ejido
UK 1999	Brunex	5, 3× 6, 7, 9	–	0 4	0.005, 0.003 < 0.002 (2)	< 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	1035/99
UK 1999	Cumlaud	6, 7, 10, 10, 8, 8	–	0 3	0.0024, 0.0029 < 0.002 (2)	< 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	1036/99
UK 1999	–	4, 5, 8, 14, 16, 17	–	0 3	0.010 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	1037/99

Country (year)	Cucumber variety	Application rate, g ai/ha	Growth stage BBCH	DAT, days	Residue Found (mg/kg)			Study; trial
					Avermectin B_{1a}	Avermectin B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
UK 1999	Cumlaud	7, 6, 8, 12, 10, 16	–	0 3	0.002, < 0.002 < 0.002 (2)	< 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	1038/99

[a] Includes the 8,9-z isomer of avermectin B_{1b}

Melons

Thirteen supervised residue trials were conducted on protected melons in Europe during 2000 to 2002 and in 2008. Melon samples were stored deep-frozen for a maximum of 23 months and residues in peel and pulp analysed by LC-MS/MS. Residues in the whole fruit were calculated from residues in peel and pulp. Results from the supervised trials on protected melons in Europe are summarized in Table 66.

Table 66 Results from protected supervised trials conducted with abamectin on melons in Europe

Country (Year)	Melon variety	Application rate, g ai/ha	Growth stage (BBCH)	DAT, days	Crop Part	Residue Found (mg/kg)			Study; trial
						Avermectin B_{1a}	Avermectin B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
France 2000	Pancha	18, 2× 19, 20	55–89	0 3	fruit fruit	0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	0032401
France 2000	Lunastar	2× 18, 19	63–81	0 3	fruit fruit	0.004 < 0.002 (2)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	0032402
France 2002	Nastar	4× 18	71–74	0 0 0 3 3 3	peel pulp fruit peel pulp fruit	0.0058 < 0.002 0.003 0.002 (2) < 0.002 (2) < 0.002 (2)	< 0.002 < 0.002 < 0.002 < 0.002 (2) < 0.002 (2) < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 (2) < 0.002 (2) < 0.002 (2)	02-1028; Montalzat
France 2002	Cyrano	4× 18	71–87	0 0 0 3 3 3	peel pulp fruit peel pulp fruit	0.010 < 0.002 0.006 0.004 (2) < 0.002 (2) 0.002 (0.003, 0.002)	< 0.002 < 0.002 < 0.002 < 0.002 (2) < 0.002 (2) < 0.002 (2)	< 0.002 < 0.002 < 0.002 < 0.002 (2) < 0.002 (2) < 0.002 (2)	02-1029; Vazecar
France 2002	Escrito	4× 18	63–81	0 0 0 3 3 3	peel pulp fruit peel pulp fruit	0.004 < 0.002 0.002 0.002, < 0.002 < 0.002 (2) < 0.002 (2)	< 0.002 < 0.002 < 0.002 < 0.002 (2) < 0.002 (2) < 0.002(2)	< 0.002 < 0.002 < 0.002 < 0.002 (2) < 0.002 (2) < 0.002(2)	02-1030; Loriol du Comtat
France 2008	Darius	4× 22	71–74	–0 –0 –0 0 0 0 1 1 1 3 3 3 7 7 7	*peel* *pulp* fruit *peel* *pulp* fruit *peel* *pulp* fruit *peel* *pulp* fruit *peel* *pulp* fruit	< 0.002 < 0.002 < 0.002 0.007 < 0.002 0.004 0.008 < 0.002 0.005 0.004 < 0.002 0.003 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	CEMS-3917; S08-00835-01

Country (Year)	Melon variety	Application rate, g ai/ha	Growth stage (BBCH)	DAT, days	Crop Part	Residue Found (mg/kg)			Study; trial
						Avermectin B_{1a}	Avermectin B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
France 2008	Darius	22, 21	73, 74	–0 –0 –0 0 0 0 1 1 1 3 3 3 7 7 7	*peel* *pulp* fruit *peel* *pulp* fruit *peel* *pulp* fruit *peel* *pulp* fruit *peel* *pulp* fruit	< 0.002 < 0.002 < 0.002 0.005 < 0.002 0.003 0.003 < 0.002 0.003 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	CEMS-3917; S08-00835-01
France 2008	Anasta	21, 3× 22	65–85	–0 –0 –0 0 0 0 1 1 1 3 3 3 7 7 7	*peel* *pulp* fruit *peel* *pulp* fruit *peel* *pulp* fruit *peel* *pulp* fruit *peel* *pulp* fruit	0.007 < 0.002 0.004 0.008 < 0.002 0.005 0.004 < 0.002 0.003 0.003 <u>< 0.002</u> <u>0.003</u> 0.005 < 0.002 0.003	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	CEMS-3916; S08-0836-1
Germany 2008	Charantaise	21, 3× 22	74–88	–0 –0 –0 0 0 0 1 1 1 3 3 3 7 7 7	*peel* *pulp* fruit *peel* *pulp* fruit *peel* *pulp* fruit *peel* *pulp* fruit *peel* *pulp* fruit	0.003 < 0.002 0.002 0.013 < 0.002 0.005 < 0.002 < 0.002 < 0.002 0.01 <u>< 0.002</u> <u>0.005</u> 0.006 < 0.002 0.003	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	CEMS-3917; S08-00835-02
Italy 2008	Honeymoon	21, 3× 22	69–75	–0 –0 –0 0 0 0 1 1 1 3 3 3 7 7 7	*peel* *pulp* fruit *peel* *pulp* fruit *peel* *pulp* fruit *peel* *pulp* fruit *peel* *pulp* fruit	< 0.002 < 0.002 < 0.002 0.009 < 0.002 0.006 0.004 < 0.002 0.003 0.002 <u>< 0.002</u> <u>0.002</u> < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	CEMS-3916; S08-0836-2
Spain	Sanch	2× 17	61–89	0	fruit	< 0.002	< 0.002	< 0.002	02-1054;

Country (Year)	Melon variety	Application rate, g ai/ha	Growth stage (BBCH)	DAT, days	Crop Part	Residue Found (mg/kg)			Study; trial
						Avermectin B_{1a}	Avermectin B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
2002	o	2× 18		3 3 3	peel pulp fruit	< 0.002 (2) <u>< 0.002</u> (2) <u>< 0.002</u> (2)	< 0.002 (2) < 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2) < 0.002 (2)	Mareny des Barraquetes
Spain 2002	Primat	3× 18	70–81	0 3 3 3	fruit peel pulp fruit	0.006 0.006, 0.004 <u>< 0.002</u> (2) <u>0.002</u> (0.003, 0.002)	< 0.002 < 0.002 (2) < 0.002 (2) < 0.002(2)	< 0.002 < 0.002 (2) < 0.002 (2) < 0.002(2)	02-1055; Sanlucar de Barrameda
Spain	Galia-F	3× 18	70–81	0 3 3 3	fruit peel pulp fruit	< 0.002 < 0.002 (2) <u>< 0.002</u> (2) <u>< 0.002</u> (2)	< 0.002 < 0.002 (2) < 0.002 (2) < 0.002(2)	< 0.002 << 0.002 (2) < 0.002 (2) < 0.002(2)	1046/01; Chipiona

Peppers

Eighteen supervised trials were carried out on protected <u>peppers</u> between 1998 and 2013 in Europe. Samples of pepper fruits were stored deep-frozen for a maximum of 11 months and residues analysed either by LC/MS/MS or HPLC-FL. Four supervised trials were carried out on open field chilli peppers in the USA in 1994. Samples were stored deep-frozen for a maximum of 5.6 months and residues analysed by HPLC-LC. Summaries of the trial results are given in Table 67.

Table 67 Results from protected supervised trials conducted with abamectin on peppers in Europe (protected) and USA (field)

Country (year)	Pepper variety	Application rate, g ai/ha	Growth stage (BBCH)	DAT, days	Residue Found (mg/kg)			Study; trial
					Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
France 1998	Sweet, Spartacus	6× 22	67–76	-0 0 3 7 14	< 0.005 0.015 <u>< 0.005</u> < 0.005 < 0.005	included	< 0.005 < 0.005 < 0.005 < 0.005 < 0.005 [a]	9830401; Ouvrouer les Champs
France 1998	Sweet, Evident	6× 22	73–78	3	< 0.005 (2)	included	< 0.005 (2) [a]	9830301; St Cyr en Val
France 1998	Sweet, Lipari	6× 22	701–705	-0 0 3 7 14	< 0.005 0.071 <u>0.051</u> 0.040 0.005	included	< 0.005 0.005 < 0.005 < 0.005 < 0.005 [a]	9830402; Monteux
France 1998	Sweet, Miami	6× 22	701–705	3	< 0.005 (2)	included	0.009, 0.010 [a]	9830302; Avignon
France 1999	Sweet, Spartacus	4× 22	65–73	0 3	0.011, 0.010 <u>0.006</u> (0.006, 0.005)	included	< 0.002 (2) < 0.002 (2) [a]	9931501; Ouvrouer les Champs
France 1999	Sweet, Evident	4× 22	64–72	0 3	0.015, 0.020 <u>0.005</u> (2)	included	< 0.002 (2) < 0.002 (2) [a]	9931502; Cyr en Val
France 2013	Vidi	5× 20	86–89	-0 0 3 7	0.013 0.020 <u>0.025</u> 0.016	< 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002	S12-04360-01
Italy	Green	4× 18	73–87	0	0.006	< 0.002	< 0.002	1042/01;

Country (year)	Pepper variety	Application rate, g ai/ha	Growth stage (BBCH)	DAT, days	Residue Found (mg/kg)			Study; trial
					Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
2001	Sienor			3	0.002	< 0.002	< 0.002	Bagnarola di Budrio
Netherlands 2013	Bell Waltz	5× 22	60–89	-0 0 3 7	0.018 0.025 0.022 0.027	< 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002	S12-04360-02
Netherlands 2013	Bell Maranello	5× 22	60–89	-0 0 3 7	0.011 0.019 0.015 0.010	< 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002	S12-04360-03
Netherlands 2013	Bell Maranello	5× 22	60–88	-0 0 3 7	0.013 0.035 0.019 0.016	< 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002	S12-04360-04
Spain 2001	Sweet, Gallego	20, 21, 22, 22	83–85	0 3	0.021 0.010 (0.012, 0.008)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	1047/01
Spain 1999	Sweet, Piquillo	2× 22 2× 23	87–89	0 3	0.051, 0.027 0.018 (0.019, 0.017)	0.004, 0.002 0.003 (2)	< 0.002 (2) < 0.002 (2)	1109/99
Spain 1999	Sweet, Itálico	21, 21, 22, 23	83–89	0 3	0.024, 0.025 0.008 (0.008, 0.009)	< 0.002 (2) 0.002, < 0.002	< 0.002 (2) < 0.002 (2)	1108/99; Sanlúcar de Barrameda
Spain 2002	Sweet, Herminio	4× 26	82	0 3	0.011 0.004 (0.002, 0.006)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	02-1053; El Mirador
Spain 2002	Sweet, Marnier	24, 25, 26, 28	61–89	0 3	0.024 0.002 (2)	< 0.002 < 0.002 (2)	0.002 < 0.002 (2)	02-1052; Mareny des Barraquets S
Switzerland 2000	Sweet, Goldflame	5× 22	63–73	0 3	0.035 0.012 (0.014, 0.010)	< 0.002 < 0.002 (2)	0.003 < 0.002 (2)	1006/00; 1006/00
Switzerland 2000	Sweet, Mazurka	5× 22	63–73	0 3	0.031 0.020 (0.020, 0.019)	< 0.002 < 0.002 (2)	0.002 < 0.002 (2)	1007/00; 1007/00
USA,TX 1994	Chilli, Jalapeño	6× 22	–	0 3 7	0.007, 0.005 < 0.005 (2) < 0.005 (2)	included	< 0.005 (2) < 0.005 (2) < 0.005 (2)	ADC 1452-1; 001-94-8000R
US, nm 1994	Chilli, Serrano	6× 22	–	0 3 7	0.012, 0.011 < 0.005 (2) < 0.005 (2)	included	< 0.005 (2) < 0.005 (2) < 0.05 (2)	ADC 1452-1; 001-94-8001R
USA AR 1994	Chilli, Serrano	6× 22	–	0 3 7	0.013, 0.012 < 0.005 (2) < 0.005 (2)	included	< 0.005 (2) < 0.005 (2) < 0.005 (2)	ADC 1452-1; 001-94-8002R
USA, CA 1994	Chilli, Jalapeño	6× 22	–	0 3 7	0.014, 0.015 < 0.005 (2) < 0.005 (2)	included	< 0.005 (2) < 0.005 (2) < 0.005 (2)	ADC 1452-1; 001-94-8003R

[a] Includes the 8,9-z isomer of avermectin B_{1b}

Tomatoes

Forty-two supervised trials were carried out on protected tomatoes in Europe in 1993, 1998, 2000, 2001, 2003, 2007 and 2008. Residues were analysed either by method 91.1 or by method REM 198.02 (equivalent to method MSD 8920 mod). Samples of tomato fruits were stored deep-frozen for a maximum of 16 months. Summaries of the trial results are given in Table 68.

Table 68 Results from supervised trials conducted with abamectin on tomato in Europe, either protected (P) or in the field (F)

Country (year)	Tomato variety (P or F)	Application rate, g ai/ha	Growth stage (BBCH)	DAT, days	Residue Found (mg/kg)			Study; trial
					Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
France 2000	Felicia (P)	4× 18	66–72	0 3	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	0031801
France 2000	Servanne (P)	4× 18	70–80	0 3	0.005 0.004 (0.003, 0.004)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	0031802
France 2000	Granitio (P)	4× 27	71–85	0 3 7	0.010 0.004 (0.004, 0.005) 0.003	< 0.002 < 0.002 (2) < 0.002	< 0.002 < 0.002 (2) < 0.002	0031901
France 2007	Sympathie (P)	2× 22	82–86	–0 0 1 3 7	0.005 0.009 0.010 0.011 0.005	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	CEMS-3518; AF/11536/SY/1
France 2007	Tornado (P)	2× 22	61–89	–0 0 1 3 7	< 0.002 0.003 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	CEMS-3519; AF/11537/SY/1
Germany 2000	Vanessa (P)	5× 11	72–84	0 3	0.005 0.004	< 0.002 < 0.002	< 0.002 < 0.002	gr 71500; Rülzheim
Germany 2001	Pannovy (P)	17, 3x 18, 22	81–82	0 3	0.0095 0.004 (2)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	gto 35301; ross Gaglow
Germany 2001	Vanessa (P)	18, 2x19, 2x20	59–82	0 3	0.004 < 0.002 (2)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	gto 55301; Eich
Germany 2007	Ochsenherz (P)	2× 20	73–83	–0 0 1 3 7	< 0.002 0.009 0.005 0.005 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	CEMS-3518; AF/11536/SY/2
Italy 2003	Naxos (P)	2× 22 2× 21	71–88	0 1 3 7 10	0.011 0.007 0.004 (0.004, 0.005) 0.002 0.002	< 0.002 < 0.002 < 0.002 (2) < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 (2) < 0.002 < 0.002	03-1025
Italy 2007	Caramba (P)	2× 22	85–87	–0 0 1 3 7	< 0.002 0.004 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	CEMS-3519; AF/11537/SY/3
Netherlands 1993	Pronto (P)	4× 22	fruiting	0 3 7	0.009, 0.005 0.007 (0.009, < 0.005) 0.007, < 0.005	included	< 0.005 (2) < 0.005 (2) < 0.005 (2) [a]	1259B; 070-93-0001 R
Netherlands 1993	Pronto (P)	4× 22	fruiting	0 3 7	0.011, < 0.005 0.004 (0.067, < 0.005) 0.064, < 0.005	included	< 0.005 (2) < 0.005 (2) < 0.005 (2) [a]	1259B; 070-93-0002 R

Country (year)	Tomato variety (P or F)	Application rate, g ai/ha	Growth stage (BBCH)	DAT, days	Residue Found (mg/kg)			Study; trial
					Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
Netherlands 1993	Pronto (P)	4× 22	fruiting	0 3 7	0.014, 0.015 0.009 (0.011, 0.007) <u>0.010</u> (0.009, 0.012)	included	< 0.005 (2) < 0.005 (2) < 0.005 (2) [a]	1259B; 070-93-0003 R
Netherlands 1993	Trust (P)	4× 22	fruiting	0 3 7	0.006, < 0.005 <u>0.006</u> (0.006, < 0.005) 0.007, < 0.005	included	< 0.005 (2) < 0.005 (2) < 0.005 (2) [a]	1259B; 070-93-0004 R
Netherlands 1993	(P)	4× 22	fruiting	0 3 7	0.019, 0.024 <u>0.014</u> (0.010, 0.017) 0.007, 0.012	included	< 0.005 (2) < 0.005 (2) < 0.005 (2) [a]	1259B; 070-93-0005 R
Netherlands 1993	Pronto (P)	4× 22	fruiting	0 3 7	0.017, 0.018 <u>0.012</u> (0.012, 0.011) 0.010, 0.008	included	< 0.005 (2) < 0.005 (2) < 0.005 (2) [a]	1259B; 070-93-0006 R
Netherlands 1998	Durintha (P)	3× 12, 14	71–83	0 3	0.003, 0.004 0.003 (2)	< 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	1124/98
Netherlands 1998	Durintha (P)	4× 12	71–83	0 3	0.002 (2) 0.003 (2)	< 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	1123/98; 1123/98
Netherlands 2000	Durinta (P)	5× 10	60–89	0 3	0.008 0.006, 0.007	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	1118/00
Netherlands 2001	Clarence (P)	9, 10, 11, 12, 11	harvest	0 3 7	0.005 0.003, 0.004 0.002	< 0.002 < 0.002 (2) < 0.002	< 0.002 < 0.002 (2) < 0.002	1113/01
Netherlands 2001	Prospero (P)	11, 14, 13, 15, 14	harvest	0 3 7	0.007 0.005, 0.006 0.0031	< 0.002 < 0.002 (2) < 0.002	< 0.002 < 0.002 (2) < 0.002	1112/01; Bleiswijk
Netherlands 2008	Korneett (P)	4× 22	60–89	–0 0 1 3 7 10	0.010 0.017 0.021 0.011 <u>0.024</u> 0.014	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	T000572-08-REG; S08-00801-01
Netherlands 2008	Brilliant (P)	4× 22	60–89	–0 0 1 3 7 10	0.010 0.011 0.010 0.014 0.018 <u>0.027</u>	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 0.002 0.003	T000572-08-REG; S08-00801-02
Netherlands 2008	Briljant (P)	4× 22	60–89	0 1 3 7 10	0.021 0.024 0.017 0.022 <u>0.027</u>	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	0.002 0.002 < 0.002 0.002 0.002	T000572-08-REG; S08-00801-03
Netherlands 2008	Tresco (P)	21, 4× 22	60–89	0 1 3 7 10	0.033 0.024 0.016 0.020 <u>0.025</u>	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	0.003 < 0.002 < 0.002 0.002 0.003	T000572-08-REG; S08-00801-04
Spain 2000	Daniela (P)	3× 18, 16	82–83	0 3 7	0.004 <u>0.002</u> < 0.002 (2)	< 0.002 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 (2)	1008/00; Cañada de Gallego

Country (year)	Tomato variety (P or F)	Application rate, g ai/ha	Growth stage (BBCH)	DAT, days	Residue Found (mg/kg)			Study; trial
					Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
Spain 2000	Bond (P)	2× 19, 17, 18	71- 85	0 3 7	0.005 <u>< 0.002</u> < 0.002 (2)	< 0.002 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 (2)	1009/00
Spain 2001	Romana (P)	23, 22, 22, 21	79–82	0 1 3 7	0.007 0.003 <u>0.003</u> < 0.002, 0.002	< 0.002 < 0.002 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 0.002, < 0.002	1107/01; Canada Gallego
Spain 2001	Bond (P)	2× 22	75–74	0 1 3 7	0.004 0.004 0.003 0.002, 0.003	< 0.002 < 0.002 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 < 0.002 (2)	1108/01
		21, 20, 24, 23	73–75	0 1 3 7	0.008 0.003 <u>0.004</u> 0.004, 0.003	< 0.002 < 0.002 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 < 0.002 (2)	
Spain 2001	Bond (P)	22, 21	85–87	0 1 3 7	0.004 0.006 <u>0.004</u> 0.002, < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 < 0.002 (2)	1109/01
		2× 22 2× 21	83–87	0 1 3 7	0.010 0.005 0.003 < 0.002 (2)	< 0.002 < 0.002 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 < 0.002 (2)	
Spain 2003	Jack (P)	2× 19 20, 22	71- 79	0 1 3 7 11	0.017 0.01 <u>0.007</u> 0.006 0.003	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	03-1019
France 2000	Promo (F)	4× 22	76–87	0 3 7	0.009 <u>< 0.002</u> (2) < 0.002	< 0.002 < 0.002 (2) < 0.002	< 0.002 < 0.002 (2) < 0.002	0032001
Italy 2000	98063 (F)	3× 18	78–81	0 3 7	0.012 <u>< 0.002</u> (2) < 0.002 (2)	< 0.002 < 0.002 (2) < 0.002 (2)	< 0.002 < 0.002 (2) < 0.002 (2)	1097/00; S.Giorgio Piacentino
Italy 2000	690 (F)	3× 18	81–89	0 3 7	0.006 <u>< 0.002</u> (2) < 0.002 (2)	< 0.002 < 0.002 (2) < 0.002 (2)	< 0.002 < 0.002 (2) < 0.002(2)	1098/00; Lombardo
Italy 2001	Falco Rosso (F)	3× 22	81–87	0 3	0.0077 <u>< 0.002</u> (2)	< 0.002 (2) < 0.002	< 0.002 (2) < 0.002	1043/01; Lagosanto
Italy 2001	Heinz 9478 (F)	3× 22	79–85	0 3	0.0071 < 0.002 (2)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	1044/01; Barbiano di Cotignola
Spain 1999	Bodar (F)	3× 22	71–73	0 3	0.006, 0.004 <u>0.002</u> (0.002, < 0.002)	< 0.002 (2) 0.002 (2)	< 0.002 (2) 0.002 (2)	1110/99; Cullera
Spain 1999	Batlle (F)	3× 22	63–73	0 3	0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	1111/99; Picaña
Spain 2000	Batlle (F)	21, 2× 22	72–74	0 3	0.010 <u>0.002</u> (2)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	1087/00; Picañia
Spain 2001	Royesca (F)	2× 21, 22	79–81	0 3	0.007 <u>0.002</u> (2)	< 0.002 0.002 (2)	< 0.002 0.002 (2)	1086/01; Massalfassar

[a] Includes the 8,9-z isomer of avermectin B_{1b}

Eggplants

Two supervised trials were carried out on protected eggplants in 1998. Samples of eggplant fruits were stored deep-frozen for a maximum of 4 months and analysed by HPLC-FL. Summaries of the trial results are given in Table 69.

Table 69 Results from protected supervised trials conducted with abamectin on eggplant in France

Location	Eggplant variety	Application rate (g ai/ha)	Growth stage BBCH	DAT, days	Residues, mg/kg		Trial
					Abamectin B_{1a} + 8,9-Z-isomer	Abamectin B_{1b} + 8,9-Z-isomer	
Ouvrouer les Champs	Madona	6× 22	61–73	3	< 0.005 (2)	< 0.005 (2)	9830201
Calvisson	Telar	6× 22	501–504	–0 0 3 7 14	< 0.005 0.015 < 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005 < 0.005 < 0.005	9830101

Lettuce

Thirty four supervised trials on protected lettuce and twelve trials on open-field lettuce were carried out in 1999 to 2008. Samples of lettuce were stored deep-frozen for a maximum of 16 months, and samples analysed by HPLC-FL or LC-MS/MS. Summaries of the trial results are given in Table 70.

Table 70 Results from supervised trials conducted with abamectin on lettuce in Europe, either protected (P) or in the field (F)

Country year	Lettuce variety (P or F)	Application		DAT (days)	Residues, mg/kg			Study, trial
		Rate, g ai/ha)	Growth Stage		Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
France 1999	Head lettuce, Angie (P)	4× (8–9)	42–48	0 3 7 14 21	0.36 0.25 0.20 0.097 0.059	included	0.014 0.009 0.008 0.004 0.002 [a]	0030301 Sandillon
France 1999	Head lettuce, Sensai (P)	4× (8–9)	19–45	0 3 7 14 21	0.340 0.100 0.050 0.020 0.006	included	0.013 0.004 0.002 < 0.002 > 0.002 [a]	0030302 St. Genouph
France 2000	Head lettuce, Kristo (P)	3, 3× 7	19–41	0 3 7 14	0.114 0.043 0.021 0.11 (0.010, 0.012)	< 0.002 0.003 < 0.002 < 0.002 (2)	0.007 0.003 < 0.002 < 0.002 (2)	1114/00
France 2000	Head lettuce, Angié (P)	2× 3 2× 6	16–47	0 3 7 14	0.151 0.048 0.026 0.005, 0.006	< 0.002 0.005 0.004 < 0.002 (2)	0.009 0.003 < 0.002, < 0.002 (2)	1115/00
France 2000	Head lettuce, Angié (P)	2, 3, 4, 7	15–41	0 3 7 13	0.115 0.032 0.008 0.004, 0.003	< 0.002 < 0.002 < 0.002 < 0.002 (2)	0.008 0.002 < 0.002 < 0.002 (2)	1116/00
France 2000	Head lettuce, Sensaï (P)	2, 3, 3, 7	15–41	0 3 7 13	0.143 0.064 0.016 0.009, 0.008	< 0.002 0.002 < 0.002 < 0.002 (2)	0.009 0.004 < 0.002 < 0.002 (2)	1117/00
France 2005	Cambria (P)	4× 9	13–19	–0 0 3 7	0.015 0.34 0.057 0.015	< 0.002 0.003 0.006 0.002	< 0.002 0.024 0.003 < 0.002	05-0501; AF/8590/SY/4

Country year	Lettuce variety (P or F)	Application		DAT (days)	Residues, mg/kg			Study, trial
		Rate, g ai/ha)	Growth Stage		Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
				14 21	0.003 < 0.002	< 0.002 < 0.002	< 0.002 < 0.002	
France 2005	Lettuce (P)	4× 9	16–46	–0 0 14	0.012 0.204 0.004	< 0.002 < 0.002 < 0.002	< 0.002 0.016 < 0.002	05-0501; AF/8590/SY/5
France 2005	Grinil (P)	4× 9	14–46	–0 0 14	0.011 0.261 0.003	< 0.002 < 0.002 < 0.002	< 0.002 0.015 < 0.002	05-0501; AF/8590/SY/6
France 2008	Head, Palomis (P)	4× 9	17–45	–0 0 3 7 14 21	0.028 0.122 0.087 0.038 0.019 0.008	0.002 < 0.002 0.005 0.002 < 0.002 < 0.002	0.003 0.015 0.009 0.003 < 0.002 < 0.002	T000573-08-REG; S08-00802-01
United Kingdom 1999	Head lettuce (P)	4× (3–4)	15–42	0 14	0.348, 0.315 < 0.002 (2)	0.005 (2) < 0.002 (2)	0.019, 0.018 < 0.002 (2)	1039/99
United Kingdom 1999	Head lettuce (P)	4× (3–4)	16–42	0 14	0.225, 0.247 < 0.002 (2)	< 0.002 (2) < 0.002 (2)	0.013 (2) < 0.002 (2)	1040/99
United Kingdom 1999	Head lettuce, Vegas (P)	4× (3–4)	16–41	0 3 7 10 14	0.162 0.060 0.026 0.016 0.010, 0.012	< 0.002 0.007 0.004 0.002 < 0.002 (2)	0.009 0.003 < 0.002 < 0.002 < 0.002 (2)	1041/99
United Kingdom 1999	Head lettuce, Frandria (P)	4× (3–4)	15–42	0 4 8 11 14	0.086 0.005 0.004 0.002 0.002 (2)	0.002 < 0.002 < 0.002 < 0.002 < 0.002 (2)	0.005 < 0.002 < 0.002 < 0.002 < 0.002 (2)	1042/99
United Kingdom 2005	Lettuce, Josephine (P)	4× 9	15–39	–0 0 3 7 14 21	0.004 0.365 0.047 0.022 0.004 < 0.002	< 0.002 < 0.002 0.003 < 0.002 < 0.002 < 0.002	< 0.002 0.015 0.003 < 0.002 < 0.002 < 0.002	05-0501; AF/8590/SY/1
United Kingdom 2005	Alexander (P)	4× 9	33–47	–0 0 14	0.037 0.132 0.012	0.003 0.003 < 0.002	0.003 0.010 < 0.002	05-0501; AF/8590/SY/2
United Kingdom 2005	Head, Brian (P)	4× 9	16–45	–0 0 14	0.019 0.301 0.007	0.003 < 0.002 < 0.002	< 0.002 0.024 < 0.002	05–0501; AF/8590/SY/3
United Kingdom 2008	Head, Whiske (P)	4× 9	33–45	–0 0 3 7 14 21	0.044 0.243 0.100 0.050 0.035 0.020	< 0.002 < 0.002 0.003 0.003 0.003 < 0.002	0.005 0.028 0.013 0.006 0.004 0.003	T000573-08-REG;S08-00802-02
United Kingdom 2008	Head, Brian (P)	4× 9	32–45	0 3 7 14 21	0.344 0.122 0.061 0.045 0.043	0.005 0.009 < 0.002 0.004 0.004	0.036 0.015 0.007 0.006 0.005	T000573-08-REG FSGD-045; S08-00802-03
United Kingdom 2008	Head, Whiske (P)	4× 9	37–45	0 3 7 14 21	0.255 0.104 0.071 0.047 0.025	0.002 0.003 0.002 0.003 < 0.002	0.027 0.012 0.008 0.005 0.003	T000573-08-REG FSGD-045; S08-00802-04
France 2007	Head, Iceberg (F)	2× 18	43–48	–0 0 1 3	< 0.002 0.193 0.016 0.003	< 0.002 < 0.002 < 0.002 < 0.002	< 0.002 0.013 < 0.002 < 0.002	CEMS-3517; AF/11534/SY/2

Country year	Lettuce variety (P or F)	Application		DAT (days)	Residues, mg/kg			Study, trial
		Rate, g ai/ha)	Growth Stage		Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
				7 14	0.003 < 0.002	< 0.002 < 0.002	< 0.002 < 0.002	
France 2000	Cos lettuce Green Tower (F)	3× 18	19–47	0 3 7	0.17 0.003 (2) < 0.002	< 0.002 < 0.002 (2) < 0.002	0.019 < 0.002 (2) < 0.002	0032102
France 2000	Cos lettuce Alisia (F)	3× 18	19–49	0 3 7	0.24 0.011, 0.010 0.003	< 0.002 < 0.002 (2) < 0.002	0.014 < 0.002 (2) < 0.002	0032101
France 2003	Lamb's, Gala (F)	9	Cotyledon	14	< 0.005	–	< 0.005	RLMA21903; RE03019
France 2003	Lamb's, Gala (F)	9	Cotyledon	14	< 0.005	–	< 0.005	RLMA21903; RE03020
France 2007	Head, Italina (F)	2× 18	19–41	–0 0 1 3 7 14	0.005 0.318 0.101 0.049 0.003 < 0.002	< 0.002 0.006 0.008 0.003 < 0.002 < 0.002	< 0.002 0.038 0.010 0.005 < 0.002 < 0.002	CEMS-3516; AF/11535/SY/1
Italy 2000	Cos lettuce Sofia (F)	3× 18	43–48	0 3 7	0.125 0.010, 0.012 0.008 (0.011, 0.006)	0.002 < 0.002 (2) < 0.002 (2)	0.008 < 0.002 (2) < 0.002 (2)	1095/00
Italy 2000	Cos lettuce Canasta Semi-open (F)	3× 18	41–49	0 3 7	0.034 0.015, 0.010 0.006 (0.005, 0.007)	0.005 0.002, < 0.002 < 0.002 (2)	0.023 < 0.002 (2) < 0.002 (2)	1096/00 Mediglia
Italy 2007	Head Gentilina Open (F)	18, 19	43–45	–0 0 1 3 7 14	0.041 0.556 0.374 0.018 < 0.002 < 0.002	< 0.002 0.011 0.008 < 0.002 < 0.002 < 0.002	0.003 0.051 0.048 < 0.002 < 0.002 < 0.002	CEMS-3516; AF/11535/SY/2
Spain 1992	Leaf lettuce Summer Blond (F)	4×22	–	0 7 14	0.198, 0.163, 0.171, 0.188 0.007 (0.007, 0.008, 0.009, 0.004) < 0.002 (4)	inlcuded	0.021, 0.018, 0.018, 0.021 < 0.002 (4) < 0.002 (4) [a]	1274-4 ADC; 065-92-0003R
		4× 43	–	0 7 14	0.361, 0.437 0.298, 0.465 0.025 (2), 0.028, 0.024 0.004, 0.005 0.002, 0.003	inlcuded	0.041, 0.045, 0.030, 0.053 0.002 (2), < 0.002 (2) < 0.002 (4) [a]	
Spain 1992	Leaf lettuce Inverna (F)	4×22	–	0 7 14	0.210, 0.166, 0.182, 0.242 0.004 (0.005, 0.004, 0.003, 0.004) 0.002, < 0.002 (3)	inlcuded	0.025, 0.019, 0.021, 0.028 < 0.002 (4) < 0.002 (4) [a]	1274-5 ADC; 065-92-0004R
		4× 43	–	0 7 14	0.396, 0.216, 0.544, 0.417 0.006, 0.005 (3) 0.003, 0.002 (2), < 0.002,	inlcuded	0.047, 0.024, 0.061, 0.048 < 0.002 (4) < 0.002 (4) [a]	
United Kingdom 2007	Head Brenson (F)	2× 18	45–47	–0 0 1 3	0.002 0.455 0.333 0.010	< 0.002 0.027 0.025 < 0.002	< 0.002 0.035 0.026 < 0.002	CEMS-3517; AF/11534/SY/1

Country year	Lettuce variety (P or F)	Application		DAT (days)	Residues, mg/kg			Study, trial
		Rate, g ai/ha)	Growth Stage		Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
				7 14	0.005 < 0.002	< 0.002 < 0.002	< 0.002 < 0.002	

[a] Includes the 8,9-z isomer of avermectin B_{1b}

Spinach

Eleven supervised trials were conducted in the USA on open field spinach in 1995, 1996, and 2007/08. Samples of spinach were stored deep-frozen for a maximum of 6 months and analysed by HPLC-FL. Summaries of the trial results on spinach are given in Table 71.

Table 71 Results from supervised trials conducted with abamectin on spinach in USA

Location year	Spinach variety	Application rate, g ai/ha	Growth stage	DAT, days	Residue Found (mg/kg)		Recovery Data
					Avermectin B_{1a} + 8,9-Z-isomer	Avermectin B_{1b} + 8,9-Z-isomer	
California 1995	Bossanova	6× 21	immature–mature	0 7 14	0.71, 0.58, 0.58, 0.40 0.028 (0.031, 0.023, 0.034, 0.024) 0.008 (2)	0.060, 0.040 0.003 (2) < 0.002 (2)	ABR-98078; 001-95-1018R
Texas 1995	Bolero	6× 21	7 in. rosette –12 in. tall	0 7 14	0.71, 0.57 0.085 (0.091, 0.079) 0.026, 0.022	0.072, 0.054 0.008, 0.007 0.002, < 0.002	ABR-98078; 001-95-8006R
Colorado 1996	Melody Firs	6× 21	1 in. tall –mature	0 7 14	0.56, 0.61 0.024 (0.021, 0.026) 0.017, 0.015	0.040, 0.041 < 0.002 (2) < 0.002 (2)	ABR-98078; 001-96-1002R
South Carolina 1996	Bloomsdale Long	6× 21	vegetative	0 7 14	0.86, 0.68 0.042 (0.046, 0.039) 0.017 (2)	0.086, 0.069 0.006, 0.004 0.003, 0.002	ABR-98078; 001-96-2000R
New Jersey 1996	Winter Bloomsdale	5× 21	1–3 in.–4–8 in. tall	0 7 14	0.28, 0.26 0.020 (0.022, 0.018) 0.011, 0.014	0.017, 0.016 < 0.002 (2) < 0.002 (2)	ABR-98078; 001-96-2001R
California 1996	Ty-ee	6× 21	first leaf-mature	0 7 14	0.81, 0.80 0.044 (0.043, 0.045) 0.024, 0.021	0.046, 0.048 0.003, 0.003 < 0.002 (2)	ABR-98078; 001-96-5014R
Virginia (2008)	Tyee F	3× 21	–	7	0.019, 0.012	< 0.002 (2)	T005593-07; E07VA078408
Oklahoma 2008	Spargo F	3× 22	BBCH 75–49	7	< 0.002 (2)	< 0.002 (2)	T005593-07; W01TX078413
Colorado 2008	Bloomsdale	3× 22	vegetative	7	< 0.002 (2)	< 0.002 (2)	T005593-07; W12CO078414
California 2007	Hybrid 7	3× 22	BBCH 49	7	0.048 (0.056, 0.040)	0.004, 0.003	T005593-07; W29CA078427
California 2008	Bloomsdale	3× 21	14–30 leaves	7	0.021 (0.022, 0.019)	< 0.002 (2)	T005593-07; W28CA078428

Beans, green with pods

Sixteen trials on protected fresh beans were carried out in Europe between 2000 and 2009. Samples of green bean were stored deep-frozen for a maximum of 22 months and analysed by LC-MS/MS. Summaries of the trial results are given in Table 72.

Table 72 Results from green house supervised trials conducted with abamectin on beans, green with pods in Europe

Country year	Bean variety	Application rate, g ai/ha	Growth stage, BBCH	DAT, days	Residue Found (mg/kg)			Study; trial
					Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
France 2008	Booster	3× 23	65–83	–0 0 1 3 7	0.018 0.042 0.028 0.029 0.026	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	CEMS 3913; S08-00832-01
	Booster	2× 22	65–83	–0 0 1 3 7	0.023 0.047 0.043 0.023 0.020	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	
Italy 2008	Oriente	23, 20, 22	76–83	–0 0 1 3 7	< 0.002 0.038 0.011 0.016 0.008	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 0.002 < 0.002 < 0.002 < 0.002	CEMS 3913; S08-00832-02
	Oriente	22, 21	77–83	–0 0 1 3 7	< 0.002 0.036 0.026 0.012 0.010	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 0.003 < 0.002 < 0.002 < 0.002	
Spain 2000	Perona	3× 18	65–81	0 3 7	0.010 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 (2)	1010/00; Emperador
Spain 2000	Perona	20, 17, 18	66–83	0 3 7	0.022 0.003 < 0.002 (2)	< 0.002 < 0.002 < 0.002 (2)	0.002 < 0.002 < 0.002 (2)	1011/00 Serratelia
Spain 2000	French	17, 18, 19	63–82	0 3 7	0.040 0.017 0.007 (2)	< 0.002 < 0.002 < 0.002 (2)	0.003 < 0.002 < 0.002 (2)	1012/00 Alberic
Spain 2000	Punxeta	3× 18	65–83	0 3 7	0.026 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 (2)	0.002 < 0.002 < 0.002 (2)	1013/00 Xereza
Spain 2001	Doma	3× 21	75–77	0 3	0.017 0.007 (2)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	1081/01 Carchuna
Spain 2002	Maite R2	3× 22	78	0 3	0.007 < 0.002 (2)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	1082/01 Motril
Spain 2001/02	Dona	13, 15, 18	71–74	0 3	0.008 < 0.002 (2)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	1083/01 El-Ejido
Spain 2002	Oriente	17, 17, 21	63–67	0 3	0.022 0.004 (0.006, 0.003)	< 0.002 < 0.002 (2)	< 0.002 < 0.002 (2)	1084/01 El-Ejido
Spain 2008	Emerite	22, 22, 21	71–85	–0 0 1 3 7	0.015 0.067 0.052 0.049 0.028	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 0.004 0.002 0.002 < 0.002	CEMS-3913 S08-00832-03
	Emerite	2× 22	72–85	–0 0 1 3 7	0.009 0.075 0.046 0.048 0.037	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 0.003 0.002 0.003 0.002	
Spain 2008	Killy	20, 22, 22	76–77	–0 0 1 3 7	0.009 0.043 0.020 0.014 0.015	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	< 0.002 0.004 0.003 0.003 0.003	CEMS-3913 S08-00832-04
	Killy	22, 21	76 77	–0 0 1	0.009 0.036 0.019	< 0.002 < 0.002 < 0.002	< 0.002 0.004 0.003	

Country year	Bean variety	Application rate, g ai/ha	Growth stage, BBCH	DAT, days	Residue Found (mg/kg)			Study; trial
					Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
				3 7	0.014 0.009	< 0.002 < 0.002	0.003 0.003	

Beans (dry)

Twelve supervised residue trials were conducted on beans in the USA during 1999. In all trials, duplicate samples of dry beans were analysed by HPLC-FL. Dry bean samples were stored deep-frozen for a maximum of 14 months. Summaries of the trial results are given in Table 73.

Table 73 Results from supervised trials conducted with abamectin on dry beans in the USA in 1999 (Study 05001)

Region	Bean variety	Application rate, g ai/ha	Growth Stage	DAT, days	Residues, mg/kg		Trial
					Avermectin B_{1a}+ 8,9-Z-isomer	Avermectin B_{1b}	
New Jersey	ETNA	3× 20	vegetative pods filled	7	< 0.002 (2)	< 0.002 (2)	NJ26
Wisconsin Arlington	Great Northern Dry Bean	21, 20, 20	fruiting mature	5	< 0.002 (2)	< 0.002 (2)	WI13
Wisconsin Hancock	Great Northern Dry Bean	22, 24, 22	flowering, fruiting	6	< 0.002 (2)	< 0.002 (2)	WI14
Wisconsin Hancock	Great Northern Dry Bean	24, 22, 21	yellow-pods drying to mature	5	< 0.002 (2)	< 0.002 (2)	WI15
N. Dakota Minot	Maverick	3× 21	mature	7	< 0.002 (2)	< 0.002 (2)	ND05
N. Dakota Minot	Maverick	3× 21	mature	7	< 0.002 (2)	< 0.002 (2)	ND06
Ohio Freemont	Avanti–navy	3× 21	bloom and fruit	7	< 0.002 (2)	< 0.002 (2)	OH*10
Ohio Freemont	Avanti–navy	3× 21	Fruit–senescing	7	< 0.002 (2)	< 0.002 (2)	OH*11
Washington Moxee	Othello	3× 22	fruiting	7	< 0.002 (2)	< 0.002 (2)	WA*14
Washington Moxee	Othello	3× 21	fruiting	7	< 0.002 (2)	< 0.002 (2)	WA*15
California	CB-46	3× 21	maturing	6	0.003 (0.004, < 0.002)	< 0.002 (2)	CA57
Idahoo	Bill Z. Pinto	3× 21	maturing –drying	7	< 0.002 (2)	< 0.002 (2)	ID04

Celeriac

Two supervised residue trials were conducted on celeriac in the USA during 1998. Duplicate samples of celeriac (roots and tops) were analysed by HPLC-FL. Celeriac samples were stored deep-frozen for a maximum of 9.4 months for roots and 10.5 months for tops. Summaries of the trial results are given in Table 74.

Table 74 Results from supervised trials conducted with abamectin on celeriac in the USA in 1998 (Study: 06593)

Location [a]	Celeriac variety	Application rate, g ai/ha	Growth stage	DAT (days)	Crop Part	Residues, mg/kg		Trial
						Avermectin B_{1a} + 8,9-Z-isomer	Avermectin B_{1b} + 8,9-Z-isomer	
Paerlier CA	Brilliant	3× 22	maturing to mature	7	roots tops	≤ 0.002 (2) 0.005, 0.004	< 0.002 (2) < 0.002 (2)	98-CA06
Paerlier CA	Brilliant	3× 22	maturing root	7	roots tops	≤ 0.002 (2) 0.015, 0.014	< 0.002 (2) < 0.002 (2)	98-CA07

[a] Same location, but conducted in periods about 2 months apart

Potatoes

Eighteen supervised residue trials were conducted on potatoes in the USA in the growing seasons 1992–1994 and 1998. Potato samples were stored deep-frozen for a maximum of 15 months and analysed by HPLC-FL. Summaries of the trial results are given in Table 75.

Table 75 Results from supervised trials conducted with abamectin on potatoes in the USA

Location year	Potato variety	Application rate, g ai/ha	Growth stage	DAT, days	Residues, mg/kg		Report; Trial
					Avermectin B_{1a}+ 8,9-Z-isomer	Avermectin B_{1b} + 8,9-Z-isomer	
New York 1992	Katahdin	6× 112	foliage to mature	0 3 7	< 0.005 (2) < 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2) < 0.005 (2)	618-0936-3671; 001-92-5017R
		6× 112	foliage to mature	0 3 7	< 0.005 (2) < 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2) < 0.005 (2)	
Pensylvania 1992	Katahdin	6× 112		0 3 7	< 0.005 (2) < 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2) < 0.005 (2)	618-0936-3671; 001-92-5018R
		6× 112		0 3 7	< 0.005 (2) < 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2) < 0.005 (2)	
Oregon 1992	Russet Burbank	6× 112		0 3 7	< 0.005 (2) < 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2) < 0.005 (2)	618-0936-3671; 001-92-5019R
		6× 112		0 3 7	< 0.005 (2) < 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2) < 0.005 (2)	
Zelwood, FL 1993	Red La Soda	6× 21		0 14	< 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2)	618-936-93671; 001-93-0002R
La Belle, FL 1993	Atlantic	6× 112		0 3 7	< 0.005 (2) < 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2) < 0.005 (2)	618-936-93671; 001-92-0038R
		6× 112		0 3 7	< 0.005 (2) < 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2) < 0.005 (2)	
Amercian Falls, ID 1993	Russet Burbank	6× 18-21	≤ 5 oz to maturity	0 14	< 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2)	618-936-93671; 001-93-1004R
Jerome, ID 1993	Russet Burbank	6× 21	75% to 90% mature	0 14	< 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2)	618-936-93671; 001-93-1005R
Mason, MI 1993	Snowden	6× 19-22	senescence to maturity	0 14	< 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2)	618-936-93671; 001-93-1007R
Washington 1993	Russet Burbank	6× 21	3–4 in. to 24–26 in. high	0 14	< 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2)	618-936-93671; 001-93-5004R
Hugson, CA 1993	Red Lasoda	6× 21	9–15 in. to 10–15 in. high	0 14	< 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2)	618-936-93671; 001-93-5005R
Bakersfield, CA 1993	Russet Norkotah	6× 21	1.5–2 in. tubers vines dry	0 14	< 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2)	618-936-93671; 001-93-5006R
Maryland 1993	White Superior	6× 21	starting to bloom mature	0 14	< 0.005 (2) < 0.005 (2)	<< 0.005 (2) < 0.005 (2)	618-936-93671; 001-93-7000R
New York 1993	White Katahdin	6× 21	18 in. high senescence starting	0 14	< 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2)	618-936-93671; 001-93-7001R
Maine 1993	FL1625	6× 21	20 in.–bloom to post bloom	0 14	< 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2)	618-936-93671; 001-93-7002R

Location year	Potato variety	Application rate, g ai/ha	Growth stage	DAT, days	Residues, mg/kg		Report; Trial
					Avermectin B_{1a}+ 8,9-Z-isomer	Avermectin B_{1b} + 8,9-Z-isomer	
North Dakota 1994	Norchip	6× 21	18–24 in. high	0 14	< 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2)	618-936-93671; 001-94-1017R
Colorado 1994	Russet Nugget	6× 112	61–76 cm	0 14	< 0.005 (4) < 0.005 (4)	< 0.005 (4) < 0.005 (4)	618-936-93671; 001-94-1022R
Washington 1998	Russet Burbank	3× 21	–	14	< 0.005 (2)	< 0.005 (2)	T000141-98; 0W-IR-601-98
N w York 1998	Katahdin	3× 21	–	15	< 0.005 (3)	< 0.005 (2)	T000141-98;05-IR-006-98

Radish

Three supervised decline trials were carried out on protected radishes in 1996 and 1999 in the Netherlands. Residues in radish (whole plant, roots, and leaves with tops) were analysed by HPLC-FL or LC-MS/MS. Samples of radish were stored deep-frozen for a maximum of 8 months. Summaries of the trial results are given in Table 76.

Table 76 Results from protected supervised trials conducted with abamectin on radishes in the Netherlands

Year	Radish variety	Application rate, g ai/ha	DAT, days	Crop Part	Residues, mg/kg			Report; trial
					Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
1999	Donar	2× 10	0 3 7 7 10 10 12 12	w. plant w. plant leaf roots leaf roots leaf roots	0.324 0.106 0.074 < 0.002 0.061 < 0.002 0.08, 0.07 < 0.002 (2)	0.016 0.01 0.007 < 0.002 0.006 < 0.002 0.007, 0.006 < 0.002 (2)	0.019 0.007 0.004 < 0.002 0.004 < 0.002 0.004 (2) < 0.002 (2)	1015/99; 1- s-Gravenzande
1996	Nevada	15	0 14 14 21 21 28 28	w. plant leaf root leaf root leaf root	0.803 0.014 < 0.002 0.013, 0.012 < 0.002 0.009 < 0.002	included	0.061 < 0.002 < 0.002 < 0.002 (2) < 0.002 < 0.002 < 0.002	MEK34/971169; 070-96-0003R
		15	0 14 14 21 21 28 28	w. plant leaf root leaf root leaf root	0.835, 0.856 0.010 < 0.002 (2) 0.012 < 0.002 0.009 < 0.002	included	0.066, 0.063 < 0.002 < 0.001 (2) < 0.002 < 0.002 < 0.002 < 0.002	
1996	Nevada	14	0 14 14 21 21 28 28	w. plant leaf root leaf root leaf root	0.794 0.014 < 0.002 0.009 < 0.002 0.007, 0.008 < 0.002	included	0.054 < 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (2) < 0.001	MEK34/971169; 070-96-0004R
		14	0 14 14	w. plant leaf root	0.789 0.006 < 0.002	included	0.059 < 0.002 < 0.002	

Year	Radish variety	Application rate, g ai/ha	DAT, days	Crop Part	Residues, mg/kg			Report; trial
					Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
			21	leaf	0.007		< 0.002	
			21	root	< 0.002		< 0.002	
			28	leaf	0.007		< 0.002	
			28	root	< 0.002		< 0.002	

Celery

Seven trials were carried out on celery in southern European in the period 1999–2002. Samples of celery whole plant and leaf stalk were stored deep-frozen for a maximum of 8 months and residues in celery analysed by LC-MS/MS. Six trials on celery were conducted in the USA in the period 1999 and 2008. Samples were stored deep-frozen for a maximum of 16 months and analysed by HPLC-FL. Summaries of the trial results are given in Table 77.

Table 77 Results from supervised trials conducted with abamectin on celery

Country year	Celery variety	Application rate, g ai/ha	Growth stage BBCH	DAT, days	Residues, mg/kg			Report; trial
					Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
Italy 2002	Elena-Francese	3× 22	41–49	0 10	0.225 0.002	0.004 < 0.002	0.013 < 0.002	02-1150; Polig-nano a Mare
Spain 1999	Utha	3× 22	33–37	0 3 7 10	0.014 0.004 0.003 (2) 0.002	< 0.002 < 0.002 < 0.002 (2) < 0.002	< 0.002 < 0.002 < 0.002 (2) < 0.002	1001/99 El Siscar
Spain 1999	Utha	3× 22	33–37	0 3 7 10	0.020 0.017 0.003, 0.004 0.006	< 0.002 < 0.002 < 0.002 (2) < 0.002	< 0.002 < 0.002 < 0.002 (2) < 0.002	1002/99 El Siscar
Spain 2000	Slow Bolting	3× 22–23	42–45	0 3 7 10	0.013 0.012 < 0.002, 0.002 0.004	< 0.002 < 0.002 < 0.002 (2) < 0.002	< 0.002 < 0.002 < 0.002 (2) < 0.002	1002/00 El Siscar
Spain 2000	Utha 52-70R	3× 22	43–45	0 3 7 10	0.014 0.011 0.004, 0.003 < 0.002	< 0.002 < 0.002 < 0.002 (2) < 0.002	< 0.002 < 0.002 < 0.002 (2) < 0.002	1003/00
Spain 2000	Utha	3× 20–22	41–45	0 3 7 10	0.026, 0.021 0.005 (2) 0.015, 0.018, 0.003, 0.004 0.004, 0.003	< 0.002 (2) < 0.002 (2) < 0.002 (4) < 0.002 (2)	0.002, < 0.002 < 0.002 (2) < 0.002 (4) < 0.002 (2)	1004/00
Spain 2000	Elne	3× 22	19–49	0 7 10	0.075 0.009, 0.0180 0.010, 0.004	0.006 < 0.002 (2) < 0.002 (2)	0.0180 < 0.002 (2) < 0.002 (2)	1085/01 Sant Boi
USA, FL 2008	Golden Pascal	3× 21	vegetative	7	0.005 (0.006, 0.004)	included	< 0.002 (2)	T005593-07 E16FL078411
USA, MI 2008	Green Bay	3× 21	BBCH 45–49	7	0.005 (0.003, 0.007)	included	< 0.002 (2)	T005593-07 C01MI078412
USA, King	G-15	3× 22	BBCH	7	0.003 (2)	included	< 0.002 (2)	T005593-07

Country year	Celery variety	Application rate, g ai/ha	Growth stage BBCH	DAT, days	Residues, mg/kg			Report; trial
					Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
City, CA2008			47–75					W32CA078415
USA, Madera, CA 2008 [a]	Salyer Sonora	3× 22	BBCH 45–49	7	0.006 (0.009, 0.004)	included	< 0.002 (2)	T005593-07 W29CA078416
USA, Madera, CA 2008 [a]	Salyer Sonora	3× 22	BBCH 47–49	0 3 7 10	0.31 0.024 0.016 (0.016, 0.015) 0.013	included	0.006 < 0.002 < 0.002 (2) < 0.002	T005593-07 W29CA078417
USA, St Maria, CA 2008	Conquistador	3× 21	BBCH 45–48	7	0.010 (0.009, 0.010)	included	< 0.002, < 0.002	T005593-07 W30CA078418

[a] Different periods

Rice

Twenty four supervised residue trials were conducted on rice in China during 2010 and 2011. Samples of rice (paddy plant, husk and grain) were stored deep-frozen for a maximum of 16 month and analysed by HPLC-FL. Only avermectin B_{1a} was analysed and the results reported as total abamectin. Summaries of the trial results are given in Table 78.

Table 78 Results from supervised trials conducted with abamectin on rice in China (Report AHKW-BG-012-2011)

Region year	Application rate, g ai/ha	DAT, days	Total abamectin residue, mg/kg
Anhui Province 2010	2× 14	14	< 0.001
		21	< 0.001
	3× 14	14	< 0.001
		21	< 0.001
	2× 20	14	< 0.001
		21	< 0.001
	3× 20	14	< 0.001
		21	< 0.001
Hunan Province 2010	2× 14	14	< 0.001
		21	< 0.001
	3× 14	14	< 0.001
		21	< 0.001
	2× 20	14	< 0.001
		21	< 0.001
	3× 20	14	< 0.001
		21	< 0.001
Guangxi Province 2010	2× 14	14 21	< 0.001 < 0.001
	3× 14	14 21	< 0.001 < 0.001
	2× 20	14 21	< 0.001 < 0.001
	3× 20	14 21	< 0.001 < 0.001
Anhui Province 2011	2× 14	14	< 0.001
		21	< 0.001
	3× 14	14	< 0.001
		21	< 0.001
	2× 20	14	< 0.001
		21	< 0.001

Region year	Application rate, g ai/ha	DAT, days	Total abamectin residue, mg/kg
	3× 20	14	0.005
		21	< 0.001
Hunan Province 2011	2× 14	14	0.002
		21	< 0.001
	3× 14	14	0.002
		21	< 0.001
	2× 20	14	0.004
		21	0.001
	3× 20	14	0.007
		21	0.003
Guangxi Province 2011	2× 14	14 21	< 0.001 < 0.001
	3× 14	14 21	< 0.001 < 0.001
	2× 20	14 21	0.002 < 0.001
	3× 20	14 21	0.005 < 0.001

Tree nuts

Thirty-two residue trials were conducted on almonds, pecans, and walnuts in the USA during the 1988 and 1989 growing seasons. Dry tree nut samples were stored deep-frozen for a maximum of 20 months and analysed by HPLC-FL. Summaries of the trial results are given in Table 79.

Table 79 Results from supervised trials conducted with abamectin on nuts in theUSA (Study 618-936-TRN)

Location year	Crop variety	Application rate, g ai/ha	Growth stage	DAT, days	Residues, mg/kg Avermectin B_{1a} + 8,9-Z-isomer	Residues, mg/kg Avermectin B_{1b} + 8,9-Z-isomer	Trial
Fresno, CA 1988	Almond Non Pareil	3× 28	hull split	0 1 3	< 0.002 (4) < 0.002 (4) < 0.002 (4)	< 0.002 (4) < 0.002 (4) < 0.002 (4)	001-88- 6028R
		3× 56	hull split	0 1 3	< 0.002 (4) < 0.002 (4) < 0.002 (4)	< 0.002 (4) < 0.002 (4) < 0.002 (4)	
Madeira, CA 1988	Almond Non Pareil	3× 28	hull split	0 1 3	< 0.002 (4) < 0.002 (4) < 0.002 (4)	< 0.002 (4) < 0.002 (4) < 0.002 (4)	001-88- 6032R
		3× 56	hull split	0 1 3	< 0.002 (4) < 0.002 (4) < 0.002 (4)	< 0.002 (4) < 0.002 (4) < 0.002 (4)	
Stanislau, CA 1988	Almond Non Pareil	3× 28	hull split Post hull split	0 1 3 7 14	< 0.002 (4) < 0.002 (4) < 0.002 (4) < 0.002 (4) < 0.002 (4)	< 0.002 (4) < 0.002 (4) < 0.002 (4) < 0.002 (4) < 0.002 (4)	001-88- 6034R
		3× 56	hull split Post hull Split	0 1 3 7 14	< 0.002 (4) < 0.002 (4) < 0.002 (4) < 0.002 (4) < 0.002 (4)	< 0.002 (4) < 0.002 (4) < 0.002 (4) < 0.002 (4) < 0.002 (4)	
Stanislau, CA 1988	Almond Non Pareil	3× 28	hull split	0 1 3	< 0.002 (4) < 0.002 (4) < 0.002 (4)	< 0.002 (4) < 0.002 (4) < 0.002 (4)	001-88- 6035R
		3× 56	hull split	0 1 3 7 14	< 0.002 (4) < 0.002 (4) < 0.002 (4) < 0.002 (4) < 0.002 (4)	< 0.002 (4) < 0.002 (4) < 0.002 (4) < 0.002 (4) < 0.002 (4)	

Location year	Crop variety	Application rate, g ai/ha	Growth stage	DAT, days	Residues, mg/kg		Trial
					Avermectin B_{1a} + 8,9-Z-isomer	Avermectin B_{1b} + 8,9-Z-isomer	
				21	< 0.002 (4)	< 0.002 (4)	
Fresno, CA 1988	Walnut Franquette	3× 28	75% husk split	14	< 0.002 (4)	< 0.002 (4)	001-88-6027R
		2× 56	75% husk split	14	< 0.002 (4)	< 0.002 (4)	
Tulare, CA 1988	Walnut Serr	3× 30	10% husk split	14	< 0.002 (4)	< 0.002 (4)	001-88-6033R
		5× 59	10% husk split	14	< 0.002 (4)	< 0.002 (4)	
Stanislau, CA 1988	Walnut Chico	3× 28)	10% husk split	14	< 0.002 (4)	< 0.002 (4)	001-88-6038R
		3× 56	10% husk split	14	< 0.002 (4)	< 0.002 (4)	
San Benito, CA 1988	Walnut Payne	3× 28	80% husk split	14	< 0.002 (4)	< 0.002 (4)	001-88-6052R
		3× 56	80% husk split	14	< 0.002 (4)	< 0.002 (4)	
Colusa, CA 1989	Almond Mission	3× 28	hull split	0 14 21	< 0.002 (4) < 0.002 (4) < 0.002 (4)	< 0.002 (4) < 0.002 (4) < 0.002 (4)	001-89-6019R
Kern, CA 1989	Almond Mission	3× 28	hull split	0 14 21	< 0.002 (4) < 0.002 (4) < 0.002 (4)	< 0.002 (4) < 0.002 (4) < 0.002 (4)	001-89-6020R
Yolo, CA 1989	Walnut Hartley	3× 28	95% husk split	14	< 0.002 (4)	< 0.002 (4)	001-89-6034R
Stanislau, CA1989	Walnut Hartley	3× 28	Post full husk split	14	< 0.002 (4)	< 0.002 (4)	001-89-6035R
Jefferson, FL1988	Pecan Kiowa	3× 28	Pre shuck split	14	< 0.002 (4)	< 0.002 (4)	001-88-0033R
		3× 56	Pre shuck split	14	< 0.002 (4)	< 0.002 (4)	
Lee, AL 1988	Pecan Cheyanne	3× 28	Pre shuck split	18	< 0.002 (4)	< 0.002 (4)	001-88-0034R
		3× 56	Pre shuck split	18	< 0.002 (4)	< 0.002 (4)	
Mitchell, GA1988	Pecan Desirable	3× 28	Pre shuck split	14	< 0.002 (4)	< 0.002 (4)	001-88-0035R
		3× 56	Pre shuck split	14	< 0.002 (4)	< 0.002 (4)	
Zavalda, TX1988	Pecan Witchita	3× 28	90% shuck split	14	< 0.002 (4)	< 0.002 (4)	001-88-3017R
		3× 56	90% shuck split	14	< 0.002 (4)	< 0.002 (4)	
St. Francis, AZ 1988	Pecan Stuart	3× 28	Full shuck split	14	< 0.002 (4)	< 0.002 (4)	001-88-3023R
		3× 56	Full shuck split	14	< 0.002 (4)	< 0.002 (4)	
Mitchell, GA1989	Pecan Schley	3× 28	Full shuck split	14	< 0.002 (4)	< 0.002 (4)	001-89-0036R
Pinal, AR 1989	Pecan Western Schley	5× 28	Full shuck split	14	< 0.002 (4)	< 0.002 (4)	001-89-1029R

Cotton

Eight supervised trials were carried out on cotton in the 1999 and 2000 in Europe. Samples were stored deep-frozen for a maximum of 12 months and analysed by LC-MS/MS. Fourteen supervised trials were carried in 2008 and 2010 in the USA. Samples of undelinted seeds were stored deep-frozen for a maximum of 10 months, cotton meal was stored for a maximum of 7 months, gin by-products

and refined oil for 14 months and cottonseed hulls for 6 months, and analysed by HPLC-FL. Summaries of the trial results are given in Table 80.

Table 80 Results from supervised trials conducted with abamectin on cotton

Country year	Cotton variety	Application rate, g ai/ha	Growth stage (BBCH)	DAT, days	Residues, mg/kg			Study; trial
					Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
Greece 1999	506 Stoneville	2× 18	81, 83	0 20	< 0.002(2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	1104/99
Greece 1999	506 Stoneville	2× 18	81 82	0 20	0.002 < 0.002 (2)	< 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	1105/99
Greece 2000	453 Stoneville	2× 18	83–84 86–87	0 3 7 14 20	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (2)	1046/00; 1–Mavrogia
Greece 2000	453 Stoneville	2× 18	83–84 86–87	0 3 7 14 20	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (2)	1047/00; 1–Ippodromos
Spain 1999	Crema 111	18, 17	87–89	0 20	< 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	< 0.002 (2) < 0.002 (2)	1114/99
Spain 1999	Carmen	2× 18	87–89	0 3 7 14 20	0.002 < 0.002 < 0.002 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (2)	1115/99
Spain 2000	Crema	2× 18	87	0 3 7 14 20	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (2)	1088/00 Alcalá del Río
Spain 2000	Crema	2× 18	87	0 3 7 14 20	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (2)	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002 (2)	1089/00; Alcalá del Río
USA Suffolk, VA, 2008	PHY 370 WR	2× 21	79, 93	20	< 0.002 (2)	included	< 0.002 (2)	T005597-07; E07VA081021
USA Proctor, AR 2008	DG2215B2RF	2× 21	mature—50% opening	20	< 0.002 (2)	included	< 0.002 (2)	T005597-07; C24AR081022
USA Proctor, AR 2008	DG2215B2RF	2× 21	mature—50% opening	10 15 20 25 30	< 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2)	included	< 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2) < 0.002 (2)	T005597-07; C24AR081023
USA Uvalde, TX	DPL 434	2× 21	82, 86	20	< 0.002 (2)	included	< 0.002 (2)	T005597-07; W07TX0810
		2×106	82, 86	20	< 0.002,	included	< 0.002 (3)	24

Country year	Cotton variety	Application rate, g ai/ha	Growth stage (BBCH)	DAT, days	Residues, mg/kg			Study; trial
					Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
2008					0.009, 0.002			
USA Levelland, TX 2008	FM9063B2F	21, 22	90% size 25% opening	20	< 0.002 (2)	included	< 0.002 (2)	T005597-07; W39TX081025
USA Groom, TX 2008	2326RF	21, 22	81, 74	20	0.005 (< 0.002, 0.008)	included	< 0.002 (2)	T005597-07; E13TX08102
		107, 108	81, 74	20	0.015, 0.010, 0.011	included	< 0.002 (3)	6
USA Claude, TX 2008	NexGen 3554RF	2× 22	80, 72	20	< 0.002 (2)	included	< 0.002 (2)	T005597-07; E13TX081027
USA Fresno, CA 2008	PHY 755 WRF Acala	2× 21	80, 82	20	0.010 (0.010, 0.011)	included	< 0.002 (2)	T005597-07; W30CA081028
USA Madera, CA 2008	Acala Riata Roundup Ready	2× 21	< 1 to 10% opening	20	< 0.002 (2)	included	< 0.002 (2)	T005597-07; W29CA081029
USA LA, 2010	Phytogen 485 WRF	21, 22	5–70% open	20	< 0.002 (2)	included	< 0.002 (2)	TK0023918; E17-0011
USA TX, 2010	Stoneville 5458B2RF	2× 21	77, 87	20	< 0.002 (2)	included	< 0.002 (2)	TK0023918; W07-0012
USA CA, 2010	PHY725RF	2× 21	77, 86	20	< 0.002 (2)	included	< 0.002 (2)	TK0023918; W28-0014

Peanuts

Four supervised residue trials were conducted on peanuts in Brazil during the growing seasons of 2009. Peanut seed samples were stored deep-frozen for a maximum of 5.7 months and analysed by HPLC-FL. Residue data from supervised trials on peanut are summarized in Table 81.

Table 81 Results from supervised trials conducted with abamectin on peanuts in Brazil in 1999 (Report: M09044)

Location	Peanut variety	Application rate, g ai/ha	Growth stage (BBCH)	DAT (days)	Residues, mg/kg		Trial
					Avermectin B_{1a} + 8,9-Z-isomer	Avermectin B_{1b}	
Minas Gerais	Tatu	3× 14	91, 93, 95	7 14 21	< 0.005 < 0.005 < 0.005	< 0.003 < 0.003 < 0.003	JJB
Paraná	Tatu	3× 14	73, 77, 81	7 14 21	< 0.005 < 0.005 < 0.005	< 0.003 < 0.003 < 0.003	LZF1
São Paulo, Eng. Coelho	Tatu	3× 14	71–73, 75–77 81–85	7 14 21	< 0.005 < 0.005 < 0.005	< 0.003 < 0.003 < 0.003	LZF2
São Paulo, Jaboticabal	Alto Oleico	3× 14	75, 77, 79	7 14 21	< 0.005 < 0.005 < 0.005	< 0.003 < 0.003 < 0.003	LZF3

Coffee

Five supervised residue trials were conducted on coffee in Brazil during the growing seasons 2009 and 2010. Coffee (bean) samples were stored deep-frozen for a maximum of 5.1 months and analysed

by HPLC-FL or LC-MS/MS. Residue data from supervised trials on coffee are summarized in Table 82.

Table 82 Results from supervised trials conducted with abamectin on coffee in Brazil

Location year	Coffee variety	Application rate, g ai/ha	Growth stage BBCH	DAT, days	Residues, mg/kg			Study; trial
					Abamectin B_{1a}	B_{1a} 8,9-Z-isomer	Abamectin B_{1b}	
Minas Gerais 2009	Catuat	7.2	88	7 14 21	< 0.002 < 0.002 < 0.002	included	< 0.001 < 0.001 < 0.001	M09030;JJB
Monte Carmelo, MG 2010	Munda Nova	9.0	91	7 14 21	< 0.001 < 0.001 < 0.001	< 0.001 < 0.001 < 0.001	< 0.0004 < 0.0004 < 0.0004	M10031;JJB1
Indianopolis, MG 2010	Munda Nova	9.0	85	7 14 21	< 0.001 < 0.001 < 0.001	< 0.001 < 0.001 < 0.001	< 0.0004 < 0.0004 < 0.0004	M10031;JJB2
E. S. do Dourado, MG 2010	Munda Nova	9.0	83	7 14 21	< 0.001 < 0.001 < 0.001	< 0.001 < 0.001 < 0.001	< 0.0004 < 0.0004 < 0.0004	M10031;LZF
Parana 2010	IAPAR 59	9.0	89	7 14 21	< 0.001 < 0.001 < 0.001	< 0.001 < 0.001 < 0.001	< 0.0004 < 0.0004 < 0.0004	M10031;AMA

Hops

Eight supervised field trials on hops were conducted in Germany and four in the USA in 1994 and 1996. Samples were stored deep-frozen for a maximum of 6 months and analysed by HPLC-FL. Summaries of the trial results are given in Table 83.

Table 83 Results from supervised trials conducted with abamectin on hops

Country year	Hop variety	Application rate, g ai/ha	Growth stage BBCH	DAT, days	Crop Part	Residues, mg/kg		Study; trial
						Avermectin B_{1a} + 8,9-Z-isomer	Avermectin B_{1b} + 8,9-Z-isomer	
Germany (Tettnang) 1994	Hallertauer Frühreifer	24, 23	47 75	0 29 29	green cones dried cones cones	0.152, 0.136 0.012 (0.011, 0.012) < 0.005 (2)	0.010, 0.009 < 0.005 (2) < 0.005 (2)	E-96-MK-936-HOP; 072-96-0011R
Germany (Pfaffenhofen) 1994	Hersbrucker	22, 23	51 75	0 30 30	green cones dried cones cones	0.172, 0.283 < 0.005 (2) < 0.005 (2)	0.011, 0.019 < 0.005 (2) < 0.005 (2)	E-96-MK-936-HOP; 072-96-0012R
Germany (Pfaffenhofen) 1994	Perle	22, 23	51 75	0 30 30	green cones dried cones cones	0.225, 0.221 0.010 (0.009, 0.011) < 0.005, 0.008	0.015, 0.015 < 0.005 (2) < 0.005 (2)	E-96-MK-936-HOP; 072-96-0013R
Germany (Weibensee) 1994	Northern Brewer	23, 21	80% height 71–75	0 28 28	green cones dried cones cones	0.120, 0.101 < 0.005 (2) < 0.005 (2)	0.008, 0.007 < 0.005 (2) < 0.005 (2)	E-96-MK-936-HOP; 072-96-0014R
Germany 1994	Hallertauer Tradition	2× 22	full height	0 14 20 21 27 28	green cones green cones green cones dried	0.231, 0.213 0.011, 0.008 0.008, 0.006 0.029, 0.031 0.006, 0.006 0.021 (0.022, 0.020)	0.026, 0.022 < 0.005 (2) < 0.005 (2) < 0.005 (2) < 0.005 (2) < 0.005 (2)	E-94-MK-936-HOP; 072-94-0005R

Country year	Hop variety	Application rate, g ai/ha	Growth stage BBCH	DAT, days	Crop Part	Residues, mg/kg		Study; trial
						Avermectin B_{1a} + 8,9-Z-isomer	Avermectin B_{1b} + 8,9-Z-isomer	
					cones green cones dried cones			
		24, 22	full height full height	0 14 20 21 27 28	green cones green cones green cones dried cones green cones dried cones	0.441, 0.817 0.022, 0.016 0.010, 0.012 0.031, 0.024 0.007, 0.006 0.022, 0.012	0.049, 0.087 < 0.005 (2) < 0.005 (2) < 0.005 (2) < 0.005 (2)	
Germany 1996	Hop (Perle)	23, 21	full height full height	0 14 20 21 27 28	green cones green cones green cones dried cones green cones dried cones	0.246, 0.292 0.015, 0.011 0.005, 0.006 0.034, 0.029 < 0.005, 0.006 0.025, 0.020	0.026, 0.031 < 0.005 (2) < 0.005 (2) < 0.005 (2) < 0.005 (2)	E-94-MK-936-HOP; 072-94-0007R
		23, 21	full height full height	0 14 20 21 27 28	green cones green cones green cones dried cones green cones dried cones	0.204, 0.348 0.016, 0.009 0.010, 0.006 0.035, 0.036 0.005, 0.006 <u>0.028</u> (0.030, 0.025)	0.021, 0.037 < 0.005 (2) < 0.005 (2) < 0.005 (2) < 0.005 (2)	
Germany 1994	Hop (Perle)	24, 22	full height full height	0 14 20 21 27 28	green cones green cones green cones dried cones green cones dried cones	0.225, 0.307 0.011, 0.018 0.008, 0.010 0.043, 0.041 < 0.005 (2) <u>0.020</u> (0.017, 0.022)	0.024, 0.031 < 0.005 (2) < 0.005 (2) < 0.005 (2) < 0.005 (2)	E-94-MK-936-HOP; 072-94-0006R
		23, 22	full height full height	0 14 20 21 27 28	green cones green cones green cones dried cones	0.400, 0.276 0.014, 0.011 0.010, 0.013 0.046, 0.044 0.006, 0.005 0.017, 0.012	0.036, 0.027 < 0.005 (2) < 0.005 (2) < 0.005 (2) < 0.0025 (2)	

Country year	Hop variety	Application rate, g ai/ha	Growth stage BBCH	DAT, days	Crop Part	Residues, mg/kg		Study; trial
						Avermectin B_{1a} + 8,9-Z-isomer	Avermectin B_{1b} + 8,9-Z-isomer	
					green cones dried cones			
Germany 1994	Hallertauer Mittelfrüh	22, 21	80% of full height full height	0 14 21 22 28 28	green cones green cones green cones dried cones dried cones green cones	0.113, 0.121 < 0.005 (2) < 0.005 (2) 0.004, 0.005 <u>< 0.005</u> (2) < 0.005 (2)	0.010, 0.012 < 0.005 (2) < 0.005 (2) < 0.005 (2) < 0.005 (2) < 0.005 (2)	E-94-MK-936-HOP; 072-94-0008R
		23, 22	80% of full height–full height	0 14 21 22 28 28	green cones green cones green cones dried cones dried cones green cones	0.238, 0.306 < 0.005 (2) < 0.005 (2) 0.004, 0.007 < 0.005 (2) < 0.005 (2)	0.025, 0.030 < 0.005 (2) < 0.005 (2) < 0.005 (2) < 0.005 (2) < 0.005 (2)	
Yakima, WA USA 1994	Galena	2× 21	18 ft	0 27	dried cones dried cones	0.59, 0.73 <u>0.061</u> (0.044, 0.078)	0.059, 0.073 < 0.005, 0.008	618-936-94035; 001-94-1005R
Ganger, WA USA 1994	Cluster	2× 21	early maturity	0 28	dried cones dried cones	0.16, 0.15 <u>0.20</u> (0.017, 0.023)	0.015, 0.015 < 0.005 (2)	618-936-94035; 001-94-1006R
ID, USA 1994	Galena	20, 22	5.2–5.5 m	0 28	dried cones dried cones	0.67, 0.59 <u>0.056</u> (0.055, 0.057)	0.072, 0.064 < 0.005 (2)	618-936-94035; 001-94-1007R
OR, USA 1994	Nugget	22, 21	5.5 m	0 28	dried cones dried cones	0.97, 0.81 <u>0.012</u> (0.009, 0.015)	0.096, 0.081 < 0.005 (2)	618-936-94035; 001-94-1008R

Feed commodities

Some trials from the studies reported previously have include the analysis of feed samples. The results are shown in Tables 84 to 91.

Table 84 Results from supervised trials conducted with abamectin on rice in China (Report AHKW-BG-012-2011). The paddy rice plant is whole plant cut just above soil level (including grain and husk).

Region	Application rate, g ai/ha	DAT (days)	Crop Part	Avermectin B1a + its 8,Z isomer, mg/kg
Anhui Province 2010	20	0.08 0.25 1	paddy plant paddy plant paddy plant	0.361 0.309 0.069

Region	Application rate, g ai/ha	DAT (days)	Crop Part	Avermectin B1a + its 8,Z isomer, mg/kg
		3	paddy plant	0.017
		5	paddy plant	0.010
		7	paddy plant	0.004
		14	paddy plant	0.001
		21	paddy plant	< 0.001
		30	paddy plant	< 0.001
	2x14	14	paddy plant	< 0.001
			husk	< 0.001
		21	paddy plant	< 0.001
			husk	< 0.001
	3× 14	14	paddy plant	< 0.001
			husk	< 0.001
		21	paddy plant	< 0.001
			husk	< 0.001
	2x 20	14	paddy plant	0.002
			husk	0.006
		21	paddy plant	< 0.001
			husk	< 0.001
	3x20	14	paddy plant	0.003
			husk	0.018
		21	paddy plant	< 0.001
			husk	< 0.001
Hunan Province	20	0.08	paddy plant	0.698
		0.25	paddy plant	0.452
		1	paddy plant	0.074
		3	paddy plant	0.025
		5	paddy plant	0.009
		7	paddy plant	0.006
		14	paddy plant	< 0.001
		21	paddy plant	< 0.001
		30	paddy plant	< 0.001
	2× 14	14	paddy plant	< 0.001
			husk	< 0.001
		21	paddy plant	< 0.001
			husk	< 0.001
	3× 14	14	paddy plant	< 0.001
			husk	0.005
		21	paddy plant	< 0.001
			husk	< 0.001
	2× 20	14	paddy plant	< 0.001
			husk	0.006
		21	paddy plant	< 0.001
			husk	< 0.001
	3x 20	14	paddy plant	0.001
			husk	0.009
		21	paddy plant	0.001
			husk	< 0.001
Guangxi Province	20	0.08	paddy plant	0.142
		0.25	paddy plant	0.140
		1	paddy plant	0.086
		3	paddy plant	0.048
		5	paddy plant	0.012
		7	paddy plant	0.004
		14	paddy plant	0.001
		21	paddy plant	< 0.001
		30	paddy plant	< 0.001
	2× 14	14	paddy plant	0.009
		14	husk	< 0.001
		21	paddy plant	< 0.001
		21	husk	< 0.001
	3× 14	14	paddy plant	0.019
		14	husk	< 0.001
		21	paddy plant	< 0.001

Region	Application rate, g ai/ha	DAT (days)	Crop Part	Avermectin B1a + its 8,Z isomer, mg/kg
		21	husk	< 0.001
	2x20	14	paddy plant	0.0171
		14	husk	0.0073
		21	paddy plant	< 0.001
		21	husk	< 0.001
	3x 20	14	paddy plant	0.033
		14	husk	0.018
		21	paddy plant	< 0.001
		21	husk	< 0.001
Anhui Province	20	0.08	paddy plant	1.983
		0.25	paddy plant	1.184
		1	paddy plant	0.272
		3	paddy plant	0.108
		5	paddy plant	0.025
		7	paddy plant	0.006
		14	paddy plant	< 0.001
		21	paddy plant	< 0.001
		30	paddy plant	< 0.001
	2× 14	14	paddy plant	< 0.001
			husk	0.008
		21	paddy plant	< 0.001
			husk	< 0.001
	2× 14	14	paddy plant	0.004
			husk	0.012
		21	paddy plant	< 0.001
			husk	< 0.001
	2x20	14	paddy plant	0.003
			husk	0.008
		21	paddy plant	< 0.001
			husk	< 0.001
	3x20	14	paddy plant	0.009
			husk	0.025
		21	paddy plant	< 0.001
			husk	< 0.001
Hunan Province	20	0.08	paddy plant	0.743
		0.25	paddy plant	0.484
		1	paddy plant	0.080
		3	paddy plant	0.027
		5	paddy plant	0.009
		7	paddy plant	0.007
		14	paddy plant	< 0.001
		21	paddy plant	< 0.001
		30	paddy plant	< 0.001
	2× 14	14	paddy plant	< 0.001
			husk	< 0.001
		21	paddy plant	< 0.001
			husk	< 0.001
	3× 14	14	paddy plant	< 0.001
			husk	0.006
		21	paddy plant	< 0.001
			husk	< 0.001
	2× 20	14	paddy plant	< 0.001
			husk	0.009
		21	paddy plant	< 0.001
			husk	< 0.001
	3× 20	14	paddy plant	0.001
			husk	0.022
	2× 20	21	paddy plant	< 0.001
			husk	< 0.001
Guangxi Province	20	0.08	paddy plant	0.683
		0.25	paddy plant	0.387
		1	paddy plant	0.112
		3	paddy plant	0.107

Region	Application rate, g ai/ha	DAT (days)	Crop Part	Avermectin B1a + its 8,Z isomer, mg/kg
		5	paddy plant	0.021
		7	paddy plant	0.003
		14	paddy plant	< 0.001
		21	paddy plant	< 0.001
		30	paddy plant	< 0.001
	2× 14	14	paddy plant	< 0.001
		14	husk	0.008
		21	paddy plant	< 0.001
		21	husk	0.006
	3× 14	14	paddy plant	0.007
		14	husk	0.010
		21	paddy plant	0.004
		21	husk	0.008
	2× 20	14	paddy plant	0.010
		14	husk	0.009
		21	paddy plant	< 0.001
		21	husk	0.008
	3× 20	14	paddy plant	0.019
		14	husk	0.016
		21	paddy plant	0.006
		21	husk	0.015

Table 85 Results from supervised trials conducted with abamectin on green beans, remaining plant (vines) (CEMS-3913; 2008)

Country	Bean variety	Application rate, g ai/ha	Growth stage	DAT, days	Residues, mg/kg			Trial
					Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
France	Booster	23, 23, 22	65–81	–0	0.279	< 0.002	0.007	S08-00832-01
				0	0.497	< 0.002	0.014	
				1	0.485	< 0.002	0.034	
				3	0.354	< 0.002	0.009	
				7	0.329	< 0.002	0.008	
	Booster	23, 22	65–83	–0	0.270	< 0.002	0.006	
				0	0.803	< 0.002	0.020	
				1	0.478	< 0.002	0.011	
				3	0.255	< 0.002	0.006	
				7	0.231	< 0.002	0.006	
Italy	Oriente	23, 20, 22	76–83	–0	0.031	< 0.002	0.002	S08-00832-02
				0	0.765	< 0.002	0.064	
				1	0.130	< 0.002	0.010	
				3	0.326	< 0.002	0.025	
				7	0.169	< 0.002	0.012	
	Oriente	22, 21	77–83	–0	0.056	< 0.002	0.004	
				0	0.471	< 0.002	0.041	
				1	0.620	< 0.002	0.047	
				3	0.329	< 0.002	0.024	
				7	0.198	< 0.002	0.014	
Spain	Emerite	22, 22, 21	71–85	–0	0.278	< 0.002	0.019	S08-00832-03
				0	0.487	< 0.002	0.012	
				1	0.556	< 0.002	0.040	
				3	0.581	< 0.002	0.040	
				7	0.435	< 0.002	0.031	
	Emerite	2× 22	72, 85		0.165	< 0.002	0.010	
				0	1.019	< 0.002	0.078	
				1	0.514	< 0.002	0.037	
				3	0.413	< 0.002	0.029	
				7	0.364	< 0.002	0.026	
Spain	Killy	20, 22, 22	76-77	–0	0.341	< 0.002	0.025	S08-00832-04
				0	0.572	< 0.002	0.049	
				1	0.531	< 0.002	0.015	
				3	0.349	< 0.002	0.023	
				7	0.250	< 0.002	0.015	

Country	Bean variety	Application rate, g ai/ha	Growth stage	DAT, days	Residues, mg/kg			Trial
					Avermectin B_{1a}	B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	
	Killy	22, 21	76, 77	–0 0 1 3 7	0.162 0.733 0.350 0.290 0.161	< 0.002 < 0.002 < 0.002 < 0.002 < 0.002	0.011 0.063 0.024 0.019 0.010	

Table 86 Results from supervised trials conducted with abamectin on almonds in the USA, showing the residues in almond hulls

Region year	Almond variety	Application rate, g ai/ha	Growth stage	DAT, days	Residues, mg/kg		Study; trial
					Avermectin B_{1a}+ 8,9-Z-isomer	B_{1b} + 8,9-Z-isomer	
Fresno, CA 1988	Non Pareil	3× 28	hull split	0 21	0.006, 0.005, 0.009, 0,016 <u>< 0.002</u> (4)	< 0.002 (4) < 0.002 (4)	618-936-TRN; 001-88-6028R
		3× 58	hull split	0 21	0.026, 0.022, 0.048, 0.041 < 0.005 (4)	< 0.005 (4) < 0.002 (4)	
Madera, CA 1988	Non Pareil	3× 28	hull split	0 3 7 14 21	0.218, 0.225 0.238, 0.266 0.095, 0.046 0.078, 0.070 0.083, 0.055 0.053, 0.061 0.037 (2), 0.046, 0.047 <u>0.035</u> (0.042, 0.030 (2), 0.038)	0.021, 0.027 0.025, 0.030 0.010, 0.005 0.010, 0.008 0.009, 0.007 0.007, 0.007 < 0.005 (4) < 0.005 (4)	618-936-TRN; 001-88-6032R
		3× 56	hull split	0 3 7 14 21	0.536, 0.642, 0.598, 0.676 0.233, 0.235, 0.305, 0.334 0.142, 0.193, 0.232, 0.178 0.144, 0.114, 0.190, 0.194 0.080, 0.107, 0.149, 0.166	0.063, 0.067, 0.066, 0.072 0.280 (2), 0.037, 0.038 0.014, 0.021(2), 0.026 0.016, 0.013, 0.020, 0.022 0.008, 0.011, 0.018 (2)	
Stanislaus, CA 1988	NonPareil	3× 28	hull split Post hull Split	0 21	0.264, 0.321, < 0.306, 0.347 <u>0.110</u> (0.070, 0.055, 0.032, 0.281)	0.030, 0.034, 0.280, 0.035 0.007, 0.006, < 0.005 (2)	618-936-TRN; 001-88-6034R
		3× 56	hull split Post hull Split	0 21	0.571, 1.096, 0.749, 1.029 0.157, 0.122, 0.098, 0.136	0.052, 0.104, 0.071, 0.100 0.016, 0.012, 0.010, 0.013	
Stanislaus, CA 1988	NonPareil	3× 28	hull split	0 21	0.064, 0.201, 0.010, 0.179 <u>0.037</u> (0.031, 0.053, 0.026, 0.041)	0.007, 0.022, 0.012, 0.019 < 0.005 (3), 0.006	618-936-TRN Trial: 001-88-6035R
		3× 56	hull split	0 21	0.198, 0.261, 0.220, 0.619 0.088, 0.113, 0.116, 0.216	0.022, 0.281, 0.023, 0.068 0.008, 0.011, 0.015, 0.023	
Colusa, CA 1989	Mission	3× 28	hull split	0 14 21	0.108, 0.091, 0.046, 0.101 0.016, 0.018, 0.011, 0.017 <u>0.012</u> (0.012, 0.013, 0.010 0.016)	0.030, 0.015 (2), 0.006 < 0.002 (4) < 0.002 (4)	618-936-TRN Trial: 001-89-6019R

Region year	Almond variety	Application rate, g ai/ha	Growth stage	DAT, days	Residues, mg/kg		Study; trial
					Avermectin B_{1a}+ 8,9-Z-isomer	B_{1b} + 8,9-Z-isomer	
Kern, CA 1989	Mission	3× 28	hull split	0 14 21	0.101, 0.204, 0.162, 0.174 0.029, 0.052, 0.021, 0.046 0.102 (0.280, 0.006, 0.021)	0.013, 0.026, 0.020, 0.022 0.005, 0.007, < 0.005, 0.008 < 0.005 (2), < 0.002	618-936-TRN Trial: 001-89-6020R

Table 87 Results from supervised trials conducted with abamectin on cotton hulls in Europe

Country year	Cotton variety	Application rate, g ai/ha	Growth stage (BBCH)	DAT, days)	Residue Found (mg/kg)				Study; trial
					Avermectin B_{1a}	Avermectin B_{1a} 8,9-Z-isomer	Avermectin B_{1b}	Total residue	
Greece 1999	Stoneville	2× 18	81–83	0 20	0.005(2) < 0.005 (2)	0.005 (2) < 0.005 (2)	0.005 (2) < 0.005 (2)	0.015 < 0.015	1104/99
Greece 1999	Stoneville	2× 18	81–82	0 20	0.008(2) < 0.005(2)	< 0.005 (2) < 0.005 (2)	< 0.005 (2) < 0.005 (2)	0.018 < 0.015	1105/99
Spain 1999	Crema 11	17, 18	87–89	0 20	0.007,< 0.005 < 0.002(2)	< 0.005 (2) < 0.002 (2)	< 0.005 (2) < 0.002 (2)	0.016 < 0.006	1114/99
Spain 1999	Carmen	2× 18	87–89	0 3 7 14 20	0.014 < 0.005 < 0.005 < 0.005 < 0.005 (2)	< 0.005 < 0.005 < 0.005 < 0.005 < 0.005 (2)	< 0.005 < 0.005 < 0.005 < 0.005 < 0.005 (2)	0.024 < 0.015 < 0.015 < 0.015 < 0.015	1115/99
Greece 2000	Stoneville	2× 18	83–87	0 3 7 14 20	0.007 < 0.005 < 0.005 < 0.005 < 0.005 (2)	< 0.005 < 0.005 < 0.005 < 0.005 < 0.005 (2)	< 0.005 < 0.005 < 0.005 < 0.005 < 0.005 (2)	0.017 < 0.015 < 0.015 < 0.015 < 0.015	1046/00; Mavrogia
Greece 2000	Stoneville	2× 18	83–87	0 3 7 14 20	0.007 < 0.005 < 0.005 < 0.005 < 0.005 (2)	< 0.005 < 0.005 < 0.005 < 0.005 < 0.005 (2)	< 0.005 < 0.005 < 0.005 < 0.005 < 0.005 (2)	0.017 < 0.015 < 0.015 < 0.015 < 0.015	1047/00; Ippodromos
Spain 2000	Crema	2× 18	87	0 3 7 14 20	0.009 < 0.005 < 0.005 < 0.005 < 0.005 (2)	< 0.005 < 0.005 < 0.005 < 0.005 < 0.005 (2)	< 0.005 < 0.005 < 0.005 < 0.005 < 0.005 (2)	0.019 < 0.015 < 0.015 < 0.015 < 0.015	1088/00
Spain 2000	Crema	2× 18	87	0 3 7 14 20	0.010 < 0.005 < 0.005 < 0.005 < 0.005 (2)	< 0.005 < 0.005 < 0.005 < 0.005 < 0.005 (2)	< 0.005 < 0.005 < 0.005 < 0.005 < 0.005 (2)	0.020 < 0.015 < 0.015 < 0.015 < 0.015	1089/00; Alcalá del Río

Fate of Residues in Processing

Four processing studies were conducted with grapes, yielding raisins, pomace, and juice, and two in plums, yielding prunes. The results are shown in Table 89. All the studies were conducted within the supervised trials. Grape processed commodities were analysed within a month after being produced.

Table 88 Processing studies of abamectin in grapes and plums

Matrix	Avermectin B_{1a} + 8,9-Z-isomer, mg/kg (mean)	Avermectin B_{1b} + 8,9-Z-isomer, mg/kg (mean)	Total residue, mg/kg	Processing factor	Study; trial
Grape fruit	0.010	< 0.002	0.012		618-244-94036; 001-94-5006R
washed fruit	0.013	< 0.002	0.015	1.25	
raisin	0.0095	< 0.002	0.012	1	
juice	< 0.002	< 0.001	< 0.003	< 0.25	

Matrix	Avermectin B_{1a} + 8,9-Z-isomer, mg/kg (mean)	Avermectin B_{1b} + 8,9-Z-isomer, mg/kg (mean)	Total residue, mg/kg	Processing factor	Study; trial
pomace, wet	0.052	0.006	0.057	4.75	
pomace, dry	0.164	0.018	0.189	15.8	
waste	0.0121	0.001	0.013	1.1	
waste	0.022	0.002	0.024	2	
Grape fruit	0.0053	< 0.002	0.007		T005598-07; E03NY081041
raisin	0.020	< 0.002	0.022	3.1	
juice	< 0.002	< 0.002	< 0.004	< 0.57	
Grape fruit	0.046	< 0.002	0.048		T005598-07; W26CA081043
raisin	0.133	< 0.002	0.135	2.8	
juice	0.067	< 0.002	0.069	1.4	
Plum	0.0035	< 0.001	0.005		ABR-98073; 001-96-4011R
prune	0.003	< 0.001	0.004	0.8	
Plum	< 0.001	< 0.001	< 0.002		ABR-98073; 001-96-4014R
prune	0.003	< 0.001	0.004	2	

Eleven processing studies were conducted with cotton, four in Europe and two in USA. The results are shown in Table 89. All the studies were conducted within the supervised trials for the main crop. Processing factors were not calculated when residues in the raw commodity was < LOQ.

Table 89 Results from processing studies conducted with abamectin on cotton

Matrix	Avermectin B_{1a} + 8,9-Z-isomer, mg/kg (mean)	Avermectin B_{1b} + 8,9-Z-isomer (mean)	Total abamectin, mg/kg	Processing factor	Study; trial
Seed	< 0.004	< 0.002	< 0.006		1104/99
press cake	< 0.004	< 0.002	< 0.006	1	
crude oil	< 0.004	< 0.002	< 0.006	1	
Seed	< 0.004	< 0.002	< 0.006		1105/99
press cake	< 0.004	< 0.002	< 0.006	1	
crude oil	< 0.004	< 0.002	< 0.006	1	
Seed	< 0.004	< 0.002	< 0.006		1046/00
press cake	< 0.004	< 0.002	< 0.006	1	
crude oil	0.002	< 0.002	0.006	–	
Seed	< 0.004	< 0.002	< 0.006		1047/00
press cake	< 0.004	< 0.002	< 0.006	1	
crude oil	< 0.004	< 0.002	< 0.006	1	
Seed	0.004	< 0.002	0.006		T005597-07;
meal	< 0.002	< 0.002	< 0.004	< 0.67	W07TX081024
refined oil	< 0.002	< 0.002	< 0.004	< 0.67	
Seed	< 0.002	< 0.002	< 0.004		
gin trash	0.015	< 0.002	0.017	–	
Seed	0.012	< 0.002	0.014		T005597-07;
meal	< 0.002	< 0.002	< 0.004	< 0.028	E13TX081026
refined oil	< 0.002	< 0.002	< 0.004	< 0.028	
Seed	0.005	< 0.002	0.007		
gin trash	0.121	0.002	0.123	–	
Seed	< 0.02	< 0.002	< 0.004		T005597-07;
gin trash	0.010	< 0.002	0.013	–	C24AR081022
Seed	< 0.02	< 0.002	< 0.004		T005597-07;
gin trash	0.012	< 0.002	0.014	–	W39TX081025
Seed	< 0.002	< 0.002	< 0.004		T005597-07;
gin trash	0.014	< 0.002	0.017	–	E13TX081027
Seed	0.011	< 0.002	0.013		T005597-07;
gin trash	0.625	0.0035	0.63	48.5	W30CA081028
Seed	< 0.002	< 0.002	< 0.004		TK0023918;
gin trash	0.0785	< 0.002	0.080	–	W07-0012

Livestock feeding studies

A feeding study in dairy cows was performed (Wehner, 1986). Twelve lactating Holstein cows were assigned to four dosing level groups (0, 0.01, 0.03 and 0.10 ppm), administered daily in gelatin capsules for 28–30 days. Milk samples were collected pre-dose, Day 1 (a.m. and p.m.), 2, 3, 5, 7, 14, and 28 (a.m. and p.m.) and liver, kidney, fat, muscle collected at sacrifice. Milk and tissue samples were analysed by HPLC-FL for avermectin B_{1a}, with an LOQ of 0.0005 mg/kg in milk and 0.01 mg/kg in tissues. The results are shown in Table 90. Levels of avermectin B_{1a} were highest in liver at all three feeding rates.

Table 90 Avermectin B_{1a} residues in tissues of treated cows

Matrix	Feeding level, ppm	Range, mg/kg	Mean, mg/kg
Muscle	0.10	0.002–0.002	0.002
Muscle	0.03	0.002–0.002	0.002
Muscle	0.01	0.001–0.002	0.002
Fat	0.10	0.0098–0.014	0.012
Fat	0.03	0.004–0.006	0.005
Fat	0.01	0.002–0.002	0.002
Liver	0.10	0.018–0.020	0.019
Liver	0.03	0.005–0.0076	0.0065
Liver	0.01	0.003–0.004	0.003
Kidney	0.10	0.004–0.005	0.004
Kidney	0.03	0.002–0.002	0.002
Kidney	0.01	0.001–0.002	0.001

Residues in control was 0.001 mg/kg in liver, fat and kidney and < 0.001 mg/kg in muscle

Residues of avermectin B_{1a} in milk are shown in Table 91. Maximum residues in milk at the highest feeding rate reached 0.004 mg/kg (Day 14).

Table 91 Residues of avermectin B_{1a} in milk from treated cows

	0.01 ppm (1×)		0.03 ppm (3×)		0.10 ppm (10×)	
Sampling time	Mean	Maximum	Mean	Maximum	Mean	Maximum
Pre-dose a.m.	–	–	–	–	(< 0.0005)	(< 0.0005)
Pre-dose p.m.	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005
Day 1 a.m.	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005
Day 1 p.m.	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005
Day 2 p.m.	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	0.001
Day 3 p.m.	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	0.001
Day 5 p.m.	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	0.001
Day 7 p.m.	< 0.0005	< 0.0005	< 0.0005	< 0.0005	0.001	0.002
Day 14 p.m.	< 0.0005	< 0.0005	< 0.0005	< 0.0005	0.002	0.004
Day 28 a.m.	< 0.0005	< 0.0005	< 0.0005	< 0.0005	0.001	0.001
Day 28 p.m.	< 0.0005	< 0.0005	< 0.0005	< 0.0005	0.001	0.001
Overall	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	0.004

Results in brackets are single determinations

APPRAISAL

Abamectin is a broad-spectrum acaricide with additional insecticidal action on a limited number of insects. Abamectin was firstly evaluated by JMPR in 1992 (T,R), and was scheduled at the Forty-sixth Session of the CCPR (2014) for the periodic re-evaluation of toxicology and residues by the 2015 JMPR. For the residue evaluation, data were submitted on physical and chemical properties, environmental fate, metabolism on plants and lactating goats, analytical methods, GAP, supervised trials on fruits, vegetables, nuts, beans, coffee, cotton and cereals, processing studies and cow feeding studies.

Abamectin is a mixture containing ≥ 80% avermectin B_{1a} and ≤ 20% avermectin B_{1b}. The absolute stereochemistry of both compounds is known and defined at each chiral centre and stereogenic carbon-carbon double bond by their IUPAC nomenclature. Abamectin (> 98% purity) has a low solubility in water (1.2 mg/L at 7.6 pH and 25 °C), is soluble in most organic solvents (23 g/L in toluene up to 470 g/L in ethyl acetate) and has a log K_{ow} of 4.4.

Abamectin is also used as an anthelmintic drug in veterinary medicine. The JECFA residue definition for the compound is avermectin B_{1a}.

The abamectin structures and the main metabolites and degradates found in water, soil, plants and animals are shown below.

<table>
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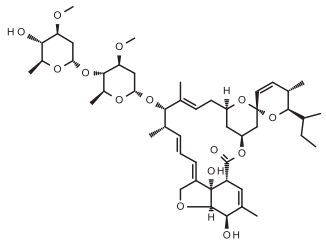
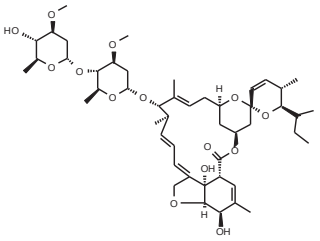
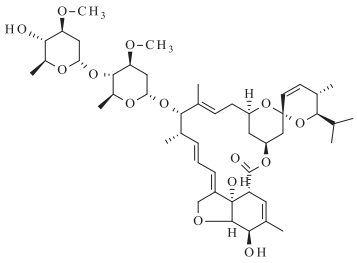
<td> 
Avermectin B_{1a}</td>
<td> 
8,9-Z isomer of avermectin B_{1a}</td>
<td> 
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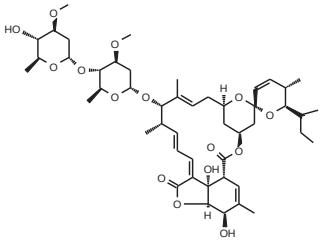
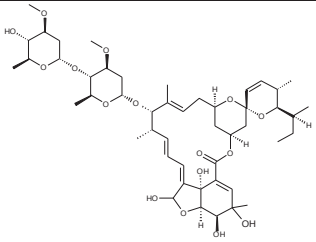
<td> 
8α-oxo-avermectin B_{1a}</td>
<td> 
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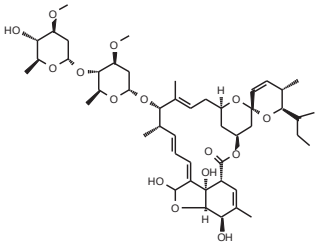
<td> 
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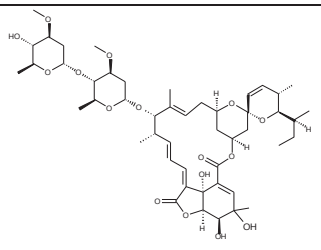
<td> 
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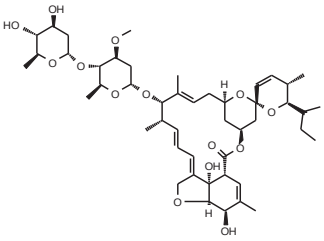
<td> 
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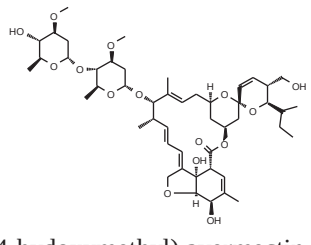
<td> 
(24-hydoxymethyl) avermectin B_{1a}</td>
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Environmental fate

Various studies were conducted to evaluate the aerobic degradation of [^{14}C- an/or ^{3}H-] avermectin B_{1a} in different non-sterile soils in the dark under various conditions (application rate, temperature and water capacity) over a period of up to 196 days. Avermectin B_{1a} degraded in soils with a half-life

ranging from 12 to 52 days, and a mean of 29 ± 14 days (n=14). The degradation pathway occurs via hydroxylation or oxidation in the C-8α position, with 8α-hydroxy-avermectin B_{1a} being the major metabolite (up to 18% of the applied radioactivity, AR), present as an equilibrium mixture between the hemiacetal and the ring cleaved aldehyde form. The oxidation product 8α-oxo-avermectin B_{1a} was found at a maximum of 14% AR. Further hydroxylation in the C-4 position resulted in two additional identified metabolites, 4,8α-dihydroxy-avermectin B_{1a} and 8α-oxo-4-hydroxy-avermectin B_{1a}, each at < 10% AR. 4,8α-dihydroxy-avermectin B_{1a} is also present in an equilibrium mixture as the hemiacetal and the aldehyde forms. At least 25 other residues were also formed at low levels, each representing < 10%. The non-extracted residues and volatile fractions (CO_2), reached their maximum at the end of the incubation period (44 and 28% AR, respectively). About 6% AR was released by harsh extraction of non-extracted residues, mostly humic, fulvic and humin acids, with only minor amounts identified as avermectin B_{1a}.

Soil photolysis studies demonstrated a similar degradation pattern, except that under the influence of light, avermectin B_{1a} initially isomerises to the 8,9-Z isomer before degrading, mainly to 8α-hydroxy-avermectin B_{1a} and 8α-oxo-avermectin B_{1a} (up to 4.7% AR). The half-life in these studies were 21–22 days. Photolysis significantly increases the rate of degradation of avermectin B_{1a}, as the dark controls showed a half-life of 119 days.

[^{3}H-avermectin B_{1a}] was stable to hydrolysis at pH 4 to 7 under sterile conditions, minimal hydrolysis was observed at pH 9 (DT_{50} of 380 days at 20 °C), with one major transient non-polar degradate 2-epi-avermectin B_{1a} being observed. At 60 °C, this degradate reached a maximum of 25%AR by Day 11 and then degraded with a DT_{50} of 1.5 days. [23-^{14}C-avermectin B_{1a}] degraded in water under light to 8,9-Z avermectin B_{1a} and 8α-oxo-avermectin B_{1a} (half-lives < 6 days).

In summary, avermectin B_{1a} degrades relatively fast in soils, with half-life < 60 days, and 8α-hydroxy- and 8α-oxo- avermectin B_{1a} being the major products. Light accelerates the degradation in water and soil, and isomerises the compound to its 8,9-Z isomer. Aqueous hydrolysis is not a significant degradation route for avermectin B_{1a} at environmentally relevant pHs and temperatures.

Plant metabolism

The metabolism of [^{14}C]avermectin B_{1a} was investigated in citrus plants kept under an open wooden frame with a fibreglass roof and treated at 18 to 40 µg ai/kg on a whole fruit basis. The [^{14}C]avermectin B_{1a} solutions, prepared in a EC formulation blank, was brushed on each fruit (0.5 mL). After 12 weeks of treatment, residues ranged from 33.3% (grapefruit) to 49.8% (lemons) of the AR. On the day of application, at least 98.4%AR was removed from the surface with methanol, and by week 12, surface residues corresponded to up to 41%TRR in oranges. No residues were detected in the pulp without the peel/pulp interface for all fruits; when the interface was included, residues reached 12–13%TRR after 8 weeks. At day 0, at least 85% TRR of the methanol rinse and acetone peel extract was avermectin B_{1a}, the level then decreased rapidly after one week (to 4.4 to 17.4%TRR) and ≤ 7.7% TRR after 12 weeks, when polar residues accounted for at least 46% TRR. The 8,9-Z isomer of avermectin B_{1a} was present in all sample extracts (0.7–4.7%TRR). Non extracted residues ranged from 40–62% TRR at week 12, but were reduced to < 10%TRR after successive treatments (Bligh-Dyer procedure, soxhlet with methanol and acid or enzyme hydrolysis). Most of the non-extracted residues were polar degradates, with avermectin B_{1a} representing 9–12% TRR, and a fraction identified as a mixture of linoleic fatty esters.

The metabolism of avermectin B_{1a} was investigated in celery in three field experiments:

1) plants treated with ^{3}H-avermectin B_{1a} at 11.2 g ai/ha

2) at 112 g ai/ha, with immature plants harvested from 0 to 43 days after the 4th application and mature plants harvested at 0 to 22 days after the 10th application

3) plants treated with [^{14}C]avermectin B_{1a} at 16.8 g ai/ha, with immature plants harvested at 0 and 14 days after the 4th application and mature plants harvested at 0 to 7 days after the 10th application.

In general, residues in immature or mature leaves and stalks decreased significantly during the study period. For example, after the 4th application at 11.2 g ai/ha, residues in immature leaves were 2.74 mg/kg eq, decreasing to 11.5 µg/kg eq 43 days later. Acetone extracts accounted for over 95% TRR in immature leaves after the 4th application at all rates, with avermectin B_{1a} accounting for 65–75% of the extracted residue. After 14 days, leaf acetone extracts were about 80%TRR, with avermectin B_{1a} accounting for 16–26% of the residues and the 8,9-Z isomer for about 5%. In general, stalks and mature leaves showed similar profiles. The 8-hydroxy avermectin B_{1a} and at least ten other unidentified minor components were also detected in the samples. Residual solids from the leaf acetone extract were mostly extracted with methanol/water and hot DMSO, being mostly polar degradates of avermectin B_{1a}. About 15% of the acetone non-extracted residues in the leaves were incorporated into glucose.

The metabolism of [^{14}C]avermectin B_{1a} was investigated in cotton in four field experiments:

1) individual leaves treated with 100 µg of [^{14}C]avermectin B_{1a} and analysed 8 days after treatment (DAT)

2) cotton plants received two foliar applications at 20 g ai/ha (100 L/ha) and mature bolls harvested at 8 DAT

3) cotton plants were grown in buckets under normal field conditions and treated three times by foliar spray at 22.4 g ai/ha

4) 3× 224 g ai/ha (467 L/ha), and the bolls harvested at 20 DAT.

Over 99.7%AR in the leaves from Experiment 1 were extracted with methanol at day 0, decreasing to 19.3% at Day 8. Avermectin B_{1a} accounted for 99.2%AR at Day 0 and 1.7% AR after 8 days. Non-extracted residues reached 26.1%AR at Day 4. Leaves from Experiments 2 to 4 contained the highest residues (up to 400 µg/kg). Seeds contained up to 85 µg/kg and lint up to 750 µg/kg; this very high level was probably due to the last application in Experiment 4, when approximately 50% of the bolls were open. Avermectin B_{1a} represented most of residues in the leaves methanol rinse from the Experiment 3, accounting for 36% AR at day 1, which decreased to 1% AR by Day 8. The 8,9-Z isomer accounted for 7% AR at 0.25 day, decreasing to 0.1% AR at Day 8. From 26 to 35% TRR in the cotton seed (Experiments 2 to 4) was extracted with hexane, and characterized as triglycerides (linoleic and palmitic acid). Methanol extracts accounted for 50 to 65% TRR and non-extracted material for up to 25% TRR (Experiment 2).

One study was conducted to compare the profile of the residues of [^{14}C]avermectin B_{1a} *in vivo* (citrus, celery and cotton) and *in vitro* photolysis conditions. In this study, a [^{14}C]avermectin B_{1a} methanol solution was dried at room temperature and placed under a 275W Suntanner bulb. Most of the residues in the cotton leaf and citrus fruit surface were of a polar nature, with avermectin B1a accounting for 5–11% TRR after 7–8 days. In stalk and leaf extracts, avermectin B_{1a} accounted for 17 and 10% TRR at 7 DAT, respectively. The *in vitro* study also showed a major decline of avermectin B_{1a} residues with time (from 37% TRR after 19 hours of exposure to light to 7.3% TRR after 30 hours). Re-chromatography of the polar residues from the three treated crops and in the photolysis experiment showed four broad peaks of multiple-oxygenated, hydrated or dehydrated and de-methylated species, which retained little of the macrocyclic characteristics of avermectin B_{1a}.

Metabolism of avermectin B_{1a} was studied in greenhouse-grown tomato plants treated with [^{14}C]avermectin B_{1a} at 5× 26 g ai/ha (sub-study 1) and 3× 281 g ai/ha (sub-study 2). The major metabolite fractions in all of the analysed samples were avermectin B_{1a} and the 8,9-Z isomer of avermectin B_{1a}, in a ratio of approximately 9:1. TRR at 28 DAT in tomato and leaves from sub-study 1 were 0.127 and 6.4 mg/kg eq., respectively, with 51 and 34% as avermectin B_{1a} + its 8,9-Z isomer (9:1), respectively. In sub-study 2, the parent compound and its isomer accounted for 75 and 50% of the residues found in tomato and leaves, respectively. 8α-oxo-avermectin B_{1a}, 8α-hydroxy-avermectin B_{1a}, and 3"-O-desmethyl-avermectin B_{1a} were present at levels < 8% TRR in tomato and leaves samples. The non-extracted radioactivity did not exceed 2% TRR in tomato fruit and 7%TRR in the leaves.

In a field study conducted at 5× 26 g ai/ha or 5× 246 g ai/ha, total residues in tomatoes were 0.017 and 0.108 mg/kg, respectively, with avermectin B_{1a} + its 8,9-Z isomer accounting for 7.1 and 25%TRR, and the 8α-oxo- and 8α-hydroxy- metabolites for less than 3%TRR. In leaves, total residues were 0.71 and 7.8 mg/kg, respectively, with avermectin $B_{1a\ and}$ its isomer accounting for 2.2 and 6.4%TRR and the two metabolites up to 1.2%TRR.

Metabolism of avermectin B_{1a} was investigated in field-grown tomatoes under similar conditions as the greenhouse studies. The major metabolite fraction in all of the analysed samples was avermectin B_{1a} and its 8,9-Z isomer, accounting for about 70–80%TRR at 0 days and decreasing over time (2–6% TRR 28 days after the 5th application). Other identified metabolites were 8α-oxo-avermectin B_{1a}, 8α-hydroxy-avermectin B_{1a}, and 3"-O-desmethyl-avermectin B_{1a}, present at levels < 7% TRR each in tomatoes and leaves at any sampling time in both experiments.

In a confined rotational crop study conducted in the field, sorghum, lettuce and carrots or turnips were planted in sandy, sandy loam and "muck" (high-organic drained swampland) soils. The soils were filled into large tubes and treated at 135 to 155% of the maximum label rate of 21.3 g ai/ha. The sandy soil received 3× 29.1 g ai/ha and sandy loam and muck soils 12× 33.6 g ai/ha. Sorghum and lettuce were planted in all soil types, turnip in the muck soil and carrot in the sand and sandy loam soils. The plant-back intervals (PBI) were 14, 123 and 365 days for the muck soil, 31, 120 and 365 days for the sandy soil and 29, 123 and 365 days for the sandy loam soil. The highest TRR was found in the lettuces samples from the muck soil (6.9 μg/kg eq.), from which extraction with acetone released only 4.4%TRR. Sorghum leaf-stem TRR ranged from 4 to 12 μg/kg eq. No identification of the residues were performed due to the low TRR levels in all samples.

In summary, the plant metabolism studies conducted in citrus, cotton, celery and tomatoes showed that the residues of avermectin B_{1a} are not significantly translocated into the plants, remaining on the surface, where it is photodegraded to its 8,9-Z isomer. The major proportion of the residues remains parent avermectin B_{1a}. The metabolism pathway include the re-arrangement to the 8,9-Z isomer, hydroxylation to 8α-hydroxy-avermectin $B_{1a,}$ further oxidation to 8α-oxo-avermectin B_{1a}, demethylation to 3"-O-desmethyl-avermectin B_{1a}, and oxidation of the 8α-hydroxy- to form the 4"-oxo-avermectin B_{1a} and 4"-,8α-di-oxo-avermectin $B_{1a.}$ The lack of uptake of radioactive material in succeeding crops indicates the non-systemic behaviour of avermectin B_{1a} and its soil degradates.

Animal metabolism

The metabolism of ^{3}H- and ^{14}C-radiolabelled abamectin B_{1a} in rats was evaluated by the WHO group. In summary, the metabolism of avermectin B_{1a} in the rat proceeded predominantly via demethylation, hydroxylation, cleavage of the oleandrosyl ring, and oxidation reactions. Unchanged avermectin B_{1a} and the metabolites 3"-O-desmethyl, 24-hydroxymethyl, 27-hydroxymethyl, 3"-O-desmethyl-24-hydroxymethyl and 3"-O-desmethyl-27-hydroxymethyl abamectin B1a represented the majority of the faecal radioactivity.

One goat metabolism study was submitted to the meeting. Six lactating goats were dosed daily for ten consecutive days with ^{3}H-avermectin B_{1a} at 0.00125 (D1), 0.0125 (D2) and 0.25 ppm (D3) (two animals per dose) and sacrificed after 24 hours. Urine and faeces were collected daily and goats were milked twice daily. The majority of the radioactivity was found in the faeces (79 to 98% AR). Milk residues plateaued by day 4–6 and were dose dependent (0.34 and 2.6 μg/kg eq. at D2 and D3, respectively). In tissues, highest residues were found in liver (mean of 0.4, 2.8 and 57.2 μg/kg eq. at D1, D2 and D3, respectively), fat (< 0.2, 1.8 and 40.9 μg/kg eq.) and kidney (0.3 to 13.8 μg/kg eq.). In muscle, residues were < 0.2, 0.32 and 5.2 μg/kg eq. Avermectin B_{1a} was the major residue in all tissues, comprising from 41–95%TRR in liver, 40–97%TRR in kidney, 73 to 96%TRR in muscle, 86–99% in fat, and 70–95%TRR in milk. Metabolite 24-hydroxymethyl-avermectin B_{1a} was a major residue in liver of the D1 goats (45.5%TRR) and was present at 2–11% TRR in milk from D3. A second metabolite, 3"-desmethyl-avermectin B_{1a}, was only isolated from Goat 5 liver (≤ 5% TRR). Fat tissue was shown to contain 24-hydroxymethyl avermectin B_{1a} in a conjugated form.

Based on the structures identified, the metabolism of avermectin B_{1a} in the goat proceeds via hydroxylation of the methyl group to 24-hydroxymethyl-avermectin B_{1a} and to a lesser extent demethylation at the 3" position. Avermectin B_{1a} is the major residues in all animal matrices. The metabolic pathway in rats showed a similar profile.

Methods of residue analysis

Abamectin residues in plant materials are analysed by two methods, one by HPLC with fluorescent detector (HPLC-FL; Exc.: 365 nm, Em.: 470 nm) and the other, used in more recent supervised trials, by LC-MS/MS. Transition ions for avermectin B1a and its isomer ($[M+Na]^+$) were m/z = 895.5 → 751.5 for quantification and m/z = 895.5 → 449.2 for confirmation.

In the HPLC-FL method, residues are extracted with acetonitrile or methanol and partitioned with hexane, the organic extract is cleaned-up in an aminopropyl solid phase extraction (SPE), and residues eluted with ethyl acetate/methanol. Fluorescent derivatives are formed by reaction with a mixture of triethylamine, trifluoroacetic anhydride and 1-methylimidazole and determined by HPLC-FL. Avermectin B_{1a} and its 8,9-Z isomer results in a single peak, and is determined as the sum of both compounds. It is the same for avermectin B_{1b} and its 8,9-Z isomer. The LOQ for the individual analytes were 0.002 or 0.005 mg/kg for most studies.

The LC-MS/MS methods quantify individually avermectin B_{1a}, avermectin B_{1b} and their 8,9-Z isomers. Residues are extracted with acetonitrile or methanol, partitioned into toluene and cleaned-up using aminopropyl, amino or C8 SPE (LOQ of 0.002 to 0.01 mg/kg), or only extracted with dichloromethane before the analysis (LOQ of 0.02 mg/kg). The method that included the clean-up step was also validated for avermectin B_{1a}, and its 8,9-Z isomer in animal matrices (LOQ of 0.002 mg/kg).

An LC-MS/MS multi-residue QuEChERS method for the determination of residues of avermectin $B_{1a,}$ avermectin B_{1b} and avermectin B_{1a} 8,9-Z isomer in lettuce, sunflower seeds, dried broad beans, wheat grain, oranges and dried hops was validated at the LOQ of 0.002 mg/kg.

Stability of residues during storage

Residues of avermectin B_{1a} in citrus peel samples fortified at levels of 0.005 or 0.025 mg/kg were stable for at least at 52 months when stored at ≤ -10 °C. Residues of avermectin B_{1a} (0.01 or 0.05 mg/kg), avermectin B_{1b} (0.004 mg/kg) and avermectin B_{1a} 8,9-Z isomer (0.009 mg/kg) were shown to be stable in tomato samples for at least 15 months, in celery and strawberry samples for at least 24 months and in pear samples for at least 35 months. Residues of the three analytes at 0.04 mg/kg were shown to be stable for at least 24 months at ≤ -18 °C when present in orange peel, green beans, sunflower seeds and potatoes. Residues of avermectin B_{1a} and its 8,9-Z isomer (0.02 mg/kg) in grapes and processed commodities were shown to be stable for at least one year under frozen conditions, with the exception of raisins, for which only 28% of avermectin B_{1a} residues remained after 12.5 years.

In summary, avermectin B_{1a} and its 8,9-Z isomer and avermectin B_{1b} were shown to be stable for at least 12 months in a variety of crop samples stored under frozen conditions, except raisins. The storage period of the samples in the residue trials guarantee the stability of the residues, unless it is specified otherwise.

Residue definition

Plant metabolism field studies conducted with ^{14}C and/or ^{3}H-avermectin B_{1a} in citrus, cotton, celery and tomatoes (also glasshouse studies) have shown that the major residue is avermectin B_{1a} (over 20% TRR), which remains on the surface of the crop and isomerizes to the 8,9-Z isomer. When present, the hydroxyl, oxo and desmethyl metabolites each accounted for < 10%TRR. Significant residues in rotational crops are not expected.

Abamectin is a mixture of ≥ 80% avermectin B_{1a} and ≤ 20% avermectin B_{1b}. In most residue trials, avermectin B_{1b} was found at levels < LOQ, and when present, the levels are significantly lower than avermectin B_{1a}. Hence, avermectin B_{1a} is an adequate marker for the use of abamectin products.

Although the HPLC-FL method used to analyse abamectin residues measure avermectin B_{1a} plus its 8,9-Z isomer together, the isomer is not expected to be a significant part of the residue (one study in tomato estimated a 9:1 ratio of both compounds) and was never detected in trials when the LC-MS/MS method was used. The toxicity of 8,9-*Z* isomer of abamectin B_{1a} is of no greater toxicity than the parent abamectin B_{1a}.

The Meeting agreed for the following residue definition for abamectin in plant commodities for enforcement and dietary risk assessment:

Avermectin B_{1a}

The metabolism of avermectin B_{1a} in lactating goats showed the parent compound as the main residue in all matrices (at least 40%TRR), with only one major metabolite (24-hydroxymethyl-avermectin B_{1a}), which accounted for 45.5%TRR in livers of the low dosed goats (0.00125 ppm) and up to 11% TRR in milk. The toxicity of 24-hydroxymethyl-avermectin B_{1a} is of no greater toxicity than the parent abamectin B_{1a}.

The Meeting agreed for the following residue definition for abamectin in animal commodities for enforcement and dietary risk assessment: Avermectin B_{1a}

Residues of avermectin B_{1a} are five times higher in fat than in muscle and the log K_{OW} is 4.4, which indicates fat solubility.

The residues are fat soluble.

Residues resulting from supervised residue trials on crops

As no trials were submitted on summer squash and watermelon, the Meeting withdraws its previous recommendations for these commodities

Citrus fruits

In the USA, GAP for abamectin in citrus is up to three applications at a maximum rate of 26 g ai/ha (max. of 53 g ai/ha per season), and 7 days PHI. Twenty one trials were conducted in the USA in citrus (grapefruit, orange, tangelo and lemon).

In nine trials conducted in oranges at GAP, abamectin residues at 7 days PHI were < 0.005 (6), 0.008, 0.010 and 0.014 mg/kg. The highest residue in a replicate samples was 0.015 mg/kg.

In two trials conducted at GAP in grapefruit, one in tangelos and one in lemons, residues were < 0.005 (4).

The median residues found in the different crops is the same, which allows the consideration of a group estimation. However, the residue populations are not similar, with residues in oranges being significantly higher than in the other crops.

Based on the residues in oranges, the Meeting estimated a maximum residue level of 0.02 mg/kg, a STMR of 0.005 mg/kg and a HR of 0.015 mg/kg for abamectin in citrus.

This estimation replaces the previous recommendation for abamectin in citrus.

Pome fruit

GAP for abamectin in pome fruit in Italy is up to 2× 22 g ai/ha and 28 days PHI. Various trials were conducted in Europe according to this GAP in apples and pears from 1986 to 2012.

In 26 trials conducted on apples in Europe according to Italian GAP, residues of abamectin were < 0.002 (20), 0.003 (2), 0.004 (2), 0.007 (2) mg/kg. The highest residue in a replicate samples was 0.010 mg/kg.

Two trials conducted in pears at GAP gave abamectin residues of < 0.002 mg/kg (2). Five trials using three applications of the GAP rate also found no residues.

Based on the residue data in apples, the Meeting estimated a maximum residue level of 0.01 mg/kg, a STMR of 0.002 mg/kg and a HR of 0.01 mg/kg for abamectin in pome fruit.

The Meeting withdraws its previous recommendations for apple and pears.

Stone fruit

GAP for abamectin in stone fruit in the USA is 2× 26 g ai/ha and 21 days PHI. Fifteen trials were conducted in cherry in USA according to this GAP, giving abamectin residues of 0.003 (2), 0.004, 0.005, 0.006, 0.007, 0.008, 0.009 (2), 0.010, 0.011, 0.015, 0.016, 0.024, 0.047 mg/kg. The highest residue in a replicate samples was 0.058 mg/kg.

Thirteen trials were conducted in peaches in the USA according to GAP, giving abamectin residues of < 0.002, 0.002 (6), 0.003, 0.004 (2), 0.005, 0.006 (2), 0.008 and 0.024 mg/kg.

Fifteen trials were conducted in plums in the USA according to GAP, giving abamectin residues of < 0.002 (7), 0.002, 0.003 and 0.004 (4) mg/kg. The highest residue in a replicate samples was 0.006 mg/kg

In Italy, GAP for abamectin in peaches is 2× 22 g ai/ha and 14 days PHI. In five trials conducted in France, Italy and Spain according to this GAP, abamectin residues in the whole fruit were < 0.002 (3), 0.004 and 0.006 mg/kg. Residues in the pulp were < 0.002 (3), 0.004 and 0.007 mg/kg

The residue populations in cherries, peaches and plums from the USA gave the highest residues and will be considered for the sub-group estimations.

The Meeting estimated a maximum residue level of 0.07 mg/kg, a STMR of 0.009 mg/kg, and a HR of 0.058 mg/kg for abamectin in cherries.

The Meeting estimated a maximum residue level of 0.03 mg/kg, a STMR of 0.002 mg/kg and a HR of 0.024 mg/kg for abamectin in peaches.

The Meeting estimated a maximum residue level of 0.005 mg/kg, a STMR of 0.004 mg/kg and a HR of 0.006 mg/kg for abamectin in plums.

Raspberry

GAP for abamectin in raspberries and blackberries in Italy is one application at 22 g ai/ha and 7 days PHI. In four trials conducted in Italy at GAP, abamectin residues were < 0.02 (2), 0.02 and 0.03 mg/kg

The Meeting estimated a maximum residue level of 0.05 mg/kg, a STMR of 0.02 mg/kg and a HR of 0.03 mg/kg for abamectin in raspberry, red, black.

The Meeting agreed to extend this estimation to blackberries.

Strawberry

In Denmark, GAP for abamectin in strawberries is greenhouse applications at 3× 22 g ai/ha and 3 days PHI. In eight greenhouse trials conducted in France and Spain according to this GAP, abamectin residues were 0.004, 0.006, 0.014, 0.020, 0.034, 0.042, 0.045 and 0.071 mg/kg. The highest residue in duplicate samples was 0.073 mg/kg.

In the USA, GAP is 4× 21 g ai/ha and 3 days PHI. In five protected trials conducted at GAP, residues were 0.005 (2), 0.006, 0.007 and 0.008 mg/kg. In seventeen field trials, residues were < 0.005 (5), 0.006 (4), 0.009 (2), 0.010 (2), 0.016, 0.020, 0.026, and 0.028 mg/kg.

Based on the protected trials conducted in Europe that gave the highest residues, the Meeting estimated a maximum residue level of 0.15 mg/kg, a STMR of 0.027 mg/kg and a HR of 0.071 mg/kg for abamectin in strawberries.

This estimation replaces the previous recommendation for abamectin in strawberries.

Grapes

GAP for abamectin in grapes in the USA is 2× 21 g ai/ha and 28 days PHI. In nineteen trials conducted in the USA at GAP, residues of abamectin were < 0.002 (10), 0.002 (4), 0.004 (3), and 0.006 (2) mg/kg. The highest residue in a replicate samples was 0.010 mg/kg

The Meeting estimated a maximum residue level of 0.01 mg/kg, a STMR of 0.002 mg/kg and a HR of 0.010 mg/kg for abamectin in grapes.

Avocado

In the USA, GAP for abamectin in avocados is 2× 26 g ai/ha and 14 days PHI. In five trials conducted at GAP in the country, residues were < 0.002, 0.003, 0.004 (2), and 0.007 mg/kg. The highest residue in a replicate samples was 0.009 mg/kg

The Meeting estimated a maximum residue level of 0.015 mg/kg, a STMR of 0.004 mg/kg and a HR of 0.009 mg/kg for abamectin in avocados.

Mango

In Brazil, GAP for abamectin in mangoes is 4× 14 g ai/ha and 7 days PHI. In five trials conducted in the country at GAP, abamectin residues were < 0.002 (3), < 0.004 and 0.004 mg/kg.

The Meeting estimated a maximum residue level of 0.01 mg/kg, a STMR of 0.002 and HR of 0.004 mg/kg for abamectin in mangoes.

Papaya

In Brazil, GAP for abamectin in papaya is 3× 22 g ai/ha and 14 days PHI. In eight trials conducted in the country at GAP, abamectin residues in papaya fruit were < 0.002, 0.002, 0.003 (2), 0.004, 0.005 (2) and 0.008 mg/kg. Residues in the pulp were < 0.002 (6) mg/kg. Six trials conducted at double rate did not show any residues in the pulp (< 0.002 mg/kg), confirming a no residue situation in the pulp when the fruit is treated at GAP.

The Meeting estimated a maximum residue level of 0.015 mg/kg, a STMR and HR of 0 mg/kg for abamectin in papaya.

Onion and shallot

GAP for onions, bulbs (include shallots) in the USA is 2× 21 g ai/ha and 30 days PHI. In eight trials conducted in the country using 3–4 applications at the GAP rate gave residues of < 0.002 (7) and 0.002 mg/kg. The highest residue in a replicate samples was 0.003 mg/kg.

Meeting estimated a maximum residue level of 0.005 mg/kg, a STMR of 0.002 and HR of 0.003 mg/kg for abamectin in onion bulbs. This estimation was extrapolated to shallots and garlic.

Leek

GAP for abamectin in leek in Belgium is 3× 9 g ai/ha and 7 days PHI. Twelve trials conducted in France and the Netherlands within this GAP gave abamectin residues of < 0.002 (10) and 0.002 (2) mg/kg. The highest residue in a replicate samples was 0.003 mg/kg.

The Meeting estimated a maximum residue level of 0.005mg/kg, a STMR of 0.002 mg/kg and HR of 0.003 mg/kg for abamectin in leek.

Cucumber/gherkin

In Denmark, GAP for abamectin in cucumbers and gherkins is four greenhouse applications at 22 g ai/ha with a 3 day PHI. Twenty-nine protected trials were conducted in Europe from 1989 to 2013. In twenty five trials (3-5 applications) conducted according to the Denmark GAP, abamectin

residues were < 0.002 (6), < 0.005 (5), 0.002 (6), 0.003, 0.004 (2), 0.005, 0.006, 0.007 (2) and 0.025 mg/kg. The highest residue in a replicate samples was 0.029 mg/kg.

The Meeting estimated a maximum residue level of 0.03 mg/kg, a STMR of 0.002 and HR of 0.029 mg/kg for abamectin in cucumbers. This estimation was extrapolated to gherkins.

Melon

In Denmark, GAP for abamectin in melons is three greenhouse applications at 22 g ai/ha and 3 days PHI. Twelve greenhouse trials (3-4 applications) were conducted in Europe from 2000 to 2008 according to this GAP, giving abamectin residues the whole fruit of < 0.002 (6), 0.002 (3), 0.003 (2) and 0.005 mg/kg. Residues in the pulp were < 0.002 (10) mg/kg.

The Meeting estimated a maximum residue level of 0.01 mg/kg, a STMR and HR of 0.002 mg/kg for abamectin in melons, except watermelon.

This estimation replaces the previous recommendation for abamectin in melons, except watermelons.

Pepper

In Denmark, GAP for abamectin in sweet or bell peppers is five greenhouse applications at 22 g ai/ha and 3 days PHI. In eighteen greenhouse trials conducted in Europe within this GAP, abamectin residues were < 0.005 (3), 0.002 (2), 0.004, 0.005, 0.006, 0.008, 0.010, 0.012, 0.015, 0.018, 0.019, 0.02, 0.025, 0.027 and 0.051 mg/kg.

In the USA, GAP for fruiting vegetables, except cucurbits, is 2× 21 g ai/ha and 7 days PHI. Four trials were conducted in chilli pepper using six applications, giving residues < 0.005 mg/kg (4).

The Meeting estimated a maximum residue level of 0.09 mg/kg, a STMR of 0.007 mg/kg and HR of 0.051 mg/kg for abamectin in peppers, sweet.

This estimation replaces the previous recommendation for abamectin in peppers, sweet.

The Meeting estimated a maximum residue level of 0.005* mg/kg, a STMR and a HR of 0.005 mg/kg for abamectin in peppers, chilli.

This estimation replaces the previous recommendation for abamectin in chilli pepper.

The Meeting withdraws its previous recommendation for pepper, chilli, dried.

Tomato and eggplant

GAP for abamectin in tomatoes in Denmark is five greenhouse applications at 22 g ai/ha and in Greece, GAP for tomatoes and eggplants is 4× 22 g ai/ha. In both countries, the PHI is 3 days. Metabolism studies have shown that abamectin degrades rapidly and the Meeting agreed that only the last applications will impact the final residues and decided to use the trials with a lower number of applications for the estimations.

In twenty six greenhouse tomato trials using two to five applications at the GAP rate gave residues of < 0.002 (5), 0.002, 0.003, 0.004 (6), 0.005, 0.006 (2), 0.007 (2), 0.010, 0.011, 0.012, 0.014, 0.24, 0.25 and 0.027 (2) mg/kg.

Nine tomato field trials were conducted in France, Italy and Spain using 3–4 applications of the GAP rate, matching the Greek GAP gave residues of < 0.002 (6) and 0.002 (3) mg/kg.

Based on the greenhouse trials, which gave the highest residues, the Meeting estimated a maximum residue level of 0.05 mg/kg, a STMR of 0.004 mg/kg and HR of 0.027 mg/kg for abamectin in tomato.

This estimation replaces the previous recommendation for abamectin in tomatoes.

In two field trials conducted in eggplants in France using six applications, no abamectin residues were detected at 3 days PHI (< 0.010 mg/kg).

As three trials is not enough for the estimations, the Meeting agreed to extend the estimations for tomatoes to eggplants.

Lettuce

Abamectin can be used in lettuce in Greece at 4× 9 g ai/ha and 14 days and in Italy (includes cos lettuce) at 3× 18 g ai/ha and 7 days PHI.

Nine field trials were conducted in Italy and France according to Italian GAP, giving abamectin residues at 7 days PHI of < 0.002, 0.003 (2) and 0.005 mg/kg in head lettuce, 0.004 and 0.007 mg/kg in leafy lettuce and < 0.002, 0.003, 0.006 and 0.008 mg/kg in cos lettuce.

In protected trials conducted in Europe according to GAP in Greece, residues at 14 days PHI in head lettuce were (n=8) 0.007, 0.011, 0.019, 0.020, 0.035, 0.045, 0.047 and 0.097 mg/kg. Residues from protected trials conducted according to GAP with unidentified lettuce type ranged from 0.003 to 0.012 mg/kg.

Protected trials conducted in head lettuce according to GAP in Greece gave the highest residues. The Meeting estimated a maximum residue level of 0.15 mg/kg, a STMR of 0.0275mg/kg and a HR of 0.097 mg/kg for abamectin in head lettuce.

The Meeting agreed that there are not enough trials to estimate a maximum residue level for abamectin in leafy lettuce and cos lettuce.

The Meeting withdraws its previous recommendation on leafy lettuce.

Corn salad (lambs lettuce)

Abamectin can be used in lambs lettuce in Italy at 3× 18 g ai/ha and 7 days PHI. Two trials were conducted in lambs lettuce in France, but they were not according to GAP.

The Meeting agreed not to estimate a maximum residue level for abamectin in lambs lettuce

Spinach

In the USA, GAP for abamectin in spinach is 2× 21 g ai/ha and 7 days PHI. Six declining trials using six application (7 days interval) and metabolism studies showed a rapid declining of the residues, indicating that the contribution of the early applications does not impact the final residue. In eleven trials conducted with 3–6 applications abamectin residues at 7 days PHI were < 0.002 (2), 0.016, 0.020, 0.021, 0.024, 0.028, 0.042, 0.044, 0.048 and 0.085 mg/kg. The highest residue in a replicate samples was 0.091 mg/kg.

The Meeting agreed to recommend a maximum residue level of 0.15 mg/kg, a STMR of 0.024 mg/kg and a HR of 0.091 mg/kg for abamectin in spinach.

The IESTI from the consumption of spinach represented 140% of the ARfD for abamectin (0.003 mg/kg bw). No alternative GAP was available to the Meeting.

Bean, green with pods

The GAP for abamectin in green beans in Spain is 3× 18 g ai/ha and 3 days PHI. In thirteen greenhouse trials conducted in Italy and Spain according to this GAP, residues in green bean with pods were < 0.002 (4), 0.003, 0.004, 0.007, 0.012, 0.014, 0.016, 0.017, 0.023, and 0.049 mg/kg

The meeting estimated a maximum residue level of 0.08 mg/kg, a STMR of 0.012 mg/kg and a HR of 0.049 mg/kg for abamectin in beans, except broad beans and soya beans (green pods and immature seeds).

Beans, dry

GAP for abamectin in beans, dry, in the USA is 2× 21 g ai/ha and 7 days PHI. In seven trials conducted in the USA using three applications, residues were < 0.002 (6) and 0.003 mg/kg.

As it is unlikely that the first application would impact the final residue, the Meeting agreed to use these trials for estimating a maximum residue level of 0.005 mg/kg and a STMR of 0.002 mg/kg for abamectin in beans, dry.

Celeriac

GAP for abamectin in celeriac in the USA is 2× 21 g ai/ha and 7 days PHI. Two trials were conducted in the country using three applications gave no residues in the root (< 0.002 mg/kg)

The Meeting agreed that two trials are not sufficient to estimate a maximum residue level for abamectin in celeriac.

Potato

In the USA, the GAP for abamectin in tuberous and corm vegetables, which include potatoes, sweet potatoes and yams, is 2× 21 g ai/ha and 14 days PHI. In thirteen potato trials conducted in the country from 1992 to 1998 using from 3-6 applicatons at GAP, no abamectin residues were detected in potato tubers (< 0.005 mg/kg). Trials conducted at 6 × 112 g ai/ha gave the same result.

The Meeting estimated a maximum residue level of 0.005* mg/kg, a STMR and a HR of 0 mg/kg for abamectin in potato. The Meeting agreed to extrapolate this recommendation to sweet potato and yams.

This estimation replaces the previous recommendation for abamectin in potatoes.

Radish

GAP for abamectin in radishes in Belgium is 2× 10 g ai/ha and 14 days PHI. In one protected trial conducted in the Netherlands in 1999 within this GAP, abamectin residues in the root were < 0.002 mg/kg.

The Meeting agreed that one trial is not sufficient to estimate a maximum residue level for abamectin in radishes.

Celery

GAP for abamectin in celery in Greece is 4× 9 g ai/ha and 14 days PHI. In seven trials conducted using three applications, samples were collected at 10 DAT.

In the USA, GAP is 2× 21 g ai/ha and 7 days PHI. Six trials conducted in the country using three applications gave residues of 0.003, 0.005 (2), 0.006 0.01 and 0.016 mg/kg

As it is unlikely that the first application would impact significantly the final residue, the Meeting agreed to use these trials to estimate a maximum residue level of 0.03 mg/kg, a STMR of 0.005 mg/kg and a HR of 0.016 for abamectin in celery.

Rice

In China, GAP for abamectin in rice is 2× 14 g ai/ha and 21 days PHI. In six trials conducted in the country according to GAP, abamectin residues in rice husked were < 0.001 mg/kg (6). Six trials conducted at 2× 20 g ai/ha rate gave residues of < 0.001 (4), 0.001 and 0.002 mg/kg. Applying the proportionally principle to this dataset, residues according to GAP are < 0.001 (5) and 0.0015 mg/kg.

Residues on the 12 trials combined are < 0.001 mg/kg (11) and 0.0015 mg/kg.

The Meeting estimated a maximum residue level of 0.002 mg/kg and a STMR of 0.001 mg/kg for abamectin in rice, husked.

Tree nuts

In the USA, GAP for abamectin in tree nuts is 2× 26 g ai/ha and 21 days PHI. In three trials conducted in almonds according to GAP, residues were <0.005 mg/kg. In another 29trials conducted

in almond, pecan and walnut using 3 applications of 28 or 56 g ai/ha, residues at 3 to 14 DAT gave the same result.

As trials conducted at higher GAP or shorter DAT do not give rise to residues in nut meat, the Meeting estimated a maximum residue level of 0.005* mg/kg, a STMR and a HR of 0 mg/kg for abamectin in tree nuts.

The Meeting withdraws its previous recommendation for almonds and walnuts.

Cotton

GAP for abamectin in cotton in Spain is 3× 18 g ai/ha and 3 days PHI. Five trials were conducted in Greece and Spain using two applications, giving abamectin residues at 3 days PHI of < 0.002 mg/kg (5).

In the USA, GAP is 2× 21 g ai/ha and 20 days PHI. In eleven trials conducted in the country according to GAP, residues were < 0.002 (9), 0.005 and 0.01 mg/kg.

The Meeting estimated a maximum residue level of 0.015 mg/kg and a STMR of 0.002 mg/kg for abamectin in cotton seed.

This estimation replaces the previous recommendation for abamectin in cotton.

Peanut

Abamectin is registered in Argentina to be used in peanuts at 1× 2 g ai/ha and 30 days PHI. Four trials were conducted in Brazil using 3× 14 g ai/ha, giving residues < 0.005 mg/kg (4).

Based on the Brazilian trials conducted at high rate and metabolism studies that showed no translocation of abamectin residues in the plant, the Meeting estimated a maximum residue level of 0.005* mg/kg, and a STMR of 0 mg/kg for abamectin in peanuts.

Coffee

Critical GAP for abamectin in coffee in Brazil is one application at 27 g ai/ha and 14 days PHI. Five trials were conducted in the country using 7–9 g ai/ha, giving residues < 0.002 mg/kg (5).

As no trials were conducted according to GAP, the Meeting could not estimate a maximum residue level for abamectin in coffee.

Hops

Abamectin is registered in hops in Slovenia and the USA to be used at 2× 21–22 g ai/ha and 28 days PHI. In seven trials conducted in Germany according to this GAP, abamectin residues in dried cones were < 0.005 (2), 0.010, 0.012 0.02, 0.021 and 0.028 mg/kg. In four trials conducted in the USA at GAP, residues were 0.012, 0.020, 0.056 and 0.061 mg/kg.

Trials conducted in the USA gave the highest residues, and the Meeting estimated a maximum residue level of 0.15 mg/kg and a STMR of 0.038 mg/kg for abamectin in hops, dry.

This estimation replaces the previous recommendation for abamectin in hops, dry.

Feed commodities

Rice husks

In six trials conducted with abamectin in rice in China according to GAP (2× 14 g ai/ha), abamectin residues in rice husks (hulls) at 21 days PHI were < 0.001 (5) mg/kg and 0.006 mg/kg.

The Meeting estimated a median residue of 0.001 mg/kg for abamectin in rice hulls.

Residues in paddy rice plant (including grain with husks) in trials according to GAP were < 0.001 mg/kg (6). Trials conducted at 20 g ai/ha gave the same results.

As no residues were found in rice plant, the Meeting estimated a maximum residue level of 0.001 mg/kg, a median and highest residue of 0.001 mg/kg for abamectin in rice straw.

Green beans

In four European trials conducted in green beans according to GAP in Spain (3× 18 g ai/ha, 3 days PHI), abamectin residues in the vines were 0.329, 0.349, 0.354, and 0.581 mg/kg.

The Meeting estimated a median residue of 0.352 mg/kg and highest residue of 0.581 mg/kg for abamectin in green bean vines.

Almond hulls

In six trials conducted in almonds in the USA at the GAP, residues in the hulls at 21 days PHI were < 0.002, 0.012, 0.035, 0.037, 0.102 and 0.11 mg/kg.

The Meeting estimated a maximum residue level of 0.2 mg/kg and a median residue of 0.036 mg/kg for abamectin in almond hulls.

Cotton hulls

As no trials were conducted in cotton according to GAP that analysed the hulls, the Meeting could not make any estimation for abamectin in cotton hulls.

Fate of residues in processing

Three processing studies were conducted in grapes, with abamectin residues in grapes of 0.012, 0.007 and 0.048 mg/kg. Although the stability study on grape processed commodities have shown that abamectin residues were not stable after 12 months in raisins, in the processed study the samples were analysed within a month after being generated, and the results are evaluated. Eleven studies were conducted in cotton, all in the context of the residue trials described before. The estimated processing factors with the respective recommendations of STMR-P, based on the recommended maximum residue level, are shown in the Table.

RAC	Processed product	PF (median or best estimate)	STMR-P, mg/kg	HR-P, mg/kg	MRL, mg/kg
Grapes	Dried grape	1, 2.8, 3.1	0.0056	0.028	0.03
MRL = 0.01 mg/kg	Grape juice	< 0.25, < 0.57, 1.4	0.0028		0.015
STMR = 0.002 mg/kg	Wet pomace	4.75	0.009		
HR = 0.01 mg/kg	dry pomace	15.8	0.0316		
Plums	Prune	0.8 [a]			
Cotton	Meal	< 0.028, < 0.067	0.000		
STMR = 0.002 mg/kg	Refined oil	< 0.028, < 0.67	0.000		

[a] Recommendation for Plums includes prunes

Residues in animal commodities

A feeding study was conducted in dairy cows (n=3) with abamectin dosed at 0.01, 0.03 and 0.10 ppm levels for 28–30 days. Avermectin B1a residues were determined by HPLC-FL, with an LOQ of 0.001 mg/kg in tissues and 0.0005 mg/kg in milk. Residues in muscle at any feeding level were < 0.01 mg/kg (traces at 0.002 mg/kg at all levels), and in kidney (traces at 0.004–0.005 mg/kg at 0.10 ppm). At this highest dose, maximum residues were 0.014 mg/kg (mean of 0.012 mg/kg) in fat and 0.020 mg/kg in liver (mean of 0.019 mg/kg). In milk, residues were only detected after 2 days dosing at 0.10 ppm (0.001 mg/kg), reaching a maximum of 0.004 mg/kg at day 14, and decreasing to the initial levels at the end of the dosing period. Overall mean was < 0.0005 mg/kg.

Farm animal dietary burden

The Meeting estimated the dietary burden of abamectin 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 JMPR Meetings.

The commodities used to estimate the dietary burden were rice, husked, rice straw, rice hulls, grape pomace dried, bean vines, almond husk, bean dry, and cotton meal. As abamectin is not registered in beans and grapes in Australia, and is unlikely that bean vines and grape pomace would be animal feed in the country, as they are not imported commodities, they were excluded in the calculation for the Australian diet.

Livestock dietary burden for abamectin, ppm of dry matter (DM) diet

	US-Canada		EU		Australia		Japan	
Commodity	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0.0003	0.0003	0.0007	0.0007	0.004	0.004	0.0006	0.0006
Dairy cattle	0.004	0.004	0.333 [a, b]	0.202 [c, d]	0.004	0.004	0.0003	0.0003
Poultry—broiler	0.0007	0.0007	0.0006	0.0006	0.002 [e]	0.002		
Poultry—layer	0.0007	0.0007	0.0007	0.0006	0.002	0.002 [f]		

[a] Highest maximum beef or dairy cattle dietary burden suitable for maximum residue level estimated for mammalian tissues

[b] Highest maximum dairy cattle dietary burden suitable for maximum residue level estimated for mammalian milk

[c] Highest mean beef or dairy cattle dietary burden suitable for STMR estimated for mammalian tissues.

[d] Highest mean dairy cattle dietary burden suitable for STMR estimated 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 maximum residue level

The calculated maximum cattle dietary burden suitable for the estimation of maximum residue level of tissues and milk is 0.333 ppm. For the estimation of STMRs, the cattle dietary burden was 0.202 ppm.

The feeding level in lactating cows was conducted in a much lower dose (up to 0.10 ppm) than the estimated dietary burden. The Meeting agreed not to make any estimation for abamectin in mammalian commodities.

The Meeting withdraws its previous recommendations for cattle fat, cattle kidney, cattle liver, cattle meat, cattle milk, goat meat, goat milk and goat, edible offal.

Currently, the existing Codex MRLs for abamectin as a veterinary drug only intended to be used in beef cattle are 0.1 mg/kg in cattle liver and cattle fat and 0.05 mg/kg in cattle kidney.

The calculated maximum poultry dietary burden suitable for maximum residue level estimated for poultry tissues and eggs was 0.002 ppm. No feeding study on poultry was submitted to the Meeting.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed in Annex 1 are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Residue definition for plant commodities for enforcement and dietary risk assessment: *Avermectin B_{1a}*

Residue definition for animal commodities for enforcement and dietary risk assessment: *Avermectin B_{1a}*

The residues are fat soluble.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
AN 0660	Almond hulls	0.2	0.1	0.036	
TN 0660	Almonds	W	0.01*		
FP 0226	Apple	W	0.01*		
FI 0326	Avocado	0.015		0.004	0.009
VP0061	Beans, except broad bean and soya bean (immature beans with pods)	0.08		0.007	0.049
VD 0771	Beans (dry)	0.005		0.002	
FB 0264	Blackberries	0.005		0.002	0.003
MF 0812	Cattle fat	W	0.1		
MO 1280	Cattle kidney	W	0.05		
MO 1281	Cattle liver	W	0.1		
MM 0812	Cattle meat	W	0.01*		
ML 0812	Cattle milk	W	0.005		
VX 0578	Celery	0.03		0.005	0.016
FS 0013	Cherries	0.07		0.009	0.058
FC 0001	Citrus fruits	0.2	0.01*	0.005	0.015
SO 0691	Cotton seed	0.015	0.01*	0.002	
VC 0424	Cucumber	0.03	0.01	0.002	0.029
VO 0440	Egg plant	0.05	0.02	0.004	0.017
VA 0381	Garlic	0.005		0.002	0.003
VC 0425	Gherkin	0.05		0.002	0.029
MM 0814	Goat meat	W	0.01*		
ML 0814	Goat milk	W	0.005		
MO 0814	Goat, edible offal of	W	0.1		
FB 0269	Grapes	0.01		0.002	0.01
DF 0269	Dried grapes (= currants, raisins and sultanas)	0.03		0.0056	0.028
JF 0269	Grape juice	0.015		0.0028	
DH 1100	Hops, dry	0.15	0.1	0.038	
VA 0384	Leek	0.005		0.002	0.003
VL 0483	Lettuce, Leaf	W	0.05		
VL 0482	Lettuce, head	0.15		0.0275	0.097
FI 0345	Mango	0.01		0.002	0.004
VC 0046	Melons, except Watermelon	0.01	0.01*	0.002	0.002
VA 0385	Onion, Bulb	0.005		0.002	0.003
FI 0350	Papaya	0.015		0	0
FS 2001	Peaches	0.03		0.004	0.024
SO 0697	Peanut	0.005*		0	
FP 0230	Pear	W	0.02		
VO 0444	Peppers, chili, dried	0.005*	0.2	0.005	0.005
VO 0445	Peppers, sweet	0.07	0.02	0.009	0.051
FS 0014	Plums (including prunes)	0.005		0.002	0.006
FP 0009	Pome fruits	0.01		0.002	0.01
VR 0589	Potato	0.005*	0.01*	0	0
DF 5263	Raisins	0.05		0.0084	0.0224
FB 0272	Raspberry, red, black	0.002		0.002	0.03
GC 0649	Rice	0.002		0.001	
AS 0646	Rice straw	0.001		0.001	0.001
VA 0388	Shallot	0.005		0.002	0.003
VL 0502	Spinach	0.15 [a]		0.024	0.091
VC 0431	Squash, summer	W	0.01*		
FB 0275	Strawberry	0.15	0.02	0.027	0.073mi
VR 0508	Sweet potato	0.005*		0	0
VO 0448	Tomato	0.05	0.02	0.004	0.017
TN 0085	Tree nuts	0.005*		0	0

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
TN 0678	Walnuts	W	0.01*		
VC 0432	Watermelon	W	0.01*		
VR 0600	Yams	0.005*		0	0
OR 0691	Cotton seed oil, edible			0	

[a] On the basis of information provided to the JMPR it was concluded that the estimated short-term intake of abamectin for the consumption of spinach may present a public health concern

DIETARY RISK ASSESSMENT

The intake assessments conducted by the Meeting did not include the uses of abamectin as a veterinary drug.

Long-term intake

The International estimated daily intakes (IEDI) of abamectin based on the STMRs estimated by this Meetings for the 17 GEMS/Food regional diets were 1–5% of the maximum ADI of 0.001 mg/kg bw (see Annex 3 to the 2015 Report). The Meeting concluded that the long-term dietary intake of residues of abamectin is unlikely to present a public health concern.

Short-term intake

The ARfD for abamectin is 0.003 mg/kg bw. The International Estimated Short-Term Intake (IESTI) of abamectin for the commodities for which STMR, HR and maximum residue levels were estimated by the current Meeting. The results are shown in Annex 4 to the 2015 Report.

For spinach, the IESTI represented 140% of the ARfD for children. No alternative GAP was available. On the basis of information provided to the Meeting, it was concluded that the short-term intake of abamectin residues from the consumption of spinach may present a public health concern.

The IESTI for the other commodities considered by the Meeting represented a maximum of 70% of the ARfD, and for these commodities, the Meeting concluded that the short-term-intake of abamectin is unlikely to present a public health concern when abamectin is used in ways considered by the Meeting.

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1107/01	Satter, P	2003u	Residue Study with Abamectin (MK936) in or on Tomatoes in Spain. Syngenta Crop Protection AG, Basel, Switzerland, 1107/01 GLP, not published.
1108/01	Satter, P	2003v	Residue Study with Abamectin (MK936) in or on Tomatoes in Spain. Syngenta Crop Protection AG, Basel, Switzerland, 1108/01 GLP, not published.
1109/01	Satter, P	2003w	Residue Study with Abamectin (MK936) in or on Tomatoes in Spain. Syngenta Crop Protection AG, Basel, Switzerland, 1109/01 GLP, not published.
1086/01	Satter, P	2003x	Residue Study with Abamectin (MK936) in or on Tomatoes in Spain. Syngenta Crop Protection AG, Basel, Switzerland, 1086/01 GLP, not published.
1081/01	Satter, P	2003y	Residue Study with Abamectin (MK936) in or on Common Beans in Spain. Syngenta Crop Protection AG, Basel, Switzerland, 1081/01 GLP, not published.
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HLA-6012-245	Siirila, A	1997	Storage Stability Study: High Performance Liquid Chromatography Fluorescence Determination for Avermectin B_1 and its Delta 8,9 Isomer in Strawberries. Syngenta Crop Protection AG, Basel, CH Hazleton Laboratories, Madison, USA, HLA- 6012-245, GLP, not published. Syngenta File No MK936/0599
gr 71500	Simon, P	2001	Determination of Residues of Abamectin in protected Tomatoes, Germany. Syngenta Crop Protection AG, Basel, Switzerland. Syngenta Agro GmbH, Maintal, Germany, gr 71500 GLP, not published.
gto35301	Simon, P	2002	Determination of Residues of Abamectin after Application of Vertimec in Protected Tomatoes in Germany. Syngenta Crop Protection AG, Basel, Switzerland. Syngenta Agro GmbH, Maintal, Germany, gto35301 GLP, not published.
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03-5073	Sole, C	2004	Residue study with Abamectin (MK936) in or on Peaches in Spain. Syngenta Crop Protection AG, Basel, CH. ADME—Bioanalyses, Vergeze, France, 03-5073 GLP, not published.
03-5074	Sole, C	2004a	Residue study with Abamectin (MK936) in or on Peaches in Spain. Syngenta Crop Protection AG, Basel, CH. ADME—Bioanalyses, Vergeze, France, 03-5074 GLP, not published.
03-5075	Sole, C	2004b	Residue study with Abamectin (MK936) in or on Peaches in Italy. Syngenta Crop Protection AG, Basel, CH. ADME—Bioanalyses, Vergeze, France, 03-5075 GLP, not published.
03-5076	Sole, C	2004c	Residue study with Abamectin (MK936) in or on Peaches in Italy. Syngenta Crop Protection AG, Basel, CH. ADME—Bioanalyses, Vergeze, France, 03-5076 GLP, not published.
03-5066	Sole, C	2004d	Residue study with Abamectin (MK936) in or on Strawberries in Northern France. Syngenta Crop Protection AG, Basel, CH. ADME—Bioanalyses, Vergeze, France, 03-5066 GLP, not published.
03-1019	Sole, C	2004e	Residue study with Abamectin (MK936) in or on Tomatoes in Spain. Syngenta Crop Protection AG, Basel, Switzerland. ADME—Bioanalyses, Vergeze, France, 03-1019 GLP, not published.
03-1025	Sole, C	2004f	Residue study with Abamectin (MK936) in or on Tomatoes in Italy. Syngenta Crop Protection AG, Basel, Switzerland. ADME—Bioanalyses, Vergeze, France, 03-1025 GLP, not published.
06593	Starner, V	2000	Abamectin—Magnitude of the Residue on Celeriac (Roots & Tops). IR-4 Project, North Brunswick, USA, 06593 GLP, not published.
1274-4	Starner, VR	1993	MK 936, Abamectin 1.8% EC, Lettuce, Spain. Novartis Crop Protection AG, Basel, Switzerland. Analytical Development Corp., Colorado Springs, USA, 1274-4 GLP, not published.
1274-5	Starner, VR	1993a	MK 936, Abamectin 1.8% EC, Lettuce, Spain. Novartis Crop Protection AG, Basel, Switzerland. Analytical Development Corp., Colorado Springs, USA, 1274-5 GLP, not published.

Code	Author	Year	Title, Institute, Report reference
00MK13	Stingelin, J	2003	Metabolism of Avermectin B1a (NOA 422601) in Greenhouse Grown Tomato Plants. Syngenta Crop Protection AG, Basel, CH, 00MK13 GLP, not published.
01MK17	Stingelin, J	2003a	Metabolism of Avermectin B1a (NOA 422601) in Field Grown Tomato Plants. Syngenta Crop Protection AG, Basel, CH, 01MK17 GLP, not published.
75494	Stulz, J	1999	Solubility in organic solvents of Abamectin tech. Syngenta Crop Protection AG, Basel, CH, 75494 GLP, not published.
HLA 6012-322	Trainor T	1990	High-Performance Liquid Chromatography Fluorescence Determination for Avermectin B_1 and its Delta 8,9 Isomer in Cucumbers. Novartis Crop Protection AG, Basel, Switzerland. Hazleton Laboratories, Madison, USA, HLA 6012-322 Not GLP, not published.
HWI 6012-358	Trainor, TJ	1991	High-Performance Liquid Chromatography Fluorescence Determination for Avermectin B_1 and its Delta 8,9 Isomer in Italian Cucumbers (1990). Novartis Crop Protection AG, Basel, Switzerland. Hazleton Laboratories, Madison, USA, HWI 6012-358 GLP, not published.
T000572-08-REG	Turnbull, G	2009	Abamectin—Residue Study on Protected Tomatoes in the Netherlands in 2008. Syngenta T000572-08-REG, FSGD-044 GLP, not published.
CA-211	Wehner, TA	1986	Abamectin (MK 936): A Study (CA-211) in Lactating Cows to Determine Milk, Tissue and Plasma Residues in Animals Exposed to Twenty-Eight Days of Oral Ingestion of Abamectin. Novartis Crop Protection AG, Basel, Switzerland. Merck & Co. Inc., Rahway NJ, USA, CA-211 Not GLP, not published.
992	Wertz, PG	1987	Storage Stability Study: Avermectin B_1 in Tomatoes. Syngenta Crop Protection AG, Basel, CH Analytical Development Corp., USA, 992 Not GLP, not published.
066-86-047R	Wertz, PG	1988	MK 936, Avermectin B_1 and its Delta 8,9 Isomer, Pears, France. Syngenta Crop Protection AG, Basel, CH Analytical Development Corp., USA, 066-86-047R Not GLP, not published.
MER/AVE/94111	White, S	1995	Validation of the analytical method for the assay of Avermectin B_{1a}, B_{1b} and 8,9-Z Avermectin B_{1a} in grape samples. Syngenta Crop Protection AG, Basel, CH ADME—Bioanalyses, Mougins, France, MER/AVE/94111 GLP, not published.
1259B	White, S & Starner, V *et al.*,	1994	Determination of the Magnitude of Residues of Abamectin and its Delta 8,9 isomer in/on raw agricultural commodity Tomato from Abamectin 1.8EC Applications by ground equipment in the Netherlands Merck & Co. Inc., Rahway NJ, USA. Analytical Development Corp., Colorado Springs, USA, 1259B, 070-90-0002R GLP, not published.
99WI21	Widmer, H	1999	Vapour pressure of Abamectin tech. Syngenta Crop Protection AG, Basel, CH, 99WI21 GLP, not published.
ADC 1452-1	Wilkes, L	1995	Determination of the Magnitude of Residues Of Avermectin B_1 and 8,9-z Avermectin B_1 in/on the Raw Agricultural Commodity, Chilli Peppers from Abamectin 0.15 Ec (agrimec 1.8%–18g/L) Applications Made With Ground Equipment. Novartis—Greensboro, Greensboro, USA. Merck Research Laboratories, Three Bridges, USA, ADC 1452-1 Not GLP, not published.
MSD-PLM 2A	Wislocki, P	1986	Fate of Avermectin B_{1a} on Cotton Plants. Merck & Co. Inc., Rahway NJ, USA, US Department of Agriculture, Colorado Springs CO, USA, MSD-PLM 2A GLP, not published.

ACETAMIPRID (246)

The first draft was prepared by Professor Mi-Gyung Lee, Andong National University, Republic of Korea

EXPLANATION

Acetamiprid is a neonicotinoid insecticide with contact and stomach action against a range of plant pests such as Hemiptera, Thysanoptera and Lepidoptera acting as an agonist of the nicotinic acetylcholine receptor in the insect central nervous system. It exhibits translaminar activity in plants and is authorized for use in a variety of crops worldwide.

Acetamiprid was evaluated for the first time by the 2011 JMPR, where an ADI of 0–0.07 mg/kg bw and an ARfD of 0.1 mg/kg bw were established, and maximum residue levels were recommended for a range of plant and animal commodities. In 2012, JMPR reconsidered acute dietary risks from maximum residue levels recommended for leafy vegetables (except spinach) and spinach and then withdrew them. Currently, there are no CXLs established for any leafy vegetables.

At the 46th Session of the CCPR (2014), acetamiprid was listed for residue evaluation for additional maximum residue levels by the 2015 JMPR. The Meeting received information on supervised residue trials for cucumber (including fruit cucumber) and tomato (including cherry tomato) from China and for sweet corn (corn-on-the-cob), mustard greens and asparagus from USA. For sweet corn (corn-on-the cob), residue trials were also conducted in Canada.

For both compliance with MRL and estimation of dietary intake, the residue is defined as acetamiprid for plant commodities and the sum of acetamiprid and desmethyl-acetamiprid for animal commodities. The residue is not fat-soluble.

Residue Analysis

Analytical methods

The Method KP-216 (considered suitable by the 2011 JMPR) was used as a reference for residue analysis of acetamiprid in asparagus, mustard greens and sweet corn (kernel plus cob with husk removed, forage and stover from USA trials). Briefly, the analytical methods for those crop samples involved extraction with methanol and water, clean-up by Strata-X (or Oasis HLB) solid phase extraction (SPE) and LC-MS/MS analysis. At fortification levels of 0.01, 0.1 and 1.0 mg/kg, the mean recoveries (n=3 or 6) ranged within 80–120% (CV, < 7.9%) in each sample matrix of asparagus, mustard greens, sweet corn, and kernel plus cob with husk removed. For forage and stover of sweet corn, the mean recoveries were 82–122% (CV, < 6.6%) and 87–124% (CV, < 6.1%), respectively at the three fortification levels. In all matrices, the limit of quantification (LOQ) for acetamiprid was 0.01 mg/kg.

For sweet corn samples (kernel plus cob with husk removed, forage and stover) from trials conducted in Canada, the analytical method involved extraction with methanol, partitioning with hexane and again with methylene chloride, clean-up by SPE Florisil column and LC-MS/MS analysis. At fortification levels of 0.01, 0.02, 0.1, 1 (only for stover) and 5 (only for forge) mg/kg, the mean recoveries (n=3) ranged within 76–99% (CV, < 17%). The LOQ for acetamiprid in matrices of sweet corn was 0.01 mg/kg.

Table 1 Analytical recoveries of acetamiprid in asparagus, mustard and sweet corn

Matrix	Fortification, mg/kg	n	Range of recoveries, %	Mean recovery, %	CV, %	Ref. method
Asparagus (spears)	0.01	3	84–90	86	3.7	Method KP-216
	0.1	3	80–82	81	1.8	
	1.0	3	90–92	91	1.1	

Matrix	Fortification, mg/kg	n	Range of recoveries, %	Mean recovery, %	CV, %	Ref. method
Mustard greens (leaves)	0.01	3	109–126	120	7.9	Method KP-216
	0.1	3	113–122	117	4.3	
	1.0	3	102–111	106	4.3	
	10	3	82–85	83	2.5	
Sweet corn, kernel plus cob with husk removed (USA)	0.01	6	101–120	108	6.4	Method KP-216
	0.1	3	99–109	103	5.0	
	1.0	3	91–97	93	3.4	
Sweet corn, forage (USA)	0.01	6	112–135	122	6.6	Method KP-216
	0.1	3	95–103	100	4.4	
	1.0	3	80–84	82	2.4	
Sweet corn, stover (USA)	0.01	6	106–120	113	4.4	Method KP-216
	0.1	3	119–133	124	6.1	
	1.0	3	80–99	87	11	
Sweet corn, kernel plus cob with husk removed (Canada)	0.01	3	89–93	91	2.2	
	0.02	3	88–91	89	2.2	
	0.1	3	85–86	85	1.2	
Sweet corn, forage (Canada)	0.01	3	87–119	99	17	
	0.02	3	82–85	84	2.4	
	0.1	3	81–82	81	1.2	
	5		71–80	77	7.8	
Sweet corn, stover (Canada)	0.01	3	76–103	89	16	
	0.02	3	69–82	78	10	
	0.1	3	77–95	84	11	
	1	3	74–78	76	2.6	

LOQs, < 0.01 mg/kg

In cucumber, acetamiprid residue was extracted with acetonitrile (mixed with acetic acid, 99:1). The extract aliquots were cleaned up by dispersive SPE (use of C_{18}, primary secondary amine and anhydrous magnesium sulphate) and analysed using LC-MS/MS. At fortification levels of 0.01, 0.2, 1.0 mg/kg, the mean recoveries (n=5) ranged within 89 and 101% (CV, < 9.9%). The LOQ was 0.01 mg/kg in cucumber (Li, Yiqiang; Report No. AC-01).

Acetamiprid residue in tomatoes was extracted with acetonitrile. Extract aliquots were purified by SPE using NH_2 cartridges and analysed by LC-MS/MS. At fortification levels of 0.01, 0.1, and 0.5 mg/kg, the mean recoveries (n=5) ranged within 82–95% (CV, < 5.9%). The LOQ was 0.01 mg/kg in tomatoes (Li, Zhou; Report No. AT-01).

Table 2 Analytical recoveries of acetamiprid in cucumber and tomato

Matrix	Fortification, mg/kg	n	Range of recoveries, %	Mean recovery, %	CV, %
Cucumber	0.01	5	79–98	89	9.9
	0.2	5	95–107	101	4.1
	1.0	5	95–96	96	0.5

Matrix	Fortification, mg/kg	n	Range of recoveries, %	Mean recovery, %	CV, %
Tomato	0.01	5	76–86	82	5.2
	0.1	5	86–101	95	5.9
	0.5	5	85–97	92	5.2

LOQs, < 0.01 mg/kg

Stability of residues in stored analytical samples

Stability of acetamiprid was tested for asparagus, cucumber, mustard greens, sweet corn and tomato stored frozen at or below –20 °C. The residue was analysed using analytical methods described above for each matrix. Maximum tested storage durations were 426 days for asparagus, 304 days for cucumber and tomato, 382 days for mustard greens and 384–391 days for sweet corn kernel plus cob with husk removed, forage and stover samples. No zero-day residues were measured except in cucumber and tomato.

The amount of acetamiprid remaining at each storage sampling interval ranged between 72% and 120% of the nominally applied amount for all matrices. Corresponding procedural recoveries ranged 76–114%. In 2011, the JMPR concluded that acetamiprid is stable for at least 12 months in apple, cabbage, cucumber, grape and tomato, and 16 months for lettuce.

Actual storage durations of the samples from residue studies were shorter than the tested storage stability durations, with an exception of asparagus (stored 473 days, tested 426 days). Based on the available information, it is considered that acetamiprid in crop samples relevant to this submission, including asparagus, was stable until analysis.

Table 3 Storage stability of acetamiprid in plant matrices

Matrix	Fortification level, mg/kg	Tested storage days	Procedural recoveries, %	Residue in fortified samples, mg/kg	Actual max. storage days
Asparagus (spears)	1.0	426	86	0.87, 0.86, 0.85	473
Cucumber	0.1	0		0.10, 0.10, 0.10	205
		31	99, 101, 105	0.095, 0.097, 0.099	
		92	98, 101, 105	0.090, 0.096, 0.096	
		182	98, 100, 104	0.096, 0.098, 0.098	
		304	100, 101, 101	0.096, 0.097, 0.098	
Mustard greens (leaves)	1.0	382	93, 94, 106	0.87, 0.91, 0.85	382
Sweet corn, kernel plus cob with husk removed (USA)	0.1	390	95, 97, 99	0.086, 0.089, 0.093	362
Sweet corn, forage (USA)	0.1	384	86, 99, 114	0.10, 0.12, 0.12	359
Sweet corn, stover (USA)	0.1	391	102, 106, 107	0.10, 0.10, 0.096	373
Tomato	0.5	0	91, 96	0.48, 0.50, 0.50	157
		30	87, 92	0.43, 0.50, 0.53	
		95	77, 87	0.46, 0.49, 0.51	
		108	108, 113	0.38, 0.41, 0.42	
		273	83, 90	0.36, 0.40, 0.46	
		304	76, 78	0.38, 0.42, 0.58	

Sweet corn trial samples conducted in Canada were stored for up to 203 days for kernel plus cob with husk removed, 212 days for forage and 194 days for stover.

USE PATTERN

Information on the registered uses of acetamiprid made available to this Meeting is shown in Table 4.

Table 4 Registered uses of acetamiprid on crops relevant to submitted residue data

Crop	Country	Form.	Method	Application			
				Rate, kg ai/ha	Max. no.	Interval days	PHI, days
Cucumber	China	200 SP (200 g ai/L)	Spray	0.090	3		2
Tomato	China	30 ME (30 g ai/L)	Spray	0.014–0.027	2		7
Sweet corn	USA	30 SG	Foliar spray	0.11 [a]	2	14	7
				0.060 [a]	4	7	1
	Canada	70 WP	Foliar spray	0.060 [b]	2	21	10
Leafy Cole crops and turnip greens (mustard greens)	USA	30 SG, 70WP	Foliar spray	0.11 [c]	4	7	3
Asparagus	USA	30 SG, 70 WP	Foliar spray	0.11 [d]	2	10	1

Formulation: SP (soluble powder), ME (micro emulsion), WP (wettable powder), SG (soluble granule)

Leafy Cole crops and turnip greens include broccoli raab, collards, cabbage (bok choy), kale, mizuna, mustard greens, mustard spinach, rape greens, and turnip greens.

[a] Do not exceed a total of 0.24 kg ai/ha/growing season; do not exceed two crop seasons per year

[b] Do not exceed a total of 0.12 kg ai/ha per season

[c] Do not exceed a total of 0.42 kg ai/ha/growing season

[d] Do not exceed a total of 0.224 kg ai/ha/growing season

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received residue trial data on asparagus, mustard greens and sweet corn from the USA, sweet corn from Canada, and on cucumber (including fruit cucumber) and tomato (cherry tomato) from China. Studies were conducted according to GLP or under the supervision of a study director.

All trials included one control plot and one treated plot. There was no acetamiprid detected above LOQ value, 0.01 mg/kg in control samples. In all trials, at least two samples were taken from a single treated plot. The average residue value was considered for estimation of the maximum residue level. The storage period of the field trial samples did not impact the residue levels, as described in the above section on *stability of residues in stored analytical samples*.

Crop group	Commodity	Table No.
Fruiting vegetables, Cucurbits	Cucumber	5, 6
Fruiting vegetables, other than Cucurbits	Tomato	7, 8
	Cherry tomato	9
	Sweet corn (corn-on-the-cob)	10, 11
Leafy vegetables (incl. Brassica leafy vegetables)	Mustard greens	12
Stalk and stem vegetables	Asparagus	13
Primary feed commodities	Sweet corn, forage	14, 15
	Sweet corn, stover	16, 17

Fruiting vegetables, Cucurbits

Cucumber

Eight residue trials on field-grown cucumbers were conducted in China (Shandong, Fujian, Jilin, Yunnan, Guangdong, Zhejiang, Hunan and Anhui) in 2013. In addition, three trials (Shandong, Fujian and Jilin) on cucumbers and fruit cucumbers were conducted under greenhouse conditions.

At each trial, one treated plot received three applications of the test substance (200 SP formulation, 200 g ai/L) 6–7 days apart. Foliar spray applications were made at growth stages of BBCH 61–71 and the application rate was 0.090 kg ai/ha. Cucumber (or fruit cucumber) in each trial was harvested 0, 1, 2, 3 and 5 days after the last application.

Two decline studies on field-grown cucumber were also conducted in the Shandong and Jujian. One application was made at a rate of 0.090 kg ai/ha and cucumber was harvested 0, 1, 3, 5, 7, 10 and 14 days after the last application.

Six samples from each trial were harvested (1.2–5 kg per sample). From the each sample, a sub sample of 200–320 g was taken and stored in a freezer at –20 °C or below. The deep-frozen sub samples were shredded in a cutter. Representative parts of the shredded samples were transferred into polystyrene box and stored at –18 °C or below until analysis. Residue analysis was made with three samples of the six.

Table 5 Residues resulting from acetamiprid application to field-grown cucumber in China (2013)

Location (Variety) Trial No./Report No.	Application						DALA	Residue, mg/kg			Mean residue, mg/kg
	kg ai/ha	n	Inter. days	L/ha	kg ai/hL	BBCH					Mean
GAP, China	0.090	3					PHI, 2 days				
Qingdao, Shandong (Luhuang No.3) SD-01/R-AC-03	0.090	3	6–8	900	0.01	61–71	0	0.076	0.060	0.048	0.061
							1	0.051	0.062	0.080	0.064
							2	0.032	0.034	0.045	0.037
							3	0.037	0.043	0.024	0.035
							5	< 0.01	< 0.01	< 0.01	< 0.01
Qingdao, Shandong (Luhuang No.3) SD-03/R-AC-05	0.090	1		900	0.01	71	0	0.078	0.080	0.062	0.073
							1	0.097	0.061	0.067	0.075
							3	0.070	0.061	0.046	0.059 [a]
							5	0.026	0.039	0.025	0.030
							7	< 0.01	< 0.01	< 0.01	< 0.01
							10	< 0.01	< 0.01	< 0.01	< 0.01
							14	< 0.01	< 0.01	< 0.01	< 0.01
Zhangzhou, Fujian (Jinyou No. 48) FJ-01/R-AC-06	0.090	3	7	900	0.01	61–69	0	0.10	0.11	0.16	0.12
							1	0.097	0.13	0.085	0.10
							2	0.12	0.10	0.15	0.12

Location (Variety) Trial No./Report No.	Application						DALA	Residue, mg/kg			Mean residue, mg/kg
	kg ai/ha	n	Inter. days	L/ha	kg ai/hL	BBCH					Mean
							3	0.066	0.049	0.015	0.043
							5	0.042	0.038	0.035	0.038
Zhangzhou, Fujian (Jinyou No. 48) FJ-03/R-AC-08	0.090	1		900	0.01	71	0	0.29	0.29	0.24	0.27
							1	0.21	0.18	0.32	0.24
							3	0.13	0.17	0.097	0.13 [a]
							5	0.071	0.085	0.071	0.076
							7	< 0.01	0.046	0.035	0.030
							10	< 0.01	< 0.01	< 0.01	< 0.01
							14	< 0.01	< 0.01	< 0.01	< 0.01
Changchun, Jilin (Lvrang) JL-01/R-AC-09	0.090	3	7	900	0.01	61–69	0	0.026	0.024	0.021	0.024
							1	0.018	0.017	0.016	0.017
							2	0.020	0.021	0.019	0.020
							3	0.013	0.013	0.014	0.013
							5	< 0.01	< 0.01	< 0.01	< 0.01
Changsha, Hunan (Shuyan No. 5) HN-01/R-AC-12	0.090	3	7	900	0.01	61–69	0	0.025	0.044	0.051	0.040
							1	0.021	0.028	0.029	0.026
							2	0.012	< 0.01	0.011	0.011
							3	< 0.01	< 0.01	< 0.01	< 0.01
							5	< 0.01	< 0.01	< 0.01	< 0.01
Hangzhou, Zhejiang (Zhexiu No. 302) ZJ-01/R-AC-13	0.090	3	7	900	0.01	61–69	0	0.11	0.099	0.12	0.11
							1	0.055	0.10	0.066	0.074
							2	0.054	0.074	0.083	0.070
							3	0.058	0.069	0.053	0.060
							5	0.036	0.029	0.024	0.030
Kunming, Yunnan (Bomei No. 2) YN-01/R-AC-14	0.090	3	7	900	0.01	61–69	0	0.11	0.14	0.11	0.12
							1	0.091	0.083	0.092	0.089
							2	0.039	0.035	0.046	0.040
							3	0.037	0.040	0.034	0.037
							5	0.063	0.031	0.032	0.042
Guangzhou, Guangdong (Dadiao)	0.090	3	7	900	0.01	61–69	0	< 0.01	< 0.01	< 0.01	< 0.01

Location (Variety) Trial No./Report No.	Application						DALA	Residue, mg/kg			Mean residue, mg/kg
	kg ai/ha	n	Inter. days	L/ha	kg ai/hL	BBCH					Mean
GD-01/R-AC-15											
							1	0.036	0.028	0.034	0.033
							2	< 0.01	< 0.01	< 0.01	< 0.01
							3	0.022	0.025	0.025	0.024
							5	< 0.01	< 0.01	< 0.01	< 0.01
Hefei, Anhui (Jinyou No. 1) AH-01/R-AC-16	0.090	3	7	900	0.01	61–69	0	0.25	0.12	0.14	0.17
							1	0.13	0.11	0.14	0.13
							2	0.13	0.10	0.14	0.12
							3	< 0.01	0.13	0.082	0.074
							5	0.066	0.062	0.063	0.064

[a] Higher residue value was selected for an estimation of maximum residue level.

Table 6 Residues resulting from acetamiprid application to cucumber in greenhouse in China (2013)

Location (Variety) Trial No./Report No.	Application						DALA	Residue, mg/kg			Mean residue, mg/kg
	kg ai/ha	n	Int. days	L/ha	kg ai/hL	BBCH					
GAP, China	0.090	3					PHI, 2 days				
Qingdao, Shandong (Budaojuncheng) SD-02/R-AC-04	0.090	3	7	900	0.01	61–69	0	0.11	0.13	0.15	0.13
							1	0.090	0.10	0.14	0.11
							2	0.081	0.077	0.11	0.089
							3	0.085	0.049	0.072	0.069
							5	0.018	0.037	0.019	0.025
	0.090	3	7	900	0.01	61–69	0	0.11	0.091	0.10	0.10
							1	0.052	0.052	0.069	0.058
							2	0.062	0.069	0.037	0.056
							3	0.065	0.091	0.040	0.065
							5	0.012	0.016	0.014	0.014
Zhangzhou, Fujian (Jinyou No. 10) FJ-02/R-AC-07	0.090	3	7	900	0.01	61–69	0	0.032	0.021	0.064	0.039
							1	0.038	0.022	0.031	0.030

Location (Variety) Trial No./Report No.	Application						DALA	Residue, mg/kg			Mean residue, mg/kg
	kg ai/ha	n	Int. days	L/ha	kg ai/hL	BBCH					
							2	0.019	0.045	0.041	0.035
							3	0.026	0.024	< 0.01	0.020
							5	0.026	0.016	0.014	0.019
	0.090	3	7	900	0.01	61–69	0	0.084	0.11	0.11	0.10
							1	0.11	0.026	0.087	0.074
							2	0.074	0.061	0.080	0.072
							3	0.048	0.063	0.057	0.056
							5	0.029	0.038	0.038	0.035
Shuangliao, Jilin (Jinchun No. 25) JL-02/R-AC-10	0.090	3	7	900	0.01	61–69	0	0.056	0.13	0.069	0.085
							1	0.082	0.077	0.062	0.074
							2	0.044	0.049	0.055	0.049
							3	< 0.01	0.066	0.090	0.055
							5	< 0.01	0.038	0.040	0.029
Changchun, Jilin (Shengchun) JL-03/R-AC-11	0.090	3		900	0.01	61–69	0	0.019	0.027	0.021	0.022
							1	0.018	0.018	0.014	0.017
							2	0.025	0.027	0.028	0.027
							3	0.010	0.010	< 0.01	0.01
							5	< 0.01	< 0.01	< 0.01	< 0.01

Fruiting vegetables, other than Cucurbits

Tomato, Cherry tomato

Eight residue trials on field-grown tomatoes were conducted in China (Shandong, Fujian, Jilin, Yunnan, Guangdong, Zhejiang, Hunan and Anhui) in 2013. In addition, three trials (Shandong, Fujian and Jilin) on tomatoes and cherry tomatoes each were conducted under greenhouse conditions.

At each trial, one treated plot received two foliar applications of the test substance (30 ME formulation, 30 g ai/L) 7 days apart. Foliar spray application was made at growth stages of BBCH 79 and 83 and the application rate were 0.027 kg ai/ha. Tomatoes (or cherry tomatoes) were harvested 3, 5, 7, 10 and 14 days after the last application. Residue trials on tomato and cherry tomato made under greenhouse conditions were carried out in the same site of each region and at the same application time.

Two decline studies on field-grown tomato were also conducted in the Shandong and Fujian regions of China. One application was made at a rate of 0.041 kg ai/ha and tomato was harvested 0, 1, 3, 5, 7, 10, 14, 21, 28 and 35 days after the last application.

Six samples from each trial were harvested (1.2–4 kg per sample). From each sample, a sub sample of 132–400 g was taken and the sub samples were shredded in a food processor. Representative parts of the shredded samples were then transferred into polystyrene boxes and stored at –18 °C or below until analysis. Residue analysis was made with three samples of the six.

Table 7 Residues resulting from acetamiprid application to field-grown tomatoes in China (2013)

Location (Variety) Trial No./Report No.	Application						DALA	Residue, mg/kg			Mean residue, mg/kg
	kg ai/ha	n	Inter. days	L/ha	kg ai/hL	BBCH					
GAP, China	0.027	2					PHI, 7 days				
Qingdao, Shandong (Qingyan No. 1) FTAT-SD-01/AT-03	0.027	2	7	900	0.003	79, 83	3	0.031	0.024	0.027	0.027
							5	0.021	0.023	0.026	0.023
							7	0.017	0.021	< 0.01	0.016
							10	0.018	0.019	0.022	0.020
							14	0.013	0.016	< 0.01	0.013
Qingdao, Shandong (Qingyan No. 1) FTAT-SD-02/AT-03	0.041	1		900	0.005	79	0	0.015	0.032	0.020	0.022
							1	0.026	0.038	0.020	0.028
							3	0.011	0.023	0.012	0.015
							5	< 0.01	0.020	0.015	0.015
							7	0.021	0.012	0.015	0.016
							14	< 0.01	0.011	< 0.01	0.010
							21	< 0.01	< 0.01	< 0.01	< 0.01
							28	< 0.01	< 0.01	< 0.01	< 0.01
							35	< 0.01	< 0.01	< 0.01	< 0.01
Zhangzhou, Fujian (Yifeng) FTAT-FJ-01/AT-04	0.027	2	7	1,110	0.002	79, 83	3	0.011	0.018	0.014	0.014
							5	0.021	0.015	0.020	0.019
							7	0.014	0.025	0.026	0.022
							10	0.016	0.019	0.012	0.016
							14	0.011	< 0.01	0.012	0.011
Zhangzhou, Fujian (Yifeng) FTAT-FJ-02/AT-04	0.041	1		1,110	0.004	79	0	0.028	0.020	0.029	0.026
							1	0.018	0.024	0.017	0.020
							3	0.029	0.014	0.017	0.020
							5	0.020	0.015	0.021	0.019

Location (Variety) Trial No./Report No.	Application						DALA	Residue, mg/kg			Mean residue, mg/kg
	kg ai/ha	n	Inter. days	L/ha	kg ai/hL	BBCH					
							7	0.013	< 0.01	0.017	0.013
							14	0.012	0.010	0.010	0.011
							21	< 0.01	< 0.01	< 0.01	< 0.01
							28	< 0.01	< 0.01	< 0.01	< 0.01
							35	< 0.01	< 0.01	< 0.01	< 0.01
Changchun, Jilin (Jiafen No. 15) FTAT-JL-01/AT-05	0.027	2	7	900	0.003	79, 83	3	0.014	0.015	0.013	0.014
							5	0.011	0.011	0.011	0.011
							7	0.011	0.010	0.010	0.010
							10	0.011	0.011	< 0.01	0.011
							14	< 0.01	< 0.01	< 0.01	< 0.01
Kunming, Yunnan (Jingang) FTAT-YN-01/AT-06	0.027	2	7	800	0.003	79, 83	3	0.015	0.016	0.015	0.015
							5	0.023	0.023	0.027	0.024
							7	0.014	0.010	0.012	0.012
							10	0.011	< 0.01	0.011	0.011
							14	0.010	0.011	< 0.01	0.010
Guangzhou, Guangdong (Naishuhong) FTAT-GD-01/AT-07	0.027	2	7	1,000	0.003	79, 83	3	0.048	0.049	0.044	0.047
							5	0.039	0.052	0.035	0.042
							7	0.030	0.013	0.023	0.022
							10	0.028	0.019	0.017	0.021
							14	< 0.01	< 0.01	< 0.01	< 0.01
Hangzhou, Zhejiang (903#) FTAT-ZJ-01/AT-08	0.027	2	7	900	0.003	79, 83	3	< 0.01	< 0.01	0.013	0.011
							5	< 0.01	< 0.01	< 0.01	< 0.01
							7	< 0.01	< 0.01	< 0.01	< 0.01
							10	< 0.01	< 0.01	< 0.01	< 0.01
							14	< 0.01	< 0.01	< 0.01	< 0.01
Changsha, Hunan (Xianghong No. 5) FTAT-HN-01/AT-09	0.027	2	7	1,333	0.002	79, 83	3	< 0.01	0.011	0.014	0.012
							5	< 0.01	< 0.01	< 0.01	< 0.01
							7	< 0.01	< 0.01	0.012	0.011
							10	< 0.01	< 0.01	< 0.01	< 0.01
							14	< 0.01	< 0.01	< 0.01	< 0.01

Location (Variety) Trial No./Report No.	Application						DALA	Residue, mg/kg			Mean residue, mg/kg
	kg ai/ha	n	Inter. days	L/ha	kg ai/hL	BBCH					
Hefei, Anhui (Hezuo No. 908) FTAT-AH-01/AT-10	0.027	2	7	1,100	0.002	79, 83	3	0.053	0.063	0.040	0.052
							5	0.039	0.026	0.036	0.034
							7	0.027	0.023	0.026	0.025
							10	0.018	0.018	0.018	0.018
							14	0.013	0.016	0.013	0.014

30 ME (micro emulsion, 30%) formulation was used

BBCH79, 83:30% of fruits show typically fully ripe colour

Table 8 Residues resulting from acetamiprid application to tomatoes in greenhouse in China (2013)

Location (Variety) Trial No./Report No.	Application						DALA	Residue, mg/kg			Mean residue, mg/kg
	kg ai/ha	n	Inter. days	L/ha	kg ai/hL	BBCH					
GAP, China	0.027	2					PHI, 7 days				
Qingdao, Shandong (Fensha) FTAT-SD-03/AT-11	0.027	2	7	1,300	0.002	79, 83	3	0.039	0.026	0.031	0.032
							5	0.027	0.019	0.032	0.026
							7	0.028	0.027	0.027	0.027
							10	0.019	0.018	0.020	0.019
							14	0.019	0.013	0.015	0.016
Zhangzhou, Fujian (Israel No. 318) FTAT-FJ-03/AT-12	0.027	2	7	900	0.003	79, 83	3	0.011	< 0.01	< 0.01	0.011
							5	< 0.01	< 0.01	< 0.01	< 0.01
							7	< 0.01	< 0.01	< 0.01	< 0.01
							10	< 0.01	< 0.01	< 0.01	< 0.01
							14	< 0.01	< 0.01	< 0.01	< 0.01
Changchun, Jilin (Jiafen No. 15) FTAT-JL-02/AT-13	0.027	2	7	900	0.003	79, 83	3	0.013	0.014	0.014	0.014
							5	0.011	0.014	0.011	0.012
							7	< 0.01	< 0.01	< 0.01	< 0.01
							10	0.010	< 0.01	0.010	0.010
							14	0.023	0.010	0.012	0.015

Table 9 Residues resulting from acetamiprid application to cherry tomatoes in greenhouse in China (2013)

Location (Variety) Trial No./Report No.	Application						DALA	Residue, mg/kg			Mean residue, mg/kg
	kg ai/ha	n	Inter. days	L/ha	kg ai/hL	BBCH					
GAP, China	0.027	2					PHI, 7 days				
Qingdao, Shandong (Caiyu No. 3) FTAT-SD-04/AT-14	0.027	2	7	1,300	0.002	79, 83	3	0.057	0.050	0.054	0.054
							5	0.065	0.060	0.054	0.060
							7	0.051	0.043	0.055	0.050
							10	0.050	0.042	0.041	0.044
							14	0.046	0.039	0.042	0.042
Zhangzhou, Fujian (Israel No. 318) FTAT-FJ-04/AT-15	0.027	2	7	900	0.003	79, 83	3	0.019	0.016	0.013	0.016
							5	0.018	< 0.01	0.017	0.018
							7	< 0.01	0.010	0.012	0.011
							10	0.016	0.016	0.022	0.018
							14	0.021	0.014	0.020	0.018
Changchun, Jilin (Taiwan Shengnv) FTAT-JL-03/AT-16	0.027	2	7	900	0.003	79, 83	3	0.030	0.032	0.027	0.030
							5	0.022	0.025	0.032	0.026
							7	0.024	0.022	0.018	0.021
							10	0.019	0.020	0.022	0.020
							14	< 0.01	0.011	< 0.01	0.010

Sweet corn (corn-on-the-cob)

Eight trials were conducted in Canada (ON, BC, QC and AB) in 2006. At each site, sweet corn plants were treated with four applications (70 WP formulation, broadcast foliar spray) 6–8 days apart. The application rate ranged from 0.059 to 0.063 kg ai/ha (total, 0.24 to 0.25 kg ai/ha/season), with the exception of Trial No. 138. In that trial, the first three applications were made at 0.083–0.087 kg ai/ha and the forth application was made at 0.060 kg ai/ha (total, 0.32 kg ai/ha/season) due to calculation error. Samples of kernel plus cob with husk removed were collected 1–2 days after the last application. At Trial No. 131, additional samples were collected 0, 3 and 7 days after the last application.

In addition, seven trials were conducted in the USA (CA, FL, GA, ID, NY, SC and WI) in 2009. One treated plot received four foliar applications of the test substance (30 SG formulation) 6–8 days apart, except in the GA*14 trial in which the intervals were as short as 4 days. The application rates were in the range 0.059 to 0.064 kg ai/ha (total, 0.24 to 0.25 kg ai/ha/season). A second treated plot received two foliar applications of the test substance (30 SG formulation) 12–16 days apart. The application rates were in the range 0.11 to 0.12 kg ai/ha (total, 0.22 to 0.24 kg ai/ha/season).

Samples of kernel plus cob with husk removed were collected one day from the four application plot and 5–8 days from the two application plot, after the last application.

Table 10 Residues resulting from acetamiprid application to sweet corn in Canada in 2006 (Report: AAFC06-034R)

Location (Variety)	Application			DALA	Residue, mg/kg	Mean residue, mg/kg	Trial No.
	kg ai/ha	n	Inter. days				
GAP, USA	0.11	2	14	PHI, 7 days			
	0.060	4	7	PHI, 1 days			
Delhi, ON (Fleet Bicolor)	0.060–0.061	4	7–8	0	< 0.01, < 0.01	< 0.01	131
				1	< 0.01, < 0.01	< 0.01	
				3	< 0.01, < 0.01	< 0.01	
				7	< 0.01, < 0.01	< 0.01	
Delhi, ON (Lancelot Bicolor)	0.061–0.063	4	7	1	< 0.01, < 0.01	< 0.01	132
London, ON (Trinity Bicolor)	0.060–0.061	4	7	1	< 0.01, < 0.01	< 0.01	133
London, ON (Accord)	0.060–0.061	4	7	1	< 0.01, < 0.01	< 0.01	134
Agassiz, BC (Gourmet Sweet Brand 276A)	0.059–0.061	4	7	2	< 0.01, < 0.01	< 0.01	135
L'Acadie, QC (Fleet)	0.059–0.062	4	7	1	< 0.01, < 0.01	< 0.01	136
L'Acadie, QC (Trinity)	0.059–0.062	4	7	1	< 0.01, < 0.01	< 0.01	137
Taber, AB (XtraSweet 82)	0.060–0.087 [a]		6–8	1	< 0.01, < 0.01	< 0.01	138

70 WP formulation was used; residue in kernel plus cob with husk removed was analysed.

[a] The first three applications were over applied due to calculation error (1st, 0.086 kg ai/ha; 2nd; 0.083 kg ai/ha; 3rd 0.087 kg ai/ha; 4th, 0.060 kg ai/ha).

Table 11 Residues resulting from acetamiprid application to sweet corn in the USA in 2009 (Report: IR-4 PR No. 10216)

Location (Variety)	Application			DALA	Residue, mg/kg	Mean residue, mg/kg	Trial No.
	kg ai/ha	n	Inter. days				
GAP, USA	0.11	2	14	PHI, 7 days			
	0.060	4	7	PHI, 1 days			
Holtville, CA (Boreal)	0.11, 0.11	2	16	7	< 0.01, < 0.01	< 0.01	CA102
	0.059–0.061	4	6–8	1	< 0.01, < 0.01	< 0.01	
Citra, FL	0.11, 0.11	2	14	7	< 0.01, < 0.01	< 0.01	FL04

Location (Variety)	Application			DALA	Residue, mg/kg	Mean residue, mg/kg	Trial No.
	kg ai/ha	n	Inter. days				
(Obsession (SH2 bicolor))							
	0.059	4	7	1	< 0.01, < 0.01	< 0.01	
Tifton, GA (XTRA-Tender Brand 270A F1 bicolor Super Sweet)	0.11, 0.11	2	12	5	< 0.01, < 0.01	< 0.01	GA*14
	0.061–0.062	4	4–7	1	< 0.01, < 0.01	< 0.01	
Kimberly, ID (Bodacious)	0.11, 0.11	2	13	7	< 0.01, < 0.01	< 0.01	ID17
	0.061–0.062	4	6–7	1	< 0.01, < 0.01	< 0.01	
North Rose, NY (Attribute)	0.11, 0.11	2	14	7	< 0.01, < 0.01	< 0.01	NY14
	0.061	4	7	1	< 0.01, < 0.01	< 0.01	
Charleston, SC (Accelerator)	0.12, 0.12	2	14	8	< 0.01, < 0.01	< 0.01	SC*01
	0.061–0.064	4	7	1	< 0.01, < 0.01	< 0.01	
Arlington, WI (Jubilee Supersweet)	0.12, 0.12	2	14	8	< 0.01, < 0.01	< 0.01	WI15
	0.061–0.063	4	7	1	< 0.01, < 0.01	< 0.01	

30 SG (30% soluble granule) formulation was used; residue in kernel plus cob with husk removed was analysed.

Leafy vegetables (incl. Brassica leafy vegetables)

Mustard greens

Eight supervised residue trials were conducted in the USA (AR, CA, GA, NC, OH, SC and TX) in 2009. At each trial, four foliar applications of the test substance (70 WP) were made 6–8 days apart, except in the GA*06 trial where a fifth application was needed because the crop was not mature after four applications. The application rates were in the range of 0.083–0.12 kg ai/ha/application. The total rate range per growing season was 0.42–0.43 kg ai/ha (GA*06 trial, 0.53 kg ai/ha). A non-ionic surfactant was included in the tank mix for each application. Samples of mustard green leaves were collected 2–4 days after the last application

Table 12 Residues resulting from acetamiprid application to mustard greens in the USA in 2009 (Report: IR-4 PR No. 09271)

Location (Variety) Year	Application			DALA	Residue, mg/kg	Mean residue, mg/kg	Trial No.
	kg ai/ha	n.	Inter. days				
GAP, USA	0.11	4	7	PHI, 3 days			
Alma, AR (Florida Broadleaf)	0.085–0.12	4	6–8	3	8.4, 10	9.2	AR07
Salinas, CA (Red Giant)	0.084–0.12	4	7–8	3	2.4 2.9	2.7	CA*51
Salinas, CA (Green Wave)	0.087–0.12	4	7	4	1.5, 1.6	1.6	CA*52
Tifton, GA (Florida Broadleaf)	0.083–0.11	5 [a]	7–8	3	2.1, 2.2	2.2	GA*06
Clinton, NC (Southern Giant Curled)	0.083–0.11	4	7–8	4	1.2, 1.2	1.2	NC10

Location (Variety) Year	Application			DALA	Residue, mg/kg	Mean residue, mg/kg	Trial No.
	kg ai/ha	n.	Inter. days				
Willard, OH (Green Wave)	0.090–0.12	4	7	2	0.20, 0.40	0.30	OH*04
Charleston, SC (Florida Broadleaf)	0.085–0.11	4	6–8	3	1.4, 2.0	1.7	SC*03
Weslaco, TX (Florida Broadleaf)	0.087–0.12	4	6–7	2	2.4, 2.4	2.4	TX*19

70 WP (70% wettable powder) was applied in all trials.

CA*51 and CA*52 trials were conducted two months apart.

[a] Extra treatment was made as samples were maturing too slowing due to excessive rain. A total rate was 0.53 kg ai/ha/season.

Stalk and stem vegetables

Asparagus

Eight supervised residue trials were conducted in the USA (CA, ID, MD, MI, WA) in 2008 and 2009. At each trial, two applications of the test substance (70 WP) were made 10–14 days apart. The application rates were in the range of 0.11–0.12 kg ai/ha/application. Non-ionic surfactant was included in the tank mix in trials CA34, ID05, MI33, MI34 and in the second application of WA06. Samples of asparagus spears were harvested one day after the last application. One decline study (CA37 trial) was conducted and samples were collected 0, 1, 4, 8 and 11 days after the last application.

Table 13 Residues resulting from acetamiprid application to asparagus in the USA (Report: IR-4 PR No. 09939)

Location (Variety) Year	Application			DALA	Residue, mg/kg	Mean residue, mg/kg	Trial No.
	kg ai/ha	n	Inter. days				
GAP, USA	0.11	2	10	PHI, 1 days			
San Ardo, CA (UC157) 2009	0.11, 0.12	2	10	1	0.21, 0.21	0.21	CA34
Merritt, CA (Apollo) 2008	0.11, 0.11	2	14	1	0.16, 0.16	0.16	CA35*
Merritt, CA (Apollo) 2008	0.11, 0.11	2	14	0	0.25, 0.25	0.25	CA37*
				1	0.26, 0.26	0.26	
				4	0.08, 0.08	0.08	
				8	0.01, 0.01	0.01	
				11	< 0.01, < 0.01	< 0.01	
Marsing, ID (Jersey King) 2008	0.11, 0.11	2	12	1	0.38, 0.43	0.41	ID05
Sailsbury, MD (Jersey Knight) 2008	0.11, 0.11	2	11	1	0.11, 0.13	0.12	MD17
East Lansing, MI (Jersey Giant) 2008	0.11, 0.11	2	12	1	0.28, 0.29	0.29	MI33
East Lansing, MI (Jersey Giant) 2008	0.11, 0.12	2	12	1	0.26, 0.26	0.26	MI34
Eltopia, WA (Jersey Knight) 2008	0.11, 0.11	2	13	1	0.25, 0.27	0.26	WA06

70 WP (70% wettable powder) was applied in all trials.

CA35* and CA37* trials were conducted at the same site however application was made 7 days apart. These trials were considered as independent as asparagus is shortly grown for 7 days.

Primary feed commodities

Sweet corn, forage and stover

Residue trials on sweet corn were conducted in Canada (eight trials in 2006) and the USA (seven trials in 2009). Application methods of test substance are described above in food commodity of sweet corn (corn-on-the-cob). Forage samples were collected on the same day as harvesting samples of kernel plus cob with husk removed in both Canada and USA.

For stover in Canada, samples (stalks with ear removed) were collected 38–89 days after the last application. This was a period after the ears were harvested and allowed to dry, free-standing in the field.

In the USA, stover samples were collected concurrently with sampling of forage (except Trial No. NY14). The samples (stalks with ear removed) were cut and dried (either in the field or in a sheltered area/low temperature "oven"). In the NY14 trial, stover samples were not cut and were allowed to dry in the field before being harvested. Harvesting was 35 days and 28 days after four and two applications of the test substance, respectively.

Table 14 Residues on forage resulting from acetamiprid application to sweet corn in Canada in 2006 (Report: AAFC06-034R)

Location (Variety)	Application			DALA	Residue, mg/kg	Mean residue, mg/kg	Trial No.
	kg ai/ha	n	Inter. days				
GAP, USA	0.11	2	14	PHI, 7 days			
	0.060	4	7	PHI, 1 days			
Delhi, On (Fleet Bicolor)	0.060–0.061	4	7–8	0	0.50, 0.72	0.61	131
				1	0.62, 0.87	0.75	
				3	0.20, 0.28	0.24	
				7	0.10, 0.17	0.14	
Delhi, On (Lancelot Bicolor)	0.061–0.063	4	7	1	0.43, 0.60	0.52	132
London, ON (Trinity Bicolor)	0.060–0.061	4	7	1	0.23, 0.25	0.24	133
London, ON (Accord)	0.060–0.061	4	7	1	0.60, 0.62	0.61	134
Agassiz, BC (Gourmet Sweet Brand 276A)	0.059–0.061	4	7	2	0.60, 0.62	0.61	135
L'Acadie, QC (Fleet)	0.059–0.062	4	7	1	1.0, 1.1	1.1	136
L'Acadie, QC (Trinity)	0.059–0.062	4	7	1	0.52, 0.62	0.57	137
Taber, AB (XtraSweet 82)	0.060–0.087[a]	4	6–8	1	0.48, 0.49	0.49	138

70 WP formulation was used.

[a] The first three applications were over applied due to calculation error (1st, 0.086 kg ai/ha; 2nd; 0.083 kg ai/ha; 3rd 0.087 kg ai/ha; 4th, 0.060 kg ai/ha).

Table 15 Residues on forage resulting from acetamiprid application to sweet corn in the USA in 2009 (Report: IR-4 PR No. 10216)

Location (Variety)	Application			DALA	Residue, mg/kg	Mean residue, mg/kg	Trial No.
	kg ai/ha	No.	Inter. days				
GAP, USA	0.11	2	14	PHI, 7 days			
	0.060	4	7	PHI, 1 days			
Holtville, CA (Boreal)	0.11, 0.11	2	16	7	8.1, 10	9.1	CA102
	0.059–0.061	4	6–8	1	6.5, 8.1	7.3	
Citra, FL (Obsession (SH2 bicolor))	0.11, 0.11	2	14	7	0.39, 0.43	0.41	FL04
	0.059	4	7	1	0.42, 0.65	0.54	
Tifton, GA (XTRA-Tender Brand 270A F1 bicolor Super Sweet)	0.11, 0.11	2	12	5	0.68, 0.84	0.76	GA*14
	0.061–0.062	4	4–7	1	0.83, 0.87	0.85	
Kimberly, ID (Bodacious)	0.11, 0.11	2	13	7	4.4, 4.9	4.7	ID17
	0.061–0.062	4	6–7	1	5.8, 6.7	6.3	
North Rose, NY (Attribute)	0.11, 0.11	2	14	7	2.1, 2.8	2.4	NY14
	0.061	4	7	1	0.95, 1.4	1.2	
Charleston, SC (Accelerator)	0.12, 0.12	2	14	8	1.2, 1.5	1.4	SC*01
	0.061–0.064	4	7	1	1.2, 1.3	1.3	
Arlington, WI (Jubilee Supersweet)	0.12, 0.12	2	14	8	1.4, 1.4	1.4	WI15
	0.061–0.063	4	7	1	3.2, 3.5	3.4	

30 SG (30% soluble granule) formulation was used.

Table 16 Residues on stover resulting from acetamiprid application to sweet corn in Canada in 2006 (Report: AAFC06-034R)

Location (Variety)	Application			DALA	Residue, mg/kg	Mean residue, mg/kg	Trial No.
	kg ai/ha	No.	Inter. days				
GAP, USA	0.11	2	14	PHI, 7 days			
	0.060	4	7	PHI, 1 days			
Delhi, On (Fleet Bicolor)	0.060–0.061	4	7–8	70	0.030, 0.035	0.033	131
				76	0.029, 0.047	0.038	
				83	0.015, 0.017	0.016	

Location (Variety)	Application			DALA	Residue, mg/kg	Mean residue, mg/kg	Trial No.
	kg ai/ha	No.	Inter. days				
				90	0.018, 0.018	0.018	
Delhi, On (Lancelot Bicolor)	0.061–0.063	4	7	83	0.029, 0.034	0.032	132
London, ON (Trinity Bicolor)	0.060–0.061	4	7	89	< 0.01, 0.016	0.013	133
London, ON (Accord)	0.060–0.061	4	7	82	< 0.01, < 0.01	< 0.01	134
Agassiz, BC (Gourmet Sweet Brand 276A)	0.059–0.061	4	7	43	0.16, 0.17	0.17	135
L'Acadie, QC (Fleet)	0.059–0.062	4	7	41	0.13, 0.19	0.16	136
L'Acadie, QC (Trinity)	0.059–0.062	4	7	41	0.20, 0.23	0.22	137
Taber, AB (XtraSweet 82)	0.060–0.087[a]		6–8	38	0.76, 0.94	0.85	138

70 WP formulation was used. Residues are expressed on a dry matter basis, ca. 83%.

[a] The first three applications were over applied due to calculation error (1st, 0.086 kg ai/ha; 2nd; 0.083 kg ai/ha; 3rd 0.087 kg ai/ha; 4th, 0.060 kg ai/ha).

Table 17 Residues on stover resulting from acetamiprid application to sweet corn in the USA in 2009 (Report: IR-4 PR No. 10216)

Location (Variety)	Application				DALA	Moisture content (%)	Residue, mg/kg	Mean residue, mg/kg	Trial No.
	kg ai/ha	No.	Growth stage	Inter. days					
GAP, USA	0.11	2		14	PHI, 7 days				
	0.060	4		7	PHI, 1 days				
Holtville, CA (Boreal)	0.11, 0.11	2	Early silk	16	7	15–20	19, 21	20	CA102
			Ears						
	0.059–0.061	4	Early silk	6–8	1		15, 16	16	
			Ear fill						
			Ears						
			Mature ears						
Citra, FL (Obsession (SH2 bicolor))	0.11, 0.11	2	Corn ear stage	14	7	20	0.21, 0.21	0.21	FL04
			Corn ear stage						
	0.059	4	Corn ear stage	7	1		0.12, 0.26	0.19	
			Corn ear stage						

Location (Variety)	Application				DALA	Moisture content (%)	Residue, mg/kg	Mean residue, mg/kg	Trial No.
	kg ai/ha	No.	Growth stage	Inter. days					
			Corn ear stage						
			Corn ear stage						
Tifton, GA (XTRA-Tender Brand 270A F1 bicolor Super Sweet)	0.11, 0.11	2	Fruiting	12	5	15–20	2.7, 3.0	2.8	GA*14
			Fruiting						
	0.061–0.062	4	Fruiting	4–7	1		3.3, 4.9	4.1	
			Fruiting						
			Fruiting						
			Fruiting						
Kimberly, ID (Bodacious)	0.11, 0.11	2	Ear growth	13	7	17	8.0, 8.7	8.4	ID17
			Maturing						
	0.061–0.062	4	Ear growth	6–7	1		10, 13	12	
			Ear growth						
			Maturing						
			Maturing						
North Rose, NY (Attribute)	0.11, 0.11	2	Early silk	14	35	–	0.43, 0.46	0.45	NY14
			Brown silk						
	0.061	4	Early silk	7	28		0.33, 0.53	0.43	
			Early kernel formation						
			Brown silk						
			Commerciall y fresh ears						
Charleston, SC (Accelerator)	0.12, 0.12	2	Blooming	14	8	15–20	2.4, 3.1	2.8	SC*01
			Fruiting						
	0.061–0.064	4	Blooming	7	1		3.0, 3.4	3.2	
			Fruiting						
			Fruiting						
			Fruiting						
Arlington, WI (Jubilee Supersweet)	0.12, 0.12	2	Reproductive	14	8	20	2.5, 2.6	2.6	WI15
			Reproductive						
	0.061–0.063	4	Reproductive	7	1		4.3, 5.2	4.8	
			Reproductive						
			Reproductive						
			Reproductive						

30 SG (30% soluble granule) formulation was used. Residues are expressed on a dry matter.

APPRAISAL

Acetamiprid was evaluated for the first time by the 2011 JMPR, where an ADI of 0–0.07 mg/kg bw and an ARfD of 0.1 mg/kg bw were established and maximum residue levels were recommended for a range of plant and animal commodities. The compound was re-evaluated by the 2012 JMPR.

At the Forty-sixth Session of the CCPR (2014), acetamiprid was listed for residue evaluation for additional maximum residue levels by the 2015 JMPR. The Meeting received information on supervised residue trials for asparagus, cucumber, mustard greens, sweet corn (corn-on-the-cob) and tomato including cherry tomatoes.

For both compliance with MRL and estimation of dietary intake, the residue is defined as acetamiprid for plant commodities, and the sum of acetamiprid and desmethyl-acetamiprid for animal commodities. The residue is not fat-soluble.

Methods of analysis

Acceptable analytical methods were developed and validated for determination of acetamiprid in asparagus, mustard greens and sweet corn. These methods were based on Method KP-216 which was considered suitable by 2011 JMPR. Other analytical methods used for sweet corn, cucumber and tomato were also fully validated. All methods used analysis by LC-MS/MS and the limits of quantification (LOQs) were 0.01 mg/kg in all matrices.

Stability of residues in stored analytical samples

In 2011, JMPR concluded that acetamiprid is stable for at least 12 months in apple, cabbage, cucumber and 16 months for lettuce.

The present Meeting received acetamiprid stability studies on asparagus, cucumber, mustard greens, sweet corn and tomato, showing that residues were stable under frozen condition for at least 426 days for asparagus, 304 days for cucumber and tomato, 382 days for mustard greens and 384–391 days for sweet corn samples (kernel plus cob with husk removed, forage and stover).

Based on the available storage stability information, the Meeting concluded that acetamiprid was stable for the period of actual storage days associated with the submitted residue trials.

Results of supervised residue trials on crops

Fruiting vegetables, Cucurbits

Cucumber

Supervised trials were conducted in China in 2013, matching the China GAP on cucumber (3 sprays applications at 0.090 kg ai/ha and a PHI of 2 days). Eight trials were conducted under field conditions. Another six trials were conducted under greenhouse conditions, two trials of which were not independent and another two trials were also not independent. Additionally, two decline studies on field-grown cucumber were conducted with one application at a rate of 0.090 kg ai/ha. The residues decreased with a half-life of 2.1 or 3.9 days.

From residue trials matching the China GAP on cucumber, acetamiprid residue values were as follows:

Field-grown cucumber (n=8): 0.011, 0.020, 0.024, 0.042, 0.059, 0.070, 0.12 and 0.13 mg/kg.

Greenhouse-grown cucumber (n=4): 0.027, 0.055, 0.072 and 0.089 mg/kg.

As the residue distributions of acetamiprid between field-grown and greenhouse-grown cucumber were similar, residue values were combined (n=12): 0.011, 0.020, 0.024, 0.027, 0.042, 0.055, 0.059, 0.070, 0.072, 0.089, 0.12 and 0.13 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg, an STMR of 0.057 mg/kg and an HR of 0.17 mg/kg (based on a highest single sample) for cucumber.

Further, the Meeting withdrew its previous recommendations for Fruiting vegetables, Cucurbits and estimated a maximum residue level of 0.2 mg/kg, an STMR of 0.05 mg/kg and an HR of 0.11 mg/kg for Fruiting vegetables, Cucurbits (except cucumber).

Fruiting vegetables, other than Cucurbits

Tomato

Supervised trials were conducted in China in 2013, matching the China GAP on tomato (2 sprays at 0.027 kg ai/ha and a PHI of 7 days). Eight trials on tomato were conducted under field conditions and an additional three trials on each of tomato and cherry tomato were conducted under greenhouse conditions. Additionally, two decline studies on field-grown tomato were conducted with one application at a rate of 0.041 kg ai/ha. The residues decreased with an average half-life of 11.6 days.

From residues trials matching the China GAP on tomato, acetamiprid residue values were as follows:

Field-grown tomato (n=8): < 0.01, 0.011, 0.011, 0.012, 0.020, 0.022, 0.022 and 0.025 mg/kg.

Greenhouse-grown tomato (n=3): < 0.01, 0.015 and 0.027 mg/kg.

Greenhouse-grown cherry tomato (n=3): 0.018, 0.021 and 0.050 mg/kg.

The 2011 JMPR recommended a maximum residue level of 0.2 mg/kg, an STMR of 0.04 mg/kg and an HR of 0.14 mg/kg for Fruiting vegetables, other than Cucurbits, based on residues in tomato (outdoor), sweet pepper and chili pepper conducted according to the US GAP (four foliar applications at 0.084 kg ai/ha and a PHI of 7 days). Since the authorization in the US represents the critical GAP, this Meeting confirmed its previous recommendations for Fruiting vegetables, other than Cucurbits (except sweet corn & mushrooms).

Sweet corn

Seven trials were conducted in the USA in 2009, matching a critical US GAP (two foliar sprays at 0.11 kg ai/ha with a 14-day retreatment interval and a PHI of 7 days). Residue concentrations in sweet corn (kernel plus cob with husk removed) from the USA trials were all < 0.01 mg/kg (n=7).

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 sweet corn (corn-on-the-cob).

Leafy vegetables (including Brassica leafy vegetables)

Mustard greens

Eight trials on mustard greens were conducted in the USA in 2009, matching the US GAP (four foliar sprays at 0.11 kg ai/ha with a 7-day retreatment interval and a PHI of 3 days).

Acetamiprid residues in mustard greens were (8): 0.30, 1.2, 1.6, 1.7, 2.2, 2.4, 2.7 and 9.2 mg/kg.

The Meeting estimated a maximum residue level of 15 mg/kg, an STMR of 2.0 mg/kg and an HR of 10 mg/kg (based on highest single sample) for mustard greens. However, this would result in an exceedance of the ARfD and an alternative GAP for mustard greens was not identified.

Stalk and stem vegetables

Asparagus

Eight trials on asparagus were conducted in the USA in 2008 and 2009, matching the US GAP (two sprays at 0.11 kg ai/ha with a 10-day retreatment interval and a PHI of 1 day).

Acetamiprid residues in asparagus were (n=8): 0.12, 0.16, 0.21, 0.26 (3), 0.29 and 0.41 mg/kg.

The Meeting estimated a maximum residue level of 0.8 mg/kg, an STMR of 0.26 mg/kg and an HR of 0.43 mg/kg (based on highest single sample) for asparagus.

Primary feed commodities

Sweet corn forage and stover

The trial conditions are described under the food commodity. For feed commodity, sweet corn forage and stover samples were harvested in the seven USA trials. In one trial, the PHI in sampling of stover did not match the US GAP.

Acetamiprid residues in sweet corn forage were (n=7): 0.41, 0.76, 1.4, 1.4, 2.4, 4.7 and 9.1 mg/kg.

Acetamiprid residues in sweet corn stover were (n=6): 0.21, 2.6, 2.8, 2.8, 8.4 and 20 mg/kg.

The Meeting estimated a median residue of 1.4 mg/kg and highest residue of 9.1 mg/kg for sweet corn forage.

The Meeting estimated a maximum residue level of 40 mg/kg, median residue level of 2.8 mg/kg and highest residue of 20 mg/kg on a dry weight basis for sweet corn stover.

Residues in animal commodities

Livestock dietary burden

Dietary burden calculations considered by the current Meeting for beef cattle and dairy cattle, incorporating sweet corn, are presented in Annex 6. Dietary burdens for poultry were not calculated as sweet corn (forage, stover and cannery waste) is not a relevant feed item.

The dietary burdens for beef cattle and dairy cattle were estimated using OECD diets listed in Appendix IX of the 2009 edition of the FAO Manual.

Summary of cattle dietary burdens (ppm of dry matter diet)

	US-Canada		EU		Australia	
	max	mean	max	mean	max	mean
Beef cattle	1.1	0.29	0.83	0.28	18[a]	2.7[b]
Dairy cattle	9.5[c]	1.6	0.84	0.29	9.0	1.7[d]

[a] Highest maximum beef or dairy cattle dietary burden suitable for maximum residue level estimates for mammalian meat and edible offal

[b] Highest mean beef of dairy cattle dietary burden suitable for STMR estimates for mammalian meat and edible offal

[c] Highest maximum dairy cattle dietary burden suitable for maximum residue level estimates for milk

[d] Highest mean dairy cattle dietary burden suitable for STMR estimates for milk

Animal commodity maximum residue levels

Livestock feeding studies involving administration of acetamiprid to dairy cows were reported in the 2011 JMPR Report.

Estimated maximum and mean dietary burdens were 18 ppm and 2.7 ppm for beef cattle and 9.5ppm and 1.7 ppm for dairy cattle, respectively. The calculation to estimate total residues

(acetamiprid plus desmethyl-acetamiprid) for 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
Maximum residue level beef or dairy cattle							
Feeding study [a]	5.77	0.063					
	17.4	0.209	17.4	0.289	0.64	0.86	0.153
Dietary burden and residue estimate	9.5	0.11	18	0.30	0.67	0.89	0.16
STMR beef or dairy cattle							
Feeding study [b]	5.77	0.063	5.77	0.048	0.15	0.24	0.037
Dietary burden and residue estimate	1.7	0.019	2.7	0.022	0.070	0.11	0.017

[a] Highest residues for tissues and mean residue for milk

[b] Mean residues for tissues and milk

For beef and dairy cattle, the Meeting estimated HR values for acetamiprid (total residue) of 0.30 mg/kg in muscle, 0.89 mg/kg in edible offal (based on kidney) and 0.16 mg/kg in fat. STMR values were estimated at levels of 0.019 mg/kg for milk, 0.022 mg/kg for muscle, 0.11 mg/kg in edible offal (based on kidney) and 0.017 mg/kg for fat.

The Meeting also estimated the following maximum residue levels to replace its previous recommendations: 0.2 mg/kg for milk, 0.5 mg/kg for meat (from mammals other than marine mammals), 0.3 mg/kg for mammalian fats (except milk fats) and 1.0 mg/kg for edible offal (mammalian).

The previous recommendations for poultry tissues and eggs are maintained.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed in Annex I are appropriate for establishing maximum residue limits and for IEDI and IESTI assessment.

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

Definition of the residue for animal commodities (for compliance with MRL and estimation of dietary intake): *sum of acetamiprid and desmethyl-acetamiprid, expressed as acetamiprid*.

The residue is not fat-soluble.

CCN	**Commodity**	**Recommended Maximum residue level (mg/kg)**		**STMR or STMR-P mg/kg**	**HR or HR-P mg/kg**
		New	**Previous**		
VS 0621	Asparagus	0.8		0.26	0.43
VC 0424	Cucumber	0.3		0.057	0.17
MO 0105	Edible offal (mammalian)	1	0.05	0.11	0.89
VC 0045	Fruiting vegetables, Cucurbits	W	0.2		
VC 0045	Fruiting vegetables, Cucurbits (except Cucumber)	0.2		0.05	0.11
MF 0100	Mammalian fats (except milk fats)	0.3	0.02	0.017	0.16
MM 0095	Meat (from mammals other than marine mammals)	0.5	0.02	0.022 (m) 0.017 (f)	0.30 (m) 0.16 (f)
ML 0106	Milks	0.2	0.02	0.019	
VL 0485	Mustard greens	15 [a]		2.0	10
VO 0447	Sweet corn (corn-on-the-cob)	0.01*		0.01	0.01

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
	Sweet corn, stover	40		2.8	20
	Sweet corn forage			1.4	9.1

[a] On the basis of information provided to the JMPR it was not possible to conclude that the estimated short-term intake of acetamiprid for consumption of mustard greens was less than the ARfD

DIETARY RISK ASSESSMENT

Long-term intake

The WHO panel of the 2011 JMPR established an ADI of 0–0.07 mg/kg bw for acetamiprid. The International Estimated Daily Intakes (IEDIs) for acetamiprid were calculated for the 17 GEMS/Food cluster diets using STMRs and STMR-Ps estimated by the current and previous Meeting. The results are shown in Annex 3 in the 2015 JMPR Report.

The calculated IEDIs represented 0–4% of the maximum ADI. The Meeting concluded that the long-term intake of residues of acetamiprid from used that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The WHO panel of the 2011 JMPR established an ARfD of 0.1 mg/kg. The International Estimated Short Term Intakes (IESTIs) for acetamiprid was calculated for the food commodities using HR/ STMR estimated by the current Meeting. The results are shown in Annex 4 in the 2015 JMPR Report.

For mustard greens, the IESTI represented 490% and 200% of the ARfD for children and general population, respectively. No alternative GAP was available. On the basis of information provided to the JMPR, the meeting concluded that the short-term intake of acetamiprid from consumption of mustard greens may present a public health concern.

Estimates of intake for the other commodities considered by the 2015 JMPR were within 0–10% ARfD. The Meeting concluded that the short-term intake of acetamiprid for these other commodities is unlikely to present a public health concern when acetamiprid is used in ways that were considered by the Meeting.

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AC-02	Li, Yiqiang	2013	Cucumber: Stability during deep freeze storage up to 10 months Acetamiprid Active substance. Institute for Control of Agrochemicals, Ministry of Agriculture, P.R. China
AT-01	Li, Zhou	2012	Method Performance Verification for the Determination of Residues of Acetamiprid in Tomato by LC-MS/MS. Institute for Control of Agrochemicals, Ministry of Agriculture, P. R. China
AT-02	Li, Zhou	2013	Tomato: Stability during deep freeze storage up to 10 months. Institute for Control of Agrochemicals, Ministry of Agriculture, P. R. China
AT-03	Li, Zhou	2013	Determination of the residues of acetamiprid in tomato after spraying of 30 g ai/L Acetamiprid ME in the open field in Shandong. Institute for Control of Agrochemicals, Ministry of Agriculture, P. R. China
AT-04	Li, Zhou	2013	Determination of the residues of acetamiprid in tomato after spraying of 30 g ai/L 7Acetamiprid ME in the open field in Fujian.

Code	Author	Year	Title, Institute, Report reference
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R-AC-03/05	Wang, Xiuguo	2013	Determination of the residues of acetamiprid on cucumber after spraying of 20% acetamiprid SP in open field in Shandong Province, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture, P. R. China
R-AC-04	Wang, Xiuguo	2013	Determination of the residues of acetamiprid on fruit cucumber after spraying of 20% acetamiprid SP in greenhouse in Shandong Province, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture, P. R. China
R-AC-04	Wang, Xiuguo	2013	Determination of the residues of acetamiprid on cucumber after spraying of 20% acetamiprid SP in greenhouse in Shandong Province, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture, P. R. China

Code	Author	Year	Title, Institute, Report reference
R-AC -06/08	Wang, Xiuguo	2013	Determination of the residues of acetamiprid on cucumber after spraying of 20% acetamiprid SP in Open Field in Fujian Province, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture, P. R. China
R-AC -07	Wang, Xiuguo	2013	Determination of the residues of acetamiprid on fruit cucumber after spraying of 20% acetamiprid SP in greenhouse in Fujian Province, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture, P. R. China
R-AC -07	Wang, Xiuguo	2013	Determination of the residues of acetamiprid on cucumber after spraying of 20% acetamiprid SP in greenhouse in Fujian Province, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture, P. R. China
R-AC-09	Wang, Xiuguo	2013	Determination of the residues of acetamiprid on cucumber after spraying of 20% acetamiprid SP in open field in Jilin Province, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture, P. R. China
R-AC -10	Wang, Xiuguo	2013	Determination of the residues of acetamiprid on cucumber after spraying of 20% acetamiprid SP in Greenhouse in Jilin Province, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture, P. R. China
R-AC -11	Wang, Xiuguo	2013	Determination of the residues of acetamiprid on fruit cucumber after spraying of 20% acetamiprid SP in greenhouse in Jilin Province, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture, P. R. China
R-AC -12	Wang, Xiuguo	2013	Determination of the residues of acetamiprid on cucumber after spraying of 20% acetamiprid SP in open field in Hunan, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture, P. R. China
R-AC -13	Wang, Xiuguo	2013	Determination of the residues of acetamiprid on cucumber after spraying of 20% acetamiprid SP in open field in Zhejiang Province, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture, P. R. China
R-AC -14	Wang, Xiuguo	2013	Determination of the residues of acetamiprid on cucumber after spraying of 20% acetamiprid SP in open field in Yunnan, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture, P. R. China
R-AC -15	Wang, Xiuguo	2013	Determination of the residues of acetamiprid on cucumber after spraying of 20% acetamiprid SP in open field in Guangdong Province, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture, P. R. China
R-AC-16	Wang, Xiuguo	2013	Determination of the residues of acetamiprid on cucumber after spraying of 20% acetamiprid SP in open field in Anhui Province, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture, P. R. China

ACETOCHLOR (280)

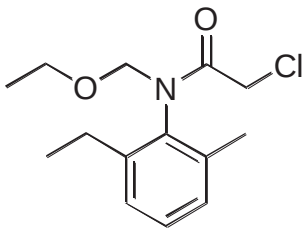
First draft prepared by Dr D.J. MacLachlan, Department of Agriculture and Water Resources, Canberra, Australia

EXPLANATION

Acetochlor is a selective herbicide, which after application is absorbed mainly by the shoots of germinating plants and to some extent by roots. Acetochlor controls annual grasses and broadleaf weeds, germinating from seeds; however, its action against perennial weeds is very limited. Acetochlor is a pre-emergence or early post-emergence soil-applied herbicide for the control of annual grasses and certain annual broadleaf weeds. At the 46th Session of the CCPR (2014), it was scheduled for evaluation as a new compound by 2015 JMPR.

The Meeting received information on the metabolism of acetochlor in lactating goats and cows, laying hens, maize, soya beans and cotton, follow crops, methods of residue analysis, freezer storage stability, GAP information, supervised residue trials on maize (forage, grain, stover and silage), sweet corn (forage, kernels plus cob with husks removed, stover and silage), cotton (gin by-products and seed), sorghum (grain, forage and stover), soya bean (meal and seed), sugar beet (dried pulp, roots, tops, sugar and molasses), peanut (hay and meal) and livestock transfer studies (lactating cows and laying hens).

IDENTITY

Common name	Acetochlor
Chemical name	
IUPAC:	2-chloro-*N*-ethoxymethyl-6'-ethylacet-*o*-toluidide
CAS:	2-chloro-*N*-(ethoxymethyl)-*N*-(2-ethyl-6-methylphenyl)acetamide
Manufacturer's code numbers:	MON 097
CAS number:	34256-82-1
CIPAC Code:	496
Molecular formula:	$C_{14}H_{20}ClNO_2$
Molecular mass:	269.77 g/mole
Structural formula:	

Specifications

Specifications for acetochlor have not been developed by the FAO.

Physical and chemical properties (pure acetochlor 99.9%)

Property	Results (method)	Reference
Appearance	Pale yellow, free-flowing liquid	
Melting point	10.6 ±0.1 °C	Pigeon 1999 MLL-31389
Boiling point	172 °C at 665 Pa	Pigeon 1999 MLL-31389

Property	Results (method)	Reference
Relative density	1.1221 g/cm³ at 20 ± 0.5 °C	Pigeon 1999 MLL-31389
pH	Non-ionisable	
Vapour pressure	2.2×10^{-5} hPa at 20 °C 4.6×10^{-5} hPa at 25 °C	Franke 2002 MLL-31685
Solubility in water	282 mg/L at 20 °C	Pigeon 1999 MLL-31389
Solubility in organic solvents (at 20 °C) (g/L)	methanol > 5000 g/L acetone > 5000 g/L n-heptane > 5000 g/L ethyl acetate > 5000 g/L p-xylene > 5000 g/L 1,2-dichloroethane > 5000 g/L	Pigeon 1999 MLL-31389
Partition coefficient n-octanol/water	log K_{ow} = 4.14 at 20 °C	Pigeon 1999 MLL-31389
Hydrolysis under sterile conditions	Stable at pH 5,7 and 9 and at 25 °C; No hydrolysis detected after 31 days	Myers 1989 WRC-88-70
Photolysis	Photolytically stable in sterile water at 25 °C	Chotalia & Weissler 1989 RJ0726B

Formulations

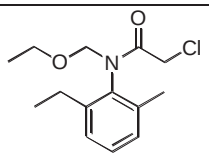
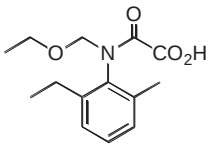
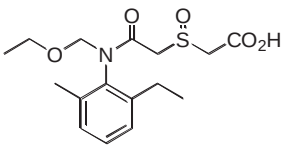
Acetochlor is available in emulsifiable concentrate (EC) and micro-encapsulated suspension (CS) formulations.

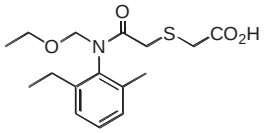
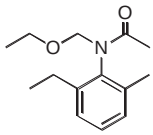
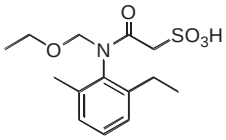
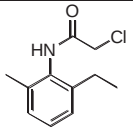
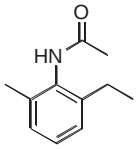
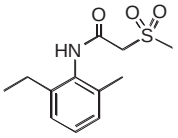
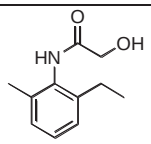
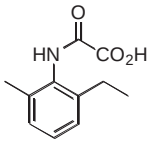
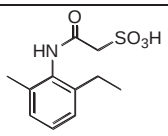
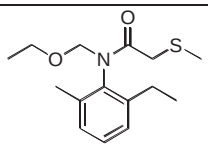
Formulations	Active ingredient content
EC	839 g/L
CS	359 g/L

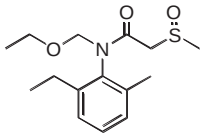
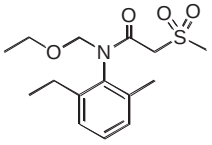
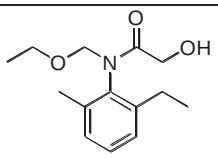
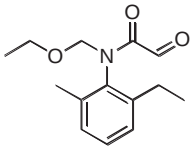
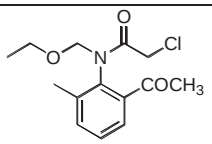
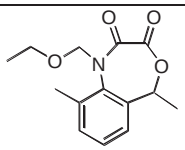
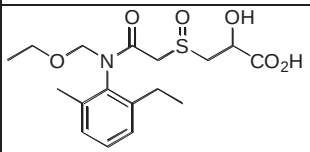
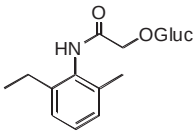
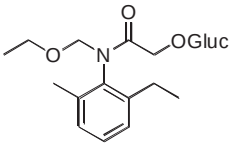
METABOLISM AND ENVIRONMENTAL FATE

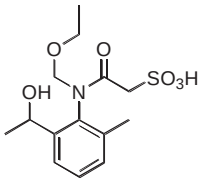
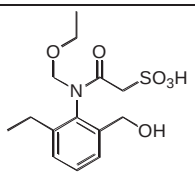
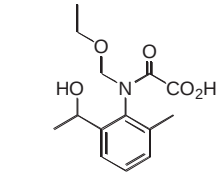
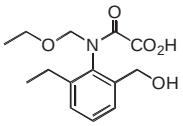
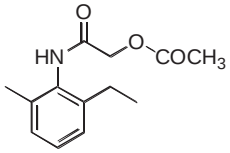
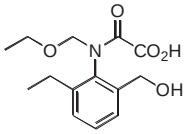
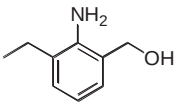
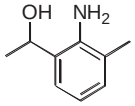
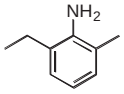
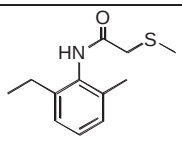
To enable interpretation of the different studies a common numbering scheme for metabolites has been developed based on one reported in the EU. The metabolite summary table provides a reference for the numbering scheme used in the current evaluation.

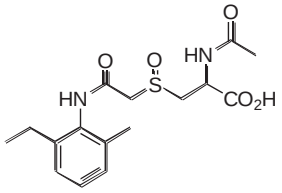
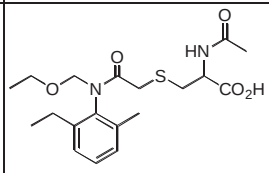
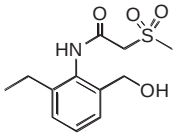
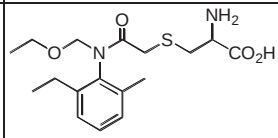
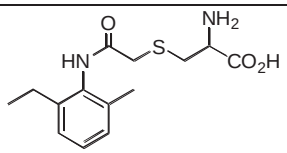
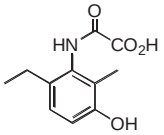
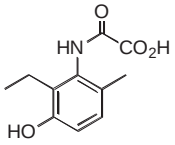
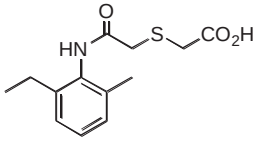
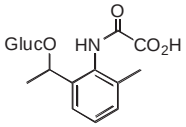
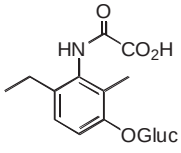
Table 1 Degradation compounds from metabolism of acetochlor in plants, animals, soil, or water

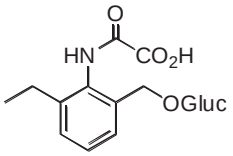
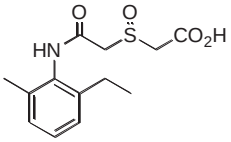
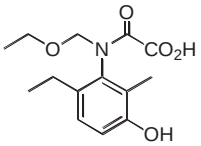
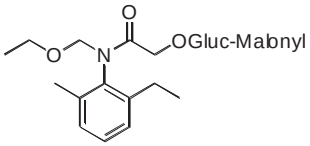
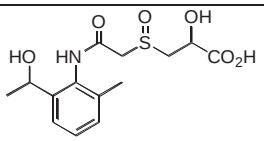
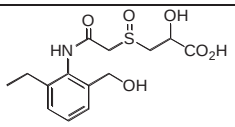
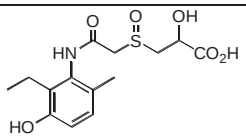
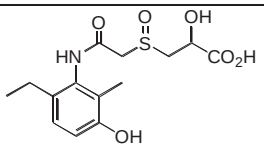
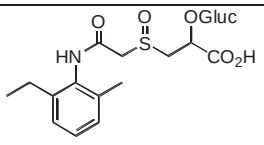
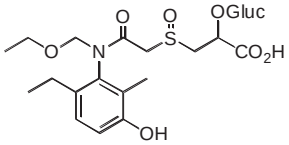
Code no	Company code	Term used in evaluation	Structure	Found in:
1		acetochlor		rat faeces goat faeces
2	R290130 MON52755 ICIA5796/17	*tert*-oxanilic acid		maize forage, stover soya bean forage, hay sediment/water, aerobic soil
3	R243797 MON52709 ICIA5796/48	*tert*-sulfinylacetic acid		maize forage, stover, grain cotton stems and leaves rotational crops sediment/water, aerobic soil

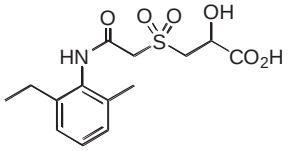
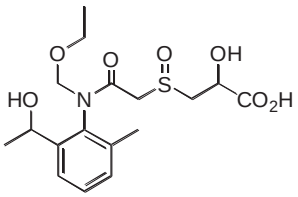
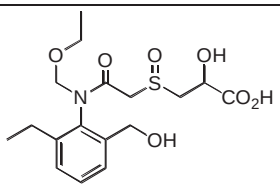
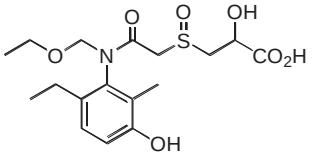
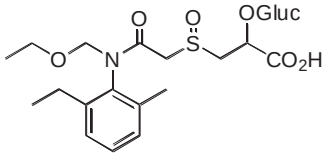
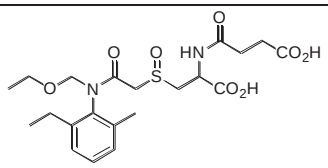
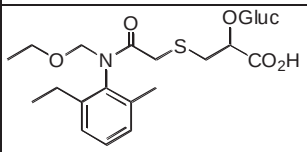
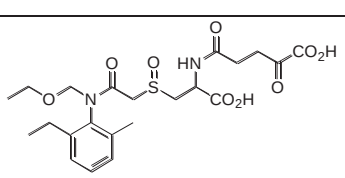
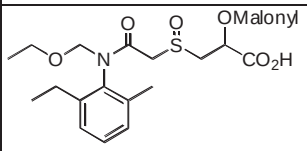
Code no	Company code	Term used in evaluation	Structure	Found in:
4		*tert*-thioacetic acid		monkey urine sediment/water, aerobic soil
6	R243661 MON52706 ICIA5796/31	*tert*-norchloroacetochlor		sediment/water, aerobic soil
7	R290131 soil MON52754 ICIA5796/2	*tert*-sulfonic acid		maize forage, stover soya bean hay rotational crops sediment/water, aerobic soil
8	ICIA5676/05	*sec*-amide chloride *s*-amide chloride *s*-acetochlor		rat urine, rat faeces, rat liver (*in vitro*) mouse urine monkey urine aerobic soil
9		*sec*-norchloroacetochlor		aerobic soil
10	ICIA5676/14	*sec*-methylsulfone		rat urine soya bean forage maize forage and stover rotational crops aerobic soil
11		*sec*-hydroxyacetochlor		maize forage, stover, grain rotational crops aerobic soil
12	CP91301	*sec*-oxanilic acid		hens maize forage and stover rotational crops aerobic soil
13	CP92428	*sec*-sulfonic acid		maize forage and stover soya bean hay cotton stems and leaves rotational crops aerobic soil
14		*tert*-methyl sulfide		aerobic soil

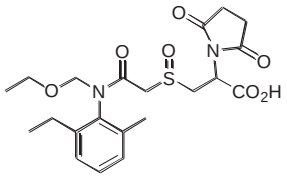
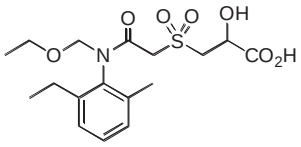
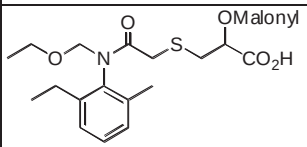
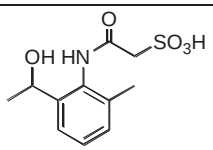
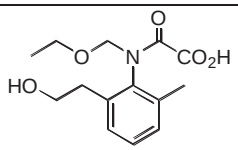
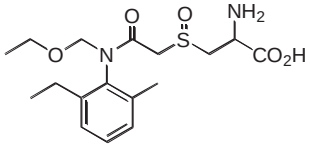
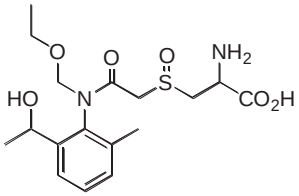
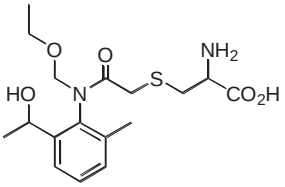
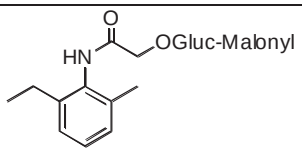
Code no	Company code	Term used in evaluation	Structure	Found in:
15		*tert*-methylsulfoxide		maize forage and stover aerobic soil
16		*tert*-methylsulfone		maize forage and stover soya bean forage rotational crops aerobic soil
17	CP68365-3	*tert*-hydroxyacetochlor		maize forage and stover rotational crops aerobic soil
18		*tert*-glyoxylic acid		aerobic soil
19		ketoethyl acetochlor		aerobic soil
20				aerobic soil
21	ICIA5676/25	*tert*- sulfinyllactic acid		maize forage, stover grain, soya bean hay
22		*sec*-hydroxy glucose conjugate		maize forage and stover soya bean hay rotational crop
23		*tert*-hydroxy glucose conjugate		maize forage and stover rotational crop

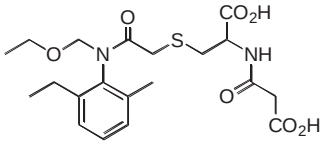
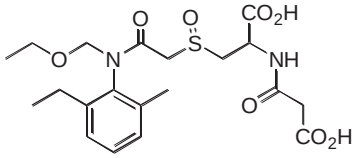
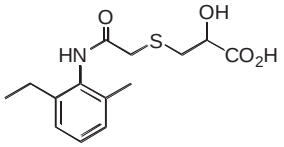
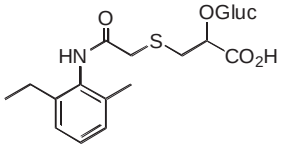
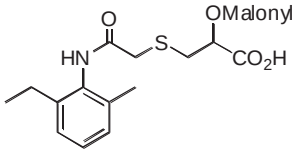
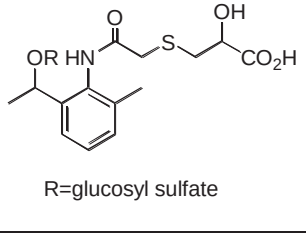
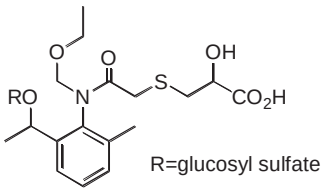
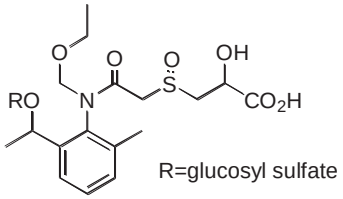
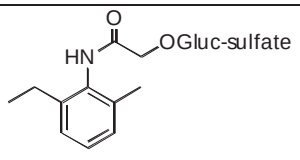
Code no	Company code	Term used in evaluation	Structure	Found in:
24		1-hydroxyethyl *tert*-sulfonic acid		Soya bean hay rotational crop
25		hydroxymethyl *tert*-sulfonic acid		rotational crops
26		1-hydroxyethyl *tert*-oxanilic acid		Soya bean forage and hay rotational crops
27		hydroxymethyl *tert*-oxanilic acid		Soya bean forage and hay rotational crops
28	CP91302	*sec*-hydroxy acetyl ester		rotational crops
31		hydroxymethyl-*tert*-oxanilic acid		Rotational crops
32		HMEA		common chemophore from hydrolysis of metabolites containing hydroxylation of the ring methyl group
33		HEMA		common chemophore from hydrolysis of metabolites containing hydroxylation at the 1-position of the ring ethyl group
34		EMA		common chemophore from hydrolysis of metabolites containing no modification of the ring methyl or ethyl groups
36	CP92422-2B ICIA5676/19	*sec*- methyl sulfide		rat urine rotational crops

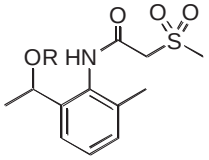
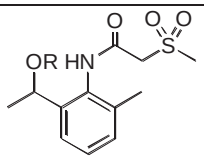
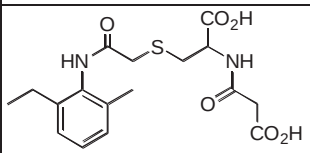
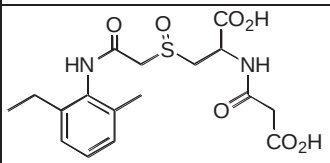
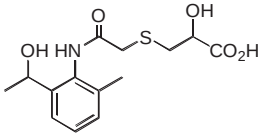
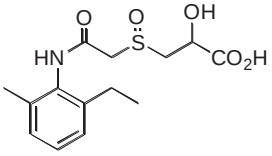
Code no	Company code	Term used in evaluation	Structure	Found in:
39	ICIA5676/47	*sec*-mercapturic acid sulfoxide		rat urine hens
44	ICIA5676/28	*tert*- mercapturic acid		rat urine rat bile goat urine monkey urine
45	ICIA5676/50	hydroxymethyl *sec*-methyl sulfone		rat urine rotational crops
56		*tert*-cysteine		rat bile, rat urine goat urine soya bean forage
67		*sec*-cysteine		rat urine goat urine hens cotton leaves and stems
68		5-hydroxy *sec*-oxanilic acid		maize forage, stover, grain rotational crops
69	ICIA5676/55	3-hydroxy *sec*-oxanilic acid		maize
72		*sec*-thioacetic acid		maize forage and stover
73		1-hydroxyethyl *sec*-oxanilic acid glucose conjugate		Maize
74		5-hydroxy *sec*-oxanilic acid glucose conjugate		Maize

Code no	Company code	Term used in evaluation	Structure	Found in:
75		hydroxymethyl *sec*-oxanilic acid glucose conjugate		Maize
76		*sec*-sulfinylacetic acid		maize forage and stover cotton stems and leaves
77		5-hydroxy *tert*-oxanilic acid		maize forage and hay
78		*tert*-hydroxy glucose malonyl conjugate		maize forage and stover
79		1-hydroxyethyl *sec*-sulfinyllactic acid		maize forage, stover, and grain, soya bean hay cotton stems and leaves
80		hydroxymethyl *sec*-sulfinyllactic acid		maize forage, stover, and grain
81		3-hydroxy *sec*-sulfinyllactic acid		maize forage, stover, and grain
82		5-hydroxy *sec*-sulfinyllactic acid		maize forage, stover, and grain soya bean hay cotton leaves and stems
83		*sec*-sulfinyllactic acid glucose conjugate		maize cotton stems and leaves
84		5-hydroxy *tert*-sulfinyllactic acid glucose conjugate		maize forage and stover

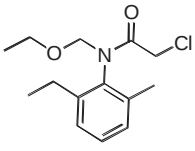
Code no	Company code	Term used in evaluation	Structure	Found in:
85		*sec*-sulfonyllactic acid		maize forage and stover
86		1-hydroxyethyl *tert*-sulfinyllactic acid		Soya bean hay maize forage and stover
87		hydroxymethyl *tert*-sulfinyllactic acid		maize forage and stover
88		5-hydroxy-*tert*-sulfinyllactic acid		maize forage and stover
89		*tert*-sulfinyllactic acid glucose conjugate		Soya bean hay, maize forage stover
90		*tert*-cysteine sulfoxide succinyl conjugate		maize forage and stover
91		*tert*-thiolactic acid glucose conjugate		maize forage and stover
92		*tert*-cysteine sulfoxide α-ketoglutaryl conjugate		maize forage and stover
94		*tert*-sulfinyllactic acid malonyl conjugate		maize forage and stover

Code no	Company code	Term used in evaluation	Structure	Found in:
95		*tert*-cysteine sulfoxide succinimide conjugate		maize forage and stover
96		*tert*-sulfonyllactic acid		maize forage and stover
97		*tert*-thiolactic acid malonyl conjugate		maize forage and stover
98		1-hydroxyethyl *sec*-sulfonic acid		Soya bean forage and hay
99		2-hydroxyethyl *tert*-oxanilic acid		Soya bean forage and hay
100		*tert*-cysteine sulfoxide		Soya bean hay
101		1-hydroxyethyl *tert*-cysteine sulfoxide		Soya bean hay
102		1-hydroxyethyl *tert*-cysteine		Soya bean hay
103		*sec*-hydroxy glucose malonyl conjugate		Soya bean hay

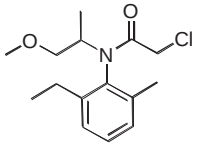
Code no	Company code	Term used in evaluation	Structure	Found in:
104		*tert*-malonylcysteine		Soya bean hay cotton stems and leaves
105		*tert*-malonylcysteine sulfoxide		Soya bean forage and hay cotton stems and leaves
106		*sec*-thiolactic acid		cotton leaves and stems
107		*sec*-thiolactic acid glucose conjugate		cotton stems and leaves
108		*sec*-thiolactic acid malonyl conjugate		cotton stems and leaves
109		1-hydroxyethyl *sec*-thiolactic acid glucosylsulfate conjugate		cotton stems and leaves
110		1-hydroxyethyl *tert*-thiolactic acid glucosylsulfate conjugate		cotton stems and leaves
111		1-hydroxyethyl *tert*-sulfinyllactic acid glucosylsulfate conjugate		cotton stems and leaves
112		*sec*-hydroxyacetochlor glucose sulfate conjugate		cotton stems and leaves

Code no	Company code	Term used in evaluation	Structure	Found in:
113		hydroxyethyl *sec*-methylsulfone glucose conjugate	 R=glucose	cotton stems and leaves
114		hydroxyethyl *sec*-methylsulfone glucose sulfate conjugate	 R=glucose sulfate	cotton stems and leaves
115		*sec*-malonylcysteine		cotton stems and leaves
116		*sec*-malonylcysteine sulfoxide		cotton stems and leaves
117		1-hydroxyethyl *sec*-thiolactic acid		cotton stems and leaves
118		*sec*-sulfinyllactic acid		maize forage, stover, and grain soya bean forage and hay cotton leaves and stems

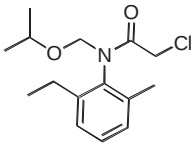
Acetochlor is a member of the chloroacetamide herbicides, a group that also includes metolachlor, propisochlor, alachlor and butachlor. The structures of these herbicides are similar, especially in the case of acetochlor, metolachlor and propisochlor, which all contain ethyl and methyl group substitutions at the 2- and 6-positions, respectively, of the phenyl group. These three herbicides share some common secondary amide metabolites that result from cleavage of their alkyl ether groups from the nitrogen.



Acetochlor



Metolachlor

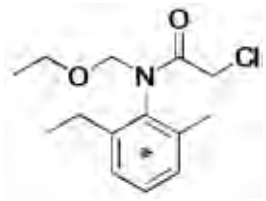


Propisochlor

The Meeting received studies on the metabolism of acetochlor in plants (maize, soya bean, and cotton), laboratory animals (rats, mice and rhesus monkey) as well as lactating goats and laying

hens. The metabolism of acetochlor in plants and animals was investigated using [14C-U-phenyl]-acetochlor. The structural formula and the positions of the 14C label are shown below. The studies on rats, mice and monkeys were evaluated by the WHO Core Assessment Group.

In addition, information is provided on the metabolic fate of acetochlor *tert*-sulfonic acid, acetochlor *tert*-oxanilic acid, acetochlor *tert*-sulfinylacetic acid, and *tert*-hydroxyacetochlor in hens and goats, the fate of acetochlor 1-hydroxyethyl *tert*-oxanilic acid in goats, and the fate of N-(6-ethyl-3-hydroxy-2-methylphenyl) oxamic acid in the lactating cow.



[^{14}C-U-phenyl]-acetochlor
Figure 1 Label positions of acetochlor: marked as * to indicate uniform labelling of the six carbons in the phenyl ring

The identification of residue components in the animal and plant metabolism studies was achieved using, where available, authentic standards of the compounds involved as well as mass spectral techniques. Additional techniques such as hydrolysis, derivatization, and enzymatic degradation were used in many cases to aid in characterizing metabolites. Individual studies utilised different numbering schemes for metabolites, sometimes even within the same study. A harmonised numbering scheme is used in this report.

In the metabolism reports that follow, acetochlor and certain metabolites are sometimes listed as occurring in multiple fractions of a single chromatogram. Hindered rotation about the amide nitrogen results in different rotational isomers (rotamers) and for some cases diastereomers.

Plant metabolism

Acetochlor is typically used for three different situations:

- Incorporation into the soil prior to planting the crop (PP)
- As a broadcast spray to weeds and bare soil after seeding but prior to crop emergence (PE)
- As a broadcast spray to weeds and the growing crop, i.e. post-emergence (PO)

The Meeting received plant metabolism studies with acetochlor following pre- and post-emergent applications to maize (corn), cotton and soya bean.

Herbicide safeners are utilised when using acetochlor for weed control in monocotyledonous cereals such as maize. The metabolism studies reviewed here for maize included the safener furilazole. Ekler *et al.* (1993) [Ekler Z, Dutka F, Stephenson GR (1993), *Safener effects on acetochlor toxicity, uptake, metabolism and glutathione S-transferase activity in maize*. *Weed Research*, 33: 311–318] noted that safeners significantly increased the uptake of [^{14}C]acetochlor, the rate of its metabolism, maize GSH content and GST activity. Seedlings receiving pre-treatment with the herbicide safener BAS-145138 metabolised almost 70% of the absorbed [^{14}C]acetochlor within 10 minutes. In contrast, Jackson *et al.* (1989) (Jackson LA, Yopp JH, Kapusta G (1989*) Absorption and distribution of flurazole and acetochlor in grain sorghum*. *Pesticide Biochemistry and Physiology* 25: 373–380.) observed that safened plants did not exhibit more rapid breakdown of acetochlor compared to non-safened plants. The metabolites formed, judged by comparison of TLC plates, were similar for plants treated with and without flurazole.

Maize

Kurtzweil (2009, MSL0020769) studied the metabolism of [^{14}C]acetochlor in maize grown in outdoor plots. The test substance consisted of [U-^{14}C-phenyl]-acetochlor and also contained a ^{13}C-label at the C-2 position of the 2-chloroacetamide moiety to aid in structure elucidation of metabolites by mass spectroscopy. Roundup Ready® corn NK603 (*Zea mays* L., hybrid DKC69-72) was planted in two plots (1.49 m^2 each) consisting of plastic-lined plywood boxes embedded in the ground to simulate field conditions. One of the plots (PE) was treated with a pre-emergence (PE) application of a [^{14}C]acetochlor spray solution immediately after seeding. After allowing the corn plants to grow to a height of 66–71 cm (growth stage V6–V7), the other plot was treated post-emergence (PO) by uniformly applying the test substance to the foliage via a spray bottle. The effective treatment rate of acetochlor test substance for the PE application 3.65 kg ai/ha and the effective treatment for the PO application was 3.52 kg ai/ha. In addition to an initial V3 thinning from the PE plot 26 days after treatment (DAT), four samplings were conducted for both the control and treated maize plots: a harvest at 95 DAT (PE) and 54 DAT (PO) of kernels plus cob with husk removed corresponding to a typical sweet corn harvest, a forage harvest at 111 DAT (PE) and 70 DAT (PO), and a final harvest of mature maize at 141 DAT (PE) and 100 DAT (PO) that sampled grain and stover.

The ^{14}C found in maize immature plants, forage, and stover (expressed as mg/kg acetochlor equivalents) is summarized in Table 2. The TRR in forage were 0.67 and 3.44 mg equiv/kg for the PE and PO treatments, respectively. In stover, the TRRs follow the same trend where they are 1.84 and 6.41 mg equiv/kg for the PE and PO treatments, respectively. In the sweet corn (KWHR) and grain were much lower (0.009–0.037 mg equiv/kg).

Table 2 TRR in maize commodities after application of [^{14}C]acetochlor

Treatment	Matrix	DAT	Matrix TRR (mg equiv/kg)
Pre-emergence	V3 immature plant (thinnings)	26	1.19
	Sweet corn (KWHR)	95	0.011
	Forage	111	0.67
	Grain	141	0.037
	Stover	141	1.84
Post-emergence	Sweet corn (KWHR)	54	0.009
	Forage	70	3.44
	Grain	100	0.022
	Stover	100	6.41

Homogenised samples were extracted with CH_3CN/H_2O (4×) (20:80 v/v for forage and immature plant 40:60 v/v in the case of grain and stover). A fifth extraction was with CH_3CN. Subsamples of the forage and stover PES were extracted sequentially with 0.1 N HCl and 0.1 N NaOH. Additional sub-samples of PES were subjected sequentially to (in order) a phosphate rinse, hydrolysis with α-amylase to produce a starch fraction, hydrolysis with protease to yield a protein fraction, EDTA extraction to produce the pectin fraction, oxidation with chlorite to yield the lignin fraction, hydrolysis with cellulase to produce the cellulose fraction and hydrolysis with strong base to yield the hemi-cellulose fraction.

CH_3CN/H_2O extracted ≥ 79% of the TRR present in immature plants, forage and stover samples. Extraction of ^{14}C present in grain with the solvent system used was lower at 58–63% TRR. The majority of the ^{14}C present in PES of forage and stover was associated with natural products, especially starch, protein, lignin and hemicellulose.

Table 3 Characterisation of ^{14}C residues in maize commodities following pre- or post-emergence application (%TRR)

		Immature plant	Forage	Grain	Stover
PE	CH_3CN/H_2O extracted [a]	94.1	87.2	58.5	79.0
	Organic layer base partition		10.09	3.36	8.45
	Organic layer acid partition		16.98	15.26	19.42
	Aqueous layer after partition		56.11	38.6	48.07

		Immature plant	Forage	Grain	Stover
	Unextracted (PES)	5.9	12.8	41.5	21.0
	Phosphate		0.62		2.9
	Starch		1.01		2.61
	Protein		1.97		2.39
	Pectin		0.96		1.37
	Lignin		4.17		6.05
	Cellulose		1.31		1.9
	Hemicellulose		2.56		3.5
	Final Pellet		0.23		0.26
PO	CH_3CN/H_2O extracted [a]	-	85.6	62.6	86
	Organic layer base partition		4.42	3.89	5.14
	Organic layer acid partition		30.27	14.57	23.29
	Aqueous layer after partition		45.34	40.14	54.2
	Unextracted (PES)	–	14.4	37.4	14
	Phosphate		1.04		1.46
	Starch		1.75		1.99
	Protein		2.93		2.29
	Pectin		1.58		1.30
	Lignin		4.60		4.21
	Cellulose		0.95		1.22
	Hemicellulose		1.39		1.39
	Final Pellet		0.16		0.14

[a] CH_3CN/H_2O extracts were adjusted to pH 8–9 and partitioned with ethyl acetate. The organic layer from the base partition contains neutral metabolites. The pH of the aqueous layer was adjusted to pH 2 and partitioned with ethyl acetate. The organic layer from the acid partition contains weak and moderate acids. Strong acids, polar and hydrophilic compounds are retained in the aqueous layer.

Analysis of extracts by reverse-phase HPLC showed that the metabolism of acetochlor in maize was extensive giving rise to a large number of metabolites with no unchanged acetochlor observed in any of the matrices. Identification was not possible for 16–25 fractions for PE and 15 fractions for PO matrices. Nearly all metabolites present at 0.05 mg/kg or higher were isolated and purified by preparative HPLC and either identified by mass spectrometry or thoroughly characterized. A fraction containing 9.4% TRR PE forage, 4.8% PE stover, 5.0% PO forage and 7.4% PO stover appeared to comprise large molecular weight material possibly phenolic conjugates of *tert*-sulfinyllactic acid as well as with some other acetochlor related metabolites.

Metabolite isolation was only conducted on forage and stover extracts as TRRs in grain were too low to permit identification or characterization of isolated metabolites. For grain, metabolite identification was based on retention time comparison of metabolites. In grain, no individual compound exceeded 10% of TRR and no discrete component characterized by chromatography exceeded 0.001 mg equiv/kg.

The acetochlor metabolites identified in PO forage and stover primarily resulted from initial glutathione conjugation of acetochlor followed by oxidation to give sulfoxide-type metabolites. One compound exceeded 10% of TRR: *tert*-sulfinyllactic acid (21) was observed at 12.6% TRR (0.434 mg equiv/kg) in forage and 11.3% of TRR (0.722 mg equiv/kg) in stover. Two other metabolites exceeded 0.1 mg equiv/kg: *sec*-sulfinyllactic acid (72) and *sec*-sulfinyllactic acid glucose conjugate (83).

In contrast, the metabolism of acetochlor in PE maize resulted in large part from the uptake of soil metabolites to give oxanilate-type metabolites. None of the individual components exceeded 10% of TRR in immature plant, forage or stover. The major metabolite was 5-hydroxy *sec*-oxanilic acid (68) present at levels of 8.4% (0.099 mg equiv/kg), 6.2% (0.042 mg equiv/kg) and 4.3% (0.080 mg equiv/kg) TRR in immature plants, forage and stover respectively.

Table 4 Identification of metabolites of acetochlor in different fractions from maize forage and stover after post-emergence (PO) application

		Matrix			
Code		PO Forage		PO Stover	
	Identification	%TRR	mg equiv/kg	%TRR	mg equiv/kg
3	*tert*-sulfinylacetic acid	0.31	0.011	0.33	0.021
7	*tert*-sulfonic acid	1.97	0.068	2.52	0.161
11	*sec*-hydroxyacetochlor	0.41	0.014	0.38	0.024
12	*sec*-oxanilic acid	0.52	0.018	0.63	0.04
13	*sec*-sulfonic acid	1.47	0.051	1.86	0.119
17	*tert*-hydroxyacetochlor	0.24	0.008	0.39	0.025
21	*tert*-sulfinyllactic acid	12.59	0.434	11.27	0.722
23	*tert*-hydroxyacetochlor glucose conjugate	0.52	0.018	0.65	0.042
118	*sec*-sulfinyllactic acid	6.37	0.219	5.94	0.381
76	*sec*-sulfinylacetic acid	0.54	0.019	0.54	0.035
85	*sec*-sulfonyllactic acid	1.23	0.042	1.32	0.085
89	*tert*-sulfinyllactic acid glucose conjugate	1.58	0.055	1.83	0.117
89	*tert*-sulfinyllactic acid glucose conjugate [a]	2.21	0.076	2.08	0.133
90	*tert*-cysteine sulfoxide succinyl conjugate	0.47	0.016	0.58	0.037
91	*tert*-thiolactic acid glucose conjugate	0.14	0.005	0.14	0.009
92	*tert*-cysteine sulfoxide α-ketoglutaryl conjugate	0.06	0.002	0.07	0.004
93	*tert*-hydroxyacetochlor glucose malonyl conjugate	0.48	0.017	0.38	0.025
94	*tert*-sulfinyllactic acid malonyl conjugate	0.71	0.025	0.57	0.036
95	*tert*-cysteine sulfoxide succinimide conjugate	0.39	0.013	0.27	0.017
96	*tert*-sulfonyllactic acid	0.24	0.008	0.31	0.02
97	*tert*-thiolactic acid malonyl conjugate	0.3	0.01	0.06	0.004
79	1-hydroxyethyl *sec*-sulfinyllactic acid	1.46	0.05	1.85	0.118
86, 87	hydroxy *tert*-sulfinyllactic acid	1.43	0.049	1.82	0.116
80	hydroxymethyl *sec*-sulfinyllactic acid	1.65	0.057	1.29	0.083
83	*sec*-sulfinyllactic acid glucose conjugate	3.28	0.113	3.44	0.221
68	5-hydroxy *sec*-oxanilic acid	1.31	0.045	1.48	0.095
81	3-hydroxy *sec*-sulfinyllactic acid	1.43	0.049	1.87	0.12
82	5-hydroxy *sec*-sulfinyllactic acid	2.02	0.07	2.03	0.13
84	5-hydroxy *tert*-sulfinyllactic acid glucose conjugate	0.38	0.013	0.43	0.027
84	5-hydroxy *tert*-sulfinyllactic acid glucose conjugate [b]	0.86	0.03	0.75	0.048
88	5-hydroxy *tert*-sulfinyllactic acid	2.09	0.072	2.07	0.133
Totals		48.65	1.676	49.51	3.174

[a] Isomer of 89

[b] Isomer of 84

Table 5 Identified metabolites in different fractions from maize grain following post-emergence (PO) application

Code	Identification	% TRR	mg equiv/kg
79	1-hydroxyethyl *sec*-sulfinyllactic acid	2.62	≤ 0.001
80	hydroxymethyl *sec*-sulfinyllactic acid	0.65	≤ 0.001
81	3-hydroxy *sec*-sulfinyllactic acid	2.75	≤ 0.001
82	5-hydroxy *sec*-sulfinyllactic acid	0.58	≤ 0.001
68	5-hydroxy *sec*-oxanilic acid	0.81	≤ 0.001
83	*sec*-sulfinyllactic acid glucose conjugate	1.68	≤ 0.001
118, 11	*sec*-sulfinyllactic acid + *sec*-hydroxyacetochlor	0.59	≤ 0.001
89	*tert*-sulfinyllactic acid glucose conjugate	0.43	≤ 0.001
89	*tert*-sulfinyllactic acid glucose conjugate	1.3	≤ 0.001
21	*tert*-sulfinyllactic acid	0.88	≤ 0.001
3	*tert*-sulfinylacetic acid	0.77	≤ 0.001
Totals		13.06	0.004

Table 6 Identification of metabolites of acetochlor in different fractions from maize forage and stover after pre-emergence (PE) application

		Matrix			
Code		PE Forage		PE Stover	
	Identification	%TRR	mg equiv/kg	%TRR	mg equiv/kg
11	*sec*-hydroxyacetochlor	0.94	0.006	1.61	0.03
15	*tert*-methylsulfoxide	0.17	0.001	0.24	0.005
16	*tert*-methylsulfone	0.66	0.004	0.47	0.009
17	*tert*-hydroxyacetochlor	0.3	0.002	0.24	0.004
2, 10	*tert*-oxanilic acid + *sec*-methylsulfone	2.04	0.014	1.96	0.036
21	*tert*-sulfinyllactic acid	1.24	0.008	1.26	0.023
22	*sec*-hydroxyacetochlor glucose conjugate	2.99	0.02	2.09	0.038
23	*tert*-hydroxyacetochlor glucose conjugate	1.03	0.007	1.65	0.03
3	*tert*-sulfinylacetic acid	1.25	0.008	0.99	0.018
7	*tert*-sulfonic acid	3.7	0.025	3.48	0.064
118	*sec*-sulfinyllactic acid	1.2	0.008	1.02	0.019
76	*sec*-sulfinylacetic acid	0.91	0.006	0.71	0.013
78	*tert*-hydroxyacetochlor glucose malonyl conjugate	1.28	0.009	0.47	0.009
73	1-hydroxyethyl *sec*-oxanilic acid glucose conjugate	1.35	0.009	1.19	0.022
75	hydroxymethyl *sec*-oxanilic acid glucose conjugate	2.45	0.016	1.91	0.035
68	5-hydroxy *sec*-oxanilic acid	6.22	0.042	4.32	0.08
74	5-hydroxy *sec*-oxanilic acid glucose conjugate	2.23	0.015	2.26	0.042
77	5-hydroxy *tert*-oxanilic acid	1.94	0.013	1.72	0.032
77	5-hydroxy *tert*-oxanilic acid +	1.68	0.011	1.98	0.037
	499 MW metabolite				
Total		33.59	0.224	29.58	0.545

Table 7 Identification of metabolites of acetochlor in different fractions of maize grain after pre-emergence (PE) application

Code	Identification	% TRR	mg equiv/kg
21	*tert*-sulfinyllactic acid	0.54	≤ 0.001
73	1-hydroxyethyl *sec*-oxanilic acid glucose conjugate	2.82	≤ 0.001
75	hydroxymethyl *sec*-oxanilic acid glucose conjugate	2.05	≤ 0.001
68	5-hydroxy *sec*-oxanilic acid	0.92	≤ 0.001
74	5-hydroxy *sec*-oxanilic acid glucose conjugate	1.08	≤ 0.001
Totals		7.41	≤ 0.001

Table 8 Identification of metabolites of acetochlor in different fractions from immature plant (V3 thinnings) after pre-emergence (PE) application

Code	Identification	% TRR	mg equiv/kg
16	*tert*-methylsulfone	0.92	0.011
17	*tert*-hydroxyacetochlor	3.53	0.042
2	*tert*-oxanilic acid	1.69	0.02
2, 10	*tert*-oxanilic acid + *sec*-methylsulfone	5.09	0.06
21	*tert*-sulfinyllactic acid	5.9	0.07
22	*sec*-hydroxyacetochlor glucose conjugate	1.7	0.02
23	*tert*-hydroxyacetochlor glucose conjugate	4.02	0.048
3	*tert*-sulfinylacetic acid	4.96	0.059
118 11	*sec*-sulfinyllactic acid +*sec*-hydroxyacetochlor	3.18	0.038
76	Unknown + *sec*-sulfinylacetic acid	3.34	0.04
78	*tert*-hydroxyacetochlor glucose malonyl conjugate	2.87	0.034
73	1-hydroxyethyl *sec*-oxanilic acid glucose conjugate	0.59	0.007
74 75	5-hydroxy *sec*-oxanilic acid glucose conjugate + hydroxymethyl *sec*-oxanilic acid glucose conjugate	2.62	0.031
68	5-hydroxy *sec*-oxanilic acid	8.37	0.099
77	5-hydroxy *tert*-oxanilic acid	2.38	0.028
77	5-hydroxy *tert*-oxanilic acid	3.04	0.036
Totals		54.18	0.643

Compounds containing an intact phenyl ring can be classified according to the aniline that would be generated in base hydrolysis. Nonhydroxylated metabolites give EMA, those hydroxylated at the 1-position of the ethyl side-chain give HEMA, those hydroxylated at the methyl side-chain HMEA, those hydroxylated at the 3, 4 or 5 positions of the phenyl ring could be classed as “OH” anilines and the remaining as “other”. The individual metabolites identified are plotted according to their aniline metabolite class for forage and stover. The major aniline metabolite class observed is EMA followed by OH (Figures 2 and 3).

The use of a common moiety may potentially be useful for residue analytical methods.

Acid pressure hydrolysis (6 M HCl, 150 °C, capped vials, > 2 hr) was used to characterize both whole CH_3CN/H_2O extracts of each matrix and isolated metabolites. This hydrolysis technique converts the relevant metabolites to their corresponding anilines. For PO forage and stover, approximately 45 to 64% of the ^{14}C residues in the extracts of forage and stover were converted to EMA with smaller amounts to an aniline corresponding to HEMA class metabolites (3.6–8.7%). PE forage and stover contained 26–28% EMA aniline class metabolites, 8.4–8.9 HEMA class, 2.5–3.1 HMEA class and 11–12% 5-OH class.

Pathways for the metabolism of acetochlor in maize from PE and PO treatments are shown in Figures 4 and 5, respectively.

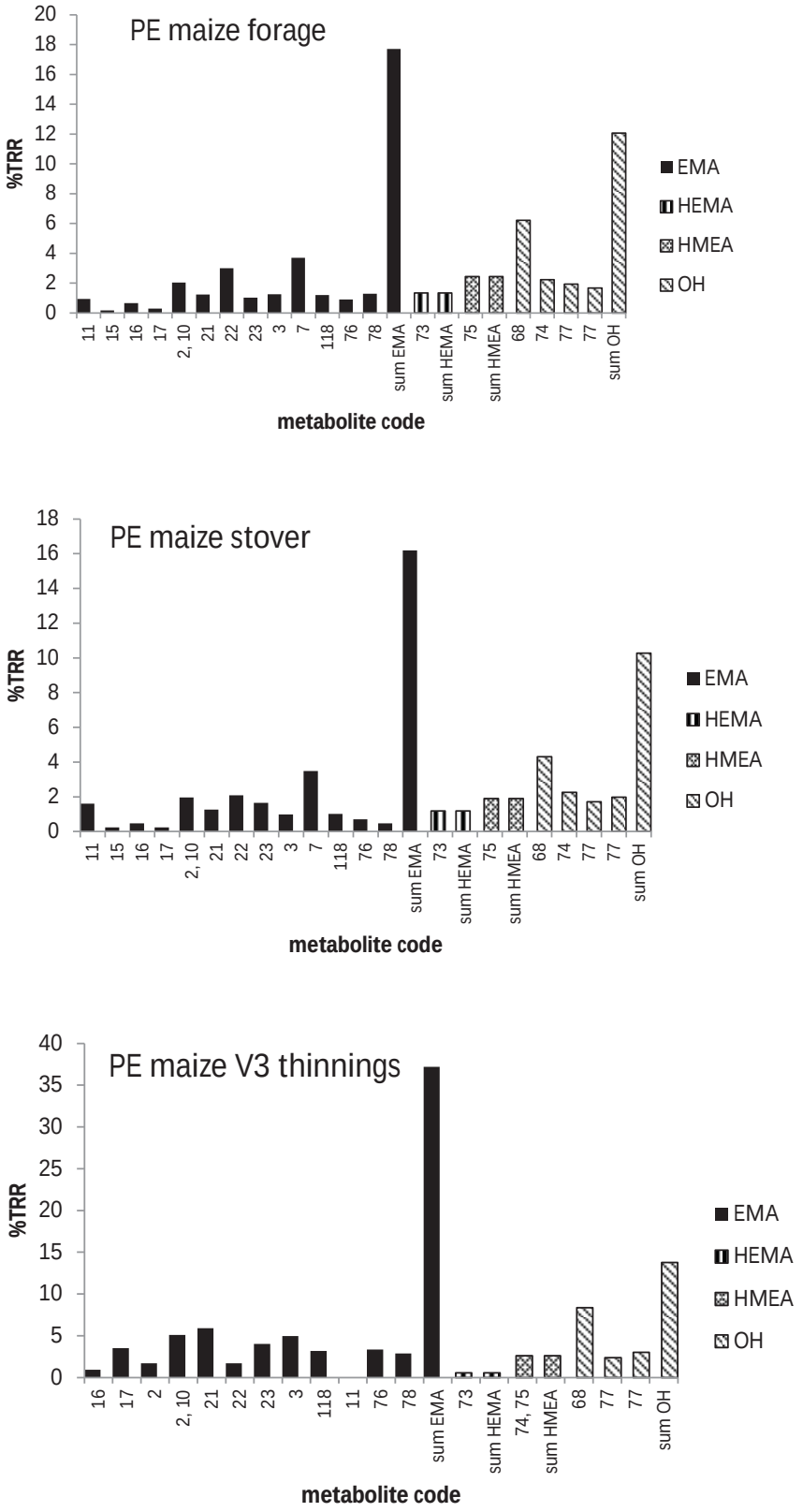


Figure 2 Aniline metabolite classes for pre-emergence application of acetochlor to maize

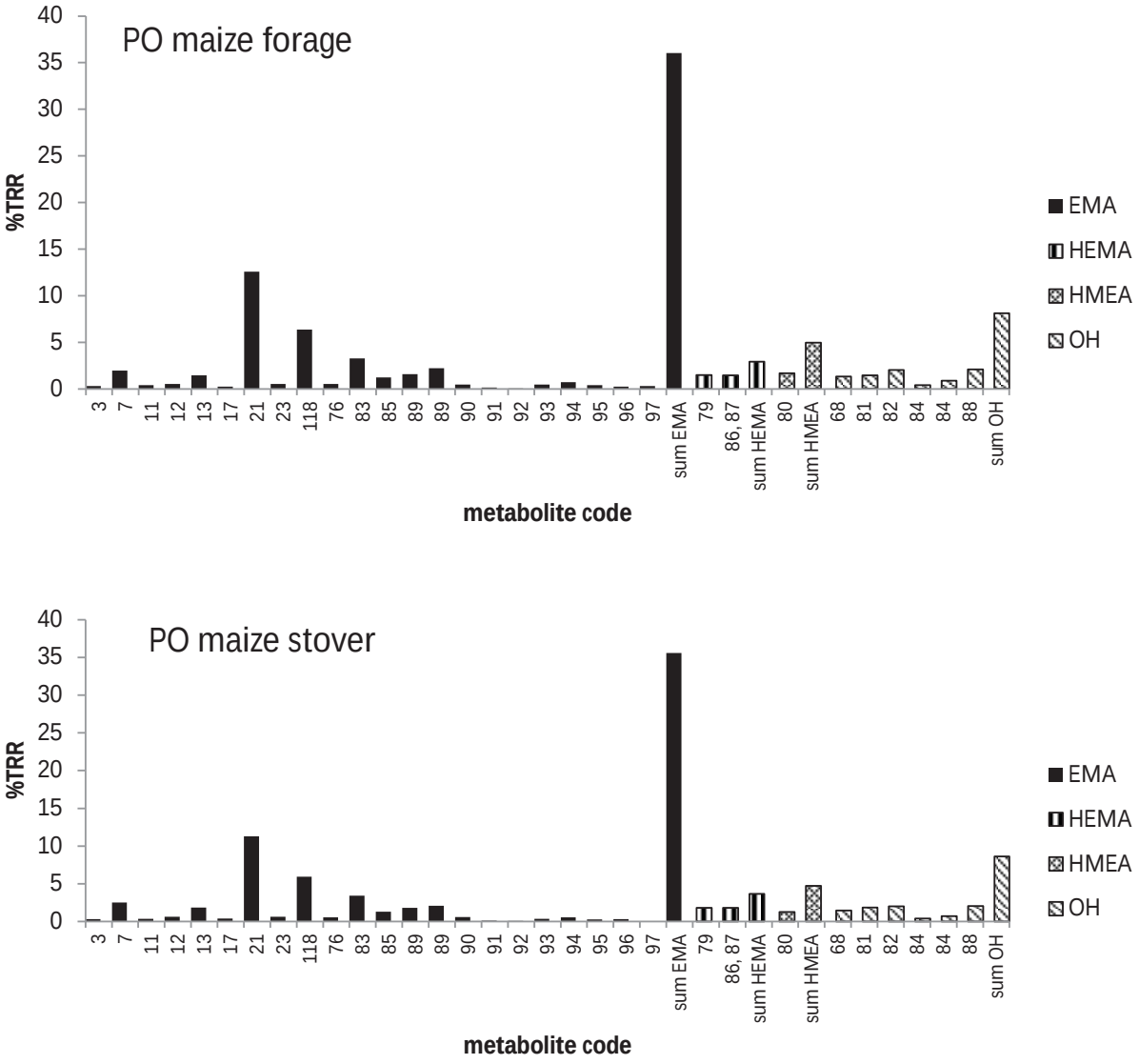


Figure 3 Aniline metabolite classes for post-emergence application of acetochlor to maize

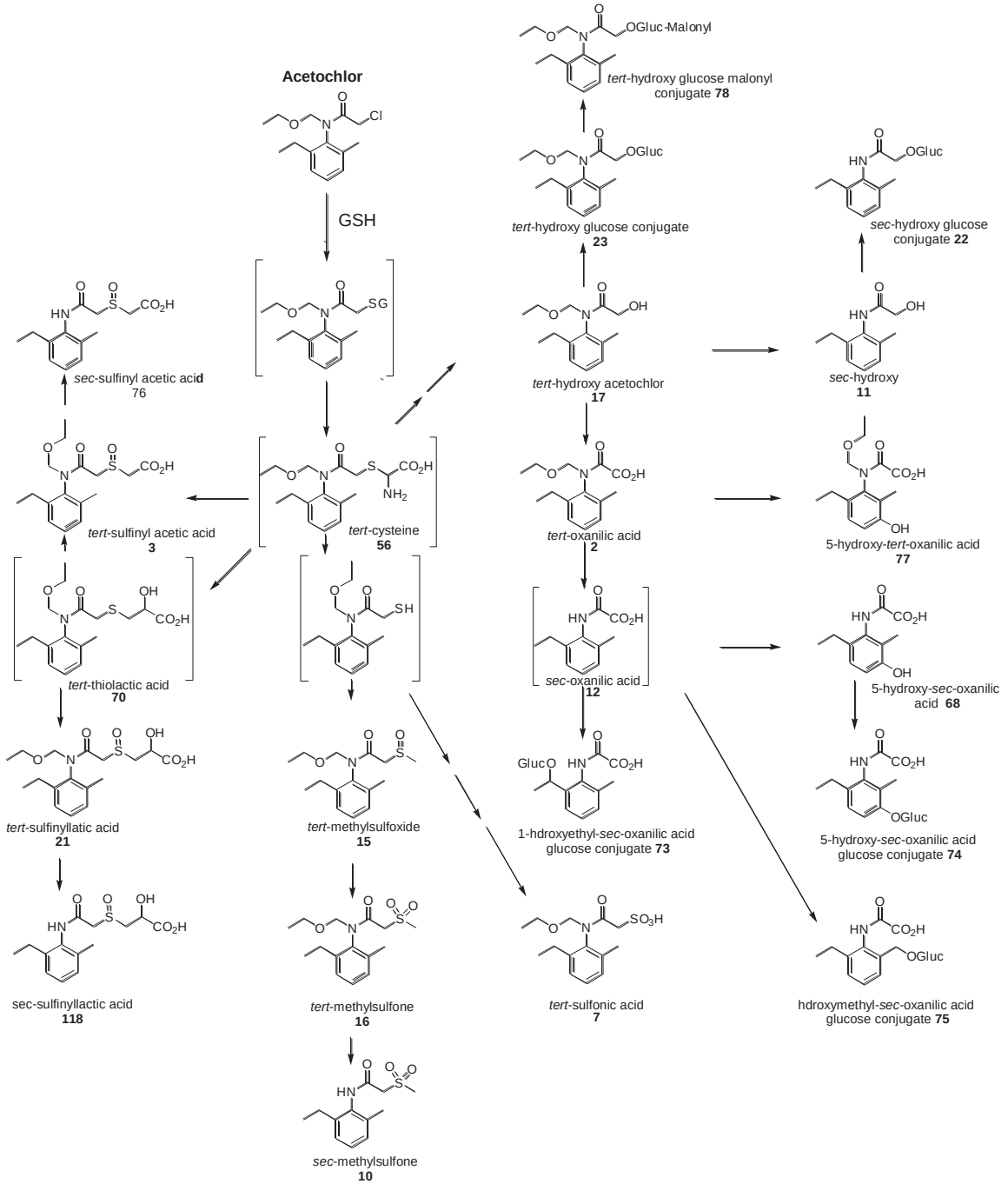


Figure 4 Proposed pathway for metabolism of acetochlor in maize after pre-emergence application

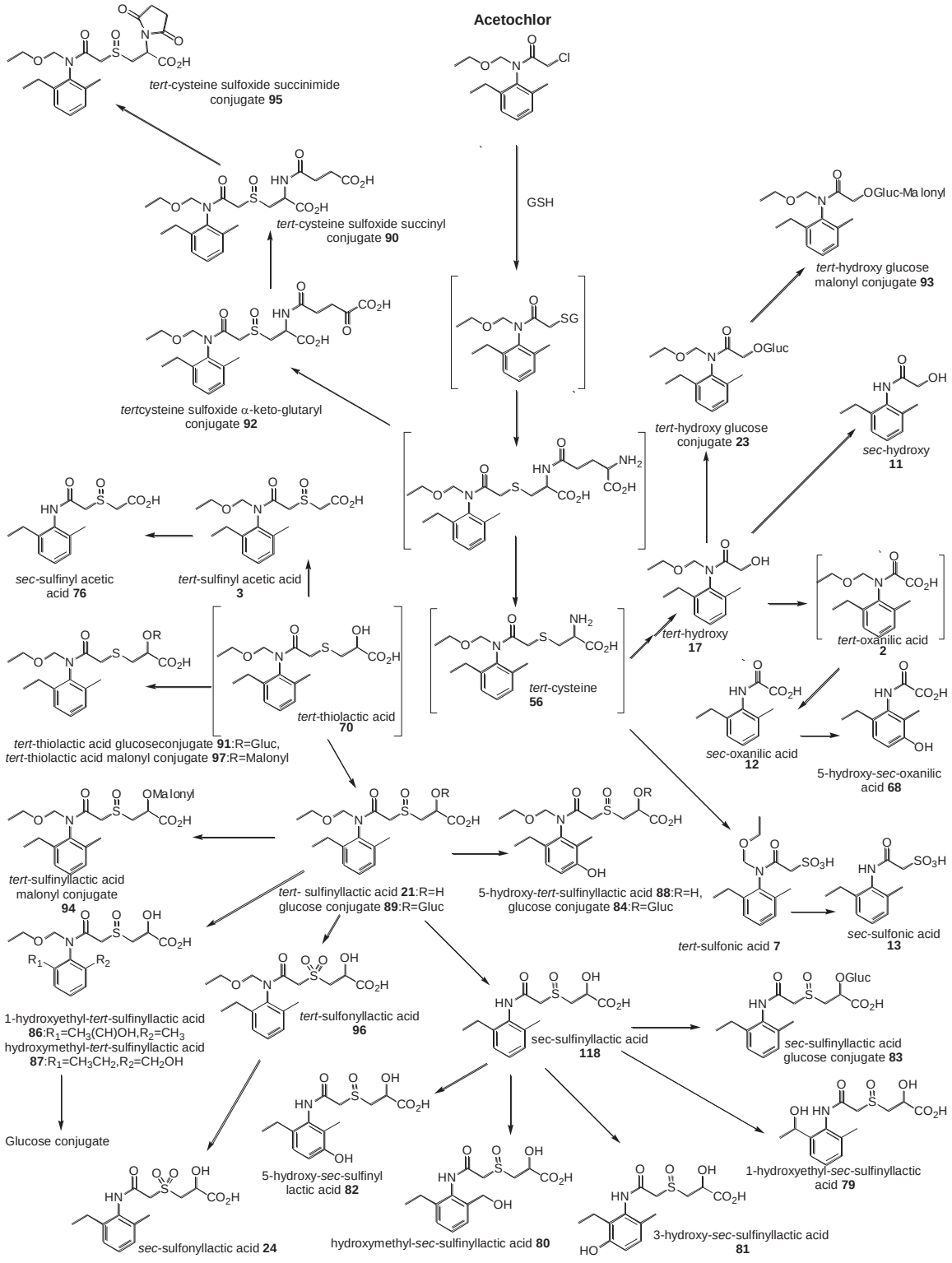


Figure 5 Proposed pathway for metabolism of acetochlor in maize after post-emergence application

Woodbury *et al.* (2009 MSL0021112) studied the metabolism of [^{14}C]acetochlor on outdoor grown soya beans following pre-plant (PP) or post-emergence (PO) application. The test substance consisted of [U-^{14}C phenyl] labelled acetochlor and also contained a ^{13}C-label at the C-2 position of the 2-chloroacetamide moiety to aid in structure elucidation of metabolites by mass

spectroscopy. The PP application was made to the soil (loamy sand) 45 days before seed planting. A separate PO application was made to a second group of plants 42 days after planting seed, when the plants were approximately at the R1–R2 growth stage. The application rates were 3.54 kg ai/ha for the PP and 3.66 kg ai/ha for the PO application.

Forage samples were harvested 91 days and 7 days after the application for PP and PO, respectively. Hay samples were harvested 122 days and 34 days after the application for PP and PO, respectively, while seed samples were harvested 191 and 101 days after the application for PP and PO, respectively. Soya bean seed was removed from pods on the day of harvest.

Harvested forage, hay, and seed samples were homogenised. Combustion analysis gave TRRs of 1.67 and 11.45 mg equiv/kg in PP and PO forage, respectively; 3.48 and 57.7 mg equiv/kg in PP and PO hay, respectively; and 0.175 and 0.192 mg equiv/kg in PP and PO seed, respectively.

Soya bean forage was extracted sequentially with CH_3CN/H_2O (3×), water (1×), 0.1 N HCl, and 0.1 N NaOH (1×). CH_3CN/H_2O extracts contained 1.64 mg equiv/kg (98.4% TRR) and 11.8 mg equiv/kg (103.2% TRR) in PP and PO treated forage, respectively. Water, 0.1 N HCl, and 0.1 N NaOH extracted 0.007 mg equiv/kg (0.4% TRR), 0.002 mg equiv/kg (0.1% TRR), and 0.015 mg equiv/kg (0.9% TRR), respectively, from PP forage. Corresponding extracts from PO forage contained 0.034 mg equiv/kg (0.3% TRR), 0.046 mg equiv/kg (0.4% TRR), and 0.12 mg equiv/kg (1.0% TRR), respectively.

Soya bean hay was also extracted sequentially with CH_3CN/H_2O (3×), water (1×), 0.1 N HCl, and 0.1 N NaOH (1×). CH_3CN/H_2O extracts contained 3.59 mg equiv/kg (103.2% TRR) and 49.51 mg equiv/kg (85.8% TRR) in PP and PO treated forage, respectively. Water, 0.1 N HCl, and 0.1 N NaOH extracted 0.028 mg equiv/kg (0.8% TRR), 0.021 mg equiv/kg (0.6% TRR), and 0.035 mg equiv/kg (1.0% TRR), respectively, from PP hay. Corresponding extracts from PO hay contained 0.58 mg equiv/kg (1.0% TRR), 0.35 mg equiv/kg (0.6% TRR), and 0.75 mg equiv/kg (1.3% TRR), respectively.

Soya bean seed was first extracted with hexane, which resulted in extraction of 0.012 mg equiv/kg (7.0% TRR) from PP seed and 0.017 mg equiv/kg (8.6% TRR) from PO seed. CH_3CN/H_2O extracts of de-fatted seed, from which lipids had been removed, contained 0.104 mg equiv/kg (59.2% TRR) and 0.154 mg equiv/kg (80.2% TRR) from PP and PO treatments, respectively. A further series of extractions with water, 0.1 N HCl (1×), and 0.1 N NaOH (1×) each extracted only a small fraction of the TRR.

Hexane extracts from soya bean seed were characterised by solvent partitioning and fractionation. Acetonitrile phases (polar-lipids or metabolites) contained 0.001 mg equiv/kg (0.6% TRR) and 0.002 mg equiv/kg (1.0% TRR) in PP and PO, respectively. The corresponding hexane phases (lipids) contained 0.011 mg equiv/kg (6.3% TRR) and 0.015 mg equiv/kg (7.8% TRR). The lipid phase was saponified and the non-saponifiable, saponifiable (fatty acids), and acidic aqueous (e.g., glycerol) fractions were quantified. In the PP samples, these fractions corresponded to < LOD, 0.002 mg equiv/kg (1.1% TRR), and 0.009 mg equiv/kg (5.1% TRR), respectively. In the PO sample, the distribution was 0.001 mg equiv/kg (0.4% TRR), 0.011 mg equiv/kg (5.7% TRR), and 0.004 mg equiv/kg (2.1% TRR), respectively. Thus, there was evidence of reincorporation of the radiolabel into natural products in the seed. Combined acetonitrile/water extracts of the seed from each treatment were concentrated and analysed. Both PP and PO seed extracts contained numerous low-level metabolites (more than 27), none of which exceeded 0.03 mg equiv/kg. PP seed metabolites were generally more polar than PO seed metabolites.

Table 9 Distribution and characterisation of ^{14}C in soya bean following pre-planting (PP) application of [^{14}C]acetochlor

	Seed		Forage		Hay	
	(mg equiv/kg)	%TRR	(mg equiv/kg)	%TRR	(mg equiv/kg)	%TRR
Extracted	0.144	82.1	1.666	99.2	3.67	105.6
CH_3CN/H_2O	0.104	59.2	1.642	98.4	3.586	103.2
H_2O	0.002	1.2	0.007	0.4	0.028	0.8
0.1N HCl	0.001	0.4	0.002	0.1	0.021	0.6
0.1N NaOH	0.025	14.3	0.015	0.9	0.035	1
Hexane extracts	0.012	7	–	–	–	–
PES	0.013	7.4	0.066	3.9	0.216	6.2
Total	0.157	89.5	1.732	103.7	3.886	111.8

Table 10 Distribution and characterisation of ^{14}C in soya bean following post-emergence (PO) application of [^{14}C]acetochlor

	Seed		Forage		Hay	
	(mg equiv/kg)	%TRR	(mg equiv/kg)	%TRR	(mg equiv/kg)	%TRR
Extracted	0.191	98.9	12.016	104.9	51.183	88.7
CH_3CN/H_2O	0.154	80.2	11.821	103.2	49.51	85.8
H_2O	0.002	0.8	0.034	0.3	0.577	1
0.1N HCl	0.001	0.7	0.046	0.4	0.346	0.6
0.1N NaOH	0.017	8.6	0.115	1	0.75	1.3
Hexane extracts	0.017	8.6	–	–	–	–
PES	0.01	5.2	0.358	3.1	2.467	4.3
Total	0.201	104.1	12.374	108	53.65	93

Combined CH_3CN/H_2O extracts from each treatment and matrix (forage or hay) were concentrated, and the residues were analysed by reverse-phase HPLC.

As was the case with maize, a large number of metabolites were detected in the solvent extracts but not unchanged acetochlor. There were notable differences in the pattern of metabolites observed following PP compared to PO application.

In PP soya bean the compounds detected resulted in large part from the uptake of soil metabolites to give oxanilate-type metabolites. None of the individual components exceeded 10% of TRR in immature plant, forage or hay. The major metabolites were *tert*-oxanilic acid (> 9.5% TRR, > 0.158 mg equiv/kg) in forage (Table 11) and *tert*-oxanilic acid combined with *tert*-sulfonic acid present at levels of > 9.7% (0.34 mg equiv/kg) in hay (Table 12).

In contrast, the metabolites identified in PO forage and hay primarily resulted from initial glutathione conjugation of acetochlor followed by oxidation to give sulfoxide-type metabolites. Five compounds exceeded 10% of TRR (Tables 13 and 14): *tert*-cysteine (39% TRR), *tert*-malonylcysteine (18–23%TRR), *tert*-sulfinyllactic acid and *tert*-malonylcysteine sulfoxide (combined 24–30%TRR). A large number of other metabolites were present at levels in excess of 0.1 mg equiv/kg.

Table 11 Summary of identified or characterised metabolites in different fractions from PP soya bean forage

Code	Identification	% TRR	mg equiv/kg
10	*sec*-methylsulfone + unknown metabolite	12.8	0.213
16	*tert*-methylsulfone + several components, the largest of which was 0.056 mg equiv/kg (3.4% of TRR)	4.4	0.073
2	*tert*-oxanilic acid	9.5	0.158
2	*tert*-oxanilic acid + several components, the largest of which was 0.024 mg equiv/kg (1.42% of TRR)	3.7	0.062
26	1-hydroxyethyl *tert*-oxanilic acid +	2	0.033

Code	Identification	% TRR	mg equiv/kg
27	hydroxymethyl *tert*-oxanilic acid +		
99	2-hydroxyethyl *tert*-oxanilic acid	5.7	0.095
98	1-hydroxyethyl *sec*-sulfonic acid	3.2	0.053
	+ several unknown metabolites	8.9	0.148
27	hydroxymethyl *tert*-oxanilic acid	6.6	0.11
26	1-hydroxyethyl *tert*-oxanilic acid		
27	hydroxymethyl *tert*-oxanilic acid +	4.9	0.082
26	1-hydroxyethyl *tert*-oxanilic acid	3.3	0.055
–	several components, the largest of which was 0.052 mg equiv/kg (3.14% of TRR)	9.9	0.165
–	several components, with a maximum of 0.031mg equiv/kg (1.83% of TRR)	2.5	0.042
	Total %identified and/or characterised	77.2	1.289

Table 12 Summary of identified or characterised metabolites in different fractions from PP soya bean hay

Code	Identification	% TRR	mg equiv/kg
7, 2	*tert*-sulfonic acid + *tert*-oxanilic acid	9.7	0.336
13	*sec*-sulfonic acid +	3.8	0.133
24	1-hydroxyethyl *tert*-sulfonic acid		
	possible sulfinyllactic acid conjugate of		
2	MW 513 + *tert*-oxanilic acid + multiple components with largest at 0.10 mg equiv/kg (2.86% of TRR)	5.4	0.189
24	1-hydroxyethyl *tert*-sulfonic acid + several radiolabelled components, the largest of which was 0.025 mg equiv/kg (0.71% of TRR)	5.9	0.205
101	1-hydroxyethyl-*tert*-cysteine sulfoxide	6.4	0.223
98	1-hydroxy *sec*-sulfonic acid + multiple	8.9	0.309
	radiolabelled metabolites, the largest of which was 0.084mg equiv/kg (2.43% of TRR)		
26	1-hydroxyethyl *tert*-oxanilic acid +	6.6	0.229
27	hydroxymethyl *tert*-oxanilic acid+		
99	2-hydroxyethyl *tert*-oxanilic acid +	5.8	0.202
	unknown component of MW 286		
–	multiple radiolabelled components, the largest of which was 0.147 mg equiv/kg (4.25% of TRR)	5.1	0.177
–	multiple components, the largest of which was 0.034 mg equiv/kg (0.96% of TRR)	3.1	0.109
–	several radiolabelled components, the	1.2	0.041
	largest being 0.077 mg equiv/kg (2.20% of TRR)	5.7	0.199
	Total %identified and/or characterised	67.7	2.352

Table 13 Summary of identified and characterised metabolites in different fractions from PO soya bean forage

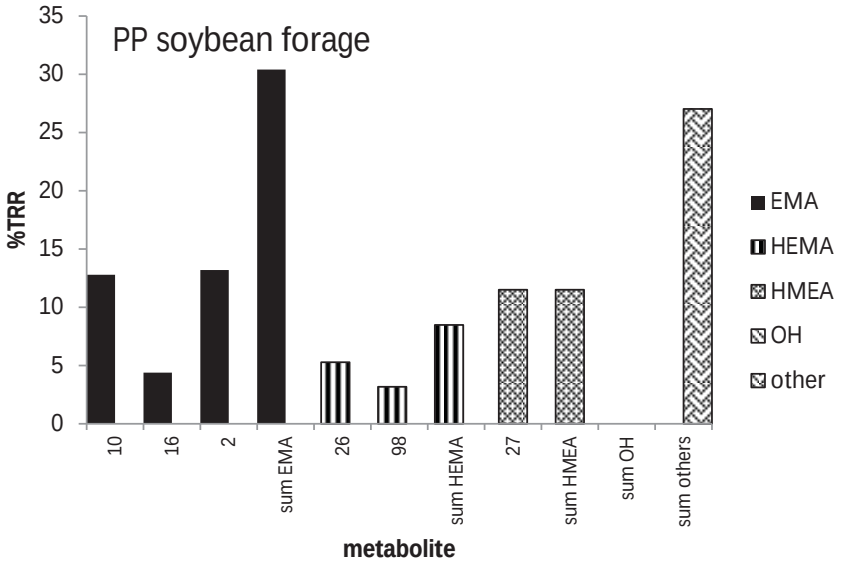
Code	Identification	% TRR	mg equiv/kg
118	*sec*-sulfinyllactic acid	4.3	0.49
56	*tert*-cysteine	8.6	0.984
56	*tert*-cysteine	30.3	3.468
21	*tert*-sulfinyllactic acid +	23.8	2.725
105	*tert*-malonylcysteine sulfoxide		
104	*tert*-malonylcysteine	22.9	2.618
	Total %identified	89.8	10.285

Table 14 Summary of identified or characterised metabolites in different fractions from PO soya bean hay

Code	Identification	% TRR	mg equiv/kg
21	*tert*-sulfinyllactic acid +	29.9	17.272
105	*tert*-malonylcysteine sulfoxide		

Code	Identification	% TRR	mg equiv/kg
22	glucose conjugate of *sec*-hydroxy acetochlor	1.7	1.005
118	*sec*-sulfinyllactic acid	7	4.042
100	*tert*-cysteine sulfoxide +	4	2.301
103	*sec*-hydroxy malonylglucose conjugate +		
	additional unknown conjugate		
103	malonylglucose conjugate of *sec*-hydroxy	1.8	1.05
104	*tert*-malonylcysteine	18.4	10.624
89	glucose conjugate of *tert*-sulfinyllactic acid	1.9	1.069
86	+ 1-hydroxyethyl *tert*-sulfinyllactic acid		
102	1-hydroxyethyl *tert*-cysteine	5.4	3.096
79	1-hydroxyethyl *sec*-sulfinyllactic acid	1.8	1.01
82	5-hydroxy *sec*-sulfinyllactic acid		
–	two unknown components, the largest of which was 2.167 mg equiv/kg (3.72% of TRR)	5	2.912
	Total %identified and/or characterised	76.9	44.387

The identified metabolites are plotted according to their aniline metabolite class for forage and hay (Figure 6). The major aniline metabolite class in soya bean commodities are EMA and "other" for PE forage, HEMA, EMA and "other" for PE hay and EMA for PO hay.



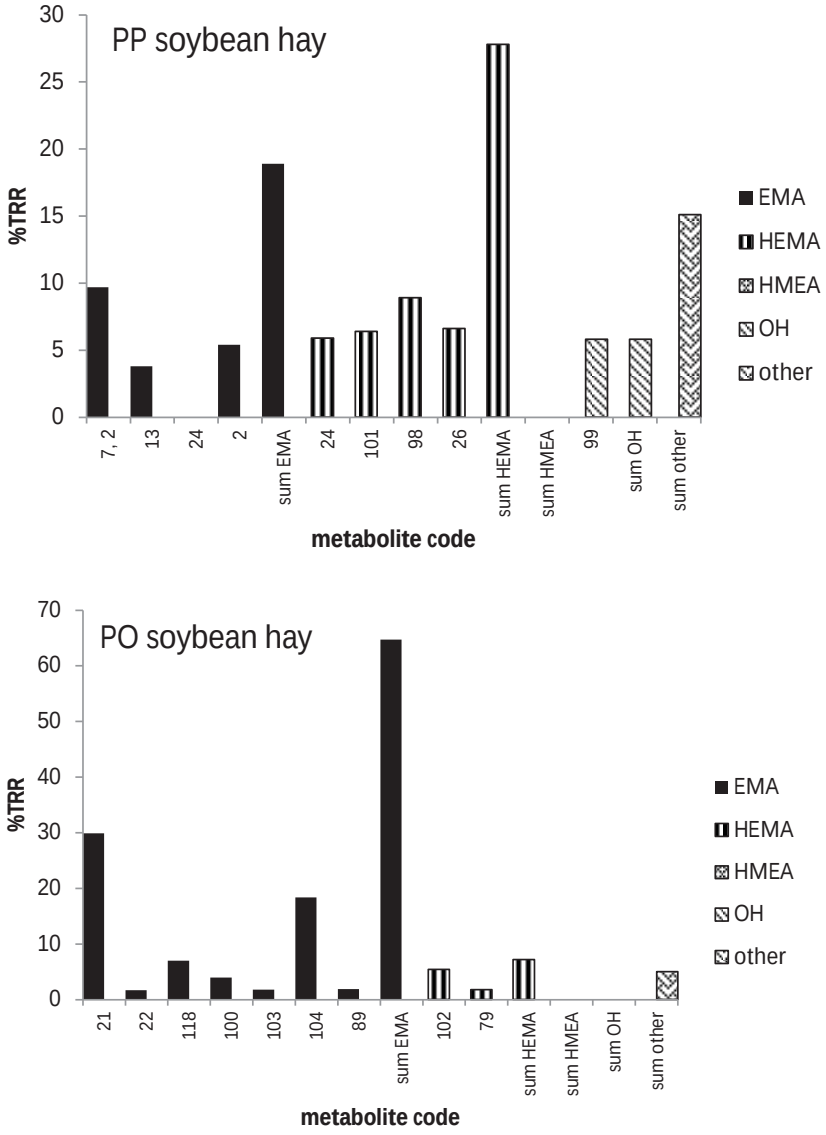


Figure 6 Aniline metabolite classes for pre- and post-emergence application of acetochlor to soya beans

A pathway for the metabolism of acetochlor in soya beans is shown in Figure 7.

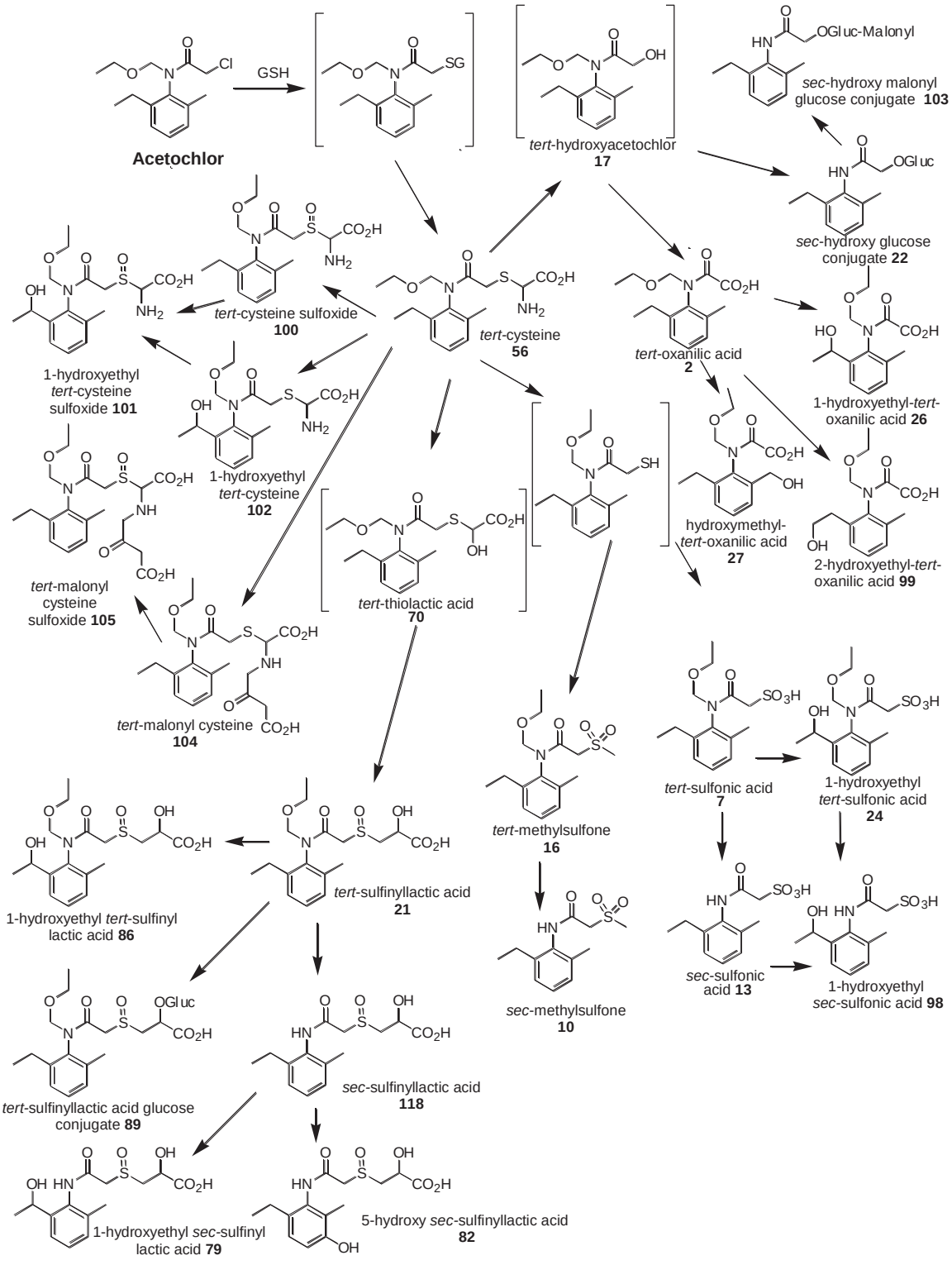


Figure 7 Proposed pathway for metabolism of acetochlor in soya bean plants

Woodbury and Baker (2008 MSL0021111) conducted a metabolism study with [^{14}C]acetochlor on cotton maintained outdoors. The test substance consisted of [U-^{14}C phenyl] labelled acetochlor and also contained a ^{13}C-label at the C-2 position of the 2-chloroacetamide moiety to aid in structure elucidation of metabolites by mass spectroscopy. A PP application was made to the soil (sandy loam) 30 days before seed planting. A separate PO application was made to a second group of plants 15 days after the majority of plants had reached their first white

flower stage. The application rates were 3.6 kg ai/ha for the PP and 3.6 kg ai/ha for the PO application.

Mature leaves/stems and seed were harvested 205 and 91 days after the application for PP and PO, respectively. Cotton was processed in a miniature gin at the field site. The leaves/stems were used as a surrogate for gin trash to increase the potential for obtaining sufficient material for metabolite identification. The use of leaves and stems provided not only a larger quantity of plant material for extraction and identification of metabolites, but also provided plant matrix with potentially higher ^{14}C levels because of the direct application of the test substance to the foliage.

Analysis of PO leaves/stems gave a TRR of 63.9 mg equiv/kg while the TRR in PP leaves/stems was much lower at 5.7 mg equiv/kg. The TRRs in seed from both treatments were both similar at 0.133 mg equiv/kg for the PO treatment and 0.103 mg equiv/kg for the PP treatment.

Cotton seed was subjected to an exhaustive extraction procedure. Hexane extracted 0.013 mg equiv/kg (12.2% TRR) from PP seed and 0.008 mg equiv/kg (5.8% TRR) from PO seed. CH_3CN/H_2O extracts of defatted seed contained 0.030 mg equiv/kg (29.3% TRR) and 0.058 mg equiv/kg (43.7% TRR) from PP and PO seeds, respectively. A further series of extractions with 0.1 N HCl (2×), 0.1 N NaOH (2×), methanol, DMSO, and THF followed by reflux with 0.1 N HCl each extracted only a small fraction of the TRR. Extraction with 24% KOH to release residues from the hemicellulose fraction was more successful and removed 0.030 mg equiv/kg (29.5% TRR) from PP seed and 0.029 mg equiv/kg (21.6% TRR) from PO seed. The latter 24% KOH extracts were characterised by partitioning with EtOAc under basic and acidic conditions that showed the majority of the radioactivity remained in the aqueous phase. This could indicate polar neutral products were released as a result of cell wall disintegration. A final treatment of the PES from the above with 72% H_2SO_4, followed by dilution with water and autoclaving, solubilised only 0.007 mg equiv/kg (6.7% TRR) and 0.009 mg equiv/kg (6.6% TRR) from PP and PO treated seed, respectively. The material following these harsh procedures still contained 0.030 mg equiv/kg (28.8% TRR) and 0.036 mg equiv/kg (26.7% TRR), respectively, in the PP and PO treatments. The general similarity of the extraction data could indicate that comparable radioactive components were formed in both PP and PO treatments. The relatively high percentage of TRR remaining in the material after exhaustive extraction may indicate covalently bound residues or reincorporation of radiolabel into natural products. Because of the low level of ^{14}C remaining, no further characterisation was conducted.

The radioactivity in hexane extracts from cotton seed was further characterised by solvent partitioning experiments. The CH_3CN phases (polar lipids or metabolites) contained 0.002 mg equiv/kg (1.9% TRR) and 0.002 mg equiv/kg (1.5% TRR) in PP and PO, respectively. The corresponding hexane phases (lipids) contained 0.011 mg equiv/kg (10.7% of TRR) and 0.006 mg equiv/kg (4.5% TRR). The lipid phase was saponified into three fractions: the non saponifiable, saponifiable (free fatty acids), and acidic aqueous (e.g., glycerol) fractions. In the PP hexane extracts, the non-saponifiable fraction represented 0.002 mg equiv/kg (1.9% TRR), saponifiable fraction 0.007 mg equiv/kg (6.8% TRR), and acidic aqueous fraction 0.002 mg equiv/kg (1.9% TRR), respectively. In the PO hexane extracts, the corresponding fractions represented < 0.001 mg equiv/kg, 0.005 mg equiv/kg (3.8% TRR), and 0.001 mg equiv/kg (0.8% TRR), respectively. Thus, there was evidence for incorporation of radiolabel into natural products, albeit at a low level.

The radioactivity in CH_3CN/H_2O extracts (polar lipids/metabolites) from cotton seed was analysed by HPLC. Both PP and PO CH_3CN/H_2O extracts contained numerous metabolites (more than 17), each < 0.01 mg equiv/kg. PP metabolites were generally more polar in character than PO metabolites.

Cotton leaves/stems were extracted sequentially with CH_3CN/H_2O, water, 0.1 N HCl, and 0.1 N NaOH. CH_3CN/H_2O extracts contained 5.098 mg equiv/kg (88.7% TRR) and

54.825 mg equiv/kg (85.8% TRR) in PP and PO treated leaves/stems, respectively. Water, 0.1 N HCl, and 0.1 N NaOH extracted 0.144 mg equiv/kg (2.5% TRR), 0.040 mg equiv/kg (0.7% TRR), and 0.161 mg equiv/kg (2.8% TRR), respectively, from PP leaves/stems. Corresponding extracts from PO leaves/stems contained 1.661 mg equiv/kg (2.6% TRR), 0.575 mg equiv/kg (0.9% TRR), and 2.173 mg equiv/kg (3.4% TRR), respectively.

CH_3CN/H_2O extracts from leaves/stems from either the PP or PO were combined and concentrated, and the residues were analysed by HPLC.

Table 15 Results for the sequential extraction of cotton seed and leaves/stems

	Seed PP		Leaves /stems	PP	Seed PO		Leaves /stems	PO
	(mg equiv/kg)	%TRR	(mg equiv/kg)	%TRR	(mg equiv/kg)	%TRR	(mg equiv/kg)	%TRR
TRR	0.103		5.748		0.133		63.9	
Extracts	0.0905	91.7	5.443	94.7	0.123	92.4	59.2	92.7
Hexane	0.013	12.2	–	–	0.008	5.8	–	–
CH_3CN/H_2O	0.03	29.3	5.098	88.7	0.058	43.7	54.825	85.8
H_2O	–	–	0.144	2.5	–	–	1.661	2.6
0.1N HCl	0.005	4.4	0.04	0.7	0.006	4.7	0.575	0.9
0.1N NaOH	0.004	4.2	0.161	2.8	0.005	3.8	2.173	3.4
Methanol	0.002	1.6	–	–	0.003	2.3	–	–
DMSO	0.001	0.9	–	–	0.001	1	–	–
THF rinse	< LOD	n/a	–	–	0.001	0.4	–	–
0.1N HCl reflux	0.003	2.9	–	–	0.003	2.5	–	–
24% KOH	0.03	29.5	–	–	0.029	21.6	–	–
H_2SO_4	0.007	6.7	–	–	0.009	6.6	–	–
PES	0.03	28.8	0.444	7.7	0.036	26.7	2.111	3.3
Total	0.124	120.7	5.886	102.4	0.158	119.1	61.345	96

In contrast to maize and soya bean, the metabolites identified following PP and PO applications were both from initial conjugation of acetochlor with glutathione, followed by subsequent loss of glutamate, then glycine. The resulting cysteinyl product underwent oxidation, deamination, dealkylation, and further conjugation with malonate or glucose to produce numerous metabolites. Only one compound exceeded 10% of TRR in PP leaves/stems: 1 hydroxyethyl-*sec*-methylsulfone glucosylsulfate conjugate (14.8%TRR) and one following PO application: *sec*-sulfinyllactic acid (20% TRR).

Table 16 Summary of identified or characterised metabolites in different fractions from PP cotton leaves/stems

Code	Identification	% TRR	mg equiv/kg
	At least five components based on hydrolysis [a]	4.3	0.247
	Multiple polar unknowns [a]	8.8	0.508
	Multiple polar unknowns [a]	7.9	0.456
	At least eight radioactive products upon acid hydrolysis, none of which was greater than 2.6% of the TRR [a]	7.7	0.442
114	glucosylsulfate conjugate of 1-hydroxyethyl-*sec*-methylsulfone	14.8	0.849
113	glucose conjugate of 1-hydroxyethyl-*sec*-methylsulfone [a]	20	1.149
114	1-hydroxyethyl-*sec*-methylsulfone glucosylsulfate conjugate [a]		
112	glucosylsulfate conjugate of *sec*-hydroxy [a]		
13	*sec*-sulfonic acid [a]		
	Total %identified and/or characterised	63.5	3.651

[a] Fraction contained multiple components

Table 17 Summary of identified and characterised metabolites in different fractions from PO cotton leaves/stems

Code	Identification	% TRR	mg equiv/kg
3	*tert*-sulfinylacetic acid	2.8	1.79
118	*sec*-sulfinyllactic acid	19.6	12.539
104	*tert*-malonylcysteine	3.5	2.223
105	*tert*-malonylcysteine sulfoxide	2.8	1.763
107	*sec*-thiolactic glucose conjugate [a]	1.5	0.947
67	*sec*-cysteine conjugate [a]	4.1	2.598
13	*sec*-sulfonic acid [a]		
83	*sec*-sulfinyllactic-glucose conjugate [a]		
112	*sec*-hydroxy glucosylsulfate [a]		
116	*sec*-malonylcysteine sulfoxide [a]	2.2	1.378
76	*sec*-sulfinylacetic acid [a]		
108	*sec*-thiolactic acid malonyl conjugate [a]	2.0	1.286
105	*tert*-malonylcysteine sulfoxide [a]		
79	1-hydroxyethyl *sec*-sulfinyllactic acid [a]	5.6	3.564
106	*sec*-thiolactic acid	4.8	3.063
111	glucosylsulfate conjugate of hydroxy *tert*-sulfinyllactic acid [a]	1.5	0.96
111	glucosylsulfate conjugate of hydroxy *tert*-sulfinyllactic acid [a]	3	1.953
117	1-hydroxyethyl-*sec*-thiolactic acid [b]	7.8	5.014
117	1-hydroxyethyl *sec*-thiolactic acid [ab]	4.7	3.031
109	glucosylsulfate conjugate of hydroxy *sec*-thiolactic acid [a]		
110	glucosylsulfate conjugate of hydroxy *sec*-thiolactic acid [a]	5.3	3.404
115	*sec*-malonylcysteine [a]		
82	5-hydroxy-*sec*-sulfinyllactic acid	8.3	5.283
	Total %identified and/or characterised	79.5	50.8

[a] Fraction contained multiple components

[b] The position of substitution of the hydroxy group has not been conclusively determined. One of these metabolites may be hydroxymethyl *sec*-thiolactic acid.

The identified metabolites are plotted according to their aniline metabolite class for leaves and stems (Figure 8). The major aniline metabolite class in cotton leaves and stems are EMA and HEMA.

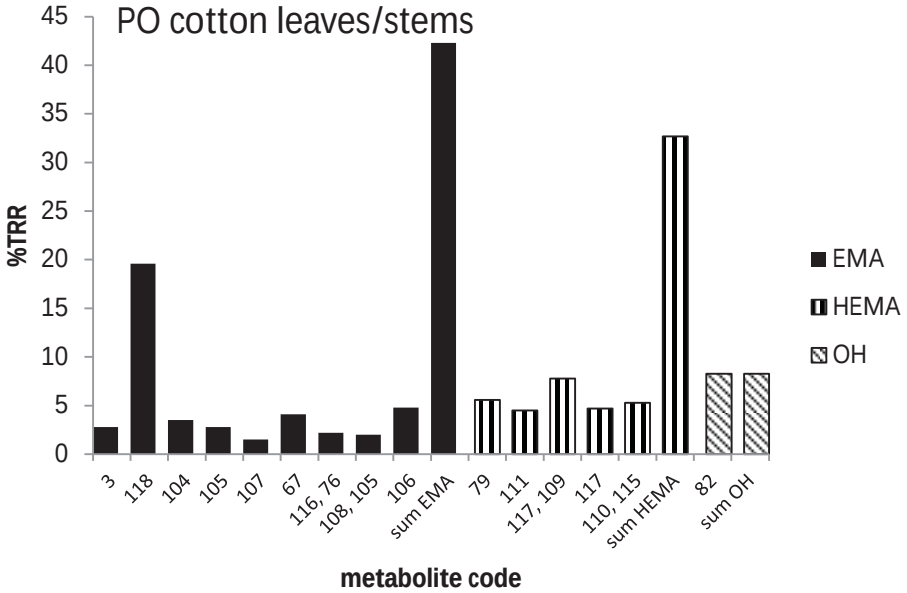


Figure 8 Aniline metabolite classes for post-emergence application of acetochlor to cotton

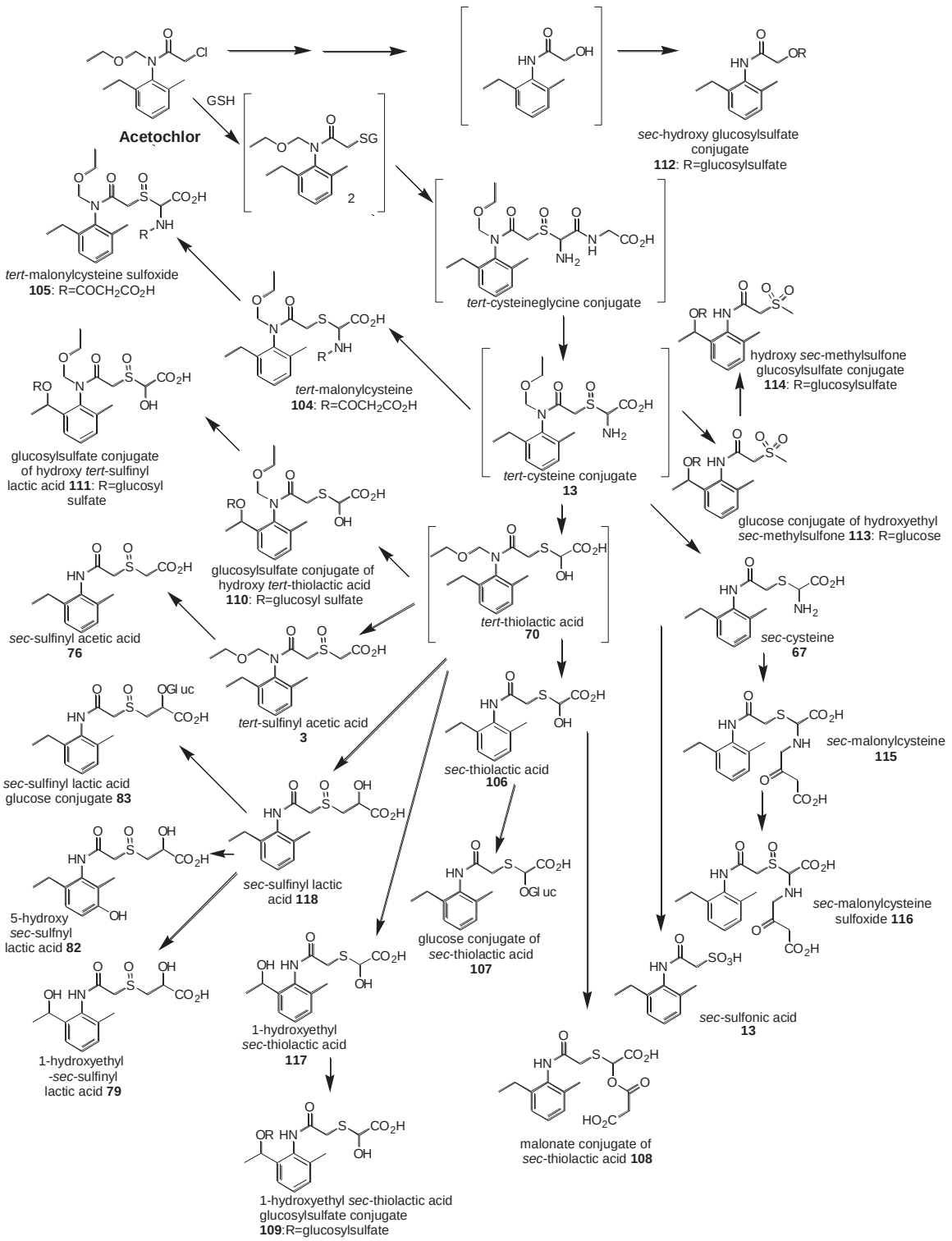


Figure 9 Proposed pathway for metabolism of acetochlor in cotton plants

Primary metabolic pathways of acetochlor in plants included:

- hydrolytic/oxidative dechlorination to form the alcohol (and conjugates) and subsequent oxidation of the alcohol to the oxanilic acid
- displacement of chlorine by glutathione (or homoglutathione) and further catabolism of the products to cysteine or lactic acid metabolites, and the S-oxides and conjugates, or to sulfonic acids and methyl sulfones
- ethyl/methyl side-chain or ring hydroxylation
- N dealkylation.

Oxanilate, sulfonic acid, and sulfone metabolites were more prevalent in pre-plant matrices. Glutathione/homoglutathione conjugation followed by catabolism to cysteine and lactic acid metabolites, and their oxidized derivatives and conjugates, was the primary metabolic pathway for acetochlor after post-emergence treatment.

The metabolism on maize, soya bean and cotton is consistent with less exhaustive studies reported in the literature for metabolism of acetochlor by other plants. Breaux (1987) [Breaux EJ (1987) *Initial Metabolism of Acetochlor in Tolerant and Susceptible Seedlings. Weed Science* 35: 463–468.] reported the initial metabolism of acetochlor in tolerant and susceptible plants (six crop and ten weed species) involved conversion of acetochlor initially to thioether conjugates. In thirteen of the species, initial conjugation was with glutathione (GSH).

Animal metabolism

Laboratory animal studies

Metabolism of acetochlor in rats, mice and monkeys was evaluated by the WHO Core Assessment Group of the 2015 JMPR.

Lactating goat

Powell and Skidmore (1991 RJ1019B) studied the metabolism of acetochlor in lactating goats (British Saanen X Nubian, 58.0–59.5 kg bw; 2.0 kg milk/d). Two goats were orally administered [^{14}C-U-phenyl]-acetochlor at 10 mg/doses, twice daily for a period of four consecutive days. Feed consumption during the dosing period was 1.7 kg/d for one goat and 2.6 kg/d for the other. The dosages were equivalent to 11.0 and 8.1 ppm in the diet. Milk production averaged 3.1 L/d. During the treatment period, milk, urine, and faeces were collected daily from both goats. Approximately 23 hours after the final dose, the goats were sacrificed and tissues were collected.

The majority of the ^{14}C residues was recovered in the excreta; between 77 and 100% of the radioactive residues were found in the excreta. Urine contained between 58 and 71% of the administered dose while faeces contained 20 to 29%.

Transfer of radioactivity into milk was very low reaching 0.016 mg equiv/L after two days. Following centrifugation and partition with hexane, the majority (98.1% TRR) of the radioactivity in the milk remained with the aqueous phase, indicating that insignificant levels of radioactivity were associated with butter fat/cream.

Further 'clean-up' of the aqueous fraction resulted in 51.3% of the total milk residue being analysed by chromatography. Up to nine individual components were observed, two of which were identified as *sec*-cysteine (67) (3.2% TRR; 0.00045 mg equiv/kg) and *tert*-cysteine (56) (18.6% TRR: 0.0026 mg equiv/kg). No other component represented > 4.4% TRR (0.00062 mg equiv/kg) of the milk residue.

For tissues, ^{14}C residues were highest in liver, (0.277–0.588 mg equiv/kg), followed by the kidney (0.247–0.479 mg equiv/kg). In general, levels of radioactivity were lowest in fat

(0.002–0.003 mg equiv/kg). Muscle tissues also featured radioactivity levels that ranged from 0.012 to 0.024 mg equiv/kg.

The majority of the residue that was found in the muscle (100%), liver (94.6%), and kidney (85.9%) tissues was bound, and was not recovered by mild extraction techniques using organic solvents or water at ambient temperatures. Incubation in the presence of β-glucuronidase also did not release the ^{14}C from these tissues.

That majority of the radioactive residue was solubilised only after acid hydrolysis at elevated temperatures (70 °C) or by digestion with a protease enzyme (papain). These results suggest that a large proportion of the radioactive residues are associated with natural proteins.

No parent acetochlor was found in the urine and tissues analysed, although small quantities were observed in the faecal samples. The metabolites of acetochlor in ruminants produce an extensive and complex mixture of components. The proposed biotransformation pathway involves the conjugation of acetochlor with glutathione or N-de-ethoxy methyl acetochlor followed by subsequent metabolism to the respective cysteine and mercapturic acid conjugates.

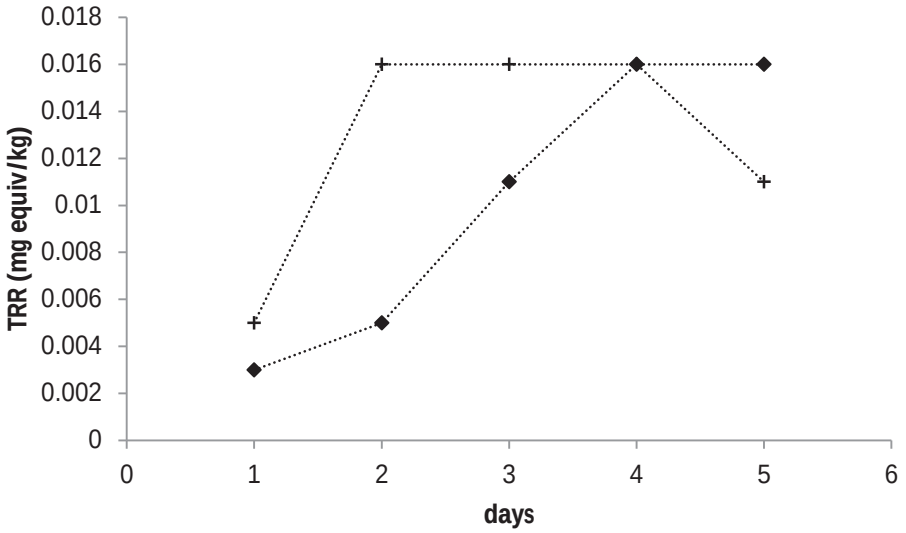


Figure 10 TRR in milk during the dosing period (♦ = goat 2, + = goat 3)

Table 18 Distribution of ^{14}C following administration of [^{14}C-U-phenyl]-acetochlor for 5 days

	Goat 2	(11 ppm)	Goat 3 (8.1 ppm)	
	%AD	mg equiv/kg	%AD	mg equiv/kg
Tissues				
Liver	0.52	0.277	0.91	0.588
Kidney	0.06	0.247	0.09	0.479
Whole milk (Day 4)		0.016		0.016
Peritoneal fat	0.006	0.003	0.008	0.003
Peri-renal fat	0.002	0.002	0.001	0.003
Subcutaneous fat		0.004		0.008
Diaphragm		0.012		0.024
Forequarter muscle	0.05	0.018	0.06	0.022
Hindquarter muscle	0.06	0.018	0.09	0.020
Excreta				
Faeces	19.7		29.3	
GIT and contents				
Urine	58.1		71.3	
Cage wash				

	Goat 2	(11 ppm)	Goat 3 (8.1 ppm)	
	%AD	mg equiv/kg	%AD	mg equiv/kg
Total	78.5		101.8	

Table 19 Summary of fractionation of [^{14}C-U-phenyl]acetochlor residues in tissues and milk from Goat 3 8.1 ppm in the diet

	Milk		Kidney		Liver		Muscle	
	%TRR	mg/L	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
TRR (mg equiv/kg)		0.014		0.458		0.60		0.019
Fraction								
Hexane	6.5	< 0.001	–		–		–	
Acetonitrile	1.3	< 0.001	3.1	0.014	1.3	0.008	–	
Acetonitrile/water	80.4	0.011	11	0.05	4	0.024	–	
Water	–		–		–		–	
Unextracted	7.6	0.001	85.9	0.39	94.6	0.0568	100	–

Table 20 Characterisation and identification of ^{14}C residues in milk, kidney and liver and excreta (Goat 3) Figures in brackets are the number of unknowns and the maximum %TRR)

Fraction	Milk		Kidney		Liver		Urine	Faeces
Compound	%TRR	mg equiv/L	%TRR	mg equiv/kg	%TRR	mg equiv/kg	%TRR	%TRR
TRR [a]	100	0.014	100	0.458	100	0.60		
Analysed solvent extract	51.3		10.7		2.5		100	100
acetochlor	–	–	–	–	–	–	–	0.8
sec-acetochlor (8)	–	–	–	–	–	–	–	6
sec-cysteine (67)	3.2	0.00045	–	–	–	–	2.9	–
tert-mercapturic acid (44)	–	–	–	–	–	–	2.6	–
tert-cysteine (56)	18.6	0.0026	–	–	–	–	23.6	–
Unknowns [c]	14.9 (7 max 4.4)	0.00209	8.4 (11 max 2.0)	0.038	0.2 (2)	0.02	12.9 (4 max 7.5)	61 (8 max 24.1)
Baseline/polar	0.6	0.00008	NA	NA	NA	NA	41.2	32.2
Remainder	14	0.002	2.3	0.01	0.8 + 1.5	0.005	16.8	–
Unanalysed aqueous soluble	7.0 (2)	0.0098	1.0 (3)	0.005	1.8 (3)	0.011	–	–
Unanalysed organic soluble	16.1 (5) [b]	0.0023	2.5 (5)	0.011	0.2 (2)	0.001	–	–
Losses during workup	13	0.0018			9.5	0.057		
Gains during workup			1.8					
Associated with solid fractions	12.6 (5)		1.4 (2)		1.9 (3)			
Residues released via papain	–	–	82.6	0.378	84.1	0.505	–	–

[a] TRR calculated from a summation of ^{14}C in extracts and PES

[b] Milk: hexane 6.5%TRR (3 unknowns), ethyl acetate 9.2%TRR (1 unknown), acetonitrile 0.4%TRR (1 unknown) total 16.1%TRR (5 unknowns)

Cell fractionation procedures were used to identify the location of ^{14}C in natural products, which also showed that the majority of the residue was associated with proteins.

Table 21 Characterisation of ^{14}C in milk, kidney and liver and distribution among cell fractions

Fraction	Milk		Kidney		Liver [a]	
Compound	%TRR	mg/L	%TRR	mg/kg	%TRR	mg/kg
Solvent extract (%lipid)			14.1		5.3	
Released by protease papain [b] (%protein)			82.6		84.1	
Cell fractionation						
Lipid			12.6		6.4	
Protein			70.2		82.5	

Fraction	Milk		Kidney		Liver [a]	
Compound	%TRR	mg/L	%TRR	mg/kg	%TRR	mg/kg
Glycans			6.8		5.2	
DNA			1.5		1.6	
Initial perchloroacetic acid (PCA)			6.6		4.4	

[a] Liver: Initial PCA, glycols and DNA were <6% and considered to be noise or low levels of radioactivity leaching into these fractions.

[b] Papain from *Papaya latex*

Due to the complex nature of the residue in animals, residue analytical methods have been developed which determined residues as common moieties, i.e. substituted anilines designated as EMA and HEMA. Strong base hydrolysis was used to convert relevant metabolites to their corresponding anilines. Radioactive residues solubilised with papain were shown to contain components containing the EMA/ HEMA moieties. In muscle, a fraction containing, on average, 29.1% of the residue was found in the extract expected to contain EMA/HEMA. Similarly, in liver and kidney an average of 43.4% and 33.8% respectively, were extracted into organic solvent after strong base hydrolysis. EMA was quantified as representing 0.07 and 0.03 mg equiv/kg in liver and kidney respectively, while HEMA was present at 0.01 and approximately 0.01 mg equiv/kg for liver and kidney respectively.

Table 22 Characterisation of class of ^{14}C metabolites present in muscle, kidney and liver

Fraction	Muscle		Kidney		Liver X Fig 18	
Compound	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
TRR		0.023		0.474		0.608
Amount papain extracts	61.2		88.4	0.419	97.9	
Aqueous phase	33.6		58.2		52.8	
Organic phase [a]	29.1	0.007	33.8	0.160	42.1	0.26
EMA		< 0.01		0.03		0.07
HEMA		< 0.01		< 0.01		0.011

[a] Organic phase = where ethylmethylaniline (EMA) and hydroxyethylmethylaniline (HEMA) expected

No acetochlor was found in the urine and tissues analysed but small quantities were observed in the faecal samples. It can be concluded that the metabolism of acetochlor in a ruminant species is extensive, with little or no potential for accumulation of metabolites in milk or tissues at the levels expected in the feed under normal agricultural practice.

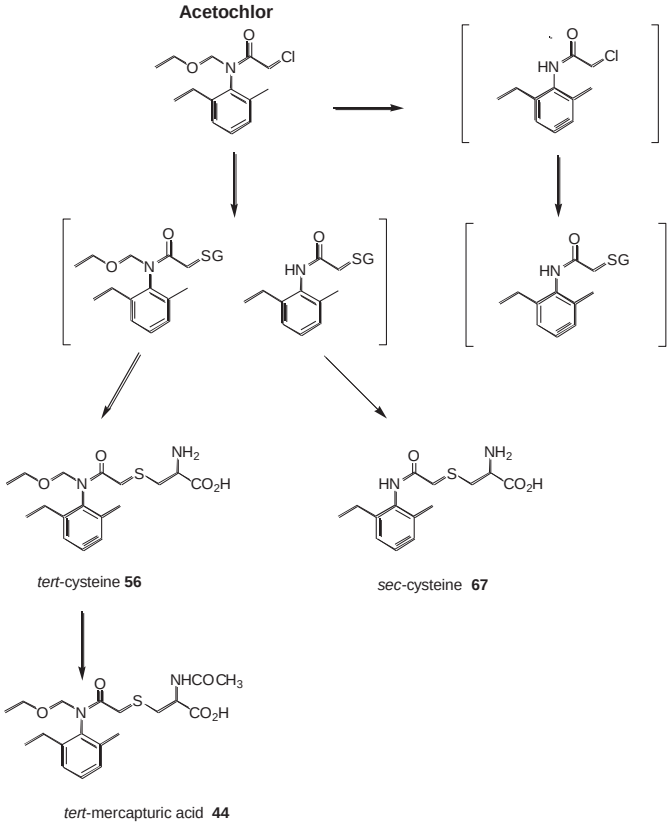


Figure 11 Metabolism of acetochlor in lactating goats

Laying hen

Hand *et al.* (1991 RJ0987B) studied the metabolism of acetochlor in laying hens. Ross Hisex Brown hens (32 weeks old, 1.8–2.1 kg bw) were dosed orally *via* capsules, once a day for seven consecutive days, with [^{14}C-U-phenyl]acetochlor at doses equivalent to 10 ppm in the diet. Feed consumption was 104–150 g/d. Laying efficiency during the dosing period was 62%. Egg samples were collected twice a day, excreta samples were also collected twice daily. Eggs were separated into egg whites and yolks. Hens were sacrificed 23 hours after the final dose, and samples of skin plus subcutaneous and peritoneal fat, leg and breast muscle, kidneys, liver, and gastrointestinal tract and contents were collected from different hens. Eggs, tissues, and excreta were extracted and analysed within five months of sampling.

Recovery of the administered dose was 73.9 to 85.8% after the dosing period based on the eggs, tissues, and excreta with the majority found in the excreta (72.3 to 68.0% AD). Carcass (3.3 to 4.5% AD) and gastrointestinal tract and contents (0.19 to 1.05% AD), eggs (0.05–0.50% AD) and edible tissues (muscle, liver, fat, and skin; 0.27–0.52% AD) accounted for relatively minor amounts of the dose.

Table 23 Percent of administered dose recovered in eggs, tissues, and excreta of laying hens following seven consecutive daily oral doses of [^{14}C-U-phenyl]acetochlor

Sample	% Dose [a]	Residue (mg equiv/kg)
Excreta	68–82	
Gastrointestinal tract and contents	0.19–1.1	
Eggs	0.05–0.5	
Liver	0.16–0.21	0.337
Kidneys	0.06–0.11	
Fat [b]	0–0.04	0.041 skin/sub cut 0.019 peritoneal
Muscle [c]	0.11–0.28	0.072 leg, 0.054 breast
Heart/gall bladder	0.02–0.04	

Sample	% Dose [a]	Residue (mg equiv/kg)
Carcass	3.3–4.5	
Total	74–86	

[a] Determined from initial combustion or solubilisation analysis of tissues, eggs, and excreta, expressed as percentage of the total activity dosed

[b] Summation of skin and subcutaneous fat and peritoneal fat

[c] Summation of leg and breast muscle

Radioactivity reached its highest level in eggs on Day 7 from the start of dosing, with average concentrations of 0.072 mg equiv/kg for yolk and 0.007 mg equiv/kg for egg whites. Mean TRR levels in edible tissues were 0.337 mg equiv/kg in liver, 0.054 mg equiv/kg in breast muscle, 0.072 mg equiv/kg in leg muscle, 0.019 mg equiv/kg in peritoneal fat, and 0.041 mg equiv/kg in skin plus subcutaneous fat.

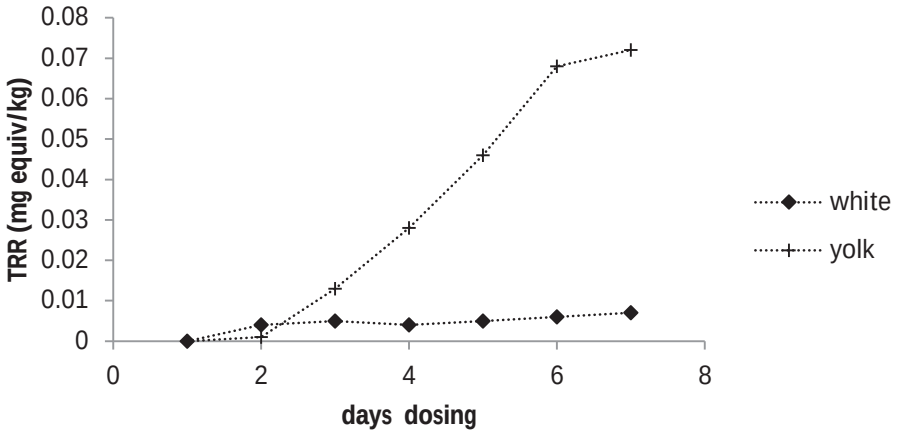


Figure 12 TRR in whole eggs of laying hens following seven consecutive daily oral doses of [^{14}C]acetochlor

Sequential extraction with CH_3CN, CH_3CN:H_2O, then water recovered 13.4 to 28.2% TRR for liver, muscle, and fat and 27.1 to 27.4% TRR for egg yolks. The extraction of excreta was nearly quantitative at 96.1%. Approximately 24–36% TRR in the fat was extracted using hexane, acetonitrile, and acetonitrile/water. Subsequent enzymatic treatment of the unextracted radioactivity in the liver, muscle, fat, and egg yolk with the protease papain (and β-glucuronidase digestion in selected samples) released an additional 21.6–56.0% TRR. The remaining radioactivity in debris and other extractions in the eggs and tissues accounted for 17.7–46.2% TRR from each tissue.

Table 24 Characterisation and distribution of ^{14}C residues in eggs and tissues from laying hens dosed with [^{14}C]acetochlor (mean values)

	Egg yolk	Liver	Muscle	Fat
TRR (mg equiv/kg)				
%TRR				
Combined CH_3CN/H_2O extracts	27–39	21–28	13–15	14.5
Protease (papain)	22–31	46–55	47–56	45
Unextracted	18–46	18–24	38–43	31.5
β-Glucuronidase extracts (*Helix pomatia*)	4.2	2.5	5.2	–
Distribution in cell fractions				
Initial perchloric acid (PCA)	21.3	23.5	13.4	10.9
Lipid	14	10.9	6	10.8
Glycans	21.6	21.2	10.2	25
Carbohydrate	3.6	3	3.5	4.3
DNA	6.5	3.8	5.6	9
Proteins	28.4	27.8	45.2	19.4

	Egg yolk	Liver	Muscle	Fat
TRR (mg equiv/kg)				
Remainder [a]	4.8	9.7	16.3	21.6
Recovery	95.3	90.3	83.8	78.4[b]

[a] The remainder is the RNA fraction (< 1%), PCA wash, and total losses throughout the method

The nature of the metabolites in the excreta showed that the biotransformation pathway of acetochlor in hens includes glutathione conjugation and metabolism to *sec*-cysteine (67), which represents 2.7% of the total excreta residues. From the cysteine conjugate, the pathway diverges to give *sec*-mercapturic acid (40) and *sec*-oxanilic acid (12), which represent 1.2% and 7.9% of the total residues respectively. The *sec*-oxanilic acid (12**)** is speculated to be formed as a result of glutathione initiated dehalogenation of the cysteine conjugate. The remainder of the residue consisted of at least 31 components, indicating that the metabolism of acetochlor in hens is extensive and complex.

The nature of the radioactive residues in the tissues and egg yolks is complex and none of the standard reference markers available co-chromatographed with ^{14}C components from either the organosoluble or the solubilized bound residues. TLC showed that at least eight organosoluble components, all < 0.01 mg equiv/kg, and that at least six "bound" components, the largest of which represented 0.018 mg equiv/kg, were extracted or digested from the liver. The muscle and egg yolk showed at least 12 and 10 organosoluble components, respectively, all of which were < 0.01 mg equiv/kg. Bound residues in the muscle and egg yolk represented < 0.05 mg equiv/kg. To obtain further characterization on the residues, tissue and egg samples were subjected to the cell fractionation procedure. This showed that, in all cases, the majority of the residue was associated with proteins, glycan, and lipid fractions. It also showed that, in the fat, the majority of the residue was associated with the skin rather than the fat itself. Results from cell fractionation in liver, muscle, egg yolk, and fat are provided in Table 23.

Due to the complex nature of the residue in animals, strong base hydrolysis was used to identify the aniline class of metabolites. From the acetochlor residues, two main anilines were generated: EMA and HEMA. Excreta contained 20.7% and 12.5% of the total residue as EMA and HEMA containing metabolites, respectively. The liver contained 16.3% EMA class metabolites (9.3% free and 6.9% bound to the solids) and 7.6% as the HEMA metabolites. Levels of both EMA and HEMA in hydrolysed extracts of muscle, fat, and egg yolks were at or below the limit of determination (0.01 mg equiv/kg). Acid hydrolysis of the papain extract of liver samples identified 5.3% EMA and 3.4% of methylaniline. Although no individual HEMA-containing molecules were identified, the presence of the moiety shows that hydroxylation of the ethyl side chain is an important metabolic pathway for acetochlor.

In summary, laying hens were dosed with [^{14}C-U-phenyl]acetochlor at a nominal rate of 10 ppm in the diet for seven consecutive days. Acetochlor and its metabolites were readily eliminated in the excreta where 68–82% of the dose was recovered. Radioactivity reached its highest average level on Day 7 of 0.072 and 0.007 mg equiv/kg in egg yolk and whites, respectively. Overall, transfer of radioactive residues to the eggs represented 0.5% or less of eliminated doses over the seven days. Low amounts of the doses were found in the liver, muscle, and fat at the time of sacrifice, indicating very little bioaccumulation potential. The highest concentration of radioactive residues in the tissues was observed in the liver. Greater than 50% of the radioactivity in the tissues and eggs could be extracted using solvents and enzyme digestions.

The nature of the metabolites is complex, and most of the metabolites were present at levels at or below the LOD necessary for identification. Unmetabolised acetochlor was not detected in the excreta, tissues, or eggs. At least 34 metabolites were observed in excreta, indicating acetochlor undergoes degradation in hens via various metabolic pathways. Three metabolites, *sec*-cysteine (67), *sec*-mercapturic acid (40), and *sec*-oxanilic acid (12), were identified in the excreta at 5.9, 1.2, and 7.9%, respectively. A major metabolic pathway of acetochlor degradation is glutathione conjugation and catabolism to the mercapturic acid with a concomitant loss of the N-ethoxymethyl group. An alternative pathway involves the glutathione

mediated reductive dechlorination resulting in the formation of *sec*-oxanilic acid (12). Additionally, base hydrolysis of acetochlor residues to EMA and HEMA moieties in excreta, eggs, and tissues indicates that hydroxylation of the ethyl side chain is another important metabolic pathway for acetochlor.

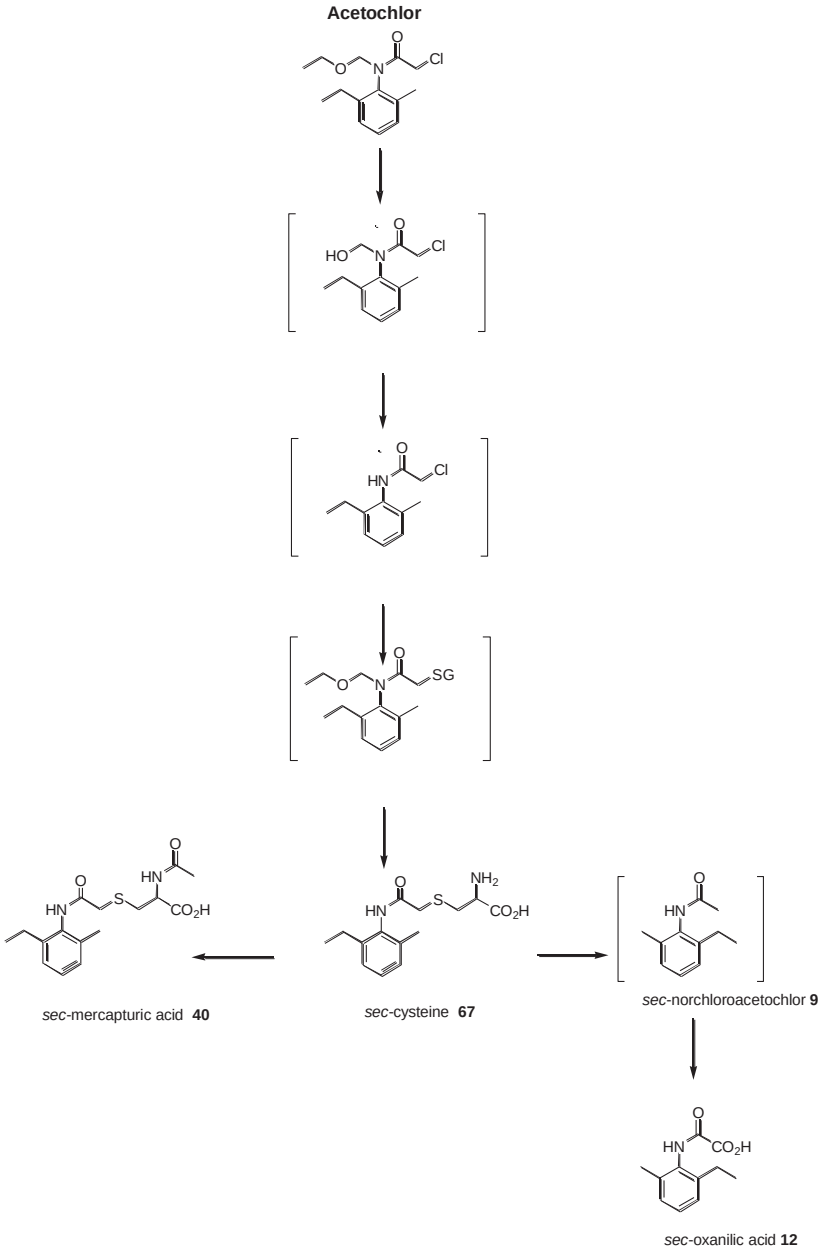
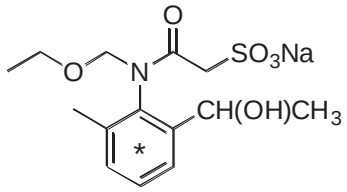


Figure 13 Metabolism of acetochlor in laying hens

The metabolism of a number of plant metabolites was also studied in livestock.

Metabolism of 1-hydroxyethyl t-sulfonic acid (CP106070) in lactating goat

Cheng (1990 MSL-10472) studied the metabolism of the benzyl hydroxylated plant metabolite ^{14}C radiolabelled 1-hydroxyethyl-*tert*-sulfonic acid (24) in a lactating goat.



acetochlor 1-hydroxyethyl t-sulphonic acid (CP106070)

In a preliminary study, one lactating goat (Preliminary Phase, 53 kg bw) was dosed orally with capsules containing ^{14}C labelled 1-hydroxyethyl-*tert*-sulfonic acid (24) for 5 consecutive days at a daily dose of 10 mg. Daily feed consumption was 1.75 kg/d. The dose was equivalent to 5.7 ppm 1-hydroxyethyl-*tert*-sulfonic acid (24) in the diet. Total production of milk, urine, and faeces were collected daily and weighed. The animals were sacrificed approximately 6 hours after the last dose, and liver, kidneys, fat, muscle, urine, gastrointestinal tract and contents, and bile samples were collected and analysed for total radioactivity content.

The total recovery was 92.3%, with 68.7% eliminated in faeces, 3.65% in urine, and 18.5% remaining in the contents of the GI tract. Less than 0.01% of the total radioactivity was detected in the entire milk production, muscle, liver, kidneys, fat, and bile. The radioactivity concentration in tissues was very low, with 0.007 mg equiv/kg (1-hydroxyethyl-*tert*-sulfonic acid (24) equivalents) in kidney, 0.003 mg equiv/kg in liver, and < LOD (0.0003 mg equiv/kg) in blood, muscle, and fat. The low radioactivity concentration in bile and tissues indicated limited absorption of 1-hydroxyethyl-*tert*-sulfonic acid (24). The majority of the radioactivity detected in kidney and urine was 1-hydroxyethyl-*tert*-sulfonic acid (24).

Table 25 Distribution of ^{14}C in tissues and excreta of a goat following dosing with 1-hydroxyethyl-*tert*-sulfonic acid

Matrix/tissue	% TRR	Residue	
		(mg 1-hydroxyethyl *tert*-sulfonic acid equivalents/kg)	(mg acetochlor equivalents/kg)
liver	< 0.01	0.003	0.002
kidney	< 0.01	0.007	0.005
blood	NA	< 0.001	< 0.001
omental and renal fat	ND	ND	< 0.002
muscle	ND	ND	< 0.002
milk	< 0.01	–	–
bile	< 0.01	0.011	0.008
gastrointestinal tract	1.29	0.144	0.11
gastrointestinal tract contents	18.53	–	–
urine	3.65	–	–
faeces	68.7	–	–
cage wash	0.09	–	–
total	92.26	–	–

NA = Not analysed

ND = Not detected

Ten lactating goats (definitive phase, 51–65 kg bw) were dosed orally with capsules containing ^{14}C labelled or non-labelled 1-hydroxyethyl-*tert*-sulfonic acid 2 **(**24) for 28 consecutive days as follows: Three animals received a daily dose of 1 mg (0.4 ppm), with one animal receiving a ^{14}C-labelled dose (2.4 kg feed/d, 1.9 L/d) and two animals receiving a non-labelled dose, three animals received a daily dose of 3 mg (1.4 ppm), with one animal receiving a ^{14}C-labelled dose (2.1 kg feed/d, 1.2 L/d) and two animals receiving a non-labelled dose, and four animals received a daily dose of 10 mg (5.6 ppm), with two animals receiving a ^{14}C-labelled (1.8 kg feed/d, 1.0 L/d) and two animals receiving a non-labelled dose. These dose levels were equivalent to approximately 0.4, 1.4, or 5.6 ppm 1-hydroxyethyl-*tert*-sulfonic acid (24) in the diet. One of the animals, which received the 10 mg ^{14}C-labelled daily dose, were maintained for a

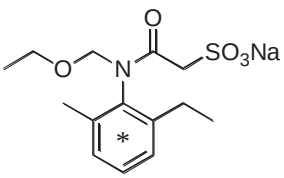
7-day depuration period following dosing and then sacrificed. The remaining nine animals were sacrificed within 24 hours after the 28th consecutive dose. Kidney, liver, fat, and muscle were collected from each animal for analysis.

The concentration in milk and tissues from animals that received the radiolabelled doses was either less than 0.001 mg equiv/kg or < LOD (0.0005 mg equiv/kg). The concentration in milk and tissues from animals that received the 10 mg non-radiolabelled doses was analysed by measuring the HEMA hydrolysis product of 1-hydroxyethyl-*tert*-sulfonic acid (24). Results indicated the residue concentration was < LOD (0.001 mg equiv/kg).

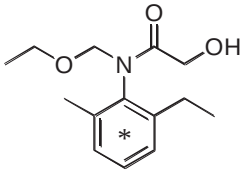
In conclusion, there was no accumulation of residues in milk and edible tissues of lactating goats after they received 28 consecutive daily oral dosings of 1-hydroxyethyl-*tert*-sulfonic acid (24) up to 10 mg/day.

Metabolism of four acetochlor metabolites co-administered to lactating goat

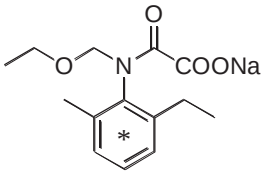
Leyes (1992, MSL-2280) studied the metabolic fate and nature of residues resulting from the metabolism of four acetochlor plant metabolites in lactating goats. Two lactating goats (Nubian, French Alpine, 49–54 kg bodyweight, 2.5 years old) were dosed with four ^{14}C-labelled synthetic plant metabolites of acetochlor twice a day for a period of five days. Milk production was 1.0–1.2 L/days. Average feed consumption was 0.6 to 0.8 kg concentrate and 2.6 to 3.4 kg hay per day. The metabolites were present in the following mass ratios: *tert*-sulfonic acid, sodium salt (7, sodium salt) (43.0%); *tert*-oxanilic acid, sodium salt (2) (33.0%); *tert*-hydroxyacetochlor (17) (22.3%); and *tert*-sulfinylacetic acid, sodium salt (3) (1.7%). The daily dose was 13.7 mg acetochlor equivalents/goat (3.2 and 4.3 ppm acetochlor equivalents for the two goats).



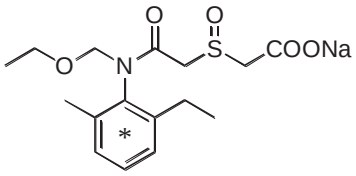
CP92429
acetochlor *t*-sulfonic acid (**7**), sodium salt (**29**)



CP68365-3
acetochlor *t*-hydroxy (**17**)



CP95200
acetochlor *t*-oxanilic acid (**2**), sodium salt



CP97290
acetochlor *t*-sulfinylacetic acid (**3**), sodium salt

Milk, urine and faeces were collected daily from each animal. On the sixth day the animals were sacrificed (12 hours after administration of the final dose) and liver, kidney, muscle, fat and blood samples were collected from each goat.

Most of the ^{14}C radioactivity was excreted (63–79% AD) with similar amounts recovered in urine (34–42% AD) and faeces (29–37% AD). Radioactivity in milk accounted for only 0.038–0.044% of the administered dose. The radioactivity present in tissues and organs was minimal.

Table 26 Distribution of ^{14}C on dosing lactating goats with a mixture containing *tert*-sulfonic acid (7), *tert*-oxanilic acid (2), *tert*-hydroxyacetochlor (17) and *tert*-sulfinylacetic acid (3)

Matrix/tissue	% AD	Mean residue
	(mean of two animals)	(mg acetochlor equivalents/kg)
liver	0.022	0.022
kidney	0.007	0.034
blood	0.024	0.004
omental and renal fat	ND	–
muscle	ND	–
milk	0.041	0.006
urine	38	–
faeces	33	–
total	71.1	

ND = not detected

Residues in milk reached a plateau by the fourth day of dosing.

Samples of raw urine and faecal extracts from each goat were profiled using reverse phase HPLC and for urine and faeces were found to be quite similar to that of the dosing solution.

To quantitate the total amount of dosing solution components or degradation products present, acid-pressure hydrolysis was performed. Hydrolyses were performed on raw urine and faecal extract samples from each of the dosed goats. In all samples subject to hydrolysis, EMA was the only aniline observed.

In conclusion, the very low levels of activity in tissues, organs, blood, and milk demonstrate that there is very little uptake of the acetochlor metabolites in the goat. The profiles of urine and faeces show that the metabolites passed through the animals with very little change. Finally, the results of this study show that the only identifiable aniline class of metabolites in the milk goat is the EMA class. This class of metabolites accounted for 83% and 77% of the recovered activity in urine and faeces, respectively, when corrected for the analytical recovery of the method.

Metabolism of four acetochlor metabolites co-administered to laying hen

Letendre *et al*. (1987 MSL-6941) studied the metabolic fate and nature of residues resulting from the metabolism of four acetochlor plant metabolites in laying hens. The dose mixture comprised an equal weight mixture of the metabolites *tert*-hydroxyacetochlor and the sodium salts *tert*-oxanilic acid, *tert*-sulfonic acid, and *tert*-sulfinylacetic acid. Each contained uniform ^{14}C labelling in the phenyl ring.

Four groups of White Leghorn hens (five per group) were dosed orally, via capsules, once a day for six consecutive days, with the acetochlor plant metabolites at an average dose of 13 ppm (acetochlor equivalents). Each metabolite used in this portion of the study was a mixture of ^{13}C-enriched and ^{14}C-labelled materials to aid in identifying metabolites. A separate group (Group 1) of five hens was dosed orally with placebo capsules for six days. Three of the four groups (Groups 2, 3, and 4), as well as the control group (Group 1) were sacrificed within 24 hours of the final dose; the fourth group (Group 5) of hens was sacrificed after a ten-day depuration period. Another group (Group 6) of three hens was dosed, via capsule, for six consecutive days at an exaggerated dose of 88 ppm (acetochlor equivalents) of the four ^{14}C-labelled metabolites and sacrificed within 24 hours of the final dose. Eggs and excreta were collected on a daily basis. The eggs were separated into egg whites (albumen) and yolk and each portion was radioassayed separately. Edible tissues were collected at the time of sacrifice and included liver, kidneys, breast muscle, thigh muscle, and abdominal fat.

The recovery of the administered dose for Groups 2–5 was 96.6–98.1%, with > 96% of the dose recovered in the excreta and cage washes. Recovery of the dose from the individual

hens from Group 6 was only slightly lower at 95.7–95.8% with the majority of the dose in the excreta and cage washes, which were pooled for the three hens to give a recovery of 95.5%.

The highest levels of ^{14}C residues were found in the gastrointestinal tract, with levels ranging from 0.211 to 0.246 mg equiv/kg for the three groups of hens (Groups 2–4) that were dosed at 13 ppm and sacrificed within 24 hours of the final dose administration. The next highest ^{14}C residues were observed in the crop and its contents, with levels ranging from 0.074 to 0.112 mg equiv/kg, respectively. The ^{14}C residue in the kidneys ranged from 0.018 to 0.020 mg equiv/kg, and for the liver ranged from 0.024 to 0.045 mg equiv/kg. The ^{14}C residue level in the gizzard ranged from 0.009 to 0.015 mg equiv/kg. The residue in the ovaries ranged from 0.021 to 0.027 mg equiv/kg. Egg whites and egg yolks collected at sacrifice had ^{14}C residue levels ranging from 0.004 to 0.007 mg equiv/kg and 0.025 to 0.033 mg equiv/kg, respectively. The ^{14}C levels in fat ranged from 0.006 to 0.011 mg equiv/kg, and the lowest levels of ^{14}C were observed in the muscle samples. Residue levels in the breast muscle ranged between < LOD and 0.010 mg equiv/kg and in thigh muscle the levels ranged from < 0.004 to 0.005 mg equiv/kg. The blood levels of ^{14}C residues taken approximately 24 hours after the final dose was administered ranged between 0.021 to 0.025 mg equiv/kg.

For the group of hens that underwent a ten-day depuration period after the last dose, the highest level of ^{14}C residues were observed in the crop at 0.044 mg equiv/kg. No residues were found in the liver and kidneys of the hens but breast and thigh muscle tissue were found to contain 0.010 and < 0.004 mg equiv/kg, respectively, and fat contained 0.006 mg equiv/kg.

The highest levels of ^{14}C found in the tissues of the hens dosed with 88 ppm (Group 6) gave similar results to the lower-dose groups. The highest levels of^{14}C were found in the gastrointestinal tract, ranging from 0.276 to 0.749 mg equiv/kg, and in the crop 0.209 to 1.03 mg equiv/kg. The ^{14}C residues in the kidneys ranged from 0.106 to 0.128 mg equiv/kg and in the liver 0.150 to 0.266 mg equiv/kg. The ^{14}C residue in the gizzard ranged from 0.031 to 0.080 mg equiv/kg. The ^{14}C content in the ovaries ranged from 0.153 to 0.162 mg equiv/kg. Egg whites and yolks collected at sacrifice had ^{14}C residue levels that ranged from 0.029 to 0.052 mg equiv/kg and from 0.192 to 0.198 mg equiv/kg, respectively. The ^{14}C residues in fat ranged from 0.049 to 0.061 mg equiv/kg. The lowest levels of ^{14}C were found in pooled breast muscle was 0.032 ppm and in thigh muscle, where the residue levels ranged from 0.024 to 0.028 mg equiv/kg.

Extractions of tissues with methanol:water (20:80) released 87% TRR in liver, 76% in kidney, 46% in breast muscle, 50% in thigh muscle, and with acetonitrile released 98% TRR in fat. Extractions conducted on eggs (90:10 CH_3CN/H_2O for whites; Bligh-Dyer method for yolks) resulted in the release of 80–97% TRR from egg whites and 31 to 63% TRR from yolks. Extracts were analysed by HPLC. In eggs, low levels of the dosing metabolites *tert*-oxanilic acid and *tert*-hydroxyacetochlor were present along with *sec*-oxanilic acid and two major unknowns. In tissues, the major component present was the dosing metabolite *tert*-hydroxyacetochlor, which represented 0 to 35% TRR in the various tissues. Small amounts of other dosing materials were also detected in some of the tissues. Analysis indicated that approximately 30 to 70% of the radioactivity contained within the tissues was associated with macromolecules. In excreta, four major components plus several minor compounds were observed. The major components were *tert*-oxanilic acid, *tert*-sulfonic acid, *tert*-hydroxyacetochlor, and *sec*-oxanilic acid; *tert*-sulfinylacetic acid was only present as a minor metabolite.

The nature of the residual metabolites was further characterised through acid hydrolysis (6 N HCl, 150 ºC, 3 hours). Acid hydrolysis of the radioactivity extracted from the liver, kidney, fat, and excreta indicates that these tissues contained one major class of metabolites that were derived from the EMA. Acid hydrolysis of breast and thigh muscle tissue produced evidence for the presence of acetochlor metabolites from the EMA, the HEMA, and HMEA aniline classes of metabolites.

Table 27 Distribution of ^{14}C residues in eggs and tissues from laying hens dosed at with four [^{14}C]-labelled acetochlor plant metabolites

Group	2 (13 ppm)		3 (13 ppm)		4 (13 ppm)		6 (88 ppm)	
	TRR [b]	%AD	TRR [b]	%AD	TRR [b]	%AD	TRR [b]	%AD
Tissues + GIT [a]		0.298		0.333		0.283		0.146
Liver	0.024	0.01	0.045	0.02	0.037	0.01	0.198	0.01
Kidney	0.019	0.003	0.02	0.003	0.018	0.002	0.119	0.003
Fat (omental and renal)	0.011	0.005	0.006	0.003	0.007	0.003	0.056	0.004
Muscle breast	< 0.004	0.001	ND	–	0.005	0.004	0.032	0.005
Muscle thigh	0.005	0.003	0.004	0.003	0.004	0.003	0.026	0.003
Ovaries	0.026	0.014	0.027	0.016	0.021	0.014	0.159	0.014
Crop	0.112	0.008	0.074	0.007	0.098	0.009	0.549	0.007
Gizzard	0.015	0.004	0.011	0.003	0.009	0.003	0.057	0.003
Egg white	0.008	0.011	0.008	0.008	0.008	0.010	0.062	0.013
Egg yolk	0.033	0.010	0.031	0.008	0.025	0.007	0.198	0.010
gastrointestinal tract	0.229	0.244	0.246	0.271	0.211	0.223	0.577	0.089
Excreta		96.7		97.6		97.3		95.5
Cage wash		0.095		0.118		0.126		0.079
Total		97.1		98.1		97.8		95.7

[a] Tissues + GIT = kidney, liver, muscle (thigh + breast), fat, ovaries, crop, gizzard, GIT and sacrifice egg yolks and whites

[b] TRR values are based on the average molecular weight of the components in the dosing mixture (309.8). A factor of 0.87 can be used to convert to acetochlor equivalents.

ND = not detected

Table 28 Identification and characterisation of ^{14}C residues in eggs and tissues from laying hens dosed at 88 ppm (Group 6) with four [^{14}C]-labelled acetochlor plant metabolites

	Egg	Egg	Liver	Kidney	Muscle [a]	Fat
	Yolk	white				
TRR [b] (mg equiv/kg)	0.198	0.052	0.266	0.128	0.030	0.061
			%TRR			
tert-oxanilic acid (2)	1.2	20.4	ND	ND	ND	1.6
tert-sulfonic acid (7)	ND	ND	ND	ND	ND	3.0
tert-sulfinylacetic acid (3)	ND	ND	ND	ND	ND	2.0
tert-hydroxy-acetochlor (17)	ND	2.9	26.0	25.0	16.7	3.9
sec-oxanilic acid (12)	6.3	ND	ND	ND	ND	ND
Total (%TRR)	7.4	23.3	26.0	25.0	16.7	10.5

[a] Combined totals for breast and thigh muscle

[b] TRR values are based on the average molecular weight of the components in the dosing mixture (309.8). A factor of 0.87 can be used to convert to acetochlor equivalents.

ND = Not detected

Table 29 Identification and characterisation of ^{14}C residues in excreta from laying hens dosed with four [^{14}C]-labelled acetochlor plant metabolites

Fraction	% ^{14}C recovery in excreta	% ^{14}C recovery in excreta
	Group 3 (13 ppm)	Group 6 (88 ppm)
tert-oxanilic acid (2)	21.6	20.6
tert-sulfonic acid (7)	21.4	21.4
tert-sulfinylacetic acid (3)	5	5.6
tert-hydroxy-acetochlor (17)	15.6	17.9
sec-oxanilic acid (12)	18	16
Total characterised by HPLC	81.6	81.5

Metabolism of oxamic acid in lactating cow

A metabolite of acetochlor in maize, 5-hydroxy-*sec*-oxanilic acid (68), uniformly labelled in the phenyl ring was used to dose a lactating cow (6 year old Friesian, 537 kg bw) at a nominal rate of 25 ppm (5-hydroxy-*sec*-oxanilic acid) in the diet for seven consecutive days (Corden *et al.* 1982 RJ1228B). Feed consumption averaged 13.7 kg/d while milk production averaged 8 L/d. Twenty-three hours after the final dose, the cow was sacrificed and the tissues collected.

Most of the administered dose was recovered from the excreta (faeces 82.5%, urine 8.4%). The majority of the urine ^{14}C was unmetabolised 5-hydroxy-*sec*-oxanilic acid (68). Four other minor components were found in the urine, each < 4.0%TRR. Two other components were found in the faeces at < 1.6%. 5-Hydroxy-*sec*-oxanilic acid (68) is rapidly excreted from cows, principally as unchanged acetochlor.

The residues in all tissues and milk were < 0.01 mg equiv/kg, except in the kidney which had a residue of 0.015 mg equiv/kg. Extraction of ^{14}C residues in kidney with $CH_3CN:H_2O$ released 70% of the TRR. Levels of ^{14}C in other tissues were too low to permit further characterisation. In the kidney 0.0068 mg equiv/kg (46.7%TRR) was unchanged 5-hydroxy-*sec*-oxanilic acid (68). The remainder of the residue was composed of unextracted material (24.5%, 0.0036 mg equiv/kg) and uncharacterized aqueous soluble material (15.0%, 0.0022 mg equiv/kg).

Table 30 Distribution of ^{14}C residues in milk and tissues from lactating cow dosed at with 5-hydroxy-*sec*-oxanilic acid

Tissue	TRR [b] (mg/kg)
Hindquarter muscle	< 0.003
Forequarter muscle	< 0.004
Subcutaneous fat	< 0.004
Peri-renal fat	0.004
Peritoneal fat	< 0.004
Liver	0.008
Kidney	0.015
Milk	0.0053 [a]

[a] Mean value of days 2 to 8 inclusive

[b] TRRs are in terms of 5-hydroxy-*sec*-oxanilic acid. A factor of 1.2 can be used to convert to acetochlor equivalents.

Table 31 Characterisation and identification of ^{14}C residues in excreta and kidney from lactating cow dosed at 25 ppm with 5-hydroxy-*sec*-oxanilic acid

Component	%TRR in urine	%TRR in faeces	%TRR in kidney	
			Method 1 [a]	Method 2 [b]
5-hydroxy-*sec*-oxanilic acid (68)	80	88.1	46.7	43.3
Unknown components (number)	11.5 (4) [c]	1.7 (2)	2.8	0.4
Baseline/polar material	–	2.6	–	–
Remainder	7.3	1.6	4.4	2.3
Radioactivity associated with solids	–	4.6	24.5 [d]	24.5 [d]
Uncharacterised aqueous soluble	1.1	–	15.0 [e]	14.3 [f]
Uncharacterised organosoluble	–	–	1.3	14.9 [g]
Losses during work-up	0.1	1.4	5.3	0.3

[a] TLC analysis of the resulting methanol fraction from the chromatographic analysis using a C_{18} column of the combined acetonitrile and acetonitrile/water extracts from the kidney sample

[b] TLC analysis of the fraction from the partition of a second subsample of the acetonitrile/water extract with ethyl acetate at neutral then acidic pH

[c] Consists of at least four components, the largest of which represents 4.0% of the total radioactive residue

[d] Consists of two fractions, 18.1 % (0.0027 mg equiv/kg) and 6.4% (0.0009 mg equiv/kg)

[e] Consists of at least four components, the largest of which represents 8% (0.0012 mg equiv/kg) of the TRR

[f] Consists of at least four components, the largest of which represents 7.3% (0.0011 mg equiv/kg) of the TRR

[g] Consists of at least three components, the largest of which represents 11.7% (0.0017 mg equiv/kg) of the TRR

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 acetochlor, supervised residue trials data are available for numerous crops. 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, hydrolysis and photolysis properties of acetochlor. Studies were also received on the behaviour of [^{14}C]acetochlor in a confined rotational crop situation.

Acetochlor residues are not persistent in soils however acetochlor residues in soils resulting from recommended uses could contribute to the residues in succeeding crops.

Confined rotational crop studies

A confined rotational crop study was conducted on a sandy loam soil (63.3% sand, 34.1% silt, 2.6% clay; pH 6.6; 2.5% organic matter; 48 meq/100 g CEC) treated with [^{14}C-U-phenyl]-acetochlor at either 2.24 or 3.36 kg ai/ha (O'Neal and Johnson 1992 MSL-12105). The lower application rate was used for rotations of 30 days while the high application rate was used with 120 and 365 day rotations. Radish (variety Red Devil B), lettuce (variety Royal Green) and wheat (variety Anza) were sown into the soil at 30, 120 and 365 (both treatment rates) days after application (DAA). Lettuce was also planted at 162 DAA. The treated boxes were maintained in a screened enclosure.

Analysis of soil extracts showed that acetochlor was metabolised to an array of metabolites, many of which were present at very low levels. In addition to acetochlor, four major soil degradates were identified as present in soil throughout the study: *tert*-oxanilic acid (2), *tert*-sulfonic acid (7), *tert*-sulfinylacetic acid (3) and hydroxyacetochlor (17). Unextracted radioactive residues in soil were characterized into soil organic components. The majority of this soil bound residue was found in the humin fraction (15–30% TRR), humic acid (10–18% TRR) and fulvic acid (5–12% TRR) of soil organic matter.

Table 32 Metabolite profile in soils at planting and at time of harvest

		30	DAA			120	DAA	162	DAA		365	DAA	
	planting	radish (56)	imm wheat (75)	wheat (162)	planting	radish (165)	wheat (276)	planting	lettuce (234)	planting	radish (402)	lettuce (421)	Wheat (452)
TRR	1.17	1.35	0.66	1.14	1.00	1.11	1.48	1.48	1.43	1.68	1.15	0.59	0.96
Extracted	68	68	72.1	43.1	53.1	58.1	39.9	54	60.2	12.1	23.2	27	24
26		0.3	0.1					0.2	0.7	0.1			0.1
24						0.4	0.2			0.1			0.1
13	1	1.8		1.3	0.5	2.3	0.8/1.4	1.7	3.1	0.6	1.4		1.2
46					0.3	0.5							
12	0.8	1.1	1.9	0.5		1.0	1.8	1.0		1.3 [a]			0.6
32						0.3			0.4	0.1	0.2		0.3
2	12.5	11.2	8.4	9.0	5.0	11.8	12.2	12.5	15.7	2.1	2.7	4.5	2.1
7	5.1	5.3	4.5			4.9	4.8	5.1		1.9	3.3	10.1	2.4
3	9.1	10.8	6.0	10.3	4.8	9.1	6.2	10.8	17.3	1.3	2.1		1.9
8		0.7	0.8		0.6	1.1					0.1		0.3
28	0.3			0.7		0.9	0.7		1.1	0.2	0.4		0.2
36	2.6	3.2	0.9	2.0		3.3	1.3	2.5	2.5	0.1			0.3
17	5.1	5.4	1.8	3.4	4.4	8.5	4.3	8.5	8.3	1.4	2.8	3.5	3.8
6	0.5	0.6	0.4	0.8		0.8		0.4					
Acetochlor	29.9	24.4	47	11.7	35.4	9.8	4.2	8.3	6.5	1.7	4.8	6.1	5.6
Unknowns	1.1 (1)	3.2 (2)	0.3 (1)	2.3 (1)	2.1 (4)	3.6 (6)	2 (1)	3.2 (3)	3.6 (4)	0.8 (6)	4.3 (6)	2.8 (1)	5.2 (7)

Acid	7	8	5	–	9	–	8	6	7	–	–		–
Base	5	4	3	–	13	–	14	8	10	–	–		–
Unextracted	26	20	2	< 1	< 1	29	26	27	28	10	39	52	138

Analysis of extracts of plant matrices showed that many of the soil degradates were also observed at significant levels in plants. Five metabolites, which were consistently present in plant extracts from all three rotation intervals were: *sec*-oxanilic acid (12), *tert*-oxanilic acid (2), *sec*-sulfonic acid (13), *tert*-sulfonic acid (7), and 1-hydroxyethyl *tert*-oxanilic acid (30).

Unextracted radioactive residues in plant matrices were characterized by cell wall fractionation. The majority of this plant bound material was incorporated into hemicellulose (3–11% of bound ^{14}C) and cellulose (32–76% bound ^{14}C) and in the case of wheat grain with starch (19% bound ^{14}C).

Table 33 Identification of ^{14}C in rotational crops following application of [^{14}C-U-phenyl]acetochlor

DAA			30				120	
Crop	radish root (56)	Radish foliage (56)	imm wheat (75)	Wheat straw (162)	wheat grain (162)	wheat chaff (162)	radish root (165)	Radish foliage (165)
TRR (mg equiv/kg)	0.30	0.52	0.14	0.98	0.05	0.78	0.19	0.67
Extracted	63	71.9	97.9	77.9	75	56.9	94.2	77
13	12.4	9.1	11.6	7				6.2
46								4.3
12	13	9.6				2.7	14.6	11.2
2	12.3	16.3		3.6		5.1	15.8	24.8
7	3.3	4.5	27.5	7.3				9.7
3	5.6	5.8	8.1	5.6		3.7	6.5	
8						0.5		
28	6.7	5.7	3.2	3.4		1.5	0.7	4.1
36	0.5	4.9				1	0.7	
17				0.6		0.1	1.9	
6								
acetochlor								
26	1.7	2.7	4.6	5.7		3.9		
24				5.5		6		
33/27			4.6	2.4		4.9	7.1	
32			5.3	4.3		2.2	3.3	
unknowns	7.5 (3)	13.3 (4)	33 (3)	32.5 (4)	75	25.3 (7)	43.6 (3)	16.7 (4)
Acid	2	6	3	6	25	10	10	10
Base	1	7	4	9	0	7	8	14
Unextracted	34	15	1	4	< 1	26	2	2

Table 34 Identification of ^{14}C in rotational crops following application of [^{14}C-U-phenyl]acetochlor

DAA			120		162				365			
Crop	Imm wheat (165)	Wheat straw (276)	Wheat grain (276)	Wheat chaff (276)	lettuce (234)	radish root (402)	Radish foliage (402)	lettuce (421)	Imm wheat	Wheat straw (452)	Wheat grain (452)	Wheat chaff (452)
TRR (mg equiv/kg)	0.27	2.88	0.10	1.37	0.08	0.14	0.23	0.09	0.38	0.97	0.05	1.01
Extracted	96.2	80	39.1	68.1	59.1	65	94.2	85.3	74.2	91.5	78	44.1
13		7.3		13.5	1.8	26.9	12.7		3.3	8.6		
46	1.9											
12	6.8	7.4			2.5	16.2	19.3	2.8	3	3.3		2.2
2	22.6	11.3	2.5	2.9	24.4	3.1	23.1	22.8	14.1	5.8	3.4	1.5
7			4.5			10.2	13.3	16.1		16.3		
3	7.7	7.7		2.9	6.8				2	8.3		7.3

DAA			120		162				365			
Crop	Imm wheat (165)	Wheat straw (276)	Wheat grain (276)	Wheat chaff (276)	lettuce (234)	radish root (402)	Radish foliage (402)	lettuce (421)	Imm wheat	Wheat straw (452)	Wheat grain (452)	Wheat chaff (452)
8		0.3			0.2					1.6		
28	1.5	6.8	0.6	1.7	7.6		2.4	11.3	6.7			1
36		0.8		0.2	2.4			3.7	0.4	1.2		
17		0.3			0.6				0.6	2.5		
6												
acetochlor		0.1										
26	9	15		5.2	1.5				2.2	6.6	3.4	4.5
24	3.1		3		0.9				3.4		6.6	4.2
33/27	2.4		5.7	2.2						5.2		
32			1.6	1.6			4.2	6.1				1.5
unknowns	41.2 (8)	23 (5)	21.2 (4)	37.9 (3)	10.4 (5)	8.6	19.2 (3)	22.5 (3)	38.5 (5)	32.8 (3)	64.6 (3)	21.9 (5)
Acid	2	6	21	12	3	13	4	4	1	13	52	24
Base	1	7	13	7	2	28	2	8	3	10	13	14
Unextracted	< 1	3	27	7	36	5	1	2	1	4	2	13

2 = *tert*-oxanilic acid
3 = *tert*-sulfinylacetic acid
6 = *tert*-norchloroacetochlor
7 = *tert*-sulfonic acid
8 = *sec*-amide chloride
12 = *sec*-oxanilic acid
13 = *sec*-sulfonic acid
17 = *tert*-hydroxyacetochlor
24 = hydroxyethyl-*tert*-sulfonic acid
26 = hydroxyethyl-*tert*-oxanilic acid
27 = hydroxymethyl-*tert*-oxanilic acid
28 = *sec*-hydroxy acetyl ester
32 = HMEA
33 = HEMA
36 = *sec*-methylsulfide
46 = *sec*-methylsulfoxide

The identified metabolites identified have are plotted according to their aniline metabolite class for various rotational crops from planting 120 days after application. The major aniline metabolite class in all crop types was EMA except wheat grain for which it was HMEA and HEMA.

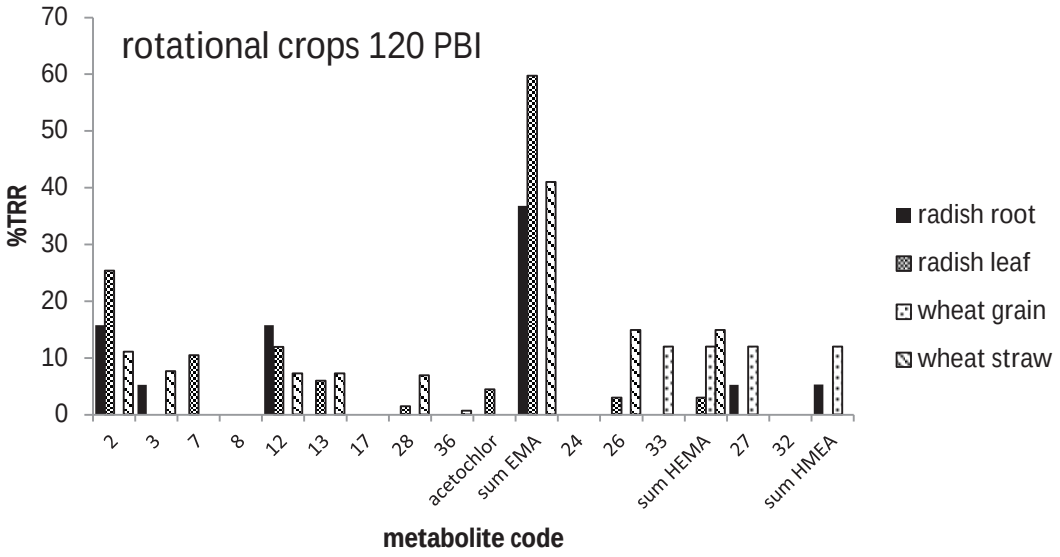


Figure 14 Aniline metabolite classes for rotational crops

In a separate study Weissler *et al.* (1995 RJ1306B) studied the residues in field confined rotational crops. [^{14}C]acetochlor was applied to the surface of a sandy loam soil at a nominal rate equivalent to 3.08 kg ai/ha. Crops typical of those rotated with corn and representative of a leafy crop, a root crop and a grain crop were planted approximately 30, 120 and 365 days after [^{14}C]acetochlor application. Soya beans were also planted approximately 30 and 365 days after application (DAA).

All crops were harvested at maturity. The grain and soya bean crops were also harvested at the immature stage (forage). Soil samples were taken at application and at each planting and harvest interval.

The radioactive residues dissipated rapidly in soil with only 22% AR remaining 30 days after application. The main identified soil metabolites were the same than those found in the laboratory aerobic soil metabolism studies, namely *tert*-oxanilic acid (2), *tert*-sulfinylacetic acid (3) and *tert*-sulfonic acid (7).

Analyses of the plant extracts showed that extensive metabolism occurred in all crops. Acetochlor was not found in any of the RACs analysed, except for day 30 turnip roots, where it accounted for 7.5% TRR (0.008 mg equiv/kg). The highest residue levels decreased from the 30 to the 365 days planting. The TRR was partially characterized and found to be comprised of up to nine different compounds, with no one above 0.01 mg equiv/kg in the edible portion of the root or cereal crop (turnip root, millet grain). The major metabolites identified in the 30 DAA rotational crops were *tert*-oxanilic acid (2), *sec*-methyl sulfone (10), *sec*-hydroxyacetochlor (11), and *tert*-methyl sulfone (16).

Further characterization of the extracted ^{14}C residue for all crops was achieved using the residue analytical methodology, and showed that the major class of metabolites was based on EMA in which no hydroxylation of the alkyl groups of the phenyl ring had occurred. A second class identified was based on HEMA and a third minor class based on HMEA.

Table 35 Identification of ^{14}C in soil at planting of rotational crops

DAA	Radioactive components in 0–15 cm soil cores (% AR)						
	acetochlor	Unknown A	*tert*-oxanilic acid (2)	*tert*-sulfonic acid (7)	*tert*-sulfinylacetic acid (3)	Unextracted	Total
1 [a]	96.2	ND	ND	ND	ND	3	97
30 [b]	6.72	1.69	0.67	0.77	0.73	7.4	22.1

DAA	Radioactive components in 0–15 cm soil cores (% AR)						
	acetochlor	Unknown A	*tert*-oxanilic acid (2)	*tert*-sulfonic acid (7)	*tert*-sulfinylacetic acid (3)	Unextracted	Total
120 [b]	1.39	1.11	0.15	0.16	0.12	9.7	15.8
365 [b]	0.65	0.47	0.09	0.04	0.05	8	10.5

[a] Day 1 figures are based on mean of results from five replicate 0–2.5 cm soil cores

[b] Figures presented are the mean of duplicate analyses

ND = Not Detected

Total = Total recovery of applied radioactivity

Table 36 Summary of ^{14}C levels in various crops and its distribution

Crop	Sample	TRR at each plant back interval (mg equiv/kg)		
		30 days	120 days	365 days
Turnip	Root	0.111	NP	0.011
	Top	0.403	NP	0.064
Mustard	Plant	0.31	0.052	0.052
Millet	Immature plant	0.235	NP	0.083
	Grain	0.192	NP	0.012
	Straw	0.374	NP	0.038
Soya bean	Immature	0.414	NP	0.164
	bean	0.08	NP	0.036
Radish	Root	NP	0.007	NP
	Top	NP	0.034	NP
Wheat	Immature plant	NP	0.054	NP
	Grain	NP	0.013	NP
	Straw	NP	0.053	NP

NP = Not planted

Table 37 Characterisation and identification of ^{14}C residues in 30–65 day rotated crops

Rotated			Metabolite code									
Crop	RAC	acetochlor	11	16	10	2	17	7	45	68	Unknown [a]	Baseline
30 DAA												
Turnip	Root	0.008 7.5%	ND	0.002 1.6%	ND	ND	ND	ND	ND	ND	0.023 (19; < 0.006)	0.002
Turnip	Tops	ND	ND	0.013 3.2%	0.013 3.2%	0.025 6.1%	ND	ND	ND	0.006 1.6%	0.18 (39; < 0.016)	0.007
Mustard	Plant	ND	0.008 2.5%	0.006 2.0%	0.008 2.6%	0.022 7.2%	ND	0.015 4.8%	ND	0.018 5.70%	0.12 (49; < 0.015)	0.007
Millet	Immature	ND	0.003 1.1%	0.002 1.0%	0.007 2.8%	ND	ND	ND	0.004 1.9%	0.007 2.9%	0.049 (21; < 0.007)	0.004
Millet	Straw	ND	0.004 1.2%	ND	0.003 0.9%	ND	ND	ND	0.003 0.8%	0.005 1.50%	0.08 (38; < 0.008)	0.006
Millet	Grain	ND	ND	ND	0.004 1.9%	ND	ND	ND	0.002 1.1%	0.007 3.4%	0.019 (11; < 0.003)	0.002
Soya bean	Immature	ND	0.003 0.8%	0.012 2.8%	0.013 3.1%	0.025 6.1%	0.003 0.7%	ND	ND	0.008 2.0%	0.131 (34; < 0.018)	0.002
Soya bean	Beans	–	–	–	–	–	–	–	–	–	–	–
120 DAA												
Wheat	Immature	ND	ND	ND	ND	ND	ND	ND	ND	0.002 3.9%	0.009 (5; < 0.003)	0
Wheat	Straw	–	–	–	–	–	–	–	–	–	–	–
365 DAA												
Turnip	Tops	ND	ND	0.001 1.9%	0.002 3.3%	ND	ND	ND	ND	0.001 1.1%	0.017 (10; < 0.003)	0.001
Mustard	Plant	ND	0.001 2.3%	< 0.001 1.8%	0.002 4.3%	0.002 4.6%	ND	0.003 5.0%	ND	0.001 2.5%	0.014 (18; < 0.003)	< 0.001
Millet	Immature	ND	ND	ND	0.007 8.7%	ND	ND	ND	ND	0.001 1.6%	0.01 (3; < 0.004)	< 0.001
Millet	Straw	–	–	–	–	–	–	–	–	–	–	–

Rotated			Metabolite code									
Crop	RAC	acetochlor	11	16	10	2	17	7	45	68	Unknown [a]	Baseline
Soya bean	Immature	ND	ND	0.003 1.7%	0.005 2.9%	0.014 8.5%	ND	ND	ND	0.004 2.3%	0.037 (14; < 0.007)	0.001

[a] Figures in parentheses indicate the number of unknowns and the magnitude of the largest unknown

2 = *tert*-oxanilic acid

7 = *tert*-sulfonic acid

10 = *sec*-methylsulfone

11 = *sec*-hydroxyacetochlor

16 = *tert*-methylsulfone

17 = *tert*-hydroxyacetochlor

45 = hydroxymethyl *sec*-amide methyl sulfone

68 = 5-hydroxy *sec*-oxanilic acid

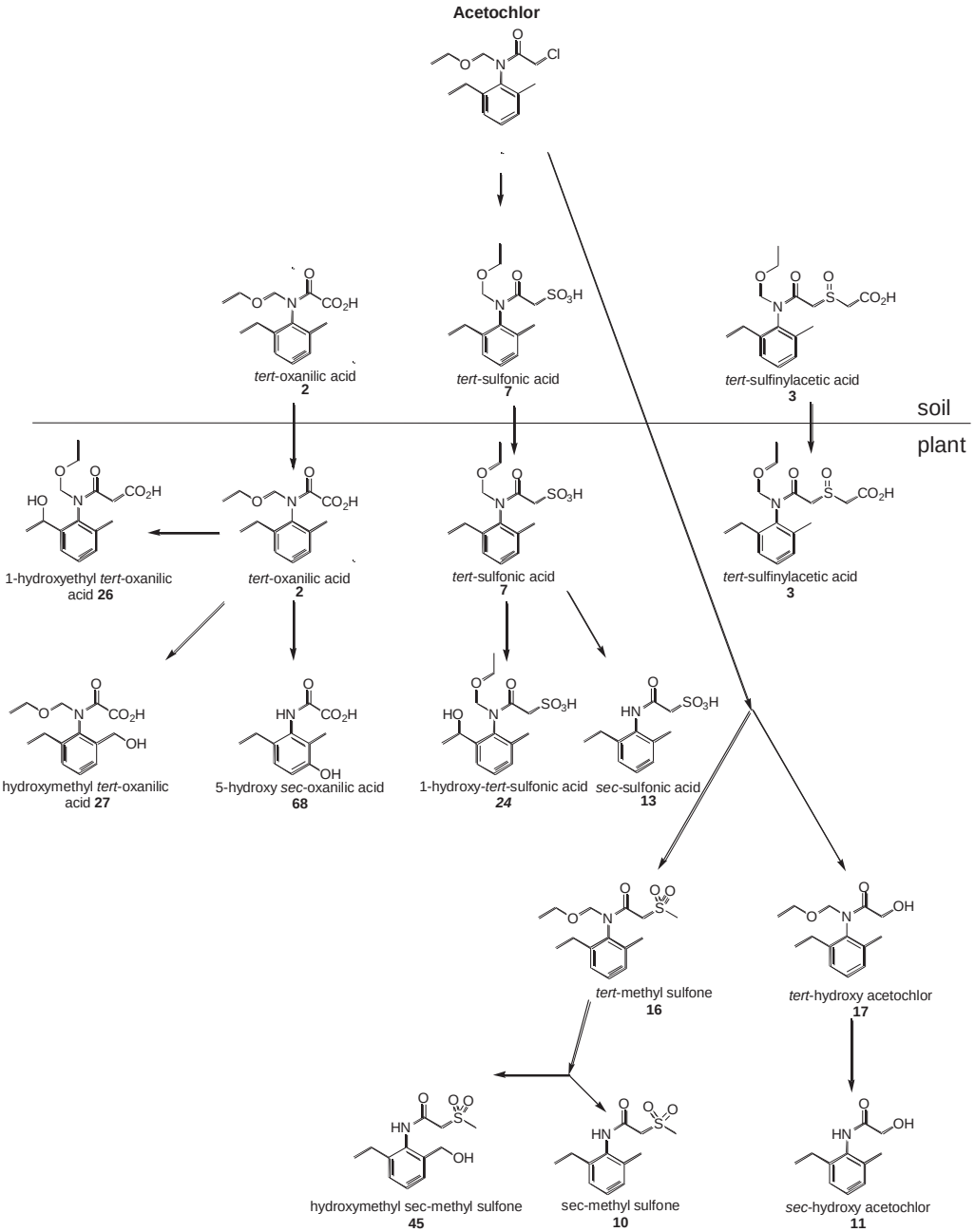


Figure 15 Proposed biotransformation pathway of acetochlor in rotated crops and soil

Field Crop Rotational Studies

A number of field crop rotational studies were made available to the meeting. From the confined rotational crop studies, low levels of residues are expected in rotational crops.

Anderson *et al*. (1998 RJ2543B, 1998 RJ2567B) studied residue levels in potatoes planted as a follow crop to maize. A potato crop rotation residue study was carried out at 10 trial locations in the USA. Magnitude of acetochlor and HEMA and EMA class residues were determined in potatoes planted in fields that had previously contained maize treated pre-emergence or pre-plant, with acetochlor (EC formulation) at a rate of 3.36 kg ai/ha. At one site, maize was treated at an exaggerated rate of 16.8 kg ai/ha pre-emergence. Residues of acetochlor and its metabolites in the rotational crop (potatoes) planted 291 to 380 DAA were < 0.01 mg/kg for acetochlor and < 0.02 mg/kg acetochlor equivalents for its metabolites HEMA and EMA. Because no detectable residues of acetochlor or HEMA and EMA class metabolites were found in any of the unprocessed tubers analysed, none of the processed fractions were analysed.

Table 38 Residues of acetochlor in potato follow crops (Anderson *et al*. (1998 RJ2543B, 1998 RJ2567B). HEMA and EMA residues are expressed in acetochlor equivalents.

Location, year, variety	Primary	crop		Follow	crop	Residue (mg/kg)			
POTATOES	Application	Rate (kg ai/ha)	Planted DAA	Sample	Harvest DAA	acetochlor	HEMA	EMA	Total
Lyons, New York, USA, 1996 Chieftan (red)	Seed	3.36	352	Tuber	452	< 0.01 < 0.01	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04
Whitakers, North Carolina, USA, 1996 Red Pontiac	Seed	3.36	334	Tuber	427	< 0.01 < 0.01	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04
Goldsboro, North Carolina, USA, 1996 Kennebec	Pre-emergence	3.36	291	Tuber	414	< 0.01 < 0.01	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04
Delavan, Wisconsin, USA, 1996 Superior	Pre-emergence	3.36	359	Tuber	448	< 0.01 < 0.01	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04
Cory, Colorado, USA, 1996 Centennial	Pre-plant	3.36	371	Tuber	524	< 0.01 < 0.01	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04
Visalia, California, USA, 1996 Chipper FL1625	Pre-emergence	3.36	362	Tuber	419	< 0.01 < 0.01	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04
Minidoka, Idaho, USA, 1996 Russet Burbank	Pre-emergence	3.36	333	Tuber	462	< 0.01 < 0.01	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04
Jerome, Idaho, USA, 1996 Russet Burbank	Pre-emergence	3.36	342	Tuber	468	< 0.01 < 0.01	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04
Hermiston, Oregon, USA, 1996 Russet Burbank	Pre-plant	3.36	357	Tuber	530	< 0.01 < 0.01	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04
Ephrata, Washington, USA, 1996 Russet Burbank	Seed	3.36	380	Tuber	509	< 0.01 < 0.01	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04
Ephrata, Washington, USA, 1996 Russet Burbank [a]	Pre-emergence	16.8	380	Tuber RAC Tuber PP	509	< 0.01 < 0.01	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04

[a] Anderson *et al.* 1998 RJ2567B

Method RAM 280/02 used for HEMA and EMA analyses

Total = EMA + HEMA

Planted DAA = follow crop planting/sowing days after application to primary maize crop
Harvest DAA = follow crop harvest days after application to primary maize crop

Anderson *et al.* (1998 RJ2560B, 1998 RJ2568B) studied residue levels in sunflowers planted as a follow crop to maize. A sunflower crop rotation residue study was carried out at nine

trial locations in the USA. Magnitude of acetochlor and HEMA and EMA residues were determined in sunflowers planted in fields that had previously contained maize treated pre-emergence or pre-plant with acetochlor (EC formulation) at a rate of 3.36 kg ai/ha. At one site, an exaggerated rate of 16.8 kg ai/ha pre-emergence was used to obtain samples for a processing study discussed later. Residues of acetochlor and its metabolites in the rotational crop (sunflowers) planted 350 to 3840 DAA were < LOQ in plots treated at 3.36 kg ai/ha (< 0.01 mg/kg for acetochlor and < 0.02 mg/kg acetochlor equivalents for its metabolites HEMA and EMA).

HEMA and EMA residues in sunflower seed harvested from sunflowers planted 338 DAT from the plot receiving the exaggerated application were 0.13–0.17 mg/kg for HEMA and 0.03 mg/kg for EMA, both expressed in acetochlor equivalents.

Table 39 Residues of acetochlor in sunflower follow crops (Anderson *et al.* 1998 RJ2560B, 1998 RJ2568B). HEMA and EMA residues are expressed in acetochlor equivalents.

Location, year, variety	Primary	crop		Follow	crop	Residue (mg/kg)			
SUNFLOWERS	Application	Rate (kg ai/ha)	Planted DAA		Harvest DAA	acetochlor	HEMA	EMA	Total
Brownton, Minnesota, USA, 1996 IS 7000	Pre-emergence	3.36	350	Seed	492	< 0.01 < 0.01	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04
Grove City, Minnesota, USA, 1996 IS 7000 Payco	Pre-emergence	3.36	360	Seed	481	< 0.01 < 0.01	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04
Doran, Minnesota, USA, 1996 IS 7000 Payco	Pre-emergence	3.36	363	Seed	499	< 0.01 < 0.01	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04
Washburn, North Dakota, USA, 1996 Pioneer 6340	seed	3.36	380	Seed	505	< 0.01 < 0.01	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04
Kulon, North Dakota, USA, 1996 Mycogen 98338	seed	3.36	371	Seed	500	< 0.01 < 0.01	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04
Mansfield, South Dakota, USA, 1996 DK3868	seed	3.36	372	Seed	503	< 0.01 < 0.01	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04
Madrid, Nebraska, USA, 1996 Triumph 546	Pre-plant	3.36	373	Seed	495	< 0.01 < 0.01	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04
Eaton, Colorado, USA, 1996 Triumph	Pre-emergence	3.36	384	Seed	519	< 0.01 < 0.01	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04
Lake Preston, South Dakota, USA, 1996 Legend LSF146 [a]	Pre-emergence	16.8	384	seed RAC seed PP	509	< 0.01 < 0.01	□□□□ □□□□	□□□□ □□□□	0.20 0.16

[a] Anderson *et al.* 1998 RJ2568B

Method RAM 280/02 used for HEMA and EMA analyses

Total = EMA + HEMA

Planted DAA = follow crop planting/sowing days after application to primary maize crop

Harvest DAA = follow crop harvest days after application to primary maize crop

Manning (1997 MSL-14117, 1997 MSL-14118) studied residues in oats grown as a follow crop to maize. This study determined residues of the HEMA and EMA classes of acetochlor metabolites in oats planted the season following pre-emergence or pre-plant incorporated treatment of sweet corn or maize with acetochlor (formulated as, an emulsifiable concentrate) at 17 sites in the USA. Magnitude of HEMA and EMA residues were determined in oats planted in fields that had previously contained maize treated pre-emergence or pre-plant with acetochlor (EC formulation) at a nominal rate of 3.36 kg ai/ha. At two sites, an exaggerated

rate of approximately 16.8 kg ai/ha pre-emergence was used to obtain samples for a processing study discussed later.

Total acetochlor residues (HEMA + EMA) in grain of crops planted 285–388 days after application to maize were < LOQ (0.035 mg/kg) in samples from all seventeen sites. The individual total acetochlor residues in the forage ranged from not detected to 0.126 mg/kg, in the hay from not detected to 0.196 mg/kg, and in the straw from not detected to 0.283 mg/kg.

Table 40 Residues of acetochlor in oat follow crops (Manning 1997 MSL-14117, 1997 MSL-14118). HEMA and EMA residues are expressed in acetochlor equivalents.

Location, year, variety	Primary	Crop		Follow crop			HEMA	EMA	Total
OATS	Application	Rate (kg ai/ha)	Planted DAA	GS	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)
Grain									
Ault, Colorado,	Pre-plant	3.36	351	Normal	Grain	456	< 0.018 [c]	< 0.017	
USA, 1995 Don	incorporated			harvest			< 0.018	< 0.017	< 0.035
Bondville,	Pre-plant	3.36	310	Normal	Grain	415	< 0.018	< 0.017	
Illinois, USA, 1995 Prairie	incorporated			harvest			< 0.018	< 0.017	< 0.035
Hamburg,	Pre-	3.46	325	Normal	Grain	439	< 0.018	< 0.017	
Pennsylvania, USA, 1995 Hercules	emergence			harvest			< 0.018	< 0.017	< 0.035
Hebron,	Pre-plant	3.72	310	Normal	Grain	429	< 0.018	< 0.017	
Maryland, USA, 1995 Southern States/Ogle	incorporated			harvest			< 0.018	< 0.017	< 0.035
Janesville,	Pre-plant	3.36	341	Normal	Grain	439	< 0.018	< 0.017	
Wisconsin, USA 1995, Certified Prairie Oats	incorporated			harvest			< 0.018	< 0.017	< 0.035
Jerseyville,	Pre-plant	3.36	285	Normal	Grain	414	< 0.018	< 0.017	
Illinois, USA, 1995 Ogle	incorporated			harvest			< 0.018	< 0.017	< 0.035
Lockbourne,	Pre-plant	3.36	312	Normal	Grain	416	< 0.018	< 0.017	
Ohio, USA, 1995 Armour	incorporated			harvest			< 0.018	< 0.017	< 0.035
Mankato,	Pre-plant	3.19	360	Normal	Grain	463	< 0.018	< 0.017	
Minnesota, USA, 1995 Troy	incorporated			harvest			< 0.018	< 0.017	< 0.035
Miller, South	Pre-plant	3.25	331	Normal	Grain	438	< 0.018	< 0.017	
Dakota, USA, 1995 Troy oats	incorporated			harvest			< 0.018	< 0.017	< 0.035
Monmouth,	Pre-	3.44	299	Normal	Grain	425-	< 0.018	< 0.017	
Illinois, USA, 1995 Ogle	emergence			harvest		426	< 0.018	< 0.017	< 0.035
New Rockford,	Pre-plant	3.21	388	Normal	Grain	471	< 0.018	0.018	
North Dakota, USA, 1995 Jerry oats	incorporated			harvest			< 0.018	< 0.017	< 0.036
Northwood,	Pre-plant	3.37	349	Normal	Grain	444	< 0.018	< 0.017	
North Dakota, USA, 1995 Jerry	incorporated			harvest			< 0.018	< 0.017	< 0.035
Spink, South	Pre-	3.37	340	Normal	Grain	454	< 0.018	< 0.017	
Dakota, USA, 1995 Ogle	emergence			harvest			< 0.018	< 0.017	< 0.035
Uvalde, Texas,	Pre-plant	3.35	292	Normal	Grain	417	< 0.018	< 0.017	
USA, 1995 Coronado	incorporated			harvest			< 0.018	< 0.017	< 0.035
Waukee, Iowa,	Pre-plant	3.63	319	Normal	Grain	437	< 0.018	< 0.017	
USA, 1995 Starter	incorporated			harvest			< 0.018	< 0.017	< 0.035

Location, year, variety	Primary	Crop		Follow crop			HEMA	EMA	Total
OATS	Application	Rate (kg ai/ha)	Planted DAA	GS	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)
West Lafayette,	Pre-plant	3.50	306	Normal	Grain	417	< 0.018	< 0.017	
Indiana, USA, 1995 Ogle	incorporated			harvest			< 0.018	< 0.017	< 0.035
Whitakers,	Pre-plant	3.33	324	Normal	Grain	426	< 0.018	< 0.017	
North Carolina, USA, 1995 Prairie	incorporated			harvest			< 0.018	< 0.017	< 0.035
Monmouth, Illinois, USA, 1996	Pre-emergence	17.1	299	Normal harvest	Grain [b] Grain [a]	425	< 0.018 0.022	0.034 0.033	< 0.054
Jerseyville, Illinois, USA, 1996	Pre-plant incorporated	16.1	285	Normal harvest	Grain [b] Grain [a]	414	< 0.018 < 0.018	< 0.017 < 0.017	< 0.035
Forage									
Ault, Colorado,	Pre-plant	3.36	351	Tillering to	Forage	393	0.022	0.047	
USA, 1995 Don	incorporated			joint			< 0.018	0.039	0.063
Bondville,	Pre-plant	3.36	310	Tillering to	Forage	360	0.024	0.036	
Illinois, USA, 1995 Prairie	incorporated			joint			0.022	0.032	0.057
Hamburg,	Pre-	3.46	325	Tillering to	Forage	380	0.025	0.058	
Pennsylvania, USA, 1995 Hercules	emergence			joint			0.024	0.063	0.085
Hebron,	Pre-plant	3.72	310	Tillering to	Forage	367	< 0.018	< 0.017	
Maryland, USA, 1995 Southern States/Ogle	incorporated			joint			< 0.018	< 0.017	< 0.035
Janesville,	Pre-plant	3.36	341	Tillering to	Forage	387	< 0.018	< 0.017	
Wisconsin, USA 1995, Certified Prairie Oats	incorporated			joint			< 0.018	< 0.017	< 0.035
Jerseyville,	Pre-plant	3.36	285	Tillering to	Forage	351	0.061	0.056	
Illinois, USA, 1995 Ogle	incorporated			joint			0.074	0.052	0.121
Lockbourne,	Pre-plant	3.36	312	Tillering to	Forage	360	< 0.018	< 0.017	
Ohio, USA, 1995 Armour	incorporated			joint			< 0.018	< 0.017	< 0.035
Mankato,	Pre-plant	3.19	360	Tillering to	Forage	410	< 0.018	0.017	
Minnesota, USA, 1995 Troy	incorporated			joint			< 0.018	< 0.017	< 0.035
Miller, South	Pre-plant	3.25	331	Tillering to	Forage	373	0.030	0.037	
Dakota, USA, 1995 Troy oats	incorporated			joint			0.030	0.034	0.066
Monmouth,	Pre-	3.44	299	Tillering to	Forage	364	< 0.018	< 0.017	
Illinois, USA, 1995 Ogle	emergence			joint			< 0.018	< 0.017	< 0.035
New Rockford,	Pre-plant	3.21	388	Tillering to	Forage	419	< 0.018	0.039	
North Dakota, USA, 1995 Jerry oats	incorporated			joint			< 0.018	0.036	< 0.056
Northwood,	Pre-plant	3.37	349	Tillering to	Forage	388	< 0.018	0.024	
North Dakota, USA, 1995 Jerry	incorporated			joint			< 0.018	0.025	< 0.042
Spink, South	Pre-	3.37	340	Tillering to	Forage	381	< 0.018	0.020	
Dakota, USA, 1995 Ogle	emergence			joint			< 0.018	0.019	< 0.038
Uvalde, Texas,	Pre-plant	3.35	292	Tillering to	Forage	364	< 0.018	< 0.017	
USA, 1995 Coronado	incorporated			joint			< 0.018	< 0.017	< 0.035
Waukee, Iowa,	Pre-plant	3.63	319	Tillering to	Forage	378	< 0.018	< 0.017	
USA, 1995 Starter	incorporated			joint			< 0.018	< 0.017	< 0.035
West Lafayette,	Pre-plant	3.50	306	Tillering to	Forage	367	0.022	0.024	
Indiana, USA, 1995	incorporated			joint			0.022	0.027	0.048

Location, year, variety	Primary	Crop		Follow crop			HEMA	EMA	Total
OATS	Application	Rate (kg ai/ha)	Planted DAA	GS	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)
Ogle									
Whitakers,	Pre-plant	3.33	324	Tillering to	Forage	373	0.022	0.018	
North Carolina, USA, 1995 Prairie	incorporated			joint			< 0.018	< 0.017	< 0.038
Hay									
Ault, Colorado,	Pre-plant	3.36	351	Early flower	Hay	405	0.044	0.035	
USA, 1995 Don	incorporated			to soft dough			0.039	0.031	0.074
Bondville,	Pre-plant	3.36	310	Early flower	Hay	391	< 0.018	0.018	
Illinois, USA, 1995 Prairie	incorporated			to soft dough			< 0.018	0.018	< 0.036
Hamburg,	Pre-	3.46	325	Early flower	Hay	410	0.028	0.064	
Pennsylvania, USA, 1995 Hercules	emergence			to soft dough			0.028	0.062	0.091
Hebron,	Pre-plant	3.72	310	Early flower	Hay	389	< 0.018	< 0.017	
Maryland, USA, 1995 Southern States/Ogle	incorporated			to soft dough			< 0.018	< 0.017	< 0.035
Janesville,	Pre-plant	3.36	341	Early flower	Hay	401	< 0.018	< 0.017	
Wisconsin, USA 1995, Certified Prairie Oats	incorporated			to soft dough			< 0.018	< 0.017	< 0.035
Lockbourne,	Pre-plant	3.36	312	Early flower	Hay	401	0.031	0.109	
Ohio, USA, 1995 Armour	incorporated			to soft dough			< 0.018	0.039	< 0.098
Mankato,	Pre-plant	3.19	360	Early flower	Hay	437	< 0.018	< 0.017	
Minnesota, USA, 1995 Troy	incorporated			to soft dough			< 0.018	< 0.017	< 0.035
Miller, South	Pre-plant	3.25	331	Early flower	Hay	414	0.052	0.029	
Dakota, USA, 1995 Troy oats	incorporated			to soft dough			0.032	0.023	0.068
Monmouth,	Pre-	3.44	299	Early flower	Hay	390	< 0.018	< 0.017	
Illinois, USA, 1995 Ogle	emergence			to soft dough			< 0.018	< 0.017	< 0.035
New Rockford,	Pre-plant	3.21	388	Early flower	Hay	447	0.058	0.138	
North Dakota, USA, 1995 Jerry oats	incorporated			to soft dough			0.034	0.081	0.156
Northwood,	Pre-plant	3.37	349	Early flower	Hay	420	0.020	0.025	
North Dakota, USA, 1995 Jerry	incorporated			to soft dough			< 0.018	0.020	< 0.042
Spink, South	Pre-	3.37	340	Early flower	Hay	430	0.018	0.043	
Dakota, USA, 1995 Ogle	emergence			to soft dough			< 0.018	0.040	< 0.060
Uvalde, Texas,	Pre-plant	3.35	292	Early flower	Hay	401	< 0.018	< 0.017	
USA, 1995	incorporated			to soft			< 0.018	< 0.017	< 0.035

Location, year, variety	Primary	Crop		Follow crop			HEMA	EMA	Total
OATS	Application	Rate (kg ai/ha)	Planted DAA	GS	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)
Coronado				dough					
Waukee, Iowa,	Pre-plant	3.63	319	Early flower	Hay	393	< 0.018	< 0.017	
USA, 1995 Starter	incorporated			to soft dough			< 0.018	< 0.017	< 0.035
West Lafayette,	Pre-plant	3.50	306	Early flower	Hay	388	< 0.018	0.018	
Indiana, USA, 1995 Ogle	incorporated			to soft dough			< 0.018	< 0.017	< 0.036
Whitakers,	Pre-plant	3.33	324	Early flower	Hay	402	0.021	0.028	
North Carolina, USA, 1995 Prairie	incorporated			to soft dough			0.018	< 0.017	0.042
Straw									
Ault, Colorado,	Pre-plant	3.36	351	Dried	Straw	456	0.023	0.019	
USA, 1995 Don	incorporated			stalks/stems			0.024	0.021	0.044
Bondville,	Pre-plant	3.36	310	Dried	Straw	415	< 0.018	< 0.017	
Illinois, USA, 1995 Prairie	incorporated			stalks/stems			< 0.018	< 0.017	< 0.035
Hamburg,	Pre-	3.46	325	Dried	Straw	439	< 0.018	0.018	
Pennsylvania, USA, 1995 Hercules	emergence			stalks/stems			< 0.018	< 0.017	< 0.036
Hebron,	Pre-plant	3.72	310	Dried	Straw	429	< 0.018	< 0.017	
Maryland, USA, 1995 Southern States/Ogle	incorporated			stalks/stems			< 0.018	< 0.017	< 0.035
Janesville,	Pre-plant	3.36	341	Dried	Straw	439	< 0.018	< 0.017	
Wisconsin, USA 1995, Certified Prairie Oats	incorporated			stalks/stems			< 0.018	< 0.017	< 0.035
Jerseyville,	Pre-plant	3.36	285	Dried	Straw	414	< 0.018	< 0.017	
Illinois, USA, 1995 Ogle	incorporated			stalks/stems			< 0.018	< 0.017	< 0.035
Lockbourne,	Pre-plant	3.36	312	Dried	Straw	416	< 0.018	< 0.017	
Ohio, USA, 1995 Armour	incorporated			stalks/stems			< 0.018	0.019	< 0.036
Mankato,	Pre-plant	3.19	360	Dried	Straw	463	< 0.018	< 0.017	
Minnesota, USA, 1995 Troy	incorporated			stalks/stems			< 0.018	< 0.017	< 0.035
Miller, South	Pre-plant	3.25	331	Dried	Straw	438	< 0.018	< 0.017	
Dakota, USA, 1995 Troy oats	incorporated			stalks/stems			< 0.018	< 0.017	< 0.035
Monmouth,	Pre-	3.44	299	Dried	Straw	425	< 0.018	< 0.017	
Illinois, USA, 1995 Ogle	emergence			stalks/stems			< 0.018	< 0.017	< 0.035
New Rockford,	Pre-plant	3.21	388	Dried	Straw	471	0.056	0.226	
North Dakota, USA, 1995 Jerry oats	incorporated			stalks/stems			0.048	0.179	0.254
Northwood,	Pre-plant	3.37	349	Dried	Straw	444	< 0.018	0.046	
North Dakota, USA, 1995 Jerry	incorporated			stalks/stems			< 0.018	0.059	< 0.070
Spink, South	Pre-	3.37	340	Dried	Straw	454	< 0.018	< 0.017	
Dakota, USA, 1995 Ogle	emergence			stalks/stems			< 0.018	< 0.017	< 0.035
Uvalde, Texas,	Pre-plant	3.35	292	Dried	Straw	417	< 0.018	< 0.017	
USA, 1995 Coronado	incorporated			stalks/stems			< 0.018	< 0.017	< 0.035
Waukee, Iowa,	Pre-plant	3.63	319	Dried	Straw	437	< 0.018	< 0.017	

Location, year, variety	Primary	Crop		Follow crop			HEMA	EMA	Total
OATS	Application	Rate (kg ai/ha)	Planted DAA	GS	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)
USA, 1995 Starter	incorporated			stalks/stems			< 0.018	< 0.017	< 0.035
West Lafayette,	Pre-plant	3.50	306	Dried	Straw	417	< 0.018	< 0.017	
Indiana, USA, 1995 Ogle	incorporated			stalks/stems			< 0.018	< 0.017	< 0.035
Whitakers,	Pre-plant	3.33	324	Dried	Straw	426	0.019	0.025	
North Carolina, USA, 1995 Prairie	incorporated			stalks/stems			0.020	0.025	0.044

Total = EMA + HEMA

[a] = Grain sampled at processing facility

[b]=Grain before sending to processing facility

Planted DAA = follow crop planting/sowing days after application to primary maize crop

Harvest DAA = follow crop harvest days after application to primary maize crop

Schneider and Schneider (1996 MSL-14276) studied residues in alfalfa and clover grown as a follow crop to field corn. Acetochlor was applied to the primary crop (maize) at 3.2 to 3.7 kg ai/ha as a post-emergent application when the maize was 13–20 cm tall. Alfalfa and clover were sown after harvest of the maize (sowing 55–355 DAA for alfalfa and 130–358 DAA for clover).

Acetochlor residues were detected in all alfalfa and clover raw agricultural commodities (RACs). Total residue levels (HEMA + EMA) in alfalfa hay and the majority of the clover hay samples were higher than the residues in the corresponding forage samples. The highest alfalfa and clover RAC residues occurred in a first cutting of hay, and residue levels in both RACs for the rotational crops tended to decline in subsequent cuttings. Maximum acetochlor residues for alfalfa forage and hay were 0.540 and 1.870 mg/kg, respectively, and the maximum residues for clover forage and hay were 0.567 and 1.244 mg/kg, respectively. Method LOQs were 0.014 mg/kg for HEMA and 0.012 mg/kg for EMA.

Table 41 Residues of acetochlor in forage from alfalfa and clover follow crops, Schneider and Schneider (1996 MSL-14276). HEMA and EMA residues are expressed in acetochlor equivalents.

Location, year, variety	Primary crop		Follow crop			HEMA	EMA	Total
ALFALFA/CLOVER	Rate (kg ai/ha)	PlantedDAA	GS	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)
Alfalfa								
Alta, Wyoming, USA, 1994 Alfalfa arrow	3.43	312	Normal harvest	Forage (1st cutting)	377	0.19	0.28	0.47
				Forage (2nd cutting)	429	0.12	0.17	0.29
Ault, Colorado, USA, 1994 Alfalfa rough-rider	3.26	300	Normal harvest	Forage (1st cutting)	414	0.16	0.38	0.54
				Forage (2nd cutting)	482	0.03	0.08	0.11
Bagley, Iowa, USA, 1994 Alfalfa Wensman	3.31	83	Normal harvest	Forage (1st cutting)	377	0.05	0.14	0.19
				Forage (2nd cutting)	501	< 0.01	0.01	< 0.02
Cunningham, Kansas, USA, 1994 Alfalfa	3.26	291		Forage (1st cutting)	371	0.10	0.25	0.35

Location, year, variety	Primary crop		Follow crop			HEMA	EMA	Total
ALFALFA/CLOVER	Rate (kg ai/ha)	PlantedDAA	GS	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)
Good as Gold				Forage (2nd cutting)	400	0.05	0.09	0.14
				Forage (3rd cutting)	423	0.06	0.10	0.16
Dayton, Idaho, USA, 1994 Alfalfa Magnum	3.44	336		Forage (1st cutting)	405	0.03	0.05	0.08
IV				Forage (2nd cutting)	447	< 0.01	0.03	0.04
Germansville, Pennsylvania, USA,	3.40	59		Forage (1st cutting)	336	< 0.01	0.04	< 0.05
1994 Alfalfa WL322HQ				Forage (2nd cutting)	386	< 0.01	0.03	< 0.04
				Forage (3rd cutting)	427	< 0.01	0.03	< 0.04
Lesterville, South Dakota, USA, 1994	3.33	355		Forage (1st cutting)	428	0.03	0.10	0.14
Alfalfa Absolute Brand				Forage (2nd cutting)	469	0.02	0.05	0.07
				Forage (3rd cutting)	516	0.02	0.06	0.08
Monmouth, Illinois, USA, 1994 Alfalfa	3.24	84		Forage (1st cutting)	363	0.04	0.47	0.51
Absolute Brand				Forage (2nd cutting)	400	0.02	0.06	0.08
				Forage (3rd cutting)	465	< 0.01	0.018	< 0.028
Northwood, North Dakota, USA, 1994	3.41	355		Forage (1st cutting)	388	0.04	0.16	0.20
Alfalfa Vernal				Forage (2nd cutting)	447	< 0.01	0.03	< 0.04
Waterloo, New York, USA, 1994 Alfalfa Edge	3.42	55		Forage (1st cutting)	344	0.05	0.24	0.29
				Forage (2nd cutting)	380	0.02	0.06	0.08
				Forage (3rd cutting)	427	< 0.01	0.04	< 0.05
York, Nebraska, USA, 1994 Alfalfa Leaf	3.24	327		Forage (1st cutting)	397	0.02	0.07	0.09
				Forage (2nd cutting)	428	0.03	0.05	0.08
				Forage (3rd cutting)	458	0.03	0.03	0.06
Clover								
Brookshire, Texas, USA, 1994 Clover Yuci Arrowleaf	3.46	130	10–20 cm. to pre-bloom	Forage (1st cutting)	306	0.02	0.15	0.17
			10–20 cm to pre-bloom	Forage (2nd cutting)	364	0.04	0.22	0.26
Conklin, Michigan, USA,	3.38	313	10–	Forage	386	0.02	0.09	0.11

Location, year, variety	Primary crop		Follow crop			HEMA	EMA	Total
ALFALFA/CLOVER	Rate (kg ai/ha)	PlantedDAA	GS	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)
1994 Clover medium red			20 cm. to pre-bloom	(1st cutting)				
			10–20 cm to pre-bloom	Forage (2nd cutting)	418	0.02	0.04	0.06
			10–20 cm to pre-bloom	Forage (3rd cutting)	469	< 0.01	0.03	< 0.04
Cunningham, Kansas, USA, 1994 Clover Kenland red	3.21	291	10–20 cm to pre-bloom	Forage (1st cutting)	371	0.05	0.30	0.35
Delavan, Wisconsin, USA, 1994 Clover	3.24	327	10–20 cm to pre-bloom	Forage (1st cutting)	378	0.03	0.15	0.17
Northup King Atlas			10–20 cm to pre-bloom	Forage (2nd cutting)	417	0.02	0.08	0.10
			10–20 cm to pre-bloom	Forage (3rd cutting)	459	< 0.01	0.04	< 0.05
La Center, Kentucky, USA, 1994 Clover Crimson	3.36	330	10–20 cm to pre-bloom	Forage (1st cutting)	412	< 0.01	0.03	< 0.03
Leonard, Missouri, USA, 1994 Clover	3.67	274	10–20 cm to pre-bloom	Forage (1st cutting)	357	0.02	0.08	0.10
Medium red			10–20 cm to pre-bloom	Forage (2nd cutting)	426	< 0.01	0.04	< 0.05
			10–20 cm to pre-bloom	Forage (3rd cutting)	497	< 0.01	0.03	< 0.04
Lesterville, South Dakota, USA, 1994	3.47	355	10–20 cm to pre-bloom	Forage (1st cutting)	407	0.03	0.07	0.10
Clover VNS			10–20 cm to pre-bloom	Forage (2nd cutting)	462	0.02	0.02	0.04
Northwood, North Dakota, USA, 1994	3.41	331	10–20 cm to pre-bloom	Forage (1st cutting)	386	0.09	0.48	0.57
Clover Arlington			10–20 cm to pre-bloom	Forage (2nd cutting)	434	0.03	0.13	0.16
York, Nebraska, USA,	3.18	327	10–	Forage	399	0.02	0.15	0.17

Location, year, variety	Primary crop		Follow crop			HEMA	EMA	Total
ALFALFA/CLOVER	Rate (kg ai/ha)	PlantedDAA	GS	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)
1994 Clover medium red (VNS)			20 cm to pre-bloom	(1st cutting)				
			10–20 cm to pre-bloom	Forage (2nd cutting)	427	0.02	0.08	0.10
			10–20 cm to pre-bloom	Forage (3rd cutting)	458	0.012	0.04	0.05

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Total = EMA + HEMA

Planted DAA = follow crop planting/sowing days after application to primary maize crop

Harvest DAA = follow crop harvest days after application to primary maize crop

Table 42 Residues of acetochlor in hay from alfalfa and clover follow crops, Schneider and Schneider (1996 MSL-14276). HEMA and EMA residues are expressed in acetochlor equivalents.

Location, year, variety	Primary crop		Follow crop			HEMA	EMA	Total
ALFALFA/CLOVER	Rate (kg ai/ha)	PlantedDAA	GS	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)
Alfalfa								
Alta, Wyoming, USA, 1994 Alfalfa arrow	3.43	312	Normal harvest	Hay (1st cutting)	382	0.48	0.49	0.97
				Hay (2nd cutting)	434	0.35	0.38	0.73
Ault, Colorado, USA, 1994 Alfalfa rough-rider	3.26	300	Normal harvest	Hay (1st cutting)	415	0.54	1.33	1.87
				Hay (2nd cutting)	490	0.07	0.12	0.19
Bagley, Iowa, USA, 1994 Alfalfa Wensman	3.31	83	Normal harvest	Hay (1st cutting)	380	0.11	0.28	0.39
				Hay (2nd cutting)	505	0.02	0.04	0.06
Cunningham, Kansas, USA, 1994 Alfalfa	3.26	291		Hay (1st cutting)	373	0.21	0.61	0.82
				Hay (2nd cutting)	406	0.12	0.22	0.34
				Hay (3rd cutting)	424	0.09	0.15	0.24
Dayton, Idaho, USA, 1994 Alfalfa Magnum	3.44	336		Hay (1st cutting)	408	0.10	0.18	0.28
				Hay (2nd cutting)	451	0.04	0.12	0.16
Germansville,	3.40	59		Hay	338	0.04	0.08	0.12

Location, year, variety	Primary crop		Follow crop			HEMA	EMA	Total
ALFALFA/CLOVER	Rate (kg ai/ha)	PlantedDAA	GS	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)
Pennsylvania, USA,				(1st cutting)				
				Hay (2nd cutting)	390	0.02	0.04	0.06
				Hay (3rd cutting)	429	< 0.01	0.03	< 0.04
Lesterville, South Dakota, USA, 1994	3.33	355		Hay (1st cutting)	433	0.07	0.22	0.29
				Hay (2nd cutting)	472	0.04	0.11	0.15
Monmouth, Illinois, USA, 1994 Alfalfa	3.24	84		Hay (1st cutting)	365	0.10	1.00	1.10
				Hay (2nd cutting)	403	0.03	0.09	0.12
				Hay (3rd cutting)	468	< 0.01	0.042	< 0.052
Northwood, North Dakota, USA, 1994	3.41	355		Hay (1st cutting)	394	0.05	0.15	0.20
				Hay (2nd cutting)	450	0.03	0.08	0.11
Waterloo, New York, USA, 1994 Alfalfa Edge	3.42	55		Hay (1st cutting)	348	0.16	0.71	0.87
				Hay (2nd cutting)	382	0.05	0.18	0.23
				Hay (3rd cutting)	433	0.03	0.08	0.11
York, Nebraska, USA, 1994 Alfalfa Leaf	3.24	327		Hay (1st cutting)	400	0.12	0.21	0.33
				Hay (2nd cutting)	431	0.08	0.10	0.18
				Hay (3rd cutting)	460	0.07	0.07	0.14
Clover								
Brookshire, Texas, USA, 1994 Clover Yuci	3.46	130	10–20 cm. to pre-bloom	Hay (1st cutting)	325	0.04	0.23	0.27
			10–20 cm to pre-bloom	Hay (2nd cutting)	366	0.10	0.48	0.58
Conklin, Michigan, USA, 1994 Clover	3.38	313	10–20 cm. to pre-bloom	Hay (1st cutting)	393	0.03	0.10	0.13
			10–20 cm to pre-bloom	Hay (2nd cutting)	426	0.04	0.08	0.12
			10–20 cm	Hay	480	0.02	0.06	0.08

Location, year, variety	Primary crop		Follow crop			HEMA	EMA	Total
ALFALFA/CLOVER	Rate (kg ai/ha)	PlantedDAA	GS	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)
			to pre-bloom	(3rd cutting)				
Cunningham, Kansas, USA, 1994 Clover	3.21	291	10–20 cm to pre-bloom	Hay (1st cutting)	387	0.12	0.64	0.76
Delavan, Wisconsin, USA, 1994 Clover	3.24	327	10–20 cm to pre-bloom	Hay (1st cutting)	394	0.07	0.37	0.44
			10–20 cm to pre-bloom	Hay (2nd cutting)	437	0.02	0.06	0.08
			10–20 cm to pre-bloom	Hay (3rd cutting)	483	< 0.01	0.03	< 0.04
La Center, Kentucky, USA, 1994 Clover	3.36	330	10–20 cm to pre-bloom	Hay (1st cutting)	422	< 0.01	< 0.01	< 0.02
Leonard, Missouri, USA, 1994 Clover	3.67	274	10–20 cm to pre-bloom	Hay (1st cutting)	392	0.07	0.23	0.30
			10–20 cm to pre-bloom	Hay (2nd cutting)	449	< 0.01	< 0.01	< 0.02
Lesterville, South Dakota, USA, 1994	3.47	355	10–20 cm to pre-bloom	Hay (1st cutting)	433	0.06	0.09	0.15
			10–20 cm to pre-bloom	Hay (2nd cutting)	472	0.02	0.06	0.08
Northwood, North Dakota, USA, 1994	3.41	331	10–20 cm to pre-bloom	Hay (1st cutting)	418	0.23	1.01	1.24
			10–20 cm to pre-bloom	Hay (2nd cutting)	456	0.09	0.39	0.48
York, Nebraska, USA, 1994 Clover medium	3.18	327	10–20 cm to pre-bloom	Hay (1st cutting)	407	0.06	0.35	0.41
			10–20 cm to pre-bloom	Hay (2nd cutting)	443	0.07	0.17	0.24
			10–20 cm to pre-bloom	Hay (3rd cutting)	470	0.04	0.09	0.13

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Total = EMA + HEMA

Planted DAA = follow crop planting/sowing days after application to primary maize crop

Harvest DAA = follow crop harvest days after application to primary maize crop

Sidhu (1992 MSL-11963) studied residues in winter wheat, soya bean and sorghum crops grown as follow crops after maize crops that had been treated with acetochlor. The primary maize crop was treated with acetochlor EC formulation at a target rate of 2.2 kg ai/ha with wheat planted 90–170 days after application to maize, soya beans 253–425 days and sorghum 253–425 days. At one site an exaggerated application rate of 16.8 kg ai/ha was used to generate material for use in a processing study if needed.

Table 43 Residues of acetochlor in wheat follow crops (Sidhu 1992 MSL-11963) HEMA and EMA residues are expressed in acetochlor equivalents

Location, year, variety	Primary crop		Follow	crop	HEMA	EMA	HMEA	Total
WINTER WHEAT	rate (kg ai/ha)	Planted DAA	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)
Forage								
Colo, Iowa, USA, 1989 Siouxland HRW	3.36	154	Forage	364	0.01	0.05	< 0.01	0.06
Dacono, Colorado, USA, 1989 Hawk	3.36	161	Forage	255	0.14	0.33	0.07	0.47
Danville, Iowa, USA, 1989 Caldwell	3.36	113	Forage	169	0.02	0.09	0.01	0.11
Delavan, Wisconsin, USA, 1989 Caldwell	3.36	141	Forage	194	0.04	0.15	0.02	0.19
Devine/Hondo, Texas, USA, 1989 MIT	2.10	90	Forage	146	0.02	0.01	< 0.01	0.03
Eakly, Oklahoma, USA, 1989 Pioneer 2157	3.36	176	Forage	229	0.02	0.04	< 0.01	0.06
Elwood, Illinois, USA, 1989 Pioneer 2550	3.59	133	Forage	179	< 0.01	< 0.01	< 0.01	< 0.02
Geneseo, Illinois, USA, 1989 Caldwell	3.47	120	Forage	189	< 0.01	0.04	< 0.01	< 0.05
Hawkinsville, Georgia, USA, 1989 Coker 9766	3.36	160	Forage	271	< 0.01	< 0.01	< 0.01	< 0.02
Hollandale, Minnesota, USA, 1989 Seward	3.36	106	Forage	154	< 0.01	0.02	< 0.01	< 0.03
Sedan, Kansas, USA, 1989 Delange 7837	3.36	119	Forage	216	0.03	0.10	0.01	0.13
Leonard, Missouri, USA, Delange 7837	3.36	148	Forage	201	0.06	0.21	0.03	0.27
Lexington, Kentucky, USA, 1989 Compton	3.18	157	Forage	214	< 0.01	0.01	< 0.01	< 0.02
New Holland, Ohio, USA, 1989 Dynasty	3.36	96	Forage	156	0.09	0.32	0.04	0.41
Noblesville, Indiana, USA, 1989 Caldwell	3.36	119	Forage	175	0.04	0.14	0.02	0.18
Lucama, North Carolina, USA, 1989 Pioneer 2555	3.27	121	Forage	178	0.01	0.03	< 0.01	0.04
Sparta, Michigan, USA, 1989 Pioneer 2550	3.36	147	Forage	359	< 0.01	0.02	< 0.01	< 0.03
York, Nebraska, USA, 1989 Brule	3.36	127	Forage	188	0.02	0.12	0.01	0.14
Straw								
Colo, Iowa, USA, 1989 Siouxland HRW	3.36	154	Straw	435	< 0.01	< 0.01	< 0.01	< 0.02
Dacono, Colorado, USA, 1989 Hawk	3.36	161	Straw	446	0.02	0.07	0.01	0.09
Danville, Iowa, USA, 1989 Caldwell	3.36	113	Straw	418	0.02	0.05	< 0.01	0.07
Delavan, Wisconsin, USA, 1989 Caldwell	3.36	141	Straw	436	0.01	0.02	< 0.01	0.03
Devine/Hondo, Texas, USA, 1989 MIT	2.10	90	Straw	315	0.04	0.03	< 0.01	0.07
Eakly, Oklahoma, USA, 1989 Pioneer 2157	3.36	176	Straw	426	< 0.01	< 0.01	< 0.01	< 0.02
Elwood, Illinois, USA, 1989 Pioneer 2550	3.59	133	Straw	405	0.03	0.04	0.02	0.07
Geneseo, Illinois, USA, 1989 Caldwell	3.47	120	Straw	455	< 0.01	0.01	< 0.01	< 0.02
Hawkinsville, Georgia, USA, 1989 Coker 9766	3.36	160	Straw	380	< 0.01	0.01	< 0.01	< 0.02
Hollandale, Minnesota, USA, 1989 Seward	3.36	106	Straw	427	0.01	0.02	< 0.01	0.03

Location, year, variety	Primary crop		Follow	crop	HEMA	EMA	HMEA	Total
WINTER WHEAT	rate (kg ai/ha)	Planted DAA	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)
Sedan, Kansas, USA, 1989 Delange 7837	3.36	119	Straw	391	0.02	0.01	< 0.01	0.03
Leonard, Missouri, USA, Delange 7837	3.36	148	Straw	426	0.04	0.06	0.02	0.10
Lexington, Kentucky, USA, 1989 Compton	3.18	157	Straw	440	< 0.01	< 0.01	< 0.01	< 0.02
New Holland, Ohio, USA, 1989 Dynasty	3.36	96	Straw	381	< 0.01	0.01	< 0.01	< 0.02
Noblesville, Indiana, USA, 1989 Caldwell	3.36	119	Straw	393	0.04	0.04	0.01	0.08
Lucama, North Carolina, USA, 1989 Pioneer 2555	3.27	121	Straw	345	< 0.01	0.01	< 0.01	< 0.02
Sparta, Michigan, USA, 1989 Pioneer 2550	3.36	147	Straw	432	0.02	0.02	< 0.01	0.04
York, Nebraska, USA, 1989 Brule	3.36	127	Straw	435	0.03	0.04	0.01	0.07
Grain								
Colo, Iowa, USA, 1989 Siouxland HRW	3.36	154	Grain	435	< 0.01	< 0.01	< 0.01	< 0.02
Dacono, Colorado, USA, 1989 Hawk	3.36	161	Grain	446	< 0.01	< 0.01	< 0.01	< 0.02
Danville, Iowa, USA, 1989 Caldwell	3.36	113	Grain	418	< 0.01	< 0.01	< 0.01	< 0.02
Delavan, Wisconsin, USA, 1989 Caldwell	3.36	141	Grain	436	< 0.01	< 0.01	< 0.01	< 0.02
Devine/Hondo, Texas, USA, 1989 MIT	2.10	90	Grain	315	< 0.01	< 0.01	< 0.01	< 0.02
Eakly, Oklahoma, USA, 1989 Pioneer 2157	3.36	176	Grain	426	< 0.01	< 0.01	< 0.01	< 0.02
Elwood, Illinois, USA, 1989 Pioneer 2550	3.59	133	Grain	405	< 0.01	< 0.01	< 0.01	< 0.02
Geneseo, Illinois, USA, 1989 Caldwell	3.47	120	Grain	455	< 0.01	< 0.01	< 0.01	< 0.02
Hawkinsville, Georgia, USA, 1989 Coker 9766	3.36	160	Grain	380	< 0.01	< 0.01	< 0.01	< 0.02
Hollandale, Minnesota, USA, 1989 Seward	3.36	106	Grain	427	< 0.01	< 0.01	< 0.01	< 0.02
Sedan, Kansas, USA, 1989 Delange 7837	3.36	119	Grain	391	< 0.01	< 0.01	< 0.01	< 0.02
Leonard, Missouri, USA, Delange 7837	3.36	148	Grain	426	< 0.01	< 0.01	< 0.01	< 0.02
Lexington, Kentucky, USA, 1989 Compton	3.18	157	Grain	440	< 0.01	< 0.01	< 0.01	< 0.02
New Holland, Ohio, USA, 1989 Dynasty	3.36	96	Grain	381	< 0.01	< 0.01	< 0.01	< 0.02
Noblesville, Indiana, USA, 1989 Caldwell	3.36	119	Grain	393	< 0.01	< 0.01	< 0.01	< 0.02
Lucama, North Carolina, USA, 1989 Pioneer 2555	3.27	121	Grain	345	< 0.01	< 0.01	< 0.01	< 0.02
	16.3	121	Grain	345	< 0.01	< 0.01	< 0.01	< 0.02
Sparta, Michigan, USA, 1989 Pioneer 2550	3.36	147	Grain	432	< 0.01	< 0.01	< 0.01	< 0.02
York, Nebraska, USA, 1989 Brule	3.36	127	Grain	435	< 0.01	< 0.01	< 0.01	< 0.02
	16.8	127	Grain	435	< 0.01	< 0.01	< 0.01	< 0.02

Storage to analysis intervals were: forage < 538 d; straw < 293 d, grain < 238 d

Total = EMA + HEMA

Planted DAA = follow crop planting/sowing days after application to primary maize crop

Harvest DAA = follow crop harvest days after application to primary maize crop

Table 44 Residues of acetochlor in soya bean follow crops (Sidhu 1992 MSL-11963). HEMA and EMA residues are expressed in acetochlor equivalents.

Location, year, variety	Primary crop		Follow	crop	HEMA	EMA	HMEA	Total
SOYA BEAN	rate (kg ai/ha)	Planted DAA	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)
Forage								
Colo, Iowa, USA, 1989 CX265	3.36	375	Forage	433	0.05	0.05	0.03	0.10
Dacono, Colorado, USA, 1989 Cargill C-285	3.36	388	Forage	446	0.06	0.16	0.03	0.22
Danville, Iowa, USA, 1989 Washington VI	3.36	380	Forage	436	0.02	0.04	< 0.01	0.06
Delavan, Wisconsin, USA, 1989 Northup King 523-12	3.36	376	Forage	424	0.07	0.16	0.04	0.23
Uvalde, Texas, USA, 1989 RA452	2.10	253	Forage	372	0.09	0.11	0.04	0.20
Eakly, Oklahoma, USA, 1989 Pioneer 2157	3.36	393	Forage	468	0.02	0.02	< 0.01	0.04
Elwood, Illinois, USA, 1989 Pioneer 2157	3.59	425	Forage	404	0.19	0.44	0.12	0.63
Geneseo, Illinois, USA, 1989 Pioneer 2157	3.47	361	Forage	476	< 0.01	< 0.01	< 0.01	< 0.02
Hawkinsville, Georgia, USA, 1989 Coker 9766	3.36	366	Forage	405	0.03	0.05	0.01	0.08
Hollandale, Minnesota, USA, 1989 Seward	3.36	367	Forage	427	0.01	0.02	< 0.01	0.04
Sedan, Kansas, USA, 1989 Delange 7837	3.36	397	Forage	459	0.01	0.03	< 0.01	0.05
Leonard, Missouri, USA, Delange 7837	3.36	396	Forage	454	0.04	0.08	0.02	0.12
Lexington, Kentucky, USA, Delange 7837	3.18	407	Forage	454	< 0.01	< 0.01	< 0.01	< 0.02
New Holland, Ohio, USA, 1989 Dynasty	3.36	362	Forage	408	0.04	0.09	0.02	0.13
Noblesville, Indiana, USA, 1989 Caldwell	3.36	372	Forage	426	0.05	0.10	0.03	0.14
Lucama, North Carolina, USA, 1989 Pioneer 2555	3.27	312	Forage	402	0.04	0.05	0.02	0.10
Sparta, Michigan, USA, 1989 Pioneer 2550	3.36	385	Forage	433	0.04	0.12	0.02	0.15
York, Nebraska, USA, 1989 Brule	3.36	396	Forage	452	0.07	0.15	0.05	0.22
Hay								
Colo, Iowa, USA, 1989 CX265	3.36	375	Hay	436	0.12	0.12	0.08	0.24
Dacono, Colorado, USA, 1989 Cargill C-285	3.36	388	Hay	457	0.31	0.73	0.15	1.04
Danville, Iowa, USA, 1989 Washington VI	3.36	380	Hay	438	0.09	0.15	0.04	0.24
Delavan, Wisconsin, USA, 1989 Northup King 523-12	3.36	376	Hay	430	0.17	0.31	0.12	0.48
Eakly, Oklahoma, USA, 1989 Pioneer 2157	3.36	393	Hay	475	0.03	0.04	0.02	0.08
Elwood, Illinois, USA, 1989 Pioneer 2157	3.59	425	Hay	409	0.12	0.19	0.08	0.31
Geneseo, Illinois, USA, 1989 Pioneer 2157	3.47	361	Hay	491	0.03	0.04	0.02	0.07
Hawkinsville, Georgia, USA, 1989 Coker 9766	3.36	366	Hay	415	0.07	0.12	0.04	0.19
Sedan, Kansas, USA, 1989 Delange 7837	3.36	397	Hay	461	0.04	0.11	< 0.01	0.14
Leonard, Missouri, USA, Delange 7837	3.36	396	Hay	457	0.13	0.24	0.06	0.37

Location, year, variety SOYA BEAN	Primary crop rate (kg ai/ha)	Planted DAA	Follow Sample	crop Harvest DAA	HEMA (mg/kg)	EMA (mg/kg)	HMEA (mg/kg)	Total (mg/kg)
Lexington, Kentucky, USA, Delange 7837	3.18	407	Hay	461	< 0.01	0.02	< 0.01	< 0.02
New Holland, Ohio, USA, 1989 Dynasty	3.36	362	Hay	410	0.12	0.30	0.05	0.42
Noblesville, Indiana, USA, 1989 Caldwell	3.36	372	Hay	432	0.20	0.35	0.12	0.55
Lucama, North Carolina, USA, 1989 Pioneer 2555	3.27	312	Hay	408	0.18	0.21	0.09	0.39
Sparta, Michigan, USA, 1989 Pioneer 2550	3.36	385	Hay	438	0.08	0.21	0.04	0.29
York, Nebraska, USA, 1989 Brule	3.36	396	Hay	458	0.18	0.24	0.13	0.41
Grain								
Dacono, Colorado, USA, 1989 Cargill C-285	3.36	388	Grain	533	0.01	0.02	< 0.01	0.03
Danville, Iowa, USA, 1989 Washington VI	3.36	380	Grain	513	< 0.01	0.01	< 0.01	< 0.02
Delavan, Wisconsin, USA, 1989 Northup King 523-12	3.36	376	Grain	519	0.03	0.01	< 0.01	0.04
Eakly, Oklahoma, USA, 1989 Pioneer 2157	3.36	393	Grain	561	< 0.01	0.01	< 0.01	< 0.02
Elwood, Illinois, USA, 1989 Pioneer 2157	3.59	425	Grain	502	0.07	0.03	0.03	0.10
Geneseo, Illinois, USA, 1989 Pioneer 2157	3.47	361	Grain	541	< 0.01	< 0.01	< 0.01	< 0.02
Hawkinsville, Georgia, USA, 1989 Coker 9766	3.36	366	Grain	524	< 0.01	< 0.01	< 0.01	< 0.02
Hollandale, Minnesota, USA, 1989 Seward	3.36	367	Grain	496	< 0.01	< 0.01	< 0.01	< 0.02
Sedan, Kansas, USA, 1989 Delange 7837	3.36	397	Grain	526	< 0.01	< 0.01	< 0.01	< 0.02
Leonard, Missouri, USA, Delange 7837	3.36	396	Grain	531	0.01	0.01	< 0.01	0.02
Lexington, Kentucky, USA, Delange 7837	3.18	407	Grain	541	< 0.01	< 0.01	< 0.01	< 0.02
New Holland, Ohio, USA, 1989 Dynasty	3.36	362	Grain	509	0.02	0.02	< 0.01	0.04
Noblesville, Indiana, USA, 1989 Caldwell	3.36	372	Grain	512	< 0.01	< 0.01	< 0.01	< 0.02
Lucama, North Carolina, USA, 1989 Pioneer 2555	3.27	312	Grain	485	< 0.01	0.02	< 0.01	< 0.03
	16.3	312	Grain	485	0.05	0.03	0.01	0.09
Sparta, Michigan, USA, 1989 Pioneer 2550	3.36	385	Grain	525	0.02	0.01	< 0.01	0.03
York, Nebraska, USA, 1989 Brule	3.36	396	Grain	521	0.04	0.02	< 0.01	0.06
	16.8	396	Grain	521	0.20	0.14	0.06	0.34

Storage to analysis intervals: forage < 146 d; ha < 162 d; grain <342 d

Total = EMA + HEMA

Planted DAA = follow crop planting/sowing days after application to primary maize crop

Harvest DAA = follow crop harvest days after application to primary maize crop

Table 45 Residues of acetochlor in sorghum follow crops (Sidhu 1992 MSL-11963). HEMA and EMA residues are expressed in acetochlor equivalents.

Location, year, variety	Primary crop		Follow	crop	HEMA	EMA	HMEA	Total
SORGHUM	Rate (kg ai/ha)	Planted DAA	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)
Forage								
Colo, Iowa, USA, 1989 DeKalb 398	3.36	375	Forage	433	< 0.01	0.02	< 0.01	< 0.03
Dacono, Colorado, USA, 1989 Cargill 577	3.36	388	Forage	446	< 0.01	0.06	< 0.01	< 0.07
Danville, Iowa, USA, 1989 Merschman 175	3.36	380	Forage	436	< 0.01	0.03	< 0.01	< 0.04
Delavan, Wisconsin, USA, 1989 Milomaster	3.36	376	Forage	424	< 0.01	0.03	< 0.01	< 0.04
Uvalde, Texas, USA, 1989 Pioneer 8333	2.10	253	Forage	372	< 0.01	< 0.01	< 0.01	< 0.02
Eakly, Oklahoma, USA, 1989 Concep II	3.36	420	Forage	475	< 0.01	0.03	< 0.01	< 0.03
Elwood, Illinois, USA, 1989	3.59	425	Forage	404	< 0.01	0.08	< 0.01	< 0.08
Geneseo, Illinois, USA, 1989	3.47	361	Forage	476	< 0.01	0.01	< 0.01	< 0.02
Hawkinsville, Georgia, USA, 1989 Delta Pineland G522OR	3.36	366	Forage	405	< 0.01	0.02	< 0.01	< 0.03
Hollandale, Minnesota, USA, 1989	3.36	366	Forage	427	< 0.01	0.03	< 0.01	< 0.04
Sedan, Kansas, USA, 1989 ORO Pronto	3.36	397	Forage	450	< 0.01	0.03	< 0.01	< 0.04
Leonard, Missouri, USA, 1989 Mustang	3.36	397	Forage	454	< 0.01	0.05	< 0.01	< 0.06
Lexington, Kentucky, USA, 1989 Funks Y42	3.18	407	Forage	454	< 0.01	< 0.01	< 0.01	< 0.02
New Holland, Ohio, USA, 1989 GA-Y101R	3.36	362	Forage	408	< 0.01	0.05	< 0.01	< 0.06
Noblesville, Indiana, USA, 1989 GA-Y101R	3.36	372	Forage	426	< 0.01	0.05	< 0.01	< 0.06
Lucama, North Carolina, USA, 1989 Pioneer 8333	3.27	312	Forage	360	< 0.01	0.02	< 0.01	< 0.03
Sparta, Michigan, USA, 1989 Staton Seed Supply Lot 2150	3.36	385	Forage	433	< 0.01	0.05	< 0.01	< 0.06
York, Nebraska, USA, 1989 Cargill 70	3.36	396	Forage	452	< 0.01	0.07	< 0.01	< 0.08
Hay								
Colo, Iowa, USA, 1989 DeKalb 398	3.36	375	Hay	436	< 0.01	0.02	< 0.01	< 0.03
Dacono, Colorado, USA, 1989 Cargill 577	3.36	388	Hay	457	0.02	0.16	0.02	0.18
Danville, Iowa, USA, 1989 Merschman 175	3.36	380	Hay	438	< 0.01	0.06	< 0.01	< 0.07
Delavan, Wisconsin, USA, 1989	3.36	376	Hay	430	0.01	0.04	< 0.01	0.05

Location, year, variety	Primary crop		Follow	crop	HEMA	EMA	HMEA	Total
SORGHUM	Rate (kg ai/ha)	Planted DAA	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)
Milomaster								
Uvalde, Texas, USA, 1989 Pioneer 8333	2.10	253	Hay	375	< 0.01	< 0.01	< 0.01	< 0.02
Eakly, Oklahoma, USA, 1989 Concep II	3.36	420	Hay	479	< 0.01	0.04	< 0.01	< 0.05
Elwood, Illinois, USA, 1989	3.59	425	Hay	409	< 0.01	0.05	< 0.01	< 0.06
Geneseo, Illinois, USA, 1989	3.47	361	Hay	491	< 0.01	0.02	< 0.01	< 0.03
Hawkinsville, Georgia, USA, 1989 Delta Pineland G522OR	3.36	366	Hay	415	< 0.01	0.03	< 0.01	< 0.04
Sedan, Kansas, USA, 1989 ORO Pronto	3.36	397	Hay	459	0.02	0.06	0.02	0.08
Leonard, Missouri, USA, 1989 Mustang	3.36	397	Hay	457	< 0.01	0.07	< 0.01	< 0.08
Lexington, Kentucky, USA, 1989 Funks Y42	3.18	407	Hay	461	< 0.01	< 0.01	< 0.01	< 0.02
New Holland, Ohio, USA, 1989 GA-Y101R	3.36	362	Hay	410	0.01	0.05	< 0.01	0.06
Noblesville, Indiana, USA, 1989 GA-Y101R	3.36	372	Hay	432	0.01	0.08	< 0.01	0.09
Lucama, North Carolina, USA, 1989 Pioneer 8333	3.27	312	Hay	366	< 0.01	0.05	< 0.01	< 0.06
Sparta, Michigan, USA, 1989 Staton Seed Supply Lot 2150	3.36	385	Hay	438	0.01	0.06	0.01	0.07
York, Nebraska, USA, 1989 Cargill 70	3.36	396	Hay	458	< 0.01	0.07	0.01	< 0.08
Silage								
Colo, Iowa, USA, 1989 DeKalb 398	3.36	375	Silage	490	< 0.01	0.01	< 0.01	< 0.02
Dacono, Colorado, USA, 1989 Cargill 577	3.36	388	Silage	509	< 0.01	0.02	< 0.01	< 0.03
Danville, Iowa, USA, 1989 Merschman 175	3.36	380	Silage	490	< 0.01	0.02	< 0.01	< 0.03
Delavan, Wisconsin, USA, 1989 Milomaster	3.36	376	Silage	479	< 0.01	0.02	< 0.01	< 0.03
Uvalde, Texas, USA, 1989 Pioneer 8333	2.10	253	Silage	398	< 0.01	< 0.01	< 0.01	< 0.02
Eakly, Oklahoma, USA, 1989 Concep II	3.36	420	Silage	517	< 0.01	0.02	< 0.01	< 0.03
Elwood, Illinois, USA, 1989	3.59	425	Silage	462	< 0.01	0.03	0.01	< 0.04
Geneseo, Illinois, USA, 1989	3.47	361	Silage	520	< 0.01	< 0.01	< 0.01	< 0.02
Hawkinsville, Georgia, USA, 1989 Delta Pineland G522OR	3.36	366	Silage	455	< 0.01	0.01	< 0.01	< 0.02
Hollandale, Minnesota, USA, 1989	3.36	366	Silage	475	0.01	0.02	< 0.01	0.04

Location, year, variety	Primary crop		Follow	crop	HEMA	EMA	HMEA	Total
SORGHUM	Rate (kg ai/ha)	Planted DAA	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)
Sedan, Kansas, USA, 1989 ORO Pronto	3.36	397	Silage	488	< 0.01	0.01	< 0.01	< 0.02
Leonard, Missouri, USA, 1989 Mustang	3.36	397	Silage	478	< 0.01	0.04	< 0.01	< 0.05
Lexington, Kentucky, USA, 1989 Funks Y42	3.18	407	Silage	511	< 0.01	< 0.01	< 0.01	< 0.02
New Holland, Ohio, USA, 1989 GA-Y101R	3.36	362	Silage	451	< 0.01	0.02	< 0.01	< 0.03
Noblesville, Indiana, USA, 1989 GA-Y101R	3.36	372	Silage	476	< 0.01	0.02	< 0.01	< 0.03
Lucama, North Carolina, USA, 1989 Pioneer 8333	3.27	312	Silage	404	< 0.01	< 0.01	< 0.01	< 0.02
Sparta, Michigan, USA, 1989 Staton Seed Supply Lot 2150	3.36	385	Silage	489	< 0.01	0.01	< 0.01	< 0.02
York, Nebraska, USA, 1989 Cargill 70	3.36	396	Silage	501	< 0.01	0.03	< 0.01	< 0.04
Fodder								
Dacono, Colorado, USA, 1989 Cargill 577	3.36	388	Fodder	533	< 0.01	0.06	0.01	< 0.07
Danville, Iowa, USA, 1989 Merschman 175	3.36	380	Fodder	513	< 0.01	0.01	< 0.01	< 0.02
Delavan, Wisconsin, USA, 1989 Milomaster	3.36	376	Fodder	523	< 0.01	0.02	< 0.01	< 0.03
Uvalde, Texas, USA, 1989 Pioneer 8333	2.10	253	Fodder	426	< 0.01	< 0.01	< 0.01	< 0.02
Eakly, Oklahoma, USA, 1989 Concep II	3.36	420	Fodder	540	< 0.01	0.03	< 0.01	< 0.04
Elwood, Illinois, USA, 1989	3.59	425	Fodder	502	< 0.01	0.05	0.01	< 0.06
Geneseo, Illinois, USA, 1989	3.47	361	Fodder	541	< 0.01	< 0.01	< 0.01	< 0.02
Hawkinsville, Georgia, USA, 1989 Delta Pineland G522OR	3.36	366	Fodder	490	< 0.01	< 0.01	< 0.01	< 0.02
Hollandale, Minnesota, USA, 1989	3.36	366	Fodder	496	< 0.01	0.02	< 0.01	< 0.03
Sedan, Kansas, USA, 1989 ORO Pronto	3.36	397	Fodder	526	< 0.01	0.01	< 0.01	< 0.02
Leonard, Missouri, USA, 1989 Mustang	3.36	397	Fodder	531	< 0.01	0.04	< 0.01	< 0.05
Lexington, Kentucky, USA, 1989 Funks Y42	3.18	407	Fodder	541	< 0.01	< 0.01	< 0.01	< 0.02
New Holland, Ohio, USA, 1989 GA-Y101R	3.36	362	Fodder	509	< 0.01	0.02	< 0.01	< 0.03
Noblesville, Indiana, USA, 1989 GA-Y101R	3.36	372	Fodder	512	< 0.01	0.02	< 0.01	< 0.03
Lucama, North Carolina, USA, 1989	3.27	312	Fodder	446	< 0.01	< 0.01	< 0.01	< 0.02

Location, year, variety	Primary crop		Follow	crop	HEMA	EMA	HMEA	Total
SORGHUM	Rate (kg ai/ha)	Planted DAA	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)
Pioneer 8333								
Sparta, Michigan, USA, 1989 Staton Seed Supply Lot 2150	3.36	385	Fodder	535	< 0.01	< 0.01	< 0.01	< 0.02
York, Nebraska, USA, 1989 Cargill 70	3.36	396	Fodder	531	< 0.01	0.04	< 0.01	< 0.04
Grain								
Dacono, Colorado, USA, 1989 Cargill 577	3.36	388	Grain	533	< 0.01	< 0.01	< 0.01	< 0.02
Danville, Iowa, USA, 1989 Merschman 175	3.36	380	Grain	513	< 0.01	< 0.01	< 0.01	< 0.02
Delavan, Wisconsin, USA, 1989 Milomaster	3.36	376	Grain	523	< 0.01	< 0.01	< 0.01	< 0.02
Uvalde, Texas, USA, 1989 Pioneer 8333	2.10	253	Grain [a]	426	< 0.01	< 0.01	< 0.01	< 0.02
			Grain [b]	426	< 0.01	< 0.01	< 0.01	< 0.02
Eakly, Oklahoma, USA, 1989 Concep II	3.36	420	Grain	540	< 0.01	< 0.01	< 0.01	< 0.02
Elwood, Illinois, USA, 1989	3.59	425	Grain	502	< 0.01	< 0.01	< 0.01	< 0.02
Geneseo, Illinois, USA, 1989	3.47	361	Grain	541	< 0.01	< 0.01	< 0.01	< 0.02
Hawkinsville, Georgia, USA, 1989 Delta Pineland G522OR	3.36	366	Grain	490	< 0.01	< 0.01	< 0.01	< 0.02
Hollandale, Minnesota, USA, 1989	3.36	366	Grain	496	< 0.01	< 0.01	< 0.01	< 0.02
Sedan, Kansas, USA, 1989 ORO Pronto	3.36	397	Grain	526	< 0.01	< 0.01	< 0.01	< 0.02
Leonard, Missouri, USA, 1989 Mustang	3.36	397	Grain	531	< 0.01	< 0.01	< 0.01	< 0.02
Lexington, Kentucky, USA, 1989 Funks Y42	3.18	407	Grain	541	< 0.01	< 0.01	< 0.01	< 0.02
New Holland, Ohio, USA, 1989 GA-Y101R	3.36	362	Grain	509	< 0.01	< 0.01	< 0.01	< 0.02
Noblesville, Indiana, USA, 1989 GA-Y101R	3.36	372	Grain	512	< 0.01	< 0.01	< 0.01	< 0.02
Lucama, North	3.27	312	Grain	446	< 0.01	< 0.01	< 0.01	< 0.02
Carolina, USA, 1989 Pioneer 8333	16.3	312	Grain	446	< 0.01	0.01	< 0.01	< 0.02
Sparta, Michigan, USA, 1989 Staton Seed Supply Lot 2150	3.36	385	Grain	535	< 0.01	< 0.01	< 0.01	< 0.02
York, Nebraska,	3.36	396	Grain	531	< 0.01	< 0.01	< 0.01	< 0.02
USA, 1989 Cargill 70	16.8	396	Grain	531	< 0.01	< 0.01	< 0.01	< 0.02

Storage to analysis intervals: forage 711 days, hay 739 d, silage < 696 d; fodder < 707 d; grain < 659 d

Total = EMA + HEMA

Planted DAA = follow crop planting/sowing days after application to primary maize crop

Harvest DAA = follow crop harvest days after application to primary maize crop

Veal and Spillner (1997a RJ2261B, 1997b RJ2262B) carried out trials on dried shelled peas and beans in the USA in which a primary crop, maize, was treated with one application of WF1301, an emulsifiable concentration of acetochlor at 3.4 kg ai/ha. The following year, beans (330–406 days after application) or peas (296–336 days after application) were planted in the plots previously treated with acetochlor and samples of dried shelled beans and peas taken at normal harvest to determine the magnitude of the residues of acetochlor and the EMA and HEMA class metabolites.

No residues of acetochlor at or above the limit of determination of 0.01 mg/kg were found in any of the samples analysed. No residues of EMA or HEMA class metabolites (LOQ 0.02 mg/kg acetochlor equivalents) were found, except for one residue of EMA at the LOQ in one sample of shelled beans and one of shelled peas.

Table 46 Residues of acetochlor in pulse (bean and pea) follow crops (Veal and Spillner 1997a RJ2261B, 1997b RJ2262B). HEMA and EMA residues are expressed in acetochlor equivalents.

Location, year, variety	Primary crop		Follow crop		acetochlor	HEMA	EMA	Total
	Rate (kg ai/ha)	Planted DAA	Sample	Harvest DAA	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)
DRIED SHELLED BEANS								
North Rose, New York,	3.36	406	Pre-plant	504	< 0.01	< 0.02	< 0.02	< 0.04
USA, 1995 California Red Kidney					< 0.01	< 0.02	< 0.02	< 0.04
Champaign, Illinois, USA,	3.36	352	Pre-	453	< 0.01	< 0.02	< 0.02	< 0.04
1995 Henry Field's Pinto			emergence		< 0.01	< 0.02	< 0.02	< 0.04
Conklin, Michigan, USA,	3.36	380	Pre-plant	483	< 0.01	< 0.02	< 0.02	< 0.04
1995 Avanti Navy Bean					< 0.01	< 0.02	< 0.02	< 0.04
Mooreton, North Dakota,	3.36	377	Pre-	511	< 0.01	< 0.02	< 0.02	< 0.04
USA, 1995 Upland			emergence		< 0.01	< 0.02	< 0.02	< 0.04
Scottsbluff, Nebraska, USA,	3.36	390	Pre-plant	484	< 0.01	< 0.02	< 0.02	< 0.04
1995 Beryl Great Northern					< 0.01	< 0.02	< 0.02	< 0.04
Ault, Colorado, USA, 1995	3.36	388	Pre-plant	498	< 0.01	< 0.02	< 0.02	< 0.04
Bill Z					< 0.01	< 0.02	< 0.02	< 0.04
Austin, Colorado, USA,	3.36	357	Pre-plant	481	< 0.01	< 0.02	< 0.02	< 0.04
1995 Bill Z					< 0.01	< 0.02	< 0.02	< 0.04
Visalia, California, USA,	3.36	330	Pre-plant	448	< 0.01	< 0.02	< 0.02	< 0.04
1995 Green Crop					< 0.01	< 0.02	< 0.02	< 0.04
Jerome, Idaho, USA, 1995	3.36	383	Pre-plant	495	< 0.01	< 0.02	< 0.02	< 0.04
Pinto					< 0.01	< 0.02	0.02	< 0.04
DRIED SHELLED PEAS								
Jerome, Idaho, USA, 1995	3.36	334	Pre-plant	444	< 0.01	< 0.02	< 0.02	< 0.04
Asgrow Cabree					< 0.01	< 0.02	< 0.02	< 0.04
Jerome, Idaho, USA, 1995	3.36	327	Pre-plant	433	< 0.01	< 0.02	< 0.02	< 0.04
Asgrow Cabree					< 0.01	< 0.02	< 0.02	< 0.04
Ephrata, Washington, USA,	3.36	301	Pre-	426	< 0.01	< 0.02	< 0.02	< 0.04
1995 Columbian			emergence		< 0.01	< 0.02	< 0.02	< 0.04
Mt. Vernon, Washington,	3.36	336	Pre-plant	443	< 0.01	< 0.02	< 0.02	< 0.04
USA, 1995 SS Alaska					< 0.01	< 0.02	< 0.02	< 0.04
Hermiston, Oregon, USA,	3.36	296	Pre-	412	< 0.01	< 0.02	< 0.02	< 0.04
1995 Fraser			emergence		< 0.01	< 0.02	0.02	< 0.04

Total = EMA + HEMA

Planted DAA = follow crop planting/sowing days after application to primary maize crop
Harvest DAA = follow crop harvest days after application to primary maize crop

In summary, residues in edible commodities (grain and tubers) of follow crops were < LOQ with the exception of soya beans. Residues were detected in livestock feeds such as forage, hay, straw and silage of alfalfa, clover and oats, wheat, sorghum and soya beans.

ENVIRONMENTAL FATE IN SOIL

Route of Degradation in Soil

Aerobic degradation in soil

A number of studies have investigated the aerobic degradation of [^{14}C]acetochlor in soil.

The rate and route of degradation of [^{14}C-U-phenyl]-acetochlor was investigated in a silty clay loam soil (Atterbury, USA, 2% sand, 67% silt, 31% clay, pH 6.9, 4.1% OM, CEC 23.6 meq/100g, % moisture holding capacity (saturation) 55.9%) under aerobic conditions by Hawkins *et al.* (1989 HRC/STR 19/881751; 1991 HRC/STR 19/901756). [^{14}C]acetochlor was applied at a nominal rate equivalent to a single application of 4.48 kg ai/ha (4.5 mg/kg soil) and 40.8 kg ai/ha (41 mg/kg soil). The soil samples were incubated under aerobic conditions in the laboratory and maintained under moist, dark conditions at 22 ± 1 °C for up to 365 days. Additional samples sterilised following autoclaving at 120 °C for 30 min were also studied at the 4.5 mg/kg soil concentration. Volatiles were collected in trapping solutions. Samples were sequentially extracted with CH3CN, CH3CN/H2O (7:3 v/v) at ambient temperature and then CH3CN/H2O under reflux using a Soxhlet apparatus.

The mean total recoveries of radioactivity for 0 to 30 DAA was 99.2% of applied radioactivity (AR) declining to 76.9% AR by 365 DAA. By 30 DAA 1.2% of the AR was mineralised to $^{14}CO_2$ with 71.9% AR extracted with the solvent systems used and 25.8 % remaining unextracted.

Acetochlor was rapidly degraded, accounting for less than 26% AR by 30 DAA. By day 365, acetochlor accounted for 1.7% AR. The only other products observed were *tert*-oxanilic acid (2), *tert*-sulfinylacetic acid (3), *tert*-sulfonic acid (7) which reached a maximum between days 30 and 180 accounting for 15.9–17.1, 4–6.5, 7.4–11.8% AR respectively and declining to 0.8–7.8% AR by day 365.

Table 47 Degradation of acetochlor (%AR) under aerobic conditions on silty clay loam soil (initial application of [^{14}C]acetochlor at 4.5 mg/kg)

Distribution of Residues	Days after application											
	0	1	3	7	14	30	60	90	120	180	275	365
Volatiles	–	0.02	0.07	0.2	0.6	1.2	2.5	3.2	3.6	4.5	7.0	9.8
Extracted	96.9	96.1	99.4	87.3	79.6	71.9	52.6	52.6	51.2	51.8	44.4	32.6
acetochlor	93.8		79.5	68.6	45.5	25.6	6.4	5.1	4.7	2.2	4.7	1.7
tert-oxanilic acid (2)	0.4	NA	1.6	4.3	12.2	15.9	17.1	16.3	14.4	14.1	9.6	6.4
tert-sulfinylacetic acid (3)	< 0.2	NA	< 0.2	1.6	3.3	6.5	4.2	4	6.5	1.9	0.7	0.8
tert-sulfonic acid (7)	–	NA	0.3	3.1	6.9	7.4	10.6	11.1	7.5	11.8	8.8	7.8
Unextracted	2.5	3.9	4.5	9.5	15.6	25.8	29.0	31.3	26.2	26.9	29.8	34.5
Total Recovered	99.4	99.9	104	97	95.9	99.0	84.2	87.2	81.2	83.2	81.3	76.9

NA = Not analysed

In sterile soils, acetochlor represented 73% of the ^{14}C present at 70 days of incubation and when compared to the approximately 19% for non-sterile, viable soils, the data indicate degradation is primarily due to microbial activity.

Table 48 Degradation of acetochlor under aerobic conditions on sterile silty clay loam soil (initial application of [^{14}C]acetochlor at 4.5 mg/kg)

Distribution of residues	Days after application	
	7	30
Volatiles	< 0.1	< 0.2
Acetochlor	94	73.2
Degradates	5.2	14.5
Unextracted	7.4	12.1
Total Recovered (as % AR)	106.6	99.8

The rate of degradation was estimated using single first-order (SFO) kinetics. The DT_{50} and DT_{90} values obtained are presented in Table 49. Degradation was slower in soils treated at the higher rate (Table 49).

Table 49 Summary of DT_{50} for acetochlor in Atterbury silty clay loam (22 °C in the dark) Hawkins *et al*. (1991, HRC/STR 19/901756)

Soil: Atterbury silty clay loam	DT_{50} (days)	DT_{90} (days)
0–60 days	13.5	44
41 ppm, 14–365 days	55	Not calculated

Hawkins *et al*. (1991, HRC/ISN 185/90535) studied the metabolism and degradation of [phenyl-U-^{14}C]acetochlor in sandy loam soil (East Jubilee Field, UK, 61.7% sand, 20.2% silt, 18.1% clay, pH 6.0, 2.9% OM, CEC 9.1 meq/100g, % moisture holding capacity (33 kPa) 17.4%, biomass 24.4 mg C/100g) under aerobic conditions. Soil samples were incubated in darkness, at an average temperature of 22.0 ± 1 °C, for periods of up to one year. Acetochlor was applied to soil at a rate equivalent to a field application rate of 3.0 kg ai/ha (sterile and non-sterile soil) and at 14 kg ai/ha (non-sterile soil). Volatiles were collected in trapping solutions. Samples were sequentially extracted with CH_3CN, CH_3CN/H_2O (7:3 v/v) at ambient temperature and for samples from 120 DAA also CH_3CN/H_2O under reflux using a Soxhlet apparatus. The 0 DAA samples were extracted with CH_3CN (4×) only.

Through the first 90 days of the study, acetochlor degraded with a half-life of 110 days; however, subsequent degradation of the remaining residue occurred more slowly. Four major metabolites of acetochlor were identified. The acetochlor degradates *tert*-oxanilic acid (**2**), *tert*-hydroxy (17), *tert*-sulfonic acid (7) and *tert*-sulfinylacetic acid (3) reached maximum levels of approximately 9%, 7%, 6% and 5% of the AR, respectively. No other single component of the extracted radioactivity (except acetochlor) accounted for more than 5% of the AR at any time. Unextracted radioactivity amounted to 14–19% of AR by 365 DAT of which $^{14}CO_2$ accounted for ca. 0.5% AR.

Table 50 Degradation of acetochlor (% AR) under aerobic conditions on sandy loam soil

Distribution of Residues	Days after application											
	0	1	3	7	14	30	60	90	120	180	275	365
Volatiles	–	0.02	0.04	0.2	0.3	0.6	1.5	1.8	2.1	2.2	2.5	2.8
Extracted												
acetochlor	96.4	94.2	99.8	88.6	88.4	72.1	62.7	43	46.9	44.3	45.9	40.9
tert-oxanilic acid (2)	ND	ND	0.5	1.3	2.1	4.4	4.7	9	7	3.9	4.6	4.2
tert-sulfinylacetic acid (3)	ND	0.1	0.3	0.8	1.2	2.5	3.9	4.3	4.4	3.5	3.1	2.3
tert-sulfonic acid (7)	ND	0.1	0.2	0.4	1	1.8	3	4.1	5	4.2	4.3	3.9
tert-hydroxy (17)	U	0.3	0.6	1	1.5	2.3	3.5	4.7	4.4	5	6.3	6.6
Unextracted	0.4	0.6	1.3	2.6	4.2	7.8	12	14.6	14.8	12.5	15.4	15.6
Total Recovered	98.3	97.3	105	99.1	103.8	97.8	98.1	89.7	97	89	94.1	88.3

ND = not determined

U = Unresolved from other peaks, total 0.6%

The fate of acetochlor was also investigated following application at an exaggerated (5×) rate. The half-life at this rate was approximately 300 days. The same metabolites were evident and unextracted and volatile components were produced in similar amounts to those found after application at the 'normal' rate.

Acetochlor was not appreciably degraded in the sterile soil over a 30-day period, indicating that the degradation of acetochlor in soil is primarily due to microbiological activity.

Campbell and Hamilton (1980, MSL-1255) studied the degradation of [carbonyl-^{14}C]acetochlor in three soils (silt loam, sandy loam, silty clay loam) maintained under aerobic conditions in the dark at a nominal temperature of 22 °C for 168 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 0.33 bar. Relevant properties of the soils used are presented in Table 51.

The aerobic degradation in sterile soils was also studied (22 °C for 28 days).

Table 51 Properties of soils used to study aerobic degradation of acetochlor Campbell and Hamilton (1980, MSL-1255)

Soil Name	Texture	%sand	%silt	%clay	%OM	pH	CEC meq/100 g	Water holding capacity (%)
Ray	Silt loam	4.6	84.2	10.0	1.2	8.1	10.4	23.9
Drummer	Silty clay loam	2.4	68.8	25.3	3.4	6.2	24.6	28.8
Spinks	Sandy loam	75.1	17.8	4.8	2.4	4.7	28.8	17.9

[Carbonyl-^{14}C]-Acetochlor was applied at a nominal rate of 3 mg/kg dry soil. Samples were analysed after 0, 7, 15, 30, 60, 90 and 120 days incubation. Soil samples were extracted with CH_3CN/H_2O (4×), once with aqueous 0.1 N ammonium hydroxide, and twice with water. The aqueous acetonitrile extracts were then partitioned with dichloromethane.

Extracted radioactivity recovered from samples declined from 98.2–102.9% at Day 0 to 63.9–67.3% at Day 56 and 50–55.3% at Day 168. Unextracted residues reached maximum levels of 64.4% at Day 56 in Ray soil, 40.6% at Day 84 in Drummer soil and 47.0% at Day 28 in Spinks soil and were 17.1–24.5% at the end of the test period. Volatile radioactivity identified as $^{14}CO_2$ represented 7.9–11.0 % AR at Day 56 and 16.5–24.5% at day 168. $^{14}CO_2$ production in sterile soils was lower than in the corresponding viable soil amounting to 0–4.4%AR after 28 days post treatment. The overall material balance ranged between *ca* 91 and 138% AR.

The amount of acetochlor recovered in the solvent extracts declined from 91, 94 and 92% AR at zero time to 0.4, 0.9 and 1.3% AR after 168 days in the Ray silt loam, Drummer silty clay loam and Spinks sandy loam soils, respectively. Three major metabolites were identified from the water soluble fraction. These metabolites reached their respective maximums between 21 and 56 days post treatment and then steadily declined through the end of the test. The *tert*-oxanilic acid (2) reached a maximum concentration of 15% AR, the *tert*-sulfinylacetic acid (3) was observed at a maximum concentration of 18% AR, and the *tert*-sulfonic acid (7) reached a maximum concentration of 11% AR. Fourteen other metabolites were identified, twelve organosoluble and two water soluble metabolites. One metabolite, the *sec*-sulfonic acid (13) exceeded 5% of AR in one soil and continued to increase at the end of the study in all soils, reaching a maximum of 9.8% AR in the Ray silt loam soil. The *tert*-hydroxy metabolite (17) exceeded 5% AR in only one soil at a single time point. No other metabolites exceeded 5% AR at any time during the study.

Table 52 Recovery of radioactivity (% AR) and distribution of [carbonyl-^{14}C] acetochlor and its main degradates in soils under aerobic conditions

Days after application	0	1	3	7	14	21	28	56	84	168
Ray silt loam										
Total recovered	100.6	102.7	105.3	121.9	125.9	138.4	125.5	136.9	107.1	93.6
CO_2	0	0.2	0.7	1.5	2.2	3.5	4.9	8.6	12.5	21.6

Days after application	0	1	3	7	14	21	28	56	84	168
Extracted CH_2Cl_2	97.1	89.8	77.1	62.4	38.1	24.6	17.7	9.6	7.4	5.2
acetochlor	91.1	83.4	68.9	54.2	29.1	15.3	8.8	1.9	1.1	0.4
sec-norchloro (9)	–	–	–	–	–	–	–	–	0.1	0.1
tert-norchloro (6)		0.4	0.6	0.8	0.8	0.8	0.9	0.6	0.6	0.4
tert-hydroxy (17)	–	–	0.9	1.1	1.2	1.3	1.4	0.3	0.5	0.2
ketoethyl (19)	–	–	1.2	1.5	1.4	1.5	1.6	0.6	0.6	0.3
(20)	–	–	0.8	2.4	1.7	1.2	0.9	0.5	0.3	0.2
sec-methylsulfone (10)	–	–	–	–	0.4	0.4	0.5	0.7	0.6	0.8
tert-methylsulfone (16)	–	–	–	0.6	0.9	1.1	1.5	2.4	1.5	1.5
Extracted water	0.8	6.2	13.5	21.3	36.5	45.0	50.0	50.5	52.6	45.7
tert-methylsulfoxide (15)	–	–	–	0.8	1.0	1.3	1.2	1.1	0.9	0.6
tert-oxanilic acid (2)	–	1.9	4.5	6.8	10.6	13	15.7	14.1	14.6	10.2
tert-sulfinylacetic acid (3)	–	1	3	5.6	10.3	12.9	12.6	11.7	11.2	8
tert-sulfonic acid (7)	–	1.5	3.5	5.4	9.1	10.7	11	10.7	11	8.2
sec-sulfonic acid (13)	–	0.1	0.3	0.9	1.7	2.3	3.2	4.6	7.2	9.8
Unextracted	1.5	5.6	12.4	34.3	46.3	62.8	50	64.4	31.5	18.7
Drummer silty clay loam										
Total recovered	104	104.9	110.4	109.9	110.9	120.2	102.8	99.4	109.8	93
CO_2	0	0.3	0.7	1.5	2.7	3.2	4.5	7.9	11	16.5
Extracted CH_2Cl_2	101.5	89.4	74.7	65.9	40.5	33.8	25.6	11.5	9.8	7.6
acetochlor	93.8	79.3	65.7	58.2	32.7	19.8	14.3	3.2	1.6	0.9
sec-norchloro (9)					0.1	0.1	0.2	0.1	0.1	0.1
tert-norchloro (6)	0.6	0.7	0.7	1.1	0.9	2.0	1.6	1.4	1.4	1.2
tert-hydroxy (17)	2.2	1.9	1.7	1	0.4	5.4	1.1	0.4	0.3	0.2
ketoethyl (19)	–	1.2	1.1	1.0	0.6	1.3	0.9	0.5	0.7	0.4
(20)	–	1.4	1.4	1.7	1.4	1.3	1.3	0.8	0.6	0.3
sec-methylsulfone (10)	–	–	0.3	0.3	0.1	0.4	0.3	0.3	0.4	0.6
tert-methylsulfone (16)	–	–	0.4	0.7	1.1	1.8	1.6	2.1	2.3	2.3
Extracted water	0.9	4.8	12.2	18.3	31.1	37.5	42.0	44.5	42.3	40.2
tert-methylsulfoxide (15)	–	–	0.8	1.0	0.7	1.7	1.4	1.1	1.1	0.7
tert-oxanilic acid (2)	–	–	3.9	6	10.7	11.7	14.7	13.6	13.1	12.1
tert-sulfinylacetic acid (3)	–	–	4.2	7.5	11.4	13.8	15.4	18	13	15.3
tert-sulfonic acid (7)	–	–	2	2.4	5.1	5.4	6.9	6.8	6.9	6
sec-sulfonic acid (13)	–	–	0.3	0.2	0.6	0.7	1	1.3	1.1	1.8
Unextracted	1.1	9.1	20.8	21	31.1	41.4	26.5	24.2	40.6	24.5
Spinks sandy loam										
Total recovered	105.8	110.5	103.6	118.4	111.4	113.3	125.1	100.7	108.8	91.6
CO_2	0	0.2	0.5	1.3	2.6	4.2	5.7	11	14.9	24.5
Extracted CH_2Cl_2	97.1	78.2	68.8	56.9	39.5	25.5	19.7	6.1	2.9	1.3
acetochlor	91.7	78.2	68.8	56.9	39.5	25.5	19.7	6.1	2.9	1.3
sec-norchloro (9)							0.2	0.1	0.1	0.1
tert-norchloro (6)	–	–	0.6	0.7	1.3	1.6	2.0	2.6	1.8	1.3
tert-hydroxy (17)	1	1.1	1.6	1.6	3.2	3.4	3.6	2.4	1.6	0.7
ketoethyl (19)	–	–	1.3	1.1	1.1	1.3	1.4	0.9	0.7	0.4
(20)	–	1.4	1.4	1.8	2.2	2.1	2.6	2.1	1.7	0.8
sec-methylsulfone (10)	–	–	–	–	0.6	0.7	0.9	0.3	0.3	0.2
tert-methylsulfone (16)	–	–	–	0.6	0.6	0.6	0.8	1.4	1.2	0.9
Extracted water	1.0	3.0	6.8	12.9	22.3	28.0	30.4	35.7	36.4	33.4
tert-methylsulfoxide (15)	–	–	–	–	–	0.2	0.2	0.4	0.4	0.2
tert-oxanilic acid (2)	–	1	2.7	4.5	7.7	11.5	11.3	12.8	12.2	11
tert-sulfinylacetic acid (3)	–	0.6	1.5	2.6	5.7	6	6.6	8.1	9.2	7
tert-sulfonic acid (7)	–	0.5	1.2	2.4	4.1	5.5	5.3	6.6	5.3	4.3
sec-sulfonic (13)	–	–	0.1	0.2	0.3	0.3	0.6	0.7	0.8	1.5
Unextracted	7.6	18.1	15.5	35	28.8	34.3	47	25	31.3	17.1

Parallel experiments using sterile soils at 22 °C indicated slower degradation of acetochlor, decreasing to 30.0–87.1 % AR at the end of the 28-day test period, compared to 8.8–19.7% AR in viable soils. The majority of the organosoluble fraction was acetochlor. Water

soluble and ammonia fractions contained less than 3% applied radioactivity, apart from the silt loam and silty clay loam soil on Day 28 when the water soluble fractions contained 29.6 and 45.8% AR, most likely due to experimental error. Metabolite identification was not conducted on these extracts.

Mason and Mills (1999 98JH113) studied the laboratory degradation of [^{14}C-U-phenyl]-acetochlor in surface and sub-soils collected from a site in Iowa, USA. Only soil from 10–20 cm below the uppermost end of each core was used in the incubation study. The 2 mm sieved samples were adjusted to their respective experimental moisture level and aliquots placed in incubation pots. These were equilibrated under the test conditions for 12 days at 20 °C (±2 °C). Following equilibration, soil pots were treated with ^{14}C-labelled acetochlor at application rates of 3.3 mg/kg for surface soil and 0.12 mg/kg for both sub-soils, equivalent to approximately 131% and 5% of the maximum agricultural application rate, of 3.5 kg ai/ha, respectively. The treated soil pots were placed back into the incubation columns, with a flow-through of moist CO_2-free air for up to 121 days. The soils, maintained at the approximately pF2 moisture level, were incubated at 20 °C (± 2°C) in the dark throughout the study.

During incubation, effluent gas from each incubation column was passed through a series of traps, including ethanolamine to trap evolved $^{14}CO_2$. At pre-determined intervals after treatment (0, 7, 14, 33, 64 days (all depths) and 92 days (mid and deep depths only) duplicate soil pots (triplicate soil pots at Day 0) were removed from each of the incubation columns. Of the duplicate pots, one was analysed for total radiochemical content, acetochlor and acetochlor metabolites.

Microbial biomass of the soils was determined at approximately the start and end of the post treatment incubation period. The microbial biomass carbon was 11.6 and 9.8 mg biomass C/100 g soil for surface soils, 7.8 and 10 for mid-depth soils, and 7.3 and 6.6 for deep soils, at the start and end of study, respectively.

Soil samples from all depths demonstrated the ability to degrade acetochlor. The rate of degradation was accurately described by the first order multi-compartment model. The DT_{50} values were 15.0 days for surface soil, 5.3 days for mid-depth soil, and 5.6 days for deep soil. Furthermore, CO_2 levels reached 1.9% AR for surface soil, 2.6% AR for mid-depth soil, and 1.9% AR for deep soil indicating some mineralisation of acetochlor had occurred in all soils. The acetochlor metabolite profile was similar to previously reported work.

This study has shown that acetochlor degradation and mineralisation can take place in both surface and sub-soils, despite the latter containing a relatively inactive microbial population.

Table 53 Recovery of radioactivity (% AR) and distribution of [^{14}C-U-phenyl] acetochlor and its main degradates in soils under aerobic conditions

DAA	0	7	14	33	64	92
Silty clay loam (Surface soil 0–30 cm)						
Extracted	94.2	85.2	79.2	70.3	64.1	NA
acetochlor	92	41.5	59.7	39.3	19.3	NA
tert-oxanilic acid (2)	0	12.8	3.3	10.4	20.4	NA
tert-hydroxy (17)	0	5.8	0	2.5	2.9	NA
tert-sulfonic acid (7)	0	7.6	13.1	4.9	6.9	NA
tert-thioacetic acid (4)	0	0	3.1	1.6	1.5	NA
tert-sulfinylacetic acid (3)	0	3.4	3.7	5.9	7.9	NA
Baseline	1.1	0.5	0.8	0.3	0.4	NA
Others	1.2	13.7	0	5.7	4.9	NA
Unextracted	2.8	11.3	15.3	21.2	26.3	NA
CO_2	na	0.1	0.3	0.9	1.9	NA
Silty clay (mid-depth soil 107-137 cm)						
Extracted	92.8	56.1	31.6	42.5	62.4	61.9
acetochlor	87.1	32.4	8.7	5.4	0	0
tert-oxanilic acid (2)	0	14.6	3.3	6	16.1	16.2
tert-hydroxy (17)	0	1.7	5.4	10.9	11.7	14
tert-sulfonic acid (7)	0	2.5	3.8	4.6	8.7	8.2

DAA	0	7	14	33	64	92
tert-thioacetic acid (4)	0	6.3	2.1	3.1	4.4	2.7
tert-norchloro (6)	0	0	1.2	1	0	0
tert-sulfinylacetic acid (3)	0	2.1	1.8	3.1	8.4	6.6
Baseline	0.3	0.1	4.9	5.3	1.2	4.2
Others	5.4	0	3.8	6	12.4	11.7
Unextracted	3.2	34.56	54.6	45.2	27	19
CO_2	na	0	0	0.5	1.3	2.6
Silty clay loam (deep soil 274–305 cm)						
Extracted	92.1	37.5	34.9	49.2	67.5	69.2
acetochlor	85.2	19.7	10.8	15.4	0	0
tert-oxanilic acid (2)	0	5	2.7	7.7	14.4	35.2
tert-hydroxy (17)	2.6	4	5.2	10.5	12.2	6.2
tert-sulfonic acid (7)	0	0.7	1	1.5	4.2	6.1
tert-thioacetic acid (4)	2.3	1.7	1.6	2.8	3.1	1.9
tert-norchloro (6)	0	0	1	3.2	4.3	0
tert-sulfinylacetic acid (3)	0	0.6	1.2	2	4.6	10.7
Baseline	0.5	1.7	6.1	8.5	7.6	1.7
Others	1.6	5.3	9.3	2	19.6	7.9
Unextracted	3.3	51.5	52.8	40.7	23.9	21.9
CO_2	na	0	0.1	0.6	1.5	1.9

Table 54 Estimated DT_{50} and DT_{90} values for aerobic degradation of acetochlor in soil

Model	SFO		FOMC	
Soil depth	DT_{50}	DT_{90}	DT_{50}	DT_{90}
Surface	26.9	89.5	14.9	302
Mid-depth	5.29	17.6	5.27	17.5
Deep	5.58	18.5	5.6	18.6

Verity *et al.* (1999 RJ2749B) studied the degradation of acetochlor under laboratory conditions in surface and sub-soils collected from an untreated area in Wisconsin, USA. Surface soil from a 0–30 cm depth (sandy loam, 1.8% OM, pH 6.6) and two sub-soils from 30–76 cm and 260–305 cm (loamy sand, 0.7% OM, pH 6.7 and sand, 0.6% OM, pH 6.9, respectively) were collected and transported to the laboratory in 10 cm diameter schedule-40 plastic tubes under cool conditions (< 12 °C). The soil samples were treated with [^{14}C-U-phenyl]-acetochlor and incubated in the laboratory to determine the degradation rate of acetochlor under two different sets of temperature and moisture conditions.

The soil was stored at 4 °C for 6 weeks prior to use. Only soil from 10–20 cm below the surface of the soil in each core was used in the incubation study. The soil was passed through a 2 mm sieve and samples prepared at two moisture contents: as "received from the field", and at approximately pF2, and aliquots placed in incubation pots. These were equilibrated under the test conditions for 5–7 days at 20 ± 2 °C.

Following equilibration, soil pots were treated with ^{14}C-labelled acetochlor at application rates of 2.0 mg/kg for surface soil and 0.1 mg/kg for both sub-soils; equivalent to approximately 100% and 5% of the normal agricultural application rate, of 2.9 kg ai/ha, respectively. The treated soil pots were placed back into the incubation columns, with a flow-through of moist CO_2 -free air for up to 122 days.

The soils maintained at approximately pF2 moisture levels were incubated at 20 ± 2 °C throughout the study. Soils maintained at field moisture were incubated at temperatures similar to those recorded by temperature probes in the field. These soils were incubated, following treatment with acetochlor, at 20 °C for the first 23 days, 18 °C for the next 46 days, 16 °C for the next 37 days and 10 °C for the last 16 days.

Microbial biomass of the soils was determined at the start and end of the incubation period. The microbial biomass carbon, as a percentage of organic matter carbon, was 1.5 and 1.7

for surface soils, 1.0 and 0.6 for mid-depth soils, and 0.3 and 0.2 for deep soils, at the start and end of study, respectively. This results indicate that end of study mid-depth and deep soil throughout would not be considered to possess an active microbial population (i.e. < 1% of organic carbon is microbial biomass carbon).

Soil samples from all depths, under both sets of laboratory incubation conditions, demonstrated the ability to degrade acetochlor.

The DT_{50} values based on modelling were 13.2 and 7.2 days for surface soil, 2.9 and 2.3 days for mid-depth soil, and 29.9 and 10.3 days for deep soil under pF2 and 'field' incubation conditions, respectively. Furthermore, CO_2 levels reached 4.1 % (of applied radioactivity) and 4.5% for surface soil, 6.1% and 4.5% for mid-depth soil, and 9.5% and 3.5% for deep soil, under pF2 and 'field' incubation conditions, respectively, indicating complete mineralisation of some acetochlor had occurred in all soils. There was evidence for both aerobic and anaerobic degradation, although aerobic degradation predominated.

Table 55 Recovery of radioactivity (% AR) and distribution of [^{14}C-U-phenyl] acetochlor and its main degradates in soils under aerobic conditions

DAA	0		7		14		28		63		98		122	
Surface soil														
Moisture	pF 2	MFT	pF 2	MFT	pF 2	MFT	pF 2	MFT	pF 2	MFT	pF 2	MFT	pF 2	MFT
acetochlor	99	99.5	71.6	65	62.3	45.4	25.2	19.6	12.1	5.1	4.6	5.2	4.5	5.9
(2)	0	0	6.1	7	7.2	13.7	19	18.8	17.6	15	22.4	20.1	17.5	15.2
(17)	0	0	2.6	3.8	2.7	2.1	2.5	2.5	2.1	2.2	1.4	3.2	0.9	1.2
(7)	0	0	2.6	2.8	3.1	4.5	6.8	6	4.7	6.6	6.7	8.6	4.9	6
(6)	0	0	0	0	0	0	0	1	0.4	3.3	1.7	0.6	0.4	0.7
(3)	0	0	1.3	1.8	1.7	3.4	4.1	4.8	3.5	3.2	5.1	6.9	4.3	5.2
Baseline	0.2	0.1	0.4	2.2	0.5	0.8	0.7	0.4	0.3	0.2	0.1	0.4	0.3	0.4
Others	1.4	1.2	4.2	4.9	4.7	6.9	9	9	8.7	7.8	9.2	8.1	9.3	8.1
Unextracted	1.1	1.1	16.1	15.9	19.6	26.2	34.6	26.5	43	44.7	44.3	42.8	45.5	43.8
CO_2	na	na	0.3	0	0.7	0.8	1.4	1.4	2.1	2.9	3.4	4.1	4.1	4.5
Mid soil														
Moisture	pF 2	MFT	pF 2	MFT	pF 2	MFT	pF 2	MFT	pF 2	MFT	pF 2	MFT	pF 2	MFT
Acetochlor	98.5	100.5	42.2	37.5	17.3	15.5	6.5	4.9	1.7	2.5	2.3	2.1	2.2	2.5
(2)	0	0	9.2	11.2	14.9	17.2	15.7	17.8	17.3	15.6	18.3	18.4	14.3	14.9
(17)	0	0	3.5	4.6	5	4.8	2.5	2.3	1.7	2.1	1.4	2.2	1.1	1.4
(7)	0	0	9.1	8.5	19.6	16.1	18.9	15.9	20.9	12.3	16	16.3	16.8	16.2
(4)	0	0	2.3	2.1	1.2	0	0	0	0	0	0.8	0.8	0	0
(6)	0	0	0	0	0	1	0.6	0.9	2.4	2.3	2.4	2.6	1	0.9
(3)	0	0	2.9	4.2	6.1	6.4	5.6	6.1	5.7	3.6	4.7	5	3.7	4.2
Baseline	0.1	0.1	0.7	0.5	0.7	0.3	0.6	0.5	0.5	0.5	0.1	1	0.3	0.3
Others	0.8	0.7	11.1	13.9	11.2	14.1	16.5	13.6	12.6	13.9	14.2	13	12	13.1
Unextracted	0.4	0.7	19.1	22.1	26.6	18.6	27	33	32.8	33.3	32	33.3	33	32.4
CO_2	na	na	0.2	0	1.5	1.2	2.9	2.3	4.4	3.8	5.4	4.3	6.1	4.5
Deep soil														
Moisture	pF 2	MFT	pF 2	MFT	pF 2	MFT	pF 2	MFT	pF 2	MFT	pF 2	MFT	pF 2	MFT
acetochlor	101.3	97.3	88.4	63.7	79.7	51.9	54.7	26.3	25.5	7.2	11.3	6.8	7.6	5.2
(2)	0	0	2.2	3.3	3.7	10	6.8	17	12.5	26.9	14.9	18.1	14.6	21.9
(17)	0	0	1.6	3.1	1.8	6.5	2.3	6.2	1.7	5.4	2.6	3.2	1.3	2.2
(7)	0	0	1.5	0.7	3.4	2	6.2	6.7	10.2	11.3	13.4	14.7	13.2	9.8
(4)	0	0	0.6	1.1	0.9	2.3	0.9	2	0.9	2.3	0	0	0	0
(6)	0	0	0	4.2	2.9	0.4	0	0.6	2.8	0	0.4	0.2	1.5	1.3
(3)	0	0	0.9	0.8	1	2	2.3	2.5	5.2	3	6.4	4.6	5.4	3.8
Baseline	0.1	0.2	0.4	0.9	0.4	1.8	0.5	1.8	0.6	0.8	0.6	1.1	1.1	0.6
Others	0.6	1.5	5.1	11.7	6.1	13.9	10.5	16	19.6	16.9	16.6	22.6	15.5	15.7
Unextracted	0.6	0.6	3.1	5.7	5.3	13.2	7.9	15.2	15	20	15.6	18.5	17.5	21.1
CO_2	na	na	0.3	0	0.8	0.5	2	1.1	5.4	1.9	8.2	3.2	9.5	3.5

2=*tert*-oxanilic acid

3=*tert*-sulfinylacetic acid

4=*tert*-thioacetic acid

6=*tert*-norchloro
7=*tert*-sulfonic acid
17=*tert*-hydroxy

Table 56 Estimated DT_{50} and DT_{90} values for aerobic degradation of acetochlor in soil

moisture	pF 2				FMT (field moisture content)			
kinetics	SFO		FOMC		SFO		FOMC	
Soil depth	DT_{50}	DT_{90}	DT_{50}	DT_{90}	DT_{50}	DT_{90}	DT_{50}	DT_{90}
Surface	26.2	87.1	13.2	65.3	28.8	95.5	7.2	50.5
Mid-depth	24.3	80.8	2.9	21.1	25.9	86.1	2.3	19.5
Deep	31.7	105.4	29.9	104.4	28.9	95.9	10.3	63.6

A proposed metabolic pathway for the aerobic degradation of acetochlor in soil is shown in Figure 16.

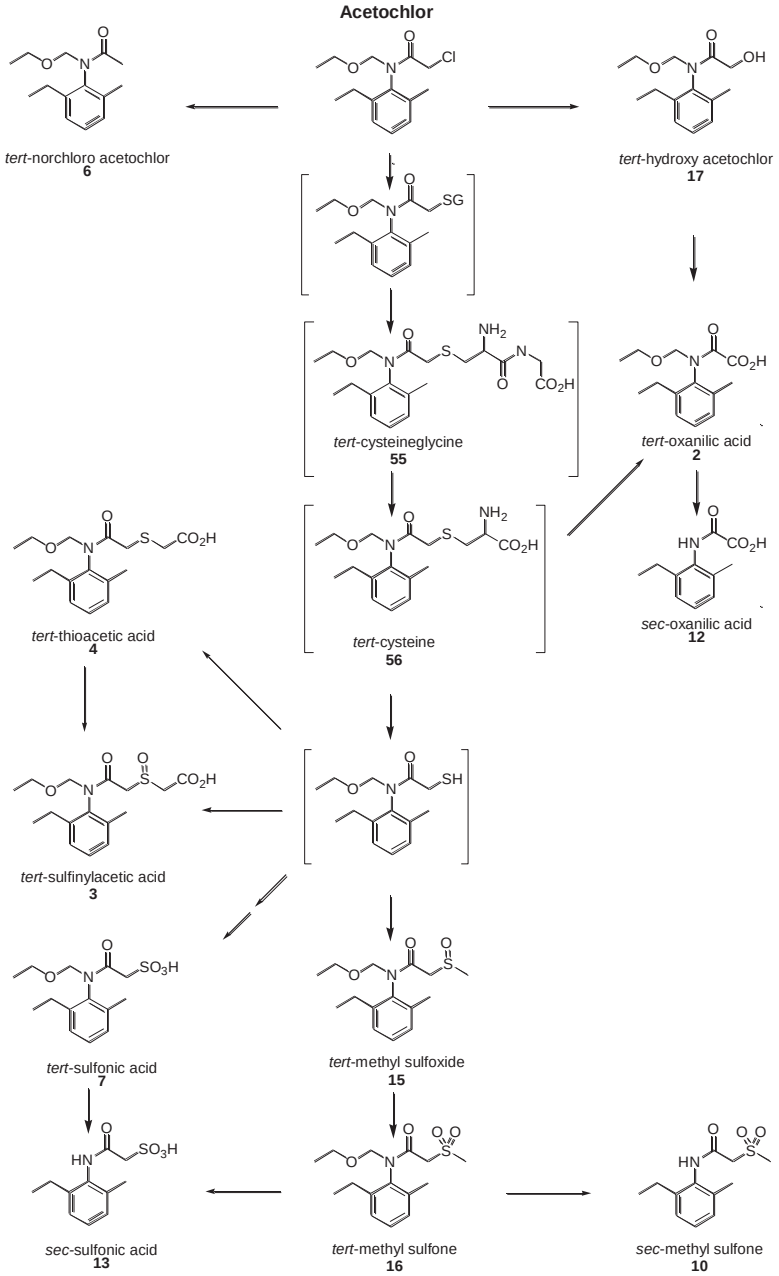


Figure 16 Proposed aerobic degradation pathway for acetochlor in soil

Rate of aerobic degradation

In a series of laboratory studies, the degradation of acetochlor was studied following a single application of [^{14}C-U-phenyl]-acetochlor to surface and subsurface soils obtained from a variety of sites in the USA Vaughan *et al.* (1999 RJ2151B) Tarry *et al*. (1998, RJ2396B). The results are summarized in Table 57 and indicate acetochlor residues *per se* should not be persistent in soil. No estimates have been calculated for acetochlor degradates and these may persist for longer periods than acetochlor.

Table 57 Summary of additional laboratory studies on the DT_{50} and DT_{90} values for aerobic degradation of acetochlor in soils (Vaughan *et al.* 1999 RJ2151B, Tarry *et al.* 1998, RJ2396B)

Soil	Application	Characteristics of soils			Incubation conditions		Best-Fit	DT_{50}	DT_{90}
	rate (mg/kg)	pH	% OM	% clay	ºC	Moisture	Model	days	(days)
Vaughan *et al.* (1999 RJ2151B									
Ohio 1	2	5	1.3	20	20	40% MHC	SFO	16.4	54.5
Ohio 1 (Low Rate)	0.04	5	1.3	20	20	40% MHC	SFO	23.8	79
Ohio 2	2	7.5	2.4	25	20	40% MHC	SFO	13.7	45.5
Ohio 2 (Low Rate)	0.04	7.5	2.4	25	20	40% MHC	SFO	12.9	43
Ohio 3	2	8	2.8	25	20	40% MHC	FOMC	9.1	55.3
Ohio 4	2				20	40% MHC	SFO	9.7	33
Wisconsin 1	2	7.1	0.7	8	20	40% MHC	SFO	5.9	22.4
Wisconsin 1 (Low Rate)	0.04	7.1	0.7	8	20	40% MHC	SFO	9.2	32
Wisconsin 2	2	7.2	1.2	8	20	40% MHC	SFO	7.7	26
Wisconsin 3	2	7.2	1	8	20	40% MHC	SFO	12.1	41.6
Wisconsin 3 (Low Rate)	0.04	7.2	1	8	20	40% MHC	SFO	12.8	42.8
Wisconsin 4	2	6.2	0.8	8	20	pF2	SFO	7.4	25.7
Tarry *et al.* (1998, RJ2396B)									
Indiana	0.04	6.3	1.7	21	20	pF2	SFO	7.9	26.4
Iowa 1	2	6	3.5	36	20	pF2	SFO	16.3	54
Iowa 1 (low rate)	3.3	6	3.5	36	20	pF2	SFO	10.3	34.2
Minnesota 1	2	6	3.5	15	20	pF2	SFO	9.4	31.4
Minnesota 1 (low rate)	0.04	6	3.5	15	20	pF2	SFO	7.9	26.2
Nebraska	0.04	7.9	1.3	27	20	pF2	SFO	3.3	11.1
Wisconsin 4	2	6.2	0.8	8	20	pF2	SFO	7.4	25.7

Field dissipation

Studies on the field dissipation of acetochlor residues were not made available to the Meeting; however, in a report in the scientific literature Oliveire *et al.* (2013) studied the persistence of acetochlor (parent compound) in Minnesota, USA where 38 locations with a wide range of soils from a single 16 ha watershed in Dakota County were sampled over a two year period (2000 and 2001). DT_{50} values ranged from 2.9 to 8.4 days (n = 74) and are in general agreement with those observed in laboratory studies.

Table 58 Descriptive statistics of soil properties and acetochlor dissipation from surface soils in the watershed study in 2000 and 2001 (Oliveire Jr RS, Koskinen WC, Graff CD, Anderson JL, Mulla DJ, Nater EA, Alonso DG (2013) Acetochlor persistence in surface and sub-surface soil samples. Water Air Soil Pollution 224: 1747)

Soil properties	n	Range	Mean	Median	SD
pH	136	5.5–7.6	6.6	6.6	0.49
OM, %	136	1.2–5.2	2.5	2.5	0.59
Clay, %	136	12.3–27.0	20.3	20.4	3.42
Silt, %	136	39.1–68.7	55.7	56.6	6.10
Slope, %	136	0–24.3	5.9	5.0	3.7
2001					
DT50 (days)	38	4.0–8.4	5.7	5.6	2.5
2001					
DT50 (days)	36	2.9–12.6	7.7	6.0	4.5

FATE AND BEHAVIOUR IN WATER

Hydrolysis

Myers (1989, WRC 88-70) studied the hydrolytic stability of acetochlor at 25 ± 0.5 °C for 31 days in dark, sterile, aqueous buffered solutions at pH 5, 7 and 9. Aqueous solutions of acetochlor showed less than 1% degradation at the end of the 31 day study period. Acetochlor is considered stable to hydrolysis at pH 5, 7 and 9 for at least 31 days.

A study to determine if hydrolysis would represent a significant degradation pathway for acetochlor in the environment was conducted (Campbell and Hamilton 1980 MSL-1255). This study showed that acetochlor was stable to hydrolysis in deionized water, in sterile buffers at pH 3, 6, and 9 and in sterilized lake water.

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

Aqueous photolysis

Chotalia and Weissler (1989 RJ0726B) studied the aqueous photolysis of [^{14}C]acetochlor at pH 7. Acetochlor accounted for 97.3% of the radioactivity at the start of the experiment and 88.8% after irradiation from a Xe arc lamp for a period equivalent to 30 days Florida summer sunlight. No degradation occurred in the dark controls. Acetochlor is considered to be essentially photolytically stable.

Environmental Fate Summary

Acetochlor degraded rapidly and extensively in soil under aerobic conditions with half-lives ranging from 3 to 30 days for soils treated at rates comparable to or lower than an equivalent maximum use rate of 3.36 kg ai/ha. The half-life from an application rate that was approximately 10-fold higher than the lower rate used in the study (4.5 mg/kg, which was equivalent to 4.48 kg ai/ha) was 55 days. Three major degradates exceeded 10% of applied radioactivity and were identified. Acetochlor *tert*-oxanilic acid (2), *tert*-sulfonic acid (7), and *tert* sulfinylacetic acid (3) reached maximum concentrations of 17, 13, and 18% of applied radioactivity, respectively, before declining by the end of the studies. No other components reached the 10% level at any time point. One metabolite, *sec*-sulfonic acid (13) reached 9.8% in one soil, and continued to increase towards the end of the incubation period. Under sterile conditions, degradation of acetochlor was significantly slower and no metabolites were observed at greater than 10% of the applied radioactivity. The results clearly indicate that degradation is principally microbially mediated.

Acetochlor is stable in sterile aqueous solutions at pH 5, 7, and 9.

Low levels of acetochlor residues are taken up by plants from soil following applications of acetochlor to soil. A confined rotational crop study conducted with radish, lettuce, and wheat resulted in TRRs of 0.05 2.88 mg/kg acetochlor equivalents at harvest of commodities from plantings 30 to 365 days after application of acetochlor to a sandy loam at 3.36 kg ai/ha. Analysis of commodities showed that residues comprised up to ten different compounds, with none exceeding 0.03 mg/kg in the edible portion of the crop (radish root and wheat grain). Five metabolites, which were consistently present in plant extracts from all three rotation intervals, were identified as *sec*-oxanilic acid (12), *tert*-oxanilic acid (2), *sec*-sulfonic acid (13), *tert*-sulfonic acid (7), and 1-hydroxyethyl *tert*-oxanilic acid (26). No acetochlor was detected in wheat or radish planted 30 and 365 days after application; however, it was detected at a level of 0.03 mg/kg in radish foliage from the 120-day planting.

A field confined rotational crop study was conducted with turnip, mustard, soya bean, millet, radish, and wheat. The study involved application of acetochlor to sandy loam soil at two different sites in the US at a rate equivalent to 3.32 kg ai/ha. Analyses of the plant extracts showed that extensive metabolism occurred in all crops. The TRR was characterised and found to be comprised of up to nine different compounds, with no one above 0.01 mg/kg in the edible portion of the root or cereal crop (turnip root, millet grain). The significant metabolites identified in the 30 DAA rotational crops were *tert*-oxanilic acid (2), *sec*-methylsulfone (10), *sec*-hydroxyacetochlor (11), and *tert*-methylsulfone (16) at 0.002–0.025 mg/kg. The 5-hydroxy-*sec*-oxanilic acid (68) appeared in the majority of crops analysed at noticeable concentrations of 0.005–0.018 mg equiv/kg. A significant percentage of TRR was not identified (e.g., in turnip tops 44.7% (0.18 mg equiv/kg), which contained at least 39 metabolites, the largest of which represented 0.016 mg equiv/kg. In the 120 DAA rotational interval, 5-hydroxy-*sec*-oxanilic acid (68) was also identified. Only *tert*-oxanilic acid (2, 0.14 mg equiv/kg), *sec*-methylsulfone (10, 0.005 mg equiv/kg), and 5-hydroxy-*sec*-oxanilic acid (68, 0.004 mg equiv/kg) were observed with levels higher than 0.002 mg equiv/kg in the 365 DAA rotational crops.

Field residue rotational crop studies involving a 3.36 kg ai/ha application of acetochlor in the previous season have been conducted on numerous crops that include the following: potatoes, sunflowers, oat, alfalfa, clover, wheat, soya bean, sorghum, dried shelled beans, and dried shelled peas. Total acetochlor residues (HEMA + EMA) were below the LOQ for potato tubers, all grain/seed commodities, beans, and peas except for soya bean seed, which reached a maximum of 0.12 mg/kg. Maximum residues in crop foliage (forage, hay, silage and straw) ranged from 0.06 to 1.19 mg/kg. Maximum residues in alfalfa and clover foliage (forage or hay) ranged from 0.54 to 1.87 mg/kg.

METHODS OF RESIDUE ANALYSIS

Analytical methods

The Meeting received descriptions and validation data for analytical methods for residues of acetochlor and metabolites in animal and plant matrices. The methods are suitable for analysis of acetochlor and metabolites in plant and animal matrices.

The metabolism of acetochlor in crops results in a complex mixture of metabolites that arise from initial glutathione (homoglutathione) conjugation. Subsequent catabolism of the glutathione conjugate via known routes along with oxidative processes and conjugation with natural products (e.g., glucose, malonic acid, etc.) or sulphite results in a wide variety of metabolites, most of which produce EMA or HEMA on base hydrolysis. The EMA-producing metabolites contain non-modified alkyl side-chains and phenyl rings and are converted to EMA upon hydrolysis. Any non-metabolised parent acetochlor that might be present would be converted to EMA. The HEMA-producing metabolites contain hydroxylation at the 1-position of the ethyl group attached directly to the phenyl ring, and a non-modified phenyl ring and are converted to HEMA upon hydrolysis. Metabolites that result from hydroxylation of the methyl group attached to the phenyl ring can also form, although generally not to a great extent. These metabolites are converted to HMEA upon hydrolysis.

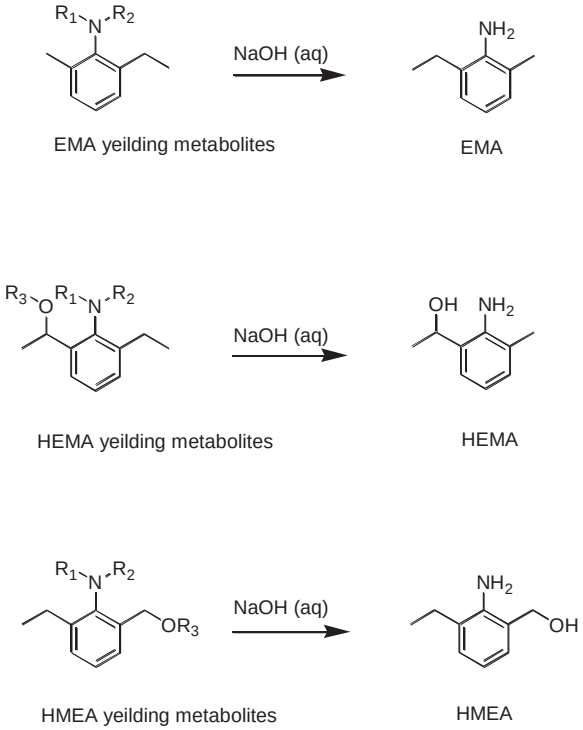


Figure 17 Aniline classes of metabolites obtained by base hydrolysis

Most of the methods developed to quantify acetochlor residues in plant commodities involve hydrolytic conversion of metabolites to the EMA and HEMA chemophores. These analytes are quantified in acetochlor equivalents and then may be summed to give total acetochlor residues. For samples from the rotational crop studies reported in MSL-11963 and the storage stability study reported in MSL-12139, residues of metabolites converted to HMEA on base hydrolysis, i.e. metabolites containing hydroxylation on the ring methyl group, were also quantified.

The methods all involve initial extraction of samples with an organic/aqueous solvent mixture, typically CH_3CN/H_2O, followed by hydrolysis of acetochlor residues with aqueous hydroxide solutions. The main differences between methods involve clean-up conditions, aniline derivatization (RES-074-93 and RAM 280 only), instrumentation for quantification, and scale.

In addition to the methods summarized for quantification of acetochlor residues in crops, a method is described for the determination of metabolites hydrolysable to EMA and HEMA in milk and animal tissues.

Representative compounds that generate EMA (*tert*-sulfonic acid) and HEMA (1-hydroxyethyl-*tert*-oxanilic acid) on hydrolysis were used as reference materials for fortification and method validation.

A method has been developed for determination of 5-hydroxy-*sec*-oxanilic acid (68) in corn and other crops that involves solvent extraction and derivatisation of (68) with N-methyl-N-(tert-butyldimethylsilyl)-trifluoroacetamide (MTBSTFA) for analysis by GC-MS.

Plant materials

RAM-280-01, RAM-280-02 (Robinson. 1996 RAM-280-01, Robinson 1998 RAM-280-02)

In summary, prepared crop samples are extracted by maceration with CH_3CN/H_2O (80:20 v/v). Samples are then filtered under vacuum or centrifuged depending on crop matrix and an aliquot is

evaporated to dryness under a stream of dry air. Saturated KOH solution and methanol are added to the samples and then the samples are refluxed for 30 minutes to 60 minutes to hydrolyse metabolites to EMA and HEMA. The hydrolysate is diluted with water and saturated NaCl and partitioned with toluene. An aliquot of the toluene extract is derivatised with heptafluorobutyric acid anhydride (HFAA) to acylate the EMA and HEMA. Excess derivatising agent is removed by partition of the derivatised samples with sodium hydrogen carbonate solution and the samples are analysed by GC-MS. The results are quantified against the acylated EMA and HEMA standards prepared in the relevant crop matrix.

Ion-monitoring for: 329, 314 amu for HEMA heptafluorobutyl derivative; 331. 162 amu for EMA heptafluorobutyl derivative. Representative compounds that generate EMA (*tert*-sulfonic acid) and HEMA (1-hydroxyethyl-*tert*-oxanilic acid) on hydrolysis are used as reference materials for fortification and method validation. The average recoveries for 1-hydroxyethyl-*tert*-oxanilic acid ranged from 65 to 120% and 73 to 115% for *tert*-sulfonic acid.

The LOQ is 0.01 mg/kg for both 1-hydroxyethyl-*tert*-oxanilic acid and *tert*-sulfonic acid, equivalent to 0.02 mg/kg acetochlor for each compound. The % RSD for 1-hydroxyethyl-*tert*-oxanilic acid at different fortification levels in different matrices ranged from 0.85 to 30%. The % RSD for *tert*-sulfonic acid at different fortification levels in different matrices ranged from 0.0 to 29.3%.

Table 59 Recovery data obtained during validation of RAM-280-01 and RAM 280-02 (*Robinson 1996 RAM-280-01, Robinson 1998 RAM-280-02*)

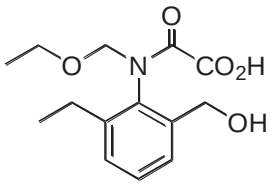
Crop matrices	Fortification level	N	Average Recovery (%)[a]	% RSD	Minimum Recovery	Maximum Recovery	Reference
	(mg/kg)				(%)	(%)	
1-hydroxyethyl-*tert*-oxanilic acid							
Sugar Beet Root	0.01	4	68	15	56	79	RAM-280-01
	0.05	2	84	0.8	83	84	
	0.1	2	86	2.5	84	87	
	0.2	2	99	4.3	96	102	
Sugar Beet Top	0.2	1	65	NA	NA	NA	RJ3114B
Pea Seed	0.02	2	80	0.9	79	80	RJ2262B
	0.1	2	104	6.8	99	109	
Potato	0.01	4	101	4	95	104	RJ2543B
	0.02	5	89	4.3	83	93	RJ2567B
	0.1	9	89	3.2	85	93	
	0.5	1	87	NA	NA	NA	
Potato Tuber	0.2	1	73	NA	NA	NA	RJ3114B
Sunflower Seed	0.01	4	96	2.3	94	99	RJ2560B
	0.02	4	88	16	72	103	RJ2568B
	0.1	11	81	9.6	65	88	
	0.5	1	99	NA	NA	NA	
Sunflower Meal	0.02	5	86	7	79	94	RJ2568B
	0.1	4	84	15	66	92	
	0.5	1	76	NA	NA	NA	
Sunflower Oil	0.02	3	115	4.1	111	120	RJ2568B
	0.1	4	120	0.8	119	121	
	0.5	1	109	NA	NA	NA	
Sweetcorn Kernel on the	0.01	4	79	9.2	72	89	RAM-280-01
Cob	0.05	2	79	12.5	72	86	
	0.1	2	91	9.3	85	97	
	0.2	2	96	2.9	94	98	
Corn Forage	0.01	4	69	14	56	79	RAM-280-01
	0.05	2	82	13	74	89	RJ2078B
	0.1	2	82	11.1	76	89	
	0.2	10	88	9.4	73	98	
Sweet Corn Forage	0.02	4	72	10	63	81	RJ2078B

Crop matrices	Fortification level	N	Average Recovery (%)[a]	% RSD	Minimum Recovery	Maximum Recovery	Reference
	(mg/kg)				(%)	(%)	
	0.05	4	80	6.3	76	87	
	0.1	4	80	30	46	96	
Sweet Corn Stover	0.02	3	89	14	78	103	RJ2078B
	0.05	5	92	12	76	106	
	0.1	4	86	12	78	101	
Sweet Corn Grain	0.02	3	82	12	71	90	RJ2078B
	0.05	3	99	19	80	118	
	0.1	3	99	20	80	120	
Dried Shelled Bean Seed	0.02	2	82	6.1	78	85	RJ2261B
	0.05	2	93	5.4	89	96	
	0.1	1	105	NA	NA	NA	
Soya bean Seed	0.01	42	99	5.4	93	104	RAM-280-01
	0.05	2	87	3.3	85	89	
	0.1	2	110	8.4	103	116	
	0.2	2	114	2.5	112	116	
tert-sulfonic acid							
Sugar Beet Root	0.01	4	84	29	50	108	RAM-280-01
	0.05	2	88	16	78	98	
	0.1	2	94	9	88	100	
	0.2	2	99	5.7	95	103	
Sugar Beet Top	0.2	1	80	NA	NA	NA	RJ3114B
Pea Seed	0.02	2	75	7.5	71	79	RJ2262B
	0.1	2	115	15	103	127	
Potato	0.01	4	92	2.9	90	96	RJ2543B
	0.02	5	87	7	77	92	RJ2567B
	0.1	9	95	7.8	86	105	
	0.5	1	96	NA	NA	NA	
Potato Tuber	0.2	1	80	NA	NA	NA	RJ3114B
Sunflower Seed	0.01	4	94	2.6	91	97	RJ2560B
	0.02	4	88	9.3	82	100	RJ2568B
	0.1	7	78	7	69	85	
	0.5	1	96	NA	NA	NA	
Sunflower Meal	0.02	5	82	6.3	77	89	RJ2568B
	0.1	4	77	14	60	83	
	0.5	1	74	NA	NA	NA	
Sunflower Oil	0.02	3	104	5.4	98	109	RJ2568B
	0.1	4	103	9.1	91	111	
	0.5	1	105	NA	NA	NA	
Sweetcorn Kernel on the	0.01	4	91	9.9	79	101	RAM-280-01
Cob	0.05	2	86	1.6	85	87	
	0.1	2	91	4.7	88	94	
	0.2	2	91	0	91	91	
Corn Forage	0.01	4	100	7.2	92	109	RAM-280-01
	0.05	2	97	5.1	93	100	RJ2078B
	0.1	2	98	2.2	96	99	
	0.2	10	89	8.3	75	99	
Sweet Corn Forage	0.02	4	92	16	72	104	RJ2078B
	0.05	4	92	13	76	104	
	0.1	4	82	16	62	90	
Sweet Corn Stover	0.02	2	89	24	74	104	RJ2078B
	0.05	5	87	18	77	114	
	0.1	4	82	16	62	90	
Sweet Corn Grain	0.02	3	76	7.5	71	82	RJ2078B
	0.05	3	84	5.2	79	87	
	0.1	3	73	26	51	84	
Dried Shelled Bean Seed	0.02	2	97	12	89	105	RJ2261B

Crop matrices	Fortification level	N	Average Recovery (%)[a]	% RSD	Minimum Recovery	Maximum Recovery	Reference
	(mg/kg)				(%)	(%)	
	0.05	2	101	12	92	109	
	0.1	1	115	NA	NA	NA	
Soya bean Seed	0.01	4	102	6.8	96	112	RAM-280-01
	0.05	2	96	15	85	106	
	0.1	2	97	5.8	93	101	
	0.2	2	87	9	81	92	

RES-004-90 (Autry and Steinmetz 1990 MSL-11963), RES-004-90 (Autry and Steinmetz 1990 MSL-12139), RES-004-90 (Kerregan and Lauer 1992 MSL-12091)

An analytical method was developed for determining acetochlor metabolites containing the EMA and HEMA moieties in wheat, sorghum and soya beans. The RAC sample is extracted with CH3CN/H2O (80:20 v/v), filtered, and concentrated on a rotary evaporator. The concentrated extract is then hydrolysed with 50% NaOH and the formed EMA and HEMA distilled into acid (2.5 N H2SO4). The distillate is partitioned with methylene chloride, made basic and partitioned again with methylene chloride. The sample is solvent exchanged from methylene chloride into the HPLC mobile phase, filtered through a 0.2 µm filter for analysis by HPLC on a SCX ion exchange column and quantitated using an electrochemical detector. Representative compounds that generate EMA (*tert*-sulfonic acid), HEMA (1-hydroxyethyl-*tert*-oxanilic acid) and HMEA (hydroxymethyl-*ter*t-oxanilic acid) on hydrolysis were used as reference materials for fortification and method validation.



HMEA (hydroxymethyl-*tert*-oxanilic acid)

Method recoveries were conducted in wheat (forage, straw, and grain), soya bean (forage, hay, and grain), and sorghum (forage, hay, silage, fodder, and grain). The average recoveries for 1-hydroxyethyl-*tert*-oxanilic acid ranged from 64.9 to 81.2%; 71.0 to 99.2% for *tert*-sulfonic acid and 62.2 to 84.7% for hydroxymethyl-*ter*t-oxanilic acid. The LOQ was 0.01 mg/kg for each analyte. The % RSD for 1-hydroxyethyl-*tert*-oxanilic acid ranged from 6.1 to 14.2%; for *tert*-sulfonic acid from 2.9 to 17.5% and for hydroxymethyl-*ter*t-oxanilic acid 1.1 to 14.4%.

Table 60 Recovery data for method *RES-004-90*

Matrix	Fortification Level (mg/kg)	N	Average Recovery (%)	% RSD	Minimum Recovery%	Maximum Recovery%	Reference
1-hydroxyethyl-*tert*-oxanilic acid							
Wheat forage	0.01–1.0	28	71.8	11	51.1	87.0	MSL-11963
	0.05	9	81.2	9	63.6	92.6	MSL-12139
Wheat straw	0.01–1.0	24	70.5	9.5	59.6	89.0	MSL-11963
	0.05	10	68.5	10.8	53.8	85.8	MSL-12139
Wheat grain	0.01–1.0	25	74.9	12.5	55.6	101	MSL-11963
	0.05	7	70.7	10.7	53.8	78.8	MSL-12139
Soya bean forage	0.01–1.0	23	80	9.8	59.9	97.3	MSL-11963
	0.05	5	64.9	6.1	58	73.4	MSL-12139
Soya bean hay	0.01–1.0	17	76.4	11.2	62.0	91.4	MSL-11963
	0.05	8	73.9	6.6	65.6	85.4	MSL-12139
Soya bean grain	0.01–1.0	21	76.6	14.2	55.2	98.3	MSL-11963
	0.05	5	74.4	13.4	58.8	89.2	MSL-12139
Sorghum forage	0.01–1.0	42	75.8	7.0	59.6	91.0	MSL-11963

Matrix	Fortification Level (mg/kg)	N	Average Recovery (%)	% RSD	Minimum Recovery%	Maximum Recovery%	Reference
Sorghum hay	0.01–1.0	40	73.2	8.2	62.9	89.0	MSL-11963
Sorghum silage	0.01–1.0	42	74.3	7.8	61.6	83.6	MSL-11963
	0.05	7	68.7	9.5	54.2	84	MSL-12139
Sorghum fodder	0.01–1.0	40	74	9.9	58.7	87.6	MSL-11963
Sorghum grain	0.01–1.0	48	76.5	7.8	64.0	88.4	MSL-11963
	0.05	7	75.2	6.1	67.4	83.8	MSL-12139
tert-sulfonic acid							
Wheat forage	0.01–1.0	28	81.9	16.2	60.8	111.7	MSL-11963
	0.05	9	90.8	9.2	72	105.6	MSL-12139
Wheat straw	0.01–1.0	24	71	13.5	52.6	92.0	MSL-11963
	0.05	10	93.5	12.4	76.8	113.2	MSL-12139
Wheat grain	0.01–1.0	25	89.4	11.9	65.3	107.7	MSL-11963
	0.05	7	89.5	11.5	73	99.2	MSL-12139
Soya bean forage	0.01–1.0	23	77.9	10.5	62	94.0	MSL-11963
	0.05	5	80.9	7.4	73.2	90.4	MSL-12139
Soya bean hay	0.01–1.0	17	82.9	9.9	69.4	98.8	MSL-11963
	0.05	8	99.2	10.9	82	112	MSL-12139
Soya bean grain	0.01–1.0	21	82.8	12.4	60.8	102.5	MSL-11963
	0.05	5	88.6	2.9	83.6	90.8	MSL-12139
Sorghum forage	0.01–1.0	42	84.6	10.3	70.7	99.6	MSL-11963
Sorghum hay	0.01–1.0	40	98	10.3	71.6	118.0	MSL-11963
Sorghum silage	0.01–1.0	42	96.4	10.7	59.8	114.8	MSL-11963
	0.05	7	89.3	17.5	71.4	120	MSL-12139
Sorghum fodder	0.01–1.0	40	97.8	8.7	81.6	123.7	MSL-11963
Sorghum grain	0.01–1.0	48	84.8	13.7	68.4	114.6	MSL-11963
	0.05	7	91.7	9	75.8	100.8	MSL-12139
hydroxymethyl-*tert*-oxanilic acid							
Wheat forage	0.01–1.0	28	75	12.1	56.7	104.0	MSL-11963
	0.05	9	81.8	12.5	59	91.4	MSL-12139
Wheat straw	0.01–1.0	24	75.7	7.9	68.3	89.3	MSL-11963
	0.05	10	67.8	14.4	43	88.6	MSL-12139
Wheat grain	0.01–1.0	25	76.5	9.4	60.5	95.5	MSL-11963
	0.05	7	76.7	5.6	66	84.4	MSL-12139
Soya bean forage	0.01–1.0	23	75.8	9.4	60.5	88.2	MSL-11963
	0.05	5	62.2	6	54.6	68.4	MSL-12139
Soya bean hay	0.01–1.0	17	72.2	8.6	61.0	79.8	MSL-11963
	0.05	8	63.5	7.6	47	73.4	MSL-12139
Soya bean grain	0.01–1.0	21	73.4	12	52.4	84.6	MSL-11963
	0.05	5	77.8	1.1	76.6	79.2	MSL-12139
Sorghum forage	0.01–1.0	42	82.5	7.7	66.2	99.6	MSL-11963
Sorghum hay	0.01–1.0	40	81.3	8.8	64.7	91.9	MSL-11963
Sorghum silage	0.01–1.0	42	82.3	6.0	72.0	91.8	MSL-11963
	0.05	7	67.9	8.4	54.2	78	MSL-12139
Sorghum fodder	0.01–1.0	40	83.1	7.5	68	102.9	MSL-11963
Sorghum grain	0.01–1.0	48	84.7	6.5	74.9	100.4	MSL-11963
	0.05	7	74.3	6.7	67.2	81.6	MSL-12139

ES-ME-1001-02 (Lauer et al. 2007 MSL-20269)

The method is a variation on earlier methods for metabolites hydrolysable to EMA and HEMA. Plant materials are cryogenically homogenized (dry ice). Residues in plant matrices are extracted with solvent (CH_3CN/H_2O 80:20 v/v), filtered, concentrated by rotary evaporation and the residue hydrolysed with base (50% NaOH) to form EMA and HEMA. The latter are steam distilled into dilute acid (2.5 N H_2SO_4) and the acid distillate partitioned with methylene chloride, discarding the organic (methylene chloride) layer. The pH of the aqueous layer is adjusted to be basic and the EMA and HEMA extracted with methylene chloride. The methylene chloride is dried over anhydrous Na_2SO_4 prior to addition of acetonitrile and rotary evaporation to remove methylene chloride. Additional

acetonitrile is added and the solution diluted with deionised water to give a solvent composition of approximately 10% acetonitrile/90% deionised water. Quantification is by LC-MS/MS using external standards (EMA 136→91 amu; HEMA 152→134 amu). Residues are converted to acetochlor equivalents using the following factors:

- Acetochlor equivalents = EMA × 1.995
- Acetochlor equivalents = HEMA × 1.784

Representative compounds that generate EMA (*tert*-sulfonic acid) and HEMA (1-hydroxyethyl-*tert*-oxanilic acid) on hydrolysis were used as reference materials for fortification and method validation.

The LOQs for 1-hydroxyethyl-*tert*-oxanilic acid are 0.002 mg/kg for sorghum grain, 0.003 mg/kg for sorghum forage, 0.01 mg/kg for sorghum stover, 0.001 mg/kg for maize grain, 0.009 mg/kg for maize forage and 0.01 mg/kg for maize stover. The LOQs for *tert*-sulfonic acid are 0.01 mg/kg for sorghum grain, 0.005 mg/kg for sorghum forage, 0.015 mg/kg for sorghum stover, 0.001 mg/kg for maize grain, 0.04 mg/kg for maize forage and 0.03 mg/kg for maize stover.

The maximum RSDs for 1-hydroxyethyl-*tert*-oxanilic acid in the different matrices was 20.25%, however, approximately two-thirds of the RSDs were < 10%. The maximum RSD for *tert*-sulfonic acid in the different matrices was 21.62%; again, approximately two-thirds of the RSDs were < 10%. The mean percent recovery values for 1-hydroxyethyl-*tert*-oxanilic acid ranged from 67.1 to 106.9%. The mean

Table 61 Recovery data from reports MSL-18670 (sorghum) and MSL-20269 (maize) for method ES-ME-1001-02

Matrix	Fortification Level (mg/kg)	N	Average Recovery (%)[a]	% RSD	Minimum Recovery %[c]	Maximum Recovery %[c]
1-hydroxyethyl-*tert*-oxanilic acid						
Sorghum Grain	0.01	7	82.0	4.0	72.1	96.4
	0.05	6	85.9	11.6		
	0.1	3	77.1	8.2		
	0.2	3	83.4	5.5		
Sorghum forage	0.01	3	78.0	18.3	64.9	92.5
	0.05	3	73.3	7.2		
	0.1	3	86.5	7.1		
	0.2	3	79.8	3.1		
	0.5	3	78.5	4.2		
	1	1	79.9	NA		
	2	1	77.0	NA		
Sorghum stover	0.01	3	78.5	20.2	65.2	96.0
	0.05	2	75.6	6.7		
	0.1	2	72.0	2.8		
	0.2	1	73.7	NA		
	0.5	3	79.9	8.5		
	1	1	79.9	NA		
	2	1	67.1	NA		
Sorghum Flour	0.01–0.05	2	86.0	NA	83.4	88.5
Sorghum Bran	0.01–0.05	2	87.5	NA	76.6	98.4
Corn Forage	0.005	1	94.1	NA	NA	NA
	0.01	3	83.4	7.9	75.8	87.7
	0.05	2	88.5	1.0	87.8	89.2
	0.1	5	87.2	5	81.1	91.2
	0.2	3	82.1	9.6	73.5	88.8
	0.5	3	85.9	15.1	76.8	100.7
	1	3	106.9	17.2	93.6	128.0
	2	4	82.3	8.9	72.0	88.9

Matrix	Fortification Level (mg/kg)	N	Average Recovery (%)[a]	% RSD	Minimum Recovery %[c]	Maximum Recovery %[c]
	5	1	88.4	NA	NA	NA
Corn Grain	0.005	4	83.6	10.2	73.4	94.2
	0.01	5	87.7	7.6	78.9	95.2
	0.05	5	86.1	17.1	74	103.8
	0.1	4	96.4	3.4	92	99
	0.2	3	76	3.5	73	78
	0.4	1	91	NA	NA	NA
	1	1	89.6	NA	NA	NA
	2	1	80.5	NA	NA	NA
Corn Stover	0.005	2	87	19.5	75	99
	0.01	4	84.3	6.3	77.7	89.1
	0.05	5	81.1	10.4	67.2	88.7
	0.1	4	83.8	5.6	76.9	87.0
	0.2	4	79.7	5.2	76	84
	0.5	3	86.8	8.9	82.4	95.8
	1	5	91.8	20.2	78.1	124.0
	2	1	71	NA	NA	NA
	5	1	83.5	NA	NA	NA
tert-sulfonic acid						
Sorghum Grain	0.01	7	100.4	5.4	87.9	108.9
	0.05	6	99.4	7.2		
	0.1	3	95.4	7.5		
	0.2	3	93.3	5.1		
Sorghum forage	0.01	3	107.5	16.7	70.0	118.7
	0.05	3	82.7	15.5		
	0.1	3	99.3	9.3		
	0.2	3	95.8	3.6		
	0.5	3	97.4	4.5		
	1	1	87.7	NA		
	2	1	94.9	NA		
Sorghum stover	0.01	3	96.8	21.6	69.45	101.2
	0.05	3	91.8	2.6		
	0.1	2	99.9	5.4		
	0.2	2	96.7	0.2		
	0.5	3	96.0	2.1		
	1	1	96.5	NA		
	2	1	87.0	NA		
Sorghum Flour	0.01–0.05	2	99.5	NA	94.5	96.6
Sorghum Bran	0.01–0.05	2	95.5	NA	90.1	109.0
Corn Forage	0.005	1	104.2	NA	NA	NA
	0.01	3	92.3	18.8	81.4	112.3
	0.05	2	93.6	6.8	89.1	98.1
	0.1	5	95.7	5.2	89	101.6
	0.2	3	87.3	13.9	73.5	96.3
	0.5	3	88.5	15.8	80.4	104.7
	1	3	98.2	15.7	81.0	110.8
	2	4	78.0	4.7	77.5	85.4
	5	1	90.8	NA	NA	NA
Corn Grain	0.005	4	95.9	4.8	89.2	99.8
	0.01	5	93.0	18.4	68.3	115
	0.05	5	86.9	12.5	72.8	98.2
	0.1	4	91.9	10.7	83.2	101
	0.2	3	75	9.7	67.5	82
	0.4	1	77.9	NA	NA	NA
	1	1	80.6	NA	NA	NA
	2	1	75	NA	NA	NA
Corn Stover	0.005	2	90.4	7.2	85.7	95
	0.01	4	91.6	6.9	84.0	99.0
	0.05	5	86.7	16.7	61.2	97.2

Matrix	Fortification Level (mg/kg)	N	Average Recovery (%)[a]	% RSD	Minimum Recovery %[c]	Maximum Recovery %[c]
	0.1	4	91.2	7.3	81.5	96.5
	0.2	4	83.7	6.3	79.0	91.2
	0.5	3	89.0	12.4	80.2	101.4
	1	5	90.8	9.8	84.5	105.9
	2	1	84.5	NA	NA	NA
	5	1	91.8	NA	NA	NA

ES-ME-1215-01 (Allan et al. 2008 MSL-20718), ES-ME-1215-02 (Allan et al. 2009 MSL-21172)

The method has been applied to cotton and soya bean commodities. Samples are cryogenically processed with dry ice (25% w/w). Storage of the homogenised sample in the freezer overnight allows the dry ice to sublime. For dry matrices, residues are extracted by blending with CH_3CN/H_2O (80:20 v/v) followed by filtration and the extract concentrated by rotary evaporation. For oily matrices, residues are extracted by shaking with CH_3CN/H_2O (80:20 v/v) followed by centrifugation to separate the phases. The CH_3CN/H_2O layer is retained and extraction process repeated on the oil layer. The combined CH_3CN/H_2O extracts are concentrated by rotary evaporation. For both dry and oily matrices, the concentrated residues are hydrolysed by adding 50% NaOH, heating and distilling the EMA and HEMA formed into 2.5 N H_2SO_4. The pH of the distillate is adjusted with $NaOH/NaHCO_3$ prior to analysis using on-line SPE (Oasis HLB) clean-up with LC-MS/MS. The following ions and transition ions are monitored:

- EMA parent ion 136 amu, product ion 91 amu
- HEMA parent ion 152 amu, product ion 134 amu.

LOQ 0.005 mg/kg (0.01 mg/kg for soy hay and forage). Range 0.005 to 5 mg/kg.

The % RSD for soya bean seed was less than 10% for both 1-hydroxyethyl-*tert*-oxanilic acid and *tert*-sulfonic acid, except for the 0.2 mg/kg *tert*-sulfonic acid fortification of seed, for which the % RSD for the two fortified samples was 22.8%. For soya bean forage and hay, maximum % RSDs were 14.4 and 14.8%, respectively, for both analytes except for *tert*-sulfonic acid in soya bean hay where levels were 29.1 and 24.6% at the 0.01 and 0.05 mg/kg fortification levels, respectively. In undelinted cotton seed, % RSDs were less than 10% for both 1-hydroxyethyl-*tert*-oxanilic acid and *tert*-sulfonic acid, except for *tert*-sulfonic acid at the 0.4 mg/kg fortification level where it was 11.9%. In gin by-products, % RSDs were less than 10% for 1-hydroxyethyl-*tert*-oxanilic acid; however, % RSDs ranged from 4.4 to 12.0% for *tert*-sulfonic acid at all levels except at the 0.005 fortification level where it was 49.6%. The mean percent recovery values for 1-hydroxyethyl-*tert*-oxanilic acid in RACs and processed fractions ranged from 71.9 to 109%. The mean percent recovery values for *tert*-sulfonic acid in the different matrices ranged from 72.6 to 96.5%.

Table 62 Recoveries for cotton (MSL-20718) and soya bean (MSL-20719) commodities obtained when using method ES-ME-1215

Matrix	Fortification level (mg/kg)	N	Average Recovery (%)	% RSD	Minimum Recovery%	Maximum Recovery%
1-hydroxyethyl-*tert*-oxanilic acid						
Gin by-products	0.005	4	74.4	3.2	71.2	76.6
	0.01	10	71.9	2.1	70.4	74
	0.4	8	76.8	7.8	70.9	90.2
	2	3	83.3	8.6	76.6	90.8
	4	5	78.7	2.3	77	81.4
Undelinted seeds	0.005	24	93.7	3.9	88	102.4

Matrix	Fortification level (mg/kg)	N	Average Recovery (%)	% RSD	Minimum Recovery%	Maximum Recovery%
	0.02	8	93	5.0	86.6	99.8
	0.4	10	94.4	6.1	83.8	101.7
	0.5	1	93.6	NA	NA	NA
	2	7	96.1	4.7	88.2	101.3
	4	1	96	NA	NA	NA
Processed ginned seeds	0.005	1	91.7	NA	NA	NA
	2	1	99.8	NA	NA	NA
Cotton hulls	0.005	1	92.4	NA	NA	NA
	2	1	99.6	NA	NA	NA
Cottonseed meal	0.005	1	103.9	NA	NA	NA
	2	1	104.8	NA	NA	NA
Cottonseed refined oil	0.005	1	101	NA	NA	NA
	2	1	108.8	NA	NA	NA
Soya bean seed	0.005	7	103.9	6.2	97.7	112.6
	0.01	4	96.7	8.1	90.3	108.2
	0.05	2	99.2	2.1	97.7	100.7
	0.1	1	93	NA	NA	NA
	0.2	2	91.3	8.0	86.1	96.5
	0.5	3	94.6	6.1	88.6	100.2
	1	2	104.2	2.1	102.7	5.7
	2	2	93.2	4.1	90.5	95.9
Soya bean forage	0.01	3	87.6	2.3	85.7	89.7
	0.05	2	96.3	2.0	95	97.7
	0.1	1	95.6	NA	NA	NA
	0.2	2	86.9	0.3	85.7	86.1
	0.5	2	89.2	7.7	84.3	94
	1	3	96.1	6.4	89.1	99.7
	2	3	87.7	6.3	81.4	91.8
	5	2	89.4	3.4	87.2	91.5
	10	2	89.6	6.5	85.4	93.6
	20	2	90.9	6.7	86.6	95.2
	50	1	101.6	NA	NA	NA
	80	1	94.2	NA	NA	NA
	100	2	90.6	7.5	85.8	95.4
	200	1	88.6	NA	NA	NA
Soya bean hay	0.01	4	90.2	13.5	79.1	107.5
	0.05	2	77.7	12.4	70.9	84.6
	0.1	1	86	NA	NA	NA
	0.2	2	88.3	10.2	81.9	94.6
	0.5	2	92.2	6.4	88	96.3
	1	2	100.6	4.0	97.7	103.5
	2	3	90.1	10.8	78.9	96.6
	5	2	89.8	5.2	83.6	90
	10	2	92.5	0.8	92	93.1
	20	2	86.4	2.1	85.2	87.7
	50	1	82.7	NA	NA	NA
	80	1	81.1	NA	NA	NA
	100	2	92.6	0.4	92.3	92.9
	200	1	83.7	NA	NA	NA
Soya bean refined oil	0.005	1	101.1	NA	NA	NA
	0.05	1	102.1	NA	NA	NA
Soya bean meal	0.01	1	91.5	NA	NA	NA
	0.1	1	96.3	NA	NA	NA
Soya bean hulls	0.05	1	98.3	NA	NA	NA
	0.5	1	97.6	NA	NA	NA
Soya bean forage	0.1	2	79.5	1.7	78.5	80.4
	1.5	2	80.8	0.8	80.3	81.2
Soya bean grain	0.05	2	84.8	7.6	80.2	89.3
	0.1	2	88.2	4.4	85.4	90.9
Soya bean stover	1.5	4	76.7	4.3	72.6	80

Matrix	Fortification level (mg/kg)	N	Average Recovery (%)	% RSD	Minimum Recovery%	Maximum Recovery%
tert-sulfonic acid						
Gin by-products	0.005	4	74.7	49.6	26	116
	0.01	10	84.9	12.0	70.7	99.5
	0.4	8	77.8	4.6	71.2	82.2
	2	3	80.1	11.1	71.1	88.9
	4	5	82.8	4.4	78.9	87.8
Undelinted seeds	0.005	24	82	9.1	66.6	97.1
	0.02	8	82.1	7.4	73.2	90.5
	0.4	10	78.1	11.9	55.7	87.4
	0.5	1	83.4	NA	NA	NA
	2	7	82	7.7	68.2	86.7
	4	1	82.3	NA	NA	NA
Processed ginned seeds	0.005	1	79.3	NA	NA	NA
	2	1	85.9	NA	NA	NA
Cotton hulls	0.005	1	82.5	NA	NA	NA
	2	1	84.3	NA	NA	NA
Cottonseed meal	0.005	1	88.5	NA	NA	NA
	2	1	92.6	NA	NA	NA
Cottonseed refined oil	0.005	1	86.5	NA	NA	NA
	2	1	92	NA	NA	NA
Soya bean seed	0.005	7	96.1	8.1	90.4	110
	0.01	4	83.7	2.2	82.6	86.5
	0.05	2	86.2	1.2	85.5	87
	0.1	1	82.4	NA	NA	NA
	0.2	2	72.6	22.8	60.9	84.4
	0.5	3	83.9	3.1	82.1	86.8
	1	2	95.7	0.4	95.5	96
	2	2	81.1	3.4	79.1	83
Soya bean forage	0.01	3	85.2	6.7	80.5	91.6
	0.05	2	78.2	14.4	70.2	86.2
	0.1	1	76.7	NA	NA	NA
	0.2	2	96.5	9.2	90.3	102.8
	0.5	2	85.8	2.7	84.1	87.4
	1	3	89	4.5	84.5	92.3
	2	3	80.2	6.5	74.5	84.6
	5	2	92.8	12.0	84.9	100.6
	10	2	81.3	0.2	81.2	81.4
	20	2	82.2	1.1	81.6	82.9
	50	1	93.5	NA	NA	NA
	80	1	89	NA	NA	NA
	100	2	81.4	0.7	81	81.8
	200	1	83.9	NA	NA	NA
Soya bean hay	0.01	4	88.7	29.1	63.6	118
	0.05	2	73.6	24.6	60.8	86.4
	0.1	1	79.5	NA	NA	NA
	0.2	2	80.9	14.8	72.5	89.4
	0.5	2	85.4	9.3	79.8	91.1
	1	2	96	2.4	94.4	97.7
	2	3	85.3	6.5	82.1	91.7
	5	2	89.4	3.8	87	91.9
	10	2	86.9	5.0	83.8	90
	20	2	83.1	0.8	82.6	83.5
	50	1	83.2	NA	NA	NA
	80	1	81.9	NA	NA	NA
	100	2	84.8	3.0	83	86.6
	200	1	81.8	NA	NA	NA
Soya bean refined oil	0.005	1	80.2	NA	NA	NA
	0.05	1	88.3	NA	NA	NA
Soya bean meal	0.01	1	84.3	NA	NA	NA
	0.1	1	89.4	NA	NA	NA

Matrix	Fortification level (mg/kg)	N	Average Recovery (%)	% RSD	Minimum Recovery%	Maximum Recovery%
Soya bean hulls	0.05	1	85.6	NA	NA	NA
	0.5	1	85.2	NA	NA	NA

AG-ME-1467 (MSL-24197), AG-ME-1467-01 (23-3.1.1-AG-ME-1467-01 Foster 2012)

Crop matrices are cryogenically milled (with dry ice) and samples extracted with methanol/water. The aqueous methanol is recovered by centrifugation and the residues hydrolysed by addition of NaOH. On completion of the hydrolysis the pH is quenched by addition of H_2SO_4. An aliquot is mixed with internal standard (^{13}C-EMA, ^{13}C-HEMA) and processed through an Oasis MCX SPE plate. The eluate is mixed with formic acid and analysed using LC-MS/MS with electrospray ionisation. The following ions and transition ions are monitored:

- EMA 136→91 amu; HEMA 152→134 amu.

Representative compounds that generate EMA (*tert*-sulfonic acid) and HEMA (1-hydroxyethyl-*tert*-oxanilic acid) on hydrolysis were used as reference materials for fortification and method validation.

Results obtained were within guideline requirements (60–120%). The LOQ for 1-hydroxyethyl-*tert*-oxanilic acid is 0.009 mg/kg for nutmeat, 0.003 mg/kg for peanut hay, 0.0015 mg/kg sugar beet roots and 0.001 mg/kg for tops. The LOQ for *tert*-sulfonic acid is 0.009 mg/kg for nutmeat, 0.003 mg/kg for peanut hay, 0.0016 mg/kg sugar beet roots and 0.004 mg/kg for tops. The %RSDs for 1-hydroxyethyl-*tert*-oxanilic acid ranged from 4.9 to 17.1% for raw agricultural commodities (RACs) and from 4.2 to 16.9% for processed fractions. The relative standard deviations for *tert*-sulfonic acid ranged from 3.4 to 16.5% for RACs and from 2.6 to 11.1% for processed fractions. The mean percent recovery values for 1-hydroxyethyl-*tert*-oxanilic acid ranged from 66.8 to 101.4% for RACs and from 80.7 to 109.3% for processed fractions. The mean percent recovery values for *tert*-sulfonic acid ranged from 66.3 to 100.7% for RACs and from 71.7 to 103.1% for processed fractions.

Table 63 Recovery data for method AG-ME-1467 from reports MSL-24197 (peanut) and MSL-24198 (sugar beet)

Matrix	Fortification level (mg/kg)	Number of tests	Average recovery (%)	% RSD	Minimum recovery	Maximum recovery
1-hydroxyethyl-*tert*-oxanilic acid						
Peanut hay	0.01	48	90.4	17.1	62	118
	0.1	48	88.6	8.0	74.5	100
	4	9	92.3	5.4	86.5	100.8
Peanut nutmeat	0.01	48	95	13.1	66.4	119
	0.1	47	101.4	7.5	88.7	123.7
	4	9	98.5	6.2	91.9	111
Sugar beet roots	0.01	60	91	8.3	70.9	108
	0.1	60	81.7	4.9	74.4	90.1
	4	9	81.3	5.7	76.3	89.3
Sugar beet tops	0.01	54	91	11.9	63.2	102
	0.1	54	71.5	8.5	60.2	80.9
	4	9	66.8	8.9	60.5	77
Peanut dry roasted	0.01	6	97.2	9.7	86	108
	0.1	6	102.9	6.6	94.7	111
Peanut meal	0.01	5	86.4	15.9	69	101
	0.1	6	101.8	4.2	95.4	108.4
Peanut butter	0.01	6	102.3	6.1	90.6	108.6
	0.1	6	98.3	9.0	83.9	108
Peanut RAC	0.01	9	84	16.3	64.9	98
	0.1	9	99.3	5.5	90.1	108.3
Peanut RBD oil	0.01	6	109.3	4.7	104	117

Matrix	Fortification level (mg/kg)	Number of tests	Average recovery (%)	% RSD	Minimum recovery	Maximum recovery
	0.1	6	93	16.9	63.9	108
Sugar beet dried pulp	0.01	12	92.5	13.3	67.9	108
	0.1	12	91.7	5.5	84	101
Sugar beet white granulated	0.01	11	96.4	6.8	89	109
sugar	0.1	12	90.5	5.2	81.2	99.5
Sugar beet molasses	0.01	12	86.7	7.4	74.3	96.5
	0.1	12	80.7	7.8	74.1	91.1
Sugar beet RAC	0.01	12	84.3	10.1	66.2	95.8
	0.1	12	79.4	5.1	74.1	84.8
tert-sulfonic acid						
Peanut hay	0.01	46	83.6	14.3	63.2	115
	0.1	48	82.8	12.4	62.9	110
	4	9	87.9	8.2	79.8	101.5
Peanut nutmeat	0.01	48	91.9	16.5	61.9	120
	0.1	47	92.6	11.6	60.9	115
	4[d]	9	100.7	8.5	86.5	114.5
Sugar beet roots	0.01	60	72	9.1	60.2	90.5
	0.1	59	69.3	7.0	60.2	78.3
	4	9	68.9	4.1	65.2	72.9
Sugar beet tops	0.01	54	71.6	11.8	61.2	94.3
	0.1	54	66.3	5.0	59.5	73.3
	4	9	69.3	3.4	66.7	72.8
Peanut dry roasted	0.01	6	100.7	2.6	97.6	105
	0.1	6	94.8	6.7	88.2	103
Peanut meal	0.01	6	96.2	8.9	84.5	110
	0.1	6	91.6	3.8	86.9	96.1
Peanut butter	0.01	6	97.4	4.7	92.8	103
	0.1	6	91.6	9.0	75.4	97.8
Peanut RAC	0.01	9	97.8	10.8	79.3	112
	0.1	9	96.5	3.3	92.5	102
Peanut RBD oil	0.01	6	103.1	9.4	93.4	112
	0.1	5	96.8	7.5	89.4	107
Sugar beet dried pulp	0.01	12	90.7	8.0	76.3	104
	0.1	12	85	7.0	75.8	96.7
Sugar beet white granulated	0.01	12	91.9	10.5	78.9	113
sugar	0.1	12	84.8	5.9	76.4	91.7
Sugar beet molasses	0.01	12	84.5	11.1	70.6	101
	0.1	12	71.7	7.8	62.4	78.9
Sugar beet RAC	0.01	12	72	8.1	61.7	80.9
	0.1	12	67.5	4.7	62.5	72.4

RES-074-93 (Arras 1995 MRL-14276), RES-074-93 (Arras and Schneider 1996 MRL-14117)

A method was developed for the analysis of metabolites hydrolysable to EMA and HEMA in crop commodities. For crop samples, the method consisted of extraction of the sample with CH_3CN/H_2O (80:20, v/v) followed by filtration and evaporation of the extract to a smaller volume. The concentrated extracts are hydrolysed with base (50% NaOH) and the resulting EMA and HEMA steam-distilled into dilute acid (2.5 N H_2SO_4). The pH of the distillate is adjusted to the basic pH range and the EMA and HEMA partitioned into methylene chloride and back-extracted into aqueous methanol/HCl. The HEMA is converted to its methoxy derivative with methanol in the presence of 4 N HCl (10:3 v/v) to form MEMA. The pH is adjusted and the residues quantified using HPLC-ECD. Residues are converted to acetochlor equivalents using the following factors:

- Acetochlor = 1.995 × EMA
- Acetochlor = 1.633 × MEMA

Representative compounds that generate EMA and HEMA on hydrolysis were used as reference materials for fortification and method validation. For plant commodities are *tert*-sulfonic acid (EMA) and 1-hydroxyethyl-*tert*-oxanilic acid (HEMA).

Method validations and recovery were conducted in oat raw agricultural commodities and processed oat fractions, alfalfa, clover and reported in MSL-14117 (oat commodities), MSL-14118 (oat processed fractions) and MSL-14276 and MSL-14134 (alfalfa and clover). The average recoveries for HEMA ranged from 71.4 to 101.8%; and 87.2 to 121.2% for *tert*- sulfonic acid. Recovery data obtained on numerous matrices from field residue studies were generally satisfactory.

The LOQ for HEMA and EMA in oat matrices was 0.018 mg/kg for HEMA and 0.017 mg/kg for EMA. For alfalfa and clover, LOQ for the HEMA were 0.014 mg/kg, and 0.012 mg/kg, respectively, for EMA.

Oat matrices

The method was validated at 0.01, 0.10, 0.50 and 1.00 mg/kg (acetochlor equivalent) for each of the two metabolite classes. Analytical recoveries from eight laboratory fortified samples of oats grain and oats straw averaged 88.2% with a standard deviation of 10.2% for *tert*-sulfonic acid; and 85.3% with a standard deviation of 9.2% for 1-hydroxyethyl-*tert*-oxanilic acid. For method recovery samples, average recoveries across all fortifications in grain, forage, straw, and processed fractions ranged from 81.6 to 101.8% for 1-hydroxyethyl-*tert*-oxanilic acid and from 102.3 to 116.8% for *tert*-sulfonic acid in all matrices except oat hulls, which had an average recovery of 121.2%. Relative standard deviations for both analytes across all matrices ranged from 1.0 to 17.7%.

Alfalfa and clover matrices

The method was validated at 0.01, 0.10, 0.20 and 0.50 mg/kg (acetochlor equivalent) for each of the two metabolite classes. Analytical recoveries from 16 laboratory fortified samples of alfalfa hay and clover forage averaged 101.0% with a standard deviation of 11.4% for *tert*- sulfonic acid; and 76.9% with a standard deviation of 8.0% for 1-hydroxyethyl-*tert*-oxanilic acid. For method recovery samples, average recoveries across all fortifications in alfalfa and clover forage and hay ranged from 69.1 to 85.5% for 1-hydroxyethyl-*tert*-oxanilic acid and from 84.2 to 108.4% for *tert*- sulfonic acid. Relative standard deviations for both analytes across all matrices ranged from 2.9 to 17.0%.

Table 64 Recovery data for method RES-074-93

Matrix	Fortification level	N	Average Recovery (%)	% RSD	Minimum Recovery %	Maximum Recovery %
1-hydroxyethyl-*tert*-oxanilic acid						
Oat grain	0.01–0.20 [a]	17	95.2	17.7	69.4	129.4
	0.01–0.10 [b]	8	94.7	8.5	84.8	108.2
Oat forage	0.01–0.10	17	92.3	13.1	73.4	122.3
Oat hay	0.04–1.00	16	89.7	10.3	72.5	109
Oat straw	0.01–1.00	17	81.6	9.2	64.5	99.2
Oat hulls	0.01–0.10	4	101.8	7.1	93.4	108.4
Oat flour	0.01–0.04	4	98.1	4.2	95.4	104.3
Oat groats	0.01–0.08	4	88.4	5.2	83.2	94.9
Alfalfa forage	0.01–2.00 [c]	6	75.6	7.3	66.8	85.6
	0.01–2.00 [d]	30	84.2	11.6	66.1	108.8
	0.10 [e]	2	69.9	3.9	68	71.9
Alfalfa hay	0.01–0.50 [c]	8	79.8	9.3	60.1	88.8
	0.01–2.00 [d]	29	81.1	11.3	64.3	106.1
Clover forage	0.01–0.50 [c]	8	72	3.9	66.2	77.6

Matrix	Fortification level	N	Average Recovery (%)	% RSD	Minimum Recovery %	Maximum Recovery %
	0.01–1.00 [d]	19	85.5	13.1	68.6	117.8
Clover hay	0.01–2.00 [c]	5	78.9	17	67.1	108.5
	0.01–2.00 [d]	18	77.1	8.5	65.9	90.9
	0.10 [f]	2	69.1	11.5	74.7	63.4
tert-sulfonic acid						
Oat grain	0.02–0.20 [a]	17	105.4	17.2	68.7	139.5
	0.01–0.10 [b]	8	113.4	4	104.2	116.6
Oat forage	0.01–0.10	17	108.3	15.6	73.2	136.2
Oat hay	0.04–1.00	16	106.4	6.5	96.5	115.8
Oat straw	0.01–1.00	17	104.6	8.4	92.5	119.5
Oat hulls	0.01–0.10	4	121.2	7.8	109.6	126.5
Oat flour	0.01–0.04	4	116.8	3.5	113	120.5
Oat groats	0.01–0.08	4	102.3	1	101.4	103.7
Alfalfa forage	0.01–2.00 [c]	6	108.1	5.9	100.7	118.1
	0.01–2.00 [d]	30	102.6	10.3	74.4	123
	0.10 [e]	2	92	2.9	90.1	93.8
Alfalfa hay	0.01–0.50 [c]	8	104.8	11.4	82	118.7
	0.01–2.00 [d]	29	99.5	9.6	77.1	115.2
Clover forage	0.01–0.50 [c]	8	93.9	9.5	83.9	109.9
	0.01–1.00 [d]	19	108.4	9.9	86.2	123.1
Clover hay	0.01–2.00 [c]	5	101.8	6.1	93.4	109.6
	0.01–2.00 [d]	18	105.1	9.4	86.5	120.2
	0.10 [e]	2	84.2	7.1	88.4	80

[a] Data from MSL-14117.

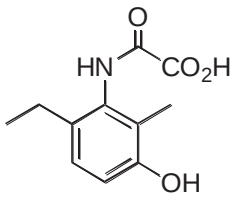
[b] Data from MSL-14118.

[c] Method validation data from Table I and Table II in MSL-14276.

[d] Method recovery data from Table V and Table VI in MSL-14276.

[e] Data from MSL-14134.

(Crook 1992 RJ1257B) 5-hydroxy-sec-oxanilic acid (68)



A method was developed for the analysis of 5-hydroxy-*sec*-oxanilic acid (68) in crops. Samples of crops are extracted by maceration with CH_3CN/H_2O (50:50, v/v), filtered under vacuum, and the filtrate concentrated by rotary evaporated to dryness and redissolved in CH_3CN/H_2O (50:50, v/v). In the case of oil process fractions, extraction involves dissolution in hexane and a water partition. In both cases, an aliquot is taken, diluted, then acidified and partitioned into ethyl acetate. Aqueous process fractions are acidified and partitioned directly into ethyl acetate. The organic layer is recovered, evaporated to dryness and reacted with isobutanol/3M HCl. Samples are again evaporated to dryness and then reacted with N-methyl-N-(tert-butyldimethylsilyl)-trifluoroacetamide (MTBSTFA) at 60–70 °C for one hour. The derivatives are then analysed by GC-MS. The LOQ was set as 0.01 mg/kg.

The % RSDs for 5-hydroxy-*sec*-oxanilic acid (68) at different fortification levels in corn commodities (grain, forage, and fodder) ranged from 7.6 to 23.9%. In all other matrices, RSDs ranged from 2.4% to 26.5%. The mean percent recovery values for 5-hydroxy-*sec*-oxanilic acid (68) at different fortification levels in corn commodities (grain, forage, and fodder) ranged from 71 to 118%. In all other matrices investigated, recoveries ranged from 55 to 120%.

Table 65 Recovery data for 5-hydroxy-*sec*-oxanilic acid in crops and processed maize fractions (Crook *et al.* 1992 RJ1257B)

Crop matrices	Fortification level (mg/kg)	N	Average recovery (%)	% RSD	Reference
Corn Grain	0.01	8	90	7.6	RJ1337B
	0.02	1	71	NA	
	0.05	6	80	20.6	
	0.1	4	84	15.1	
	0.2	1	112	NA	
	0.5	1	118	NA	
	1	1	83	NA	
Corn Forage	0.01	6	94	12.3	RJ1337B
	0.02	1	88	NA	
	0.05	4	88	16.5	
	0.1	5	90	7.6	
	0.2	1	94	NA	
	0.5	1	112	NA	
	1	1	95	NA	
Corn Fodder	0.01	7	105	16.5	RJ1337B
	0.03	1	97	NA	
	0.05	6	97	23.9	
	0.1	6	92	20.6	
	0.2	1	110	NA	
	0.5	1	102	NA	
	1	1	101	NA	
Corn Silage	0.01	2	88	2.4	RJ1337B
	0.02	1	91	NA	
	0.05	2	88	13.7	
	0.1	4	86	26.5	
	0.2	1	72	NA	
Turnip Roots	0.01	3	99	16.7	RJ1257B
	0.05	1	91	NA	
	0.1	1	83	NA	
	0.2	1	80	NA	
	0.5	1	110	NA	
	1	1	99	NA	
Turnip Tops	0.01	4	103	12.3	RJ1257B
	0.05	1	92	NA	
	0.1	1	79	NA	
	0.2	1	91	NA	
	0.5	1	111	NA	
	1	1	101	NA	
Lettuce	0.01	4	99	15.4	RJ1257B
	0.05	1	86	NA	
	0.1	1	89	NA	
	0.2	1	84	NA	
	0.5	1	96	NA	
	1	1	103	NA	
Soy Seed	0.01	3	62	9.8	RJ1257B
	0.05	1	61	NA	
	0.1	1	61	NA	
	0.5	1	70	NA	
	1	1	67	NA	
Soy Hay	0.01	4	71	12.2	RJ1257B
	0.1	1	62	NA	
	0.2	1	72	NA	
	0.5	1	83	NA	
	1	1	96	NA	
Flour	0.01	4	106	15	RJ1257B
	0.05	1	73	NA	
	0.1	1	84	NA	

Crop matrices	Fortification level (mg/kg)	N	Average recovery (%)	% RSD	Reference
	0.2	1	86	NA	
	0.5	1	114	NA	
	1	1	110	NA	
Process Water	0.01	4	95	9.2	RJ1257B
	0.05	1	108	NA	
	0.1	1	102	NA	
	0.5	1	120	NA	
	1	1	107	NA	
Gluten	0.01	4	88	6.1	RJ1257B
	0.05	1	79	NA	
	0.1	1	72	NA	
	0.2	1	78	NA	
	0.5	1	103	NA	
	1	1	97	NA	
Bleached Oil	0.01	3	58	15.5	RJ1257B
	0.05	1	78	NA	
	0.1	1	55	NA	
	0.2	1	74	NA	
	0.5	1	86	NA	
	1	1	69	NA	

Residues in food of animal origin

RES-074-93 (Arras 1995 MRL-14276), RES-074-93 (Arras and Schneider 1996 MRL-14117)

A method has been developed for the analysis of metabolites hydrolysable to EMA in animal commodities. The sample preparations for the different matrices is as follows:

- Milk—Homogenize the sample thoroughly extract residues with CH_3CN, centrifuge and concentrate the CH_3CN/H_2O phase by rotary evaporation
- Fat—Homogenise with hexane, and extract with CH_3CN/H_2O (80:20 v/v). Centrifuge and retain the aqueous acetonitrile phase, which is concentrated on a rotary evaporator.
- Muscle, Liver, Kidney—add CH_3CN/H_2O (80:20 v/v) and homogenise the partially frozen tissue, centrifuge and retain the supernatant which is concentrated by rotary evaporation.

The concentrated extracts are hydrolysed with base (50% NaOH) and the resulting EMA and HEMA steam-distilled into dilute acid (2.5 N H_2SO_4). The pH of the distillate is adjusted to the basic pH range and the EMA and HEMA partitioned into methylene chloride and back-extracted into aqueous methanol/HCl. The HEMA is methylated with methanol in the presence of 4 N HCl (10:3 v/v) to form MEMA. The pH is adjusted and the residues quantified using HPLC-ECD. Residues are converted to acetochlor equivalents using the following factors:

- Acetochlor = 1.995 × EMA
- Acetochlor = 1.633 × MEMA

Representative compounds that generate EMA and HEMA on hydrolysis were used as reference materials for fortification and method validation. For animal commodities these are *tert*-oxanilic acid (EMA) and 1-hydroxyethyl-*tert*-sulfonic acid (HEMA/MEMA).

The LOQ is 0.01 to 0.02 mg/kg for *tert*-oxanilic acid in milk, beef fat, muscle, liver and kidneys, eggs, chicken muscle, fat, liver and kidney.

Summarized recovery data were available for samples of milk, liver, kidney, muscle and fat fortified at 0.01 and 0.1 mg/kg. Average *tert*-oxanilic acid recoveries were 94 ± 3.7% for muscle, 88 ± 4.2% for fat, 79 ± 4.6 for kidney, 95 ± 16.8% for liver and 101 ± 14.7% for milk. Corresponding values for 1-hydroxyethyl-*tert*-sulfonic acid were 80 ± 2.4 (muscle), 77 ± 4.1

(fat), 74 ± 7 (kidney), 87 ± 5.2 (liver) and 95 ± 4.3% (milk). Recoveries reported by an independent laboratory for samples fortified at 0.01 and 0.05 mg/kg were *tert*-oxanilic acid: 86 ± 8 (muscle), 89 ± 9.5% (fat), 84 ± 4.6 (kidney), 89 ± 2.4 (liver), 91 ± 11.8 (milk); 1-hydroxyethyl-*tert*-sulfonic acid 77 ± 3.4 (muscle), 78 ± 5.7 (fat), 83 ± 4.6 (kidney), 75 ± 2.5 (liver) and 78 ± 5.6 (milk).

Method verification/validation and method recovery data were also obtained in numerous animal matrices and reported in MSL-2285 (beef muscle, liver, kidney and fat, and milk), MSL-2287 (chicken kidney, liver, fat, muscle, and eggs), and MSL-4537 (beef muscle, liver, kidney, fat, milk, chicken liver, eggs, and pig liver). Average method verification recoveries obtained from 0.02 or 0.20 mg/kg fortifications in the feeding studies, MSL-2285 and MSL-2287, ranged from 61.5 to 77.2%. Average method recoveries for samples fortified at multiple levels ranging from 0.02 to 0.2 mg/kg and analysed concurrently with treated samples in the feeding studies ranged from 69.4 to 82.1%; % RSDs ranged from 5.4 to 13.6%. Average method recoveries for beef liver, muscle, kidney, fat, and milk, and chicken liver and eggs, from fortifications at 0.10 mg/kg in the storage stability study on animal matrices (MSL-4537) ranged from 78.2 to 89.2%; and % RSDs ranged from 8.3 to 16.3%.

The mean relative standard deviations for method recovery samples fortified at levels ranging from 0.02 to 0.20 mg/kg in different animal matrices ranged from 5.4 to 16.3%. The mean percent recovery values for method recovery samples fortified at levels ranging from 0.02 to 0.20 mg/ ranged from 69.4 to 89.2%.

Table 66 Recovery data for *tert*-oxanilic acid (EMA class) in animal commodities analysed using method *RES-074-93*

Matrix	Fortification Level (mg/kg)	N	Average Recovery (%)	% RSD	Minimum Recovery%	Maximum Recovery%	Reference
Beef muscle	0.02, 0.20	5	71.6	11.5	60	80	MSL-2285
	0.02–0.20	9	70.6	NA	NA	NA	MSL-2285
	0.10	16	82	8.4	70.9	100.8	MSL-4537
Beef liver	0.02, 0.20	7	69.4	9	61	80	MSL-2285
	0.02–0.20	13	61.5	NA	NA	NA	MSL-2285
	0.10	14	78.4	13.8	60.4	116	MSL-4537
Beef kidney	0.02, 0.20	9	73.8	13.6	55	84	MSL-2285
	0.02–0.20	26	65.7	NA	NA	NA	MSL-2285
	0.10	17	84.8	16	59.2	122.1	MSL-4537
Beef fat	0.02, 0.20	5	77.5	9.1	70	85	MSL-2285
	0.02–0.20	9	74.9	NA	NA	NA	MSL-2285
	0.10	16	78.7	16.3	57.5	110.9	MSL-4537
Chicken kidney	0.02	4	79.8	5.4	74	83	MSL-2287
Chicken	0.02	4	72.8	6.2	67	78	MSL-2287
liver	0.02, 0.20	6	61.6	NA	NA	NA	MSL-2287
	0.10	16	78.2	13.3	57	104.5	MSL-4537
Chicken fat	0.02	4	77	6.1	72	81	MSL-2287
	0.02, 0.20	8	75	NA	NA	NA	MSL-2287
Chicken	0.02	4	75.8	7.1	70	83	MSL-2287
muscle	0.02, 0.20	4	64.7	NA	NA	NA	MSL-2287
Milk	0.02	36	76.9	12.1	52	91	MSL-2285
	0.02–0.20	18	77.2	NA	NA	NA	MSL-2285
	0.10	16	89.2	11.3	66.8	108.2	MSL-4537
Eggs	0.02	22	82.1	8.2	66	95	MSL-2287
	0.02–0.20	10	66.5	NA	NA	NA	MSL-2287
	0.10	17	80.6	8.3	58.2	92	MSL-4537

Applicability of multi-residue methods

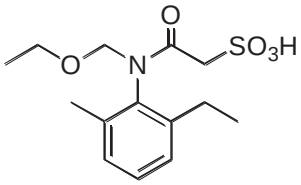
Acetochlor is not suitable for inclusion in multi-residue methods.

Stability of residues in stored analytical samples

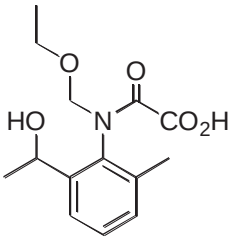
The freezer storage stability of acetochlor in homogenised plant, animal tissues, milk, and eggs samples fortified with acetochlor and/or metabolites was studied.

Stability of residues in plant products

Studies on the metabolism of acetochlor have shown that the parent compound is metabolised to a large number of compounds, none of which is the dominant contributor to residues. Methods of analysis have been developed that convert metabolites containing the ethylmethylaniline moiety to EMA and the hydroxyethylmethylaniline moiety to HEMA. To study the freezer storage stability of residues the metabolites *tert*-sulfonic acid [7] and 1-hydroxyethyl-*tert*-oxanilic acid [26] selected as representative as of the EMA and HEMA classes of acetochlor metabolites. The two compounds were mixed in equal proportions on the basis of acetochlor equivalents.



EMA-producing *tert*-sulfonic acid (7)



HEMA-producing 1-hydroxyethyl-*tert*-oxanilic acid (26)

Horton *et al.* (1996 MSL-14134) studied the stability of acetochlor residues in alfalfa forage and clover hay. Samples of ground alfalfa forage and clover hay were spiked with 1-hydroxyethyl-*tert*-oxanilic acid (HEMA class) and *tert*-sulfonic acid (EMA class) metabolites of acetochlor (94% and 95% purity, respectively) at a level of 0.10 mg/kg acetochlor equivalents each at approximately four-week intervals over 330 days (11 months). Samples were stored at approximately 20 °C. Samples were analysed using the same method as that employed in rotational alfalfa and clover crop residue studies (RES-074-93, v. 2). Results are expressed in terms of acetochlor equivalents.

Under the conditions of the study, total residues did not significantly decrease in alfalfa forage and clover hay. 1-hydroxyethyl-*tert*-oxanilic acid (HEMA-class) and *tert*-sulfonic acid (EMA class) residues on alfalfa forage and clover hay were stable at approximately –20 °C for 330 days.

Table 67 Stability of *tert*-sulfonic acid (EMA class) and 1-hydroxyethyl-*tert*-oxanilic acid (HEMA class) residues of acetochlor in alfalfa forage and clover hay on storage at approximately –20 °C

Alfalfa forage				Clover hay			
HEMA		EMA		HEMA		EMA	
Days of storage	(mg/kg)	Days of storage	(mg/kg)	Days of storage	(mg/kg)	Days of storage	(mg/kg)
0	0.071	0	0.092	0	0.072	0	0.087
1	0.077	1	0.092	1	0.073	1	0.089
29	0.078	29	0.096	27	0.075	27	0.092
57	0.075	57	0.091	57	0.075	57	0.093
85	0.075	85	0.088	85	0.074	85	0.088
113	0.076	113	0.088	113	0.072	113	0.088
145	0.077	145	0.093	145	0.071	145	0.087
176	0.075	176	0.089	176	0.075	176	0.091
204	0.078	204	0.094	204	0.074	204	0.089
239	0.077	239	0.093	238	0.077	238	0.090

Alfalfa forage				Clover hay			
HEMA		EMA		HEMA		EMA	
Days of storage	(mg/kg)	Days of storage	(mg/kg)	Days of storage	(mg/kg)	Days of storage	(mg/kg)
267	0.076	267	0.092	265	0.074	265	0.090
302	0.077	302	0.090	302	0.072	302	0.087
330	0.068	330	0.079	330	0.071	330	0.085

In a freezer storage stability study, Mannion and Steinmetz (1992 MSL-12139) spiked samples of ground soya bean forage, hay, and grain; wheat forage, straw, and grain; and sorghum silage and grain with three representative acetochlor metabolites: 1-hydroxyethyl-*tert*-oxanilic acid (HEMA class), *tert*-sulfonic acid (EMA class), and hydroxymethyl-*tert*-oxanilic acid (HMEA class) at a level of 0.05 mg/kg acetochlor equivalents each and stored for periods of up to 390 days (soya bean forage), 391 days (soya bean hay), 382 days (soya bean grain), 741 days (wheat forage), 741 days (wheat straw), 734 days (wheat grain), 739 days (sorghum silage), or 732 days (sorghum grain). Samples were stored at less than –17.8 °C. Samples were analysed at various time points using the same method as that employed in rotational soya bean, wheat, and sorghum crop residue studies (RES 004 90, v1 and v3). Results are expressed in terms of acetochlor equivalents.

Under the conditions of the study, total residues did not significantly decrease in soya bean forage, soya bean hay, and sorghum silage. A small but noticeable decreasing trend of residues with storage time was observed in soya bean grain, wheat forage, wheat straw, wheat grain, and sorghum grain. The average percent of residues remaining was 73% in soya bean grain after 382 days, 87% in wheat forage after 741 days, 78% in wheat straw after 741 days, 85% in wheat grain after 734 days, and 82% in sorghum grain after 732 days in frozen storage. The data reported indicate that 1-hydroxyethyl-*tert*-oxanilic acid, *tert*-sulfonic acid, and hydroxymethyl-*tert*-oxanilic acid residues on soya bean forage, soya bean hay, and sorghum silage were stable at less than –17.8 °C for 390, 391, and 739 days, respectively. Residues of 1-hydroxyethyl-*tert*-oxanilic acid, *tert*-sulfonic acid, and hydroxymethyl-*tert*-oxanilic acid on soya bean grain, wheat forage, wheat straw, wheat grain, and sorghum grain appear to degrade slowly with time, although the average percent remaining was still > 70% at the end of the storage periods.

Table 68 Stability of 1-hydroxyethyl-*tert*-oxanilic acid (HEMA class), *tert*-sulfonic acid (EMA class), and hydroxymethyl-*tert*-oxanilic acid (HMEA class) residues in soya bean forage, hay, and grain; wheat forage, straw, and grain; and sorghum silage and grain on storage at less than –17.8 °C

Days storage	HMEA	Procedural recovery (%)	HEMA	Procedural recovery (%)	EMA	Procedural recovery (%)
Sorghum grain						
0	0.034		0.041		0.047	
65	0.032 0.037	82 82	0.032 0.033	70 72	0.041 0.042	96 98
93	0.031	68	0.034	82	0.040	94
126	0.032 0.034	82 82	0.028 0.032	70 72	0.038 0.040	96 98
154	0.031	68	0.033	82	0.037	94
187	0.034 0.037	82 82	0.027 0.031	70 72	0.039 0.041	96 98
215	0.033	68	0.034	82	0.037	94
254	0.035 0.036	70 80	0.035 0.036	78 84	0.042 0.042	94 100
315	0.032 0.037	70 80	0.032 0.036	78 84	0.040 0.042	94 100
376	0.033 0.037	70 80	0.033 0.034	78 84	0.041 0.042	94 100
459	0.025 0.032	68 74	0.027 0.034	74 68	0.033 0.040	76 82
545	0.036 0.035	68 74	0.036 0.035	74 68	0.039 0.040	76 82
638	0.030 0.034	68 74	0.028 0.034	74 68	0.035 0.036	76 82
732	0.029 0.033	68 74	0.029 0.032	74 68	0.034 0.038	76 82
Sorghum silage						
0	0.027		0.033		0.038	
66	0.036 0.037	66 78	0.030 0.031	54 66	0.048 0.051	84 120
93	0.037	54	0.041	66	0.048	76
127	0.025 0.035	66 78	0.020 0.028	54 66	0.033 0.053	84 120

Days storage	HMEA	Procedural recovery (%)	HEMA	Procedural recovery (%)	EMA	Procedural recovery (%)
154	0.030	54	0.033	66	0.034	76
188	0.034 0.037	66 78	0.027 0.027	54 66	0.050 0.055	84 120
215	0.036	54	0.038	66	0.045	76
255	0.040 0.041	70 78	0.037 0.038	78 84	0.042 0.043	94 96
316	0.039 0.039	70 78	0.038 0.038	78 84	0.042 0.043	94 96
377	0.036 0.039	70 78	0.032 0.035	78 84	0.041 0.043	94 96
466	0.030 0.034	62 66	0.035 0.038	66 68	0.037 0.038	72 72
552	0.034 0.034	62 66	0.035 0.037	66 68	0.038 0.039	72 72
645	0.032 0.034	62 66	0.033 0.035	66 68	0.033 0.033	72 72
739	0.031 0.034	62 66	0.034 0.035	66 68	0.035 0.035	72 72
Soya bean forage						
0	0.031		0.037		0.045	
67	0.027 0.031	68 68	0.027 0.032	82 86	0.032 0.036	82 86
105	0.033	62	0.040	74	0.043	90
128	0.027 0.032	68 68	0.028 0.031	82 86	0.031 0.038	82 86
166	0.034	62	0.039	74	0.043	90
189	0.027 0.033	68 68	0.024 0.032	82 86	0.035 0.039	82 86
227	0.033	62	0.036	74	0.043	90
268	0.037 0.037	54 58	0.034 0.035	58 60	0.045 0.048	74 74
329	0.038 0.038	54 58	0.035 0.035	58 60	0.045 0.045	74 74
390	0.036 0.038	54 58	0.031 0.031	58 60	0.043 0.045	74 74
Soya bean grain						
0	0.038		0.045		0.045	
67	0.031 0.036	78 78	0.030 0.030	58 62	0.038 0.041	84 90
105	0.037	76	0.033	90	0.036	90
128	0.032 0.032	78 78	0.027 0.027	58 62	0.035 0.036	84 90
166	0.036	76	0.030	90	0.032	90
189	0.032 0.034	78 78	0.025 0.026	58 62	0.036 0.038	84 90
227	0.038	76	0.034	90	0.035	90
260	0.026 0.035	76 80	0.031 0.033	80 84	0.037 0.039	90 90
321	0.029 0.031	76 80	0.034 0.036	80 84	0.038 0.039	90 90
382	0.025 0.025	76 80	0.029 0.031	80 84	0.036 0.038	90 90
Soya bean hay						
0	0.031 0.033 0.037		0.034 0.036 0.038		0.043 0.047 0.055	
71	0.029	62 66 74	0.033	68 72 76	0.037	86 94 110
107	0.030	46 66	0.036	66 86	0.043	82 106
132	0.029	62 66 74	0.032	68 72 76	0.038	86 94 110
168	0.029	46 66	0.035	66 86	0.040	82 106
193	0.038 0.038	62 66 74	0.036 0.038	68 72 76	0.043 0.046	86 94 110
229	0.027	46 66	0.031	66 86	0.040	82 106
269	0.033 0.035	62 64 68	0.033 0.038	68 76 80	0.046 0.055	98 104 112
330	0.031 0.032	62 64 68	0.031 0.037	68 76 80	0.043 0.053	98 104 112
391	0.035 0.039	62 64 68	0.032 0.033	68 76 80	0.053 0.053	98 104 112
Wheat forage						
0	0.040		0.046		0.053	
73	0.041 0.044	86 92	0.043 0.043	90 92	0.045 0.046	96 98
109	0.029	80	0.036	92	0.046	106
134	0.037 0.038	86 92	0.042 0.043	90 92	0.042 0.046	96 98
170	0.034	80	0.044	92	0.049	106
195	0.040 0.044	86 92	0.040 0.042	90 92	0.044 0.048	96 98
231	0.038	80	0.040	92	0.050	106
336	0.044 0.045	88 90	0.039 0.040	80 82	0.041 0.043	88 92
464	0.037 0.037	88 92	0.034 0.035	78 78	0.039 0.046	88 88
586	0.037 0.037	88 92	0.035 0.035	78 78	0.045 0.047	88 88
619	0.024	60 62	0.027	64 78	0.030	72 90
680	0.030	60 62	0.035	64 78	0.038	72 90
741	0.027	60 62	0.032	64 78	0.036	72 90
Wheat grain						

Days storage	HMEA	Procedural recovery (%)	HEMA	Procedural recovery (%)	EMA	Procedural recovery (%)
0	0.037		0.038		0.050	
72	0.035 0.036	74 78	0.026 0.027	54 56	0.040 0.043	86 98
107	0.037	74	0.038	76	0.041	100
133	0.027 0.035	74 78	0.023 0.026	54 56	0.037 0.043	86 98
168	0.036	74	0.035	76	0.041	100
194	0.034 0.035	74 78	0.023 0.025	54 56	0.038 0.042	86 98
229	0.035	74	0.041	76	0.045	100
270	0.041 0.041	80 84	0.034 0.034	78 78	0.044 0.044	98 98
331	0.034 0.035	80 84	0.030 0.031	78 78	0.039 0.040	98 98
392	0.034 0.037	80 84	0.030 0.032	78 78	0.042 0.043	98 98
612	0.037	78 76	0.038	78 76	0.033	72 78
673	0.036	78 76	0.038	78 76	0.033	72 78
734	0.034	78 76	0.034	78 76	0.031	72 78
Wheat straw						
0	0029 0.033		0.027 0.030		0.044 0.044	
79	0.030 0.031	62 70 74	0.029 0.031	64 68 70	0.038 0.039	84 94 98
109	0.031	58 66	0.028	54 60	0.035	88 88
140	0.030 0.030	62 70 74	0.028 0.030	64 68 70	0.033 0.036	84 94 98
170	0.028	58 66	0.029	54 60	0.035	88 88
201	0.028 0.029	62 70 74	0.027 0.027	64 68 70	0.037 0.039	84 94 98
231	0.028	58 66	0.028	54 60	0.037	88 88
273	0.034 0.035	82 82 88	0.025 0.026	80 82 86	0.035 0.048	106 108 114
334	0.034 0.034	82 82 88	0.025 0.026	80 82 86	0.038 0.044	106 108 114
395	0.032 0.036	82 82 88	0.021 0.022	80 82 86	0.043 0.044	106 108 114
619	0.027	42 54	0.030	60 60	0.033	76 80
680	0.021	42 54	0.029	60 60	0.036	76 80
741	0.020	42 54	0.028	60 60	0.034	76 80

In a freezer storage stability study (White 2001 RJ3114B), samples of potato tubers and sugar beet tops were spiked with acetochlor (99.8% purity) as well as 1-hydroxyethyl-*tert*-oxanilic acid (HEMA class) and *tert*-sulfonic acid (EMA class) at a level of 0.2 mg/kg acetochlor equivalents each. Samples were stored at–18 °C for up to 295 days (9 months), and duplicate samples were analysed for acetochlor and HEMA and EMA metabolite class residues after 0, 3, 7 (acetochlor only), 8 (HEMA and EMA metabolite class only), and 9 months. Samples were analysed for HEMA and EMA metabolite class residues using analytical method RAM 280/02; parent acetochlor was analysed using RAM 244/02. Results were expressed as total acetochlor residues (HEMA + EMA) in acetochlor equivalents.

Under the conditions of the study, total residues did not significantly decrease in potato tubers or sugar beet tops, indicating that acetochlor and the HEMA and EMA metabolite class residues in these matrices were stable at–18 °C for up to 9 months.

Table 69 Stability of acetochlor, 1-hydroxyethyl-*tert*-oxanilic acid (HEMA class) and *tert*-sulfonic acid (EMA class) residues in potato tubers and sugar beet tops on storage at –18 °C

		Potato tubers			Beet tops	
	acetochlor	HEMA	EMA	acetochlor	HEMA	EMA
Days	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)
0	0.20 0.21	0.20 0.20	0.20 0.20	0.22 0.21	0.21 0.20	0.22 0.21
98	0.20 0.20			0.19 0.21		
104		0.20 0.20	0.24 0.19		0.21 0.20	0.21 0.20
216	0.20 0.20			0.20 0.17		
251		0.18 0.19	0.19 0.20		0.20 0.19	
286		0.20 0.19	0.22 0.21		0.18 0.20	
294				0.22 0.23		
295	0.21 0.20					

Procedural recoveries were 91% for acetochlor in potato day 295, 73% for 1-hydroxyethyl-*tert*-oxanilic acid (HEMA) day 286, 80% for *tert*-sulfonic acid (EMA) day 286 and for sugar beet tops 89% for acetochlor day 294, 65% for 1-hydroxyethyl-*tert*-oxanilic acid (HEMA) day 286 and 80% for *tert*-sulfonic acid (EMA) day 286.

Crook (1995 RJ1984B) studied the storage stability of 5-hydroxy-*sec*-oxanilic acid (68) in maize, soya bean, turnip and lettuce commodities. Samples were fortified with ^{14}C-phenyl-radiolabelled 5-hydroxy-*sec*-oxanilic acid at 0.09 mg/kg and then deep frozen at < –10 °C. Actual fortification levels were 0.09 mg/kg for maize fractions and lettuce and 0.1 mg/kg for turnip and soya bean fractions, except for 0 month samples that were all fortified at 0.09 mg/kg. Residues of 5-hydroxy-*sec*-oxanilic acid (68) were shown to be stable in deep frozen field maize grain forage and fodder for a storage period of at least 24 months.

Table 70 5-hydroxy-*sec*-oxanilic acid (68) residues in maize grain, forage and fodder samples fortified at 0.09 mg/kg and held in frozen storage

Commodity	Storage period (months)	Residue (mg/kg)	Commodity	Storage period (months)	Residue (mg/kg)
Maize grain	0	0.06	Turnip roots	0	0.07
	6	0.07		6	0.09
	12	0.06		12	0.08
	18	0.07		18	0.07
	24	0.07		24	0.07
Maize forage	0	0.07	Turnip tops	0	0.07
	6	0.08		6	0.09
	12	0.07		12	0.08
	18	0.07		18	0.08
	24	0.08		24	0.07
Maize grain	0	0.07	Soya bean seed	0	0.06
	6	0.08		6	0.07
	12	0.07		12	0.06
	18	0.07		18	0.08
	24	0.08		24	0.07
Lettuce	0	0.08	Soya bean hay	0	0.06
	6	0.09		6	0.07
	12	0.08		12	0.08
	18	0.08		18	0.08
	24	0.07		24	0.06

Hay and Wujcik (2009 MSL-21172) studied the frozen storage stability of 1-hydroxyethyl-*tert*-oxanilic acid (HEMA class) and *tert*-sulfonic acid (EMA class) in corn forage, grain and stover for a period of one year. Samples were stored at < –18 °C, which represents conditions typical of those used for frozen storage of residue samples. Samples were analysed using a common moiety method of analysis which converts metabolites in the EMA- and HEMA-classes to the analytes by base hydrolysis. Results are expressed in acetochlor equivalents.

Residues of the *tert*-sulfonic acid and 1-hydroxyethyl-*tert*-oxanilic acid were stable in forage, stover and grain after one year.

Table 71 Recovery of *tert*-sulfonic acid residues after fortification and frozen storage

	Grain		Forage		Stover
Days of storage	Residue (mg/kg)	Days of storage	Residue (mg/kg)	Days of storage	Residue (mg/kg)
0	0.087 0.094	0	1.22 1.25	0	1.26 1.27
1	0.087 0.083	1	1.10 1.21	1	1.03 1.14
43	0.080 0.085	40	1.05 1.18	35	1.15 1.14
93	0.083 0.081	86	1.04 1.16	82	1.18 1.14
133	0.084 0.080	131	1.12 1.17	128	1.19 1.18
182	0.084 0.084	175	1.12 1.21	175	1.13 1.21
222	0.078 0.078	222	1.04 1.07	210	1.18 1.17
268	0.088 0.086	267	1.13 1.16	261	1.25 1.20
315	0.088 0.087	315	1.20 1.11	309	1.20 1.14
356	0.076 0.071	357	0.95 1.06	351	1.27 1.20

Table 72 Recovery of 1-hydroxyethyl-*tert*-oxanilic acid residues after fortification and frozen storage

	Grain		Forage		Stover
Days of storage	Residue (mg/kg)	Days of storage	Residue (mg/kg)	Days of storage	Residue (mg/kg)
0	0.085 0.091	0	1.20 1.22	0	1.20 1.13 1.09 1.18
1	0.081 0.076	1	1.10 1.18	1	0.93 0.98
43	0.075 0.077	40	1.03 1.03	35	0.98 1.01
93	0.075 0.075	86	1.04 1.07	82	1.04 1.05
133	0.075 0.073	131	1.09 1.17	128	1.11 0.99
182	0.077 0.074	175	1.05 1.12	175	1.02 1.09
222	0.076 0.070	222	1.03 1.01	210	1.07 1.04
268	0.074 0.074	267	1.10 1.08	261	1.12 1.02
315	0.077 0.076	315	1.15 1.02	309	1.05 1.03
356	0.060 0.057	357	0.87 0.86	351	1.10 1.09

Animal matrices

In a freezer storage stability study (Wilson 1986 MSL-4537), samples of ground or blended eggs, whole milk, chicken liver, pig liver, beef liver, muscle, fat, and kidneys were spiked with four metabolites of acetochlor (*tert*-hydroxyacetochlor, *tert*-oxanilic acid, *tert*-sulfonic acid and *tert*-sulfinylacetic acid, 99% purity each) at a level of 0.025 mg/kg each, for a total of 0.10 mg/kg, and stored at–23 °C for at least 130 weeks and up to 146 weeks. Samples were analysed at several time intervals using the same method as that employed in egg, milk, chicken tissue and beef tissue residue studies ("Analytical Residue Method for Four Metabolites of Acetochlor in Milk, Beef Tissues, Hog Liver and Chicken Liver"). Residues are measured as EMA and expressed in terms of acetochlor equivalents.

Under the conditions of the study, total residues did not significantly decrease in eggs, milk, chicken liver, pig liver, beef liver, muscle, fat, and kidneys. The data reported indicate that residues of the four acetochlor metabolites in eggs, milk, chicken liver, pig liver, beef liver, muscle, fat and kidneys were stable when samples are stored at–23 °C for at least 130 weeks and up to 146 weeks.

Table 73 Stability of combined residues of *tert*-hydroxyacetochlor, *tert*-oxanilic acid, *tert*-sulfonic acid and *tert*-sulfinylacetic acid in fortified samples of tissues, eggs and milk on frozen storage

	Eggs			Milk		Chicken liver		Pig liver	
Weeks of storage	EMA (mg/kg)	Procedural recovery	Weeks of storage	EMA (mg/kg)	Procedural recovery	EMA (mg/kg)	Procedural recovery	EMA (mg/kg)	Procedural recovery
0	0.085 0.083		0	0.093 0.086		0.057 0.071		0.066 0.072	
1	0.082 0.086	85 83	1	0.079 0.072	93 86	0.080 0.092	57 71	0.063 0.141	66 71
2	0.075 0.081	89	2	0.095 0.086	67 99	0.053 0.077	64 86	0.062	81
4	0.085 0.091	58 84	4	0.092 0.079	76	0.054 0.054	74	0.065 0.074	61 78
8	0.079 0.043	71 89	8	0.092 0.081	87	0.097 0.080	83	0.050 0.067	64 44
16	0.085 0.080	76 72	16	0.091 0.096	92 95	0.052 0.057	80 89	0.063 0.079	87 82
32	0.065 0.071	74 80	32	0.090 0.074	96 90	0.068 0.056	89 86	0.045 0.067	78 78
64	0.099 0.078	88 92	64	0.087 0.088	77 76	0.043 0.052	74 71	0.070 0.063	72 69
142	0.116 0.118	82 81	130	0.078 0.115	108 107	0.106 0.075	95 104	0.098 0.091	82 89
	Beef liver			Beef muscle		Beef fat		Beef kidney	

Weeks of storage	EMA (mg/kg)	Procedural recovery	EMA (mg/kg)	Procedural recovery	EMA (mg/kg)	Procedural recovery	EMA (mg/kg)	Procedural recovery
0	0.083 0.078		0.077 0.074		0.063		0.061 0.089	
1	0.065 0.070	82 78	0.078 0.078	77 74	0.057	63	0.093 0.089	61 89
2	0.067 0.058	60	0.076 0.080	76 83	0.090 0.099	111 102	0.062 0.083	93 74
4					0.099 0.147 [a]	85 72	0.075 0.081	89 84
8	0.079 0.090	83 88	0.092 0.083	87 91	0.079 0.088	85 74	0.089 0.063	89 94
16	0.068 0.068	79 79	0.083 0.086	87 71	0.077 0.081	86 73	0.076 0.087	59
32	0.067 0.065	64 75	0.103 0.087	89 82	0.072 0.074	77 74	0.072 0.066	93 88
64	0.060 0.072	65	0.078 0.080	80 74	0.085 0.075	71 58	0.026 0.088	74 79
130	0.071 0.069	65 116	0.104 0.102	92 100	0.110 0.100	106 60	0.112 0.074	104 122

Introduction to Use Patterns

Acetochlor is an herbicide used to control annual grasses and broadleaf weeds. Acetochlor controls weeds by inhibiting growth of seedling shoots. It needs to be applied before weeds germinate to be effective; therefore, it is typically applied just before or after planting of the crop. Acetochlor is in the chloroacetanilide herbicide family. It is in herbicide Site of Action Group 15, known as long-chain fatty acid inhibitors. The product is mixed with water and applied as a ground broadcast spray prior to planting, after planting but pre-emergence to the crop, or post-emergence to the crop and pre-emergence to the weeds using ground equipment equipped for conventional spraying on crops.

Table 74 Selected registered uses of acetochlor

Crop	Country	Form g ai/L	GS	Rate kg ai/ha	Water L/ha	No	Interval (days)	PHI (days)
Sweet corn	USA	839 g/L EC	Apply pre-plant or pre-emergence only. Do not apply post emergence.	1.47–2.95 max 3.36 kg ai/ha per year	≥ 93.6	1		Not specified
Soya bean	USA	359 g/L CS	Apply pre-plant or pre-emergence or post-emergence but before R2 GS	1.05–1.68 max 3.36 kg ai/ha per year	≥ 93.6	1–2		Not specified
Sugar beet	USA	359 g/L CS	Apply pre-plant, at-planting, pre-emergence, or post emergence (2-leaf to the 8-leaf stage)	1.05–1.68 max 3.36 kg ai/ha per year	≥ 93.6	1–3	7	70
Field corn	USA	839 g/L EC	Apply pre-plant or pre-emergence or post-emergence (until corn reaches 28 cm in height)	1.47–2.95 max 3.36 kg ai/ha per year	≥ 93.6	1–2		Not specified
Field corn	USA	359 g/L CS	Apply pre-plant or pre-emergence or post-emergence (until corn reaches 76 cm in height)	1.26–2.52 max 3.36 kg ai/ha per year	≥ 93.6	1–2		Not specified
Sorghum	USA	359 g/L CS	Apply pre-plant incorporated, pre-emergence, or post-emergence before the crop exceeds 28 cm in height (generally 5–6 leaf)	1.26–2.52 max 3.36 kg ai/ha per year	≥ 93.6	1–2		Not specified

Crop	Country	Form g ai/L	GS	Rate kg ai/ha	Water L/ha	No	Interval (days)	PHI (days)
Cotton	USA	359 g/L CS	Apply pre-plant, at-planting, pre-emergence or post-emergence (but before first bloom)	1.05–1.68 max 3.36 kg ai/ha per year	≥ 93.6	1–2		Not specified
Peanut	USA	359 g/L CS	Apply pre-plant, at-planting, pre-emergence or post-emergence (but before flowering)	1.05–1.68 max 3.36 kg ai/ha per year	≥ 93.6	1–3	7	Not specified

Cotton—do not graze treated area or feed treated cotton forage to livestock following application

Maize—do not graze treated area or feed treated forage to livestock for 40 days following application

Do not use Warrant CS herbicide on sweet corn.

Peanut—allow a minimum of 90 days between last application and grazing or harvest and feeding of peanut hay to livestock.

Sorghum—do not graze treated area or feed treated sorghum forage to livestock for 60 days following application

If sorghum seed is not properly treated with seed protectant or safener, pre-plant and pre-emergence applications will severely injure the crop.

Soya bean post-emergence use—do not graze treated area or feed treated forage to livestock

Sugar beet allow a minimum of 70 days between last application and harvest of sugar beet, and grazing or harvest and feeding of sugar beet tops to livestock.

Rotational crops (CS formulation). Do not graze or harvest winter cover crops for food or animal feed for a minimum of 18 months following last application of acetochlor.

Rotational crops:

- If a treated crop is lost, corn (all types), cotton, soya beans, and milo (sorghum), may be replanted immediately, but could result in crop injury. When planting milo (sorghum), only use seed properly treated with seed protectant or safener. Do not exceed a total of 3.4 kg ai/ha/year if additional applications are made.
- Non grass animal feeds such as alfalfa, clover, kudzu, lespedeza, lupin, sainfoin, trefoil, velvet bean, and Vetch spp. may be planted 9 months after application. Wheat may be planted 4 months after application. .
- Rotate the next season to the following crops: soya beans, corn (all types), milo (sorghum), cotton, tobacco, sugar beets, sunflowers, potatoes, barley, buckwheat, millet (pearl and proso), oats, rye, teosinte, triticale, wild rice, dried shelled bean group Lupinus spp. (including grain lupin, sweet lupin and white lupin); Phaseolus spp. (includes field bean, kidney bean, lima bean (dry), navy bean, pinto bean, tepary bean); bean, Vigna spp. (includes adzuki bean, black-eyed pea, catjang, cowpea, Crowder pea, moth bean, mung bean, rice bean, southern pea and urd bean); broad bean (dry) chickpea, guar, lab lab bean, lentil, pea (Pisum spp., includes field pea); pigeon pea.

Residues studies

The Meeting received information on supervised field trials for acetochlor on the following crops or crop groups:

Crop	Table No.
Sweet corn	Table 76
Soya bean	Table 77
Sugar beet	Table 78
Maize	Table 79–82
Sorghum	Table 83
Cotton	Table 84
Peanut	Table 85

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.

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 75 Summary of sprayers, plot sizes and field sample sizes in the supervised trials

Location	Year	Sprayer	Plot size	Sample size	Sample to analysis interval (days)
Sweet corn USA RJ2078B	1995	Tractor mounted boom sprayer, backpack sprayer, or all-terrain vehicle sprayer. The granular formulation was applied directly using a Gandy air flow granule applicator or a broadcast spreader	> 93–1115 m^2	Grain ≥ 12 ears > 5 lb Forage > 2.5 lb Stover 12 plts > 2.5 lb	≤ 182 d (6 mo)
Soya bean USA MSL20719	2007	Backpack, hand-held or tractor-mounted sprayers	93–557 m^2	Forage > 1.3 lb Hay > 1.1 lb 0.3 kg Seed > 1.2 lb	≤ 330 d (11 mo)
Sugar beet USA MSL-24198	2011	Backpack sprayer, tractor with boom	60–372 m^2	12 plants	≤ 210 d (7 mo)
Maize USA MSL-6843	1985	Bicycle sprayer, tractor with boom	> 93 m^2	Ns	Forage ≤ 669 d Fodder ≤ 624 d Grain ≤ 542 d
Maize USA RJ1337B	1991				< 11 mo
Maize USA MSL-11794	1990	Backpack sprayer, tractor with boom	181–2926 m^2		Silage 300 d (10 mo) Forage 240 d (8 mo) Fodder 240 d (8 mo) Grain 195 d (6.5 mo)
Maize USA MSL-20269	2006	Backpack sprayer, tractor with boom	70–149 m^2	Forage 1.1–4.1, 12 plants Grain 1.0–3.3 Stover 0.2–1.8, 12 plants	≤ 374 + 26 ≤ 175 + 11 ≤ 357 + 32
Sorghum USA MSL-18670	2003	backpack, ATV or tractor-mounted sprayers	93–248 m^2, 297 m^2 NE-1, 858 m^2 OK-2	Forage 1.5 kg Grain 1.07 kg Stover 12 plants, 0.16 kg (GA) else 0.57 kg	≤ 222 d forage ≤ 221 d stover ≤ 211 d grain
Cotton USA MSL-20718	2007	Backpack, hand-held or tractor-mounted sprayer	93–705 m^2	Seed > 1 kg Gin bp > 0.7 kg	Seed 112–209 d Gin byp 171–249 d
Peanut USA MSL-24197	2011	Backpack sprayer, tractor with boom	64–446 m^2	Nutmeat 0.2 to > 1 kg Hay 20–24 plants	≤ 240 d

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).

Sweet corn

Crook and French (1996 RJ2078B) conducted fourteen trials in the USA during 1995, on the pre-emergence and pre-plant incorporated use of acetochlor formulations containing the safener dichlormid (R-25788) in sweet corn. Plots containing sweet corn were treated pre-emergence or pre-plant incorporated with either an emulsifiable concentrate (EC), a water dispersible micro-encapsulated suspension (CS) or a dry granular (GR) formulation of acetochlor at a rate of 3.4 kg ai/ha. The method used was RAM 280/01 for which the LOQ is 0.01 mg/kg for both EMA-class and HEMA-class compounds.

Table 76 Residues in sweet corn following a single application of an EC, CS or GR acetochlor formulation (kernels + cob with husk removed) (Crook and French 1996 RJ2078B) HEMA and EMA residues are expressed in acetochlor equivalents.

Location, year, variety	Form	Growth stage at	Rate	DALA	Residues (mg/kg)			Total
SWEET CORN		application	kg ai/ha		Acetochlor	HEMA	EMA	(mg/kg)
North Rose, New York, USA 1995 Crusader	EC	Pre-plant incorporated	3.36	88	< 0.01	< 0.02	< 0.02	< 0.04
4399 LF	CS	Pre-plant incorporated	3.36	88	< 0.01	< 0.02	< 0.02	< 0.04
	GR	Pre-plant incorporated	3.36	88	< 0.01	< 0.02	< 0.02	< 0.04
Boone, Iowa, USA 1995 Illini Xtra Sweet	EC	Pre-plant incorporated	3.36	76	< 0.01	< 0.02	< 0.02	< 0.04
	CS	Pre-plant incorporated	3.36	76	< 0.01	< 0.02	< 0.02	< 0.04
	GR	Pre-plant incorporated	3.36	76	< 0.01	< 0.02	< 0.02	< 0.04
Whitakers, North	EC	Pre-emergence	3.36	80	< 0.01 [a]	< 0.02 [a]	< 0.02 [a]	< 0.04 [a]
Carolina, USA 1995	CS	Pre-emergence	3.36	80	< 0.01	< 0.02	< 0.02	< 0.04
Silver Queen	GR	Pre-emergence	3.36	80	< 0.01	< 0.02	< 0.02	< 0.04
Champaign, Illinois,	EC	Pre-emergence	3.36	58	< 0.01	< 0.02	< 0.02	< 0.04
USA 1995 Early Choice	CS	Pre-emergence	3.36	58	< 0.01	< 0.02	< 0.02	< 0.04
	GR	Pre-emergence	3.36	58	< 0.01	< 0.02	< 0.02	< 0.04
Northwood, North	EC	Pre-plant	3.36	103	< 0.01 [b]	< 0.02 [b]	< 0.02 [b]	< 0.04 [b]
Dakota, USA 1995 Golden Bantam		incorporated			< 0.01 [b]	< 0.02 [b]	< 0.02 [b]	< 0.04 [b]
Janesville, Wisconsin,	EC	Pre-plant	3.36	87	< 0.01 [b]	< 0.02 [b]	< 0.02 [b]	< 0.04 [b]
USA 1995 More		incorporated			< 0.01 [b]	< 0.02 [b]	< 0.02 [b]	< 0.04 [b]
Hebron, Maryland, USA	EC	Pre-plant	3.36	81	< 0.01 [b]	< 0.02 [b]	< 0.02 [b]	< 0.04 [b]
1995 Snow Belle		incorporated			< 0.01 [b]	< 0.02 [b]	< 0.02 [b]	< 0.04 [b]
Hamburg, Pennsylvania,	EC	Pre-emergence	3.36	72	< 0.01 [b]	< 0.02 [b]	< 0.02 [b]	< 0.04 [b]
USA 1995 Stars-N-Stripes					< 0.01 [b]	< 0.02 [b]	< 0.02 [b]	< 0.04 [b]
Loxley, Alabama, USA	EC	Pre-emergence	3.36	81	< 0.01 [a]	< 0.02 [a]	< 0.02 [a]	< 0.04 [a,b]
1995 Silver Queen					< 0.01 [a]	< 0.02 [a]	< 0.02 [a]	< 0.04 [a,b]
Monmouth, Illinois, USA	EC	Pre-emergence	3.36	61	< 0.01 [b]	< 0.02 [b]	< 0.02 [b]	< 0.04 [b]
1995 Pioneer 3395 IR					< 0.01 [b]	< 0.02 [b]	< 0.02 [b]	< 0.04 [b]
Visalia, California, USA	EC	Pre-emergence	3.36	83	< 0.01	< 0.02	< 0.02	< 0.04
1995 Supersweet	EC	Pre-plant incorporated	3.36	83	< 0.01	< 0.02	< 0.02	< 0.04
Ephrata, Washington,	EC	Pre-plant incorporated	3.36	91	< 0.01	< 0.02	< 0.02	< 0.04

Location, year, variety	Form	Growth stage at	Rate	DALA	Residues (mg/kg)			Total
SWEET CORN		application	kg ai/ha		Acetochlor	HEMA	EMA	(mg/kg)
USA 1995 Jubilee	EC	Pre-emergence	3.36	91	< 0.01	< 0.02	< 0.02	< 0.04
Oviedo, Florida, USA 1995 Florida Stay Sweet	EC	Pre-plant incorporated	3.36	65	< 0.01 [a]	< 0.02 [a]	< 0.02 [a]	< 0.04 [a]
	EC	Pre-emergence	3.36	65	< 0.01 [a]	< 0.02 [a]	< 0.02 [a]	< 0.04 [a]
Mt. Vernon, Washington, USA 1995	EC	Pre-plant incorporated	3.36	113	< 0.01	< 0.02	< 0.02	< 0.04
Jubilee	EC	Pre-emergence	3.36	113	< 0.01	< 0.02	< 0.02	< 0.04

[a] Samples of kernels only and not kernels + cob with husk removed

[b] Replicate samples from same plot

Soya bean

Hay *et al.* (2008 MSL-20719) studied residues of acetochlor in soya beans following application of a micro-encapsulated formulation as a single post-emergent application at 3.4 kg ai/ha made at growth stage R1-R2 (beginning flowering–full flowering) or as three applications of 1.1 kg ai/ha each made pre-plant (45 d prior to planting), and post-emergent at growth stages V3 (3rd trifolate leaf) and at R1-R2. One composite sample was collected from each untreated control plot and two composite samples were collected from each of the treated plots. Hay was left in the field to dry for 1 to 7 days before sampling as this was needed to allow moisture levels to reach that of commercial hay. Residues were quantified using LC-MS/MS analytical method ES-ME-1215-01.

Table 77 Residues in soya bean following application of a CS acetochlor formulation (Hay *et al.* 2008 MSL-20719) HEMA and EMA residues are expressed in acetochlor equivalents. Replicate samples

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SOYA BEAN		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Proctor, Arkansas, USA	1	3.36	V5/R1–R2	90	Seed	0.054	0.190	
2007 AG4403RR						0.056	0.193	
					mean	0.055	0.192	0.247
	3	1.12	Bare ground	90	Seed	0.037	0.071	
	(71	1.12	V3			0.037	0.064	
	12)	1.12	V5/R1–R2		mean	0.037	0.067	0.104
Newport, Arkansas, USA	1	3.37	R2	83	Seed	0.103	0.378	
2007 JG55R505C						0.110	0.489	
					mean	0.106	0.434	0.540
	3 (65	1.13	Pre-plant	83	Seed	0.049	0.087	
	32)	1.11	V3			0.052	0.088	
		1.12	R2		mean	0.051	0.087	0.138
Richland, Iowa, USA	1	3.36	R1	83	Seed	0.021	0.043	
2007 Asgrow 3101						0.020	0.041	
					mean	0.021	0.042	0.063
				90	Seed	0.027	0.056	
						0.026	0.053	
					mean	0.026	0.054	0.080
				96	Seed	0.022	0.050	
						0.022	0.048	
					mean	0.022	0.049	0.071
				104	Seed	0.023	0.048	
						0.022	0.048	
					mean	0.023	0.048	0.071
	3 (76	1.17	Pre-plant	90	Seed	0.008	0.013	
	28)	1.10	V3			0.008	0.011	
		1.11	R1		mean	0.008	0.012	0.020
Ollie, Iowa, USA	1	3.38	R1	97	Seed	0.015	0.036	
2007 AG 3802						0.016	0.034	
					mean	0.015	0.035	0.050

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SOYA BEAN		kg ai/ha	at application			HEMA	EMA	(mg/kg)
	3 (68	1.12	Pre-plant	97	Seed	0.004	0.005	
	30)	1.11	V3			0.004	0.005	
		1.13	R1		mean	0.004	0.005	0.009
Milford, Iowa, USA	1	3.33	R1–R2	100	Seed	0.022	0.076	
2007 NK S19-L7						0.028	0.109	
					mean	0.025	0.093	0.118
	3 (74	1.11	Pre-plant	100	Seed	0.017	0.036	
	33)	1.10	V3			0.018	0.040	
		1.09	R1–R2		mean	0.017	0.038	0.055
Bagley, Iowa, USA	1	3.37	R2	83	Seed	0.074	0.235	
2007 92M52						0.072	0.212	
					mean	0.073	0.223	0.296
	3 (74	1.14	Pre-plant	83	Seed	0.041	0.046	
	24)	1.11	V3			0.039	0.043	
		1.14	R2		mean	0.040	0.044	0.084
Carlyle, Illinois, USA	1	3.36	R1–R2	73	Seed	0.276	0.602	
2007 5N382 RR						0.283	0.599	
					mean	0.279	0.600	0.879
				80	Seed	0.299	0.641	
						0.306	0.752	
					mean	0.302	0.696	0.998
				87	Seed	0.193	0.618	
						0.213	0.695	
					mean	0.203	0.657	0.860
				94	Seed	0.205	0.616	
						0.160	0.506	
					mean	0.183	0.561	0.744
	3 (72	1.13	Pre-plant	80	Seed	0.155	0.283	
	25)	1.13	V3			0.170	0.295	
		1.11	R1–R2		mean	0.162	0.289	0.451
Carlyle, Illinois, USA	1	3.4	R1–R2	91	Seed	0.058	0.157	
2007 NK 37N4						0.062	0.178	
					mean	0.060	0.167	0.227
	3 (71	1.12	Pre-plant	91	Seed	0.038	0.082	
	27)	1.12	V3			0.034	0.078	
		1.14	R1–R2		mean	0.036	0.080	0.116
Mason, Illinois, USA	1	3.41	R2	73	Seed	0.085	0.183	
2007 Trisler T-3463 RR						0.083	0.192	
					mean	0.084	0.188	0.272
	3 (88	1.12	Pre-plant	73	Seed	0.061	0.073	
	15)	1.15	BBCH 14/V3			0.062	0.077	
		1.13	R2		mean	0.061	0.075	0.136
Wyoming, Illinois, USA	1	3.45	R1–R2	78	Seed	0.073	0.222	
2007 AG3101						0.081	0.250	
					mean	0.077	0.236	0.313
	3 (78	1.16	Pre-plant	78	Seed	0.046	0.060	
	23)	1.12	V3			0.037	0.051	
		1.08	R1–R2		mean	0.041	0.056	0.097
Rockville, Indiana, USA	1	3.43	R1	90	Seed	0.043	0.132	
2007 T-3463RR						0.044	0.125	
					mean	0.044	0.129	0.173
	3 (74	1.22	Pre-plant	90	Seed	0.027	0.038	
	21)	1.11	BBCH 14/V3			0.027	0.036	
		1.12	R1		mean	0.027	0.037	0.064
New Ross, Indiana, USA	1	3.5	R1	93	Seed	0.041	0.081	
2007 T-3463RR						0.041	0.082	
					mean	0.041	0.082	0.123
	3 (74	1.15	Pre-plant	93	Seed	0.016	0.028	
	21)	1.13	V3			0.015	0.025	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SOYA BEAN		kg ai/ha	at application			HEMA	EMA	(mg/kg)
		1.10	R1		mean	0.016	0.027	0.043
Washington, Louisiana,	1	3.35	R2	77	Seed	0.099	0.283	
USA 2007 AG 5905						0.097	0.289	
					mean	0.098	0.286	0.384
	3 (70	1.15	Pre-plant	77	Seed	0.040	0.095	
	28)	1.13	V3			0.036	0.093	
		1.10	R2		mean	0.038	0.094	0.132
Paynesville, Minnesota,	1	3.38	R2	86	Seed	< 0.006	< 0.006	
USA 2007 90M60-N201						< 0.006	< 0.006	
					mean	< 0.006	< 0.006	< 0.012
	3 (88	1.11	Pre-plant	86	Seed	< 0.006	< 0.006	
	36)	1.11	V3			< 0.006	< 0.006	
		1.12	R2		mean	< 0.006	< 0.006	< 0.012
Geneva, Minnesota, USA	1	3.41	R2	82	Seed	0.035	0.098	
2007 Pioneer 91M30						0.047	0.117	
					mean	0.041	0.107	0.148
	3 (70	1.12	Pre-plant	82	Seed	0.026	0.032	
	19)	1.12	V3			0.025	0.033	
		1.11	R2		mean	0.025	0.032	0.057
La Plata, Missouri, USA	1	2.69	R1–R2	96	Seed	0.056	0.169	
2007 Asgrow AG3802						0.061	0.177	
					mean	0.059	0.173	0.232
	3 (76	1.14	Pre-plant	96	Seed	0.034	0.059	
	32)	1.12	V4 (90% V3)			0.037	0.065	
		1.12	R1–R2		mean	0.035	0.062	0.097
Pikeville, North Carolina,	1	3.41	R1, beginning	103	Seed	0.039	0.097	
USA 2007 NK 565-M3			to flower			0.038	0.101	
					mean	0.038	0.099	0.137
	3 (76	1.13	Pre-plant	103	Seed	0.121	0.116	
	40)	1.11	BBCH 14/V3			0.100	0.091	
		1.12	R1/flower start		mean	0.110	0.103	0.213
York, Nebraska, USA	1	3.36	BBCH 61/R1	87	Seed	0.078	0.077	
2007 WW152201						0.078	0.077	
					mean	0.078	0.077	0.155
	3 (79	1.13	Pre-plant	87	Seed	0.040	0.033	
	14)	1.11	BBCH 15/ late third trifoliate			0.038	0.033	
		1.12	BBCH 61/ R1		mean	0.039	0.033	0.072
New Holland, Ohio, USA	1	3.43	R1–R2	78	Seed	0.044	0.119	
2007 Crop Plan RC 3935						0.057	0.184	
					mean	0.051	0.152	0.203
	3 (85	1.13	Pre-plant	78	Seed	0.060	0.056	
	21)	1.13	V3			0.051	0.052	
		1.13	R1–R2		mean	0.055	0.054	0.109
New Holland, Ohio, USA	1	3.43	R1–R2	78	Seed	0.078	0.098	
2007 Crows 3518 R						0.081	0.097	
					mean	0.079	0.097	0.176
	3 (85	1.13	Pre-plant	78	Seed	0.107	0.130	
	21)	1.12	V3			0.093	0.122	
		1.12	R1–R2		mean	0.100	0.126	0.226
Elko, South Carolina,	1	3.38	R2	99	Seed	0.290	0.403	
USA 2007 97M50						0.262	0.394	
					mean	0.276	0.399	0.675
	3 (73	1.15	Pre-plant	99	Seed	0.132	0.127	
	40)	1.12	V3			0.102	0.107	
		1.13	R2		mean	0.117	0.117	0.234

Growth Stages

VE Emergence—cotyledons have been pulled through the soil surface

VC Unrolled unifoliolate leaves—unfolding of the unifoliolate leaves

V1 First trifoliolate—one set of unfolded trifoliolate leaves

V2 Second trifoliolate—two sets of unfolded trifoliolate leaves

V4 Fourth trifoliolate—four unfolded trifoliolate leaves

V(n) nth trifoliolate—V stages continue with the unfolding of trifoliolate leaves. The final number of trifoliolates depends on the soya bean variety and the environmental conditions

R1 Beginning flowering—plants have at least one flower on any node

R2 Full flowering—there is an open flower at one of the two uppermost nodes

R3 Beginning pod—pods are 5 mm at one of the four uppermost nodes

R4 Full pod—pods are 2 cm at one of the four uppermost nodes

R5 Beginning seed—seed is 3 mm long in the pod at one of the four uppermost nodes on the main stem

R6 Full seed—pod containing a green seed that fills the pod capacity at one of the four uppermost nodes on the main stem

R7 Beginning maturity—one normal pod on the main stem has reached its mature pod colour

R8 Full maturity—95% of the pods have reached their full mature colour

Sugar beet

Fifteen supervised residue trials were conducted on sugar beet in the USA and Canada in 2011. At each sites a plot was treated with CS formulations (Mueth and Foster 2012 MSL-24198). A non-ionic surfactant (0.5% v/v) and 2 kg ammonium sulphate/100L were added to the spray mixtures for all applications. One composite sample was collected from each untreated control plot and two composite samples were collected from each of the treated plots. Samples were analysed for residues of acetochlor using method AG-ME-1467. Roots EMA LOD 0.0005 mg/kg, LOQ 0.0016 mg/kg; HEMA 0.0005 mg/kg, LOQ 0.0015 mg/kg; Tops: EMA LOD 0.0012 mg/kg LOQ 0.0037 mg/kg HEMA LOD 0.00037 mg/kg, LOQ 0.0011 mg/kg.

Table 78 Residues in sugar beet following application of a CS acetochlor formulation (Mueth and Foster 2012 MSL-24198). HEMA and EMA residues are expressed in acetochlor equivalents. Replicate samples.

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SUGAR BEET		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Conklin, Michigan,	2	1.65	Pre-emergence	108	Roots	0.005	0.013	
USA 2011 18RR26		1.67	6-leaf			0.006	0.019	
					mean	0.005	0.016	0.021
	2	1.68	2-leaf	108	Roots	0.004	0.011	
		1.67	6-leaf			0.005	0.014	
					mean	0.005	0.012	0.017
	1	3.37	6-leaf	108	Roots	0.006	0.021	
						0.006	0.021	
					mean	0.006	0.021	0.027
				101	Roots	0.006	0.022	
						0.005	0.024	
						0.007	0.034	
						0.006	0.122	
					mean	0.006	0.051	0.057
				108	Roots	0.006	0.021	
						0.006	0.021	
					mean	0.006	0.021	0.027
				115	Roots	0.006	0.050	
						0.006	0.034	
					mean	0.006	0.042	0.048
				122	Roots	0.005	0.029	
						0.006	0.031	
						0.005	0.026	
						0.006	0.069	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SUGAR BEET		kg ai/ha	at application			HEMA	EMA	(mg/kg)
					mean	0.006	0.039	0.044
				129	Roots	0.006	0.036	
						0.006	0.036	
					mean	0.006	0.036	0.042
Richland, Iowa, USA	2	1.69	Pre-emergence	107	Roots	< 0.002	0.005	
2011 SX Triton		1.67	6-leaf			< 0.002	0.007	
					mean	< 0.002	0.006	< 0.008
	2	1.66	2-leaf	107	Roots	< 0.002	0.005	
		1.67	6-leaf			< 0.002	0.007	
					mean	< 0.002	0.006	< 0.008
	1	3.35	6-leaf	107	Roots	< 0.002	0.005	
						< 0.002	0.007	
					mean	< 0.002	0.006	< 0.008
York, Nebraska, USA	2	1.68	Pre-emergence	122	Roots	< 0.002	0.005	
2011 Hilleshog 9093 RR		1.66	6-leaf			< 0.002	0.006	
					mean	< 0.002	0.005	< 0.007
	2	1.66	2-leaf	122	Roots	0.004	0.008	
		1.64	6-leaf			< 0.002	0.004	
					mean	< 0.003	0.006	< 0.009
	1	3.33	6-leaf	122	Roots	0.004	0.008	
						0.004	0.012	
					mean	0.004	0.010	0.014
Geneva, Minnesota, USA	2	1.68	Pre-emergence	89	Roots	0.004	0.013	
2011 3035 RZ		1.67	6-leaf			0.004	0.011	
					mean	0.004	0.012	0.016
	2	1.67	2-leaf		Roots	0.004	0.011	
		1.67	6-leaf			0.004	0.012	
					mean	0.004	0.012	0.016
	1	3.40	6-leaf		Roots	0.004	0.017	
						0.005	0.029	
						< 0.002	0.008	
						0.003	0.009	
					mean	< 0.003	0.016	< 0.019
Perley, Minnesota, USA	2	1.73	Pre-emergence	103	Roots	0.003	0.011	
2011 SX Uplander RR		1.67	6-leaf			0.003	0.008	
					mean	0.003	0.010	0.013
	2	1.66	2-leaf	103	Roots	0.002	0.007	
		1.69	6-leaf			0.003	0.013	
						0.002	0.008	
						0.004	0.037	
					mean	0.003	0.016	0.019
	1	3.33	6-leaf	103	Roots	< 0.002	0.012	
						0.004	0.038	
						< 0.002	0.011	
						0.003	0.014	
					mean	< 0.003	0.019	< 0.022
Gardner, North Dakota,	2	1.70	Pre-emergence	103	Roots	0.003	0.009	
USA SV36812 RR		1.71	6-leaf			0.003	0.007	
					mean	0.003	0.008	0.011
	2	1.68	2-leaf	103	Roots	0.003	0.006	
		1.75	6-leaf			0.003	0.005	
					mean	0.003	0.005	0.008
	1	3.41	6-leaf	103	Roots	< 0.002	0.006	
						0.003	0.006	
						< 0.002	0.010	
						0.003	0.011	
					mean	< 0.002	0.008	< 0.010
Norwich, North Dakota,	2	1.69	Pre-emergence	93	Roots	0.003	0.016	
USA 2011 Crystal R434		1.70	6-leaf			0.004	0.026	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SUGAR BEET		kg ai/ha	at application			HEMA	EMA	(mg/kg)
						0.003	0.037	
						0.005	0.087	
					mean	0.003	0.042	0.045
	2	1.67	2-leaf	93	Roots	0.004	0.020	
		1.70	6-leaf			0.004	0.023	
						< 0.002	0.002	
						0.004	0.043	
					mean	0.003	0.022	0.025
	1	3.46	6-leaf	93	Roots	0.005	0.049	
						0.004	0.042	
					mean	0.004	0.045	0.049
Velva, North Dakota,	2	1.70	Pre-emergence	93	Roots	0.002	0.041	
USA Crystal R308		1.71	6-leaf			0.006	0.071	
						0.004	0.037	
						0.005	0.040	
					mean	0.004	0.047	0.051
	2	1.67	2-leaf	93	Roots	0.004	0.022	
		1.73	6-leaf			0.005	0.031	
					mean	0.004	0.027	0.031
	1	3.47	6-leaf	93	Roots	0.003	0.034	
						0.005	0.040	
						< 0.002	0.004	
						0.004	0.036	
					mean	< 0.003	0.029	< 0.032
Grand Island, Nebraska,	2	1.68	Pre-emergence	113	Roots	0.007	0.020	
USA 2011 Hilleshog		1.69	6-leaf			0.007	0.017	
Monogen 9093 RR					mean	0.007	0.018	0.025
	2	1.68	2-leaf	113	Roots	0.006	0.014	
		1.69	6-leaf			0.008	0.021	
					mean	0.007	0.017	0.024
	1	3.36	6-leaf	113	Roots	0.006	0.015	
						0.007	0.019	
					mean	0.006	0.017	0.023
Larned, Kansas, USA	2	1.70	Pre-emergence	63	Roots	0.004	0.013	
2011 Am Crystal R308		1.69	6-leaf			0.005	0.011	
					mean	0.005	0.012	0.017
	2	1.68	2-leaf	63	Roots	0.005	0.010	
		1.69	6-leaf			0.005	0.014	
					mean	0.005	0.012	0.017
	1	3.44	6-leaf	63	Roots	0.005	0.015	
						0.004	0.018	
					mean	0.004	0.017	0.021
Jerome, Idaho, USA	2	1.68	Pre-emergence	119	Roots	0.003	0.011	
2011 Grystal RR876		1.70	6-leaf			0.003	0.015	
					mean	0.003	0.013	0.016
	2	1.70	2-leaf	119	Roots	0.004	0.014	
		1.70	6-leaf			0.004	0.013	
					mean	0.004	0.014	0.018
	1	3.37	6-leaf	119	Roots	0.003	0.017	
						0.004	0.019	
					mean	0.003	0.018	0.021
Porterville, California,	2	1.70	Pre-emergence	83	Roots	0.015	0.078	
USA 2011 Pheonix		1.71	6-leaf			0.013	0.066	
					mean	0.014	0.072	0.086
	2	1.70	2-leaf	83	Roots	0.013	0.057	
		1.70	6-leaf			0.016	0.073	
					mean	0.014	0.065	0.079
	1	3.33	6-leaf	83	Roots	0.029	0.300	
						0.020	0.198	
					mean	0.025	0.249	0.274

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SUGAR BEET		kg ai/ha	at application			HEMA	EMA	(mg/kg)
				76	Roots	0.013	0.133	
						0.015	0.176	
						0.021	0.177	
						0.026	0.235	
					mean	0.019	0.180	0.199
				83	Roots	0.029	0.300	
						0.020	0.198	
					mean	0.025	0.249	0.274
				90	Roots	0.023	0.216	
						0.031	0.332	
					mean	0.027	0.274	0.301
				98	Roots	0.015	0.143	
						0.015	0.195	
					mean	0.015	0.169	0.184
				104	Roots	0.017	0.186	
						0.023	0.284	
					mean	0.020	0.235	0.255
Ephrata, Washington,	2	1.68	Pre-emergence	131	Roots	0.006	0.018	
USA Crystal RR876		1.68	6-leaf			0.005	0.013	
					mean	0.005	0.016	0.021
	2	1.68	2-leaf	131	Roots	0.003	0.015	
		1.69	6-leaf			0.003	0.017	
					mean	0.003	0.016	0.019
	1	3.35	6-leaf	131	Roots	0.004	0.016	
						0.003	0.017	
					mean	0.003	0.017	0.020
Rupert, Idaho, USA 2011	2	1.60	Pre-emergence	113	Roots	0.003	0.007	
Crystal RR929		1.69	6-leaf			0.003	0.010	
					mean	0.003	0.008	0.011
	2	1.69	2-leaf	113	Roots	0.003	0.006	
		1.65	6-leaf			0.004	0.008	
					mean	0.003	0.007	0.010
	1	3.34	6-leaf	113	Roots	0.003	0.012	
						0.003	0.018	
					mean	0.003	0.015	0.018
Minto, Manitoba, Canada	2	1.74	Pre-emergence	89	Roots	0.004	0.009	
2011 SVDH 66854		1.84	6-leaf			0.002	0.016	
						0.002	0.009	
						0.004	0.016	
					mean	0.003	0.012	0.015
	2	1.70	2-leaf	89	Roots	0.003	0.007	
		1.67	6-leaf			0.003	0.009	
					mean	0.003	0.008	0.011
	1	3.35	6-leaf	89	Roots	0.002	0.007	
						0.003	0.008	
					mean	0.002	0.007	0.009

Maize

Oppenhuizen and Wilson (1989 MSL-6843) studied residues of acetochlor in corn (field and sweet) from 12 different trial sites in the USA. An EC formulation (MON-097 in tank mixed with MON-4666 ratio 1:10) was applied a single pre-emergence application at 1.7, 3.4 or 6.7 kg ai/ha. Forage samples (4.5 kg) were collected 8 weeks after application and fodder (4.5 kg) and grain (11 kg) at commercial harvest. The LOD for corn forage is 0.005 mg/kg for EMA-class metabolites and 0.006 mg/kg for HEMA-class metabolites. The LOQ is 0.017 mg/kg for EMA-class metabolites and

0.018 mg/kg for HEMA-class metabolites. Results are corrected for the average analytical recovery of the method.

Table 79 Residues in maize and sweet corn (two trials only) following application of an acetochlor EC formulation (Oppenhuizen and Wilson 1989 MSL-6843) HEMA and EMA residues are expressed in acetochlor equivalents. Results are for samples analysed in duplicate.

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg) [a]	Total [a]
MAIZE		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Gretna, NE 1985 DK Xl73	1	1.7	Pre-emergent	147	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	147	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	147	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
Isleton, CA 1985 Funks	1	1.7	Pre-emergent	163	Grain	< 0.02	< 0.02	
4438						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	163	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	163	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
Otterbein, IN 1985 Sweet	1	1.7	Pre-emergent	86	Grain	< 0.02	< 0.02	
Corn						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	86	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	86	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
Princeton, IA 1985	1	1.7	Pre-emergent	151	Grain	< 0.02	< 0.02	
Pioneer 33/78						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	151	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	151	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
Redfield, IA 1985 Lynks	1	1.7	Pre-emergent	149	Grain	< 0.02	< 0.02	
4330						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	149	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	149	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
Reeds Corner, NY 1985	1	1.7	Pre-emergent	167	Grain	< 0.02	< 0.02	
Cargil 815						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	167	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	167	Grain	< 0.02	< 0.02	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg) [a]	Total [a]
MAIZE		kg ai/ha	at application			HEMA	EMA	(mg/kg)
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
Reevesville, SC 1985	1	1.7	Pre-emergent	138	Grain	< 0.02	< 0.02	
PN3320						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	138	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	138	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
Trenton, TN 1985 O's	1	1.7	Pre-emergent	157	Grain	< 0.02	< 0.02	
Gold 3344						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	157	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	157	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
Waseca, MN 1985 Sweet	1	1.7	Pre-emergent	84	Grain	< 0.02	< 0.02	
corn Jubilee						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	84	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	84	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
Waukee, IA 1985 Funks	1	1.7	Pre-emergent	168	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	168	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	168	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
Williamston, MI 1985	1	1.7	Pre-emergent	163	Grain	< 0.02	< 0.02	
DK2120						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	163	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	163	Grain	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04

[a] Results are corrected for the average analytical recovery of the method

Ralph *et al.* (1992 RJ1337B) studied residues of 5-hydroxy *sec*-oxanilic acid (68) in 14 trials conducted in the USA where acetochlor, formulated as an EC formulation incorporating the safener R25788, was applied to the soil surface immediately after planting field corn. All treatments were made as a single application at a rate of 2.8 kg ai/ha, with the exception of one trial carried out in Colorado, which was mistakenly treated at 4.5 kg ai/ha. The analytical method used was reported in RJ1257B. Samples were analysed in duplicate.

Table 80 Residues of 5-hydroxy *sec*-oxanilic acid (68) in maize following application of an acetochlor EC formulation as a single pre-emergent application at 2.8 kg ai/ha (Ralph *et al.* 1992 RJ1337B) Single field samples analysed in duplicate.

Country/ location MAIZE	Crop growth stage	Sample	DALA	Analysis 1	Analysis 2	mean
Whitakers North Carolina USA 1991	Pre-emergence	Grain	137	< 0.01	< 0.01	< 0.01
Visalia, California, USA 1991	Pre-emergence	Grain	122	< 0.01	< 0.01	< 0.01
Champaign Illinois USA 1991	Pre-emergence	Grain	139	< 0.01	< 0.01	< 0.01
Ephrata Washington, USA 1991	Pre-emergence	Grain	165	< 0.01	< 0.01	< 0.01
Paynesville Minnesota USA 1991	Pre-emergence	Grain	148	< 0.01	< 0.01	< 0.01
York Nebraska, USA 1991	Pre-emergence	Grain	126	< 0.01	< 0.01	< 0.01
Iconium Iowa USA 1991	Pre-emergence	Grain	145	< 0.01	< 0.01	< 0.01
Berthoud Colorado USA 1991	Pre-emergence	Grain	161 [a]	< 0.01	< 0.01	< 0.01
Noblesville Indiana USA 1991	Pre-emergence	Grain	147	< 0.01	< 0.01	< 0.01
Sudlerville Maryland USA 1991	Pre-emergence	Grain	175	< 0.01	< 0.01	< 0.01
Fabius New York USA 1991	Pre-emergence	Grain	153	< 0.01	< 0.01	< 0.01
Fabius New York USA 1991	Pre-emergence	Grain	147	< 0.01	< 0.01	< 0.01
Germansville Pennsylvania USA 1991	Pre-emergence	Grain	138	< 0.01	< 0.01	< 0.01
Pulaski Pennsylvania USA 1991	Pre-emergence	Grain	138	< 0.01	< 0.01	< 0.01

[a] Application was 4.5 kg ai/ha

Lau (1992 MSL-11794) conducted 14 trials on maize in the USA. At each site, a CS formulation of acetochlor was applied to field corn as a pre-emergent application (all sites) or pre-plant incorporation (six sites) application at a nominal rate of 3.4 kg ai/ha. Pre-emergent applications were also done at 1.7 kg ai/ha at two sites. The CS formulation was applied as a tank-mix with MON 13900 (3-(dichloroacetyl)-5-(2-furyl)-2,2-dimethyloxazolidine, 2,2-dichloro-1-[5-(2-furyl)-2,2-dimethyl-oxazolidin-3-yl]ethanone = safener). Forage samples were collected six to twelve weeks after planting. Silage samples were taken at the dent stage, and grain and fodder collected at normal harvest.

Table 81 Residues in maize following application of a CS acetochlor formulation Lau (1992 MSL-11794) HEMA and EMA residues are expressed in acetochlor equivalents. Single field samples analysed in duplicate.

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Colo, Iowa, USA	1	3.4	Pre-emergent	124	Grain	< 0.01	< 0.01	
1990 DK535						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
	1	3.4	Pre-plant	136	Grain	< 0.01	< 0.01	
						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
	1	1.7	Preemergent	130	Grain	< 0.01	< 0.01	
						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
Conklin, Michigan, USA	1	3.4	Pre-emergent	145	Grain	< 0.01	< 0.01	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE		kg ai/ha	at application			HEMA	EMA	(mg/kg)
1990 Pioneer 3751						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
Danville, Iowa, USA	1	3.4	Pre-emergent	156	Grain	< 0.01	< 0.01	
1990 Dockendorf 7670						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
Delavan, Wisconsin,	1	3.4	Pre-emergent	173	Grain	< 0.01	< 0.01	
USA 1990 RK627						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
Elwood, Illinois USA	1	3.4	Pre-emergent	142	Grain	< 0.01	< 0.01	
1990 Pioneer 3615						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
Geneseo, Illinois USA	1	3.4	Pre-emergent	147	Grain	< 0.01	< 0.01	
USA 1990 Pioneer 3615						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
	1	3.4	Pre-plant	147	Grain	< 0.01	< 0.01	
						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
	1	1.7	Pre-emergent	147	Grain	< 0.01	< 0.01	
						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
Hollandale, Minnesota,	1	3.4	Pre-emergent	138	Grain	< 0.01	< 0.01	
USA 1990 Pioneer 3751						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
	1	3.4	Pre-plant	138	Grain	< 0.01	< 0.01	
						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
Elk City Kansas, USA	1	3.4	Pre-emergent	143	Grain	< 0.01	< 0.01	
1990 Cargil 6127						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
Leonard, Missouri, USA	1	3.4	Pre-emergent	127	Grain	< 0.01	< 0.01	
1990 McAllister						< 0.01	< 0.01	
SX8611RFR					mean	< 0.01	< 0.01	< 0.02
	1	3.4	Pre-plant	127	Grain	< 0.01	< 0.01	
						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
New Holland Ohio, USA	1	3.4	Pre-emergent	189	Grain	< 0.01	< 0.01	
1990 Pioneer 3343						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
	1	3.4	Pre-plant	191	Grain	< 0.01	< 0.01	
						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
Noblesville Indiana,	1	3.4	Pre-emergent	148	Grain	< 0.01	< 0.01	
USA 1990 Pioneer 3744						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
Sioux Falls South Dakota	1	3.4	Pre-emergent	135	Grain	< 0.01	< 0.01	
USA 1990 Moews 3140						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
Uvalde Texas, USA	1	3.4	Pre-emergent	132	Grain	< 0.01	< 0.01	
1990 Pioneer 3192						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
York Nebraska, USA	1	3.4	Pre-emergent	162	Grain	< 0.01	< 0.01	
1990 Pioneer 3379						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
	1	3.4	Pre-plant	162	Grain	< 0.01	< 0.01	
						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02

Values have been corrected for analytical method recoveries and expressed as acetochlor equivalents for either EMA (ethylmethylaniline producing) or HEMA (hydroxyethylrnethylaniline producing) residues

Maize

Twenty-one supervised residue trials were conducted on maize in the USA in 2006. At each sites a plot was treated with either CS (microencapsulated) or EC formulations (Maher 2007 MSL-20269). An herbicide safener, furilazole, was used in the spray mix for each application. A single control and duplicate treated samples of corn forage, grain, and stover were collected from each test plot. The interval between sampling and extraction for the acetochlor samples was 175 days for grain, 374 days for forage and 359 days for stover. Samples were analysed for residues of acetochlor using the LC-MS/MS method ES-ME-1001-02.

Table 82 Residues in maize following application of an EC or a CS acetochlor formulation (Maher 2007 MSL-20269). HEMA and EMA residues are expressed in acetochlor equivalents. Replicate samples.

Location, year, variety	Form	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE			kg ai/ha	at application			HEMA	EMA	(mg/kg)
Richland, Iowa, USA	EC	1	3.10 PO	V8	103	Grain	0.001	0.003	
2006 Dekalb DKC51-39				68–81 cm			0.001	0.002	
						mean	0.001	0.002	0.003
	CS	1	3.33 PO	V8	103	Grain	0.001	0.007	
				68–81 cm			0.001	0.007	
						mean	0.001	0.007	0.008
Hedrick, Iowa, USA	EC	1	2.97 PO	V7–V8	106	Grain	< 0.001	0.007	
2006 Pioneer 34A16				71–84 cm			< 0.001	0.006	
						mean	< 0.001	0.006	< 0.007
	CS	1	3.17 PO	V7–V8	106	Grain	< 0.001	0.008	
				71–84 cm			< 0.001	0.007	
						mean	< 0.001	0.008	< 0.009
Richland, Iowa, USA	EC	1	2.96 PO	V8	108	Grain	< 0.001	0.002	
2006 Middle Koop 2212				71–86 cm			< 0.001	0.002	
						mean	< 0.001	0.002	< 0.003
	CS	1	3.18 PO	V8	108	Grain	< 0.001	0.002	
				71–86 cm			0.002	0.001	
						mean	< 0.002	0.002	< 0.004
Perry, Iowa, USA 2006	EC	1	2.96 PO	V8	96	Grain	0.003	0.013	
Pioneer 36B10				69–86 cm			0.002	0.011	
							0.002	0.011	
							0.002	0.010	
						mean	0.002	0.011	0.013
	EC	1	1.47 PE	V8	96	Grain	0.002	0.007	
			1.50 PO	69–86 cm			0.002	0.005	
						mean	0.002	0.006	0.008
	EC	1	2.88 PO	V6	108	Grain	0.004	0.002	
				46–51 cm			0.002	0.001	
						mean	0.003	0.002	0.005
	CS	1	3.27 PO	V8	96	Grain	0.002	0.007	
				69–86 cm			0.003	0.007	
						mean	0.002	0.007	0.009
Bagley, Iowa, USA 2006	EC	1	2.96 PO	V8	97	Grain	0.001	0.003	
Pioneer 33P65				66–89 cm			< 0.001	0.003	
						mean	< 0.001	0.003	< 0.004
	CS	1	3.31 PO	V8	97	Grain	< 0.001	0.001	
				66–89 cm			< 0.001	< 0.001	
						mean	< 0.001	< 0.001	< 0.002
Carlyle, Illinois, USA	EC	1	2.89 PO	V9	121	Grain	< 0.001	0.007	
2006 DKC61-45				66–86 cm			< 0.001	0.007	
						mean	< 0.001	0.007	< 0.008

Location, year, variety	Form	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE			kg ai/ha	at application			HEMA	EMA	(mg/kg)
	CS	1	3.13 PO	V9	121	Grain	0.002	0.003	
				66–86 cm			0.001	0.008	
						mean	0.002	0.006	0.008
Mason, Illinois, USA	EC	1	3.06 PO	BBCH 18	100	Grain	0.001	0.011	
2006 Midland mg 606RR				66–91 cm			0.001	0.009	
						mean	0.001	0.010	0.011
	CS	1	3.32 PO	BBCH 18	100	Grain	0.004	0.015	
				66–91 cm			0.004	0.015	
						mean	0.004	0.015	0.019
Wyoming, Illinois, USA	EC	1	2.95 PO	V8	114	Grain	0.001	0.005	
2006 Burns 644 RWR				74–79 cm			0.001	0.005	
						mean	0.001	0.005	0.006
	CS	1	3.15 PO	V8	114	Grain	< 0.001	0.002	
				74–79 cm			0.001	0.002	
						mean	< 0.001	0.002	< 0.003
Danville, Indiana, USA	EC	1	2.82 PO	BBCH 18	140	Grain	< 0.001	0.002	
2006 Wyffels W5531				66–91 cm			< 0.001	0.001	
						mean	< 0.001	0.002	< 0.003
	CS	1	3.19 PO	BBCH 18	140	Grain	< 0.001	0.001	
				61–91 cm			< 0.001	0.001	
						mean	< 0.001	0.001	< 0.002
Rockville, Indiana, USA	EC	1	2.82 PO	BBCH 18	130	Grain	< 0.001	0.003	
2006 Pioneer 33NO8				66–86 cm			< 0.001	0.004	
						mean	< 0.001	0.004	0.005
	CS	1	3.33 PO	BBCH 18	130	Grain	0.001	0.004	
				71–91 cm			0.002	0.005	
						mean	0.002	0.004	0.006
Paynesville, Minnesota,	EC	1	2.93 PO	V8	123	Grain	< 0.001	< 0.001	
USA 2006 Dekalb				71–86 cm			< 0.001	< 0.001	
DKC47-10 RR2						mean	< 0.001	< 0.001	< 0.002
	CS	1	3.19 PO	V8	123	Grain	< 0.001	< 0.001	
				71–86 cm			< 0.001	< 0.001	
						mean	< 0.001	< 0.001	< 0.002
Hawick, Minnesota, USA	EC	1	2.87 PO	76 cm	123	Grain	< 0.001	< 0.001	
2006 Dekalb DKC47-10							< 0.001	< 0.001	
RR2						mean	< 0.001	< 0.001	< 0.002
	CS	1	3.22 PO	76 cm	123	Grain	< 0.001	< 0.001	
							< 0.001	< 0.001	
						mean	< 0.001	< 0.001	< 0.002
LaPlata, Missouri, USA	EC	1	3.04 PO	V8	103	Grain	< 0.001	0.001	
2006 Dekalb DKC61-42				71–79 cm			< 0.001	< 0.001	
						mean	< 0.001	< 0.001	< 0.002
	EC	2	1.43 PE	V8	103	Grain	< 0.001	0.002	
			1.49 PO	71–79 cm			< 0.001	< 0.001	
						mean	< 0.001	< 0.002	< 0.003
	EC	1	2.90 PO	V6	110	Grain	< 0.001	0.001	
				46–51 cm			< 0.001	0.001	
						mean	< 0.001	0.001	< 0.002
	CS	1	3.26 PO	V8	103	Grain	< 0.001	< 0.001	
				71–79 cm			< 0.001	0.001	
						mean	< 0.001	< 0.001	< 0.002
	CS	2	1.61 PE	V8	103	Grain	< 0.001	< 0.001	
			1.61 PO	71–79 cm			< 0.001	< 0.001	
						mean	< 0.001	< 0.001	< 0.002
	CS	1	3.19 PO	V6	110	Grain	0.001	0.002	

Location, year, variety	Form	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE			kg ai/ha	at application			HEMA	EMA	(mg/kg)
				46–51 cm			< 0.001	< 0.001	
						mean	< 0.001	< 0.002	< 0.003
Seven Springs, North	EC	1	2.96 PO	BBCH 33	83	Grain	0.001	0.006	
Carolina, USA 2006				71–86 cm			< 0.001	0.004	
Garst 8377						mean	< 0.001	0.005	< 0.006
	CS	1	3.24 PO	BBCH 33	83	Grain	0.002	0.007	
				71–86 cm			0.002	0.007	
						mean	0.002	0.007	0.009
York, Nebraska, USA	EC	1	2.94 PO	BBCH 18	106	Grain	0.002	0.002	
2006 Pioneer				69–81 cm			0.002	0.002	
34N45 RR2/YGCB						mean	0.002	0.002	0.004
	CS	1	3.18 PO	BBCH 18	106	Grain	< 0.001	0.003	
				69–81 cm			< 0.001	0.002	
						mean	< 0.001	0.002	< 0.003
Osceola, Nebraska, USA	EC	1	2.94 PO	BBCH 18	103	Grain	0.001	0.006	
2006 N73-F7 RR/LL/CB				66–81 cm			0.002	0.007	
						mean	0.002	0.006	0.008
	CS	1	3.16 PO	BBCH 18	103	Grain	0.001	0.002	
				66–81 cm			0.001	0.003	
						mean	0.001	0.002	< 0.003
Baptistown, New Jersey,	EC	1	3.00 PO	V8	97	Grain	< 0.001	0.002	
USA TA5750/ 401169				61–91 cm			< 0.001	0.002	
						mean	< 0.001	0.002	< 0.003
	CS	1	3.26 PO	V8	97	Grain	< 0.001	0.002	
				61–91 cm			< 0.001	0.002	
						mean	< 0.001	0.002	< 0.003
Washington, Ohio, USA	EC	1	2.97 PO	V8–V9	110	Grain	0.001	0.008	
2006 SC 11RR06				71–84 cm			< 0.001	0.007	
						mean	< 0.001	0.008	< 0.009
	CS	1	3.14 PO	V8–V9	110	Grain	0.002	0.005	
				71–84 cm			0.001	0.003	
						mean	0.002	0.004	0.006
New Holland, Ohio, USA	EC	1	3.00 PO	V8	120	Grain	< 0.001	0.006	
2006 Crows 515Z R				71–79 cm			< 0.001	0.003	
						mean	< 0.001	0.004	< 0.005
	CS	1	3.17 PO	V8	120	Grain	< 0.001	0.003	
				71–84 cm			< 0.001	0.002	
						mean	< 0.001	0.002	< 0.003
Dill City, Oklahoma, USA	EC	1	2.90 PO	V8–V9	89	Grain	0.001	0.008	
2006 DK C48-53				74–81 cm			0.001	0.007	
						mean	0.001	0.008	0.009
	CS	1	3.19 PO	V8–V9	89	Grain	< 0.001	0.003	
				71–81 cm			< 0.001	0.003	
						mean	< 0.001	0.003	< 0.004
Delavan, Wisconsin, USA	EC	1	2.89 PO	V8	124	Grain	< 0.001	0.001	
2006 Dekalb DKC51-39				74–79 cm			< 0.001	0.002	
						mean	< 0.001	0.002	< 0.003
	CS	1	3.09 PO	V8	124	Grain	< 0.001	< 0.001	
				74–79 cm			0.001	0.003	
						mean	< 0.001	< 0.002	< 0.003

PE = pre-emergent

PO = post-emegent

EMA LOD 0.0006 mg/kg, LOQ 0.0012 mg/kg

HEMA LOD 0.0007 mg/kg, LOQ 0.0012 mg/kg

Growth Stages

VE Corn emergence, coleoptiles break through soil surface

V1 First leaf fully emerged and leaf collar visible

V2 Second leaf fully emerged and leaf collar visible

V(n) n^{th} leaf fully emerged

VT last branch visible but silks not emerged

R1 Beginning silking—silk visible outside of husk

R2 Blister stage—kernel is white and shaped like a blister

R3 Milk stage—kernel is yellow with white milky inner liquid

R4 Dough stage—inner fluid begins to thicken due to stach accumulation

R5 Dent stage—kernels begin to dry down from the top of the kernel toward the cob. Each kernel will have a dent at the top.

R6 Full maturity—black layer forms where kernel attaches the cob. Kernel moisture is at 30–35%.

BBCH 18 leaf development: 18^{th} leaf unfolded

BBCH 33 stem elongation: 3 nodes detectable

Sorghum

Moran (2004 MSL-18670) studied residues in sorghum in 13 field trials conducted in 2003. Acetochlor as a CS formulation (controlled release suspension capsule) was applied in side-by-side tests at each trial site as either a Pre-emergence or an early post-emergence (plants $\leq$ 28 cm) application at a rate of 2.8 kg ai/ha. All applications were made using ground equipment in spray volumes of 94 to 188 L/ha. At two sites, food grade (white) sorghum was planted and used for processing while at the others typical animal feed sorghum varieties were used. Because sorghum is sensitive to acetochlor, the test substance included the safener furilazole at 0.50%. At crop maturity, single control and duplicate treated samples of grain (90 to 171 DAT) and stover (93 to 177 DAT) were collected from each test. Stover was left in the field to dry at four sites (Plains Georgia, York, Osceola and Grand Island Nebrasha) where the stover was too green and contained too much moisture at the appropriate grain harvest time. Samples were stored frozen for durations of up to 222 days prior to analysis of acetochlor residues. The LC/MS/MS Method ES-ME-1001-01 used to determine residues of EMA and HEMA class metabolites in sorghum forage, grain, and stover. The LOQs for EMA were 0.005 mg/kg in forage, 0.005 mg/kg in grain, and 0.015 mg/kg in stover, while the LOQs for HEMA were 0.003 mg/kg in forage, 0.003 mg/kg in grain, and 0.011 mg/kg in stover.

Table 83 Residues in sorghum following application of an EC or a CS acetochlor formulation Moran (2004 MSL-18670). HEMA and EMA residues are expressed in acetochlor equivalents. Replicate samples.

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SORGHUM		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Plains, Georgia	1	2.77	PE	107	Grain	0.006	< 0.005	
USA 2003 A571						0.006	0.006	
					mean	0.006	0.006	0.012
	1	2.79	PO	93	Grain	0.008	0.008	
			15–20 cm			0.009	0.009	
					mean	0.008	0.008	0.016
Cord, Arkansas, USA	1	2.78	PE	123	Grain	< 0.005	< 0.005	
2003 Garst 5515						< 0.005	< 0.005	
					mean	< 0.005	< 0.005	< 0.010
	1	2.80	PO	104	Grain	< 0.005	< 0.005	
			23 cm			< 0.005	< 0.005	
					mean	< 0.005	< 0.005	< 0.010
Carlyle, Illinois, USA	1	2.87	PE	133	Grain	< 0.005	< 0.005	
2003 KS 585						< 0.005	< 0.005	
					mean	< 0.005	< 0.005	< 0.010

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SORGHUM		kg ai/ha	at application			HEMA	EMA	(mg/kg)
	1	2.89	PO	104	Grain	< 0.005	< 0.005	
			25 cm			< 0.005	< 0.005	
					mean	< 0.005	< 0.005	< 0.010
New Holland, Ohio, USA	1	2.73	PE	160	Grain	< 0.005	< 0.005	
2003 A571						< 0.005	< 0.005	
					mean	< 0.005	< 0.005	< 0.010
	1	2.78	PO	121	Grain	< 0.005	< 0.005	
			25 cm			< 0.005	< 0.005	
					mean	< 0.005	< 0.005	< 0.010
York, Nebraska,	1	2.79	PE	138	Grain	0.011	0.018	
USA 2003 Eclipse						0.007	0.008	
					mean	0.009	0.013	0.022
	1	2.80	PO	112	Grain	0.015	0.021	
			13–15 cm			0.013	0.018	
					mean	0.014	0.019	0.033
Richland, Iowa, USA					Grain	c0.005	c0.007	c0.012
2003 Dekaalb AS71	1	2.86	PE	134	Grain	< 0.005	< 0.005	
						< 0.005	< 0.005	
					mean	< 0.005	< 0.005	< 0.010
	1	2.80	PO	104	Grain	< 0.005	< 0.005	
			28 cm			< 0.005	< 0.005	
					mean	< 0.005	< 0.005	< 0.010
Osceola, Nebraska, USA	1	2.80	PE	147	Grain	0.006	0.008	
2003 NC+6B50						< 0.005	< 0.005	
					mean	< 0.006	< 0.006	< 0.012
	1	2.81	PO	115	Grain	0.006	0.008	
			15–20 cm			0.010	0.014	
					mean	0.008	0.011	0.019
Colony, Oklahoma, USA	1	2.77	PE	133	Grain	< 0.005	< 0.005	
2003 Cherokee						< 0.005	< 0.005	
					mean	< 0.005	< 0.005	< 0.010
	1	2.82	PO	96	Grain	0.010	0.021	
			30–35 cm			0.011	0.020	
					mean	0.010	0.020	0.030
East Bernard, Texas,	1	2.79	PE	113	Grain	0.005	0.010	
USA 2003 DKS36-00						0.005	0.010	
					mean	0.005	0.010	0.015
	1	2.86	PO	90	Grain	0.007	0.012	
			25–28 cm			0.006	0.012	
					mean	0.006	0.012	0.018
Grand Island, Nebraska,	1	2.79	PE	148	Grain	0.004	0.012	
USA 2003 NC+6B50						0.004	0.012	
					mean	0.004	0.012	0.016
	1	2.80	PO	120	Grain	0.006	0.018	
			13–15 cm			0.008	0.022	
					mean	0.007	0.020	0.027
Dill City, Oklahoma,	1	2.89	PE	133	Grain	0.006	0.006	
USA 2003 Eclipse						0.006	0.005	
					mean	0.006	0.006	0.012
	1	2.80	PO	97	Grain	0.006	0.010	
			28–36 cm			0.007	0.011	
					mean	0.006	0.010	0.016
Claude, Texas, USA	1	2.81	PE	171	Grain	< 0.005	< 0.005	
2003 Y363						< 0.005	< 0.005	
					mean	< 0.005	< 0.005	< 0.010
	1	2.84	PO	158	Grain	< 0.005	< 0.005	
			15 cm			< 0.005	< 0.005	
					mean	< 0.005	< 0.005	< 0.010

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SORGHUM		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Levelland, Texas, USA	1	2.86	PE	119	Grain	< 0.005	0.010	
2003 F-270E						< 0.005	0.009	
					mean	< 0.005	0.010	< 0.015
	1	2.84	PO	98	Grain	< 0.005	< 0.005	
			15–28 cm			< 0.005	0.008	
					mean	< 0.005	< 0.006	< 0.011

PE = pre-emergent

PO = post-emegent

Cotton

Hay *et al.* 2008 (MSL-20718) studied residues in cotton seed following pre-plant/post-emergence or post-emergence applications of either CS (microencapsulated) or EC formulations of acetochlor. Both formulations contain a safener although this is not effective in the case of cotton. Applications were made as a single spray at 3.4 kg ai/ha late post-emergence (1st flower) or at the 8-leaf stage or as a split treatment with an application made pre-plant (about 30 days before planting) and another at the 8-leaf stage. Cotton was harvested by commercial-type equipment (stripper or mechanical picker) at all but two sites (Dill City and Tulare), where handheld clippers were used. A single control and duplicate treated samples of seed and gin by-products were collected from each test Samples were analysed for residues using LC-MS/MS method ES-ME-1215-01. The LOQs are 0.005 mg/kg for EMA and HEMA in seed and 0.06 mg/kg for EMA in forage and 0.014 mg/kg for HEMA in forage.

Table 84 Residues in cotton seed following application of a micro-encapsulated (CS) acetochlor formulation (Hay *et al.* 2008 MSL-20718). HEMA and EMA residues are expressed in acetochlor equivalents. Replicate samples.

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
COTTON		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Proctor, Arkansas, USA	1	3.37	16 nodes,	74	Undelinted	0.026	0.095	
2007 ST4554B2RF			midbloom		seed	0.047	0.172	
					mean	0.036	0.133	0.169
	1	3.37	8 nodes	107	Undelinted	< 0.005	0.006	
					seed	< 0.005	0.009	
					mean	< 0.005	0.008	< 0.013
	2	1.67	Pre-plant	107	Undelinted	< 0.005	< 0.005	
		1.68	8 nodes		seed	< 0.005	0.009	
					mean	< 0.005	< 0.007	< 0.012
Newport, Arkansas, USA	1	3.41	BBCH 65	83	Undelinted	0.17	0.205	
2007 DP 143 B2RF					seed	0.075	0.135	
					mean	0.123	0.17	0.293
	1	3.35	BBCH 18	122	Undelinted	< 0.005	< 0.005	
					seed	< 0.005	< 0.005	
					mean	< 0.005	< 0.005	< 0.010
	2	1.69	Pre-plant	122	Undelinted	< 0.005	< 0.005	
		1.67	BBCH 18		seed	< 0.005	< 0.005	
					mean	< 0.005	< 0.005	< 0.010
Yuma, Arizona, USA	1	3.44	BBCH 64	107	Undelinted	0.057	0.266	
2007 DP 44S BG/ RR					seed	0.071	0.336	
					mean	0.064	0.301	0.365
	1	3.34	BBCH 17–18	134	Undelinted	0.01	0.055	
					seed	0.018	0.088	
					mean	0.014	0.071	0.085
	2	1.68	Pre-plant	134	Undelinted	0.009	0.046	
		1.68	BBCH 17–18		seed	0.013	0.057	
					mean	0.011	0.051	0.063
Porterville, California,	1	3.36	BBCH 64	91	Undelinted	0.066	0.239	
USA 2007 Roundup					seed	0.042	0.165	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
COTTON		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Ready / Bollgard					mean	0.054	0.202	0.256
	1	3.38	BBCH 18	113	Undelinted	0.011	0.036	
					seed	0.013	0.037	
					mean	0.012	0.037	0.049
	2	1.68	Pre-plant	113	Undelinted	0.006	0.014	
		1.68	BBCH 18		seed	0.006	0.015	
					mean	0.006	0.014	0.021
Tulare, California, USA	1	3.35	BBCH 60 first	133	Undelinted	0.101	0.297	
2007 Phytogen 725RR			flowers opened		seed	0.056	0.170	
					mean	0.079	0.233	0.312
	1	3.36	BBCH 18	154	Undelinted	< 0.005	0.012	
					seed	0.006	0.037	
					mean	< 0.006	0.024	< 0.030
	2	1.68	Pre-plant	154	Undelinted	< 0.005	0.008	
		1.68	BBCH 18		seed	< 0.005	< 0.005	
					mean	< 0.005	0.006	< 0.011
Chula, Georgia, USA	1	3.35	1st white flower	91	Undelinted	0.005	0.016	
2007 782-A-5091-61A			+ 7° days		seed	0.006	0.014	
					mean	0.006	0.015	0.021
	1	3.40	7–8 leaf stage	123	Undelinted	< 0.005	< 0.005	
					seed	0.005	0.005	
					mean	< 0.005	< 0.005	< 0.010
	2	1.68	Pre-plant	123	Undelinted	< 0.005	< 0.005	
		1.69	7–8 leaf stage		seed	< 0.005	< 0.005	
					mean	< 0.005	< 0.005	< 0.010
Cheneyville, Louisiana,	1	3.37	BBCH 65	116	Undelinted	< 0.005	0.035	
USA 2007 DPL143RRF/			mid-flower		seed	0.005	0.045	
BII					mean	< 0.005	0.04	< 0.045
	1	3.34	BBCH 19	153	Undelinted	< 0.005	< 0.005	
					seed	< 0.005	< 0.005	
					mean	< 0.005	< 0.005	< 0.010
	2	1.77	Pre-plant	153	Undelinted	< 0.005	< 0.005	
		1.68	BBCH 19		seed	< 0.005	< 0.005	
					mean	< 0.005	< 0.005	< 0.010
Dill City, Oklahoma, USA	1	3.34	BBCH 65	84	Undelinted	0.047	0.187	
2007 ST 4554 B2RF					seed	0.055	0.224	
					mean	0.051	0.206	0.257
	1	3.36	BBCH 18	118	Undelinted	0.008	0.008	
					seed	0.01	0.011	
					mean	0.009	0.009	0.019
	2	1.67	Pre-plant	118	Undelinted	< 0.005	< 0.005	
		1.66	BBCH 18		seed	0.005	0.006	
					mean	< 0.005	< 0.006	< 0.011
Uvalde, Texas, USA 2007	1	3.34	BBCH 65	84	Undelinted	< 0.005	0.007	
DP 143 B2RF					seed	< 0.005	0.010	
					mean	< 0.005	0.009	< 0.014
	1	3.34	BBCH 18	119	Undelinted	< 0.005	< 0.005	
					seed	< 0.005	< 0.005	
					mean	< 0.005	< 0.005	< 0.010
	2	1.68	Pre-plant	119	Undelinted	< 0.005	< 0.005	
		1.67	BBCH 18		seed	< 0.005	< 0.005	
					mean	< 0.005	< 0.005	< 0.010
LaPryor, Texas, USA	1	3.31	BBCH 65	65	Undelinted	0.012	0.079	
2007 Delta Pine 117			mid bloom		seed	0.014	0.092	
B2RF					mean	0.013	0.086	0.098
	1	3.36	BBCH 18–19	100	Undelinted	< 0.005	0.016	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
COTTON		kg ai/ha	at application			HEMA	EMA	(mg/kg)
					seed	< 0.005	0.011	
					mean	< 0.005	0.014	< 0.019
	2	1.67	Pre-plant	100	Undelinted	< 0.005	< 0.005	
		1.66	BBCH 18–19		seed	< 0.005	0.005	
					mean	< 0.005	< 0.005	< 0.010
Levelland, Texas, USA	1	3.36	BBCH 63	70	Undelinted	0.064	0.238	
2007 FM 9063B2F					seed	0.075	0.292	
					mean	0.07	0.265	0.335
				76	Undelinted	0.023	0.097	
					seed	0.023	0.11	
						0.023	0.1	
						0.03	0.142	
					mean	0.025	0.112	0.137
				83	Undelinted	0.03	0.138	
					seed	0.029	0.13	
					mean	0.029	0.134	0.163
				91	Undelinted	0.026	0.14	
					seed	0.026	0.12	
					mean	0.026	0.13	0.156
	1	3.36	BBCH 18	112	Undelinted	0.005	0.01	
					seed	< 0.005	0.007	
					mean	< 0.005	0.009	< 0.014
	2	1.69	Pre-plant	112	Undelinted	< 0.005	0.007	
		1.66	BBCH 18		seed	0.005	0.007	
					mean	< 0.005	0.007	< 0.012
Wolfforth, Texas, USA	1	3.32	BBCH 63	86	Undelinted	0.016	0.087	
2007 ST 45357 B2RF					seed	0.016	0.084	
					mean	0.016	0.085	0.101
	1	3.50	BBCH 19	121	Undelinted	< 0.005	0.011	
					seed	0.006	0.017	
					mean	< 0.005	0.014	< 0.019
	2	1.71	Pre-plant	121	Undelinted	< 0.005	0.008	
		1.69	BBCH 19		seed	< 0.005	0.01	
					mean	< 0.005	0.009	< 0.014
Claude, Texas, USA	1	3.36	BBCH 65	64	Undelinted	0.02	0.134	
2007 NG3550					seed	0.018	0.111	
					mean	0.019	0.123	0.142
	1	3.36	BBCH 18	106	Undelinted	< 0.005	0.024	
					seed	< 0.005	0.026	
					mean	< 0.005	0.025	< 0.030
	2	1.68	Pre-plant	106	Undelinted	< 0.005	0.009	
		1.69	BBCH 18		seed	< 0.005	0.016	
					mean	< 0.005	0.013	< 0.018

BBCH 17 7th true leaf unfolded

BBCH 18 8th true leaf unfolded

BBCH 19 9th true leaf unfolded

BBCH 51 First floral buds detectable (“pin-head square”)

BBCH 52 First floral buds visible (“match-head square”)

BBCH 55 Floral buds distinctly enlarged

BBCH 59 Petals visible: floral buds still closed

BBCH 60 First flowers opened (sporadically within the population)

BBCH 61 Beginning of flowering (“Early bloom”): 5–6 blooms / 7.5 meter of row

BBCH 65 Full flowering: (“Mid bloom”): 11 and more blooms / 7.5 meter of row

Peanuts

Mueth and Foster (2012 MSL-0024197) studied residues in peanuts at 13 trial sites in the USA. Treatments included one pre-plant (10–15 days before planting) or one pre-emergence application and a post-emergence (about 40 days after planting but prior to flowering) applications or a single post-emergent application. The formulation used was a CS (microencapsulated, 359 g ai/L) formulation of acetochlor. The tank mix included a non-ionic surfactant (0.5% v/v) and in the case of the post-emergence applications 2 kg ammonium sulphate/100 L spray solution. One composite sample was collected from each untreated control plot and two composite samples were collected from each of the treated plots. The residues were quantified by LC-MS/MS, method AG-ME-1467. For nutmeat the LOD and LOQs were 0.003 and 0.009 mg/kg for EMA and 0.003 and 0.009 mg/kg for HEMA. For hay the LOD and LOQs were 0.001 and 0.003 mg/kg for EMA and 0.001 and 0.003 mg/kg for HEMA.

Table 85 Residues in nutmeat following application of a micro-encapsulated (CS) acetochlor formulation to peanuts Mueth and Foster (2012 MSL-0024197). HEMA and EMA residues are expressed in acetochlor equivalents. Replicate samples.

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
PEANUTS		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Suffolk, Virginia,	2	1.70 PP	Bare soil	100 (116)	Nutmeat	0.023	0.061	
USA 2011 Champs		1.70 PO	BBCH 55			0.024	0.054	
					mean	0.023	0.058	0.081
	2	1.71 PE	BBCH 00	100 (116)	Nutmeat	0.028	0.063	
		1.71 PO	BBCH 55			0.029	0.066	
					mean	0.028	0.064	0.092
	1	4.43 PO	BBCH 55	100 (116)	Nutmeat	0.027	0.064	
						0.025	0.066	
					mean	0.026	0.065	0.091
Hertford, North Carolina,	–			–	Nutmeat	c< 0.009	c0.011	
USA 2011 Champs						c< 0.009	c0.012	
					mean	c< 0.009	c0.012	c< 0.021
Nutmeat 0.18–0.25 kg	2	1.67 PP	Pre-plant	98 (104)	Nutmeat	0.011	0.019	
		1.71 PO	BBCH 59 1st			0.010	0.025	
			bloom		mean	0.010	0.022	0.032
	2	1.66 PE		98 (104)	Nutmeat	0.015	0.026	
		1.65 PO	BBCH 59 1st			0.016	0.030	
			bloom		mean	0.016	0.028	0.043
	1	3.28 PO	BBCH 59 1st	98 (104)	Nutmeat	0.015	0.035	
						0.016	0.036	
			bloom		mean	0.016	0.036	0.051
Seven Springs, North	2	1.68 PP	BBCH 00	126 (133–134)	Nutmeat	0.014	0.032	
Carolina, USA 2011		1.64 PO	BBCH 51			0.014	0.030	
Champs [a]			Flower buds visible		mean	0.014	0.031	0.045
	2	1.67PE	BBCH 00	126 (133–134)	Nutmeat	0.010	0.026	
		1.63 PP	BBCH 51			0.011	0.024	
			Flower buds visible		mean	0.011	0.025	0.035

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
PEANUTS		kg ai/ha	at application			HEMA	EMA	(mg/kg)
	1	3.37 PO	BBCH 51	126 (133)	Nutmeat	0.016	0.034	
			Flower buds visible			0.015	0.030	
					mean	0.015	0.032	0.047
Seven Springs, North	2	1.68 PP	BBCH 00	128 (134)	Nutmeat	0.018	0.030	
Carolina, USA 2011 Perry [a]		1.69 PO	BBCH 55			0.019	0.036	
			Flower buds visible		mean	0.018	0.033	0.051
	2	1.71 PE	BBCH 00	128 (134)	Nutmeat	0.012	0.023	
		1.69 PO	BBCH 55			0.011	0.021	
			Flower buds visible		mean	0.011	0.022	0.033
	1	3.38 PO	BBCH 55	128 (134)	Nutmeat	0.012	0.024	
			Flower buds visible			0.013	0.024	
					mean	0.013	0.024	0.037
Blackville, South Carolina,	2	1.66 PP	Pre-plant	113 (125)	Nutmeat	0.016	0.029	
USA 2011 Gregory		1.70 PO	BBCH 18			0.018	0.026	
					mean	0.017	0.028	0.044
	2	1.69 PE	BBCH 00	113 (125)	Nutmeat	0.032	0.042	
		1.66 PO	BBCH 18			0.025	0.036	
					mean	0.029	0.039	0.067
	1	3.37 PO	BBCH 18	113 (125)	Nutmeat	0.017	0.028	
						0.019	0.027	
					mean	0.018	0.028	0.046
Abbeville, Georgia, USA	2	1.70 PP	Bare soil	103 (107)	Nutmeat	0.012	0.018	
2011 GA 07W		1.67 PO	BBCH 25			0.011	0.023	
					mean	0.011	0.021	0.032
Nutmeat 0.2–0.6 kg	2	1.66 PE	Bare soil	103 (107)	Nutmeat	0.014	0.019	
		1.70 PO	BBCH 25			0.015	0.024	
					mean	0.014	0.021	0.036
	1	3.35 PO	BBCH 25	103 (107)	Nutmeat	0.035	0.042	
						0.027	0.039	
					mean	0.031	0.041	0.072
Chula, Georgia, USA	2	1.67 PP	Bare soil	111 (123)	Nutmeat	< 0.009	0.010	
2011 GA06		1.68 PO	BBCH 25			< 0.009	0.011	
						< 0.009	0.012	
						0.010	0.012	
					mean	< 0.009	0.011	< 0.020
	2	1.69 PE	Bare soil	111 (123)	Nutmeat	0.011	0.023	
		1.68 PO	BBCH 25			0.012	0.024	
						< 0.009	0.014	
						< 0.009	0.014	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
PEANUTS		kg ai/ha	at application			HEMA	EMA	(mg/kg)
					mean	< 0.010	0.019	< 0.029
	1	3.36 PO	BBCH 25	104 (109)	Nutmeat	0.010	0.016	
						0.009	0.019	
						0.021	0.023	
						0.018	0.026	
					mean	0.014	0.021	0.036
				111 (123)	Nutmeat	0.011	0.023	
						0.009	0.016	
					mean	0.010	0.020	0.030
				118 (127)	Nutmeat	0.011	0.020	
						0.011	0.017	
					mean	0.011	0.018	0.029
				125 (132)	Nutmeat	0.012	0.018	
						0.012	0.018	
					mean	0.012	0.018	0.030
				132 (138)	Nutmeat	0.016	0.021	
						0.014	0.022	
					mean	0.015	0.021	0.036
Lenox, Georgia, USA	2	1.67 PP	Bare soil	106 (112)	Nutmeat	0.012	0.019	
2011 06-GA		1.68 PO	BBCH 25			0.010	0.025	
					mean	0.011	0.022	0.033
	2	1.68 PE	Bare soil	106 (112)	Nutmeat	0.015	0.021	
		1.66 PO	BBCH 25			0.015	0.019	
					mean	0.015	0.020	0.035
	1	3.32 PO	BBCH 25	106 (112)	Nutmeat	0.011	0.017	
						0.014	0.019	
					mean	0.013	0.018	0.031
Newberry, Florida,	2	1.69 PP	Bare ground	111 (119)	Nutmeat	0.010	0.020	
USA 2011 GA 06		1.64 PO	BBCH 25			0.009	0.023	
					mean	0.009	0.021	0.031
	2	1.67 PE	Bare ground	111 (119)	Nutmeat	0.014	0.034	
		1.67 PO	BBCH 25			0.014	0.029	
					mean	0.014	0.032	0.045
	1	3.32 PO	BBCH 25	111 (119)	Nutmeat	0.029	0.057	
						0.033	0.066	
					mean	0.031	0.061	0.092
Charlotte, Texas, USA	2	1.66 PP	Pre-plant	98 (108)	Nutmeat	< 0.009	0.014	
2011 Georgia 09		1.68 PO	BBCH 55			< 0.009	0.016	
					mean	< 0.009	0.015	< 0.024
	2	1.69 PE	Pre-plant	98 (108)	Nutmeat	0.012	0.018	
		1.69 PO	BBCH 55			0.012	0.024	
					mean	0.012	0.021	0.033
	1	3.37 PO	BBCH 55	98 (108)	Nutmeat	0.010	0.021	
						< 0.009	0.021	
						0.010	0.017	
						0.016	0.036	
					mean	< 0.011	0.024	< 0.035

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
PEANUTS		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Hinton, Oklahoma, USA	2	1.73 PP	Pre-plant	98 (102)	Nutmeat	0.015	0.034	
2011 Tamnut OL06		1.69 PO	BBCH 59			0.011	0.024	
					mean	0.013	0.029	0.042
	2	1.70 PE	Pre-plant	98 (102)	Nutmeat	0.012	0.023	
		1.70 PO	BBCH 59			0.015	0.030	
					mean	0.014	0.027	0.040
	1	3.38 PO	BBCH 59	91–98	Nutmeat	0.012	0.031	
						0.012	0.023	
					mean	0.012	0.027	0.039
				98 (102)	Nutmeat	0.013	0.027	
						0.014	0.030	
					mean	0.014	0.029	0.042
				105 (109)	Nutmeat	0.010	0.023	
						0.013	0.028	
					mean	0.011	0.025	0.036
				115 (118)	Nutmeat	0.010	0.027	
						0.015	0.034	
					mean	0.012	0.030	0.043
				119 (126)	Nutmeat	0.015	0.031	
						0.017	0.027	
					mean	0.016	0.029	0.045
Dill City, Oklahoma,	2	1.67 PP	Pre-plant	99 (104)	Nutmeat	0.014	0.025	
USA Tamnut OL06		1.67 PO	BBCH 59			0.014	0.030	
					mean	0.014	0.028	0.041
	2	1.70 PE	Pre-emergence	99 (104)	Nutmeat	0.015	0.031	
		1.66 PO	BBCH 59			0.016	0.025	
					mean	0.016	0.028	0.044
	1	3.35 PO	BBCH 59	99 (104)	Nutmeat	0.016	0.024	
						0.018	0.030	
					mean	0.017	0.027	0.044
Levelland, Texas, USA	2	1.68 PP	Not applicable	99 (104)	Nutmeat	0.020	0.051	
Tamnut OL06		1.69 PO	Pre-bloom			0.018	0.050	
					mean	0.019	0.050	0.069
Nutmeat 0.3–0.9 kg	2	1.67 PE	Pre-emergence	99 (104)	Nutmeat	0.017	0.046	
		1.69 PO	Pre-bloom			0.016	0.043	
					mean	0.017	0.045	0.061
	1	3.35 PO	Pre-bloom	99 (104)	Nutmeat	0.019	0.083	
						0.021	0.089	
						0.018	0.050	
						0.018	0.051	
					mean	0.019	0.068	0.087

DALA = harvest (digging) interval, figure in brackets is sampling interval (after drying in field)

PP = pre-planting

PE = pre-emergent

PO = post-emegent

[a] Seven Springs trials planting dates 20/5 and 12/5. Application dates: PP 6/5 and 29/4; PE 20/5 and 13/5; PO 20/6 and 15/6. Trials can be considered as a single site as the trial location and application timings are too similar.

BBCH 18 8th true leaf (pinnate) unfolded

BBCH 25 5th side shoot visible

BBCH 55 First individual flower buds visible

BBCH 59 First flower petals visible. Flower buds still closed

BBCH 61 Beginning of flowering

BBCH 62 First carpophore pegs visible

BBCH 63 Continuation of flowering

Animal feeds

Table 86 Residues in peanut fodder (hay) following application of a micro-encapsulated (CS) acetochlor formulation to peanuts (Mueth and Foster 2012 MSL-0024197). HEMA and EMA residues are expressed in acetochlor equivalents. Replicate samples.

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
PEANUT HAY		kg ai/ha	at application	[a]		HEMA	EMA	(mg/kg)
Suffolk, Virginia, USA	2	1.70	Bare soil	100 (116)	Hay	0.911	1.98	
2011 Champs		1.70	BBCH 55			1.00	1.94	
					mean	0.96	1.96	2.92
	2	1.71	BBCH 00	100 (116)	Hay	0.77	1.89	
		1.71	BBCH 55			1.1	2.16	
					mean	0.94	2.03	2.96
	1	4.43	BBCH 55	100 (116)	Hay	1.21	3.69	
						1.30	3.56	
					mean	1.26	3.63	4.88
Hertford, North Carolina,	2	1.67	Pre-plant	98 (104)	Hay	0.795	2.99	
USA 2011 Champs		1.71	BBCH 59 1st bloom			0.781	2.97	
					mean	0.79	2.98	3.77
	2	1.66	Pre-plant	98 (104)	Hay	0.567	1.74	
		1.65	BBCH 59 1st bloom			0.604	1.68	
					mean	0.59	1.71	2.3
	1	3.28	BBCH 59 1st bloom	98 (104)	Hay	0.413	1.6	
						0.466	1.84	
					mean	0.44	1.72	2.16
Seven Springs, North	2	1.68	BBCH 00	126 (133–134)	Hay	0.717	0.884	
Carolina, USA 2011		1.64	BBCH 51			0.772	0.853	
Champs			Flower buds visible		mean	0.74	0.87	1.61
	2	1.67	BBCH 00	126	Hay	0.43	0.558	
		1.63	BBCH 51	(133–134)		0.372	0.439	
			Flower buds visible		mean	0.4	0.5	0.9
	1	3.37	BBCH 51	126	Hay	0.71	1.16	
			Flower buds visible	(133–134)		0.761	1.19	
					mean	0.74	1.18	1.91
Seven Springs, North	2	1.68	BBCH 00 PRE	128	Hay	1.51	1.49	
Carolina, USA 2011 Perry		1.69	BBCH 55	(134–135)		1.21	1.07	
			Flower buds visible		mean	1.36	1.28	2.64
	2	1.71	BBCH 00 PRE	128	Hay	0.817	0.866	
		1.69	BBCH 55	(134–135)		0.824	0.795	
			Flower buds		mean	0.82	0.83	1.65

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
PEANUT HAY		kg ai/ha	at application	[a]		HEMA	EMA	(mg/kg)
			visible					
	1	3.38	BBCH 55	128	Hay	0.817	0.827	
			Flower buds visible	(134–135)		0.816	0.898	
					mean	0.82	0.86	1.68
Blackville, South	2	1.66	Pre-plant	113 (125)	Hay	0.474	0.69	
Carolina, USA 2011		1.70	BBCH 18			0.388	0.56	
Gregory					mean	0.43	0.63	1.06
	2	1.69	BBCH 00	113 (125)	Hay	0.416	0.626	
		1.66	BBCH 18			0.397	0.642	
					mean	0.41	0.63	1.04
	1	3.37	BBCH 18	113 (125)	Hay	0.381	1.02	
						0.387	1	
					mean	0.38	1.01	1.39
Abbeville, Georgia,	2	1.70	Bare soil	103 (107)	Hay	0.16	0.487	
USA 2011 GA 07W		1.67	BBCH 25			0.131	0.348	
					mean	0.15	0.42	0.56
	2	1.66	Bare soil	103 (107)	Hay	0.196	0.479	
		1.70	BBCH 25			0.196	0.492	
					mean	0.2	0.49	0.68
	1	3.35	BBCH 25	103 (107)	Hay	0.228	0.542	
						0.291	0.694	
					mean	0.26	0.62	0.88
Chula, Georgia, USA	2	1.67	Bare soil	111 (123)	Hay	0.287	0.32	
2011 GA06		1.68	BBCH 25			0.288	0.336	
						0.177	0.212	
						0.197	0.247	
					mean	0.24	0.28	0.52
	2	1.69	Bare soil	111 (123)	Hay	0.25	0.65	
		1.68	BBCH 25			0.228	0.664	
						0.144	0.321	
						0.151	0.372	
					mean	0.19	0.5	0.7
	1	3.36	BBCH 25	104 (109)	Hay	0.0918	0.43	
						0.0938	0.471	
						0.336	0.842	
						0.336	0.846	
					mean	0.21	0.65	0.86
				111 (123)	Hay	0.128	0.504	
						0.16	0.435	
					mean	0.14	0.47	0.61
				118 (127)	Hay	0.226	0.877	
						0.184	0.784	
					mean	0.21	0.83	1.04
				125 (132)	Hay	0.118	0.588	
						0.131	0.662	
						0.248	0.862	
						0.236	0.932	
					mean	0.18	0.76	0.94
				132 (138)	Hay	0.159	0.518	
						0.234	0.584	
					mean	0.2	0.55	0.75
Lenox, Georgia, USA	2	1.67	Bare soil	106 (112)	Hay	0.296	0.643	
2011 06-GA		1.68	BBCH 25			0.233	0.685	
					mean	0.26	0.66	0.93
	2	1.68	Bare soil	106 (112)	Hay	0.225	0.632	
		1.66	BBCH 25			0.239	0.667	
					mean	0.23	0.65	0.88
	1	3.32	BBCH 25	106 (112)	Hay	0.277	0.859	
						0.327	0.999	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
PEANUT HAY		kg ai/ha	at application	[a]		HEMA	EMA	(mg/kg)
					mean	0.3	0.93	1.23
Newberry, Florida,	2	1.69	Bare ground	111 (119)	Hay	0.24	0.692	
USA 2011 GA 06		1.64	BBCH 25			0.233	0.742	
					mean	0.24	0.72	0.95
	2	1.67	Bare ground	111 (119)	Hay	0.213	0.797	
		1.67	BBCH 25			0.215	0.793	
					mean	0.21	0.8	1.01
	1	3.32	BBCH 25	111 (119)	Hay	0.513	1.69	
						0.498	1.76	
					mean	0.51	1.73	2.23
Charlotte, Texas, USA	2	1.66	Pre-plant	98 (108)	Hay	0.0692	0.245	
2011 Georgia 09		1.68	BBCH 55			0.0716	0.245	
					mean	0.07	0.25	0.32
	2	1.69	Pre-plant	98 (108)	Hay	0.0746	0.36	
		1.69	BBCH 55			0.0748	0.37	
						0.117	0.626	
						0.117	0.633	
					mean	0.1	0.5	0.59
	1	3.37	BBCH 55	98 (108)	Hay	0.0886	0.415	
						0.0955	0.534	
					mean	0.09	0.47	0.57
Hinton, Oklahoma, USA	2	1.73	Pre-plant	98 (102)	Hay	0.522	1.07	
2011 Tamnut OL06		1.69	BBCH 59			0.529	1.1	
					mean	0.53	1.09	1.61
	2	1.70	Pre-plant	98 (102)	Hay	0.424	1.13	
		1.70	BBCH 59			0.344	0.943	
					mean	0.38	1.04	1.42
	1	3.38	BBCH 59	91 (98)	Hay	0.31	0.957	
						0.331	0.949	
					mean	0.32	0.95	1.27
				98 (102)	Hay	0.264	0.896	
						0.317	0.969	
					mean	0.29	0.93	1.22
				105 (109)	Hay	0.233	0.67	
						0.278	0.914	
					mean	0.26	0.79	1.05
				115 (118)	Hay	0.296	0.936	
						0.206	0.663	
					mean	0.25	0.8	1.05
				119 (126)	Hay	0.143	0.572	
						0.18	0.646	
					mean	0.16	0.61	0.77
Dill City, Oklahoma,	2	1.67	Pre-plant	99 (104)	Hay	0.239	0.673	
USA Tamnut OL06		1.67	BBCH 59			0.252	0.749	
					mean	0.25	0.71	0.96
	2	1.70	Pre-emergence	99 (104)	Hay	0.227	0.681	
		1.66	BBCH 59			0.287	0.849	
					mean	0.26	0.77	1.02
	1	3.35	BBCH 59	99 (104)	Hay	0.298	0.773	
						0.271	0.653	
					mean	0.28	0.71	1
Levelland, Texas,	2	1.68	Not applicable	98 (104)	Hay	0.254	1.03	
USA Tamnut OL06		1.69	Pre-bloom			0.234	1.06	
					mean	0.24	1.05	1.29
	2	1.67	Pre-emergence	98 (104)	Hay	0.28	1.3	
		1.69	Pre-bloom			0.336	1.45	
					mean	0.31	1.38	1.68
	1	3.35	Pre-bloom	98 (104)	Hay	0.307	1.57	
						0.275	1.35	
					mean	0.29	1.46	1.75

[a] Peanuts are typically dug (harvest date) and the field dried until they are ready for shelling and bagging (sampling date). Peanuts were sampled 3–16 days after harvesting. The pre-harvest intervals reported are the days between last application and the harvest date (digging date)

Table 87 Residues in forage following application of an EC, micro-encapsulated or GR acetochlor formulation to sweet corn (Crook and French 1996 RJ2078B). HEMA and EMA residues are expressed in acetochlor equivalents.

Location, year, variety	Form	Growth stage	Rate	DALA	Sample		Residue	(mg/kg)	Total
SWEET CORN FORAGE		at application	kg ai/ha			acetochlor	HEMA	EMA	(mg/kg)
North Rose, New York, USA 1995	EC	Pre-plant incorporated	3.36	88	Forage	< 0.01	0.14	0.3	0.44
Crusader 4399 LF)	CS	Pre-plant incorporated	3.36	88	Forage	< 0.01	0.13	0.25	0.38
	GR	Pre-plant incorporated	3.36	88	Forage	< 0.01	0.12	0.22	0.34
Boone, Iowa, USA 1995 Illini Xtra	EC	Pre-plant incorporated	3.36	76	Forage	< 0.01	0.08	0.14	0.22
Sweet	CS	Pre-plant incorporated	3.36	76	Forage	< 0.01	0.08	0.11	0.19
	GR	Pre-plant incorporated	3.36	76	Forage	< 0.01	0.05	0.09	0.14
Whitakers,	EC	Pre-emergence	3.36	80	Forage	< 0.01	0.12	0.17	0.29
North Carolina, USA	CS	Pre-emergence	3.36	80	Forage	< 0.01	0.09	0.18	0.27
1995 Silver Queen	GR	Pre-emergence	3.36	80	Forage	< 0.01	0.19	0.29	0.48
Champaign, Illinois,	EC	Pre-emergence	3.36	58	Forage	< 0.01	< 0.02	< 0.02	< 0.04
USA 1995 Early	CS	Pre-emergence	3.36	58	Forage	< 0.01	< 0.02	< 0.02	< 0.04
Choice	GR	Pre-emergence	3.36	58	Forage	< 0.01	< 0.02	< 0.02	< 0.04
Northwood, North Dakota, USA	EC	Pre-plant incorporated	3.36	103	Forage	< 0.01 [a]	< 0.02 [a]	0.04 [a]	
1995 Golden Bantam						< 0.01 [a]	0.03 [a]	0.09 [a]	
				Mean		< 0.01 [a]	< 0.025 [a]	0.065 [a]	< 0.09 [a]
Janesville, Wisconsin, USA	EC	Pre-plant incorporated	3.36	87	Forage	< 0.01 [a]	0.15 [a]	0.04 [a]	
1995 More						< 0.01 [a]	< 0.02 [a]	0.03 [a]	
				mean		< 0.01 [a]	< 0.085 [a]	0.04 [a]	< 0.12 [a]
Hebron, Maryland, USA 1995 Snow	EC	Pre-plant incorporated	3.36	81	Forage	< 0.01 [a,b]	0.4 [a,b]	0.43 [a,b]	
Belle						< 0.01 [a,b]	0.39 [a,b]	0.32 [a,b]	
				mean		< 0.01 [a,b]	0.395 [a,b]	0.375 [a,b]	0.77 [a,b]
Hamburg,	EC	Pre-emergence	3.36	72	Forage	< 0.01 [a]	0.03 [a]	0.1 [a]	
Pennsylvania, USA 1995						< 0.01 [a]	0.03 [a]	0.11 [a]	
				Mean		< 0.01 [a]	0.03 [a]	0.11 [a]	0.14 [a]
Loxley, Alabama,	EC	Pre-emergence	3.36	60	Forage	< 0.01	0.02	0.04	0.06
USA 1995 Silver				67		< 0.01	0.02	0.08	0.1
Queen				74		< 0.01	0.02	0.05	0.07
				81		< 0.01	0.02	0.05	0.07
				81		< 0.01	< 0.02	0.04	< 0.06
				88		< 0.01	< 0.02	0.03	< 0.05

Location, year, variety	Form	Growth stage	Rate	DALA	Sample		Residue	(mg/kg)	Total
SWEET CORN FORAGE		at application	kg ai/ha			acetochlor	HEMA	EMA	(mg/kg)
Monmouth, Illinois,	EC	Pre-emergence	3.36	41	Forage	< 0.01	0.02	0.03	0.05
USA 1995 Pioneer				48		< 0.01	0.02	0.05	0.07
3395 IR				55		< 0.01	< 0.02	< 0.02	< 0.04
				61		< 0.01	0.02	0.04	0.06
				61		< 0.01	0.03	0.05	0.08
				69		< 0.01	< 0.02	< 0.02	< 0.04
Visalia, California,	EC	Pre-emergence	3.36	83	Forage	< 0.01	0.2	0.77	0.97
USA 1995 Supersweet	EC	Pre-plant incorporated	3.36	83	Forage	< 0.01	< 0.02	< 0.02	< 0.04
Ephrata, Washington, USA 1995 Jubilee	EC	Pre-plant incorporated	3.36	91	Forage	< 0.01	< 0.02	0.02	< 0.04
	EC	Pre-emergence	3.36	91	Forage	< 0.01	< 0.02	0.04	< 0.06
Oviedo, Florida, USA 1995 Florida	EC	Pre-plant incorporated	3.36	65	Forage	< 0.01	0.05	0.19	0.24
Stay Sweet	EC	Pre-emergence	3.36	65	Forage	< 0.01	0.04	0.15	0.19
Mt. Vernon, Washington, USA	EC	Pre-plant incorporated	3.36	113	Forage	< 0.01	< 0.02	0.06	< 0.08
1995 Jubilee	EC	Pre-emergence	3.36	113	Forage	< 0.01	< 0.02	0.06	< 0.08

[a] Replicate samples

[b] Leaves only

Table 88 Residues in sweet corn stover following application of an EC, micro-encapsulated or GR acetochlor formulation to sweet corn as single pre-plant incorporated or pre-emergent applications at 3.4 kg ai/ha (Crook and French 1996 RJ2078B). HEMA and EMA residues are expressed in acetochlor equivalents.

Location, year, variety	Form	Growth stage	DALA	Sample		Residue	(mg/kg)	Total	
SWEET CORN FODDER		at application			acetochlor	HEMA	EMA	(mg/kg)	% moisture
North Rose, New York, USA 1995	EC	Pre-plant incorporated	130	Stover	< 0.01	0.03	0.06	0.09	65.4
Crusader 4399 LF	CS	Pre-plant incorporated	130	Stover	< 0.01	0.04	0.07	0.11	63.8
	GR	Pre-plant incorporated	130	Stover	< 0.01	0.05	0.08	0.13	60.6
Boone, Iowa, USA 1995 Illini Xtra	EC	Pre-plant incorporated	111	Stover	0.02	0.03	0.05	0.08	64.4
Sweet	CS	Pre-plant incorporated	111	Stover	< 0.01	0.03	0.04	0.07	63.6
	GR	Pre-plant incorporated	111	Stover	< 0.01	0.04	0.06	0.1	60.5
Whitakers, North	EC	Pre-emergence	121	Stover	< 0.01	0.25	0.17	0.42	24.1
Carolina, USA	CS	Pre-emergence	121	Stover	< 0.01	0.17	0.12	0.29	26.5
1995 Silver Queen	GR	Pre-emergence	121	Stover	< 0.01	0.25	0.15	0.4	32.2
Champaign, Illinois,	EC	Pre-emergence	99	Stover	< 0.01	< 0.02	0.02	< 0.04	40.3
USA 1995 Early	CS	Pre-emergence	99	Stover	< 0.01	< 0.02	0.02	< 0.04	35.0

Location, year, variety	Form	Growth stage	DALA	Sample		Residue	(mg/kg)	Total	
SWEET CORN FODDER		at application			acetochlor	HEMA	EMA	(mg/kg)	% moisture
Choice	GR	Pre-emergence	99	Stover	< 0.01	< 0.02	0.02	< 0.04	35.4
Northwood, North	EC	Pre-plant	143	Stover	< 0.01 [a]	< 0.02 [a]	< 0.02 [a]		60.2
Dakota, USA 1995		incorporated			< 0.01 [a]	< 0.02 [a]	< 0.02 [a]		62.3
Golden Bantam			mean		< 0.01 [a]	< 0.02 [a]	< 0.02 [a]	< 0.04 [a]	61.8
Janesville,	EC	Pre-plant	135	Stover	< 0.01 [a]	< 0.02 [a]	< 0.02 [a]		68.0
Wisconsin, USA		incorporated			< 0.01 [a]	< 0.02 [a]	< 0.02 [a]		65.7
1995 More			Mean		< 0.01 [a]	< 0.02 [a]	< 0.02 [a]	< 0.04 [a]	66.8
Hebron, Maryland,	EC	Pre-plant	123	Stover	< 0.01 [a]	0.02 [a]	0.02 [a]		45.3
USA 1995 Snow		incorporated			< 0.01 [a]	0.02 [a]	0.03 [a]		39.2
Belle			mean		< 0.01 [a]	0.02 [a]	0.02 [a]	0.04 [a]	42.2
Hamburg,	EC	Pre-emergence	111	Stover	< 0.01 [a]	0.03 [a]	0.04 [a]		58.9
Pennsylvania, USA					< 0.01 [a]	0.05 [a]	0.08 [a]		55.2
1995			mean		< 0.01 [a]	0.04 [a]	0.06 [a]	0.10 [a]	57.0
Loxley, Alabama,	EC	Pre-emergence	128	Stover	< 0.01 [a]	< 0.02 [a]	0.03 [a]		31.9
USA 1995 Silver					< 0.01 [a]	< 0.02 [a]	0.03 [a]		32.1
Queen			mean		< 0.01 [a]	< 0.02 [a]	0.03 [a]	< 0.05 [a]	32.0
Monmouth, Illinois,	EC	Pre-emergence	112	Stover	< 0.01 [a]	< 0.02 [a]	0.03 [a]		40.4
USA 1995 Pioneer					< 0.01 [a]	< 0.02 [a]	0.04 [a]		37.8
3395 IR			mean		< 0.01 [a]	< 0.02 [a]	0.04 [a]	< 0.06 [a]	39.2
Visalia, California,	EC	Pre-emergence	133	Stover	< 0.01	0.21	0.7	0.91	46.9
USA 1995 Supersweet	EC	Pre-plant incorporated	133	Stover	< 0.01	0.12	0.25	0.37	54.9
Ephrata, Washington, USA	EC	Pre-plant incorporated	126	Stover	< 0.01	0.02	0.11	0.13	73.7
1995 Jubilee	EC	Pre-emergence	126	Stover	< 0.01	0.02	0.1	0.12	70.8
Oviedo, Florida, USA 1995 Florida	EC	Pre-plant incorporated	101	Stover	< 0.01	0.03	0.1	0.13	62.6
Stay Sweet	EC	Pre-emergence	101	Stover	< 0.01	< 0.02	0.03	< 0.05	54.6
Mt. Vernon, Washington, USA	EC	Pre-plant incorporated	169	Stover	< 0.01	< 0.02	< 0.02	< 0.04	74.4
1995 Jubilee	EC	Pre-emergence	169	Stover	< 0.01	< 0.02	< 0.02	< 0.04	75.2

[a] Replicate samples

Table 89 Residues in maize forage following application of an EC or a CS acetochlor formulation (Maher 2007 MSL-20269). HEMA and EMA residues are expressed in acetochlor equivalents. Replicate samples

Location, year, variety	Form	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE FORAGE			kg ai/ha	at application			HEMA	EMA	(mg/kg)
Richland, Iowa, USA	EC	1	3.10	V8	59	Forage	0.071	1.170	
2006 NK N51-V9				68–81 cm			0.069	1.170	
						mean	0.070	1.170	1.240
					66	Forage	0.093	1.940	
							0.095	1.860	
						mean	0.094	1.900	1.994
					74	Forage	0.051	0.709	
							0.049	0.713	
						mean	0.050	0.711	0.761
					81	Forage	0.030	0.377	
							0.032	0.353	

Location, year, variety	Form	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE FORAGE			kg ai/ha	at application			HEMA	EMA	(mg/kg)
						mean	0.031	0.365	0.396
					87	Forage	0.014	0.156	
							0.014	0.157	
						mean	0.014	0.157	0.171
	CS	1	3.33	V8	66	Forage	0.166	0.876	
				68–81 cm			0.182	0.961	
						mean	0.174	0.919	1.093
Hedrick, Iowa, USA	EC	1	2.97	V7–V8	79	Forage	0.084	1.400	
2006 Pioneer 34A16				71–84 cm			0.077	1.280	
						mean	0.080	1.340	1.420
	CS	1	3.17	V7–V8	79	Forage	0.079	0.376	
				71–84 cm			0.085	0.402	
						mean	0.082	0.389	0.471
Richland, Iowa, USA	EC	1	2.96	V8	79	Forage	0.045	0.719	
2006 Middle Koop 2212				71–86 cm			0.044	0.709	
						mean	0.044	0.714	0.758
	CS	1	3.18	V8	79	Forage	0.040	0.179	
				71–86 cm			0.044	0.197	
						mean	0.042	0.188	0.230
Perry, Iowa, USA 2006	EC	1	2.96PO	V8	52	Forage	0.107	1.760	
Pioneer 36B10				69–86 cm			0.075	1.290	
						mean	0.091	1.525	1.616
	EC	2	1.47PE	V8	52	Forage	0.086	1.320	
			1.50PO	69–86 cm			0.081	1.250	
						mean	0.084	1.285	1.369
	EC	1	2.88PO	V6	64	Forage	0.034	0.131	
				46–51 cm			0.033	0.131	
						mean	0.034	0.131	0.165
	CS	1	3.27PO	V8	52	Forage	0.217	1.440	
				69–86 cm			0.204	1.410	
						mean	0.211	1.425	1.636
	CS	2	1.60PE	V8	52	Forage	0.053	0.263	
			1.61PO	69–86 cm			0.054	0.284	
						mean	0.053	0.274	0.327
	CS	1	3.23PO	V6	64	Forage	0.017	0.256	
				46–51 cm			0.019	0.256	
						mean	0.018	0.256	0.274
Bagley, Iowa, USA 2006	EC	1	2.96	V8	54	Forage	0.086	1.450	
Pioneer 33P65				66–89 cm			0.071	1.270	
						mean	0.078	1.360	1.438
	CS	1	3.31	V8	54	Forage	0.066	0.427	
				66–89 cm			0.064	0.390	
						mean	0.065	0.409	0.474
Carlyle, Illinois, USA	EC	1	2.89	V9	48	Forage	0.013	0.178	
2006 DKC61-45				66–86 cm			0.016	0.208	
						mean	0.015	0.193	0.208
					55	Forage	0.201	2.470	
							0.205	2.490	
						mean	0.203	2.480	2.683
					62	Forage	0.166	1.920	
							0.159	1.950	
						mean	0.163	1.935	2.098
					69	Forage	0.266	2.950	
							0.225	3.280	
						mean	0.246	3.115	3.361
					76	Forage	0.216	2.320	
							0.212	2.400	
						mean	0.214	2.360	2.574

Location, year, variety	Form	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE FORAGE			kg ai/ha	at application			HEMA	EMA	(mg/kg)
	CS	1	3.13	V9	55	Forage	0.230	1.160	
				66–86 cm			0.239	1.190	
						mean	0.235	1.175	1.410
Mason, Illinois, USA	EC	1	3.06	BBCH 18	55	Forage	0.061	1.370	
2006 Midland mg 606RR				66–91 cm			0.057	1.360	
						mean	0.059	1.365	1.424
	CS	1	3.32	BBCH 18	55	Forage	0.149	1.040	
				66–91 cm			0.168	0.998	
						mean	0.159	1.019	1.178
Wyoming, Illinois, USA	EC	1	2.95	V8	64	Forage	0.051	0.923	
2006 Burns 644 RWR				74–79 cm			0.053	0.889	
						mean	0.052	0.906	0.958
	CS	1	3.15	V8	64	Forage	0.096	0.495	
				74–79 cm			0.102	0.536	
						mean	0.099	0.516	0.614
Danville, Indiana, USA	EC	1	2.82	BBCH 18	61	Forage	0.027	0.380	
2006 Wyffels W5531				66–91 cm			0.025	0.386	
						mean	0.026	0.383	0.409
	CS	1	3.19	BBCH 18	61	Forage	0.083	0.509	
				66–91 cm			0.084	0.506	
						mean	0.083	0.508	0.591
Rockville, Indiana, USA	EC	1	2.82	BBCH 18	57	Forage	0.215	3.350	
2006 Pioneer 33NO8				66–86 cm			0.198	3.160	
						mean	0.207	3.255	3.462
	CS	1	3.33	BBCH 18	57	Forage	0.246	2.050	
				66–86 cm			0.221	1.920	
						mean	0.234	1.985	2.219
Paynesville, Minnesota,	EC	1	2.93	V8	67	Forage	0.012	1.410	
USA 2006 Dekalb				71–86 cm			0.011	1.420	
DKC47-10 RR2						mean	0.012	1.415	1.427
	CS	1	3.19	V8	67	Forage	0.004	1.040	
				71–86 cm			0.004	0.921	
						mean	0.004	0.981	0.984
Hawick, Minnesota, USA	EC	1	2.87	76 cm	67	Forage	0.009	0.964	
2006 Dekalb DKC47-10							0.009	1.040	
RR2						mean	0.009	1.002	1.011
	CS	1	3.22	76 cm	67	Forage	0.004	0.368	
							0.004	0.392	
						mean	0.004	0.380	0.384
LaPlata, Missouri, USA	EC	1	3.04	V8	68	Forage	0.072	0.620	
2006 Dekalb DKC61-42				71–79 cm			0.073	0.612	
						mean	0.072	0.616	0.688
	EC	2	1.43PE	V8	68	Forage	0.019	0.341	
			1.49PO	71–79 cm			0.019	0.333	
						mean	0.019	0.337	0.356
	EC	1	2.90	V6	75	Forage	0.005	0.028	
				46–51 cm			0.005	0.027	
						mean	0.005	0.028	0.033
	CS	1	3.26	V8	68	Forage	0.098	0.965	
				71–79 cm			0.093	0.944	
						mean	0.096	0.955	1.050
	CS	2	1.61PE	V8	68	Forage	0.024	0.256	
			1.61PO	71–79 cm			0.024	0.230	
						mean	0.024	0.243	0.267
	CS	1	3.19	V6	75	Forage	0.005	0.032	

Location, year, variety	Form	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE FORAGE			kg ai/ha	at application			HEMA	EMA	(mg/kg)
				46–51 cm			0.005	0.029	
						mean	0.005	0.031	0.036
Seven Springs, North	EC	1	2.96	BBCH 33	38	Forage	0.161	2.310	
Carolina, USA 2006				71–86 cm			0.148	2.380	
Garst 8377						mean	0.155	2.345	2.500
	CS	1	3.24	BBCH 33	38	Forage	0.293	0.020	
				71–86 cm			0.285	0.018	
						mean	0.289	0.019	0.308
York, Nebraska, USA	EC	1	2.94	BBCH 18	69	Forage	0.010	0.035	
2006 Pioneer 34N45				69–81 cm			0.010	0.035	
RR2/YGCB						mean	0.010	0.035	0.045
	CS	1	3.18	BBCH 18	69	Forage	0.011	0.079	
				69–81 cm			0.012	0.084	
						mean	0.012	0.081	0.093
Osceola, Nebraska, USA	EC	1	2.94	BBCH 18	68	Forage	0.051	0.605	
2006 N73-F7 RR/LL/CB				66–81 cm			0.045	0.551	
						mean	0.048	0.578	0.626
	CS	1	3.16	BBCH 18	68	Forage	0.115	0.709	
				66–81 cm			0.113	0.714	
						mean	0.114	0.712	0.826
Baptistown, New Jersey,	EC	1	3.00	V8	58	Forage	0.088	1.580	
USA TA5750/ 401169				61–91 cm			0.089	1.530	
						mean	0.089	1.555	1.644
	CS	1	3.26	V8	58	Forage	0.158	1.730	
				61–91 cm			0.158	1.750	
						mean	0.158	1.740	1.898
Washington, Ohio, USA	EC	1	2.97	V8–V9	50	Forage	0.177	3.930	
2006 SC 11RR06				71–84 cm			0.151	3.500	
						mean	0.164	3.715	3.879
	CS	1	3.14	V8–V9	50	Forage	0.188	1.360	
				71–84 cm			0.188	1.340	
						mean	0.188	1.350	1.538
New Holland, Ohio, USA	EC	1	3.00	V8	58	Forage	0.080	1.630	
2006 Crows 515Z R				71–79 cm			0.072	1.440	
						mean	0.076	1.535	1.611
	CS	1	3.17	V8	58	Forage	0.101	0.701	
				71–79 cm			0.093	0.660	
						mean	0.097	0.681	0.778
Dill City, Oklahoma, USA	EC	1	2.90	V8–V9	47	Forage	0.136	1.590	
2006 DK C48-53				74–81 cm			0.124	1.630	
						mean	0.130	1.610	1.740
	CS	1	3.19	V8–V9	47	Forage	0.104	0.594	
				74–81 cm			0.102	0.551	
						mean	0.103	0.573	0.676
Delavan, Wisconsin, USA	EC	1	2.89	V8	66	Forage	0.017	0.224	
2006 Dekalb DKC51-39				74–79 cm			0.017	0.231	
						mean	0.017	0.228	0.244
	CS	1	3.09	V8	66	Forage	0.047	0.290	
				74–79 cm			0.047	0.286	
						mean	0.047	0.288	0.335

Table 90 Residues in maize and sweet corn forage (two trials only) following application of an acetochlor EC formulation (Oppenhuizen and Wilson 1989 MSL-6843). HEMA and EMA residues are expressed in acetochlor equivalents. Results are for samples analysed in duplicate

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE FORAGE		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Gretna, NE 1985 DK Xl73	1	1.7	Pre-emergent	49	Forage	< 0.02	0.03	
						< 0.02	0.04	
					mean	< 0.02	0.04	< 0.06
	1	3.4	Pre-emergent	49	Forage	0.03	0.11	
						0.03	0.09	
					Mean	0.03	0.10	0.13
	1	6.7	Pre-emergent	49	Forage	0.06	0.17	
						0.06	0.18	
					Mean	0.06	0.18	0.24
Isleton, CA 1985 Funks	1	1.7	Pre-emergent	55	Forage	< 0.02	< 0.02	
4438						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	55	Forage	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	55	Forage	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
Jerome, ID 1985 Cenex	1	1.7	Pre-emergent	59	Forage	0.02	0.07	
98d						0.02	0.07	
					mean	0.02	0.07	0.09
	1	3.4	Pre-emergent	59	Forage	0.03	0.14	
						0.03	0.14	
					mean	0.03	0.14	0.17
	1	6.7	Pre-emergent	59	Forage	0.06	0.24	
						0.06	0.24	
					mean	0.06	0.24	0.30
Otterbein, IN 1985 Sweet	1	1.7	Pre-emergent	56	Forage	< 0.02	< 0.02	
Corn						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	56	Forage	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	56	Forage	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
Princeton, IA 1985	1	1.7	Pre-emergent	60	Forage	< 0.02	< 0.02	
Pioneer 33/78						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	60	Forage	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	60	Forage	< 0.02	0.02	
						< 0.02	0.03	
					mean	< 0.02	0.03	< 0.05
Redfield, IA 1985 Lynks	1	1.7	Pre-emergent	57	Forage	< 0.02	< 0.02	
4330						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	57	Forage	< 0.02	< 0.02	
						< 0.02	0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	57	Forage	< 0.02	0.02	
						< 0.02	0.03	
					mean	< 0.02	0.03	< 0.05
Reeds Corner, NY 1985	1	1.7	Pre-emergent	62	Forage	< 0.02	0.02	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
Cargil 815						< 0.02	0.02	
					mean	< 0.02	0.02	< 0.04
	1	3.4	Pre-emergent	62	Forage	0.02	0.05	
						0.02	0.05	
					mean	0.02	0.05	0.07
	1	6.7	Pre-emergent	62	Forage	0.03	0.10	
						0.03	0.10	
					mean	0.03	0.10	0.13
Reevesville, SC 1985	1	1.7	Pre-emergent	56	Forage	< 0.02	0.02	
PN3320						< 0.02	0.02	
					mean	< 0.02	0.02	< 0.04
	1	3.4	Pre-emergent	56	Forage	0.05	0.13	
						0.06	0.14	
					mean	0.06	0.14	0.20
	1	6.7	Pre-emergent	56	Forage	0.15	0.45	
						0.15	0.44	
					mean	0.15	0.45	0.60
Trenton, TN 1985 O's	1	1.7	Pre-emergent	61	Forage	0.02	0.04	
Gold 3344						0.02	0.04	
					mean	0.02	0.04	0.06
	1	3.4	Pre-emergent	61	Forage	0.03	0.05	
						0.03	0.04	
					mean	0.03	0.05	0.08
	1	6.7	Pre-emergent	61	Forage	0.05	0.09	
						0.05	0.07	
					mean	0.05	0.08	0.13
Waseca, MN 1985 Sweet	1	1.7	Pre-emergent	64	Forage	< 0.02	< 0.02	
corn Jubilee						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	64	Forage	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	64	Forage	< 0.02	0.03	
						< 0.02	0.03	
					mean	< 0.02	0.03	< 0.05
Waukee, IA 1985 Funks	1	1.7	Pre-emergent	58	Forage	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	58	Forage	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	58	Forage	< 0.02	0.03	
						< 0.02	0.03	
					mean	< 0.02	0.03	< 0.05
Williamston, MI 1985	1	1.7	Pre-emergent	50	Forage	0.02	0.05	
DK2120						0.02	0.05	
					mean	0.02	0.05	0.07
	1	3.4	Pre-emergent	50	Forage	0.02	0.08	
						0.02	0.09	
					mean	0.02	0.09	0.11
	1	6.7	Pre-emergent	50	Forage	0.10	0.42	
						0.09	0.35	
					mean	0.10	0.39	0.49

Results are corrected for the average analytical recovery of the method

Table 91 Residues of 5-hydroxy *sec*-oxanilic acid (68) in maize forage following application of an acetochlor EC formulation as a single pre-emergent application (Ralph *et al.* 1992 RJ1337B) Results are for samples analysed in duplicate

Country/ location MAIZE	Crop growth stage	Sample	DALA	Analysis 1	Analysis 2	mean
Visalia, California, USA 1991	Pre-emergence	Forage	59	0.04	0.04	0.04
Champaign Illinois USA 1991	Pre-emergence	Forage	68	0.01	0.01	0.01
Ephrata Washington, USA 1991	Pre-emergence	Forage	82	0.02	0.02	0.02
Paynesville Minnesota USA 1991	Pre-emergence	Forage	71	< 0.01	< 0.01	< 0.01
York Nebraska, USA 1991	Pre-emergence	Forage	63	0.12	0.11	0.12
Iconium Iowa USA 1991	Pre-emergence	Forage	68	< 0.01	< 0.01	< 0.01
Berthoud Colorado USA 1991 [a]	Pre-emergence	Forage	120	0.06 c0.01	0.05 c0.01	0.06
Noblesville Indiana USA 1991	Pre-emergence	Forage	69	0.08	0.08	0.08
Sudlerville Maryland USA 1991	Pre-emergence	Forage	91	0.02	0.01	0.02
Fabius New York USA 1991	Pre-emergence	Forage	67	0.06	0.05	0.06
Fabius New York USA 1991	Pre-emergence	Forage	70	0.04	0.04	0.04
Germansville Pennsylvania USA 1991	Pre-emergence	Forage	66	< 0.01	< 0.01	< 0.01
Pulaski Pennsylvania USA 1991	Pre-emergence	Forage	67	–	–	–

[a] Application rate 4.5 kg ai/ha

Table 92 Residues in maize forage following application of a CS acetochlor formulation Lau (1992 MSL-11794). HEMA and EMA residues are expressed in acetochlor equivalents. Results are for samples analysed in duplicate.

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE FORAGE		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Colo, Iowa, USA	1	3.4	Pre-emergent		Forage	< 0.01	0.02	
1990 DK535						< 0.01	0.02	
						< 0.01	0.02	
					mean	< 0.01	0.02	< 0.03
	1	3.4	Pre-plant	73	Forage	0.02	0.03	
						0.02	0.03	
						0.02	0.03	
					mean	0.02	0.03	0.05
Conklin, Michigan, USA	1	3.4	Pre-emergent	62	Forage	0.01	0.04	
1990 Pioneer 3751						0.02	0.04	
					mean	0.01	0.04	0.05
Danville, Iowa, USA	1	3.4	Pre-emergent	89	Forage	< 0.01	0.01	
1990 Dockendorf 7670						< 0.01	0.01	
					mean	< 0.01	0.01	< 0.02
Delavan, Wisconsin,	1	3.4	Pre-emergent	78	Forage	< 0.01	0.01	
USA 1990 RK627						< 0.01	0.01	
					mean	< 0.01	0.01	< 0.02
Elwood, Illinois USA	1	3.4	Pre-emergent	63	Forage	0.01	0.03	
1990 Pioneer 3615						< 0.01	0.03	
					mean	< 0.01	0.03	< 0.04
Geneseo, Illinois USA	1	3.4	Pre-emergent	88	Forage	< 0.01	< 0.01	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE FORAGE		kg ai/ha	at application			HEMA	EMA	(mg/kg)
USA 1990 Pioneer 3615						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.01
	1	3.4	Pre-plant	88	Forage	< 0.01	0.01	
						< 0.01	0.01	
					mean	< 0.01	0.01	< 0.02
Hollandale, Minnesota,	1	3.4	Pre-emergent	78	Forage	< 0.01	0.02	
USA 1990 Pioneer 3751						< 0.01	0.02	
					mean	< 0.01	0.02	< 0.03
	1	3.4	Pre-plant	78	Forage	< 0.01	0.03	
						< 0.01	0.04	
					mean	< 0.01	0.04	< 0.05
Elk City Kansas, USA	1	3.4	Pre-emergent	64	Forage	< 0.01	0.02	
1990 Cargil 6127						< 0.01	0.02	
					mean	< 0.01	0.02	< 0.03
Leonard, Missouri, USA	1	3.4	Pre-emergent	78	Forage	< 0.01	0.01	
1990 McAllister						< 0.01	0.01	
SX8611RFR					mean	< 0.01	0.01	< 0.02
	1	3.4	Pre-plant	78	Forage	< 0.010	0.02	
						0.01	0.02	
					mean	< 0.01	0.02	< 0.03
New Holland Ohio, USA	1	3.4	Pre-emergent	55	Forage	< 0.010	0.06	
1990 Pioneer 3343						0.01	0.06	
					mean	< 0.01	0.06	< 0.08
	1	3.4	Pre-plant	57	Forage	0.01	0.08	
						0.02	0.09	
					mean	0.02	0.09	0.10
Noblesville Indiana,	1	3.4	Pre-emergent	74	Forage	0.02	0.02	
USA 1990 Pioneer 3744						0.01	0.03	
					mean	0.02	0.03	0.05
Sioux Falls South Dakota	1	3.4	Pre-emergent	87	Forage	0.01	0.03	
USA 1990 Moews 3140						0.02	0.03	
					mean	0.01	0.03	0.04
Uvalde Texas, USA	1	3.4	Pre-emergent	47	Forage	0.01	0.04	
1990 Pioneer 3192						0.01	0.04	
					mean	0.01	0.04	0.05
York Nebraska, USA	1	3.4	Pre-emergent	91	Forage	0.01	0.01	
1990 Pioneer 3379						0.01	0.02	
					mean	0.01	0.01	0.03
	1	3.4	Pre-plant	91	Forage	0.02	0.03	
						0.02	0.03	
					mean	0.02	0.03	0.05

Values have been corrected for analytical method recoveries and expressed as acetochlor equivalents for either EMA (ethylmethylaniline producing) or HEMA (hydroxyethylrnethylaniline producing) residues

Table 93 Residues of 5-hydroxy *sec*-oxanilic acid (68) in maize silage following application of an acetochlor EC formulation as a single pre-emergent application (Ralph *et al.* 1992 RJ1337B) Results are for samples analysed in duplicate.

Country/ location MAIZE SILAGE	Crop growth stage	Sample	DALA (days)	Analysis 1	Analysis 2	mean
Whitakers North Carolina USA 1991	Pre-emergence	Silage	96	0.37	0.41	0.39
Visalia, California, USA 1991	Pre-emergence	Silage	82	0.01	0.01	0.01
Champaign Illinois USA 1991	Pre-emergence	Silage	104	0.05	0.05	0.05
Ephrata Washington, USA 1991	Pre-emergence	Silage	118	0.04	0.04	0.04

Country/ location MAIZE SILAGE	Crop growth stage	Sample	DALA (days)	Analysis 1	Analysis 2	mean
Paynesville Minnesota USA 1991	Pre-emergence	Silage	110	< 0.01	< 0.01	< 0.01
York Nebraska, USA 1991	Pre-emergence	Silage	105	0.18	0.17	0.18
Iconium Iowa USA 1991	Pre-emergence	Silage	98	< 0.01	< 0.01	< 0.01
Berthoud Colorado USA 1991 [a]	Pre-emergence	Silage	132	0.04 c0.02	0.04	0.04
Noblesville Indiana USA 1991	Pre-emergence	Silage	103	0.11	0.11	0.11
Sudlerville Maryland USA 1991	Pre-emergence	Silage	103	0.02	0.01	0.02
Fabius New York USA 1991	Pre-emergence	Silage	116	0.04	0.04	0.04
Fabius New York USA 1991	Pre-emergence	Silage	110	0.05	0.05	0.05
Germansville Pennsylvania USA 1991	Pre-emergence	Silage	109	< 0.01	< 0.01	< 0.01
Pulaski Pennsylvania USA 1991	Pre-emergence	Silage	80	0.05	0.05	0.05

[a] Application rate 4.5 kg ai/ha

Table 94 Residues in maize silage following pre-emergent or pre-plant application of a CS acetochlor formulation Lau (1992 MSL-11794). HEMA and EMA residues are expressed in acetochlor equivalents. Results are for samples analysed in duplicate.

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE SILAGE		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Colo, Iowa, USA	1	3.4	Pre-emergent	89	Silage	< 0.01	0.02	
1990 DK535						< 0.01	0.02	
					mean	< 0.01	0.02	< 0.03
	1	3.4	Pre-plant	101	Silage	0.02	0.02	
						0.02	0.02	
					mean	0.02	0.02	0.03
Conklin, Michigan, USA	1	3.4	Pre-emergent	102	Silage	0.02	0.03	
1990 Pioneer 3751						0.02	0.03	
					mean	0.02	0.03	0.05
Danville, Iowa, USA	1	3.4	Pre-emergent	131	Silage	< 0.01	< 0.01	
1990 Dockendorf 7670						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
Delavan, Wisconsin,	1	3.4	Pre-emergent	129	Silage	< 0.01	0.01	
USA 1990 RK627						< 0.01	0.01	
					mean	< 0.01	0.01	< 0.02
Elwood, Illinois USA	1	3.4	Pre-emergent	103	Silage	0.01	0.01	
1990 Pioneer 3615						0.01	0.01	
					mean	0.01	0.01	0.02
Geneseo, Illinois USA	1	3.4	Pre-emergent	117	Silage	< 0.01	< 0.01	
USA 1990 Pioneer 3615						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
	1	3.4	Pre-plant	117	Silage	< 0.01	0.01	
						< 0.01	0.01	
					mean	< 0.01	0.01	< 0.02
Hollandale, Minnesota,	1	3.4	Pre-emergent	119	Silage	< 0.01	0.01	
USA 1990 Pioneer 3751						< 0.01	0.01	
					mean	< 0.01	0.01	< 0.02
	1	3.4	Pre-plant	119	Silage	< 0.01	0.01	
						< 0.01	0.01	
					mean	< 0.01	0.01	< 0.02
Elk City Kansas, USA	1	3.4	Pre-emergent	86	Silage	0.03	0.05	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE SILAGE		kg ai/ha	at application			HEMA	EMA	(mg/kg)
1990 Cargil 6127						0.03	0.05	
					mean	0.03	0.05	0.08
Leonard, Missouri, USA	1	3.4	Pre-emergent	104	Silage	< 0.01	0.01	
1990 McAllister						< 0.01	0.01	
SX8611RFR					mean	< 0.01	0.01	< 0.02
	1	3.4	Pre-plant	104	Silage	0.02	0.01	
						0.02	0.02	
					mean	0.02	0.01	0.03
New Holland Ohio, USA	1	3.4	Pre-emergent	144	Silage	< 0.01	< 0.01	
1990 Pioneer 3343						< 0.01	< 0.01	
					mean	< 0.01	< 0.01	< 0.02
	1	3.4	Pre-plant	146	Silage	< 0.01	0.01	
						< 0.01	0.01	
					mean	< 0.01	0.01	< 0.02
Noblesville Indiana,	1	3.4	Pre-emergent	112	Silage	0.02	0.03	
USA 1990 Pioneer 3744						0.02	0.04	
					mean	0.02	0.03	0.05
Sioux Falls South Dakota	1	3.4	Pre-emergent	119	Silage	0.02	0.02	
USA 1990 Moews 3140						0.02	0.03	
					mean	0.02	0.02	0.05
Uvalde Texas, USA	1	3.4	Pre-emergent	90	Silage	0.01	0.02	
1990 Pioneer 3192						0.01	0.02	
					mean	0.01	0.02	0.03
York Nebraska, USA	1	3.4	Pre-emergent	128	Silage	0.01	0.02	
1990 Pioneer 3379						0.02	0.02	
					mean	0.01	0.02	0.03
	1	3.4	Pre-plant	128	Silage	0.02	0.03	
						0.02	0.03	
					mean	0.02	0.03	0.05

Values have been corrected for analytical method recoveries and expressed as acetochlor equivalents for either EMA (ethylmethylaniline producing) or HEMA (hydroxyethylrnethylaniline producing) residues

Table 95 Residues in maize and sweet corn fodder (two trials only) following pre-emergent application of an acetochlor EC formulation (Oppenhuizen and Wilson 1989 MSL-6843). HEMA and EMA residues are expressed in acetochlor equivalents. Results are for samples analysed in duplicate.

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE FODDER		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Gretna, NE 1985 DK Xl73	1	1.7	Pre-emergent	147	Fodder	< 0.02	0.02	
						< 0.02	0.02	
					mean	< 0.02	0.02	< 0.04
	1	3.4	Pre-emergent	147	Fodder	0.03	0.04	
						0.02	0.04	
					Mean	0.03	0.04	0.07
	1	6.7	Pre-emergent	147	Fodder	0.05	0.08	
						0.05	0.07	
					Mean	0.05	0.08	0.13
Isleton, CA 1985 Funks	1	1.7	Pre-emergent	164	Fodder	< 0.02	< 0.02	
4438						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	164	Fodder	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	164	Fodder	< 0.02	< 0.02	
						< 0.02	0.02	
					mean	< 0.02	< 0.02	< 0.04

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
Otterbein, IN 1985 Sweet	1	1.7	Pre-emergent	86	Fodder	< 0.02	0.03	
Corn						< 0.02	0.02	
					mean	< 0.02	0.03	< 0.05
	1	3.4	Pre-emergent	86	Fodder	< 0.02	0.03	
						< 0.02	0.02	
					mean	< 0.02	0.03	< 0.05
	1	6.7	Pre-emergent	86	Fodder	< 0.02	0.02	
						< 0.02	0.02	
					mean	< 0.02	< 0.02	< 0.04
Princeton, IA 1985	1	1.7	Pre-emergent	151	Fodder	< 0.02	< 0.02	
Pioneer 33/78						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	151	Fodder	< 0.02	0.03	
						< 0.02	0.03	
					mean	< 0.02	0.03	< 0.05
	1	6.7	Pre-emergent	151	Fodder	< 0.02	0.02	
						< 0.02	0.03	
					mean	< 0.02	0.03	< 0.05
Redfield, IA 1985 Lynks	1	1.7	Pre-emergent	149	Fodder	< 0.02	< 0.02	
4330						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	149	Fodder	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	149	Fodder	< 0.02	0.03	
						< 0.02	0.03	
					mean	< 0.02	0.03	< 0.05
Reeds Corner, NY 1985	1	1.7	Pre-emergent	167	Fodder	< 0.02	0.02	
Cargil 815						< 0.02	0.07	
					mean	< 0.02	0.05	< 0.07
	1	3.4	Pre-emergent	167	Fodder	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	167	Fodder	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
Reevesville, SC 1985	1	1.7	Pre-emergent	138	Fodder	< 0.02	< 0.02	
PN3320						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	138	Fodder	0.03	0.04	
						0.03	0.04	
					mean	0.03	0.04	0.07
	1	6.7	Pre-emergent	138	Fodder	0.05	0.06	
						0.04	0.05	
					mean	0.05	0.06	0.11
Trenton, TN 1985 O's	1	1.7	Pre-emergent	157	Fodder	< 0.02	0.02	
Gold 3344						< 0.02	0.02	
					mean	< 0.02	0.02	< 0.04
	1	3.4	Pre-emergent	157	Fodder	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	157	Fodder	0.04	0.05	
						0.04	0.05	
					mean	0.04	0.05	0.09
Waseca, MN 1985 Sweet	1	1.7	Pre-emergent	84	Fodder	< 0.02	< 0.02	
corn Jubilee						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	84	Fodder	< 0.02	0.02	
						< 0.02	0.03	
					mean	< 0.02	0.03	< 0.05
	1	6.7	Pre-emergent	84	Fodder	< 0.02	0.04	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
						< 0.02	0.07	
					mean	< 0.02	0.06	< 0.08
Waukee, IA 1985 Funks	1	1.7	Pre-emergent	168	Fodder	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	168	Fodder	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	6.7	Pre-emergent	168	Fodder	< 0.02	< 0.02	
						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
Williamston, MI 1985	1	1.7	Pre-emergent	163	Fodder	< 0.02	< 0.02	
DK2120						< 0.02	< 0.02	
					mean	< 0.02	< 0.02	< 0.04
	1	3.4	Pre-emergent	163	Fodder	< 0.02	0.03	
						< 0.02	0.02	
					mean	< 0.02	0.03	< 0.05
	1	6.7	Pre-emergent	163	Fodder	< 0.02	0.06	
						< 0.02	0.06	
					mean	< 0.02	0.06	< 0.08

Results are corrected for the average analytical recovery of the method

Table 96 Residues of 5-hydroxy *sec*-oxanilic acid (68) in maize fodder following a single pre-emergent application of an acetochlor EC formulation (Ralph *et al.* 1992 RJ1337B) Results are for samples analysed in duplicate.

Country/ location MAIZE FODDER	Crop growth stage	Sample	DALA	Analysis 1	Analysis 2	mean
Whitakers North Carolina USA 1991	Pre-emergence	Fodder	137	0.04	0.04	0.04
Visalia, California, USA 1991	Pre-emergence	Fodder	122	0.01	0.01	0.01
Champaign Illinois USA 1991	Pre-emergence	Fodder	139	0.06	0.06	0.06
Ephrata Washington, USA 1991	Pre-emergence	Fodder	165	0.02	0.02	0.02
Paynesville Minnesota USA 1991	Pre-emergence	Fodder	148	< 0.01	< 0.01	< 0.01
York Nebraska, USA 1991	Pre-emergence	Fodder	126	0.16	0.15	0.16
Iconium Iowa USA 1991	Pre-emergence	Fodder	145	< 0.01	< 0.01	< 0.01
Berthoud Colorado USA 1991 [a]	Pre-emergence	Fodder	161	0.04	0.04	0.04
Noblesville Indiana USA 1991	Pre-emergence	Fodder	147	0.06	0.05	0.06
Sudlerville Maryland USA 1991	Pre-emergence	Fodder	175	< 0.01	< 0.01	< 0.01
Fabius New York USA 1991	Pre-emergence	Fodder	153	0.02	0.02	0.02
Fabius New York USA 1991	Pre-emergence	Fodder	147	0.02	0.01	0.02
Germansville Pennsylvania USA 1991	Pre-emergence	Fodder	138	< 0.01	< 0.01	< 0.01
Pulaski Pennsylvania USA 1991	Pre-emergence	Fodder	138	< 0.01	< 0.01	< 0.01

[a] Application rate 4.5 kg ai/ha

Table 97 Residues in maize fodder following application of a CS acetochlor formulation Lau (1992 MSL-11794). HEMA and EMA residues are expressed in acetochlor equivalents. Results are for samples analysed in duplicate.

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE FODDER		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Colo, Iowa, USA	1	3.4	Pre-emergent	126	Fodder	< 0.01	0.02	
1990 DK535						< 0.01	0.02	
					mean	< 0.01	0.02	< 0.03
	1	3.4	Pre-plant	138	Fodder	< 0.01	0.02	
						0.01	0.03	
					mean	< 0.01	0.03	< 0.04
Conklin, Michigan, USA	1	3.4	Pre-emergent	145	Fodder	0.01	0.05	
1990 Pioneer 3751						0.02	0.05	
					mean	0.02	0.05	0.07
Danville, Iowa, USA	1	3.4	Pre-emergent	156	Fodder	< 0.01	0.02	
1990 Dockendorf 7670						< 0.01	0.02	
					mean	< 0.01	0.02	< 0.03
Delavan, Wisconsin,	1	3.4	Pre-emergent	173	Fodder	< 0.01	0.02	
USA 1990 RK627						< 0.01	0.02	
					mean	< 0.01	0.02	< 0.03
Elwood, Illinois USA	1	3.4	Pre-emergent	142	Fodder	< 0.01	< 0.010	
1990 Pioneer 3615						< 0.01	0.02	
					mean	< 0.01	< 0.02	< 0.05
Geneseo, Illinois USA	1	3.4	Pre-emergent	147	Fodder	< 0.01	0.01	
USA 1990 Pioneer 3615						< 0.01	0.02	
					mean	< 0.01	0.02	< 0.03
	1	3.4	Pre-plant	147	Fodder	< 0.01	0.03	
						< 0.01	0.04	
					mean	< 0.01	0.04	< 0.05
Hollandale, Minnesota,	1	3.4	Pre-emergent	138	Fodder	< 0.01	0.01	
USA 1990 Pioneer 3751						< 0.01	0.01	
					mean	< 0.01	0.01	< 0.02
	1	3.4	Pre-plant	138	Fodder	< 0.01	0.02	
						< 0.01	0.02	
					mean	< 0.01	0.02	< 0.03
Elk City Kansas, USA	1	3.4	Pre-emergent	143	Fodder	< 0.01	0.01	
1990 Cargil 6127						< 0.01	0.02	
					mean	< 0.01	0.02	< 0.03
Leonard, Missouri, USA	1	3.4	Pre-emergent	127	Fodder	< 0.010	0.01	
1990 McAllister						0.01	0.02	
SX8611RFR					mean	< 0.01	0.02	< 0.03
	1	3.4	Pre-plant	127	Fodder	0.01	0.02	
						0.02	0.02	
					mean	0.01	0.02	0.04
New Holland Ohio, USA	1	3.4	Pre-emergent	189	Fodder	< 0.01	0.01	
1990 Pioneer 3343						< 0.01	0.02	
					mean	< 0.01	0.02	< 0.03
	1	3.4	Pre-plant	191	Fodder	< 0.01	0.01	
						< 0.01	0.01	
					mean	< 0.01	0.01	< 0.02
Noblesville Indiana,	1	3.4	Pre-emergent	148	Fodder	< 0.01	0.02	
USA 1990 Pioneer 3744						< 0.01	0.02	
					mean	< 0.01	0.02	< 0.03
Sioux Falls South Dakota	1	3.4	Pre-emergent	135	Fodder	0.03	0.04	
USA 1990 Moews 3140						0.03	0.05	
					mean	0.03	0.05	0.08
Uvalde Texas, USA	1	3.4	Pre-emergent	132	Fodder	0.01	0.02	
1990 Pioneer 3192						0.02	0.02	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE FODDER		kg ai/ha	at application			HEMA	EMA	(mg/kg)
					mean	0.01	0.02	0.03
York Nebraska, USA	1	3.4	Pre-emergent	162	Fodder	0.02	0.09	
1990 Pioneer 3379						0.03	0.13	
					mean	0.02	0.11	0.13
	1	3.4	Pre-plant	162	Fodder	0.02	0.07	
						0.02	0.07	
					mean	0.02	0.07	0.09

Values have been corrected for analytical method recoveries and expressed as acetochlor equivalents for either EMA (ethylmethylaniline producing) or HEMA (hydroxyethylrnethylaniline producing) residues

Table 98 Residues in maize fodder following post-emergent application of an EC or a CS acetochlor formulation (Maher 2007 MSL-20269). HEMA and EMA residues are expressed in acetochlor equivalents. Replicate samples.

Location, year, variety	Form	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE FODDER			kg ai/ha	at application			HEMA	EMA	(mg/kg)
Richland, Iowa, USA	EC	1	3.10	V8	103	Stover	0.07	0.69	
2006 NK N51-V9				68–81 cm			0.07	0.74	
						mean	0.07	0.72	0.79
	CS	1	3.33	V8	103	Stover	0.18	1.14	
				68–81 cm			0.20	1.19	
						mean	0.19	1.17	1.36
Hedrick, Iowa, USA	EC	1	2.97	V7–V8	106	Stover	0.05	0.57	
2006 Pioneer 34A16				71–84 cm			0.03	0.39	
						mean	0.04	0.48	0.52
	CS	1	3.17	V7–V8	106	Stover	0.02	0.09	
				71–84 cm			0.02	0.10	
							0.03	0.18	
							0.04	0.23	
						mean	0.03	0.15	0.18
Richland, Iowa, USA	EC	1	2.96	V8	108	Stover	0.04	0.53	
2006 Middle Koop 2212				71–86 cm			0.01	0.13	
							0.01	0.13	
						mean	0.02	0.26	0.28
	CS	1	3.18	V8	108	Stover	0.03	0.11	
				71–86 cm			0.02	0.12	
						mean	0.03	0.11	0.14
Perry, Iowa, USA 2006	EC	1	2.96PO	V8	96	Stover	0.02	0.16	
Pioneer 36B10				69–86 cm			0.03	0.29	
							0.01	0.12	
							0.02	0.23	
						mean	0.02	0.20	0.22
	EC	2	1.47PE	V8	96	Stover	0.01	0.12	
			1.50PO	69–86 cm			0.02	0.21	
							0.01	0.07	
							0.01	0.13	
						mean	0.01	0.13	0.14
	EC	1	2.88PO	V6	108	Stover	0.01	0.05	
				46–51 cm			0.02	0.08	
							0.01	0.02	
							0.01	0.03	
						mean	0.01	0.04	0.05
	CS	1	3.27PO	V8	96	Stover	0.03	0.20	
				69–86 cm			0.05	0.32	
						mean	0.04	0.26	0.30

Location, year, variety	Form	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE FODDER			kg ai/ha	at application			HEMA	EMA	(mg/kg)
	CS	2	1.60PE	V6	96	Stover	0.02	0.06	
			1.61PO	46–51 cm			0.02	0.11	
							0.03	0.11	
							0.04	0.19	
						mean	0.03	0.12	0.15
	CS	1	3.23PO	V8	108	Stover	< 0.01	0.02	
				69–86 cm			< 0.01	0.02	
						mean	< 0.01	0.02	0.02
Bagley, Iowa, USA 2006	EC	1	2.96	V8	97	Stover	0.08	1.09	
Pioneer 33P65				66–89 cm			0.09	1.22	
						mean	0.08	1.16	1.24
	CS	1	3.31	V8	97	Stover	0.07	0.39	
				66–89 cm			0.06	0.31	
						mean	0.07	0.35	0.42
Carlyle, Illinois, USA	EC	1	2.89	V9	121	Stover	0.03	0.25	
2006 DKC61-45				66–86 cm			0.02	0.18	
						mean	0.02	0.22	0.24
	CS	1	3.13	V9	121	Stover	0.06	0.32	
				66–86 cm			0.13	0.42	
						mean	0.10	0.37	0.47
Mason, Illinois, USA	EC	1	3.06	BBCH 18	100	Stover	0.05	0.47	
2006 Midland mg 606RR				66–91 cm			0.04	0.38	
						mean	0.05	0.42	0.47
	CS	1	3.32	BBCH 18	100	Stover	0.22	1.31	
				66–91 cm			0.19	1.21	
						mean	0.20	1.26	1.46
Wyoming, Illinois, USA	EC	1	2.95	V8	114	Stover	0.03	0.50	
2006 Burns 644 RWR				74–79 cm			0.04	0.58	
							0.01	0.04	
							0.02	0.16	
						mean	0.03	0.32	0.35
	CS	1	3.15	V8	114	Stover	0.04	0.19	
				74–79 cm			0.05	0.23	
						mean	0.05	0.21	0.26
Danville, Indiana, USA	EEC	1	2.82	BBCH 18	140	Stover	0.01	0.07	
2006 Wyffels W5531				66–91 cm			0.01	0.08	
							0.02	0.17	
							0.02	0.18	
						mean	0.01	0.13	0.14
	CS	1	3.19	BBCH 18	140	Stover	0.02	0.13	
				66–91 cm			0.02	0.14	
							0.02	0.23	
							0.02	0.22	
						mean	0.02	0.18	0.20
Rockville, Indiana, USA	EC	1	2.82	BBCH 18	130	Stover	0.02	0.22	
2006 Pioneer 33NO8				66–86 cm			0.02	0.21	
						mean	0.02	0.22	0.24
	CS	1	3.33	BBCH 18	130	Stover	0.02	0.24	
				66–86 cm			0.03	0.24	
							0.04	0.53	
						mean	0.03	0.34	0.37
Paynesville, Minnesota,	EC	1	2.93	V8	123	Stover	< 0.01	0.03	
USA 2006 Dekalb				71–86 cm			< 0.01	0.04	
DKC47-10 RR2						mean	< 0.01	0.03	0.03
	CS	1	3.19	V8	123	Stover	< 0.01	0.24	

Location, year, variety	Form	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE FODDER			kg ai/ha	at application			HEMA	EMA	(mg/kg)
				71–86 cm			< 0.01	0.16	
						mean	< 0.01	0.20	0.20
Hawick, Minnesota, USA	EC	1	2.87	76 cm	123	Stover	< 0.01	0.07	
2006 Dekalb DKC47-10							< 0.01	0.12	
RR2						mean	< 0.01	0.09	0.10
	CS	1	3.22	76 cm	123	Stover	< 0.01	0.20	
							< 0.01	0.21	
						mean	< 0.01	0.20	0.20
LaPlata, Missouri, USA	EC	1	3.04	V8	103	Stover	0.04	0.51	
2006 Dekalb DKC61-42				71–79 cm			0.03	0.30	
						mean	0.04	0.41	0.45
	EC	2	1.43PE	V8	103	Stover	0.01	0.12	
			1.49PO	71–79 cm			0.02	0.19	
						mean	0.01	0.15	0.16
	EC	1	2.90	V6	110	Stover	< 0.01	0.02	
				46–51 cm			0.01	0.01	
						mean	< 0.01	0.02	0.02
	CS	1	3.26	V8	103	Stover	0.04	0.27	
				71–79 cm			0.12	0.75	
						mean	0.08	0.51	0.59
	CS	2	1.61PE	V8	103	Stover	0.02	0.16	
			1.61PO	71–79 cm			0.02	0.16	
						mean	0.02	0.16	0.18
	CS	1	3.19	V6	110	Stover	0.01	0.03	
				71–79 cm			0.01	0.05	
							0.01	0.03	
							0.01	0.05	
						mean	0.01	0.04	0.05
Seven Springs, North	EC	1	2.96	BBCH 33	83	Stover	0.03	0.17	
Carolina, USA 2006				71–86 cm			0.03	0.21	
Garst 8377						mean	0.03	0.19	0.22
	CS	1	3.24	BBCH 33	83	Stover	0.04	0.18	
				71–86 cm			0.05	0.25	
						mean	0.04	0.22	0.26
York, Nebraska, USA	EC	1	2.94	BBCH 18	106	Stover	0.01	0.05	
2006 Pioneer 34N45				69–81 cm			0.01	0.06	
RR2/YGCB						mean	0.01	0.05	0.06
	CS	1	3.18	BBCH 18	106	Stover	0.01	0.08	
				69–81 cm			0.01	0.10	
						mean	0.01	0.09	0.10
Osceola, Nebraska, USA	EC	1	2.94	BBCH 18	103	Stover	0.01	0.20	
2006 N73-F7 RR/LL/CB				66–81 cm			0.01	0.13	
						mean	0.01	0.17	0.18
	CS	1	3.16	BBCH 18	103	Stover	0.03	0.18	
				66–81 cm			0.04	0.24	
						mean	0.04	0.21	0.25
Baptistown, New Jersey,	EC	1	3.00	V8	97	Stover	0.01	0.10	
USA TA5750/ 401169				61–91 cm			0.01	0.09	
							0.02	0.18	
							0.02	0.19	
						mean	0.01	0.14	0.15
	CS	1	3.26	V8	97	Stover	0.03	0.53	
				61–91 cm			0.03	0.61	

Location, year, variety	Form	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
MAIZE FODDER			kg ai/ha	at application			HEMA	EMA	(mg/kg)
						mean	0.03	0.57	0.60
Washington, Ohio, USA	EC	1	2.97	V8–V9	110	Stover	0.05	0.96	
2006 SC 11RR06				71–84 cm			0.03	0.66	
						mean	0.04	0.81	0.85
	CS	1	3.14	V8–V9	110	Stover	0.02	0.32	
				71–84 cm			0.03	nr	
						mean	0.03	0.32	0.35
New Holland, Ohio, USA	EC	1	3.00	V8	120	Stover	0.02	0.27	
2006 Crows 515Z R				71–79 cm			0.01	0.22	
						mean	0.01	0.24	0.25
	CS	1	3.17	V8	120	Stover	0.05	0.74	
				71–79 cm			0.05	0.60	
							0.01	0.38	
							0.02	0.67	
						mean	0.03	0.60	0.63
Dill City, Oklahoma, USA	EC	1	2.90	V8–V9	89	Stover	0.24	2.57	
2006 DK C48-53				74–81 cm			0.27	3.24	
						mean	0.26	2.91	3.17
	CS	1	3.19	V8–V9	89	Stover	0.13	0.80	
				74–81 cm			0.13	0.94	
							0.06	0.28	
							0.05	0.26	
						mean	0.09	0.57	0.66
Delavan, Wisconsin, USA	EC	1	2.89	V8	124	Stover	0.01	0.18	
2006 Dekalb DKC51-39				74–79 cm			0.02	0.20	
						mean	0.01	0.19	0.20
	CS	1	3.09	V8	124	Stover	0.05	0.27	
				74–79 cm			0.08	0.38	
						mean	0.07	0.33	0.40

PE = pre-emergent

PO = post-emergent

Table 99 Residues in soya bean forage following application of a CS acetochlor formulation (Hay *et al.* 2008 MSL-20719). HEMA and EMA residues are expressed in acetochlor equivalents. Replicate samples.

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SOYA BEAN FORAGE		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Proctor, Arkansas, USA	1	3.36	V5/R1–R2	7	Forage	3.14	93.45	
2007 AG4403RR						3.23	89.08	
					mean	3.19	91.27	94.45
	3 (71 12)	1.12	Bare ground	7	Forage	1.51	34.20	
		1.12	V3			1.51	34.75	
		1.12	V5/R1–R2		mean	1.51	34.48	35.98
Newport, Arkansas,	1	3.37	R2	7	Forage	1.72	74.55	
USA 2007 JG55R505C						1.92	72.80	
					mean	1.82	73.68	75.50
	3 (65 32)	1.13	Pre-plant	7	Forage	0.97	14.35	
		1.11	V3			0.97	14.45	
		1.12	R2		mean	0.97	14.40	15.37

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SOYA BEAN FORAGE		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Richland, Iowa, USA	1	3.36	R1	0	Forage	0.06	97.32	
2007 Asgrow 3101						0.09	99.05	
					mean	0.07	98.19	98.26
				7	Forage	1.29	39.79	
						1.15	32.90	
					mean	1.22	36.35	37.57
				14	Forage	1.12	23.60	
						1.22	29.08	
					mean	1.17	26.34	27.51
				21	Forage	0.87	11.32	
						1.10	14.02	
					mean	0.98	12.67	13.65
	3 (76 28)	1.17	Pre-plant	7	Forage	0.30	6.08	
		1.10	V3			0.26	7.08	
		1.11	R1		mean	0.28	6.58	6.87
Ollie, Iowa, USA	1	3.38	R1	8	Forage	1.77	30.20	
2007 AG 3802						2.00	34.65	
					mean	1.88	32.43	34.31
	3 (68 30)	1.12	Pre-plant	8	Forage	0.56	7.19	
		1.11	V3			0.80	7.13	
		1.13	R1		mean	0.68	7.16	7.84
Milford, Iowa, USA	1	3.33	R1–R2	8	Forage	1.14	26.56	
2007 NK S19-L7						1.40	26.85	
					mean	1.27	26.71	27.97
	3 (74 33)	1.11	Pre-plant	8	Forage	0.57	9.12	
		1.10	V3			0.57	9.89	
		1.09	R1–R2		mean	0.57	9.51	10.07
Bagley, Iowa, USA	1	3.37	R2	7	Forage	1.09	35.91	
2007 92M52						1.30	43.61	
					mean	1.20	39.76	40.96
	3 (74 24)	1.14	Pre-plant	7	Forage	0.93	16.03	
		1.11	V3			0.93	17.85	
		1.14	R2		mean	0.93	16.94	17.87
Carlyle, Illinois, USA	1	3.36	R1–R2	0	Forage	0.20	123.60	
2007 5N382 RR						0.17	123.70	
					mean	0.19	123.65	123.84
				7	Forage	1.85	84.47	
						1.33	55.85	
					mean	1.59	70.16	71.75
				14	Forage	2.52	39.83	
						2.21	37.06	
					mean	2.37	38.45	40.81
				21	Forage	2.34	25.98	
						2.60	24.32	
					mean	2.47	25.15	27.62
	3 (72 25)	1.13	Pre-plant	7	Forage	0.79	15.32	
		1.13	V3			0.66	14.80	
		1.11	R1–R2		mean	0.72	15.06	15.78
Carlyle, Illinois, USA	1	3.4	R1–R2	7	Forage	1.58	48.16	
2007 NK 37N4						2.10	49.95	
					mean	1.84	49.06	50.89
	3 (71 27)	1.12	Pre-plant	7	Forage	1.06	18.23	
		1.12	V3			0.88	15.71	
		1.14	R1–R2		mean	0.97	16.97	17.94
Mason, Illinois, USA	1	3.41	R2	7	Forage	3.17	70.96	
2007 Trisler T-3463 RR						2.60	57.27	
					mean	2.89	64.12	67.00
	3 (88 15)	1.12	Pre-plant	7	Forage	2.41	29.88	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SOYA BEAN FORAGE		kg ai/ha	at application			HEMA	EMA	(mg/kg)
		1.15	BBCH 14/V3			1.98	28.33	
		1.13	R2		mean	2.20	29.11	31.30
Wyoming, Illinois,	1	3.45	R1–R2	7	Forage	2.00	62.04	
USA 2007 AG3101						2.17	73.94	
					mean	2.08	67.99	70.07
	3 (78 23)	1.16	Pre-plant	7	Forage	0.85	18.44	
		1.12	V3			0.84	17.68	
		1.08	R1–R2		mean	0.85	18.06	18.91
Rockville, Indiana,	1	3.43	R1	7	Forage	2.53	92.10	
USA 2007 T-3463RR						3.53	84.64	
					mean	3.03	88.37	91.40
	3 (74 21)	1.22	Pre-plant	7	Forage	1.06	24.79	
		1.11	BBCH 14/V3			0.87	22.66	
		1.12	R1		mean	0.96	23.73	24.69
New Ross, Indiana,	1	3.5	R1	7	Forage	1.39	51.16	
USA 2007 T-3463RR						1.57	52.63	
					mean	1.48	51.90	53.37
	3 (74 21)	1.15	Pre-plant	7	Forage	0.84	21.39	
		1.13	V3			0.89	19.29	
		1.10	R1		mean	0.86	20.34	21.20
Washington, Louisiana,	1	3.35	R2	7	Forage	1.84	45.89	
USA 2007 AG 5905						1.97	46.95	
					mean	1.90	46.42	48.32
	3 (70 28)	1.15	Pre-plant	7	Forage	0.71	10.40	
		1.13	V3			0.71	10.38	
		1.10	R2		mean	0.71	10.39	11.10
Paynesville, Minnesota,	1	3.38	R2	7	Forage	0.00	0.53	
USA 2007						0.01	0.66	
90M60-N201					mean	0.01	0.60	0.60
	3 (88 36)	1.11	Pre-plant	7	Forage	0.00	0.28	
		1.11	V3			0.00	0.22	
		1.12	R2		mean	0.00	0.25	0.25
Geneva, Minnesota,	1	3.41	R2	7	Forage	1.26	38.03	
USA 2007 Pioneer						1.23	39.06	
91M30					mean	1.25	38.55	39.79
	3 (70 19)	1.12	Pre-plant	7	Forage	0.60	13.77	
		1.12	V3			0.59	14.52	
		1.11	R2		mean	0.59	14.15	14.74
La Plata, Missouri, USA	1	2.69	R1–R2	7	Forage	1.33	70.17	
2007 Asgrow AG3802						1.49	70.31	
					mean	1.41	70.24	71.65
	3 (76 32)	1.14	Pre-plant	7	Forage	0.61	22.98	
		1.12	V3–V4 (90% V3)			0.66	24.94	
		1.12	R1–R2		mean	0.64	23.96	24.60
Pikeville, North	1	3.41	R1, beginning	6	Forage	2.14	72.11	
Carolina, USA 2007			to flower			1.84	59.27	
NK 565-M3					mean	1.99	65.69	67.68
	3 (76 40)	1.13	Pre-plant	6	Forage	1.20	17.72	
		1.11	BBCH 14/V3			1.11	15.26	
		1.12	R1/beginning to flower		mean	1.15	16.49	17.64
York, Nebraska, USA	1	3.36	BBCH61/R1	7	Forage	1.05	26.38	
2007 WW152201						1.21	28.33	
					mean	1.13	27.36	28.49
	3 (79 14)	1.13	Pre-plant	7	Forage	1.06	14.54	
		1.11	BBCH 15/ late 3rd			0.89	13.95	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SOYA BEAN FORAGE		kg ai/ha	at application			HEMA	EMA	(mg/kg)
			trifoliate					
		1.12	BBCH 61/ R1		mean	0.97	14.25	15.22
New Holland, Ohio,	1	3.43	R1–R2	6	Forage	2.24	85.54	
USA 2007 Crop Plan						2.65	90.16	
RC 3935					mean	2.44	87.85	90.29
	3 (85 21)	1.13	Pre-plant	6	Forage	1.11	23.82	
		1.13	V3			1.13	24.24	
		1.13	R1–R2		mean	1.12	24.03	25.15
New Holland, Ohio,	1	3.43	R1–R2	7	Forage	1.98	60.71	
USA 2007 Crows						2.47	66.93	
3518 R					mean	2.23	63.82	66.05
	3 (85 21)	1.13	Pre-plant	7	Forage	1.33	18.02	
		1.12	V3			1.36	22.01	
		1.12	R1–R2		mean	1.34	20.02	21.36
Elko, South Carolina,	1	3.38	R2	7	Forage	2.10	54.93	
USA 2007 97M50						2.55	62.06	
					mean	2.33	58.50	60.82
	3 (73 40)	1.15	Pre-plant	7	Forage	0.97	15.40	
		1.12	V3			0.91	15.41	
		1.13	R2		mean	0.94	15.41	16.34

Table 100 Residues in soya bean hay following application of a CS acetochlor formulation (Hay *et al.* 2008 MSL-20719). HEMA and EMA residues are expressed in acetochlor equivalents. Replicate samples.

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SOYA BEAN HAY		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Proctor, Arkansas, USA	1	3.36	V5/R1–R2	40 (41)	Hay	2.58	25.67	
2007 AG4403RR						2.56	25.93	
					mean	2.57	25.80	28.37
	3 (71 12)	1.12	Bare ground	40 (41)	Hay	1.51	12.54	
		1.12	V3			1.46	12.40	
		1.12	V5/R1–R2		mean	1.49	12.47	13.96
Newport, Arkansas,	1	3.37	R2	22 (25)	Hay	8.98	97.54	
USA 2007 JG55R505C						9.34	100.90	
					mean	9.16	99.22	108.38
	3 (65 32)	1.13	Pre-plant	22 (25)	Hay	5.05	36.79	
		1.11	V3			4.96	37.37	
		1.12	R2		mean	5.01	37.08	42.09
Richland, Iowa, USA	1	3.36	R1	21 (26)	Hay	3.24	43.33	
2007 Asgrow 3101						3.99	48.62	
					mean	3.62	45.98	49.59
				26 (29)	Hay	3.12	32.94	
						3.18	32.89	
					mean	3.15	32.92	36.06
				33 (36)	Hay	1.42	12.31	
						1.21	10.76	
					mean	1.32	11.54	12.85
				40 (44)	Hay	1.31	11.53	
						1.48	12.11	
					mean	1.39	11.82	13.21
	3 (76 28)	1.17	Pre-plant	26 (29)	Hay	0.77	6.84	
		1.10	V3			0.77	6.70	
		1.11	R1		mean	0.77	6.77	7.54
Ollie, Iowa, USA	1	3.38	R1	25 (27)	Hay	2.22	22.06	
2007 AG 3802						2.09	21.85	
					mean	2.16	21.96	24.11

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SOYA BEAN HAY		kg ai/ha	at application			HEMA	EMA	(mg/kg)
	3 (68 30)	1.12	Pre-plant	25 (27)	Hay	0.65	5.25	
		1.11	V3			0.71	6.17	
		1.13	R1		mean	0.68	5.71	6.39
Milford, Iowa, USA	1	3.33	R1–R2	28 (33)	Hay	1.30	13.95	
2007 NK S19-L7						1.74	16.80	
					mean	1.52	15.38	16.89
	3 (74 33)	1.11	Pre-plant	28 (33)	Hay	0.90	6.13	
		1.10	V3			0.67	4.99	
		1.09	R1–R2		mean	0.79	5.56	6.35
Bagley, Iowa, USA	1	3.37	R2	19 (24)	Hay	3.56	65.90	
2007 92M52						3.30	68.75	
					mean	3.43	67.33	70.76
	3 (74 24)	1.14	Pre-plant	19 (24)	Hay	1.72	20.29	
		1.11	V3			1.95	25.03	
		1.14	R2		mean	1.83	22.66	24.49
Carlyle, Illinois, USA	1	3.36	R1–R2	21 (24)	Hay	6.33	66.87	
2007 5N382 RR						6.37	69.55	
					mean	6.35	68.21	74.56
				28 (31)	Hay	5.31	42.70	
						5.20	41.53	
					mean	5.26	42.12	47.37
				35 (38)	Hay	4.29	30.59	
						4.43	31.54	
					mean	4.36	31.07	35.42
				42 (45)	Hay	2.84	19.79	
						3.41	22.02	
					mean	3.12	20.91	24.03
	3 (72 25)	1.13	Pre-plant	28 (31)	Hay	2.50	14.46	
		1.13	V3			2.67	16.25	
		1.11	R1–R2		mean	2.59	15.36	17.94
Carlyle, Illinois, USA	1	3.4	R1–R2	28 (31)	Hay	2.50	32.60	
2007 NK 37N4						3.16	33.55	
					mean	2.83	33.08	35.90
	3 (71 27)	1.12	Pre-plant	28 (31)	Hay	2.11	15.28	
		1.12	V3			1.38	9.71	
		1.14	R1–R2		mean	1.75	12.50	14.24
Mason, Illinois, USA	1	3.41	R2	17 (20)	Hay	6.79	81.34	
2007 Trisler T-3463 RR						6.09	79.14	
					mean	6.44	80.24	86.68
	3 (88 15)	1.12	Pre-plant	17 (20)	Hay	3.52	30.98	
		1.15	BBCH 14/V3			3.72	31.97	
		1.13	R2		mean	3.62	31.48	35.09
Wyoming, Illinois,	1	3.45	R1–R2	16 (20)	Hay	6.53	121.10	
USA 2007 AG3101						7.07	123.80	
					mean	6.80	122.45	129.25
	3 (78 23)	1.16	Pre-plant	16 (20)	Hay	2.00	28.27	
		1.12	V3			2.03	29.76	
		1.08	R1–R2		mean	2.01	29.02	31.03
Rockville, Indiana,	1	3.43	R1	17 (23)	Hay	4.96	67.06	
USA 2007 T-3463RR						5.84	63.85	
					mean	5.40	65.46	70.86
	3 (74 21)	1.22	Pre-plant	17 (23)	Hay	1.65	18.38	
		1.11	BBCH 14/V3			1.84	21.33	
		1.12	R1		mean	1.74	19.86	21.60
New Ross, Indiana,	1	3.5	R1	17 (23)	Hay	1.69	20.44	
USA 2007 T-3463RR						1.76	20.91	
					mean	1.73	20.68	22.40
	3 (74 21)	1.15	Pre-plant	17 (23)	Hay	2.34	27.37	
		1.13	V3			1.84	22.92	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SOYA BEAN HAY		kg ai/ha	at application			HEMA	EMA	(mg/kg)
		1.10	R1		mean	2.09	25.15	27.23
Washington, Louisiana,	1	3.35	R2	25 (28)	Hay	5.72	53.08	
USA 2007 AG 5905						5.64	49.32	
					mean	5.68	51.20	56.88
	3 (70 28)	1.15	Pre-plant	25 (28)	Hay	1.83	11.86	
		1.13	V3			1.80	14.14	
		1.10	R2		mean	1.82	13.00	14.82
Paynesville, Minnesota,	1	3.38	R2	30 (32)	Hay	0.00	0.75	
USA 2007						0.01	1.41	
90M60-N201					mean	0.01	1.08	1.09
	3 (88 36)	1.11	Pre-plant	30 (32)	Hay	0.01	0.29	
		1.11	V3			0.00	0.15	
		1.12	R2		mean	0.01	0.22	0.23
USA 2007 Pioneer				17 (20)	Hay	4.08	70.57	
91M30						4.81	78.92	
					mean	4.45	74.75	79.19
	3 (70 19)	1.12	Pre-plant	17 (20)	Hay	1.03	17.42	
		1.12	V3			1.10	18.83	
		1.11	R2		mean	1.07	18.13	19.19
La Plata, Missouri, USA	1	2.69	R1–R2	26 (29)	Hay	3.12	32.72	
2007 Asgrow AG3802						3.50	34.49	
					mean	3.31	33.61	36.92
	3 (76 32)	1.14	Pre-plant	26 (29)	Hay	1.59	16.06	
		1.12	V3–V4, (90% V3),			1.40	13.93	
		1.12	R1–R2		mean	1.50	15.00	16.49
Pikeville, North Carolina,	1	3.41	R1, beginning	32 (34)	Hay	4.82	34.96	
USA 2007 NK 565-M3			to flower			6.05	43.89	
					mean	5.44	39.43	44.86
	3 (76 40)	1.13	Pre-plant	32 (34)	Hay	2.11	10.78	
		1.11	BBCH 14/V3			2.62	12.02	
		1.12	R1/beginning to flower		mean	2.36	11.40	13.76
York, Nebraska, USA	1	3.36	BBCH61/R1	27 (32)	Hay	2.13	23.45	
2007 WW152201						2.40	23.60	
					mean	2.27	23.53	25.79
	3 (79 14)	1.13	Pre-plant	27 (32)	Hay	1.10	10.20	
		1.11	BBCH 15/ late 3rd trifoliate			1.14	11.10	
		1.12	BBCH 61/ R1		mean	1.12	10.65	11.77
New Holland, Ohio,	1	3.43	R1–R2	28 (30)	Hay	4.55	39.87	
USA 2007						4.81	42.31	
Crop Plan RC 3935					mean	4.68	41.09	45.77
	3 (85 21)	1.13	Pre-plant	28 (30)	Hay	1.26	9.71	
		1.13	V3			1.44	10.89	
		1.13	R1–R2		mean	1.35	10.30	11.65
New Holland, Ohio,	1	3.43	R1–R2	28 (30)	Hay	3.13	24.59	
USA 2007						2.77	20.44	
Crows 3518 R					mean	2.95	22.52	25.47
	3 (85 21)	1.13	Pre-plant	28 (30)	Hay	1.47	8.11	
		1.12	V3			1.81	10.77	
		1.12	R1–R2		mean	1.64	9.44	11.08
Elko, South Carolina,				12 (19)	Hay	9.51	115.30	
USA 2007 97M50						9.28	111.10	
					mean	9.39	113.20	122.59
	3 (73 40)	1.15	Pre-plant	12 (19)	Hay	4.39	37.52	
		1.12	V3			4.06	33.08	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SOYA BEAN HAY		kg ai/ha	at application			HEMA	EMA	(mg/kg)
		1.13	R2		mean	4.22	35.30	39.52

Table 101 Residues in sugar beet tops following application of a CS acetochlor formulation (Mueth and Foster 2012 MSL-24198). HEMA and EMA residues are expressed in acetochlor equivalents. Replicate samples.

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SUGAR BEET TOPS		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Conklin, Michigan,	2	1.65	Pre-emergence	108	Tops	0.019	0.027	
USA 2011 18RR26		1.67	6-leaf			0.023	0.032	
					mean	0.021	0.030	0.051
	2	1.68	2-leaf	108	Tops	0.013	0.020	
		1.67	6-leaf			0.013	0.019	
					mean	0.010	0.019	0.033
	1	3.37	6-leaf	108	Tops	0.025	0.046	
						0.030	0.054	
					mean	0.027	0.050	0.077
				101	Tops	0.030	0.049	
						0.030	0.049	
						0.020	0.030	
						0.033	0.050	
					mean	0.028	0.045	0.073
				108	Tops	0.025	0.046	
						0.030	0.054	
					mean	0.027	0.050	0.077
				115	Tops	0.021	0.034	
						0.023	0.034	
					mean	0.022	0.034	0.056
				122	Tops	0.026	0.044	
						0.030	0.046	
					mean	0.028	0.045	0.073
				129	Tops	0.019	0.027	
						0.021	0.029	
					mean	0.020	0.028	0.048
Richland, Iowa, USA	2	1.69	Pre-emergence	107	Tops	0.005	0.016	
2011 SX Triton		1.67	6-leaf			0.005	0.014	
					mean	0.005	0.015	0.020
	2	1.66	2-leaf	107	Tops	0.006	0.022	
		1.67	6-leaf			0.005	0.023	
					mean	0.010	0.022	0.028
	1	3.35	6-leaf	107	Tops	0.004	0.021	
						0.009	0.030	
					mean	0.006	0.025	0.032
York, Nebraska, USA	2	1.68	Pre-emergence	122	Tops	0.003	0.005	
2011 Hilleshog 9093 RR		1.66	6-leaf			0.004	0.006	
					mean	0.004	0.005	0.009
	2	1.66	2-leaf	122	Tops	0.004	0.006	
		1.64	6-leaf			0.004	0.005	
					mean	0.004	0.005	0.009
	1	3.33	6-leaf	122	Tops	0.005	0.007	
						0.005	0.007	
					mean	0.005	0.007	0.012
Geneva, Minnesota, USA	2	1.68	Pre-emergence	89	Tops	0.008	0.019	
2011 3035 RZ		1.67	6-leaf			0.007	0.022	
						0.008	0.026	
						0.015	0.034	
					mean	0.009	0.025	0.035
	2	1.67	2-leaf		Tops	0.006	0.016	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SUGAR BEET TOPS		kg ai/ha	at application			HEMA	EMA	(mg/kg)
		1.67	6-leaf			0.009	0.022	
					mean	0.008	0.019	0.027
	1	3.40	6-leaf		Tops	0.007	0.024	
						0.008	0.022	
					mean	0.008	0.023	0.030
Perley, Minnesota, USA	2	1.73	Pre-emergence	103	Tops	0.005	0.009	
2011 SX Uplander RR		1.67	6-leaf			0.006	0.010	
					mean	0.005	0.009	0.014
	2	1.66	2-leaf	103	Tops	0.005	0.008	
		1.69	6-leaf			0.004	0.007	
					mean	0.005	0.007	0.012
	1	3.33	6-leaf	103	Tops	0.005	0.012	
						0.005	0.010	
					mean	0.005	0.011	0.016
Gardner, North Dakota,	2	1.70	Pre-emergence	103	Tops	0.006	0.012	
USA SV36812 RR		1.71	6-leaf			0.008	0.015	
						0.005	0.009	
						0.011	0.017	
					mean	0.007	0.013	0.020
	2	1.68	2-leaf	103	Tops	0.010	0.016	
		1.75	6-leaf			0.011	0.019	
					mean	0.010	0.018	0.028
	1	3.41	6-leaf	103	Tops	0.007	0.015	
						0.009	0.024	
						0.012	0.020	
						0.011	0.021	
					mean	0.010	0.020	0.030
Norwich, North Dakota,	2	1.69	Pre-emergence	93	Tops	0.009	0.019	
USA 2011 Crystal R434		1.70	6-leaf			0.011	0.017	
					mean	0.010	0.018	0.028
	2	1.67	2-leaf	93	Tops	0.011	0.031	
		1.70	6-leaf			0.020	0.034	
						0.014	0.021	
						0.014	0.027	
					mean	0.015	0.028	0.043
	1	3.46	6-leaf	93	Tops	0.011	0.036	
						0.014	0.052	
					mean	0.012	0.044	0.056
Velva, North Dakota,	2	1.70	Pre-emergence	93	Tops	0.011	0.042	
USA Crystal R308		1.71	6-leaf			0.014	0.044	
					mean	0.012	0.043	0.056
	2	1.67	2-leaf	93	Tops	0.007	0.025	
		1.73	6-leaf			0.009	0.026	
					mean	0.008	0.026	0.033
	1	3.47	6-leaf	93	Tops	0.007	0.026	
						0.008	0.027	
					mean	0.007	0.026	0.034
Grand Island, Nebraska,	2	1.68	Pre-emergence	113	Tops	0.024	0.034	
USA 2011 Hilleshog		1.69	6-leaf			0.029	0.040	
Monogen 9093 RR					mean	0.026	0.037	0.063
	2	1.68	2-leaf	113	Tops	0.028	0.034	
		1.69	6-leaf			0.030	0.035	
					mean	0.029	0.035	0.063
	1	3.36	6-leaf	113	Tops	0.023	0.027	
						0.022	0.023	
					mean	0.022	0.025	0.047
Larned, Kansas, USA	2	1.70	Pre-emergence	63	Tops	0.011	0.016	
2011 Am Crystal R308		1.69	6-leaf			0.015	0.023	
					mean	0.013	0.020	0.033
	2	1.68	2-leaf	63	Tops	0.017	0.029	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SUGAR BEET TOPS		kg ai/ha	at application			HEMA	EMA	(mg/kg)
		1.69	6-leaf			0.024	0.034	
						0.010	0.017	
						0.011	0.021	
					mean	0.016	0.025	0.041
	1	3.44	6-leaf	63	Tops	0.012	0.028	
						0.017	0.038	
						0.007	0.016	
						0.009	0.017	
					mean	0.011	0.025	0.036
Jerome, Idaho, USA	2	1.68	Pre-emergence	119	Tops	0.011	0.019	
2011 Grystal RR876		1.70	6-leaf			0.011	0.019	
						0.012	0.020	
						0.019	0.034	
					mean	0.001	0.023	0.036
	2	1.70	2-leaf	119	Tops	0.023	0.027	
		1.70	6-leaf			0.019	0.030	
					mean	0.021	0.029	0.050
	1	3.37	6-leaf	119	Tops	0.010	0.013	
						0.011	0.017	
					mean	0.010	0.015	0.025
Porterville, California,	2	1.70	Pre-emergence	83	Tops	0.054	0.343	
USA 2011 Pheonix		1.71	6-leaf			0.131	0.587	
						0.099	0.421	
						0.107	0.474	
					mean	0.098	0.456	0.554
	2	1.70	2-leaf	83	Tops	0.056	0.148	
		1.70	6-leaf			0.042	0.172	
					mean	0.049	0.160	0.209
	1	3.33	6-leaf	76	Tops	0.175	0.839	
						0.199	0.993	
					mean	0.187	0.916	1.100
				83	Tops	0.128	0.808	
						0.127	0.899	
						0.045	0.243	
						0.042	0.248	
					mean	0.085	0.550	0.635
				90	Tops	0.124	0.662	
						0.145	0.866	
						0.205	1.000	
						0.223	1.010	
					mean	0.174	0.885	1.060
				98	Tops	0.050	0.226	
						0.049	0.236	
						0.053	0.435	
						0.058	0.449	
					mean	0.052	0.337	0.389
				104	Tops	0.018	0.101	
						0.021	0.116	
						0.037	0.148	
						0.027	0.181	
					mean	0.026	0.137	0.162
Ephrata, Washington,	2	1.68	Pre-emergence	131	Tops	0.061	0.123	
USA Crystal RR876		1.68	6-leaf			0.040	0.072	
					mean	0.050	0.097	0.147
	2	1.68	2-leaf	131	Tops	0.033	0.086	
		1.69	6-leaf			0.033	0.107	
						0.019	0.054	
						0.022	0.066	
					mean	0.027	0.079	0.105
	1	3.35	6-leaf	131	Tops	0.022	0.088	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SUGAR BEET TOPS		kg ai/ha	at application			HEMA	EMA	(mg/kg)
						0.030	0.130	
						0.039	0.118	
						0.050	0.154	
					mean	0.035	0.122	0.158
Rupert, Idaho, USA 2011	2	1.60	Pre-emergence	113	Tops	0.011	0.022	
Crystal RR929		1.69	6-leaf			0.008	0.018	
					mean	0.009	0.020	0.030
	2	1.69	2-leaf	113	Tops	0.010	0.019	
		1.65	6-leaf			0.010	0.021	
					mean	0.010	0.020	0.030
	1	3.34	6-leaf	113	Tops	0.009	0.027	
						0.011	0.030	
					mean	0.010	0.029	0.039
Minto, Manitoba, Canada	2	1.74	Pre-emergence	89	Tops	0.007	0.013	
2011 SVDH 66854		1.84	6-leaf			0.006	0.011	
					mean	0.006	0.012	0.018
	2	1.70	2-leaf	89	Tops	0.006	0.012	
		1.67	6-leaf			0.006	0.013	
					mean	0.006	0.013	0.019
	1	3.35	6-leaf	89	Tops	0.008	0.015	
						0.011	0.019	
					mean	0.009	0.017	0.026

Table 102 Residues in sorghum forage following pre-emergent or early post-emergent application of an EC or a CS acetochlor formulation Moran (2004 MSL-18670). HEMA and EMA residues are expressed in acetochlor equivalents. Replicate samples.

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SORGHUM FORAGE		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Plains, Georgia,	1	2.77	PE	85	Forage	0.030	0.153	
USA 2003 A571						0.031	0.160	
					mean	0.031	0.157	0.187
	1	2.79	PO	71	Forage	0.042	0.195	
			15–20 cm			0.036	0.168	
					mean	0.039	0.182	0.221
Cord, Arkansas, USA	1	2.78	PE	87	Forage	0.032	0.141	
2003 Garst 5515						0.034	0.152	
					mean	0.033	0.147	0.180
	1	2.80	PO	68	Forage	0.018	0.090	
			23 cm			0.020	0.037	
					mean	0.019	0.064	0.083
Carlyle, Illinois, USA	1	2.87	PE	84	Forage	< 0.003	0.015	
2003 KS 585						< 0.003	0.015	
					mean	< 0.003	0.015	< 0.018
	1	2.89	PO	55	Forage	0.008	0.050	
			25 cm			0.007	0.048	
					mean	0.007	0.049	0.056
New Holland, Ohio, USA	1	2.73	PE	116	Forage	0.009	0.058	
2003 A571						0.006	0.046	
					mean	0.008	0.052	0.060
	1	2.78	PO	77	Forage	0.016	0.094	
			25 cm			0.020	0.096	
					mean	0.018	0.095	0.113
York, Nebraska,	1	2.79	PE	92	Forage	0.020	0.168	
USA 2003 Eclipse						0.018	0.128	
					mean	0.019	0.148	0.167
	1	2.80	PO	48	Forage	0.007	0.077	

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SORGHUM FORAGE		kg ai/ha	at application			HEMA	EMA	(mg/kg)
			13–15 cm			0.009	0.106	
					mean	0.008	0.091	0.099
				59		0.008	0.142	
						0.009	0.166	
					mean	0.009	0.154	0.163
				66		0.016	0.084	
						0.016	0.084	
					mean	0.016	0.084	0.100
				66		0.019	0.213	
						0.018	0.184	
					mean	0.018	0.199	0.217
				73		0.023	0.166	
						0.026	0.083	
					mean	0.024	0.124	0.149
Richland, Iowa, USA	1	2.86	PE	82	Forage	0.010	0.057	
2003 Dekaalb AS71						0.014	0.073	
					mean	0.012	0.065	0.077
	1	2.80	PO	52	Forage	0.010	0.067	
			28 cm			0.009	0.063	
					mean	0.010	0.065	0.074
Osceola, Nebraska, USA	1	2.80	PE	96	Forage	0.031	0.147	
2003 NC+6B50						0.027	0.136	
					mean	0.029	0.141	0.170
	1	2.81	PO	64	Forage	0.042	0.234	
			15–20 cm			0.040	0.232	
					mean	0.041	0.233	0.274
Colony, Oklahoma, USA	1	2.77	PE	106	Forage	0.055	0.405	
2003 Cherokee						0.061	0.510	
					mean	0.058	0.458	0.515
	1	2.82	PO	69	Forage	0.133	0.852	
			30–35 cm			0.108	0.682	
					mean	0.121	0.767	0.888
East Bernard, Texas,	1	2.79	PE	88	Forage	0.039	0.217	
USA 2003 DKS36-00						0.041	0.230	
					mean	0.040	0.223	0.263
	1	2.86	PO	65	Forage	0.079	0.443	
			25–28 cm			0.063	0.374	
					mean	0.071	0.408	0.480
Grand Island, Nebraska,	1	2.79	PE	100	Forage	0.016	0.122	
USA 2003 NC+6B50						0.017	0.117	
					mean	0.017	0.120	0.137
	1	2.80	PO	72	Forage	0.022	0.146	
			13–15 cm			0.025	0.176	
					mean	0.023	0.161	0.184
Dill City, Oklahoma,	1	2.89	PE	103	Forage	0.044	0.092	
USA 2003 Eclipse						0.048	0.095	
					mean	0.046	0.093	0.139
	1	2.80	PO	67	Forage	0.064	0.361	
			28–36 cm			0.078	0.407	
					mean	0.071	0.384	0.454
Claude, Texas, USA	1	2.81	PE	99	Forage	0.028	0.260	
2003 Y363						0.025	0.181	
					mean	0.027	0.220	0.247
	1	2.84	PO	86	Forage	0.042	0.272	
			15 cm			0.045	0.318	
					mean	0.043	0.295	0.338
Levelland, Texas, USA	1	2.86	PE	90	Forage	0.015	0.089	
2003 F-270E						0.015	0.089	
					mean	0.015	0.089	0.104

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SORGHUM FORAGE		kg ai/ha	at application			HEMA	EMA	(mg/kg)
	1	2.84	PO	69	Forage	0.013	0.027	
			15–28 cm			0.013	0.037	
					mean	0.013	0.032	0.045

Table 103 Residues in sorghum fodder following pre-emergent or early post-emergent application of an EC or a CS acetochlor formulation Moran (2004 MSL-18670). HEMA and EMA residues are expressed in acetochlor equivalents. Replicate samples.

Location, year, variety	N	Rate	Growth stage	DALA	Sample	Residue	(mg/kg)	Total
SORGHUM FODDER		kg ai/ha	at application			HEMA	EMA	(mg/kg)
Plains, Georgia, USA 2003 A571	1	2.77	PE	107 (142)	Stover	0.09	0.61	
						0.08	0.49	
					mean	0.08	0.55	0.63
	1	2.79	PO 15–20 cm	93 (128)	Stover	0.09	0.46	
						0.07	0.37	
					mean	0.08	0.41	0.49
Cord, Arkansas, USA 2003 Garst 5515	1	2.78	PE	123	Stover	0.02	0.12	
						0.03	0.14	
					mean	0.02	0.13	0.15
	1	2.80	PO 23 cm	104	Stover	0.02	0.09	
						0.02	0.09	
					mean	0.02	0.09	0.11
Carlyle, Illinois, USA 2003 KS 585	1	2.87	PE	133	Stover	< 0.01	< 0.02	
						< 0.01	< 0.02	
					mean	< 0.01	< 0.02	< 0.02
	1	2.89	PO 25 cm	104	Stover	0.01	0.03	
						0.01	< 0.02	
					mean	0.01	0.02	0.02
New Holland, Ohio, USA 2003 A571	1	2.73	PE	160 (177)	Stover	0.01	0.09	
						0.01	0.10	
					mean	0.01	0.09	0.10
	1	2.78	PO 25 cm	121 (138)	Stover	0.02	0.18	
						0.02	0.14	
					mean	0.02	0.16	0.18
York, Nebraska, USA 2003 Eclipse	1	2.79	PE	139 (144)	Stover	0.03	0.11	
						0.03	0.17	
					mean	0.03	0.14	0.17
	1	2.80	PO 13–15 cm	113 (118)	Stover	0.03	0.20	
						0.03	0.20	
					mean	0.03	0.20	0.23
Richland, Iowa, USA 2003 Dekaalb AS71	1	2.86	PE	140	Stover	0.01	0.05	
						0.01	0.06	
					mean	0.01	0.05	0.06
	1	2.80	PO 28 cm	110	Stover	0.01	0.06	
						0.01	0.05	
					mean	0.01	0.05	0.07
Osceola, Nebraska, USA 2003 NC+6B50	1	2.80	PE	139 (141)	Stover	0.03	0.23	
						0.03	0.22	
					mean	0.03	0.22	0.25
	1	2.81	PO 15–20 cm	107 (109)	Stover	0.04	0.32	
						0.04	0.32	
					mean	0.04	0.32	0.36
Colony, Oklahoma, USA 2003 Cherokee	1	2.77	PE	140	Stover	0.06	0.53	
						0.10	0.80	
					mean	0.08	0.66	0.74
	1	2.82	PO 30–35 cm	103	Stover	0.16	1.16	
						0.13	0.84	

Location, year, variety SORGHUM FODDER	N	Rate kg ai/ha	Growth stage at application	DALA	Sample	Residue (mg/kg) HEMA	EMA	Total (mg/kg)
					mean	0.14	1.00	1.14
East Bernard, Texas, USA 2003 DKS36-00	1	2.79	PE	116	Stover	0.04	0.17	
						0.04	0.18	
					mean	0.04	0.17	0.21
	1	2.86	PO 25–28 cm	93	Stover	0.06	0.23	
						0.07	0.25	
					mean	0.07	0.24	0.30
Grand Island, Nebraska, USA 2003 NC+6B50	1	2.79	PE	148 (152)	Stover	0.02	0.15	
						0.02	0.11	
					mean	0.02	0.13	0.15
	1	2.80	PO 13–15 cm	120 (124)	Stover	0.03	0.20	
						0.02	0.18	
					mean	0.02	0.19	0.21
Dill City, Oklahoma, USA 2003 Eclipse	1	2.89	PE	142	Stover	0.07	0.44	
						0.07	0.48	
					mean	0.07	0.46	0.54
	1	2.80	PO 28–36 cm	106	Stover	0.08	0.65	
						0.12	0.95	
					mean	0.10	0.80	0.90
Claude, Texas, USA 2003 Y363	1	2.81	PE	177	Stover	0.01	0.12	
						0.02	0.15	
					mean	0.01	0.13	0.15
	1	2.84	PO 15 cm	164	Stover	0.02	0.20	
						0.02	0.19	
					mean	0.02	0.19	0.21
Levelland, Texas, USA 2003 F-270E	1	2.86	PE	126	Stover	0.02	0.08	
						0.02	0.08	
					mean	0.02	0.08	0.11
	1	2.84	PO 15–28 cm	105	Stover	0.02	0.21	
						0.02	0.21	
					mean	0.02	0.21	0.23

Table 104 Residues in cotton gin by-products following application of a micro-encapsulated (CS) acetochlor formulation (Hay *et al.* 2008 MSL-20718). HEMA and EMA residues are expressed in acetochlor equivalents. Replicate samples.

Location, year, variety Gin by-products	N	Rate kg ai/ha	Growth stage at application	DALA	Sample	Residue (mg/kg) HEMA	EMA	Total (mg/kg)
Proctor, Arkansas, USA 2007 ST4554B2RF	1	3.37	16 nodes, midbloom	74	Cotton gin by-products	0.34	1.28	
						0.38	1.57	
					mean	0.36	1.43	1.78
	1	3.37	8 nodes	107	Cotton gin by-products	0.02	< 0.06	
						0.02	< 0.06	
					mean	0.02	< 0.06	< 0.08
	2	1.67	Pre-plant	107	Cotton gin by-products	0.02	< 0.06	
		1.68	8 nodes			0.02	< 0.06	
					mean	0.02	< 0.06	< 0.08
Newport, Arkansas, USA 2007 DP 143 B2RF	1	3.41	BBCH 65	83	Cotton gin by-products	0.20	1.21	
						0.24	1.68	
					mean	0.22	1.45	1.67
	1	3.35	BBCH 18	122	Cotton gin	0.02	< 0.06	

Location, year, variety Gin by-products	N	Rate kg ai/ha	Growth stage at application	DALA	Sample	Residue (mg/kg) HEMA	EMA	Total (mg/kg)
					by-products	0.02	0.07	
					mean	0.02	0.06	0.08
	2	1.69	Pre-plant	122	Cotton gin	0.01	< 0.06	
		1.67	BBCH 18		by-products	0.01	< 0.06	
					mean	0.01	< 0.06	< 0.07
Uvalde, Texas, USA	1	3.34	BBCH 65	84	Cotton gin	0.03	0.24	
2007 DP 143 B2RF					by-products	0.04	0.33	
					mean	0.04	0.28	0.32
	1	3.34	BBCH 18	119	Cotton gin	0.01	< 0.06	
					by-products	0.01	< 0.06	
					mean	0.01	< 0.06	< 0.07
	2	1.68	Pre-plant	119	Cotton gin	< 0.01	< 0.06	
		1.67	BBCH 18		by-products	< 0.01	< 0.06	
					mean	< 0.01	< 0.06	< 0.07
LaPryor, Texas, USA	1	3.31	BBCH 65	65	Cotton gin	0.09	1.09	
2007 Delta Pine 117			mid bloom		by-products	0.08	0.94	
B2RF					mean	0.09	1.01	1.10
	1	3.36	BBCH 18–19	100	Cotton gin	0.02	0.11	
					by-products	0.02	0.11	
					mean	0.02	0.11	0.13
	2	1.67	Pre-plant	100	Cotton gin	0.01	0.06	
		1.66	BBCH 18–19		by-products	0.01	< 0.06	
					mean	0.01	< 0.06	< 0.07
Levelland, Texas, USA	1	3.36	BBCH 63	70	Cotton gin	0.78	3.78	
2007 FM 9063B2F					by-products	0.69	3.66	
					mean	0.74	3.72	4.45
				76	Cotton gin	0.29	1.65	
					by-products	0.33	1.74	
						0.38	2.02	
						0.37	2.11	
					mean	0.34	1.88	2.22
				83	Cotton gin	0.33	1.87	
					by-products	0.32	1.98	
					mean	0.32	1.92	2.24
				91	Cotton gin	0.42	2.64	
					by-products	0.43	2.79	
					mean	0.43	2.72	3.14
	1	3.36	BBCH 18	112	Cotton gin	0.07	0.20	
					by-products	0.09	0.21	

Location, year, variety Gin by-products	N	Rate kg ai/ha	Growth stage at application	DALA	Sample	Residue (mg/kg) HEMA	EMA	Total (mg/kg)
					mean	0.08	0.20	0.28
	2	1.69	Pre-plant	112	Cotton gin	0.06	0.16	
		1.66	BBCH 18		by-products	0.07	0.17	
					mean	0.07	0.17	0.23
Wolfforth, Texas, USA	1	3.32	BBCH 63	86	Cotton gin	0.32	2.14	
2007 ST 45357 B2RF					by-products	0.32	2.19	
					mean	0.32	2.17	2.48
	1	3.50	BBCH 19	121	Cotton gin	0.10	0.29	
					by-products	0.09	0.31	
					mean	0.09	0.30	0.39
	2	1.71	Pre-plant	121	Cotton gin	0.06	0.22	
		1.69	BBCH 19		by-products	0.06	0.23	
					mean	0.06	0.23	0.29
Claude, Texas, USA	1	3.36	BBCH 65	64	Cotton gin	0.14	1.58	
2007 NG3550					by-products	0.15	1.79	
					mean	0.14	1.69	1.83
	1	3.36	BBCH 18	106	Cotton gin	0.02	0.18	
					by-products	0.03	0.23	
					mean	0.03	0.21	0.23
	2	1.68	Pre-plant	106	Cotton gin	0.02	0.12	
		1.69	BBCH 18		by-products	0.02	0.14	
					mean	0.02	0.13	0.15

Fate of residues in processing

Peanuts

Peanuts from two trial locations were processed. Following receipt and inventory, the peanuts were placed into frozen storage until preparation for processing. The untreated and treated peanuts were oven dried (54–71 °C) to 7–12% moisture, then cleaned by aspiration and screening. The unshelled peanuts were then fed into a huller to crack the hull and liberate the nutmeat. The nutmeat was separated from the hulls by aspiration. Fractions of hull material and nutmeat were collected and placed in frozen storage. If necessary, the nutmeat material for processing was oven dried (54–71 °C) to 7–10% moisture. Following drying the nutmeat was separated into two portions (dry roasted peanuts/peanut butter and peanut meal/peanut oil) for further processing.

Peanut oil and meal: Kernel moisture was adjusted to 12% and the material heated to 85–104 °C and pressed in an expeller to remove the majority of the oil. The press cake was milled and solvent extracted with hexane (49–60 °C) and after 30 minutes the miscella was drained and the process repeated (2×). Solvent was removed from the meal by forcing warm air through. Hexane was removed from the miscella by vacuum evaporation (91–96 °C) to obtain crude oil. The free fatty acid content of the crude oil was determined and an appropriate amount of NaOH added with mixing, initially at 20–24 °C and then at 60–67 °C after which the mixture was allowed to settle for one hour prior to refrigeration for 12 hours. The neutralised refined oil was decanted from the soapstock. The refined oil was bleached by adding activated bleaching earth,

heating to 85–100 °C for 10–15 minutes and filtering. The bleached oil was heated under vacuum to 220–230 °C for 30 minutes and cooled to 135–150 °C before addition of 0.5% citric acid. The oil was cooled and filtered to produce refined bleached deodorised oil.

Dry roasted nuts were prepared by heating raw shelled peanuts 160–171 °C for 2–7 minutes.

Oil roasting: raw shelled nuts were submerged in a deep fryer containing peanut oil heated to 171–182 °C for 2 minutes and drained.

Peanut butter: Skins were removed from dry roasted nuts, which were chopped and fed through a peanut butter machine. Peanut oil and salt were added to the material exiting the machine.

Table 105 Residues in peanut processed commodities (Mueth and Foster 2012 MSL-0024197). HEMA and EMA residues are expressed in acetochlor equivalents.

Location	Rate g ai/ha	DALA	Sample	HEMA	EMA	Total	PF
Seven Springs, North	1×3.37	126	Peanut	0.035	0.034	0.069	
Carolina, USA 2011			RAC	0.039	0.038	0.077	
Champs			mean	0.037	0.036	0.073	
			Dry roasted	0.024	0.035	0.059	
			nuts	0.026	0.037	0.063	
34.5 kg batch			mean	0.025	0.036	0.061	0.8
			Meal	0.067	0.079		
				0.072	0.084		
			mean	0.070	0.081	0.151	2.1
			Peanut	0.022	0.036	0.058	
			butter	0.028	0.037	0.065	
			mean	0.025	0.036	0.061	0.9
			RBD oil	< 0.009	< 0.009		
				< 0.009	< 0.009		
			mean	< 0.009	< 0.009	< 0.009	< 0.3
Lenox, Georgia, USA	1×3.32	106	Peanut	0.017	0.018	0.035	
2011 06-GA			RAC	0.017	0.022	0.039	
			mean	0.017	0.02	0.037	
			Dry roasted	0.016	0.023	0.039	
34.5 kg batch			nuts	0.016	0.024	0.04	
			mean	0.016	0.023	0.039	1.1
			Meal	0.050	0.068		
				0.048	0.071		
			mean	0.049	0.070	0.118	3.2
			Peanut	0.019	0.024	0.043	
			butter	0.018	0.03	0.048	
			mean	0.018	0.027	0.046	1.2
			RBD oil	< 0.009	< 0.009		
				< 0.009	< 0.009		
			mean	< 0.009	< 0.009	< 0.009	< 0.5

Soya bean

Harvested soya beans from one trial were processed into refined oil, soya bean meal, and hulls. Samples were processed to simulate commercial practice as closely as possible. Samples were dried in an oven at 54–71 °C until the moisture content was 7–10%. Light impurities were removed by aspiration and the samples screened to separate large and small foreign particles. The cleaned soya beans were fed into a roller mill to crack the hull and release the kernel. The hulls and kernels were separated by aspiration. The kernels were adjusted to a moisture content of 13.5% and heated to 71–79 °C, flaked (0.02–0.03 cm) and extruded into collets by direct steam injection and compression (exit temperature 93–121 °C). After extrusion the collects were dried at 66–82 °C for 30–40 minutes before being immersed in hexane 49–60 °C for 30 minutes, a process repeated a further 2×. The

solvent extracted meal was heated to 99–104 °C. The hexane was removed from the miscella under vacuum (91–96 °C) and filtered. An appropriate amount of NaOH was added and the oil mixed for 90 minutes at 20–24 °C and then 20 minutes at 63–67 °C. The neutralised oil was centrifuged to separate the refined oil from the soapstock.

Total HEMA and EMA residues in soya bean were reduced in refined oil and hulls relative to the unprocessed seeds by processing factors of 0.11 and 0.72, respectively. Total HEMA and EMA residues were slightly increased in meal relative to the unprocessed seeds with a processing factor of 1.2. The results show that no concentration occurred in soya bean refined oil or hulls, but a slight concentration of residues did occur in soya bean meal.

Table 106 Residues in soya bean processed commodities (Hay *et al.* 2008 MSL-20719). HEMA and EMA residues are expressed in acetochlor equivalents.

Location	Rate g ai/ha	DALA	Sample	HEMA	EMA	Total	PF
Carlyle, Illinois, USA	1×8.95	205	Seed for	0.299	0.893	1.192	
2007 NK 37N4			processing	0.291	0.871	1.163	
			mean	0.295	0.882	1.177	-
			Refined	0.063	0.064	0.127	
10.4 kg batch			oil	0.067	0.062	0.129	
			mean	0.065	0.063	0.128	0.11
			Soya bean	0.361	1.106	1.467	
			meal	0.349	1.04	1.389	
			mean	0.355	1.073	1.428	1.2
			Hulls	0.189	0.631	0.82	
				0.218	0.662	0.88	
			mean	0.204	0.647	0.85	0.72

Sugar beet

Sugar beets from two trials were processed. Following receipt and inventory, the sugar beets were placed into frozen storage until preparation for processing. Samples were weighed and cleaned and a representative sample of the sugar beet RAC was collected. The cleaned beets were then chopped, diffused at 68–74 °C, and the raw juice was sieved to remove pieces of beet from the juice.

Diffused material was dewatered with a hydraulic press and beet pulp was dried to ≤ 15% moisture. The beet pulp fraction was then placed in freezer storage. Raw juice was mixed and heated, and the pH was adjusted to separate the mud and juice by centrifugation and filtration. After concentration, the thick juice was seeded with sugar to begin the crystallization process. Sugar and molasses were separated, centrifuged, and steam was added to facilitate separation. Sugar and molasses fractions were placed in freezer storage.

Table 107 Residues in sugar beet processed commodities (Mueth and Foster 2012 MSL-24198). HEMA and EMA residues are expressed in acetochlor equivalents.

Location	Rate g ai/ha	DALA	Sample	HEMA	EMA	Total	PF
York, Nebraska, USA			RAC	0.005	0.006		
2011 Hilleshog 9093 RR				0.005	0.006		
			mean	0.005	0.006	0.011	
			Dried	0.008	0.016		
			pulp	0.009	0.017		
53.1 kg batch			mean	0.008	0.016	0.025	2.3
			Molasses	0.021	0.025		
				0.022	0.025		
			mean	0.022	0.025	0.046	4.2
			Refined	0.002	0.003		
			sugar	0.002	0.003		
			mean	0.002	0.003	0.005	0.5
Rupert, Idaho, USA 2011			RAC	0.003	0.013		

Location	Rate g ai/ha	DALA	Sample	HEMA	EMA	Total	PF
Crystal RR929				0.003	0.014		
			mean	0.003	0.013	0.016	
			Dried	0.003	0.011		
			pulp	0.003	0.011		
63.8 kg batch			mean	0.003	0.011	0.014	0.9
			Molasses	0.005	0.013		
				0.005	0.014		
			mean	0.005	0.014	0.018	1.1
			Refined	ND	ND		
			sugar	ND	ND		
			mean	< 0.002	< 0.002	< 0.004	< 0.25

Sorghum

Harvested sorghum grain from two sites was processed into cleaned grain, flour, and bran. Samples were processed to simulate commercial practice as closely as possible.

Grain was dried to target moisture content, aspirated to remove light impurities, screened to clean the seed prior to decortication to produce decorticated grain, bran and grits (small grits and large grits). The decorticated grain was further processed to produce flour.

Grain sorghum was dried (if necessary) in an oven at 54–71 °C to a moisture content of 10–13%. The light impurities were separated using an aspirator. After aspiration, the sample was screened in a two screen cleaner to separate large and small foreign particles (screening) from the grain sorghum.

The cleaned grain was milled in an abrasion mill to remove most of the bran from the seed. Bran was separated using a sample sifter equipped with a 12 TMS screen. The grain was decorticated until approximately 15% or more of the bran passed through the 12 TMS screen. The material on top was again classified in the sample sifter utilizing 8 TMS and 10 TMS screens. The decorticated grain was collected from the top of the 8 TMS screen. Large grits passed through the 8 TMS and were collected on top of the 10 TMS screen. Small grits passed through the 8 and 10 TMS screens and were collected in the pan.

The decorticated grain was ground in a mill fitted with a 0.31 cm, 0.17 cm or similar size screen. The through product wais then reground in the mill with a 0.015 cm. Decorticated grain was milled into flour. Ground material was sifted with a sample sifter equipped with a US 34 screen.

Table 108 Residues in sorghum processed commodities (Moran 2004 MSL-18670). HEMA and EMA residues are expressed in acetochlor equivalents.

Location	Rate g ai/ha	DALA	Sample	HEMA	EMA	Total	PF
York, Nebraska,	2.80	112	Grain	0.014	0.019	0.033	
USA 2003 Eclipse			Cleaned grain	0.012	0.018	0.030	
32 kg batch			Flour	nd	0.010	0.010	0.33
			Bran	0.052	0.049	0.101	3.1
Dill City, Oklahoma,	2.80	97	Grain	0.007	0.010	0.017	
USA 2003 Eclipse			Cleaned grain	0.009	0.012	0.021	
39 kg batch			Flour	< 0.01	< 0.01	< 0.01	< 0.59
			Bran	0.040	0.052	0.092	4.4

Cotton

Undelinted seed were processed into hulls, cottonseed meal, and refined oil. Samples were processed to simulate commercial practice as closely as possible. Prior to ginning, the seed cotton was cleaned with an attached stick extractor to remove gin trash (gin by-products). Seed was saw-ginned to remove most of the lint (ginned cottonseed). With approximately 11–15% remaining lint, the undelinted cottonseed was saw delinted to produce delinted cottonseed (ca. 3% lint remaining). The delinted seed was mechanically cracked on a roller mill followed by screening to separate the hulls from the kernel. The kernel material was processed into meal and crude oil by heating kernels to 79–91 °C for 15–30 minutes, after which the kernel material was flaked (roll gap 0.02 cm) and the flaked material fed into a continuous processor (extruder). As material moved through the extruder steam was injected directly on the product. The maximum temperature of the exiting collets was 118 °C. Collets were dried in an oven at 65–82 °C for 30–40 minutes and then solvent extracted in batches (hexane 49–60 °C). After 30 minutes the hexane was drained, fresh hexane added and the extraction process repeated, three times in total. After final draining, the spent collets (meal) were heated to 99–104 °C to remove residual hexane. The miscella (crude oil + hexane) was passed through a vacuum evaporator (91–96 °C) to remove the hexane and filtered prior to refining. Crude oil and NaOH were mixed at 20–24 °C for 15 minutes, the temperature increased to 63–67 °C for a further 12 minutes. The neutralised oil was centrifuged to remove the solids (soapstock) and the refined oil decanted and vacuum filtered.

The processing factors range from 0.083 to 0.438, indicating that no concentration occurred in processed fractions.

Table 109 Residues in cotton seed processed commodities (Hay *et al.* 2008 MSL-20718). HEMA and EMA residues are expressed in acetochlor equivalents.

Location	Rate g ai/ha	DALA	Sample	HEMA	EMA	Total	PF
Uvalde, Texas, USA 2007	1.66	218	Undelinted	0.007	0.045	0.052	
DP 143 B2RF	8.89 [a]		seed for	0.006	0.037	0.044	
			processing				
52 kg batch			mean	0.007	0.041	0.048	
			Hulls	0.002	0.01	0.011	
				0.003	0.013	0.016	
			mean	0.002	0.012	0.014	0.29
			Cottonseed	0.004	0.015	0.02	
			meal	0.006	0.017	0.023	
			mean	0.005	0.016	0.021	0.44
			Refined	0.001	0..003	0.004	
			Oil	0.001	0.002	0.003	
			mean	0.001	0.003	0.004	0.08

[a] 1×EC pre-plant + 1×CS (1st flower)

Sunflower

The whole sunflower samples were dried in an oven at 54–71 °C until a final moisture content of 7–10% was reached. The light impurities were separated using an aspirator. After aspiration, the sample was screened in a two screen cleaner. Large and small foreign particles (screenings) were separated from the sunflower.

The whole sunflower was fed into a disc mill to crack the hull and liberate the kernel material. After hulling, the material was passed through the aspirator to separate the hull and kernel material (some whole-seed and kernel remain after separation).

Kernel material was moisture conditioned to 12%, heated to 88–104 °C and pressed in an expeller to liberate a portion of the crude oil. The press cake from the expeller was placed in stainless steel tanks and submerged in 49–60 °C solvent (hexane). After 30 minutes, the hexane was drained and fresh hexane added to repeat the cycle two more times. The final two washes

were for 15 minutes each. After the final draining, warm air was forced through the solvent extracted press cake (meal) to remove any residual hexane.

The miscella (crude oil and hexane) was passed through a recovery unit to separate the crude oil and hexane. Crude oil was heated to 73–90 °C for hexane removal.

Crude oil recovered from the expeller and solvent extraction was combined and refined. After refining, the refined oil and soap stock were separated.

A subsample of the seed was taken and processed into sunflower meal and oil. Samples were processed to simulate commercial practice as closely as possible. The processing factor was 1.44 for sunflower meal, indicating concentration may have occurred. The processing factor for sunflower oil was 0.22, indicating no concentration occurred

Table 110 Residues in sunflower processed commodities (Anderson 1996 RJ2568B). HEMA and EMA residues are expressed in acetochlor equivalents.

Location	Primary crop Rate kg ai/ha	DAA	Sample	acetochlor	HEMA	EMA	Total	PF
South Dakota, USA 1996	3.4	477	Seed	< 0.01	0.17	0.03	0.20	
Legend LSF146			Seed [a]	< 0.01	0.13	0.03	0.16	
			Meal	< 0.01	0.18	0.05	0.23	1.44
22 kg batch			Oil	< 0.01	< 0.02	< 0.02	< 0.04	0.22

[a] Bulk pre-processing

Oats

The oat samples were dried in an oven with a temperature range of 85–93 °C to a moisture content of 7.4–9.9%. The light impurities were separated using an aspirator, after which the sample was screened in a two screen cleaner. Large and small screenings were separated from the oats. The cleaned oats were passed through a mill to dehull the oat sample. During dehulling, the groats (hulled oats) and hull were separated using the aspiration system on the mill. The groats and unhulled seed were separated using a gravity table until the amount of unhulled seed was less than 5% (visual inspection). The groats and fine material (oat feed) were separated with a sample sifter equipped with a US 24 screen. A fraction of the groats was ground in a mill and sifted in a sifter equipped with a US 34 screen. The groats were ground until 80–90% of the material passed through the screen. Resulting fractions were bran (top of the screen) and flour (through the screen).

There was no residue concentration in any of the processed commodities.

Table 111 Residues in oat processed commodities (Manning 1997 MSL-14118). HEMA and EMA residues are expressed in acetochlor equivalents.

Location	Primary crop Rate kg ai/ha	DAA	Sample	HEMA	EMA	Total	PF
Monmouth Illinois USA	3.4	425	Grain initial	< 0.018	0.034	< 0.052	
1996 Ogle			Grain final	0.022	0.033	0.055	
			Hulls	< 0.018	< 0.017	< 0.035	
> 22 kg batches			Flour	< 0.018	< 0.017	< 0.035	
			Groats	< 0.018	< 0.017	< 0.035	
Jerseyville Illinois, USA	3.4	414	Grain initial	< 0.018	< 0.017	< 0.035	
1996 Ogle			Grain final	< 0.018	< 0.017	< 0.035	
			Hulls	< 0.018	0.028	< 0.046	
> 22 kg batches			Flour	< 0.018	< 0.017	< 0.035	
			Groats	< 0.018	< 0.017	< 0.035	

Table 112 Summary of acetochlor processing factors

	Processed Fraction	Processing Factor
Soya bean	Refined oil	0.11
	Soya bean meal	1.2
	Hulls	0.72
Sugar beet	Dried pulp	2.3, 0.9
	Molasses	4.2, 1.1
	Refined sugar	0.5, < 0.25
Sorghum	Cleaned grain	–
	Flour	0.33, < 0.59
	Bran	3.1, 4.4
Cotton	Hulls	0.29
	Cottonseed meal	0.44
	Refined oil	0.08
Peanut	Dry roasted nuts	0.8, 1.1
	Meal	2.1, 3.2
	Peanut butter	0.9, 1.2
	Refined oil	< 0.3, < 0.5
Sunflower	Meal	1.4
	Oil	0.22

PFs are based on combined EMA- and HEMA-class metabolites

PRIMARY FEED COMMODITIES OF PLANT ORIGIN

Fate on processing

Livestock feeding studies

Dairy cow feeding study

The transfer of acetochlor metabolites from feed to tissues and milk of dairy cows was studied by Wilson (1982, MSL-2285). A synthetic mixture representing the four classes of metabolites was used for dosing. The four metabolites were *tert*-hydroxy (17) and the sodium salts of *tert*-sulfonic acid (7), *tert*-oxanilic acid (2) and *tert*-sulfinylacetic acid (3) and were present in the dose material in equal parts by weight.

A mixture of the four acetochlor metabolites was administered orally to four groups of three Holstein cattle (1.9–5.8 years old; 441–598 kg bw) by gelatine capsule for 28 days. Mean daily feed consumption for the dose groups during the exposure period were 21.4–22.6 kg DM (hay, *ad libitum* and 6.3 kg/day protein concentrate). Mean daily milk yield for the dose groups during the exposure period were 15.6 to 17.3 kg/cow/day. Based on mean daily feed consumption, the exposure was equivalent to 5, 15 and 50 ppm in the feed. 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 acetochlor metabolites. Muscle, liver, kidney and fat samples were collected at sacrifice 22–24 hours after the last dose; or 28 days in the case of the depuration animals. 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. Samples were analysed using the analytical method "Analytical Residue Method for Four Metabolites of Acetochlor in Milk and Beef Tissues". Milk, muscle, liver, kidney, and fat were analysed for residues of acetochlor metabolites containing the EMA moiety.

The analytical method determines of compounds hydrolysable to EMA. The procedure consists of extraction of the beef matrix with solvents (90% CH_3CN/H_2O for milk, muscle, liver and kidney; hexane followed by 90% CH_3CN/H_2O), centrifugation, filtration, and evaporation. The extracted residue is digested first in acid (12 N H_2SO_4, reflux 20 min) to remove the

ethoxymethyl group, then in base (50% NaOH) followed by distillation to recover the liberated EMA. The recovered aniline is quantified by GC-NPD. Residues are expressed as acetochlor equivalents. The LOQ is 0.02 mg/kg for tissues and milk.

Milk samples taken throughout the 28-day dosing period and muscle and fat samples taken at sacrifice time showed residues < 0.02 mg/kg. Kidneys showed a maximum residue of 0.09 mg/kg at the 50 ppm dosing level, 0.04 mg/kg at the 15 ppm dosing level and < 0.02 mg/kg at the 5 ppm dosing level. Residues of 0.02 mg/kg in liver were seen only at the 50 ppm dosing level. All tissues and milk from animals allowed a 28-day withdrawal period were < 0.02 mg/kg.

Table 113 Concurrent recovery results for acetochlor metabolites for the dairy cow feeding study

Fortification (mg/kg)	Average EMA % recovery (number of replicates)				
	Milk	Muscle	Liver	Kidney	Fat
0.02	76.5 (36)	69.8 (4)	70.0 (6)	73.1 (8)	78.3 (4)
0.2	–	79.0 (1)	66.0 (1)	84.0 (1)	74.5 (1)
Average	76.5 (36)	71.6 (5)	69.5 (7)	73.7 (9)	77.5 (5)

Table 114 Residues of acetochlor metabolites in tissues of cows dosed with a mixture of four metabolites

	Dose group (ppm)	EMA (mg/kg), expressed as acetochlor	
		28 day dosing	28 day dosing + 28 day withdrawal
Milk (–1, 1, 4, 8, 14, 21, 28 d) [a]	50	< 0.02	< 0.02
Muscle	50	< 0.02 (< 0.02 (3))	< 0.02
Fat	50	< 0.02 (< 0.02 (3))	< 0.02
Liver	15	< 0.02 (< 0.02 (3))	NA
	50	0.02 (0.02 (3))	< 0.02
Kidney	5	< 0.02 (< 0.02 (3))	NA
	15	0.03 (0.02 0.04 0.04)	NA
	50	0.07 (0.09 0.06 0.06)	< 0.02

[a] Individual samples of milk from the days listed were analysed separately and were all < 0.02 mg/kg.

The method converts the metabolites to the common moiety EMA.

NA = not analysed

Laying hen feeding study

Wilson (1982 MSL-2287) studied the transfer of acetochlor metabolites to chicken tissues and eggs. A residue feeding study was conducted in laying hens (White Leghorn, 20–24 weeks old, 1.4–1.84 kg bw, lay efficiency 0.98, 0.96 and 0.96 eggs/d) with acetochlor metabolites to provide a basis for establishing tolerances for acetochlor in eggs and chicken tissues. Four representative metabolites of acetochlor, equal parts by weight, dissolved in absolute ethanol were fed by oral gavage to 100 laying hens in a single daily oral dose. The four metabolites were *tert*-hydroxy (17) and the sodium salts of *tert*-sulfonic acid (7), *tert*-oxanilic acid (2) and *tert*-sulfinylacetic acid (3). The 100 chickens were divided into four dosing groups (control, 5, 15, and 50 ppm) and dosed once daily for 28 days. Mean feed consumption was 110 g/d (actual dosing period, 117, 114, 115 g/d but 90% DM so 130, 128 128 g/d). Mean laying efficiency for the three dose groups were 98%, 96% and 96% respectively. After the dosing, one-half of the birds were sacrificed for tissue samples while the remaining hens were allowed a 28 day withdrawal period prior to sacrifice.

The analytical method, "Analytical Residue Method for Four Metabolites of Acetochlor in Eggs and Chicken Tissues" and is essentially the same method as used for the lactating cow transfer study. The residue method converts the metabolites to the common EMA moiety. Tissues from all three dose levels were analysed at one time. The tissues from all the birds in each treatment level group were composited. This was necessary to ensure a large enough analytical sample for some of the tissues.

Table 115 Concurrent recovery results for acetochlor metabolites for the laying hen feeding study

Matrix	Spiking level (mg/kg)	Average Recovery (%)	Standard Deviation	RSD (%)	Replicates
Egg	0.02	82.1	6.8	8.2	22
Muscle	0.02	75.8	5.4	7.1	4
Liver	0.02	72.8	4.5	6.2	4
Kidney	0.02	79.8	4.3	5.4	4
Fat	0.02	77	4.7	6.1	4

The LOQ was 0.02 mg/kg for all tissues except kidneys, for which the LOQ was 0.05 mg/kg due to the small sample size. Egg samples collected throughout the 28-day dosing period and muscle, liver, and fat samples taken at sacrifice time showed non-detectable residues (< 0.02 mg/kg) from all feeding levels. Kidney samples also showed non-detectable residues (< 0.05 mg/kg). All tissues and eggs from birds allowed a 28-day withdrawal period also had non-detectable residues from all feeding levels.

NATIONAL RESIDUE DEFINITIONS

Canada Acetochlor

China Acetochlor

Japan Acetochlor

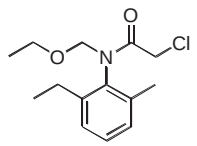
Korea Acetochlor

USA Acetochlor and its HEMA- and EMA-producing metabolites, calculated as parent equivalents

APPRAISAL

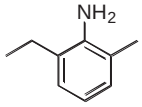
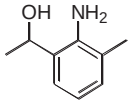
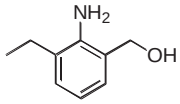
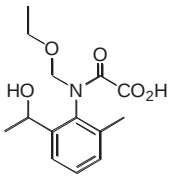
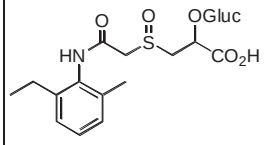
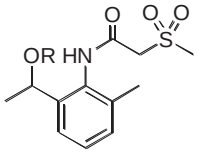
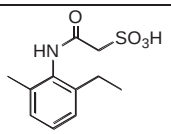
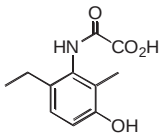
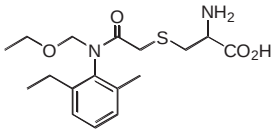
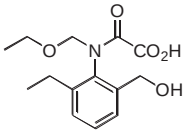
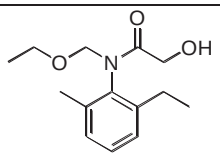
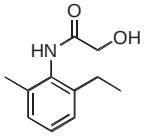
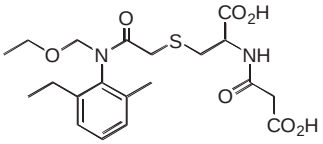
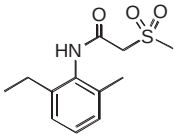
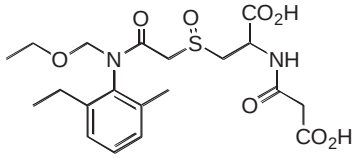
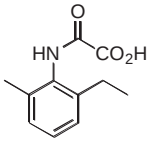
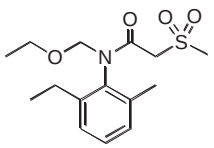
Acetochlor is a selective herbicide which, after application, is absorbed mainly by the shoots of germinating plants, and to some extent, by roots. Acetochlor is used as a pre-emergence or early post-emergence soil-applied herbicide. Acetochlor controls annual grasses and broadleaf weeds, germinating from seeds; however, its action against perennial weeds is very limited. At the Forty-sixth Session of the CCPR (2014), it was scheduled for the evaluation as a new compound by 2015 JMPR.

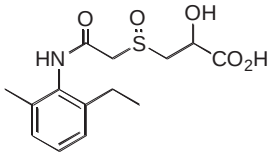
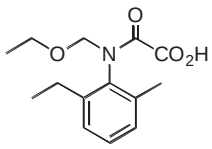
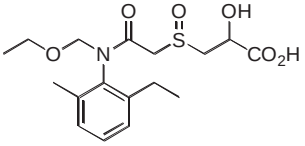
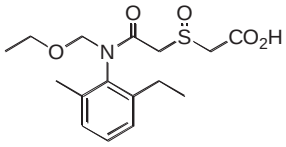
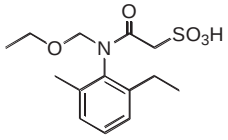
The Meeting received information on the metabolism of acetochlor in maize, soya beans and cotton, lactating goats and cows, laying hens, follow crops, methods of residue analysis, freezer storage stability, GAP information, supervised residue trials on maize (forage, grain, stover and silage), sweet corn (forage, kernels plus cob with husks removed, stover and silage), cotton (gin by-products and seed), sorghum (grain, forage and stover), soya bean (meal and seed), sugar beet (dried pulp, roots, tops, sugar and molasses), peanuts (hay and meal) and livestock transfer studies (lactating cows and laying hens).



Acetochlor is 2-chloro-*N*-(ethoxymethyl)-*N*-(2-ethyl-6-methylphenyl)acetamide

Metabolites referred to in the appraisal were addressed by their common names with the corresponding aniline metabolite class (EMA, HEMA, HMEA or OH) indicated in brackets.

2-ethyl-6-methyl aniline = EMA		2-(1-hydroxyethyl)-6-methyl aniline = HEMA	
2-ethyl-6-hydroxymethyl aniline = HMEA			
1-hydroxyethyl *tert*-oxanilic acid (HEMA class)		*sec*-sulfinyllactic acid glucose conjugate (EMA class)	
1-hydroxyethyl-*sec*-methylsulfone glucosylsulfate conjugate (HEMA class)	 R=glucose sulfate	*sec*-sulfonic acid (EMA class)	
5-hydroxy-*sec*-oxanilic acid (OH class)		*tert*-cysteine (EMA class)	
hydroxymethyl-*tert*-oxanilic acid (HMEA class)		*tert*-hydroxyacetochlor (EMA class)	
sec-hydroxyacetochlor (EMA class)		*tert*-malonylcysteine (EMA class)	
sec-methylsulfone (EMA class)		*tert*-malonylcysteine sulfoxide (EMA class)	
sec-oxanilic acid (EMA class)		*tert*-methylsulfone (EMA class)	

sec-sulfinyllactic acid (EMA class)		*tert*-oxanilic acid (EMA class)	
tert-sulfinyllactic acid (EMA class)		*tert*-sulfinylacetic acid (EMA class)	
tert-sulfonic acid (EMA class)			

Plant metabolism

Acetochlor is typically used for three different situations:

- Incorporation into the soil prior to planting the crop (PP)
- As a broadcast spray to weeds and bare soil after seeding but prior to crop emergence (PE)
- As a broadcast spray to weeds and the growing crop, i.e. post-emergence (PO).

The Meeting received plant metabolism studies with acetochlor following pre-plant, pre- and post-emergent applications to maize (corn), cotton and soya bean.

Maize

The metabolism of [^{14}C-U-phenyl]-acetochlor in maize grown outdoors was studied following either a pre-emergence (PE) application immediately after seeding or post-emergence after allowing the corn plants to grow to a height of 66–71 cm (growth stage V6 to V7, i.e., 6–7 leaves fully emerged) before spraying. The effective treatment rates were 3.6 kg ai/ha for the PE application and 3.5 kg ai/ha for the PO application.

Total radioactive residues in PE forage, grain and stover were 0.67, 0.04 and 1.84 mg equiv/kg while those in PO forage, grain and stover were higher at 3.44, 0.022 and 6.41 mg equiv/kg respectively.

Solvent (CH_3CN/H_2O) extracted ≥ 79% of the TRR present in immature plants, forage and stover samples. Extraction of ^{14}C present in grain was lower at 58–63% TRR. The majority of the ^{14}C present in the solids after extraction were associated with natural products, especially starch, protein, lignin and hemicellulose. A large number of metabolites were detected in the solvent extracts but not unchanged acetochlor. There were notable differences in the pattern of metabolites observed following PE compared to PO application.

The metabolites identified in PO forage and stover primarily resulted from initial glutathione conjugation of acetochlor followed by oxidation to give sulfoxide-type metabolites. Only one compound exceeded 10% of TRR: *tert*-sulfinyllactic acid was observed at 12.6% TRR (0.43 mg equiv/kg) in forage and 11.3% of TRR (0.72 mg equiv/kg) in stover. Two other metabolites exceeded 0.1 mg equiv/kg: *sec*-sulfinyllactic acid and *sec*-sulfinyl lactic acid glucose conjugate.

In contrast, in PE maize the compounds detected resulted largely from the uptake of soil metabolites to give oxanilate-type metabolites. None of the individual components exceeded

10% of TRR in immature plant, forage or stover. The major component was 5-hydroxy *sec*-oxanilic acid present at levels of 8.4% (0.099 mg equiv/kg) 6.2% (0.042 mg equiv/kg) and 4.3% (0.080 mg equiv/kg) TRR in immature plants, forage and stover respectively.

In grain from PE or PO application, no individual compound exceeded 10% of TRR and no discrete component characterized by chromatography exceeded 0.001 mg equiv/kg.

Compounds containing an intact phenyl ring can be classified according to the aniline that would be generated on base hydrolysis. Non-hydroxylated metabolites give EMA, those hydroxylated at the 1-position of the ethyl side-chain give HEMA, those at the hydroxylated at the methyl side-chain HMEA and those hydroxylated at the 3, 4 or 5 positions of the phenyl ring could be classed as "OH" anilines. The major aniline metabolite class observed in maize (PE and PO) is EMA followed by OH.

Soya bean

The metabolism of [^{14}C-U-phenyl]-acetochlor in soya beans grown outdoors following either a pre-plant (PP) or post-emergence (PO) application was studied. The PP application was made to the soil (loamy sand) 45 days before seed planting while the PO application was made to a second group of plants 42 days after planting seed when the plants were approximately at the R1–R2 growth stage (beginning flowering to full flowering). The application rates were 3.5 kg ai/ha for the PP and 3.7 kg ai/ha for the PO application.

Levels of radioactivity were higher in PO treated plants compared to PP application. TRRs were 1.67 and 11.4 mg equiv/kg in PP and PO forage, respectively; 3.48 and 57.7 mg equiv/kg in PP and PO hay; and 0.175 and 0.192 mg equiv/kg in PP and PO seed.

Solvent (CH_3CN/H_2O) extracted ≥ 86% of the TRR present in forage and hay samples. Extraction of ^{14}C present in grain was lower at 59–80% TRR.

As was the case with maize, a large number of metabolites were detected in the solvent extracts but not unchanged acetochlor. There were also notable differences in the patterns of metabolites observed following PP compared to PO application.

Like maize, the metabolites identified in PO soya bean forage and hay primarily resulted from initial glutathione conjugation of acetochlor followed by oxidation to give sulfoxide-type metabolites. Five compounds exceeded 10% of TRR: *tert*-cysteine (forage 39% TRR, 4.45 mg equiv/kg), *tert*-malonylcysteine (forage and hay 18–23%TRR, 2.62–10.6 mg equiv/kg), *tert*-sulfinyllactic acid and *tert*-malonylcysteine sulfoxide (forage and hay; combined 24–30%TRR, 2.72–17.3 mg equiv/kg). A large number of other metabolites were present at levels in excess of 0.1 mg equiv/kg.

In contrast, in PP soya bean forage or hay the compounds detected resulted largely from the uptake of soil metabolites to give oxanilate-type metabolites. None of the individual components exceeded 10% of TRR in immature plant, forage or hay. The major metabolites were *tert*-oxanilic acid (> 9.5% TRR, > 0.158 mg equiv/kg) in forage and *tert*-oxanilic acid combined with *tert*-sulfonic acid present at levels of > 9.7% (0.34 mg equiv/kg) in hay.

Both PP and PO seed extracts contained numerous low-level metabolites (≥ 27), none of which exceeded 0.03 mg equiv/kg. PP seed metabolites were generally more polar than PO seed metabolites.

The major aniline metabolite classes in soya bean commodities are EMA and "other" for PP forage, HEMA and EMA for PP hay and EMA for PO hay.

Cotton

The metabolic fate of [^{14}C-U-phenyl]-acetochlor in cotton maintained outdoors was examined following either a pre-plant (PP) soil (sandy loam) application 30 days before seed planting or as a separate application (PO) made to plants 15 days after the majority of plants had reached their first white flower stage. The application rates were 3.6 kg ai/ha for the PP and for the PO application.

TRR in PO leaves/stems were 63.9 mg equiv/kg whilst the TRR in PP leaves/stems were much lower at 5.7 mg equiv/kg. The TRRs in seed from both treatments were similar at 0.13 mg equiv/kg for the PO treatment and 0.10 mg equiv/kg for the PP treatment.

Solvent (CH_3CN/H_2O) extracted $\geq$ 88% of the TRR present in leaf/stem samples. Extraction of ^{14}C present in seed was lower at 29–44% TRR.

In contrast to maize and soya bean, the metabolites identified following PP and PO applications were both from initial conjugation of acetochlor with glutathione, followed by subsequent loss of glutamate, then glycine. The resulting cysteinyl product underwent oxidation, deamination, dealkylation, and further conjugation with malonate or glucose to produce numerous metabolites. Only one compound exceeded 10% of TRR in PP leaves/stems: 1 hydroxyethyl-*sec*-methylsulfone glucosylsulfate conjugate (> 15%TRR, > 0.85 mg equiv/kg) and one following PO application: *sec*-sulfinyllactic acid (20% TRR, 12.5 mg equiv/kg). Levels of ^{14}C in cotton seed were too low to allow identification of the numerous metabolites present, none of which individually exceeded 5.3% TRR or 0.007 mg equiv/kg.

The major aniline metabolite classes in cotton leaves and stems are EMA and HEMA.

In summary, the metabolism of acetochlor by plants is well understood. Primary metabolic pathways of acetochlor in plants included:

1) hydrolytic/oxidative dechlorination to form the alcohol (and conjugates) and subsequent oxidation of the alcohol to the oxanilic acid

2) displacement of chlorine by glutathione (or homoglutathione) and further catabolism of the products to cysteine or lactic acid metabolites, and the S-oxides and conjugates, or to sulfonic acids and methyl sulfones

3) ethyl/methyl side-chain or ring hydroxylation; and 4) N dealkylation. Oxanilate, sulfonic acid, and sulfone metabolites were more prevalent in PP and PE matrices. Glutathione/homoglutathione conjugation followed by catabolism to cysteine and lactic acid metabolites, and their oxidized derivatives and conjugates, was the primary metabolic pathway for acetochlor after PO treatment.

Animal metabolism

The plant metabolism studies show that livestock are unlikely to be exposed to parent acetochlor. Rather, animals will be exposed to a range of metabolites, none of which is considered likely to be a major component of the residue. A range of livestock metabolism studies were made available to the meeting including the metabolism of acetochlor in lactating goats and laying hens as well as the metabolism of a range of plant metabolites administered individually or as a combination to lactating animals (goats and cows) or laying hens.

Acetochlor

Lactating goats were orally dosed twice daily for four consecutive days with [^{14}C-U-phenyl]-acetochlor at a dose equivalent to 8.1 to 11 ppm in the feed. The majority of the ^{14}C residues was recovered in the excreta (urine 58–71%AD, faeces 20–29% AD). For tissues, ^{14}C residues were highest in liver, (0.277–0.588 mg equiv/kg), followed by the kidney (0.247–0.479 mg equiv/kg), muscle TRR ranged from (0.012 to 0.024 mg equiv/kg) and fat (0.002–0.003 mg equiv/kg). TRR in milk reached 0.016 mg equiv/kg after two days of dosing. No intact acetochlor was detected in tissues or milk. The majority of the residues were not recovered by mild extraction techniques using organic solvents or water at ambient temperatures. Cell fractionation confirmed the ^{14}C in the solids had been incorporated into natural products, principally proteins.

Laying hens were orally dosed once a day for seven consecutive days with [^{14}C-U-phenyl]-acetochlor at a dose equivalent to 10 ppm in the feed. The majority of the ^{14}C residues was recovered in the excreta (68–72.3%AD). Radioactivity reached its highest level in eggs on Day 7 from the start of dosing, with average concentrations of 0.072 mg equiv/kg for yolk and

0.007 mg equiv/kg for egg whites. Mean levels of TRR were 0.337 mg equiv/kg in liver, 0.054 mg equiv/kg in breast muscle, 0.072 mg equiv/kg in leg muscle, 0.019 mg equiv/kg in peritoneal fat, and 0.041 mg equiv/kg in skin plus subcutaneous fat. No intact acetochlor was detected in tissues or eggs. The majority of the residue was associated with natural products; proteins, glycan, and lipid fractions.

Metabolism of selected acetochlor plant metabolites by livestock

1-hydroxyethyl-tert-sulfonic acid

Groups of lactating goats were dosed orally with ^{14}C-[1-hydroxyethyl-*tert*-sulfonic acid] for five or 28 consecutive days at a dose equivalent to 0.4 to 5.7 ppm in the feed. In an animal dosed at the equivalent of 5.7 ppm for five days, most of the ^{14}C was recovered in the excreta (faeces 68.7%AD, and urine 3.65% AD). TRR in tissues was very low, with 0.007 mg equiv/kg (1-hydroxyethyl-*tert*-sulfonic acid equivalents) in kidney, 0.003 mg equiv/kg in liver, and < 0.0003 mg equiv/kg in muscle and fat. For animals dosed for 28 days, ^{14}C residues in milk and tissues were < 0.001 mg equiv/kg.

Metabolism of four acetochlor plant metabolites co-administered to lactating goat

Two lactating goats were orally dosed with a mixture of metabolites (*tert*-sulfonic acid, *tert*-oxanilic acid, *tert*-hydroxyacetochlor and *tert*-sulfinylacetic acid ratio 25:19:13:1 based on weight) uniformly labelled in the phenyl ring at 13.7 mg acetochlor equivalents/goat twice daily for five days equivalent to 3.2 and 4.3 ppm (acetochlor equivalents) in the feed. Most of the ^{14}C was excreted (63–79% AD) with similar amounts recovered in urine (34–42% AD) and faeces (29–37% AD). Residues in milk reached a plateau by the fourth day of dosing. Levels of ^{14}C were highest in kidney (0.034 mg acetochlor equiv/kg) followed by liver (0.022 mg equiv/kg) with levels in muscle and fat below the limit of detection. Levels of ^{14}C in milk were 0.006 mg equiv/kg. The HPLC profile of urine and faeces was similar to the dosing solution suggesting limited transformation occurs. Due to the low levels of ^{14}C present in tissue, analysis was by high pressure acid hydrolysis to form anilines. The only aniline metabolite class observed was EMA, the same as the dosing compounds.

Laying hens were dosed with the same mixture of metabolites (but in ratios 1:1:1:1 based on weight) for five to six days at doses equivalent to 13 to 88 ppm (acetochlor equivalents) in the feed. Excreta and cage wash accounted for ≥ 96% AD. The highest levels of ^{14}C found in the tissues of the hens dosed with 88 ppm were in liver (0.150–0.266 mg equiv/kg) followed by kidneys (0.106–0.128 mg equiv/kg) with much lower levels found in fat (0.049–0.061 mg equiv/kg) and muscle (0.024–0.032 mg equiv/kg). Egg whites and yolks collected at sacrifice had ^{14}C residue levels that ranged from 0.029 to 0.052 mg equiv/kg and from 0.192 to 0.198 mg equiv/kg, respectively.

The main components of ^{14}C detected in tissues and eggs were unchanged *tert*-hydroxyacetochlor (2.9–26%TRR) and *tert*-oxanilic acid 1.2–20.4% TRR) as well as *sec*-oxanilic acid (6.3% TRR yolk).

Metabolism of 5-hydroxy-sec-oxanilic acid in lactating cow

A metabolite of acetochlor in maize, 5-hydroxy-*sec*-oxanilic acid, uniformly labelled in the phenyl ring was used to dose a lactating cow at a nominal rate of 25 ppm (30 ppm if expressed in acetochlor equivalents) in the diet for seven consecutive days. Most of the administered dose was recovered from the excreta (faeces 82.5% and urine 8.4%).

The residues in all tissues and milk were < 0.01 mg 5-hydroxy-sec-oxanilic acid equiv/kg, except in the kidney which had a residue of 0.015 mg equiv/kg. Extraction of ^{14}C residues in kidney with CH_3CN:H_2O released 70% of the TRR. In kidney unchanged 5-hydroxy-*sec*-oxanilic acid accounted for 46.7%TRR with the remainder composed of unextracted material (24.5%TRR) and uncharacterized aqueous soluble residues (15.0%TRR).

The metabolism of acetochlor and selected plant metabolites (*tert*-oxanilic acid, *tert*-sulfonic acid, *tert*-sulfinylacetic acid, *sec*-sulfonic acid, *tert*-norchloroacteochlor, 5-hydroxy-*sec*-

oxanilic acid?) in laboratory animals (rats) was summarized and evaluated by the WHO panel of the JMPR in the present meeting.

In summary, the metabolism of acetochlor in goats is similar to metabolism in laboratory animals. Studies on a limited number of plant metabolites suggests, at least for these plant metabolites, that following oral dosing they remain the major component of the ^{14}C residues.

Environmental fate

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

The degradation of acetochlor in soil maintained under aerobic conditions is rapid with four major degradates identified; *tert*-oxanilic acid, *tert*-hydroxy, *tert*-sulfonic acid and *tert*-sulfinylacetic acid. While parent acetochlor is degraded relatively quickly in soils the degradates formed are moderately persistent. In the laboratory studies, soil DT_{50} values for parent acetochlor ranged from 3.3 to 55 days while for field dissipation studies DT_{50} values ranged from 2.9 to 12.6 days.

Acetochlor was stable to hydrolysis in aqueous solutions at pH 5, 7 and 9 (25 °C) suggesting hydrolysis plays a negligible role in its degradation. Similarly negligible degradation was observed in an aqueous photolysis study suggesting photolysis is not a major route of degradation.

In a confined rotational crop study with lettuce, radish and wheat, a plot of sandy loam soil was treated with [^{14}C-U-phenyl]-acetochlor at the equivalent of 2.24 or 3.36 kg ai/ha and crops sown 30, 120 and 365 days after the soil application. Analysis of soil extracts prior to planting showed that acetochlor was degraded to an array of compounds, many of which were present at very low levels. In addition to acetochlor, four major soil degradates were identified as present in soil throughout the study: *tert*-oxanilic acid, *tert*-sulfonic acid, *tert*-sulfinylacetic acid and *tert*-hydroxyacetochlor.

Five compounds, which were consistently present in plant extracts from all three rotation intervals were: *sec*-oxanilic acid (0–11%TRR; < LOD–0.075 mg equiv/kg; not observed in grain), *tert*-oxanilic acid (0–25%TRR; < LOD–0.17 mg equiv/kg; up to 0.003 mg equiv/kg in grain), *sec*-sulfonic acid (0–27%TRR; < LOD–0.21 mg equiv/kg; not observed in grain), *tert*-sulfonic acid (0–16%TRR; < LOD–0.072 mg equiv/kg; up to 0.0045 mg equiv/kg in grain), and 1-hydroxyethyl *tert*-oxanilic acid (0–15%TRR; < LOD–0.43 mg equiv/kg; up to 0.0017 mg equiv/kg in grain). Unextracted radioactive residues in plant matrices were characterized by cell wall fractionation. The majority of this plant bound material was incorporated into hemicellulose and cellulose and in the case of wheat grain into starch.

The major aniline metabolite class in rotational crop types was EMA except in wheat grain for which it was HMEA and HEMA.

In a separate study [^{14}C-U-phenyl]-acetochlor was applied to the surface of a sandy loam soil at a nominal rate equivalent to 3.08 kg ai/ha. Mustard, turnip and millet were planted approximately 30, 120 and 365 days after [^{14}C]acetochlor application. Soya beans were planted approximately 30 and 365 days after treatment. The radioactive residues dissipated rapidly in soil with only 22% AR remaining 30 days after application. The main identified soil degradates were *tert*-oxanilic acid, *tert*-sulfinylacetic acid and *tert*-sulfonic acid.

Analyses of the plant extracts showed that extensive metabolism occurred in all crops. Acetochlor was not found in any of the RACs analysed, With the exception of Day 30 turnip roots, acetochlor was not found in RACs. The ^{14}C residue levels decreased in crops from the 30 day compared to the 365 days planting. The TRR was partially characterized and found to be comprised of up to nine different compounds, with not one above 0.01 mg equiv/kg in the edible portion of the root or cereal crop (turnip root and millet grain). The major metabolites identified

in crops planted 30 DAA were *tert*-oxanilic acid, *sec*-methyl sulfone, *sec*-hydroxyacetochlor, and *tert*-methyl sulfone.

The major aniline class of metabolites was EMA in which no hydroxylation of the alkyl groups of the phenyl ring had occurred with HEMA class metabolites was also significant.

In summary, acetochlor related residues in soil may contribute to residues observed in rotational and primary crops.

Methods of Analysis

The metabolism of acetochlor in crops results in a complex mixture of metabolites, most of which produce EMA or HEMA on base hydrolysis. Any non-metabolised parent acetochlor that might be present would be converted to EMA upon hydrolysis.

Consequently most of the methods developed to quantify acetochlor residues in animal and plant commodities involve hydrolytic conversion of metabolites to the EMA and HEMA chemophores. These analytes are quantified and expressed in acetochlor equivalents and then may be added to give total acetochlor residues. LOQs are typically 0.01 mg/kg each for EMA and HEMA.

The methods all involve initial extraction of samples with an organic/aqueous solvent mixture, typically CH_3CN/H_2O, followed by hydrolysis of residues with aqueous hydroxide solutions. The main differences between methods involve clean-up conditions and instrumentation for quantification, LC-MS/MS in more recent versions.

Representative compounds that generate EMA (*tert*-sulfonic acid) and HEMA (1-hydroxyethyl-*tert*-oxanilic acid) on base hydrolysis are used as reference materials for fortification and method validation.

The methods are suitable for analysis of acetochlor and related metabolites in plant and animal matrices.

Multi-residue methods are currently not validated for acetochlor and its metabolites.

Stability of pesticide residues in stored analytical samples

The Meeting received information on the stability of acetochlor and example metabolites hydrolysable to EMA (*tert*-sulfonic acid) and HEMA (1-hydroxyethyl-*tert*-oxanilic acid) and for some matrices HMEA (hydroxymethyl-*tert*-oxanilic acid) and OH-class (5-hydroxy-*sec*-oxanilic acid) in various matrices on freezer storage (–18 °C).

Residues of parent acetochlor were stable in potato tubers for at least 295 days and sugar beet tops for at least 294 days storage.

Residues of *tert*-sulfonic acid (EMA-class) and 1-hydroxyethyl-*tert*-oxanilic acid (HEMA class) measured using a common moiety method, were stable in alfalfa forage and clover hay for at least 330 days freezer storage, soya bean forage for 390 days, soya bean hay for 391 days, soya bean grain for 382 days, wheat forage for 741 days, wheat straw for 741 days, wheat grain for 734 days, sorghum silage for 739 days, sorghum grain for 732 days, potato tubers for 286 days, sugar beet tops for 286 days, maize grain for 356 days, maize forage for 357 days and maize stover for 351 days.

Residues of hydroxymethyl-*tert*-oxanilic acid (HMEA class) measured using a common moiety method, were stable in sorghum grain, silage for at least 732 days, soya bean grain, forage and hay for at least 380 days and wheat grain, forage and straw for at least 734 days.

Residues of 5-hydroxy-*sec*-oxanilic acid (OH-class) measured using a common moiety method, were stable in maize grain, forage and stover, lettuce, turnip roots and leaves and soya bean seed and hay for at least 730 days.

Residues of a mixture of *tert*-hydroxyacetochlor, *tert*-oxanilic acid, *tert*-sulfonic acid and *tert*-sulfinylacetic acid (EMA-class) in equal proportions measured using a common moiety method, were stable in eggs, milk, chicken liver, pig liver, beef liver, muscle, fat, and kidneys for at least 910 days.

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

Definition of the residue

Following application of acetochlor to crops (maize, soya bean and cotton) a large number of metabolites were detected, but not unchanged acetochlor. There were notable differences in the pattern of metabolites observed following applications (PP and PE) to soil prior to crop emergence compared to applications made when the crop is present (PO).

The metabolites identified in forage, stover and hay following PO application to maize and soya beans are mainly sulfoxide-type metabolites. Significant metabolites (> 10%TRR) were *tert*-sulfinyllactic acid (13% TRR 0.43 mg equiv/kg maize forage; 11% TRR 0.72 mg equiv/kg maize stover), *tert*-cysteine (39% TRR soya hay), *tert*-malonylcysteine (18–23%TRR soya bean forage and hay), *tert*-sulfinyllactic acid and *tert*-malonylcysteine sulfoxide (combined 24–30%TRR soya bean forage and hay).

In contrast, in PE maize and PP soya beans the compounds detected resulted largely from the uptake of soil metabolites to give oxanilate-type metabolites. None of the individual components exceeded 10% of TRR in immature plant, forage, stover or hay. The major metabolite in PE maize was 5-hydroxy *sec*-oxanilic acid present at levels of 8.4% (0.099 mg equiv/kg) 6.2% (0.042 mg equiv/kg) and 4.3% (0.080 mg equiv/kg) TRR in immature plants, forage and stover respectively. The major metabolites in soya bean were *tert*-oxanilic acid (> 9.5% TRR) in forage and combined with *tert*-sulfonic acid present at levels of > 9.7% (0.34 mg equiv/kg) in hay.

Metabolism of acetochlor in cotton differed compared to maize and soya bean in that the metabolites identified following both PP and PO applications were from initial conjugation of acetochlor with glutathione, followed by subsequent loss of glutamate, then glycine. Only one compound exceeded 10% of TRR in PP leaves/stems: 1 hydroxyethyl-*sec*-methylsulfone glucosylsulfate conjugate (14.8%TRR) and one following PO application: *sec*-sulfinyllactic acid (20% TRR).

Negligible residues were detected in seeds and grain. Metabolites detected in maize were individually present at < 0.001 mg/kg. Identification of individual metabolites was not achieved in soya bean grain and cotton seed. In both cases extracts contained numerous metabolites, each present at < 0.03 mg equiv/kg (soya bean grain) or < 0.01 mg equiv/kg (cotton seed).

There is no obvious candidate compound for use as a residue definition for compliance, nor is there a small group of compounds that combined could usefully be used to monitor compliance. It is noted that the majority of the residue in crops can be classified according to the aniline class formed on base hydrolysis. As such a common moiety residue definition would allow residues to be monitored in all crops and derived commodities.

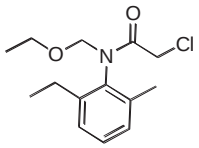
The major aniline metabolite class observed in maize (PE and PO) is EMA followed by OH, in soya bean commodities EMA and “other” for PE soya bean forage, HEMA and EMA for PE soya bean hay and EMA for PO soya bean hay and in cotton leaves and stems EMA and HEMA.

Validated analytical methods are available for the determination of compounds hydrolysable with base to EMA and HEMA in crop matrices.

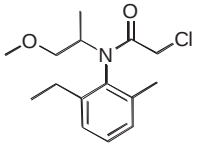
Residues derived from acetochlor may also occur in rotational (follow) crops. Five metabolites, which were consistently present in plant extracts from all rotation intervals studied were: *sec*-oxanilic acid, *tert*-oxanilic acid, *sec*-sulfonic acid, *tert*-sulfonic acid, and 1-

hydroxyethyl *tert*-oxanilic acid. The major aniline metabolite class in rotational crop types studied was EMA except wheat grain for which it was HMEA and HEMA.

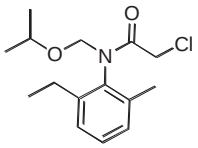
The Meeting also noted that acetochlor is a member of the chloroacetamide herbicides, a group that also includes metolachlor and propisochlor. The structures of these herbicides are similar to acetochlor and they are expected to share a number of common metabolites on cleavage of the ether side-chain.



Acetochlor

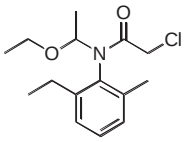


Metolachlor



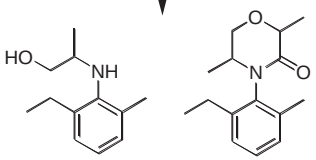
Propisochlor

A common moiety method of analysis has been developed for metolachlor that involves hydrolysis in 6N HCl. The resulting compounds differ from those produced by acetochlor and where required re-analysis of samples using the metolachlor method could be used to distinguish acetochlor from metolachlor residues.



metolachlor

6N HCl



CGA-37913 CGA-49751

No naturally occurring compounds hydrolysable to EMA and HEMA have been identified in crops likely to be treated or grown as follow crops.

The Meeting decided the residue definition for compliance with MRLs and estimation of dietary intake in plants should be the sum of compounds converted to EMA and HEMA, expressed in terms of acetochlor

Livestock may be exposed to acetochlor-derived residues present in feeds. Due to the extensive metabolism of acetochlor in plants, exposure to unchanged parent compound is not expected. Additionally the extensive metabolism combined with metabolite profiles that differ with application type (pre-emergence or post-emergence) and also crops complicate the choice of metabolite mixtures that might usefully typify the metabolite profiles present in feed, and therefore the nature of residues in livestock commodities. Available studies involving a limited number of plant metabolites suggest the major components of the residues in livestock commodities are the dosing compounds. Therefore, as for plant commodities, it is proposed the residue definition for compliance in animals be compounds converted to EMA and HEMA. Analytical methods are available for animal matrices.

Residues hydrolysable to EMA and HEMA and captured by the residue definition are comprised of a range of hydroxylated acetochlor-derived compounds as well as conjugates, all reactions that are expected to increase water solubility. Taken as a whole, the Meeting considered that residues encompassed by the residue definition for acetochlor are not fat soluble.

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):

Sum of compounds converted to EMA and HEMA, expressed in terms of acetochlor.

The residue is not fat soluble.

Results of supervised residue trials on crops

Supervised residue trial data for were available for acetochlor on maize, sweet corn, cotton, sorghum, soya bean, sugar beet and peanuts. With the exception of one series of trials on maize where 5-hydroxy *sec*-oxanilic acid was analysed, residues were measured as compounds hydrolysable with base to EMA and HEMA. Residues listed below are for the sum of compounds hydrolysed to EMA and HEMA expressed in acetochlor equivalents.

The following indicates how the residues were combined when residues were reported as < LOQ for one or both of the components.

EMA	HEMA	Total residues (EMA+HEMA)
< 0.05	< 0.05	< 0.1
0.1	< 0.05	< 0.15
0.1	0.06	0.16

Sweet corn

The Meeting received supervised residue trial data for acetochlor on sweet corn from the USA. GAP in the USA is applications pre-plant or pre-emergence at up to 3.0 kg ai/ha with a PHI not required. The maximum rate per year is 3.4 kg ai/ha. In trials approximating critical GAP in the USA residues in sweet corn were (n=14): < 0.04 (14) mg/kg (kernels with husks removed).

The Meeting estimated a maximum residue level, STMR and HR or 0.04 (*), 0.04 and 0.04 mg/kg respectively for sweet corn (corn-on-the-cob).

Soya bean

In the USA acetochlor is approved for use on soya beans. GAP in the USA is applications pre-plant, pre-emergence or post-emergence but before the R2 growth stage (full flowering) at up to 1.7 kg ai/ha with a PHI not required. The maximum rate per year is 3.4 kg ai/ha. None of the trials matched critical GAP (2× 1.7 kg ai/ha post-emergence applications) and none were suitable for applying the proportionality approach.

Sugar beet

Supervised residue trial data for acetochlor on sugar beet were made available. GAP in the USA is applications pre-plant, pre-emergence or post-emergence (2 to 8 leaf stage) at up to 1.7 kg ai/ha with a PHI of 70 days. The maximum rate per year is 3.4 kg ai/ha. In trials approximating critical GAP in the USA residues in sugar beet roots were (n=15): < 0.008, < 0.009, 0.011, 0.011, 0.015, 0.016, 0.017, 0.018, 0.019, 0.021, 0.021, 0.025, 0.045, 0.051 and 0.086 mg/kg.

The Meeting estimated a maximum residue level and STMR of 0.15 and 0.018 mg/kg respectively for sugar beet roots.

Maize

The Meeting received supervised residue trial data for acetochlor on maize. GAP in the USA is applications pre-plant, pre-emergence or post-emergence (28 cm height) at up to 3.0 kg ai/ha with a PHI not specified for the EC formulation and pre-plant, pre-emergence or post-emergence (76 cm height) at up to 2.5 kg ai/ha for the CS formulation. The maximum rate per year is 3.4 kg ai/ha. The

Meeting considered trials with the CS formulation where the last application can be made closer to harvest but at a lower rate compared to the EC trials where applications are made earlier but at a higher rate to give rise to higher residues and represent critical GAP. Critical GAP was considered to be pre-emergent application at 0.9 kg ai/ha followed by post-emergence application at 2.5 kg ai/ha. In trials with the CS formulation, maize was treated with a single post-emergence application rate at approximately 3.2 kg ai/ha (1.28× the maximum label rate). The Meeting agreed to utilise the proportionality approach to estimate residues matching *c*GAP noting that residues from pre-emergence applications do not contribute to final residues and that a single post-emergence application at 2.5 kg ai/ha should be targeted for use in estimating maximum residue levels. The following scaled residues (n=21) matched *c*GAP:

Trial application rate (kg ai/ha)	Scaling factor = 2.5/trial application rate	Trial residue (mg/kg)	Scaled residue =scaling factor × trial residue (mg/kg) [a]
3.31	0.755	< 0.002	< 0.002
3.19	0.784	< 0.002	< 0.002
3.19	0.784	< 0.002	< 0.002
3.22	0.776	< 0.002	< 0.002
3.15	0.794	0.003	< 0.002
3.19	0.784	0.003	< 0.002
3.18	0.786	0.003	< 0.002
3.16	0.791	0.003	< 0.002
3.26	0.767	0.003	< 0.002
3.17	0.789	0.003	< 0.002
3.09	0.809	0.003	< 0.002
3.18	0.786	0.004	< 0.003
3.19	0.784	0.004	< 0.003
3.14	0.796	0.006	0.005
3.33	0.751	0.006	0.005
3.33	0.751	0.008	0.006
3.13	0.799	0.008	0.006
3.17	0.789	0.009	0.007
3.27	0.765	0.009	0.007
3.24	0.772	0.009	0.007
3.32	0.753	0.019	0.014

[a] LOQ for combined residues is 0.002 mg/kg.

The Meeting estimated a maximum residue level, STMR and HR of 0.02 and 0.002 mg/kg respectively for maize.

Sorghum

Acetochlor is approved in the USA for use on sorghum. GAP in the USA is applications pre-plant, pre-emergence or post-emergence (28 cm height) at up to 2.5 kg ai/ha with a PHI not specified. The maximum rate per year is 3.4 kg ai/ha. No trials matched cGAP (2× 1.7 kg ai/ha POST) and the data were not suitable for use of the proportionality approach.

Cotton

The Meeting received supervised residue trial data for acetochlor on cotton. GAP in the USA is applications pre-plant, pre-emergence or post-emergence (before 1st bloom) at up to 1.7 kg ai/ha with a PHI not specified. The maximum rate per year is 3.4 kg ai/ha. Two-post-emergence applications made closest to the latest growth stage permitted lead to highest residues. No trials utilising post-emergence application matched critical GAP in the USA.

Peanut

Supervised residue trial data for acetochlor on peanuts were available. GAP in the USA is applications pre-plant, pre-emergence or post-emergence (before flowering) at up to 1.7 kg ai/ha with a PHI not specified. The maximum rate per year is 3.4 kg ai/ha. In trials conducted in the USA plots

were treated pre-plant and post-emergence (1.7 PP + 1.7 PO kg ai/ha), pre-emergent and post-emergent (1.7 PE + 1.7 POST kg ai/ha) or post-emergent (3.4 PO kg ai/ha). No trials matched cGAP (2× 1.7 PO kg ai/ha) and the data were not suitable for use of the proportionality approach.

Animal feeds

Peanut fodder

GAP in the USA is applications pre-plant, pre-emergence or post-emergence (before flowering) at up to 1.7 kg ai/ha with a PHI not specified. The maximum rate per year is 3.4 kg ai/ha. GAP in the USA is to allow a minimum of 90 days between last application and grazing or harvest and feeding of peanut hay to livestock. No trials matched *c*GAP (2× 1.7 POST kg ai/ha).

Soya bean forage

In the USA there are restraints on the grazing and feeding of post-emergence treated soya bean forage to livestock.

Soya bean fodder

In the USA acetochlor is approved for use on soya beans. GAP in the USA is applications pre-plant, pre-emergence or post-emergence but before the R2 growth stage (full flowering) at up to 1.7 kg ai/ha with a PHI not required. None of the trials matched *c*GAP and none were suitable for use of the proportionality approach.

Corn and maize forage

GAP in the USA is applications pre-plant, pre-emergence or post-emergence (28 cm height) at up to 3.0 kg ai/ha with a PHI not specified for the EC formulation and pre-plant, pre-emergence or post-emergence (76 cm height) at up to 2.5 kg ai/ha for the CS formulation. The maximum rate per year is 3.4 kg ai/ha. GAP for maize (field corn) in the USA requires that treated areas are not grazed and treated forage not fed to livestock for 40 days following application. No trials matched *c*GAP.

GAP in the USA for sweet corn is applications of an EC formulation pre-plant or pre-emergence at up to 3.0 kg ai/ha with a PHI not required. The maximum rate per year is 3.4 kg ai/ha. Residues in sweet corn forage from field trials performed in the USA approximating cGAP in the USA were (n=13): < 0.04, < 0.06, < 0.08, 0.08, < 0.09, 0.1, < 0.12, 0.14, 0.22, 0.24, 0.29, 0.44 and 0.97 mg/kg (on an as received basis). Sweet corn forage contains approximately 48% DM.

The Meeting estimated median and highest residues of 0.25 and 2.02 mg/kg for sweet corn forage (on a dry matter basis).

Corn and maize fodder

For maize (field corn), GAP in the USA is applications pre-plant, pre-emergence or post-emergence (28 cm height) at up to 3.0 kg ai/ha with a PHI not specified for the EC formulation and pre-plant, pre-emergence or post-emergence (76 cm height) at up to 2.5 kg ai/ha for the CS formulation. The maximum rate per year is 3.36 kg ai/ha. The Meeting considered trials with the CS formulation where the last application can be made closer to harvest but at a lower rate compared to the EC trials where applications are made earlier but at a higher rate to give rise to higher residues and represent critical GAP. Critical GAP was considered to be pre-emergent application at 0.9 kg ai/ha followed by post-emergence application at 2.5 kg ai/ha. No trials matched cGAP.

Residues in sweet corn fodder from field trials performed in the USA approximating cGAP in the USA were (n=14): < 0.04, < 0.04, < 0.04, < 0.04, 0.04, < 0.05, < 0.06, 0.08, 0.09, 0.10, 0.13, 0.13, 0.42 and 0.91 mg/kg (on an as received basis). Sweet corn fodder contains approximately 83% DM.

The Meeting estimated a maximum residue level and median and highest residues of 1.5, 0.07 and 0.91 mg/kg for sweet corn fodder (on as received matter basis) or 1.5, 0.084 and 1.096 mg/kg (dry matter basis) assuming 83% dry matter (DM).

Sorghum forage

GAP in the USA requires that treated areas are not grazed and treated forage not fed to livestock for 60 days following application. In the USA applications are made pre-plant, pre-emergence or post-emergence (28 cm height) at up to 2.5 kg ai/ha. No trials matched *c*GAP.

Sorghum fodder (stover)

GAP in the USA is applications pre-plant, pre-emergence or post-emergence (28 cm height) at up to 2.5 kg ai/ha with a PHI not specified. No trials matched cGAP.

Cotton gin by-products

No trials on cotton matched GAP in the USA.

Sugar beet tops

GAP in the USA is applications pre-plant, pre-emergence or post-emergence (2 to 8 leaf stage) at up to 1.7 kg ai/ha with a 70 day interval between the last application and grazing or harvest of sugar beet tops. The maximum seasonal application is 3.4 kg ai/ha/year. Residues in sugar beet tops from field trials performed in the USA approximating cGAP in the USA were (n=15): 0.009, 0.014, 0.019, 0.028, 0.028, 0.030, 0.035, 0.041, 0.043, 0.050, 0.051, 0.056, 0.063, 0.147 and 0.554 mg/kg (on an as received basis). Sugar beet tops contain approximately 23% DM.

The Meeting estimated a maximum residue level and median residues of 3 and 0.178 mg/kg for sugar beet tops (on dry matter basis).

Rotational crop residues

Soil residues of acetochlor related compounds are moderately persistent. The use-pattern (USA GAP) specifies plant-back intervals for certain follow-crops as well as crops that may be rotated following application:

- Non-grass animal feeds such as alfalfa, clover, kudzu, lespedeza, lupin, sainfoin, trefoil, velvet bean, and Vetch spp. may be planted 9 months (270 days) after application.
- Wheat may be planted 4 months (120 days) after application.
- Rotate the next season to the following crops—soya beans, corn (all types), milo (sorghum), cotton, sugar beets, sunflowers, potatoes, barley, buckwheat, millet (pearl and proso), oats, rye, teosinte, triticale, wild rice, dried shelled bean group Lupinus spp. (including grain lupin, sweet lupin and white lupin), Phaseolus spp. (includes field beans, kidney beans, lima beans (dry), navy beans, pinto bean and tepary beans), bean Vigna spp. (includes adzuki beans, black-eyed peas, catjang, cowpeas, Crowder peas, moth beans, mung beans, rice beans, southern peas and urd beans), broad beans (dry), chickpeas, guar, lab lab beans, lentils, peas (Pisum spp., includes field peas) and pigeon peas.

Field crop rotation residue trials are available for representative crops that may be rotated. In these trials follow crops were planted after harvesting of maize that had been treated with acetochlor as a pre-plant, pre-emergence or seed treatment at 3.4 kg ai/ha equivalent to the maximal seasonal rate in the USA. The Meeting considered these trials reflect likely residues in crops grown in rotation following application at the maximum seasonal rate (3.4 kg ai/ha/year).

Legume animal feed as a follow crop

Residues in follow crops of alfalfa and clover as representative legume feed commodities were made available to the Meeting. Alfalfa was sown 274–355 days after pre-emergent application to maize. Clover was sown 274–355 days after pre-emergent application to maize. Residues are listed below:

Alfalfa forage (n=17): < 0.04, 0.04, 0.06, 0.07, 0.08, 0.08, 0.08, 0.09, 0.11, 0.14, 0.14, 0.16, 0.20, 0.29, 0.35, 0.47 and 0.54 mg/kg (fresh weight basis).

Clover forage (n=18): < 0.03, < 0.04, < 0.04, 0.04, < 0.05, < 0.05, 0.05, 0.06, 0.10, 0.10, 0.10, 0.10, 0.11, 0.16, 0.17, 0.17, 0.35 and 0.57 mg/kg (fresh weight basis).

The Meeting estimated median and highest residues in legume forage of 0.10 and 0.57 mg/kg (as received basis) or 0.333 and 1.9 mg/kg when expressed on a dry matter basis (assuming 35%DM for alfalfa and 30%DM for clover).

Alfalfa hay (n=16): 0.11, 0.14, 0.15, 0.16, 0.18, 0.19, 0.20, 0.24, 0.28, 0.29, 0.33, 0.34, 0.73, 0.82, 0.97, and 1.87 mg/kg (fresh weight basis).

Clover hay (n=17): < 0.02, < 0.02, < 0.04, 0.08, 0.08, 0.08, 0.12, 0.13, 0.13, 0.15, 0.24, 0.30, 0.41, 0.44, 0.48, 0.76 and 1.24 mg/kg (fresh weight basis).

The median residues in the clover and alfalfa hay datasets differed by less than a factor of five and the Meeting decided to recommend a group maximum residue level for legume animal feeds. In deciding which data set to use for the recommendation, as a Mann Whitney U-test indicated that the residue populations were not different it was decided to combine the data sets.

Residues in alfalfa and clover fodder (hay) of follow crops ranged from < 0.02 to 1.87 mg/kg (as received basis). Alfalfa and clover hay contains approximately 89%DM.

The Meeting estimated maximum residue levels, median and highest residues of [2, 0.20 and 1.87 mg/kg fresh weight basis] 3, 0.225, and 2.101 mg/kg (dry matter basis) for legume animal feeds.

Wheat (forage, straw, grain)

Wheat may be planted as a follow crop four months after application. Residues (as received basis) in follow wheat crops planted 90–176 days after pre-emergent application to maize were:

- Forage (n=18): < 0.02, < 0.02, < 0.02, < 0.03, < 0.03, 0.03, 0.04, < 0.05, 0.06, 0.06, 0.11, 0.13, 0.14, 0.18, 0.19, 0.27, 0.41 and 0.47 mg/kg (fresh weight basis).

The Meeting estimated median and highest residues in wheat forage of 0.06 and 0.47 mg/kg (fresh weight basis) or 0.24 and 1.88 mg/kg when expressed on a dry matter basis (assuming 25%DM).

- Straw (n=18): < 0.02, < 0.02, < 0.02, < 0.02, < 0.02, < 0.02, < 0.02, 0.03, 0.03, 0.03, 0.04, 0.07, 0.07, 0.07, 0.07, 0.08, 0.09 and 0.10 mg/kg.

The Meeting estimated maximum residue levels, median and highest residues of 0.2, 0.034, and 0.114 mg/kg for wheat straw and fodder (dry matter basis) assuming wheat straw contains 88% dry matter.

- Grain (n=18): < 0.02 (18) mg/kg.

The Meeting estimated maximum residue levels and median residues of 0.02 (*) and 0.02 mg/kg for wheat grain.

Other cereals (forage, hay, straw, grain)

In the USA, a number of cereal and grass-like crops (other than wheat, maize, sorghum) may be planted approximately one year after last application. Residues (on an as received basis) in follow oat crops planted the next season after pre-emergent application to maize:

- Forage (n=18): < 0.035 (7), < 0.038, < 0.038, < 0.042, 0.048, 0.056, 0.057, 0.063, 0.066, 0.085 and 0.121 mg/kg (fresh weight basis).

The Meeting estimated median and highest residues in oat forage of 0.04 and 0.121 mg/kg (as received basis) or 0.13 and 0.40 mg/kg when expressed on a dry matter basis (assuming 30%DM).

- Hay (n=16): < 0.035 (6), < 0.036, < 0.036, < 0.042, 0.042, 0.060, 0.068, 0.074, 0.091, < 0.098 and 0.156 mg/kg (fresh weight basis).

The Meeting estimated median and highest residues of 0.039, and 0.173 mg/kg for oat hay (dry matter basis).

- Straw (n=17): < 0.035 (11), < 0.036, < 0.036, 0.044, 0.044, 0.070, and 0.254 mg/kg (fresh weight basis).

The Meeting estimated maximum residue levels, median and highest residues of 0.3, 0.039, and 0.282 mg/kg for oat straw (dry matter basis) assuming straw contains 90% dry matter.

- Grain (n=17): < 0.035 (16) and < 0.036 mg/kg.

The Meeting estimated a maximum residue level and STMR of 0.04 (*) and 0.035 mg/kg for oat grain.

The Meeting agreed to extrapolate the results for oats to other cereals that are permitted in the USA as follow crops and not treated directly—barley, buckwheat, millet (pearl and proso), rye, teosinte, triticale and wild rice commodities. The Meeting decided not to extrapolate the results to follow rice crops as the cultivation practices for rice differ from those of other cereal crops and this may impact on residues.

Sunflowers

Sunflowers are a permitted follow crop when planted the following year. Residues in seed of follow sunflower crops planted 350–384 days after pre-emergent application to maize were all < 0.04 (8) mg/kg. The Meeting estimated a maximum residue level and STMR of 0.04 (*) and 0.04 mg/kg for sunflower seed.

Potato

Potatoes are a permitted follow crop when planted the following year. Residues in tubers of follow potato crops (planted 291–380 days after pre-emergent application to maize) were all < 0.04 (10) mg/kg. The Meeting estimated a maximum residue level, STMR and HR of 0.04 (*), 0.04, and 0.04 mg/kg for potatoes.

Beans (dry), Peas (dry)

A number of legume grains are permitted follow crops to be planted the next season (about one year after the last application). Residues in grain of follow bean and pea crops were all < 0.02 mg/kg, nine bean trials and five pea trials. The Meeting estimated maximum residue levels of 0.02 (*) for beans and peas (dry) and STMRs or 0.02 mg/kg. The two maximum residue levels would cover residues in follow *Phaseolus* spp as well as *Vigna* spp and *Pisum* spp.

Rotational crop trials were available for follow soya beans. The observed residues are higher than reported for beans and peas (dry) and residues in follow soya beans could be used as a representative crop for the remaining pulses permitted to be rotated in the USA—*Lupinus* spp., broad beans, chickpeas, Hyacinth beans (lab lab beans), lentils, and pigeon peas.

Residues in seed of follow soya beans were (n=16): < 0.02 (8), 0.02, < 0.03, 0.03, 0.03, 0.04, 0.04, 0.06 and 0.10 mg/kg.

The Meeting agreed to extrapolate to residues in seed of follow soya bean to *Lupinus* spp., broad beans (dry), chickpeas, Hyacinth beans, dry (lab lab beans), lentils, and pigeon peas and estimated maximum residue limits of 0.15 and STMRs of 0.02 mg/kg for these seeds.

Fate of residues during processing

The Meeting received information on the fate of incurred residues of acetochlor during the processing of soya beans, sugar beets, sorghum, cotton, peanuts and sunflower seeds. A study of the nature of the residue of acetochlor under simulated processing conditions (pasteurization, baking/brewing/boiling, sterilization) showed acetochlor, if present, is stable.

Summaries of relevant acetochlor processing factors are provided below.

	Processed Fraction	Processing Factor	Best estimate PF	RAC STMR or median	STMR × PF = STMR-P	RAC HR or highest	HR × PF = HR-P
Sugar beet	Dried pulp	2.3, 0.9	1.6	0.018	0.029	0.086	0.138
	Molasses	4.2, 1.1	2.65		0.048		0.228
	Refined sugar	0.5, < 0.25	0.375		0.0068		0.032
Sunflower	Meal	1.4	1.4	0.04	0.056	0.04	0.056
	Oil	0.22	0.22		0.0088		0.0088

PFs are based on combined EMA and HEMA aniline class metabolites: PFs calculated as EMA + HEMA, expressed as acetochlor in processed commodity divided by EMA + HEMA in the RAC

The Meeting recommended a maximum residue level of 0.3 mg/kg for sugar beet molasses and a median residue of 0.048 mg/kg. For sugar beet pulp (dry) the Meeting recommended a maximum residue level of 0.3 mg/kg and a median residue of 0.029 mg/kg.

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 a mixture of four EMA class acetochlor plant metabolites (*tert*-hydroxy and the sodium salts of *tert*-sulfonic acid, *tert*-oxanilic acid and *tert*-sulfinylacetic acid and present in equal proportions) at the equivalent of 5, 15 and 50 ppm acetochlor equivalents in the feed for 28 consecutive days. Based on HPLC retention times for extracts in plant metabolism studies, it is concluded that the properties of the dosing compounds encompass the range of polarities of the majority of compounds observed in the plant metabolism studies (log K_{ow} of dosing compounds ranged from –3.2 to 2.2). The studies are considered to cover the likely transfer of acetochlor-related residues, including those from different aniline metabolite classes, from feed to livestock.

Residues in milk were < 0.02 mg/kg (acetochlor equivalents) for the 50 ppm dose group for all sample intervals.

In kidney mean residues were < 0.02, 0.03, and 0.07 mg/kg (acetochlor equivalents) for the 5, 15, and 50 ppm dose groups respectively. Mean residues liver residues were < 0.02 and 0.02 mg/kg for the 15 and 50 ppm dose groups while mean residues in fat and muscle were < 0.02 mg/kg for all samples in the 50 ppm dose group. As no residues were observed at the highest dose level samples muscle and fat from other dose groups were not analysed.

Laying hens dosed at the equivalent of 5, 15 and 50 ppm acetochlor with a mixture of *tert*-hydroxy and the sodium salts of *tert*-sulfonic acid, *tert*-oxanilic acid and *tert*-sulfinylacetic acid for 28 days. No residues above the LOQ were detected in any tissues or eggs, LOQ 0.05 mg/kg for kidney and LOQ 0.02 mg/kg for other tissues and eggs.

Estimation of livestock dietary burdens

Dietary burden calculations for beef cattle, 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 legume fodder, cereal forage and fodder, sugar beet tops and various grains.

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

	US-Canada		EU		Australia		Japan	
	max	mean	Max	mean	max	Mean	max	Mean
Beef cattle	0.4	0.1	2.0	0.3	2.1	0.3	0.2	0.03
Dairy cattle	1.4	0.2	1.6	0.2	2.1 [a, b]	0.3 [c, d]	0.6	0.09
Broilers	0.03	0.02	0.05	0.04	0.02	0.02	0.1	0.03
Layers	0.03	0.02	0.54 [e]	0.10 [f]	0.02	0.02	0.01	0.01

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

	Feed level	Residues	Feed level	Residues (mg/kg) in			
	(ppm) for milk residues	(mg/kg) in milk	(ppm) for tissue residues	Muscle	Liver	Kidney	Fat
MRL beef or dairy cattle							
Feeding study [a]	5	< 0.02	15	< 0.02	< 0.02	0.04	< 0.02
Dietary burden and high residue	2.1	< 0.008	2.1	< 0.003	< 0.003	0.0056	< 0.003
STMR beef or dairy cattle							
Feeding study [b]	5	< 0.02	15	< 0.02	< 0.02	0.03	< 0.02
Dietary burden and median residue estimate	0.3	< 0.0012	0.3	< 0.0004	< 0.0004	0.0006	< 0.0004

[a] Highest residues for tissues and mean residues for milk

[b] Mean residues for tissues and mean residues for milk

The Meeting estimated the following maximum residue levels: milk 0.02* mg/kg; meat (mammalian except marine mammals) 0.02* mg/kg, mammalian fat (except milk fat) 0.02* mg/kg andedible offal 0.02* mg/kg.

For poultry no residues were observed in eggs and tissues on dosing laying hens at up to 50 ppm in the diet for 28 days. The Meeting estimated the following maximum residue levels for poultry commodities: poultry meat 0.02* mg/kg; poultry edible offal 0.02* mg/kg and eggs 0.02* mg/kg. The Meeting estimated the following STMR and HR values: poultry meat 0 mg/kg; poultry fat 0 mg/kg; poultry edible offal 0 mg/kg and eggs 0 mg/kg.

RECOMMENDATIONS FURTHER WORK OR INFORMATION

On the basis of the data obtained from supervised residue trials the Meeting concluded that the residue levels listed in Annex 1 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):

Sum of compounds hydrolysable with base to 2-ethyl-6-methylaniline (EMA) and 2-(1-hydroxyethyl)-6-methylaniline (HEMA), expressed in terms of acetochlor.

The residue is not fat soluble.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
GC 0640	Barley	0.04 *		0.035	0.036
AS 0640	Barley straw and fodder, dry	0.3		0.039 dw[a]	0.282 dw
VP 0061	Beans, except broad bean and soya bean	0.02 *		0.02	0.02
VD 0523	Broad bean (dry)	0.15		0.02	0.1
GC 0641	Buckwheat	0.04 *		0.035	0.036
AS 0641	Buckwheat fodder	0.3		0.039 dw	0.282 dw
VD 0524	Chick-pea (dry)	0.15		0.02	0.1
MO 0105	Edible offal (mammalian)	0.02 *		0.0004 liver 0.0006 kidney	0.003 liver 0.0056 kidney
PE 0112	Eggs	0.02 *		0	0
VD 0531	Hyacinth bean (dry)	0.15		0.02	0.1
AL 0157	Legume animal feeds	3		0.225 dw	2.101 dw
VD 0533	Lentil (dry)	0.15		0.02	0.1
VP 0545	Lupin (dry)	0.15		0.02	0.1
GC 0645	Maize	0.02		0.002	
MF 0100	Mammalian fats (except milk fats)	0.02 *		0.0004	0.003
MM 0095	Meat (from mammals other than marine mammals)	0.02 *		0.0004	0.003
ML 0106	Milks	0.02 *		0.0012	0.008
GC 0646	Millet	0.04 *		0.035	0.036
AS 0646	Millet fodder, dry	0.3		0.039 dw	0.282 dw
AS 0647	Oat straw and fodder, dry	0.3		0.039 dw	0.282 dw
GC 0647	Oats	0.04 *		0.035	0.036
VD 0072	Peas (dry)	0.02 *		0.02	0.02
VD 0537	Pigeon pea (dry)	0.15		0.02	0.1
VR 0589	Potato	0.04 *		0.04	0.04
PF 0111	Poultry fats	0.02 *		0	0
PM 0110	Poultry meat	0.02 *		0	0
PO 0111	Poultry, Edible offal of	0.02 *		0	0
GC 0650	Rye	0.04 *		0.035	0.036
AS 0650	Rye straw and fodder, dry	0.3		0.039 dw	0.282 dw
VR 0596	Sugar beet	0.15		0.018	0.086
AV 0596	Sugar beet leaves or tops	3		0.178 dw	2.409 dw
DM 0596	Sugar beet molasses	0.3		0.048	0.228
AB 0596	Sugar beet pulp, dry	0.3		0.058	0.275
SO 0702	Sunflower seed	0.04 *		0.04	0.04
VO 0447	Sweet corn (corn-on-the-cob)	0.04 *		0.04	0.04
	Sweet corn fodder	1.5		0.084 dw	1.096 dw
GC 0657	Teosinte	0.04 *		0.035	
AS 0657	Teosinte fodder	0.3		0.039 dw	0.282 dw
GC 0653	Triticale	0.04 *		0.035	

GC 0654	Wheat	0.02 *		0.02	
AS 0654	Wheat straw and fodder, dry	0.2		0.034 dw	0.114 dw
GC 0655	Wild rice	0.04 *		0.035	
	Sugar beet, refined sugar			0.0068	
OC 0702	Sunflower seed oil, edible			0.0088	
	Legume forage			0.333 dw	1.9 dw
AF 0647	Oat forage (green)			0.13 dw	0.40 dw
AF 0650	Rye forage (green)			0.13 dw	0.40 dw
	Sugar beet, refined sugar			0.0068	
	Sweet corn forage			0.25 dw	2.02 dw
	Wheat forage			0.24 dw	1.88 dw

DIETARY RISK ASSESSMENT

Long-term intake

The 2015 JMPR established an Acceptable Daily Intake (ADI) of 0–0.01 mg/kg bw for acetochlor.

The evaluation of acetochlor 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 results are shown in Annex 3 to the 2015 Report.

The IEDIs in the seventeen Cluster Diets, based on the estimated STMRs were 0–4% of the maximum ADI (0.01 mg/kg bw). The Meeting concluded that the long-term intake of residues of acetochlor from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The 2015 JMPR established an Acute Reference Dose (ARfD) of 1 mg/kg bw for acetochlor. The IESTI of acetochlor for the commodities for which STMR, HR and maximum residue levels were estimated by the current Meeting are shown in Annex 4 to the 2015 Report. The IESTI represented 0–0% of the ARfD.

The Meeting concluded that the short-term intake of residues of acetochlor resulting from uses that have been considered by the JMPR is unlikely to present a public health concern.

BIFENTHRIN (178)

The first draft was prepared by Professor Mi-Gyung Lee, Andong National University, Republic of Korea

EXPLANATION

Bifenthrin is a pyrethroid insecticide and miticide. It was first evaluated for residues and toxicology by the JMPR in 1992 and re-evaluated in 2009 (T) and 2010 (R). The 46th Session of the CCPR (2014) listed bifenthrin for the evaluation of additional MRLs.

Currently, an ADI of 0–0.01 mg/kg bw and an ARfD of 0.01 mg/kg bw are established. The residue definition for compliance with the MRL and for estimation of dietary intake (for animal and plant commodities) is bifenthrin (sum of isomers). The residue is fat-soluble.

The Meeting received information on supervised residue trials for blueberries, grapes, head lettuce, spinach, celery, peas, snap beans and lima beans.

Analytical methods

Grape, Head lettuce (IR-4 trial), Spinach (manufacturer), Celery, Peas, Snap bean, Lima bean

Analytical methods used for analysis of bifenthrin residues involved an extraction with acetone, an aqueous/acetone partition, a clean-up using florisil and analysis by GC-ECD (Ref. method, Ridler 1989, Report P-2132M; evaluated acceptable for recoveries of the residue in maize samples by 2010 JMPR). For spinach, 4'-hydroxy bifenthrin was analysed as well as bifenthrin using the analytical method. For lima bean (seed), methylene chloride was used instead of hexane in the partition step. The limit of quantification (LOQ) of bifenthrin was 0.05 mg/kg in all matrices. The recoveries and CV (%) values at various fortification levels were in an acceptable range of 70–120% and 20%, respectively, with exceptions of head lettuce 23% CV and celery 25% CV at a fortification level of 0.05 mg/kg. The LOQ of 4'-hydroxy bifenthrin in spinach was 0.05 mg/kg and recoveries at fortification levels of 0.05–2.0 mg/kg were in a range of 79–100% (in total, n=9).

Blueberry, Spinach (IR-4 trial)

Bifenthrin residues were extracted with hexane in an automated extraction unit. The extract was cleaned up on a florisil column and subjected to GC-ECD for analysis. For spinach, the hexane extract was subjected to GC-ECD, omitting the florisil clean-up step. The LOQ was 0.05 mg/kg in the matrices. Recoveries at fortification levels of 0.05, 0.5 and 2.0 or 5.0 mg/kg were in a range of 80–100% (CV, < 5.3%).

Head lettuce (manufacturer)

Bifenthrin residues in head lettuce were extracted with acetone after adding sodium chloride. The extract was partitioned with hexane and cleaned up using an aminopropyl SPE cartridge. GC-MSD was used for determination of the analyte and the LOQ was 0.05 mg/kg. At four fortification levels in the range of 0.05–1.0 mg/kg, recoveries of bifenthrin were 90–120% (in total, n=6; CV, 16%).

A summary of recovery data with the methods used for residue trial samples in this submission are shown in Table 1.

Table 1 Analytical recoveries of bifenthrin in some plant commodities

Matrix	Fortification, mg/kg	n	Range of recoveries, %	Mean recovery, %	CV, %	Ref. method
Blueberry	0.05	3	90–100	96	5.3	
	0.5	4	84–94	89	5.2	

Matrix	Fortification, mg/kg	n	Range of recoveries, %		Mean recovery, %		CV, %		Ref. method
	5.0	3	93–96		94		2.1		
Grape [a]	0.05	7	73–112, 63 (one value)		94		20		Report P-2132M
	0.1	1	110		110		–		
	0.5	6	89–112		100		11		
	1.0	1	98		98		–		
	5	6	73–99		90		14		
	12	1	121		121		–		
Head lettuce (IR-4) [b]	0.05	6	77–111, 152 (one value)		104		23		Report P-2132M
	0.1	4	71–104		87		16		
	0.5	1	96		–		–		
	1.0	4	78–97		90		11		
	5.0	1	95		–		–		
Head lettuce (manufacturer) [b]	0.05	3	90–120		102		16		
	0.1	1	110		–		–		
	0.2	1	95		–		–		
	1.0	1	94		–		–		
Spinach(IR-4) [b]	0.05	3	80–82		81		1.2		
	0.5	3	84–91		88		3.4		
	2.0	3	92–94		93		1.1		
Spinach (manufacturer) [b, c]	0.05	2	101, 103	91, 100	102	96	–	–	Report P-2132M[d]
	0.25	2	100, 107	79, 97	104	88	–	–	
	0.5	2	91, 99	79, 90	95	80	–	–	
	1.0	2	93, 107	61 (90), 81	100	71 (86)	–	–	
	2.0	1	97	94	97	94	–	–	
Celery (IR-4)	0.05	6	77–86		80		2.9		Report P-2132M
	0.5	3	88–99		95		6.4		
	5.0	3	94–100		97		3.1		
Celery (IR-4)	0.04	3	92–118		106		12		Report P-2132M
	0.05	3	69–109		84		25		
	0.4	3	89–94		91		3.3		
	4.0	3	78–103		89		13		
Peas with pods [b]	0.05	5	83–90		86		3.3		Report P-2132M
	0.5	2	79, 106		93		–		
	1.0	2	71, 76		74		–		
	2.0	3	88–91		90		1.7		Report P-2132M
Peas without pods [b]	0.05	2	84, 86		85		–		
	0.2	1	75		75		–		
	0.5	3	76–79		78		1.9		
Snap bean with pods [b]	0.05		105–119		109		8.0		Report P-2132M

Matrix	Fortification, mg/kg	n	Range of recoveries, %	Mean recovery, %	CV, %	Ref. method
	0.5		88–116	103	14	
	0.6		98	–	–	
	5.0		101–111	105	5.2	
Lima bean, seed	0.05		87–105	93	11	Report P-2132M
	0.5		104–108	106	1.9	

LOQs, < 0.05 mg/kg

[a] Including concurrent recoveries

[b] Concurrent recoveries

[c] Recoveries underlined are for 4'-hydroxy bifenthrin. The value in parenthesis is from a repeat analysis.

[d] In the study report, P-2715 was mentioned as a reference method, however, the used method was very similar to the P-2132M.

Stability of residues in stored analytical samples

The 2010 JMPR evaluated that bifenthrin residues were stable for the period of at least 18 months in oranges, 49 months in apples, 7 months in strawberries, 24 months in bananas, 36 months in lettuce, potatoes and pecans, 15 months in peas, dry, 34 months in maize grain and up to 24 months in cotton seed.

In this Meeting, additional information was available that showed the residues were stable for at least 176 days in grapes, 300 days in head lettuce, 561 days in celery, 210 days in peas with pods, 142 days in snap beans and up to 196 days in lima beans. In these storage stability tests, zero-day residues were not determined, except for lima beans.

Based on the available information, it is considered that residues in all samples relevant to this submission were stable under frozen conditions until extraction and analysis. Table 2 includes results of storage stability tests and actual storage days for field trial samples.

Table 2 Storage stability of bifenthrin in some plant matrices

Matrix	Fortification level, mg/kg	Tested storage days	Residue in fortified samples, mg/kg	Procedural recoveries, %	Actual max. storage days
Blueberry	–	–	–	–	81
Grape	0.10	176	0.080	110	172
	10	176	8.7	121% at 12 ppm	
Head lettuce	0.05	300	0.062	152	280 days or 11 months
	0.1	300	0.11	104	
	0.5	300	0.55	97% at ca. 1 ppm	
Spinach	–	–	–	–	57 days or 4 months
Celery	0.5	561	0.38, 0.38, 0.30 (60%)	69, 76, 109% at 0.05 ppm	229 or 349
Peas with pods	0.05	192	0.030 (58%), 0.049	88	178
	0.5	192	0.34 (68%), 0.39	106	
	0.16 [a]	210	0.17	[b]	
	0.17 [a]	210	0.15	[b]	
Snap bean	0.5	142	0.38, 0.47, 0.36	98% at 0.6 ppm	135 or 150
Lima bean, seed	0.5	1	0.52, 0.53, 0.54	81% at 0.05 ppm	61
		35	0.54, 0.54	81% at 0.05 ppm	
		68	0.49, 0.50, 0.50	87% at 0.05 ppm	
		98	0.42, 0.46, 0.46	61% at 0.05 ppm	

Matrix	Fortification level, mg/kg	Tested storage days	Residue in fortified samples, mg/kg	Procedural recoveries, %	Actual max. storage days
		117	0.48, 0.48, 0.48	195 at 0.05 ppm	
		196	0.40, 0.43, 0.46	120% at 0.05 ppm	

[a] Pea samples from a field residue trial were analysed again seven months after the initial analysis. The initial residue concentrations were 0.16 mg/kg and 0.17 mg/kg, with overall concurrent recoveries of 79–91% at fortification levels of 0.05, 0.5 and 2.0 mg/kg.

[b] Mean procedural recoveries were 88% and 106% at fortification levels of 0.05 mg/kg and 0.5 mg/kg, respectively. At the same date, fortified sample at 192 days and field trial sample at 210 days were analysed.

USE PATTERNS

Bifenthrin is registered in many countries for control of insect pests on fruit, vegetables, cereals, oilseeds and forage crops. This Meeting received information on registered uses from the USA regarding the submitted residue trial, which is summarized in the Table 3.

Table 3 Registered uses of bifenthrin in the USA on crops relevant to this submission

Crop	Form.	Method	Application			
			Rate, kg ai/ha	Max. no.	Interval days	PHI, days
Bushberries (blueberry)	WSB, 2EC	Foliar, G or A [a]	0.11 (0.56 kg ai/ha/season)		7	1
Grapes	WSB, 2EC	Foliar, G or A	0.11 (0.11 kg ai/ha/season)			30
Leafy petiole vegetables (celery)	WSB, 2EC	Foliar, G or A	0.11 (0.56 kg ai/ha/season)		7	7
Lettuce, head	WSB, 2EC	Foliar, G or A	0.11 (0.56 kg ai/ha/season)		7	7
Spinach	WSB, 2EC	Soil at planting; foliar, G or A	0.11 (0.45 kg ai/ha/season)	4	7	40
Succulent peas and beans (pea, snap bean, lima bean)	WSB, 2EC	At planting time [b]; foliar use, G or A [b]	0.11 (0.22 kg ai/ha/season)			3

Succulent peas and beans include as follows: pea (Pisum spp.: dwarf pea, English pea, garden pea, etc.), bean (Phaseolus spp.: broadbean, succulent, lima bean, green, snap bean, etc.), bean (Vigna spp.: asparagus bean, cowpea, moth bean, etc.), jackbean, soybean, immature seed and sword bean.

Bushberries include blueberry, high-bush and low-bush, currant, elderberry, gooseberry and huckleberry.

Leafy petiole vegetables include celery, cardoon, Chinese celery, celtuce, Florence fennel, rhubarb, Swiss chard.

The formulation, WSB (water soluble bags; ai, 10%) is a type of wettable powder. The formulation 2EC is a type of emulsifiable concentrate (ai, 25.1%).

[a] By ground or air

[b] Apply in-furrow with the seed or transplant

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Supervised residue trials were conducted in the USA for the following crops: blueberry, grape, head lettuce, spinach, celery, peas, snap bean and lima bean. The results of these residue trials are summarized in the following tables.

Crop group	Commodity	Table No.
Berries and other small fruits	Blueberry	4
	Grape	5
Leafy vegetables	Head lettuce	6
	Spinach	7

Crop group	Commodity	Table No.
Stalk and stem vegetables	Celery	8
Legume vegetables	Peas	9, 10
	Snap bean	11
	Lima bean	12

In all trials, there were no residues detected above the LOQ, 0.05 mg/kg, in control samples. Procedural recoveries of bifenthrin residues were satisfactory in all analytical sets. Bifenthrin residues were demonstrated to be stable for the period of frozen storage for all samples (See section of stability of residue in stored analytical samples).

For estimation of a maximum residue level, residue values from the trials conducted according to the maximum GAP were used. In cases where multiple samples were taken from a single plot, the mean residue from that plot was selected. In cases where separate plots were found not to be independent, the highest residue value was selected for estimating a maximum residue level. Those selected values are underlined in the tables.

Berries and Other Small Fruit

Bushberries—Blueberry

Nine trials were conducted in the USA (CA, ME, MI, NC, NJ and OR) in 2004. At each trial, five foliar applications, 6–8 days apart, were made with the 2 EC formulation (emulsifiable concentrate, 240 g/L), except in Trial ID, NJ10 (in which two of the applications were made at 4-day intervals because the crop was maturing rapidly). In four of the trials (ME01, MI06, NC10, and OR06), separate plots received treatments with the 10 WP formulation (10% wettable powder) as five foliar applications, 6–8 days apart. In all treatments, the application rate was 0.11–0.12 kg ai/ha (0.55–0.57 kg ai/ha/season). Samples of blueberries were collected 1 day after the last application.

Table 4 Residues resulting from bifenthrin application to blueberries in the USA (Report: IR-4 PR No. 08736)

Location (Variety) Year	Application				DALA	Residue, mg/kg			Trial ID
	Form.	kg ai/ha	No.	Inter. days		Repl.1	Repl. 2	Mean	
GAP, USA		0.11 (0.56 kg ai/ha /season)		7	PHI, 1 days				
Tulare, CA (Misty) 2004	2 EC	0.11–0.12	5	7	1	0.50	0.36	0.43	CA33
Jonesboro, ME (Lowbush) 2004	2 EC	0.11	5	6–8	1	1.1	1.6	1.4	ME01
	10 WP	0.11	5	6–8	1	0.64	1.0	0.84	
Fennville, MI (Rubel) 2004	2 EC	0.11	5	7	1	0.95	0.88	0.92	MI06 [a]
	10 WP	0.11	5	6–7	1	1.1	0.76	0.91	
Fennville, MI (Rubel) 2004	2 EC	0.11	5	7	1	0.87	1.4	1.2	MI07 [a]
Fennville, MI (Rubel) 2004	2 EC	0.11	5	6–8	1	0.80	0.96	0.88	MI08 [a]
Castle Hayne, NC (Croatan) 2004	2 EC	0.11	5	7	1	0.42	0.54	0.48	NC10

Location (Variety) Year	Application				DALA	Residue, mg/kg			Trial ID
	Form.	kg ai/ha	No.	Inter. days		Repl.1	Repl. 2	Mean	
	10 WP	0.11	5	7	1	0.48	0.39	0.44	
Bridgeton, NJ (Blueray) 2004	2 EC	0.11–0.12	5	6	1	0.71	0.60	0.66	NJ09 [b]
Bridgeton, NJ (Duke) 2004	2 EC	0.11–0.12	5	4–6	1	0.79	0.89	0.84	NJ10 [b]
Aurora, OR (Bluecrop) 2004	2 EC	0.11–0.12	5	6–8	1	0.52	0.48	0.50	OR06
	10 WP	0.11	5	6–8	1	0.43	0.37	0.40	

2 EC (emulsifiable concentrate; ai, 25.1%), 10 WP (wettable powder, 10%)

Duplicate sampling in each trial was made.

[a, b] The trials were conducted at the same site and on the same dates of application.

Small fruit vine climbing—Grapes

Seven trials on grapes were conducted in the USA (NC, MI, WA, NJ, NY and OH) from 1994 to 1996. One foliar application (2 EC) was made at the rate of 0.10 or 0.11 kg ai/ha. Grape samples were harvested 28 to 32 days after the application.

Table 5 Residues resulting from bifenthrin application to grapes in USA (Report: IR-4 PR No. 05335)

Location (Variety) Year	Application		DALA	Residue, mg/kg [a]			Trial ID
	kg ai/ha	No.		Repl.1	Repl. 2	Mean	
GAP, USA	0.11 (0.11 kg ai/ha/season)		PHI, 30 days				
Raleigh, NC (Muscadine) 1994	0.11	1	29	0.11	0.14	0.13	NC14
Fennville, MI (Concord) 1994	0.11	1	30	< 0.05	< 0.05	< 0.05	MI30
Prosser, WA (Concord) 1994	0.11	1	28	< 0.05	0.070	0.060	WA50
Ferrell, NJ (Concord) 1995	0.11	1	32	0.050	0.050	0.050	NJ29
Mattituck, NY (Chardonnay) 1995	0.11	1	31	0.10	0.13	0.12	NY14
Wooster, OH (Ives) 1995	0.11	1	29	0.060	0.060	0.060	OH*25
Riverhead, NY (Lemberger) 1996	0.10	1	29	0.070	0.070	0.070	NY02

[a] 2 EC was applied and duplicate sampling in each trial was made.

Leafy vegetables

Lettuce, head

Six residue trials were conducted in the USA (CA, OH, NJ, FL and TX) in 1993 and 1994. At each trial, five applications (six at TX*25) timed 5–11 days apart were made with the 2 EC formulation.

The rate of application was approximately 0.11 kg ai/ha (0.55 kg ai/ha/season; 0.66 kg ai/ha/season for TX*25 trial). Head lettuce samples with and without wrapper leaves were taken 7–8 days after the last treatment.

The 'Mesa' variety used in Trial NJ17 has a 'frilled' morphology, which suggests that the leaf edges may not stay as close to the head as in other varieties, perhaps resulting in water and residues being retained.

In 2004, four additional trials were conducted in California and Arizona. At each trial, five foliar applications were made at a rate of 0.11 kg ai/ha (0.55 kg ai/ha/season), 5–10 days apart, with the 2 EC formulation. Samples with and without wrapper leaves were collected 6–8 days after the last treatment. In Trial 04, additional samples were collected at 1, 3 and 14 days after the last application to determine the residue decline pattern of bifenthrin.

Table 6 Residues resulting from bifenthrin application to head lettuce in the USA (Report: IR-4 PR No. 05274; P-3723)

Location (Variety) Year	Application			DALA	Portion analysed	Residue, mg/kg			Trial ID
	kg ai/ha	No.	Inter. days			Repl.1	Repl. 2	Mean	
GAP, USA	0.11 (0.56 kg ai/ha /season)		7	PHI, 7 days					
Salinas, CA (Salinas) 1993	0.11	5	6–7	7	w/ w. leaves	0.78	0.84	0.81	CA*46
					wo/ w. leaves	< 0.05	0.09	0.070	
Willard, OH (Ithaca) 1993	0.11	5	6–8	7	w/ w. leaves	0.38	0.51	0.45	OH*12
					wo/ w. leaves	< 0.05	< 0.05	< 0.05	
Bridgeton, NJ (Mesa) [a]1993	0.11	5	7–9	7	w/ w. leaves	1.7	1.8	1.8	NJ17
					wo/ w. leaves	0.48	0.85	0.67	
Zellwood, FL (South Burg) 1993	0.11	5	6–9	8	w/ w. leaves	0.56	0.85	0.71	FL42
					wo/ w. leaves	< 0.05	< 0.05	< 0.05	
Weslaco, TX (Golden State) 1993	0.11	6	7–11	8	w/ w. leaves	1.6	1.9	1.7	TX*25
					wo/ w. leaves	< 0.05	< 0.05	< 0.05	
Holtville, CA (Empire) 1994	0.11	5	5–10	7	w/ w. leaves	0.33		0.33	CA19
					wo/ w. leaves	< 0.05		< 0.05	
San Ardo, CA (Shape Shooter) 2003	0.11	5	5–6	8	w/ w. leaves	< 0.05	< 0.05	< 0.05	01
					wo/ w. leaves	< 0.05	< 0.05	< 0.05	
Hughson, CA (Bayview) 2003	0.11	5	5	6	w/ w. leaves	0.21	0.25	0.23	02
					wo/ w. leaves	0.20	0.24	0.22	
Yuma, AZ (Telluride) 2004	0.11	5	5–10	7	w/ w. leaves	0.54	0.58	0.56	03
					wo/ w. leaves	0.38	0.41	0.40	

Location (Variety) Year	Application			DALA	Portion analysed	Residue, mg/kg			Trial ID
	kg ai/ha	No.	Inter. days			Repl.1	Repl. 2	Mean	
Visalia, CA (Salinas M.I.) 2004	0.11	5	7	1	w/ w. leaves	0.39	0.43	0.41	04
					wo/ w. leaves	0.40	0.41	0.41	
				3	w/ w. leaves	0.14	0.16	0.15	
					wo/ w. leaves	0.14	0.14	0.14	
				7	w/ w. leaves	0.11	0.14	0.13	
					wo/ w. leaves	0.14	0.14	0.14 [b]	
				14	w/ w. leaves	0.070	0.080	0.075	
					wo/ w. leaves	0.070	0.080	0.075	

2 EC formulation was used and duplicate sampling in each trial was made.

Residues were analysed for heads with wrapper leaves and heads without wrapper leaves.

[a] Variety with a “frilled” appearance

[b] Higher residue value was selected.

Spinach

Five trials were conducted in the USA (MD, NJ and TX) in 1999. At each trial, foliar spray was made once at a rate of 0.45–0.47 kg ai/ha with the 2 EC formulation. A single treatment was made because spinach in some areas developed too rapidly to accommodate a use pattern of four applications and a 40-day PHI. Spinach samples were taken 36–41 days after the application.

Three additional trials were conducted in California and Arizona in 1999. The 2 EC formulation was applied to spinach as four foliar sprays (aerial or ground spray) 4–13 days apart. Each application was at 0.11 kg ai/ha (0.55 kg ai/ha/season), and spinach samples were collected 20, 39 or 40 days after the last application. In these three trials, the metabolite 4'-hydroxy bifenthrin was also analysed along with bifenthrin.

Table 7 Residues resulting from bifenthrin application to spinach in the USA (Report: IR-4 PR No.07088; P-2839)

Location (Variety) Year	Application			DALA	Residue, mg/kg			Trial ID
	kg ai/ha	No.	Inter. days		Repl.1	Repl. 2	Mean	
GAP, USA	0.11 (0.45 kg ai/ha/season)		7	PHI, 40 days				
Salisbury, MD (Vienna) 1999	0.454	1		37	< 0.05	< 0.05	< 0.05	MD01
Bridgeton, NJ (Melody) 1999	0.448	1		36	< 0.05	< 0.05	< 0.05	NJ07
Weslaco, TX (Olympia) 1999	0.448	1		41	< 0.05	< 0.05	< 0.05	TX07
Weslaco, TX (Fall Green) 1999	0.448	1		39	< 0.05	< 0.05	< 0.05	TX*08
Weslaco, TX (Olympia) 1999	0.467	1		39	< 0.05	< 0.05	< 0.05	TX24
Yuma, AZ (St. Helens) 1999	0.11	4	7–13	20	0.89	1.4	1.1	01

Location (Variety) Year	Application			DALA	Residue, mg/kg			Trial ID
	kg ai/ha	No.	Inter. days		Repl.1	Repl. 2	Mean	
			4–10	40	0.14	0.16	0.15	
Imperial, CA (St. Helens) 1999	0.11 aerial spray	4	5–8	20	0.44	0.50	0.47	02
Imperial, CA (St. Helens) 1999	0.11 ground spray	4	5–8	20	1.0	1.1	1.0	03
			4–7	39	0.040	0.060	0.050	

2 EC formulation was used and duplicate sampling in each trial was made.

For Trial ID, 01, 02 and 03, the metabolite, 4'-hydroxy bifenthrin was analysed as well as bifenthrin. The metabolite was not determined in any samples, i.e., less than LOQ, 0.05 mg/kg.

Stalk and stem vegetables

Celery

Four trials were conducted in the USA (Florida and California) in 1997 and 1998. Celery plots were treated five times with the 2 EC formulation 6–8 days apart. Foliar application was made at a rate of 0.11–0.12 kg ai/ha (0.55–0.57 kg ai/ha/season). Samples were taken 6–8 days after the last treatment. In one trial (CA*03), additional samples were taken 1, 5, 9 and 14 days after the last treatment.

In 2004, four additional trials were conducted in California, Florida and Ohio. Each trial included two treated plots treated with the 2EC or 10 WP formulation. Five foliar applications, 6–8 days apart, were made at the rate of 0.11–0.12 kg ai/ha (0.56–0.58 kg ai/ha/season). Samples were harvested 6–7 days after the last application.

Table 8 Residues resulting from bifenthrin application to celery in the USA (Report: IR-4 PR No. A4945, B4945)

Location (Variety) Year	Application				DALA	Residue, mg/kg			Trial ID
	Form.	kg ai/ha	No.	Inter. days		Repl.1	Repl. 2	Mean	
GAP, USA		0.11 (0.56 kg ai/ha/ season)		7	PHI, 7 days				
Gainesville, FL (June Belle) 1997	2 EC	0.11	5	6–7	6	0.26	0.31	0.29	FL03
Holtville, CA (Conquistador) 1997–98	2 EC	0.11	5	6–8	8	0.87	0.91	0.89	CA04
Salinas, CA (Conquistador) 1997	2 EC	0.11	5	6–8	1	1.2	2.3	1.8	CA*03
					5	0.74	1.1	0.91	
					7	0.28	0.61	0.45	
					9	0.81	1.1	0.97	
					14	0.66	1.6	1.1	
Salinas, CA (52–75) 1998	2 EC	0.11–0.12		7–8	7	0.11	0.22	0.17	CA*45
Irvine, CA (Conquistador 1703) 2004	2 EC	0.11–0.12	5	6–8	7	1.8	1.2	1.5	CA31

Location (Variety) Year	Application				DALA	Residue, mg/kg			Trial ID
	Form.	kg ai/ha	No.	Inter. days		Repl.1	Repl. 2	Mean	
	10 WP	0.11–0.12		6–8	7	0.97	1.2	1.1	
Salinas, CA (Conquistador) 2004	2 EC	0.11–0.12	5	6–8	6	0.65	0.71	0.68	CA*32
	10 WP	0.11–0.12		6–8	6	0.43	0.47	0.45	
Citra, FL (M9) 2004	2 EC	0.11	5	6–8	7	0.69	0.73	0.71	FL21
	10 WP	0.11		6–8	7	0.58	0.69	0.64	
Celeryville, OH (Ventura) 2004	2 EC	0.11–0.12	5	6–7	7	0.11	0.15	0.13	OH05
	10 WP	0.11–0.12		6–7	7	0.06	0.13	0.095	

2 EC (emulsifiable concentrate, 240 g/L), 10 WP (wettable powder, 10%)

Two sampling in each trial was made and duplicate sampling in each trial was made.

Residues in stalks and leaves were analysed.

Legume vegetables

Peas

Six trials were conducted in the USA (MN, WI, MD, NY and WA) from 1992 to 1994. At each trial, two foliar applications (2 EC) were made with a 7-day retreatment interval at a rate of 0.11 kg ai/ha (0.22 kg ai/ha/season). Peas and shelled pea samples were collected 3 days after the second application. In three of the six trials, forage samples were collected (one trial for hay), however, residues were not analysed.

Table 9 Residues resulting from bifenthrin application to peas (with pods) in the USA (Report: IR-4 PR No. 05237)

Location (Variety) Year	Application			DALA	Portion analysed	Residue, mg/kg			Trial ID
	kg ai/ha	No.	Inter. days			Repl.1	Repl. 2	Mean	
GAP, USA	0.11 (0.22 kg ai/ha /season)			PHI, 3 days					
Springfield, MN (Del Monte 5063) 1992	0.11	2	7	3	w/ pods	0.16	0.17	0.17	MN02
Columbus, WI (DLM 2601) 1992	0.11	2	7	3	w/ pods	0.19	0.48	0.34	WI20
Salisbury, MD (Rigo) 1993	0.11	2	7	3	w/ pods	0.17	0.17	0.17	MD06
Geneva, NY (Wando) 1993	0.11	2	7	3	w/ pods	0.47	0.50	0.49	NY17
Yakima, WA (Puget) 1993	0.11	2	7	3	w/ pods	0.18	0.22	0.20	WA*22
Yakima, WA (Puget) 1994	0.11	2	7	3	w/ pods	0.25		0.25	WA*17

2 EC formulation was used and duplicate sampling in each trial was made.

Table 10 Residues resulting from bifenthrin application to peas (pea, shelled) in the USA (Report: IR-4 PR No. 05237)

Location (Variety) Year	Application			DALA	Portion analysed	Residue, mg/kg			Trial ID
	kg ai/ha	No.	Inter. days			Repl.1	Repl. 2	Mean	
GAP, USA	0.11 (0.22 kg ai/ha /season)			PHI, 3 days					
Springfield, MN (Del Monte 5063) 1992	0.11	2	7	3	wo/ pods	< 0.05	< 0.05	< 0.05	MN02
Columbus, WI (DLM 2601) 1992	0.11	2	7	3	wo/ pods	< 0.05	< 0.05	< 0.05	WI20
Salisbury, MD (Rigo) 1993	0.11	2	7	3	wo/ pods	< 0.05	< 0.05	< 0.05	MD06
Geneva, NY (Wando) 1993	0.11	2	7	3	wo/ pods	< 0.05	< 0.05	< 0.05	NY17
Yakima, WA (Puget) 1993	0.11	2	7	3	wo/ pods	< 0.05	< 0.05	< 0.05	WA*22
Yakima, WA (Puget) 1994	0.11	2	7	3	wo/ pods	< 0.05	< 0.05	< 0.05	WA*17

2 EC formulation was used and duplicate sampling in each trial was made.

Snap bean

Six residue trials on snap beans (beans with pods) conducted in the USA (1996 and 1997), previously evaluated by the 2010 JMPR were re-submitted for evaluation by this Meeting. The 2010 JMPR did not estimate a maximum residue level as the trials submitted were not in accordance with the GAP.

At each trial, three foliar applications (2EC) were made, with 7-day intervals, at a rate of 0.090, 0.090, and 0.045 kg ai/ha for the 1st, 2nd and 3rd applications, respectively. The total rate was 0.23 kg ai/ha during growing the season. The bean samples were harvested 2-4 days after the last application.

Table 11 Residues resulting from bifenthrin application to snap bean in the USA (Report: IR-4 PR No. 06423)

Location (Variety) Year	Application			DALA	Residue, mg/kg			Trial ID
	kg ai/ha	No.	Inter. days		Repl.1	Repl. 2	Mean	
GAP, USA	0.11 (0.22 kg ai/ha/season)			PHI, 3 days				
Live Oak, FL (Magnum) 1996	0.090, 0.090, 0.045	3	7	3	0.12	0.15	0.14	FL50
Kimberly, ID (Idelif) 1996	0.090, 0.090, 0.045	3	7	3	< 0.05	0.05	0.050	ID11
West Lafaytte, IN (Espada) 1996	0.090, 0.090, 0.045	3	7	3	0.050	0.060	0.055	IN04
Geneva, NY (Labrador) 1996	0.090, 0.090, 0.045	3	7	3	0.090	0.13	0.11	NY09
Plower, WI (Del Monte 0488) 1996	0.090, 0.090, 0.045	3	7	3	< 0.05	0.05	0.050	WI16
Charleston, SC	0.090, 0.090, 0.045	3	7	4	< 0.05	< 0.05	< 0.05	SC*09

Location (Variety) Year	Application			DALA	Residue, mg/kg			Trial ID
	kg ai/ha	No.	Inter. days		Repl.1	Repl. 2	Mean	
(Bush Blue Lake) 1997								

Re-submitted data to this Meeting.

2 EC formulation was used and duplicate sampling in each trial was made.

Residues in snap bean with pods were analysed.

Lima bean

Seven residue trials on lima beans, without pods, conducted in the USA (1997), previously evaluated by the 2010 JMPR, were re-submitted for evaluation by this Meeting. The 2010 JMPR did not estimate a maximum residue level as the trials submitted were not in accordance with the GAP.

At each trial, three foliar applications (2EC) were made with 6–7 day intervals at the rate of 0.087–0.091, 0.089–0.092, 0.043–0.047 kg ai/ha for the 1st, 2nd and 3rd applications, respectively. The total rate was 0.22–0.23 kg ai/ha during the growing season. The bean samples were harvested 2–4 days after the last application.

Table 12 Residues resulting from bifenthrin application to lima bean in USA (Report: IR-4 PR No. 06252)

Location (Variety) Year	Application			DALA	Residue, mg/kg			Trial ID
	kg ai/ha	No.	Inter. days		Repl.1	Repl. 2	Mean	
GAP, USA	0.11 (0.22 kg ai/ha/season)			PHI, 3 days				
Parlier, CA (Jackson Wonder) 1997	0.090, 0.090, 0.045	3	6	3	< 0.05	< 0.05	< 0.05	CA01
Salisbury, MD (Maffei) 1997	0.087, 0.092, 0.045	3	7	3	< 0.05	< 0.05	< 0.05	MD01
Bridgeton, NJ (Baby Fordhook) 1997	0.090, 0.090, 0.047	3	6-7	3	< 0.05	< 0.05	< 0.05	NJ01
Charleston, SC (Henderson Bush) 1997	0.090, 0.090, 0.045	3	6-7	4	< 0.05	< 0.05	< 0.05	SC*05
Moxee, WA (Ford Hook 242) 1997	0.091, 0.090, 0.045	3	6	3	< 0.05	< 0.05	< 0.05	WA*10
Hancock, WI (Improved Kingston) 1997	0.090, 0.089, 0.044	3	7	3	< 0.05	< 0.05	< 0.05	WI03
Arlington, WI (Improved Kingston) 1997	0.091, 0.091, 0.043	3	6	2	< 0.05	< 0.05	< 0.05	WI04

Re-submitted data to this Meeting

2 EC formulation was used and duplicate sampling in each trial was made.

The 3rd application rate was 0.045 kg ai/ha in all trials.

Residues in lima bean without pods, were analysed.

APPRAISAL

Bifenthrin is a pyrethroid insecticide and miticide. It was first evaluated for residues and toxicology by the JMPR in 1992 and re-evaluated in 2009 (T) and 2010 (R) under the periodic review

programme of the CCPR. The forty-sixth Session of the CCPR (2014) listed bifenthrin for the evaluation of additional maximum residue levels by the 2015 JMPR.

Currently, an ADI of 0–0.01 mg/kg bw and an ARfD of 0.01 mg/kg bw are established. The residue definition for compliance with the MRL and for estimation of dietary intake (for animal and plant commodities) is bifenthrin (sum of isomers). The residue is fat-soluble.

The Meeting received information on supervised residue trials for blueberry, grape, head lettuce, spinach, celery, peas, snap bean and lima bean.

Methods of analysis

Acceptable analytical methods were developed and validated for determination of bifenthrin in residue trial samples. All methods involved an analysis by GC-ECD, except one method using GC-MSD. The limit of quantification (LOQ) of bifenthrin was 0.05 mg/kg in all matrices.

Stability of residues in stored analytical samples

At the 2010 JMPR, bifenthrin was shown to be stable in lettuce under frozen storage condition for at least 36 months. This Meeting received additional storage stability studies on grape, head lettuce, celery, peas, snap bean and lima bean, showing that bifenthrin was stable for the period of storage of the supervised trial samples. Bifenthrin residues in blueberry (81 days) and spinach (4 months) were considered to be stable for the storage period based on all available information.

Results of supervised residue trials on crops

Berries and Other Small Fruit

Bushberries-Blueberry

Nine trials were conducted in the USA in 2004, matching the US GAP on bushberries (0.11 kg ai/ha with 7-day intervals and a PHI of 1 day; 0.56 kg ai/ha/season). Six independent trials matched the GAP.

Bifenthrin residues in blueberry were (n=6): 0.43, 0.48, 0.50, 0.84, 1.2 and 1.4 mg/kg.

The Meeting estimated a maximum residue level of 3 mg/kg, an STMR of 0.67 mg/kg and an HR of 1.6 mg/kg (based on a highest single sample) for blueberries. The Meeting noted that an extrapolation to the group of bushberries was not possible because of a high acute intake resulting from the consumption of currents.

Small fruit vine climbing-Grapes

Seven trials were conducted in the USA from 1994 to 1996 that matched the US GAP on grapes (0.11 kg ai/ha with a PHI of 30 days; 0.11 kg ai/ha/season).

Bifenthrin residues in grapes were (n=7): < 0.05, 0.050, 0.060, 0.060, 0.070, 0.12 and 0.13 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg, an STMR of 0.060 mg/kg and an HR of 0.14 mg/kg (based on a highest single sample) for grapes.

Leafy vegetables

Lettuce, head

Ten trials were conducted in the USA in 1993–1994 (six trials) and 2003 (four trials), matching the US GAP on lettuce, head (0.11 kg ai/ha with 7-day intervals and a PHI of 7 days; 0.56 kg ai/ha/season).

Bifenthrin residues in head lettuce with wrapper leaves were (n=10): < 0.05, 0.14, 0.23, 0.33, 0.45, 0.56, 0.71, 0.81, 1.7 and 1.8 mg/kg.

The Meeting estimated a maximum residue level of 4 mg/kg for lettuce, head, an STMR of 0.51 mg/kg and an HR of 1.9 mg/kg (based on a highest single sample). However, this would result in an exceedance of the ARfD and an alternative GAP for head lettuce was not identified.

Spinach

Eight trials were conducted in the USA in 1999, two trials of which matched the US GAP on spinach (by ground or aerial spray, a rate of 0.11 kg ai/ha with 7-day intervals and a PHI of 40 days; 0.45 kg ai/ha/season).

Bifenthrin residues were 0.05 and 0.15 mg/kg.

The Meeting did not estimate a maximum residue level as the number of trials was not sufficient.

Stalk and stem vegetables

Celery

Eight trials, including one decline trial, were conducted in 1997 (3 trials), 1998 (one trial) and 2004 (four trials) matching the US GAP on leafy petiole vegetables (0.11 kg ai/ha with 7-day intervals and a PHI of 7 days; 0.56 kg ai/ha/season).

Bifenthrin residues were (n=8): 0.13, 0.17, 0.29, 0.68, 0.71, 0.89, 1.1 and 1.5 mg/kg.

The Meeting estimated a maximum residue level of 3 mg/kg, an STMR of 0.70 mg/kg and an HR of 1.8 mg/kg (based on a highest single sample). However, this would result in an exceedance of the ARfD and an alternative GAP for celery was not identified.

Legume vegetables

Peas

Six trials were conducted in the USA from 1992 to 1994 that matched the US GAP on succulent peas and beans (0.11 kg ai/ha with a PHI of 3 days; 0.22 kg ai/ha/season).

Bifenthrin residues in peas with pods were (n=6): 0.17, 0.17, 0.20, 0.25, 0.34 and 0.49 mg/kg.

The Meeting estimated a maximum residue level of 0.9 mg/kg, an STMR of 0.23 mg/kg and an HR of 0.50 mg/kg (based on a highest single sample) for peas (pods and succulent=immature seed).

Bifenthrin residues in peas without pods were (n=6): < 0.05 (6) 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).

Beans

Data from six trials on snap bean (beans with pods) were re-submitted. The 2010 JMPR did not estimate a maximum residue level as the trials were not conducted in accordance with the US GAP (0.11 kg ai/ha with a PHI of 3 days; 0.22 kg ai/ha/season). The trials were conducted in the USA in 1996 and 1997 with three applications 7 days apart, 0.090 kg ai/ha (1st), 0.090 kg ai/ha (2nd) and 0.045 kg ai/ha (3rd) and with a 3-day PHI. Residue values in snap beans with pods were < 0.05, 0.050, 0.050, 0.055, 0.11 and 0.14 mg/kg.

None of the data matched the GAP and the data were not suitable for application of the proportionality approach.

Data from seven trials on lima bean, without pods (conducted in the USA in 1997) were re-submitted. The 2010 JMPR did not estimate a maximum residue level as the trials were not conducted in accordance with the US GAP. The trials were conducted with three applications (approximately 0.090 kg ai/ha at the 1st and 2nd application, and 0.045 kg ai/ha at the 3rd application), 6–7 days apart, and a 2 to 4-day PHI. Residue concentrations in lima bean, shelled (succulent seeds) were all less than 0.05* mg/kg (n=7).

None of the data matched the GAP and the data were not suitable for application of the proportionality approach.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed in Annex I are appropriate 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 plant and animal commodities: *bifenthrin (sum of isomers).*

The residue is fat-soluble.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FB 0020	Blueberries	3		0.67	1.6
FB 0269	Grapes	0.3		0.06	0.14
VL 0482	Lettuce, Head	4 [a]		0.51	1.9
VS 0624	Celery	3 [a]		0.7	1.8
VP 0063	Peas (pods and succulent=immature seed)	0.9		0.23	0.5
VP 0064	Peas, shelled	0.05*		0	

[a] On the basis of information provided to the JMPR it was concluded that the estimated short-term intake of bifenthrin for the consumption of head lettuce and celery may present a public health concern

DIETARY RISK ASSESSMENT

Long-term intake

The 2009 JMPR established an ADI of 0–0.01 mg/kg bw for bifenthrin.

The International Estimated Daily Intakes (IEDIs) of bifenthrin were calculated for the 17 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the current and previous Meeting. The results are shown in Annex 3 to the 2015 JMPR Report.

The calculated IEDIs were 9–30% of the maximum ADI. The Meeting concluded that the long-term intake of residues of bifenthrin from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The 2009 JMPR established an ARfD of 0.01 mg/kg bw for bifenthrin. The International Estimated Short Term Intakes (IESTIs) for bifenthrin were calculated for the food commodities using HRs/STMRs estimated by the current Meeting. The results are shown in Annex 4 to the 2015 JMPR Report.

For celery the IESTI represented 600% and 360% of the ARfD for children and general population, respectively. For head lettuce the IESTI represented 430% and 190% of the ARfD for children and general population, respectively. No alternative GAP for celery and head lettuce was available. On the basis of information provided to the JMPR, the Meeting concluded that the short-term intake of residues of bifenthrin from consumption of celery and head lettuce may present a public health concern.

Estimates of intake for the other commodities considered by the 2015 JMPR were within 0-100% ARfD. The Meeting concluded that the short-term intake of bifenthrin for the other commodities is unlikely to present a public health concern when bifenthrin is used in ways that were considered by the Meeting.

REFERENCES

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P-2839	Kim, I	1993	Magnitude of the Residue of Bifenthrin and 4-Hydroxy Bifenthrin in/on Spinach Treated with Capture 2EC Insecticide-Miticide. FMC Corporation, Agricultural Products Group, not published
P-2132M	Ridler, JE	1989	Analytical method for the determination of bifenthrin in/on various crops and soils. FMC Corporation, Agricultural Products Group, not published
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IR-4 PR No. 08736	Samoil, K	2006	Bifenthrin: Magnitude of the Residue on Blueberry. IR-4 Project HQ, Rutgers, The State University of New Jersey. GLP, not published
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CHLOROTHALONIL (081)

The first draft was prepared by Mr Christian Sieke, Federal Institute for Risk Assessment, Berlin, Germany

EXPLANATION

Chlorothalonil is a non-systemic fungicide first evaluated by JMPR in 1974 and a number of times subsequently. It was recently reviewed for toxicology by the 2009 and 2010 JMPR within the periodic review program of the CCPR. For the parent substance an ADI of 0-0.02 mg/kg bw and an ARfD of 0.6 mg/kg bw were established. In addition to the parent substance an ADI of 0-0.008 mg/kg bw and an ARfD of 0.03 mg/kg bw were established for the metabolite SDS-3701. In 2010 the JMPR also considered the toxicity of the soil metabolite R611965, however due to the lower toxicity compared to the parent compound, estimation of a separate ADI and ARfD was considered unnecessary.

The 2010 JMPR recommended the following residue definition for chlorothalonil:

Definition of the residue for compliance with MRL for plant commodities: *chlorothalonil*

Definition of the residue for estimation of dietary intake for plant commodities: *chlorothalonil*

SDS-3701 (2,5,6-trichloro-4-hydroxyisophthalonitrile), all considered separately.

Definition of the residue for compliance with MRL and for estimation of dietary intake for animal commodities: *SDS-3701 (2,5,6-trichloro-4-hydroxyisophthalonitrile)*.

In 2012 the JMPR evaluated additional uses for chlorothalonil in banana, chard, chicory, endive, spring onion, spinach, and peas.

The current Meeting received new information on use patterns for chlorothalonil in multiple crops supported by additional analytical methods, storage stability data and supervised field trials.

RESIDUE ANALYSIS

Analytical methods

For chlorothalonil and its metabolite SDS-3701 two additional analytical methods were provided for plant matrices.

Method GRM005.01A (Chaggar, 2006, CLTA10_269 & CLTA10_270)
Crop samples are extracted by homogenisation with acetone; 5M sulphuric acid solution (95:5 v/v) and then centrifuged. For chlorothalonil determination, aliquots were diluted with water followed by solid phase extraction (SPE) clean-up. Chlorothalonil was analysed by gas chromatography with mass selective detection (GC-MSD). For the determination of R182281, aliquots were diluted with acetonitrile:water and quantified by high performance liquid chromatography with triple-quadrupole mass spectrometric detection (LC-MS/MS). Target markers are m/z: 266 → 264 and m/z: 266 → 268 for chlorothalonil and m/z: 245 → 182 and m/z: 245 → 175 for SDS-3701.

Table 1 Recovery data for method GRM005.01A measuring chlorothalonil and SDS-3701 plant matrices

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Analyte, reference
Apple	0.01	5	92-98	95	2	Chlorothalonil, Chaggar, 2006, CLTA10_269 & CLTA10_270, m/z: 266 → 264
	0.1	5	75-81	78	4	
Peach	0.01	5	103-109	105	2	
	0.1	5	91-111	100	8	
Grape	0.01	5	83-94	88	6	
	0.1	5	96-103	100	3	

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Analyte, reference
Strawberry	0.01	5	88-100	93	5	
	0.1	5	91-106	99	6	
Orange, skin	0.01	5	86-95	92	4	
	0.1	5	83-91	88	4	
Orange, flesh	0.01	5	72-92	85	9	
	0.1	5	92-98	94	3	
Olive	0.01	5	77-85	81	4	
	0.1	5	76-80	78	2	
Banana, skin	0.01	5	92-97	95	2	
	0.1	5	96-105	101	3	
Banana, flesh	0.01	5	99-103	101	1	
	0.1	5	99-110	105	4	
Potato, tuber	0.01	5	66-77	72	6	
	0.1	5	92-101	96	4	
Carrot	0.01	5	97-104	100	3	
	0.1	5	90-104	99	5	
Onion	0.01	5	94-100	96	3	
	0.1	5	84-105	96	8	
Cabbage	0.01	5	90-96	94	2	
	0.1	5	84-96	94	4	
Cauliflower	0.01	5	103-114	108	4	
	0.1	5	97-107	101	4	
Leek	0.01	5	79-99	89	9	
	0.1	5	88-97	93	4	
Pea, fresh seed	0.01	5	80-102	92	9	
	0.1	5	77-91	86	6	
Pea, dry seed	0.01	5	90-102	96	4	
	0.1	5	99-107	104	3	
French bean	0.01	5	69-87	79	11	
	0.1	5	77-87	82	4	
Tomato	0.01	5	77-82	79	3	
	0.1	5	84-86	85	1	
Melon, flesh	0.01	5	90-124	100	14	
	0.1	5	85-92	86	8	
Cereal, grain	0.01	5	79-94	86	8	
	0.1	4	102-109	106	2	
Cereal, straw	0.01	5	85-94	90	4	
	0.1	5	93-97	95	2	
Cereal, forage	0.01	5	95-104	101	4	
	0.1	5	93-103	98	4	
Potato, foliage	0.01	5	88-110	95	9	
	0.1	5	81-99	91	8	
Peanut, nutmeat	0.01	5	84-92	88	4	
	0.1	5	85-91	89	3	
Melon, flesh	0.01	5	91-113	100	9	
	0.1	5	87-100	92	6	
Wheat, grain	0.01	5	98-108	105	5	SDS-3701, Chaggar, 2006, CLTA10_269 & CLTA10_270, m/z: 245 → 182
	0.1	5	95-109	100	7	
Wheat, straw	0.01	5	84-96	87	10	
	0.1	5	87-102	95	7	
Leek	0.01	5	85-120	91	19	
	0.1	5	76-95	88	8	
Cabbage	0.01	5	101-114	108	5	
	0.1	5	97-109	104	5	
Olive	0.01	5	82-104	94	11	
	0.1	5	93-99	95	2	
Oranges	0.01	5	94-108	103	5	
	0.1	5	87-104	96	7	
Wheat, grain	0.01	5	94-115	105	8	SDS-3701, Chaggar, 2006, CLTA10_269 & CLTA10_270, m/z: 245 → 175
	0.1	5	94-112	101	6	
Wheat, straw	0.01	5	82-98	94	7	
	0.1	5	88-96	94	4	

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Analyte, reference
Leek	0.01	5	76-96	90	10	
	0.1	5	78-94	87	8	
Cabbage	0.01	5	96-119	106	10	
	0.1	5	103-111	98	3	
Olive	0.01	5	84-112	101	11	
	0.1	5	95-101	98	3	
Oranges	0.01	5	96-121	105	9	
	0.1	5	98-107	102	3	

Method "Cornell Laboratory" (Thompson, 2007, CLTA10_277 & CLTA10_278)
Crop samples are ground whilst frozen, then extracted with acidified acetone. Extracts are partitioned against petroleum ether, the organic phase containing chlorothalonil and the aqueous SDS-3701. The organic phase is evaporated and the residue cleaned up on a Florisil column, eluting with dichloromethane/hexane and dichloromethane/hexane/acetonitrile. The aqueous phase is adjusted to a pH below 2 and extracted with ether. The sample is then methylated with diazomethane and cleaned up on an alumina column, eluting with dichloromethane. The organic and aqueous extracts were analysed by GC/EC to determine residues of chlorothalonil and SDS-3701 respectively.

Table 2 Recovery data for method "Cornell Laboratory" in plant matrices by GC-ECD

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Analyte, reference
Bell pepper	0.03	7	83-133	117	19	Chlorothalonil
	0.5	16	80-124	92	15	
	1	10	84-94	91	3	
	2	13	80-115	98	13	
	3	1	87	-	-	
	4	1	80	-	-	
Non-bell pepper	0.03	3	80-93	87	7	
	0.5	5	84-92	89	4	
	2	7	85-100	91	5	
Horseradish	0.02	6	70-100	81	17	
	0.2	3	75-85	80	6	
	2	3	78-84	81	4	
Rhubarb	0.02	7	90-120	103	11	
	0.2	3	80-90	85	6	
	1	3	84-86	85	1	
	5	3	100	100	0	
Bell pepper	0.03	35	61-141	98	22	SDS-3701
	0.5	4	122-140	130	7	
	2	3	130-145	138	6	
Non-bell pepper	0.03	9	63-110	84	19	
	0.5	3	70-86	80	11	
Horseradish	0.02	6	85-105	95	8	
	0.2	3	80-95	88	9	
	2.0	3	99-100	100	1	
Rhubarb	0.02	6	90-110	100	9	
	0.2	3	95-100	98	3	
	1	3	92-100	95	5	

Stability of pesticides in stored analytical samples

Plant matrices

Two additional studies on the storage stability of chlorothalonil and SDS-3701 in stored plant commodities were submitted for incurred residues and fortified residues in cranberries.

Anderson (2007, CLTA10_271)

A study was conducted to investigate the storage stability of field-incurred residues of chlorothalonil and its metabolite SDS-3701 in a wide range of crops (tomato, cucumber, whole melon, whole orange, carrot leaves, carrots, barley straw, barley grain and soya bean) when prepared without acidification or the addition of dry ice and stored deep frozen for up to 24 months.

In this study, all field treated crops were prepared by chopping large quantities of semi-frozen crop without acidification or the addition of dry ice. Untreated samples of these matrices were acidified and chopped semi-frozen without the addition of dry ice and used as control samples and procedural recoveries. Field treated and untreated barley grain and soya bean samples were stored frozen and dispensed into sample pots with no preparation; frozen barley straw was chopped into small pieces and finally prepared in a knife mill. The 0 month samples were analysed immediately after preparation and samples for the 3, 6 12 and 24 month storage intervals were stored deep frozen for the appropriate period up to 24 months.

Samples were analysed by method GRM005.01A, using LC-MS/MS.

Table 3 Recovered chlorothalonil and SDS-3701 incurred residues in stored plant commodities after storage up to 24 months (Anderson, 2007, CLTA10_271)

Interval (days)	Chlorothalonil			SDS-3701		
	Recovered residue (mg/kg)	Percent remaining (%)	Procedural recovery (%)	Recovered residue (mg/kg)	Percent remaining (%)	Procedural recovery (%)
Tomato						
0	2.7, 2.8, 3.0 (2.8)	100	89	2×0.007, 3×0.008, 2×0.009, 0.010 (0.008)	100	92
98	2.7, 3.0, 3.1 (3.0)	106	102	0.008, 0.009 (0.008)	102	90
211	1.8, 1.9, 2.1 (1.9)	69	73	0.006, 0.007, 0.007 (0.007)	83	71
385	2.5, 2.6, 2.7 (2.6)	93	90	0.008, 0.01, 0.009 (0.009)	113	108
786	2.3, 2.5, 2.5 (2.5)	88	87	0.008, 0.009, 0.01 (0.009)	110	100
Cucumber						
7	1.5, 1.8, 2.3 (1.9)	100	106	2×0.002, 2×0.003, 3×0.004, 0.005 (0.004)	100	106
104	1.6, 1.6, 1.6 (1.6)	86	99	0.008, 0.010, 0.010 (0.009)	264	103
209	1.4, 1.5, 1.5 (1.5)	78	90	0.014, 0.017, 0.020 (0.017)	482	101
383	1.4, 1.4, 1.5 (1.4)	76	93	0.021, 0.016, 0.021 (0.019)	556	91
784	1.3, 1.3, 1.6 (1.4)	76	85	0.026, 0.024, 0.025 (0.025)	714	98
Melon						
0	0.57, 0.65, 0.65, 0.79, 0.62 (0.66)	100	95	2×0.003, 3×0.005 (0.004)	100	86
99	0.55, 0.52, 1.02 (0.7)	106	97	0.004, 0.003, 0.005 (0.004)	97	103
216	0.7, 0.66, 0.71 (0.69)	104	113	0.005, 0.005, 0.006 (0.006)	140	105
378	0.71, 0.41, 0.51 (0.54)	83	93	0.005, 0.003, 0.006 (0.004)	111	103
779	0.69, 0.53, 0.8 (0.68)	103	96	0.009, 0.008, 0.008 (0.009)	220	106
Orange						
0	11, 8.2, 8.9, 11, 11 (10)	100	87	0.024, 0.014, 0.015, 0.028, 0.029 (0.022)	100	92
102	8.0, 8.7, 8.6 (8.4)	84	100	0.022, 0.021, 0.028 (0.024)	109	94
223	7.6, 8.1, 8.3 (8.0)	80	95	0.020, 0.019, 0.019 (0.019)	86	108
404	8.5, 8.6, 8.2 (8.4)	84	97	0.016, 0.020, 0.018 (0.018)	82	100
788	8.0, 8.5, 7.8 (8.1)	81	97	0.016, 0.017, 0.018 (0.017)	77	94
Carrot roots						
0	0.73, 0.69, 0.70, 0.71, 0.64 (0.69)	100	97	3×0.030, 2×0.033 (0.031)	100	80
97	0.67, 0.62, 0.74 (0.68)	98	91	0.048, 0.042, 0.043 (0.044)	143	104
216	0.60, 0.62, 0.57 (0.6)	86	99	0.050, 0.047, 0.047 (0.048)	154	95
405	0.60, 0.61, 0.60	87	94	0.059, 0.063, 0.061 (0.061)	196	102

Interval (days)	Chlorothalonil			SDS-3701		
	Recovered residue (mg/kg)	Percent remaining (%)	Procedural recovery (%)	Recovered residue (mg/kg)	Percent remaining (%)	Procedural recovery (%)
	(0.6)					
781	0.50, 0.52, 0.53 (0.51)	74	92	0.084, 0.076, 0.081 (0.08)	259	99
Carrot tops						
0	101, 85, 94, 92, 87 (92)	100	93	0.28, 0.24, 0.25, 0.26, 0.26 (0.26)	100	101
92	92, 89, 87 (89)	97	101	0.45, 0.41, 0.42, 0.37, 0.42, 0.36 (0.4)	157	114
211	79, 80, 73 (77)	84	91	0.42, 0.38, 0.38 (0.39)	153	99
400	90, 101, 94 (95)	103	95	0.50, 0.49, 0.51 (0.5)	194	108
784	77, 77, 73 (75)	82	100	0.60, 0.70, 0.58 (0.62)	243	105
Barley straw						
0	25, 25, 28, 24, 26 (26)	100	101	1.1, 4×1.2 (1.2)	100	105
104	21, 21, 20 (20)	80	100	1.3, 1.4, 1.3 (1.3)	111	98
209	18, 18, 20 (18)	72	97	1.4, 1.5, 1.4 (1.4)	121	103
406	19, 18, 17 (18)	70	95	1.6, 1.6, 1.7 1.6)	138	104
790	15, 15, 16 (15)	59	95	1.9, 2.0, 2.0 (2.0)	166	102
840	13, 14, 15 (14)	53	89	1.3, 1.0, 2.0 (1.4)	119	117
Barley grain						
0	0.71, 0.80, 0.73, 0.74, 0.83 (0.76)	100	91	0.052, 0.053, 0.053, 0.056, 0.057 (0.054)	100	90
92	0.82, 0.82, 0.88 (0.84)	110	83	0.066, 0.075, 0.072 (0.071)	131	94
203	0.67, 0.80, 0.65 (0.71)	93	90	0.114, 0.124, 0.112 (0.117)	215	106
391	0.79, 0.76, 0.77 (0.77)	101	94	0.067, 0.069, 0.068 (0.068)	125	94
770	0.81, 0.58, 0.85 (0.74)	98	92	0.089, 0.093, 0.097 (0.093)	172	98
Soya beans						
0	1.4, 1.4, 1.3, 1.3, 1.4 (1.4)	100	84	0.024, 0.021, 0.022, 0.036, 0.032, 0.035, 0.031, 0.032 (0.026)	100	89
91	1.4, 1.3, 1.4 (1.4)	100	73	0.022, 0.020, 0.015 (0.019)	84	105
202	1.5, 1.6, 1.6 (1.6)	115	85	0.026, 0.029, 0.028 (0.027)	122	110
390	1.5, 1.4, 1.4 (1.5)	106	75	0.018, 0.024, 0.020 (0.021)	92	91
770	1.2, 0.69, 0.84 (0.91)	68	83	0.016, 0.014, 0.015 (0.015)	65	93
810	0.97, 1.2, 1.5 (1.2)	88	77	0.022, 0.022, 0.029 (0.024)	100	109

Mean values are expressed in parenthesis

Corley (2013, CLTA10_272)

Samples of cranberries were fortified with either chlorothalonil or the metabolite SDS-3701 at a concentration of 0.2 mg/kg and stored under the same conditions as those used for the residues trials samples, i.e. -20 °C in the dark. Samples were analysed after 295 days of storage. Analysis of the samples was performed according to the method GRM005.01A.

Table 4 Recovered residues in cranberries fortified with chlorothalonil or SDS-3701 at 0.2 mg/kg after storage for 295 days

Analyte	Storage Period (days)	Recovered residue (%)	Mean storage stability recovery (%)	Procedural Recoveries (%)
Chlorothalonil	295	55, 64, 70	63	58-64
SDS-3701		38, 38, 39	38	66-74

USE PATTERN

Chlorothalonil is a non-systemic protectant fungicide. The Meeting received numerous uses involving foliar spray applications mainly before harvest in 2010, amended by additional uses in 2015. The following table lists all additional GAPs only; however the labels provided cover a broader spectrum of uses.

Table 5 List of additional uses of chlorothalonil submitted in 2015

Crop	Country	Application detail					
		Indoor/ Outdoor	Type	kg ai/ha	Growth stage at last treatment	No	PHI
Pome fruit							
Pear	KR	Outdoor	Foliar	0.04 kg ai/hL	At infestation	4	14
Stone fruit							
Cherry	CA	Outdoor	Foliar	4.5	Shuck split (BBCH 71)	3	40
Peaches	CA	Outdoor	Foliar	4.5	Shuck period (BBCH 71)	3	60
Cherry	US	Outdoor	Foliar	3.5	Shuck split (BBCH 71)	4	0
Peaches	US	Outdoor	Foliar	3.5	Shuck split (BBCH 71)	4	0
Berries and other small fruit							
Cranberries	CA	Outdoor	Foliar	5.8	Late bloom	3	50
Cranberries	USA	Outdoor	Foliar	5.5	At infestation	3	50
Bulb vegetables							
Onions, dry	CA	Outdoor	Foliar	2.8	At infestation	3	7
Onions, green	CA	Outdoor	Foliar	2.8	At infestation	5	14
Onion, dry	PL	Outdoor	Foliar	1.0	At infestation	2	14
Leek	US	Outdoor	Foliar	2.5	At infestation	3	14
Onions, dry	US	Outdoor	Foliar	2.5	At infestation	7	7
Onions, green	US	Outdoor	Foliar	2.5	At infestation	3	14
Shallots	US	Outdoor	Foliar	2.5	At infestation	3	14
Fruiting vegetables, other than cucurbits							
Bell pepper	BR	Outdoor	Foliar	0.2 kg ai/hL (up to 1.8 kg ai/ha)	At infestation	2	7
Mushroom	US	Indoor	Soil drench	12.7 + 6.4	Not specified	2	7
Fruiting vegetables (except tomatoes)	US	Outdoor	Foliar	1.3	At infestation	8	3
Tomato	PL	Indoor	Foliar	0.1 kg ai/hL (up to 1 kg ai/ha and application)	At infestation	2	3
Root and tuber vegetables							
Ginseng	CA	Outdoor	Foliar	2.4	At infestation	6	14 (do not feed to livestock)
Ginseng	US	Outdoor	Foliar	1.7	At infestation	8	14
Horseradish	US	Outdoor	Foliar	2.5	At infestation	8	14
Stalk and stem vegetables							
Asparagus	CA	Outdoor	Foliar	1.7	After harvest, to the fern	3	190
Asparagus	US	Outdoor	Foliar	3.4	After harvest, to the fern	3	190
Rhubarb	US	Outdoor	Foliar	2.5	At infestation	6	30
Tree nuts							
Pistachios	US	Outdoor	Foliar	5.0	Full bloom (BBCH 65)	5	14

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Residue levels were reported as measured. Application rates were always reported as chlorothalonil equivalents. When residues were not detected they are shown as below the LOQ, e.g., < 0.01 mg/kg. Application rates, spray concentrations and mean residue results 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.

Chlorothalonil - supervised residue trials

Commodity	**Indoor/Outdoor**	**Treatment**	**Countries**	**Table**
Pear	Outdoor	Foliar	Korea	Table 6
Cherries	Outdoor	Foliar	USA	Table 7
Peaches	Outdoor	Foliar	USA	Table 8
Cranberries	Outdoor	Foliar	USA	Table 9
Onions, bulb	Outdoor	Foliar	USA	Table 10
Onions, green	Outdoor	Foliar	USA	Table 11
Peppers	Outdoor	Foliar	Brazil, USA	Table 12
Tomatoes	Indoor	Foliar	France, Germany, Spain, United Kingdom	Tomatoes Table 13
Mushroom	Indoor	Drench	USA	Table 14
Ginseng	Outdoor	Foliar	USA	Table 15
Horseradish	Outdoor	Foliar	USA	Table 16
Asparagus	Outdoor	Foliar	USA	Table 17
Rhubarb	Outdoor	Foliar	USA	Table 18
Pistachio	Outdoor	Foliar	USA	Table 19

Pear

Table 6 Residues of chlorothalonil and SDS-3701 in pears (HPLC-UV (230nm), LOQ: 0.03 mg/kg (76–110% Recovery, n=5), storage interval: 4 months)

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No., Reference
South Korea, Sangju 2012 (Singo)	SC	4	1.8	0.04	85	Fruit, after removal of hilum and core parts	0 3 7 14 21 28 35	0.82 1.1 0.77 0.59 0.45 0.44 0.3	Not analysed	S-14-04-2-FOD-009-0-D, Trial 1, Park (2014, CLTA10_294)
South Korea, Gyeongju 2012 (Mansu)	SC	4	1.8	0.04	85	Fruit, after removal of hilum and core parts	0 3 7 14 21 28 35	0.9 0.8 0.48 0.45 0.38 0.36 0.25	Not analysed	S-14-04-2-FOD-009-0-D, Trial 2, Park (2014, CLTA10_294)
South Korea, Yesan 2013	SC	4	1.8	0.04	85	Fruit, after removal of hilum and core parts	0 3 7 14	1.0 0.85 0.86 0.68	Not analysed	S-14-04-2-FOD-009-0-D, Trial 3, Park (2014, CLTA10_294) months

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No., Reference
(Singo)							21 28 35	0.39 0.34 0.17		
South Korea, Naju 2013 (Singo)	SC	4	1.8	0.04	85	Fruit, after removal of hilum and core parts	0 3 7 14 21	1.4 1.0 0.98 0.56 0.28	Not analysed	S-14-04-2-FOD-009-0-D, Trial 4, Park (2014, CLTA10_294)
South Korea, Anseong 2013 (Singo)	SC	4	1.8	0.04	85	Fruit, after removal of hilum and core parts Juice	0 3 7 14 21 28 35 14	1.6 1.2 0.87 0.62 0.41 0.34 0.1 0.15	Not analysed	S-14-04-2-FOD-009-0-D, Trial 5, Park (2014, CLTA10_294)
South Korea, Wonju 2013 (Singo)	SC	4	1.8	0.04	85	Fruit, after removal of hilum and core parts	0 3 7 14 21 28 35	2.3 1.6 1.2 0.85 0.49 0.35 0.22	Not analysed	S-14-04-2-FOD-009-0-D, Trial 6, Park (2014, CLTA10_294)

DAT: days after last treatment
BBCH 85: 50% of fruits show typical fully ripe colour

Cherries

Table 7 Residues of chlorothalonil and SDS-3701 in cherries (GRM005.01A, Storage interval: 13-16 months

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No., Reference
USA, Alton (NY) 2013 (Montmorency)	SC	3	3.5	0.37	72	Whole fruit	39	0.038, 0.042 (0.04)	2× < 0.01 (< 0.01)	TK0119272-01 McDonald (2014, CLTA10_273)
USA, Conklin (MI) 2013 (Montmorency)	SC	3	3.5	0.37	74	Whole fruit	40	0.22, 0.33 (0.28)	2× < 0.01 (< 0.01)	TK0119272-02 McDonald (2014, CLTA10_273)
(Sams)	SC	3	3.5	0.4	74	Whole fruit	40	0.05, 0.053 (0.052)	2× < 0.01 (< 0.01)	TK0119272-06 McDonald (2014, CLTA10_273)
USA, Casnovia (MI) 2013 (Montmorency)	SC	3	3.5	0.37	74	Whole fruit	40	0.049, 0.097 (0.073)	2× < 0.01 (< 0.01)	TK0119272-03 McDonald (2014, CLTA10_273)
USA, Fremont (MI) 2013 (Montmorency)	SC	3	3.5	0.4	74	Whole fruit	39	1.1, 1.2 (1.2)	2× < 0.01 (< 0.01)	TK0119272-04 McDonald (2014, CLTA10_273)
USA, Hart (MI) 2013 (Montmorency)	SC	3	3.5	0.4	74	Whole fruit	39	0.86, 1.8 (1.3)	< 0.01, 0.012 (0.011)	TK0119272-05 McDonald (2014, CLTA10_273)
(Hudson)	SC	3	3.5	0.4	74	Whole fruit	39	0.24, 0.25 (0.24)	2× < 0.01 (< 0.01)	
USA, Perry (UT) 2013 (Montmorency)	SC	3	3.5	0.2	75	Whole fruit	37	0.11, 0.15 (0.13)	2× < 0.01 (< 0.01)	TK0119272-06 McDonald (2014, CLTA10_273)
USA, Tulare (CA) 2013 (Brooks)	SC	3	3.5	0.14	72	Whole fruit	40	0.11, 0.14 (0.12)	2× < 0.01 (< 0.01)	TK0119272-09 McDonald (2014, CLTA10_273)
USA, Plainview	SC	3	3.5	0.55	73	Whole	40	0.43, 0.57	2× < 0.01	TK0119272-10

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No., Reference
(CA) 2013 (Rainier)						fruit		(0.5)	(< 0.01)	McDonald (2014, CLTA10_273)
USA, Ephrata (WA) 2013 (Bing)	SC	3	3.5	0.37	81	Whole fruit	36	0.65, 0.95 (0.8)	2× < 0.01 (< 0.01)	TK0119272-11 McDonald (2014, CLTA10_273)
USA, Weiser (ID) 2013 (Benton)	SC	3	3.6	0.25	75	Whole fruit	40	0.59, 0.9 (0.74)	0.026, 0.035 (0.03)	TK0119272-12 McDonald (2014, CLTA10_273)
USA, Hotchkiss (CO) 2012 (Montmorency)	SC	5	3.5	0.4	Note a	Cherries w/o stem and stone	14	2.7, 5.1 (3.9)	2× < 0.02 (< 0.02)	12-CO01, Jolly (2014, CLTA10_274)
							20	23, 24 (24)	2× < 0.02 (< 0.02)	
						Washed cherries w/o stem and stone	14	2.3, 2.7 (2.5)	2× < 0.02 (< 0.02)	
							20	4.7, 6.5 (5.6)	2× < 0.02 (< 0.02)	
USA, Buhl (ID) 2012 (Montmorency)	SC	5	3.5	0.36	Note a	Cherries w/o stem and stone	7	9.3, 10 (9.7)	2× < 0.02 (< 0.02)	12-ID06, Jolly (2014, CLTA10_274)
							14	8.8, 9.3 (9.0)	2× < 0.02 (< 0.02)	
							22	9.0, 9.2 (9.1)	2× < 0.02 (< 0.02)	
							28	4.0, 6.4 (5.2)	2× < 0.02 (< 0.02)	
						Washed cherries w/o stem and stone	7	1.2, 1.6 (1.4)	2× < 0.02 (< 0.02)	
							14	0.96, 1.3 (1.1)	2× < 0.02 (< 0.02)	
							22	1.6, 1.7 (1.6)	2× < 0.02 (< 0.02)	
							28	1.3, 2.0 (1.8)	2× < 0.02 (< 0.02)	
USA, Fennville (MI) 2013 (Montmorency) Note B	SC	5	3.5	0.36	Note a	Cherries w/o stem and stone	7	2.6, 3.4 (3.0)	2× < 0.02 (< 0.02)	13-MI05, Jolly (2014, CLTA10_274)
							14	1.0, 1.4 (1.2)	2× < 0.02 (< 0.02)	
							21	0.81, 0.82 (0.82)	2× < 0.02 (< 0.02)	
							28	0.73, 0.76 (0.74)	2× < 0.02 (< 0.02)	
						Washed cherries w/o stem and stone	7	1.1, 1.2 (1.2)	2× < 0.02 (< 0.02)	
							14	0.48, 0.52 (0.5)	2× < 0.02 (< 0.02)	
							21	0.3, 0.37 (0.34)	2× < 0.02 (< 0.02)	
							28	0.25, 0.27 (0.26)	2× < 0.02 (< 0.02)	

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No., Reference
USA, Fennville (MI) 2013 (Balaton) Note B	SC	5	3.4	1.2	Note a	Cherries w/o stem and stone	14	3.7, 4.1 (3.9)	2× < 0.02 (< 0.02)	13-MI36, Jolly (2014, CLTA10_274)
							21	1.4, 1.6 (1.5)	2× < 0.02 (< 0.02)	
						Washed cherries w/o stem and stone	14	1.5, 1.7 (1.6)	2× < 0.02 (< 0.02)	
							21	1.2, 1.4 (1.3)	2× < 0.02 (< 0.02)	
	SC	5	3.4	0.6	Note a	Cherries w/o stem and stone	14	2.9, 3.1 (3.0)	2× < 0.02 (< 0.02)	13-MI37, Jolly (2014, CLTA10_274)
							21	1.4, 1.6 (1.5)	2× < 0.02 (< 0.02)	
						Washed cherries w/o stem and stone	14	0.39, 0.5 (0.44)	2× < 0.02 (< 0.02)	
							21	0.37, 0.42 (0.4)	2× < 0.02 (< 0.02)	
USA, Fennville (MI) 2013 (Montmorency) Note B	SC	5	3.5	0.5	Note a	Cherries w/o stem and stone	13	4.2, 4.9 (4.5)	2× < 0.02 (< 0.02)	13-MI38, Jolly (2014, CLTA10_274)
							20	3.0, 3.0 (3.0)	2× < 0.02 (< 0.02)	
						Washed cherries (0.8 L/min) w/o stem and stone	13	2.5, 3.1 (2.8)	2× < 0.02 (< 0.02)	
							20	1.7, 1.7 (1.7)	2× < 0.02 (< 0.02)	
						Washed cherries (1.5 L/min) w/o stem and stone	13	2.4, 2.9 (2.6)	2× < 0.02 (< 0.02)	
							20	1.7, 1.8 (1.8)	2× < 0.02 (< 0.02)	
						Washed cherries (3.2 L/min) w/o stem and stone	13	1.8, 2.4 (2.1)	2× < 0.02 (< 0.02)	
							20	1.7, 1.8 (1.8)	2× < 0.02 (< 0.02)	
USA, Fennville (MI) 2013 (not reported) Note B	SC	5	3.5	0.27	Note a	Cherries w/o stem and stone	14	1.8, 2.1 (2.0)	2× < 0.02 (< 0.02)	13-MI39, Jolly (2014, CLTA10_274)
							21	2.8, 4.4 (3.6)	2× < 0.02 (< 0.02)	
						Washed cherries w/o stem and stone	14	0.68, 0.81 (0.74)	2× < 0.02 (< 0.02)	
							21	2.0, 2.2 (2.1)	2× < 0.02 (< 0.02)	
USA, Lansing (NY) 2013 (not reported)	SC	5	3.5	3.1	Note a	Cherries w/o stem and stone	14	0.41, 0.42 (0.42)	2× < 0.02 (< 0.02)	13-NY01, Jolly (2014, CLTA10_274) GRM005.01A, Storage interval: 16 months
							20	0.52, 0.61 (0.56)	2× < 0.02 (< 0.02)	
						Washed cherries w/o stem and stone	14	0.028, 0.03 (0.029)	2× < 0.02 (< 0.02)	
							20	0.029, 0.13 (0.08)	2× < 0.02 (< 0.02)	

DAT: days after last treatment
BBCH 72-74: 1st-4th fruit has reached typical size
A: BBCH not provided, plants were in "fruiting" growth stage at last application
B: Trials conducted at the same location were considered independent when the difference in treatment dates was at least one week

Peaches

Table 8 Residues of chlorothalonil and SDS-3701 following foliar application to peaches (GRM005.01A, Storage interval: 13 months)

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No., Reference
USA, Alton (NY) 2013 (Venture)	SC	3	3.5	0.37	77	Whole fruit	60	0.11, 0.14 (0.12)	2× < 0.01 (< 0.01)	TK0119271-01, McDonald (2014, CLTA10_275)
USA, Byron (GA) 2013 (Summer Lady)	SC	3	3.5	0.56	76	Whole fruit	62	0.13, 0.14 (0.13)	2× < 0.01 (< 0.01)	TK0119271-02, McDonald (2014, CLTA10_275)
USA, Athens (GA) 2014 (Contender)	SC	3	3.5	0.56	74	Whole fruit	57	0.086, 0.16 (0.12)	2× < 0.01 (< 0.01)	TK0119271-03, McDonald (2014, CLTA10_275)
USA, Plains (GA) 2013 (Red skin)	SC	3	3.5	0.37	77	Whole fruit	59	< 0.01, 0.018 (0.014)	2× < 0.01 (< 0.01)	TK0119271-04, McDonald (2014, CLTA10_275)
USA, Shorter (AL) 2013 (Flame Prince)	SC	3	3.5	0.56	77	Whole fruit	59	0.24, 0.25 (0.24)	2× < 0.01 (< 0.01)	TK0119271-05, McDonald (2014, CLTA10_275)
USA, Boyce (LA) 2013 (June Prince)	SC	3	3.5	0.19	78	Whole fruit	60	0.66, 1.1 (0.9)	< 0.01, 0.011 (0.01)	TK0119271-06, McDonald (2014, CLTA10_275)
USA, Hondo (TX) 2013 (Flamin' Fury)	SC	3	3.4	0.37	73	Whole fruit	60	< 0.01, < 0.01 (< 0.01),	2× < 0.01 (< 0.01)	TK0119271-08, McDonald (2014, CLTA10_275)
USA, Madera (CA) 2014 (Spring Crest)	SC	3	3.4	0.25	73	Whole fruit	58	< 0.01, 0.01 (0.01)	2× < 0.01 (< 0.01)	TK0119271-09, McDonald (2014, CLTA10_275)
USA, Los Molinos (CA) 2013 (Halford)	SC	3	3.5	0.37	72	Whole fruit	60	< 0.01, < 0.01 (< 0.01)	2× < 0.01 (< 0.01)	TK0119271-10, McDonald (2014, CLTA10_275)
USA, Porterville (CA) 2013 (Fey Elberta)	SC	3	3.5	0.19	79	Whole fruit	60	0.18, 0.18 (0.18)	2× < 0.01 (< 0.01)	TK0119271-11, McDonald (2014, CLTA10_275)
USA, Kingsburg (CA) 2013 (Klamt Cling)	SC	3	3.5	0.37	79	Whole fruit	58	0.2, 0.4 (0.3)	2× < 0.01 (< 0.01)	TK0119271-12, McDonald (2014, CLTA10_275)
USA, Ringwood (OK) 2014 (Loring)	SC	3	3.5	0.19	76	Whole fruit	56	0.052, 0.074 (0.063)	2× < 0.01 (< 0.01)	TK0119271-13, McDonald (2014, CLTA10_275)

DAT: days after last treatment
BBCH 71-79: 1st-9th fruit has reached typical size

Cranberry

Table 9 Residues of chlorothalonil and SDS-3701 following foliar application to cranberries (GRM005.01A, Storage interval: 7 months)

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No., Reference
USA, Warehan (MA) 2012 (Howes)	SC	3	5.6	1	A	fruits	51	1.1, 1.7 (1.4)	2× < 0.02 (< 0.02)	MA01, Corley (2013, CLTA10_272)
USA, Creamridge (NJ) 2012 (Stevens)	SC	3	5.6	2.2	B	fruits	49	2.9, 3.4 (3.2)	2× < 0.02 (< 0.02)	NJ03, Corley (2013, CLTA10_272)
USA, Langlois (OR) 2012 (Stevens)	SC	3	5.6	1.5	C	fruits	52	5.4, 5.4 (5.4)	2× < 0.02 (< 0.02)	OR16, Corley (2013, CLTA10_272)
USA, Warrens (WI) 2012 (Stevens)	SC	3	5.6	1.5	A	fruits	50	2.5, 2.8 (2.6)	2× < 0.02 (< 0.02)	WI05, Corley (2013, CLTA10_272)
USA, Wisconsis Rapids (WI) 2012 (Norman LeMunyon)	SC	3	5.6	0.26	A	fruits	50	2.7, 3.7 (3.2)	2× < 0.02 (< 0.02)	WI06, Corley (2013, CLTA10_272)

DAT: days after last treatment
A: fruiting
B: green fruit
C: fruiting, white-pink

Bulb onions

Table 10 Residues of chlorothalonil and SDS-3701 after foliar application to bulb onions (GRM005.01A, Storage interval: 1-11 months

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No., Reference
USA, Lyons (NY) 2013 (Bridger F1)	SC	3	2.5	0.89	48	Bulb, dry	7	0.19, 0.26 (0.22)	0.023, 0.028 (0.026)	TK0119273-01, McDonald (2014, CLTA10_276)
USA,Fresno (CA) 2013 (Stockton Yellow)	SC	3	2.5	1.1	49	Bulb, dry	7	0.37, 0.46 (0.4)	2× < 0.01 (< 0.01)	TK0119273-05, McDonald (2014, CLTA10_276)
USA, Portersville (CA) 2013 (Walla Walla)	SC	3	2.5	1.1	49	Bulb, dry	7	0.38, 0.42 (0.4)	2× < 0.01 (< 0.01)	TK0119273-06, McDonald (2014, CLTA10_276)
USA, Payette (ID) 2013 (Vaquero)	SC	3	2.5	1.1	49	Bulb, dry	7	0.34, 0.78 (0.56)	2× < 0.01 (< 0.01)	TK0119273-07, McDonald (2014, CLTA10_276)
USA, Hillsboro (OR) 2013 (Bridger)	SC	3	2.5	1.1	88	Bulb, dry	7	0.66, 0.69 (0.68)	2× < 0.01 (< 0.01)	TK0119273-08, McDonald (2014, CLTA10_276)
USA,Lenexa (KS) 2013 (Stuttgarter Yellow)	SC	3	2.5	1.1	48	Bulb, dry	7	0.054, 0.11 (0.083)	2× < 0.01 (< 0.01)	TK0119273-12, McDonald (2014, CLTA10_276)
USA, Uvalde (TX) 2013 (Obsession)	SC	3	2.5	1.3	49	Bulb, dry	6	0.34, 0.61 (0.48)	2× < 0.01 (< 0.01)	TK0119273-13, McDonald (2014, CLTA10_276)
USA, Larned (KS) 2013 (Candy Sweet Onion)	SC	3	2.5	1.2	48	Bulb, dry	6	0.061, 0.074 (0.068)	2× < 0.01 (< 0.01)	TK0119273-14, McDonald (2014, CLTA10_276)

DAT: days after last treatment
BBCH 48: Leaves bent over in 50% of plants
BBCH 49: Leaves dead, bulb top dry

Green Onions

Table 11 Residues of chlorothalonil and SDS-3701 after foliar application to green onions (GRM005.01A, Storage interval: 3-9 months

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No., Reference
USA, Athens (GA) 2013 (Texas Sweet)	SC	5	1.5	0.65	17	Whole plant	14	0.37, 0.47 (0.42)	0.046, 0.069 (0.058)	TK0119273-09, McDonald (2014, CLTA10_276)
USA, Portersville (CA) 2013 (Texas Sweet)	SC	5	1.5	0.65	49	Whole plant	14	35, 44 (39)	0.052, 0.066 (0.059)	TK0119273-11, McDonald (2014, CLTA10_276)
USA, Richland (LA) 2013 (Texas Sweet)	SC	5	1.5	0.8	18	Whole plant	14	0.27, 0.31 (0.29)	< 0.01, 0.013 (0.012)	TK0119273-15, McDonald (2014, CLTA10_276)

DAT: days after last treatment
BBCH 18-19: 9 or more leaves clearly visible
BBCH 49: Growth complete; length and stem diameter typical for variety reached

Peppers

Table 12 Residues of chlorothalonil and SDS-3701 after foliar application to peppers

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No., Reference
Bell peppers										
Brazil, Uberlandia 2005 (Natali)	SC	4		0.2	85	Fruit	0 3 5 7 14	6.6 7.5 5.4 2.7 <u>2.9</u>	NA	M03019-JJB, Baptista (2006, CLTA10_280)
	SC	4		0.4	85	Fruit	0 3 5 7 14	15.3 13.1 10.6 9.9 5.8	NA	POPIT MET.109 & 150, Recovery: Mean=103% RSD=6% Storage interval: 12 months
Brazil, Piepade 2005 (Natalie Rogers)	SC	4		0.2	82	Fruit	0 3 5 7 14	3.0 3.8 2.8 0.64 <u>0.74</u>	NA	M03019-LZF, Baptista (2006, CLTA10_280)
	SC	4		0.4	82	Fruit	0 3 5 7 14	12.9 15.6 14.3 11.5 1.5	NA	
Brazil, Sao José dos Pinhais 2005 (Magali)	SC	4		0.2	79	Fruit	0 3 5 7 14	1.6 0.17 0.12 0.12 <u>0.15</u>	NA	M03019-DMO, Baptista (2006, CLTA10_280)
	SC	4		0.4	79	Fruit	0 3 5 7 14	2.2 1.9 0.72 0.19 0.15	NA	
Brazil, Engenheiro Coehlo (SP) 2012 (Ikeda)	SC	3	1.8	0.2	78	Fruit	0 1 3 5 7	0.74 0.56 0.21 0.16 <u>0.16</u>	< 0.01 < 0.01 < 0.01 < 0.01 < 0.<u>01</u>	M13003-FSB1, Matarazzo (2014, CLTA10_281)

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No., Reference
Brazil, Ponta Grossa (PR) 2012 (Magali R)	SC	3	1.8	0.2	76	Fruit	0 1 3 5 7	0.83 0.47 0.41 0.49 0.22	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	M13003-FSB2, Matarazzo (2014, CLTA10_281)
Brazil, Planaltina (DF) 2012 (Paloma)	SC	3	1.8	0.2	75	Fruit	0 1 3 5 7	3.2 2.6 2.8 2.1 1.9	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	M13003-MFG, Matarazzo (2014, CLTA10_281)
Brazil, Palmeira (PR) 2012 (Magali R)	SC	3	1.8	0.2	76	Fruit	0 1 3 5 7	1.0 0.64 0.65 0.57 0.28	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	M13003-RWC1, Matarazzo (2014, CLTA10_281)
Brazil, Lavras (MG) 2012 (Magali)	SC	3	1.8	0.2	85	Fruit	0 7	1.6 0.44	< 0.01 < 0.01	M13003-RWC2, Matarazzo (2014, CLTA10_281) POPIT MET.109 & 150, Recovery: Mean=96-99% RSD=3-11% Storage interval: 12 months
USA, Bridgeton (NJ) 1997 (King Arthur Hybrid)	SC	8	1.3	0.21	89	Fruit	3 7 14 29	2.6, 3.1 (2.8) 2.1, 2.2 (2.2) 1.3, 1.4 (1.4) 0.56, 0.99 (0.78)	2× < 0.03 (< 0.03) 2× < 0.03 (< 0.03) 2× < 0.03 (< 0.03) 2× < 0.03 (< 0.03)	97-NJ15, Thompson (2007, CLTA10_277)
USA, Live Oka (FL) 1997 (Capistrano)	SC	8	1.3	0.46	85	Fruit	3	1.7, 1.7 (1.7)	2× < 0.03 (< 0.03)	97-FL17, Thompson (2007, CLTA10_277)
USA, Weslaco (TX) 1998 (Capistrano)	SC	8	1.2	0.28	89	Fruit	3	2.3, 3.5 (2.9)	2× < 0.03 (< 0.03)	97-TX15, Thompson (2007, CLTA10_277)
USA, Charleston (SC) 1997 (Camelot)	SC	8	1.3	0.25	37 leaf stage	Fruit	3	1.3, 1.6, (1.4)	2× < 0.03 (< 0.03)	97-SC13, Thompson (2007, CLTA10_277)
USA, Freemont (OH) 1997 (King Arthur)	SC	8	1.3	0.19	89	Fruit	2	0.69, 0.82 (0.76)	2× < 0.03 (< 0.03)	97-OH12, Thompson (2007, CLTA10_277)
USA, Salinas (CA) 1997 (Cal Wonder) Note A	SC	8	1.3	0.23	85	Fruit	2 6 13 27	0.33, 0.66 (0.5) 0.18, 0.2 (0.19) 0.2, 0.23 (0.22) 0.055, 0.06 (0.058)	2× < 0.03 (< 0.03) 2× < 0.03 (< 0.03) 2× < 0.03 (< 0.03) 2× < 0.03 (< 0.03)	97-CA45, Thompson (2007, CLTA10_277)
USA, Salinas (CA) 1997 (Gusto) Note A	SC	8	1.3	0.23	85	Fruit	2	0.45, 0.53 (0.49)	2× < 0.03 (< 0.03)	97-CA46, Thompson (2007, CLTA10_277)

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No., Reference
USA, Gainesville (FL) 1997 (Capristrano)	SC	8	1.2	0.23	85	Fruit	3 7 14 28	1.6, 1.6 (1.6) 1.0, 1.4 (1.2) 0.75, 0.9 (0.82) 0.2, 0.22 (0.21)	2× < 0.03 (< 0.03) 2× < 0.03 (< 0.03) 2× < 0.03 (< 0.03) 2× < 0.03 (< 0.03)	97-FL41, Thompson (2007, CLTA10_277)
USA, Tifton (GA) 1997 (Camelot)	SC	7	1.3	0.28	89	Fruit	2 7 13	0.62, 0.94 (0.78) 0.96, 0.99, 1.2 (1.0) 0.4, 0.46 (0.43)	2× < 0.03 (< 0.03) 2× < 0.03 (< 0.03) 2× < 0.03 (< 0.03)	98-GA17, Thompson (2007, CLTA10_277)
Non-bell peppers										
USA, Bridgeton (NJ) 1999 (Biscayne)	WG	8	1.2	0.21	89	Fruit	3 8 14	1.4, 1.8 (1.6) 0.8, 1.2 (1.0) 0.4, 0.6 (0.5)	2× < 0.03 (< 0.03) 2× < 0.03 (< 0.03) 2× < 0.03 (< 0.03)	99-NJ21, Thompson (2007, CLTA10_278)
USA, Gainesville (FL) 1999 (Mesilla Cayenne)	WG	8	1.3	0.35	85	Fruit	3	0.42, 0.82 (0.62)	2× < 0.03 (< 0.03)	99-FL30, Thompson (2007, CLTA10_278)
USA, Weslaco (TX) 1999 (Sonora Anaheim)	WG	8	1.3	0.35	85	Fruit	2	1.5, 1.7 (1.6)	2× < 0.03 (< 0.03)	99-TX17, Thompson (2007, CLTA10_278)
USA, Fremont (OH) 1999 (Milta Jalapeno)	WG	8	1.3	5× 0.35 + 3× 0.25	85	Fruit	2	0.62, 0.78 (0.7)	2× < 0.03 (< 0.03)	99-OH12, Thompson (2007, CLTA10_278)
USA, El Centro (CA) 1999 (Fresno)	WG	6	1.3 1.3 3.8 1.3 1.3 1.3	0.3 0.3 1.0 0.3 0.3 0.3	85	Fruit	2 7 14	0.64, 1.1 (0.87) 2× < 0.03 (< 0.03) 0.93, 1.1 (1.0)	2× < 0.03 (< 0.03) 2× < 0.03 (< 0.03) 2× < 0.03 (< 0.03)	99-CA51, Thompson (2007, CLTA10_278)
USA, Weslcao(TX) 1999 (Veracruz)	WG	8	1.3	0.28	89	Fruit	2 6 13	0.6, 0.64 (0.62) 0.24, 0.26 (0.25) 0.18, 0.24 (0.21)	2× < 0.03 (< 0.03) 2× < 0.03 (< 0.03) 2× < 0.03 (< 0.03)	99-TX28, Thompson (2007, CLTA10_278)
USA, Las Cruces (NM) 2008 (Big Jim)	SC	8	1.3	0.45	85	Fruit	2	0.18, 0.32 (0.25)	0.028, 0.031 (0.03)	08-NM, Homa (2011, CLTA10_279)
	WG	8	1.3	0.45	85	Fruit	2	0.2, 0.31 (0.26)	0.028, 0.03 (0.029)	

Matarazzo (2014, CLTA10_281): POPIT MET.109 & 150, Recovery: Mean=96-99% RSD=3-11%, Storage interval: 12 months
Baptista (2006, CLTA10_280): POPIT MET.109 & 150, Recovery: Mean=103% RSD=6%, Storage interval: 12 months
Thompson (2007, CLTA10_277): "Cornell Method", Storage interval: 2-10 months
Thompson (2007, CLTA10_278): "Cornell Method", Storage interval: 24-25 months
A: Trials considered not independent, since same location and treatment date was used. Different variety was not considered sufficiently different to justify a independent trial result
B: Trials were conducted in the same area but at significantly different dates (two week difference). These trials are considered independent
DAT: days after last treatment
NA: not analysed
BBCH 71-79: 1st-9th fruit has reached typical size
BBCH 81-88: 10-80% of fruits show typical fully ripe colour
BBCH 89: Fully ripe: fruits have typical fully ripe colour

Tomatoes

Table 13 Residues of chlorothalonil and SDS-3701 in protected cherry tomatoes following foliar spraying (GRM005.01A, Storage interval: 6 months)

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No. Reference
cGAP: Poland, 2 × 0.1 kg ai/hL, PHI: 3 d										
France (North), Dampierre en burly 2011 (Lucinda, *Cherry tomato*)	SC	2	1.0	0.17	87	Fruit	0 1 3	1.6 1.8 1.6	0.01 0.02 0.01	S11-00518-REG-02, North (2012, CLTA10_283)
France (South), Elne 2011 (Swift, *Cherry tomato*)	SC	2	1.0	0.17	87	Fruit	0 1 3	2.8 4.0 3.1	0.02 0.04 0.04	S11-00519-REG-01, North (2012, CLTA10_284)
Germany, Unterriexingen 2012 (Favorita, *Cherry tomato*)	SC	2	1.5 1.6	0.2	87	Fruit	3	3.4	0.03	S12-01287-01, Schulz (2012, CLTA10_285)
Germany, Heidelberg 2012 (Amoah EZ, *Cherry tomato*)	SC	2	0.94 0.96	0.2	88	Fruit	3	0.99	0.01	S12-01287-02, Schulz (2012, CLTA10_285)
Spain, Conil de la frontera 2012 (Lupita, *Cherry tomato*)	SC	2	1.6	0.2	82	Fruit	3	2.2	0.03	S12-01288-01, Schulz (2013, CLTA10_286)
Spain, Puerto de Mazarrón 2012 (Katalina, *Cherry tomato*)	SC	2	1.3 1.2	0.2	82	Fruit	3	5.5	0.07	S12-01288-02, Schulz (2013, CLTA10_286)
Spain, Conil de la frontera 2011 (Lupita, *Cherry tomato*)	SC	2	1.0	0.13	85	Fruit	0 1 3	1.1 1.6 0.59	< 0.01 0.01 < 0.01	S11-00519-REG-02, North (2012, CLTA10_284)
United Kingdom, Suffolk 2011 (Conchita, *Cherry tomato*)	SC	2	1.0	0.17	74	Fruit	0 1 3	2.3 1.5 1.8	0.01 0.01 0.02	S11-00518-REG-01, North (2012, CLTA10_283)

DAT: days after last treatment
BBCH 71-79: 1st-9th fruit has reached typical size
BBCH 81-88: 10-80% of fruits show typical fully ripe colour
BBCH 89: Fully ripe: fruits have typical fully ripe colour

Mushrooms

Table 14 Residues of chlorothalonil and SDS-3701 in mushroom following soil drench application (Analytical method 3136-88-0138-MD-001 (see JMPR Report 2010), Storage interval: 1 month)

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No., Reference
cGAP: USA, 12.7 kg ai/ha + 6.4 kg ai/ha, PHI: 7 d										
USA, Fleetwood (PA) 1994 (Spawn: Lambert 900) Note D	SC	2	12 6.1	0.24 0.12	A	mushroom	5	0.33, 0.4 (0.36)	0.052, 0.086 (0.069)	PA03, Thompson (1995, CLTA10_287)
							7	0.35, 0.51 (0.43)	0.15, 0.17 (0.16)	
						mushroom (washed)	5	0.024, 0.037 (0.03)	0.024, 0.046 (0.035)	
	SC	2	12 6.1	0.24 0.12	B	mushroom	5	0.092, 0.2 (0.14)	0.031, 0.034 (0.032)	PA04, Thompson (1995, CLTA10_287)
						mushroom (washed)	5	0.014, 0.022 (0.018)	0.038, 0.027 (0.032)	
USA, Morgan Hill (CA) (Crop # 4143, Strain 2000)	SC	2	12 6.1	0.24 0.12	C	mushroom	5	0.031, 0.11 (0.070)	2× < 0.01 (< 0.01)	CA98, Thompson (1995, CLTA10_287)
							7	0.03, 0.15 (0.09)	2× < 0.01 (< 0.01)	
							13	0.033, 0.12 (0.076)	2× < 0.01 (< 0.01)	
						mushroom (washed)	5	0.022 (0.012, 0.032)	2× < 0.01 (< 0.01)	

DAT: days after last treatment
A: "Pin to ¼ inch diameter buttons"
B: "Pin to ¾ inch diameter buttons"
C: "Pin"
D: These trials were conducted in the same room and at the same date. The use of a different mushroom bed is not considered sufficient to justify independent results

Ginseng

Table 15 Residues of chlorothalonil and SDS-3701 in ginseng following foliar application (Analytical method 3136-88-0138-MD-001 (see JMPR Report 2010), Storage interval: 19 month)

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No., Reference
USA, Marathon County (WI) 2004 (American Ginseng) Note D	SC	8	1.7	0.26	A	Root, washed and dried to 10-30% moisture content	6	0.33, 0.37 (0.35)	0.26, 0.33 (0.3)	WI20, Corley (2007, CLTA10_289)
USA, Marathon County (WI) 2004	SC	8	1.7	0.26	B	Root, washed and dried to 10-30%	7	0.55, 1.0 (0.78)	0.47, 0.75 (0.61)	WI21, Corley (2007, CLTA10_289)

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No., Reference
(American Ginseng) Note D						moisture content				
USA, Marathon County (WI) 2004 (American Ginseng) Note D	SC	8	1.7	0.26	C	Root, washed and dried to 10-30% moisture content	8	0.19, 0.19 (0.19)	0.17, 0.21 (0.19)	WI28, Corley (2007, CLTA10_289)

DAT: days after last treatment
A: "Mature berries"
B: "Most berries dropped"
C: "Berries dropping"
D: Trials were conducted at the same date but farm locations differed by at least 15 miles. The Meeting considered these trials as independent

Horseradish

Table 16 Residues of chlorothalonil and SDS-3701 in horseradish following foliar application ("Cornell Method", Storage interval: 3 months)

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No., Reference
USA, Salisbury (MD) 2002 (no variety reported)	SC	8	2.5	1	A	Roots	13	< 0.02, 0.044 (0.031)	0.025, 0.029 (0.027)	MD02, Thompson (2007, CLTA10_290)
USA, Bridgeton (NJ) 2002 (no variety reported)	SC	8	2.5	1.3	B	Roots	12	0.24, 0.26 (0.25)	0.22, 0.28 (0.25)	NJ16, Thompson (2007, CLTA10_290)
USA, Arlington (WI) 2002 (Big Top Western)	SC	8	2.5	1.9	B	Roots	15	0.29, 0.48 (0.38)	0.13, 0.15 (0.14)	WI04, Thompson (2007, CLTA10_290)

DAT: days after last treatment
A: "Mature"
B: "Vegetative"

Asparagus

Table 17 Residues of chlorothalonil and SDS-3701 in asparagus following foliar application (GRM005.01A, Storage interval: 6 months)

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	Stage	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No. Reference
USA, Comstock Park (MI) 2013 (Jersey Giant)	SC	3	3.4	4.4	fern	Spear	228	2× < 0.01 (< 0.01)	2× < 0.01 (< 0.01)	TK0119274-01, McDonald (2014, CLTA10_291)
USA, Verona (WI) 2013 (Jersey Supreme)	SC	3	3.4	3.4	fern	Spear	231	2× < 0.01 (< 0.01)	2× < 0.01 (< 0.01)	TK0119274-02, McDonald (2014, CLTA10_291)
Canada, Paris (Ontario) 2013 (Mellennium)	SC	3	3.4	4.2	fern	Spear	230	2× < 0.01 (< 0.01)	2× < 0.01 (< 0.01)	TK0119274-03, McDonald (2014, CLTA10_291)
USA, Stockton (CA) 2013 (Colossal)	SC	3	3.4	4.4	fern	Spear	120	2× < 0.01 (< 0.01)	2× < 0.01 (< 0.01)	TK0119274-04, McDonald (2014, CLTA10_291)
USA, Delta (CA) 2013 (Pacific Purple)	SC	3	3.4	4.4	fern	Spear	120	2× < 0.01 (< 0.01)	2× < 0.01 (< 0.01)	TK0119274-05, McDonald (2014,

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	Stage	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No. Reference
										CLTA10_291)
USA, Porterville (CA) 2013 (UC157)	SC	3	3.4	3.4	fern	Spear	121	2× < 0.01 (< 0.01)	2× < 0.01 (< 0.01)	TK0119274-06, McDonald (2014, CLTA10_291)
USA, King City (CA) 2013 (UC157)	SC	3	3.4	3.0	Fern	Spear	121	2× < 0.01 (< 0.01)	2× < 0.01 (< 0.01)	TK0119274-07, McDonald (2014, CLTA10_291)
USA, New Plymouth (ID) 2013 (Apollo)	SC	3	3.4	3.4	fern	Spear	195	2× < 0.01 (< 0.01)	2× < 0.01 (< 0.01)	TK0119274-08, McDonald (2014, CLTA10_291

DAT: days after last treatment

Rhubarb

Table 18 Residues of chlorothalonil and SDS-3701 in rhubarb following foliar treatment ("Cornell Method", Storage interval: 6 months)

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No. Reference
USA, Clarksville (MI) 2002 (Reeds Early Superb)	SC	6	2.6	1.7	A	Petiole (stalk)	31	0.09, 1.0 (0.55)	2× < 0.02 (< 0.02)	MI13, Thompson (2007, CLTA10_292)
USA, Aurora (OR) 2002 (Crimson Red) Note C	SC	6	2.6	0.99	B	Petiole (stalk)	34	1.6, 3.9 (2.8)	2× < 0.02 (< 0.02)	OR14, Thompson (2007, CLTA10_292)
USA, Aurora (OR) 2002 (Crimson) Note C	SC	6	2.7	2.0	B	Petiole (stalk)	28	0.17, 0.58 (0.38)	2× < 0.02 (< 0.02)	OR15, Thompson (2007, CLTA10_292)
	SC	6	2.7	2.0	B	Petiole (stalk)	27	0.33, 0.45 (0.39)	2× < 0.02 (< 0.02)	OR13, Thompson (2007, CLTA10_292)

DAT: days after last treatment
A: "blooming"
B: "8-10 inch petioles"
C: Trial OR14 was conducted at sufficiently different treatment dates and location to justify independent results. Trials OR13 and OR15 were treated at the same location and same date.

Pistachio nuts

Table 19 Residues of chlorothalonil and SDS-3701 in pistachios following foliar application (Analytical method 3136-88-0138-MD-001 (see JMPR Report 2010), Storage interval: 17 month)

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	Chloro-thalonil	SDS-3701	Report/Trial No. Reference
USA, Chico (CA) 1992 (Kerman)	SC	5	5.0	1.3	NR	Nutmeat	14	0.08, 0.14 (0.11)	2× < 0.01 (< 0.01)	CA68, Thompson (1996, CLTA10_293)
USA, Madera (CA) 1992 (Peter, Kerman)	SC	5	5.0	-	A	Nutmeat	14	0.073, 0.091 (0.082)	2× < 0.01 (< 0.01)	CA69, Thompson (1996, CLTA10_293)
USA, Bowie (AZ) 2002 (Kerman)	SC	5	5.0	-	A	Nutmeat	14	2× < 0.01 (< 0.01)	2× < 0.01 (< 0.01)	AZ01, Thompson (1996, CLTA10_293)

DAT: days after last treatment
NS: not reported A: full size nuts

APPRAISAL

Chlorothalonil is a non-systemic fungicide first evaluated by JMPR in 1974 and a number of times subsequently. It was recently reviewed for toxicology by the 2009 and 2010 JMPR within the periodic review program of the CCPR. For the parent substance an ADI of 0–0.02 mg/kg bw and an ARfD of 0.6 mg/kg bw were established. In addition to the parent substance, an ADI of 0–0.008 mg/kg bw and an ARfD of 0.03 mg/kg bw were established for the metabolite SDS-3701.

The 2010 JMPR recommended the following residue definition for chlorothalonil:

Definition of the residue for compliance with MRL for plant commodities: *chlorothalonil*

Definition of the residue for estimation of dietary intake for plant commodities: *chlorothalonil*

SDS-3701 (2,5,6-trichloro-4-hydroxyisophthalonitrile), all considered separately.

Definition of the residue for compliance with MRL and for estimation of dietary intake for animal commodities: *SDS-3701 (2,5,6-trichloro-4-hydroxyisophthalonitrile)*.

In 2012 the JMPR evaluated additional uses for chlorothalonil in banana, chard, chicory, endive, spring onion, spinach, and peas.

The current Meeting received new information on use patterns for chlorothalonil in multiple crops supported by additional analytical methods, storage stability data and supervised field trials.

Methods of analysis

The Meeting received two analytical methods for chlorothalonil not previously evaluated by the Meeting. Both methods were used in the supervised field trials newly submitted and are not intended for monitoring purposes.

Method GRM005.01A is applicable to plant matrices and used homogenisation with acetone and 5M sulphuric acid solution (95:5 v/v). Following solid phase extraction (SPE) clean-up, chlorothalonil was analysed by gas chromatography with mass selective detection (GC-MSD). The metabolite R182281 was quantified by high performance liquid chromatography with triple-quadrupole mass spectrometric detection. The method was successfully validated (70–110% recovery, RSD < 20%) for both analytes for matrices with high water, high acid, high oil and high starch content.

The second method (“Cornell-Method”) is an in-house method using acidified acetone and partitioning against petroleum ether. The organic phase contains chlorothalonil and the aqueous, its metabolite SDS-3701. The sample is then methylated with diazomethane and cleaned up on an alumina column, eluting with dichloromethane. The organic and aqueous extracts were analysed by GC/ECD to determine residues of chlorothalonil and SDS-3701 respectively. The method was successfully validated (70–110% recovery, RSD < 20%) for both analytes for matrices with high water and high acid content.

Stability of residues in stored analytical samples

The Meeting received two additional studies on the storage stability to support the newly submitted supervised field trials not previously evaluated.

In the first study chlorothalonil and its metabolite SDS-3701 were proven to be stable for at least 24 months in stored samples of tomato, cucumber, melon, oranges, carrots (roots and tops), barley (grain and straw) and soya bean seeds.

In a second study cranberries fortified with chlorothalonil and SDS-3701 were analysed after 10 months. The stored triplicate samples indicated a significant decline with average recoveries of 63% of chlorothalonil and 38% of SDS-3701 remaining. The Meeting concluded that both analytes may degrade in cranberries. Since no intermediate samples were analysed, no acceptable storage interval above one month could be identified by the Meeting.

Results of supervised residue trials on crops

The Meeting received supervised trial data for applications of chlorothalonil on various fruit and vegetable crops conducted in Brazil, Europe, Rep. of Korea and the USA.

Residues of SDS-3701 may potentially be taken up by succeeding crops after application of chlorothalonil in the previous year. For annual crops considered by this year, JMPR only estimated median and highest residue values following primary treatment, as these are intermediate values in the establishment of the final STMR and HR values which need to take into account the additional contribution by soil uptake; refer to the rotational crop section.

Pear

Chlorothalonil is registered in Rep. of Korea on pears at a rate of 4×0.04 kg ai/hL with a PHI of 14 days. Six supervised field trials from Rep. of Korea matching this GAP were submitted.

In the trials submitted samples were prepared for analysis by removal of the stem and the core, which were discarded before homogenisation. The Meeting concluded the sample preparation did not comply with the Codex Sampling Guideline, and would have had a significant influence on the residue concentration, making these trials unsuitable for the estimation of maximum residue levels or STMR and HR values.

Cherries

Chlorothalonil is registered in Canada on cherries with a rate of 3×4.5 kg ai/ha with a PHI of 40 days. Supervised field trials from the USA matching this GAP were submitted.

In cherries following treatment with chlorothalonil according to Canadian GAP, residues were (n=10): 0.04, 0.073, 0.12, 0.13, 0.28, 0.5, 0.74, 0.8, 1.2, 1.3 mg/kg.

The corresponding residues of SDS-3701 were (n=10): < 0.01(8), 0.011, 0.03 mg/kg

The Meeting estimated a maximum residue level, an STMR and an HR value of 3 mg/kg, 0.39 mg/kg and 1.8 mg/kg (based on a single highest field sample) for chlorothalonil in cherries, respectively.

For dietary intake purposes the Meeting also estimated an STMR of 0.01 mg/kg and an HR of 0.035 mg/kg (based on a single highest field sample) for SDS-3701 in cherries.

Peaches and nectarines (subgroup)

Chlorothalonil is registered in Canada on peaches and nectarins with a rate of 3×4.5 kg ai/ha with a PHI of 60 days. Supervised field trials from the USA matching the GAP were submitted.

In peaches following treatment with chlorothalonil according to Canadian GAP residues were (n=12): < 0.01, < 0.01, 0.01, 0.014, 0.063, 0.12, 0.12, 0.13, 0.18, 0.24, 0.3, 0.9 mg/kg.

The corresponding residues of SDS-3701 were (n=12): < 0.01(11), 0.01 mg/kg

The Meeting estimated a maximum residue level, an STMR and an HR value of 1.5 mg/kg, 0.12 mg/kg and 1.1 mg/kg (based on a single highest field sample) for chlorothalonil in peaches, respectively.

For dietary intake purposes the Meeting also estimated an STMR of 0.01 mg/kg and an HR of 0.011 mg/kg (based on a single highest field sample) for SDS-3701 in peaches (including nectarines and apricots).

Cranberry

Chlorothalonil is registered in Canada on cranberries with a rate of 3×5.5 kg ai/ha with a PHI of 50 days.

Supervised field trials from the USA matching the GAP were submitted; however supportive storage stability data indicated a substantial loss of residues after the seven month storage interval of the field samples. The Meeting concluded that the data could not be used for assessment.

Bulb onions

Chlorothalonil is registered in the USA on dry onions and shallots with a rate of 3×2.5 kg ai/ha with a PHI of 7 days. Supervised field trials from the USA matching this GAP were submitted.

In bulb onions following treatment with chlorothalonil according to USA GAP residues were (n=8): 0.068, 0.083, 0.22, 0.4, 0.4, 0.48, 0.56, 0.68 mg/kg.

The corresponding residues of SDS-3701 were (n=8): < 0.01(7), 0.026 mg/kg.

The Meeting estimated a maximum residue level, and STMR and an HR value of 1.5 mg/kg, 0.4 mg/kg and 0.69 mg/kg (based on a single highest field sample) for chlorothalonil in bulb onions, respectively.

For dietary intake purposes the Meeting also estimated a STMR of 0.01 mg/kg and an HR of 0.028 mg/kg (based on a single highest field sample) for SDS-3701 in bulb onions.

The Meeting agreed to extrapolate the results to shallots.

Green onions

Chlorothalonil is registered in the USA on green onions with a rate of 3×2.5 kg ai/ha with a PHI of 14 days.

Three supervised field trials from the USA matching the GAP application rate and PHI were submitted. However, one of these trials was conducted at a late growth stage of BBCH 49 which showed substantially higher residues (39 mg/kg) than the two other trials treated at BBCH 17–18 (0.29 mg/kg and 0.42 mg/kg).

The Meeting concluded that the total dataset available is inadequate and no recommendation on green onions can be made.

Peppers

Chlorothalonil is registered in Brazil on pepper with a rate of 2×0.2 kg ai/hL with a PHI of 7 days. Supervised field trials from Brazil matching this GAP were submitted to the 2010 Meeting and supported by additional trials this year.

Residues of chlorothalonil in peppers following treatment according to Brazilian GAP based on trials submitted to the 2010 JMPR were (n=4): 1.1, 1.5, 1.7 and 4.4 mg/kg.

Additional trials submitted this year on peppers gave chlorothalonil residues of (n=8): 0.15, 0.16, 0.22, 0.28, 0.44, 0.74, 1.9, 2.9 mg/kg

Total residues (2010+2015 data) in peppers following treatment according to Brazilian GAP were (n=12): 0.15, 0.16, 0.22, 0.28, 0.44, 0.74, 1.1, 1.5, 1.7, 1.9, 2.9 and 4.4 mg/kg.

The corresponding residues of SDS-3701 (when analysed) were (n=5): < 0.01(5) mg/kg.

In the USA chlorothalonil is registered on peppers with a rate of 8×1.3 kg ai/ha with a PHI of 3 days. Supervised field trials from the USA matching this GAP were submitted.

In bell peppers following treatment with chlorothalonil according to USA GAP residues were (n=8): 0.5, 0.76, 1.0, 1.4, 1.6, 1.7, 2.8, 2.9 mg/kg. The corresponding residues of SDS-3701 were (n=8): < 0.03(8) mg/kg.

In non-bell peppers following treatment with chlorothalonil according to USA GAP residues were (n=7): 0.26, 0.62, 0.62, 0.7, 1.0, 1.6, 1.6 mg/kg. The corresponding residues of SDS-3701 were (n=7): 0.029, < 0.03(6) mg/kg.

The Meeting recognized that chlorothalonil residues in peppers treated according to Brazilian GAP resulted in the highest residue and estimated a maximum residue level of 7 mg/kg based on this dataset for peppers.

For dietary intake purposes of chlorothalonil the Meeting concluded that the STMR value for bell peppers treated according to US GAP was higher than the STMR according to the Brazilian GAP. Since both GAPs were supported by a sufficient number of trial data, the higher STMR of 1.5 mg/kg was selected for dietary intake purposes. An HR of 4.4 mg/kg was estimated based on the Brazilian GAP.

Residues of SDS-3701 were generally below the LOQs of 0.01 mg/kg to 0.03 mg/kg except for one finite residue at 0.029 mg/kg. The Meeting estimated both an STMR and HR of 0.03 mg/kg for SDS-3701 in peppers based on the more critical US dataset.

For the extrapolation from sweet pepper to dried chili pepper a default processing factor of 10 was taken into account. The Meeting estimated a maximum residue level of 70 mg/kg for chlorothalonil in dried chili pepper as wells as a STMR of 15 mg/kg and a HR of 44 mg/kg. For SDS-3701 both a STMR and HR of 0.3 mg/kg were estimated.

Tomato

Chlorothalonil is registered in Poland on tomatoes under protected conditions with a rate of 2 × 0.1 kg ai/hL (up to 1 kg ai/ha per application) with a PHI of 3 days. Protected supervised field trials on cherry tomatoes from various European countries approximating the GAP but with higher spray concentrations of 0.13 kg ai/hL to 0.2 kg ai/hL were submitted.

Compared to the Polish GAP all supervised field trials involved treatment at exaggerated spray concentrations, however the rates applied approximate the GAP maximum of 1 kg ai/ha and application. Since in the field trials submitted tomatoes were cultivated as high crops, the Meeting concluded that the spray concentration is the most sensitive parameter in terms of residues and decided to use the proportionality approach based on the spray concentration.

In protected tomatoes following treatment with 0.13 kg ai/hL (scaling factor 0.77) chlorothalonil residues were 0.45 mg/kg (0.77×0.59 mg/kg) and SDS-3701 residues were < 0.01 mg/kg (unscaled).

In protected tomatoes following treatment with 0.17 kg ai/hL (scaling factor 0.59) chlorothalonil residues were 0.94, 1.1, 1.8 mg/kg (0.59×1.6, 1.8 and 3.1 mg/kg) and SDS-3701 residues were 0.006, 0.012, 0.024 mg/kg (0.59×0.01, 0.02 and 0.04 mg/kg).

In protected tomatoes following treatment with 0.2 kg ai/hL (scaling factor 0.5) chlorothalonil residues were 0.5, 1.1, 1.7, 2.8 mg/kg (0.5×0.99, 2.2, 3.4 and 5.5 mg/kg) and SDS-3701 residues were 0.005, 0.015, 0.015, 0.035 mg/kg (0.5×0.01, 0.03, 0.03 and 0.07 mg/kg).

Total scaled residues of chlorothalonil were (n=8): 0.45, 0.5, 0.94, 1.1, 1.1, 1.7, 1.8 and 2.8 mg/kg

Total scaled residues of SDS-3701 were (n=8): 0.005, 0.006, < 0.01, 0.012, 0.015, 0.015, 0.024 and 0.035 mg/kg

The Meeting estimated a maximum residue level, an STMR and an HR value of 5 mg/kg, 1.1 mg/kg and 2.8 mg/kg for chlorothalonil in tomatoes, respectively.

For dietary intake purposes the Meeting also estimated a STMR of 0.0135 mg/kg and an HR of 0.035 mg/kg for SDS-3701 in tomatoes.

Mushroom

Chlorothalonil is registered in the USA on mushrooms for soil drench application with a rate of 12.7 kg ai/ha as a first treatment followed by 6.4 kg ai/ha as second treatment with a PHI of 7 days. Supervised field trials from the USA matching the GAP were submitted.

In mushrooms following treatment with chlorothalonil according to USA GAP residues were (n=2): 0.09, 0.43 mg/kg.

The corresponding residues of SDS-3701 were (n=2): < 0.01, 0.16 mg/kg.

The Meeting concluded that the data submitted for mushroom was insufficient upon which to make recommendations.

Ginseng

Chlorothalonil is registered in the USA on ginseng with a rate of 8×1.7 kg ai/ha with a PHI of 14 days. Supervised field trials from the USA matching the GAP were submitted.

In ginseng roots (washed and dried) following treatment with chlorothalonil according to USA GAP residues were (n=3): 0.19, 0.35, 0.78 mg/kg.

The corresponding residues of SDS-3701 were (n=3): 0.19, 0.3, 0.61 mg/kg.

The Meeting estimated a maximum residue level, and STMR and an HR value of 2 mg/kg, 0.35 mg/kg and 1.0 mg/kg (based on a single highest field sample) for chlorothalonil in dried ginseng (including red ginseng), respectively.

For dietary intake purposes the Meeting also estimated an STMR of 0.3 mg/kg and an HR of 0.61 mg/kg (based on a single highest field sample) for SDS-3701 in dried ginseng (including red ginseng).

Horseradish

Chlorothalonil is registered in the USA on horseradish with a rate of 8×2.5 kg ai/ha with a PHI of 14 days. Supervised field trials from the USA matching this GAP were submitted.

In horseradish roots following treatment with chlorothalonil according to USA GAP residues were (n=3): 0.031, 0.25, 0.38 mg/kg.

The corresponding residues of SDS-3701 were (n=3): 0.027, 0.14, 0.25 mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR value of 1 mg/kg, 0.25 mg/kg and 0.48 mg/kg (based on a single highest field sample) for chlorothalonil in horseradish, respectively.

For dietary intake purposes the Meeting also estimated an STMR of 0.14 mg/kg and an HR of 0.28 mg/kg (based on a single highest field sample) for SDS-3701 in horseradish.

Root and tuber vegetables, except horseradish

In 2010 the Meeting recommended a maximum residue level for root and tuber vegetables of 0.3 mg/kg. Due to the higher maximum residue level of 1 mg/kg for chlorothalonil in horseradish, the Meeting decided to exclude horseradish from the group maximum residue level.

The Meeting estimated a maximum residue level of 0.3 mg/kg for root and tuber vegetables, except horseradish. In 2010 the Meeting decided to accommodate for the uncertainty involved with the residue data by basing the dietary risk assessment (chronic and acute) on the maximum residue level also.

The Meeting withdraws its previous recommendation of 0.3 mg/kg for chlorothalonil in root and tuber vegetables.

Asparagus

Chlorothalonil is registered in the USA on asparagus with a rate of 3×3.4 kg ai/ha applied after harvest to the fern with a PHI of 190 days. Supervised field trials from the USA matching the GAP were submitted.

In asparagus spears following treatment with chlorothalonil according to USA GAP residues were (n=8): < 0.01(8) mg/kg.

The corresponding residues of SDS-3701 were (n=8): < 0.01(8) mg/kg.

The Meeting estimated a maximum residue level of 0.01* mg/kg for chlorothalonil in asparagus.

For dietary intake purposes the Meeting concluded that the application of chlorothalonil after harvest to the fern does not lead to significant residues in asparagus spears in the next growing season. Therefore the STMR and HR for both chlorothalonil and SDS-3701 were estimated at 0 mg/kg, although no trials conducted at exaggerated rates were submitted.

Rhubarb

Chlorothalonil is registered in the USA on rhubarb with a rate of 6×2.5 kg ai/ha with a PHI of 30 days. Supervised field trials from the USA matching this GAP were submitted.

In rhubarb stalks following treatment with chlorothalonil according to USA GAP residues were (n=3): 0.39, 0.55, 2.8 mg/kg.

The corresponding residues of SDS-3701 were (n=3): < 0.02(3) mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR value of 7 mg/kg, 0.55 mg/kg and 3.9 mg/kg (based on a single highest field sample) for chlorothalonil in rhubarb, respectively.

For dietary intake purposes the Meeting also estimated an STMR and an HR of 0.02 mg/kg for SDS-3701 in rhubarb.

Pistachio nut

Chlorothalonil is registered in the USA on pistachio nuts with a rate of 5×5.0 kg ai/ha and a PHI of 14 days. Supervised field trials from the USA matching the GAP were submitted.

In pistachio nutmeat following treatment with chlorothalonil according to USA GAP residues were (n=3): < 0.01, 0.082, 0.11 mg/kg.

The corresponding residues of SDS-3701 were (n=3): < 0.01(3) mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR value of 0.3 mg/kg, 0.082 mg/kg and 0.14 mg/kg (based on a single highest field sample) for chlorothalonil in pistachios, respectively.

For dietary intake purposes the Meeting also estimated an STMR and an HR of 0.01 mg/kg for SDS-3701 in pistachios.

Residues in rotational crops

Following application of chlorothalonil the major metabolite SDS-3701 has a potential to be taken up by succeeding crops. However, the additional uses evaluated by this JMPR either involve treatment of permanent crops not being subject to crop rotation or their total seasonal rate is lower than the maximum seasonal rate of 20 kg ai/ha used in 2010 to estimate residues in rotational crops. The

Meeting concluded that the assessment of SDS-3701 residues in rotational crops, as evaluated in 2010, also covers uses evaluated this year.

For primary uses evaluated this year on crops being subject to crop rotation, the Meeting decided to take into account the soil uptake of SDS-370 on crop residues. STMR and HR values following direct treatment were added to the corresponding values estimated for rotational crops to address the potential use of chlorothalonil in previous years.

For bulb onions and shallots STMR and HR values of 0.01 mg/kg and 0.028 mg/kg were identified after treatment according to current GAP. In 2010 STMR and HR values of 0.01 mg/kg and 0.04 mg/kg were estimated for SDS-3701 in rotated bulb vegetables. For the dietary intake assessment the Meeting estimated overall STMR and HR values of 0.02 mg/kg and 0.068 mg/kg, respectively.

In peppers grown as rotational crop (see fruiting vegetables) the 2010 Meeting estimated an STMR and an HR value of 0.015 mg/kg and 0.06 mg/kg for SDS-3701, respectively. The current Meeting evaluated uses on peppers (STMR and HR: 0.03 mg/kg each) and estimated overall STMR and HR-values of 0.045 mg/kg and 0.09 mg/kg. For dried chili pepper a default processing factor of 10 was applied, resulting in STMR and HR values of 0.45 mg/kg and 0.9 mg/kg for SDS-3701.

Uses on tomatoes evaluated by the current Meeting are only related to protected conditions and therefore not subject to crop rotation.

In horseradish grown as rotational crop (see root and tuber vegetables) the 2010 Meeting estimated an STMR and an HR value of 0.02 mg/kg and 0.03 mg/kg for SDS-3701, respectively. The current Meeting evaluated uses on horseradish (STMR: 0.14 mg/kg and HR: 0.28 mg/kg) and estimated overall STMR and HR-values of 0.16 mg/kg and 0.31 mg/kg for SDS-3701.

Asparagus, cherries, ginseng, peaches, pistachio nuts and protected tomatoes were not considered relevant in terms of residues derived from crop rotation.

Fate of residues during processing

In 2010 the JMPR Meeting concluded that under simulated processing conditions in sterile buffer solutions at pH 4 chlorothalonil residues were relatively stable with > 90% remaining at 90 °C and 73% remaining at 120 °C. At pH 5 and 100 °C a moderate degradation was observed in all samples, leaving approx. 80% of the initial chlorothalonil. The major degradation product was identified as SDS-3701 at 19% of the initial residue. For pH6 at 120 °C chlorothalonil is quickly degraded. Under addition of a sodium acetate buffer, less than 4% of the chlorothalonil remained. Main degradation products were SDS-3701 (48%) and an artefact (28%, identified as 4-amino-2,5,6-trichloroisophthalonitrile). In sterile water without buffer approx. 26% of the chlorothalonil remained. SDS-3701 constituted 59% of the residue while there was no formation of the artefact.

In contrast to the results obtained from sterile buffer solutions processing studies involving background matrices gave much lower levels of SDS-3701 after processing. The 2010 Meeting decided that besides the normal processing factors for chlorothalonil, yield factors for the conversion of parent substance into SDS-3701 should be taken into account for the estimation of the dietary intake. Depending on the outcome, the higher processing factor of SDS-3701 → SDS-3701 or chlorothalonil → SDS-3701 is used for the overall estimation of STMR-P and HR-P for SDS-3701 in the processed product.

Raw commodity (chlorothalonil)	Processed commodity	Chlorothalonil → Chlorothalonil (see 2010 JMPR Evaluation)		
		Individual processing factors	Mean or best estimate processing factor	STMR-P in mg/kg
Tomato (STMR: 1.1 mg/kg)	Juice, raw	0.3	See juice, bottled	See juice, bottled
	Juice, bottled	0.09, 0.1, 0.11, 0.13	0.1	0.11
	Puree	< 0.01(4)	0.01	0.011
	Canned/preserve	< 0.01(4)	0.01	0.011

	pomace, wet	0.01, 0.32	See pomace, dry	See pomace, dry
	pomace, dry	1.0, 1.3, 1.3, 1.4	1.3	1.4

Raw commodity (SDS-3701)	Processed commodity	SDS-3701 → SDS-3701 (see 2010 JMPR Evaluation)		
		Individual processing factors	Mean or best estimate processing factor	STMR-P in mg/kg
Tomato (STMR: 0.0135 mg/kg)	Juice, raw	0.5	See juice, bottled	See juice, bottled
	Juice, bottled	1.0, 1.0, 1.0, 1.5	1.0	0.0135
	Puree	5.5, 6, 6.5, 7.5	6.3	0.085
	Canned/preserve	1.0, 2.0, 2.0, 2.5	2.0	0.027
	pomace, wet	1.5, 19	See pomace, dry	See pomace, dry
	pomace, dry	13, 14, 16, 18	15	0.2

Raw commodity (chlorothalonil)	Processed commodity	Chlorothalonil → SDS-3701 (see 2010 JMPR Evaluation)		
		Individual processing factors	Mean or best estimate processing factor	STMR-P in mg/kg
Tomato (STMR: 1.1 mg/kg)	Juice, raw	0.001	See juice, bottled	See juice, bottled
	Juice, bottled	0.002(4)	0.002	0.0022
	Puree	0.01(3), 0.02	0.01	0.011
	Canned/preserve	0.002, 0.004, 0.004, 0.005	0.004	0.0044
	pomace, wet	0.003, 0.04	See pomace, dry	See pomace, dry
	pomace, dry	0.03(3), 0.04	0.03	0.033

For chlorothalonil in processed tomato products, based on an STMR value of 1.1 mg/kg, the Meeting estimated STMR-P values of 0.11 mg/kg for tomato juice, 0.011 mg/kg for tomato puree and canned tomatoes and 1.4 mg/kg for tomato dry pomace.

For SDS-3701, based on processing factor from SDS-3701 → SDS-3701 and an STMR value of 0.0135 mg/kg, the Meeting estimated STMR-P values of 0.0135 mg/kg for tomato juice, 0.085 mg/kg for tomato puree, 0.027 mg/kg for canned tomatoes and 0.2 mg/kg for tomato dry pomace.

Residues in animal commodities

For all uses under evaluation in this JMPR for chlorothalonil only tomato pomace was identified as a relevant feed item to livestock animals. Since residues in tomato pomace in the dietary feed burden are superseded by residues of grape pomace being in the same Codex feed item group, no increase in the dietary burden for SDS-3701 by the uses evaluated this year compared to 2010 can be expected.

RECOMMENDATIONS

The Meeting estimated the STMR, HR and MRL values shown in Annex 1.

Definition of the residue for compliance with MRL for plant commodities: *chlorothalonil*

Definition of the residue for estimation of dietary intake for plant commodities: chlorothalonil SDS-3701 (2,5,6-trichloro-4-hydroxyisophthalonitrile), all considered separately.

Definition of the residue for compliance with MRL and for estimation of dietary intake for animal commodities: *SDS-3701 (2,5,6-trichloro-4-hydroxyisophthalonitrile)*.

The residue is considered not fat-soluble.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
VS 0621	Asparagus	0.01*	-	Chlorothalonil: 0 SDS-3701: 0	Chlorothalonil: 0 SDS-3701: 0
FS 0013	Cherries	3	-	Chlorothalonil: 0.39 SDS-3701: 0.01	Chlorothalonil: 1.8 SDS-3701: 0.035
DV 0604	Dried ginseng (including red ginseng)	2	-	Chlorothalonil: 0.35 SDS-3701: 0.3 [a]	Chlorothalonil: 1.0 SDS-3701: 0.61[a]
VR 0583	Horseradish	1	-	Chlorothalonil: 0.25 SDS-3701: 0.16 [b]	Chlorothalonil: 0.48 SDS-3701: 0.31 [b]
VA 0385	Onion, bulb	1.5	-	Chlorothalonil: 0.4 SDS-3701: 0.02 [b]	Chlorothalonil: 0.69 SDS-3701: 0.068 [b]
FS 0247	Peaches (including nectarines and apricots)	1.5	-	Chlorothalonil: 0.12 SDS-3701: 0.01	Chlorothalonil: 1.1 SDS-3701: 0.011
VO 0051	Peppers	7	-	Chlorothalonil: 1.5 SDS-3701: 0.045 [b]	Chlorothalonil: 4.4 SDS-3701: 0.09[b]
VO 0440	Peppers, Chili (dry)	70	-	Chlorothalonil: 15 SDS-3701: 0.45 [b]	Chlorothalonil: 44 SDS-3701: 0.9[b]
TN 0675	Pistachio nut	0.3	-	Chlorothalonil: 0.082 SDS-3701: 0.01	Chlorothalonil: 0.14 SDS-3701: 0.01
VS 0627	Rhubarb	7	-	Chlorothalonil: 0.55 SDS-3701: 0.02	Chlorothalonil: 3.9 SDS-3701: 0.02
VR 0075	Root and tuber vegetables	W	0.3	-	-
VR 0075	Root and tuber vegetables, except horseradish	0.3	-	Chlorothalonil: 0.3 SDS-3701: 0.02 [c]	Chlorothalonil: 0.3 SDS-3701: 0.03 [c]
VA 0388	Shallot	1.5	-	Chlorothalonil: 0.4 SDS-3701: 0.02 [b]	Chlorothalonil: 0.69 SDS-3701: 0.068 [b]
VO 0448	Tomato	5	-	Chlorothalonil: 1.1 SDS-3701: 0.0135	Chlorothalonil: 2.8 SDS-3701: 0.035
JF 0048	Tomato juice			Chlorothalonil: 1.1 SDS-3701: 0.0135	
MW 0448	Tomato purée			Chlorothalonil: 1.1 SDS-3701: 0.0185	
	Tomato canned			Chlorothalonil: 1.1 SDS-3701: 0.027	
	Tomato dry pomace			Chlorothalonil: 1.4 SDS-3701: 0.2	

[a] The contribution of SDS-3701 by uptake from soil cannot be estimated for dried ginseng.

[b] STMR and HR values represent the sum of SDS-3701 found after direct application and in crops grown as rotational crop (see Residues in rotational crops

[c] Based on 2010 Evaluation

DIETARY RISK ASSESSMENT

Long-term intake

The evaluation of chlorothalonil 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 this years estimated STMRs and previous STMRs from 2010 and 2012 were in the range 10–50% of the maximum ADI of 0.02 mg/kg bw.

The evaluation of SDS-3701 has resulted in recommendations for STMRs for raw and processed commodities following primary treatment and after uptake from soil as rotational crop.

The International Estimated Daily Intakes for the 17 GEMS/Food cluster diets, based on this years estimated STMRs and previous STMRs from 2010 and 2012 were in the range 4–10% of the maximum ADI of 0.008 mg/kg bw.

The results are shown in Annex 3 to the 2015 Report.

The Meeting concluded that the long-term intake of residues of chlorothalonil and its metabolite SDS-3701, 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 chlorothalonil and its metabolite SDS-3701 were separately 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 2015 Report.

The IESTI for chlorothalonil varied from 0–30% of the ARfD (0.6 mg/kg bw) and the IESTI for its metabolite SDS-3701 from 0–10% of the ARfD (0.03 mg/kg bw). The Meeting concluded that the short-term intake of residues of chlorothalonil and SDS-3701, from uses that have been considered by the JMPR, is unlikely to present a public health concern.

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CYANTRANILIPROLE (263)

The first draft was prepared by Dr Guibiao Ye, Institute for the Control of Agrochemicals, Ministry of Agriculture, Beijing, China

EXPLANATION

Cyantraniliprole is a diamide insecticide with a mode of action (ryanodine receptor activation) similar to chlorantraniliprole and flubendiamide. It has root systemic activity with some translaminar movement and is effective against the larval stages of lepidopteran insects; and on thrips, aphids, and some other chewing and sucking insects.

Cyantraniliprole was first evaluated for toxicology and residues by JMPR in 2013 and an ADI of 0–0.03 mg/kg bw/day was established. An ARfD was deemed to be unnecessary. Residue definitions were also established:

- Definition of residue for compliance with MRL for both animal and plant commodities: *cyantraniliprole*.
- Definiton of residue for estimation of dietary intake for unprocessed plant commodities: *cyantraniliprole*.
- Definition of residue for estimation of dietary intake for processed plant commodities: *sum of cyantraniliprole and IN-J9Z38, expressed as cyantraniliprole.*
- Definition of residue for estimation of dietary intake for animal commodities—*sum of cyantraniliprole, 2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]-3,4-dihydro-3,8-dimethyl-4-oxo-6-quinazolinecarbonitrile [IN-J9Z38], 2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]-1,4-dihydro-8-methyl-4-oxo-6-quinazolinecarbonitrile [IN-MLA84], 3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2-(hydroxymethyl)-6-[(methylamino)carbonyl]phenyl]-1H-pyrazole-5-carboxamide [IN- N7B69] and3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2[[(hydroxymethyl)amino]carbonyl]-6-methylphenyl]-1H-pyrazole-5-carboxamide [IN-MYX98], expressed a cyantraniliprole.*
- The residue is not fat soluble.

At the 46th Session of the CCPR(2014) cyantraniliprole was scheduled for evaluation of additional use patterns by 2015 JMPR.

The Meeting received the residue data for citrus fruits, strawberries, grapes, pomegranates, olives, cucumber, squash, melons, beans, peas, soya beans, carrots, radishes, artichokes, corn, rice, tree nuts, cotton, tea, coffee and tobacco, and information on proposed/registered uses of cyantraniliprole on corresponding crops, and the processing studies of oranges, grapes, olives, and cotton. Some of these studies had been submitted to and evaluated by 2013 JMPR.

USE PATTERNS

Cyantraniliprole is registered in many countries for the control of insect pests on fruits, vegetables and cereals. Cyantraniliprole is intended for use as foliar applications in a wide range of fruit and vegetable crops, tree crops and oil seed crops. Other applications include seed treatments and pre-plant soil application. The information available to the Meeting on registered uses is summarized in the following table.

Table 1 Registered uses of cyantraniliprole

Crop	Country	Formulation		Application				PHI (days)	Remarks
		g ai./L or g ai/kg	type	Method	Rate (g ai/ha)	Water L/ha	No		
Citrus fruit (Group 002)									
Citrus	USA	200	SC	Soil application	220–438			1	Label, maximum seasonal application rate 450 g ai/ha
	USA	100	SE	Spraying	100–150	935–1400		1	Label, maximum seasonal application rate 450 g ai/ha
	Japan	100	SE	Spraying	40–140	2000–7000	3	1	Label
Berries and other small fruits (Group 004)									
Grape	India	100	OD	foliar	70	1000	3	5	Label
	Japan	100	SE	foliar	40–280	2000–7000	3	1	Label
Assorted tropical and sub-tropical fruits—inedible peel (Group 006)									
Pomegranate	India	100	OD	Spraying	75–90	1000	3	5	Label
Fruiting vegetables—Cucurbits (Group 011)									
Vegetables, cucurbit	Canada	100	SE	Foliar	25–150	100	4	1	Label
	USA	200	SC	Soil application	70–197		2	1	Label, maximum seasonal application rate 450 g ai/ha
	USA	100	SE	Spraying	50–150	93–935		1	Label, maximum seasonal application rate 450 g ai/ha
Legume Vegetables (Group 014)									
Legume (Bean, pea, soya bean)	Canada	100	SE	Spraying	25–150	100	4	1	Label, new
Pulses (Group 015)									
Legume (Bean, pea, soya bean)	Canada	100	SE	Spraying	25–150	100	4	7	Label, new
Root and Tuber Vegetables (Group 016)									
Radish	Canada	200	SC	Soil application	75–100		1	21	Label, new
	Canada	100	SE	Spraying	25–150	100	4	7	Label, new
Carrot	Canada	100	SE	Spraying	25–150	100	4	7	Label, new
	Canada	200	SC	Soil application	75–100		1	21	Label, new
Stalk and Stem Vegetables (017)									
Artichoke	Canada	100	SE	spraying	25–150	100	4	7	Label, Tuberous and corm vegetable
	USA	100	OD	Spraying	40–150	93		7	Label, Tuberous and corm vegetable,< 450 g ai/ha
	USA	200	SC	Soil application	90–197			N/A	Label, maximum seasonal application rate 450 g ai/ha
Cereal Grain (Group 020)									
Maize (field and pop)	Canada	600	FORTENZA	Seed treatment	12–24				Label, 50–100 g ai/100kg seed

Crop	Country	Formulation		Application				PHI (days)	Remarks
		g ai./L or g ai/kg	type	Method	Rate (g ai/ha)	Water L/ha	No		
Tree Nuts (Group 022)									
Tree nut Almond, pecan	Canada	100	SE	Spraying	50–100	450	4	5	label
	US	100	OD	Spraying	60–150	935–1400	3	5	Label, Maximum seasonal application rate 450 g ai/ha
	US	100	SE	Spraying	60–150	935–1400	3	5	Label, Maximum seasonal application rate 450 g ai/ha
Oilseed (Group 023)									
Cotton	USA	100	OD	Spraying	50–150	93–468	3	7	Label, Maximum seasonal application rate 450 g ai/ha
	Columbia	100	OD	Spraying	50–100		2	7	label
rapeseed and sunflower	USA	100	OD	Spraying	50–150		3	7	Label, Maximum seasonal application rate 450 g ai/ha
	Canada	100	OD	Spraying	25–100		4	7	Label, Maximum seasonal application rate 450 g ai/ha
Seed for Beverage and Sweets (Group 024)									
Coffee	Columbia	100	OD	Spraying	60–175		2	7	label
Derived Products of Plant Origin (Group 066)									
Tea	Japan	100	SE	Spraying	100–200	2000–4000	1	7	label

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on supervised field trials following foliar, drip irrigation or seed treatment applications of cyantraniliprole to the following crops: strawberries, cucumbers (greenhouse), beans, peas, soya beans, artichokes, corn, almonds, pecans and tea.

The supervised trials were 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 samples 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. The notation for these two sampling times in the data tables is '–0' and '0' respectively.

When multiple applications were made to a crop, the application rates, spray concentrations and spray volumes 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.

The analytical methods used in the field trials were capable of analysing both cyantraniliprole and from one to seven metabolites (among them, four metabolites are considered in the residue definition). In most cases, residues of these metabolites were not detected (LOD of 0.003 mg/kg in most trials) or in some cases were reported at levels below the LOQ of 0.01 mg/kg. Where metabolite residues were present at levels above the LOQ, these values are recorded in the following tables using the abbreviations listed below:

- M1 = IN-J9Z38 2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]-3,4-dihydro-3,8-dimethyl-4-oxo-6-quinazolinecarbonitrile
- M2 = IN-MYX98 3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2[[(hydroxymethyl)amino]carbonyl]-6-methylphenyl]-1*H*-pyrazole-5-carboxamide
- M3 = IN-N7B69 3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2-(hydroxymethyl)-6-[(methylamino)carbonyl]phenyl]-1*H*-pyrazole-5-carboxamide
- M4 = IN-MLA84 2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]-1,4-dihydro-8-methyl-4-oxo-6-quinazolinecarbonitrile
- M5 = IN-JCZ384-[[[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]carbonyl]amino]-N′3′,5-dimethyl-1,3-benzenedicarboxamide
- M6 = IN-N5M09 6-Chloro-4-methyl-11-oxo-11*H*-pyrido[2,1-*b*]quinazoline-2-carbonitrile
- M7 = IN-F6L99 3-Bromo-N-methyl-1*H*-pyrazole-5-carboxamide

Citrus fruits

All trials from Europe and the USA on oranges, grapefruit, lemons and mandarins submitted to the Meeting were evaluated by the 2013 Meeting.

Table 2 Residues in oranges from supervised trials in the USA following three foliar applications of cyantraniliprole, SE formulation, (data previously reviewed by the 2013 JMPR)

ORANGE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Clermont, FL USA, 2009 (Hamlin)	3	0.15	535	28	7	1	peel pulp whole	0.37, 0.19 0.033, 0.028 0.19, 0.11	0.28 0.03 0.15		DP-27554 Test 01
Clermont, FL USA, 2009 (Mid Sweet)	3	0.15	535	28	7	1	peel pulp whole	0.56, 0.69 0.054, 0.074 0.31, 0.38	0.63 0.064 0.35		DP-27554 Test 02
Mascotte, FL USA, 2009 (Valencia—Early)	3	0.15	535	28	7	1	peel pulp whole	0.54, 0.39 0.08, 0.092 0.31, 0.24	0.46 0.086 0.28		DP-27554 Test 03
Oviedo, FL USA, 2009 (Navel)	3	0.15	11	1400	7	1	peel pulp whole	0.36, 0.36 0.053, 0.039 0.17, 0.17	0.36 0.046 0.17		DP-27554 Test 04
Oviedo, FL USA, 2009 (Hamlin)	3	0.15	11	1400	7	1	peel pulp whole	0.27, 0.14 0.026, 0.029 0.15, 0.085	0.21 0.027 0.12		DP-27554 Test 05
Mims, FL USA, 2009 (Hamlin)	3	0.15	20	700	7	1	peel pulp whole	0.48, 0.64 0.036, 0.043 0.26, 0.35	0.56 0.04 0.3		DP-27554 Test 06

ORANGE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Holopaw, FL USA, 2009 (Valencia)	3	0.15	21	700	7	1	peel pulp whole	0.34, 0.47 0.041, 0.045 0.18, 0.24	0.41 0.043 0.21		DP-27554 Test 07
Chuluota, FL USA, 2009 (Hamlin)	3	0.15	11	1400	7	1	peel pulp whole	0.69, 0.7 0.081, 0.092 0.37, 0.4	0.7 0.086 0.39		DP-27554 Test 08
Alamo, TX USA, 2009 (Valencia)	3	0.15	25	610	7	1	peel pulp whole	0.86, 0.91 0.071, 0.066 0.22, 0.23	0.88 0.069 0.22		DP-27554 Test 09
Sanger, CA USA, 2009 (Fisher)	3	0.15	25	610	7	1	peel pulp whole	0.23, 0.28 0.016, 0.02 0.087, 0.11	0.25 0.018 0.098	M1 = 0.01	DP-27554 Test 10 2009/02/25
Sanger, CA USA, 2009) (Campbell)	3	0.15	25	610	7	1	peel pulp whole	0.45, 0.35 0.017, 0.01 0.14, 0.1	0.4 0.013 0.12		DP-27554 Test 14 1009/04/08
Sanger, CA USA, 2009) (Navel)	3	0.15	8	1870	7	1	peel pulp whole	0.21, 0.21 0.038, 0.035 0.1, 0.1	0.21 0.036 0.1		DP-27554 Test 25 2009/09/18
Sanger, CA USA, 2009) (Washington Navel)	3	0.15	0.01	1550	7	1	peel pulp whole	0.7, 0.64 0.019, 0.024 0.2, 0.2	0.67 0.021 0.2		DP-27554 Test 26 2009/03/16

M1: Average residues of metabolite IN-J9Z38 reported in peel

Table 3 Residues in lemons from supervised trials in the USA following three foliar applications of cyantraniliprole, SE formulation, (data previously reviewed by the 2013 JMPR)

LEMON Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Newman, CA USA, 2009/2010 (Lisbon)	3	0.15	8	1870	7	1	peel pulp whole	0.42, 0.44 0.11, 0.11 0.21, 0.22	0.43 0.11 0.21		DP-27554 Test 19
Sanger, CA USA, 2009 (Lisbon)	3	0.15	25	610	7	1	peel pulp whole	0.3, 0.45 0.022, 0.024 0.13, 0.2	0.37 0.023 0.16		DP-27554 Test 20
Sanger, CA USA, 2009 (Frost Lisbon)	3	0.15	10	1560	7	1	peel pulp whole	0.62, 0.63 0.068, 0.057 0.31, 0.3	0.63 0.063 0.3		DP-27554 Test 21 2009/04/02
Sanger, CA USA, 2009/2010 (Eureka)	3	0.15	33	470	7	1	peel pulp whole	0.34, 0.39 0.069, 0.071 0.18, 0.2	0.36 0.07 0.19		DP-27554 Test 22 2009/01/04
Sanger, CA USA, 2009 (Lizbon 8A)	3	0.16	8	1870	7	1	peel pulp whole	0.32, 0.39 0.059, 0.066 0.14, 0.17	0.35 0.063 0.16		DP-27554 Test 23 2009/01/04
Elderwood, CA USA, 2009 (Lizbon)	3	0.15	32	470	7	1	peel pulp whole	0.24, 0.42 0.037, 0.077 0.11, 0.21	0.33 0.057 0.16		DP-27554 Test 24

Table 4 Residues in grapefruit from supervised trials in the USA following three foliar applications of cyantraniliprole, 100 SE formulation, (data previously reviewed by the 2013 JMPR)

GRAPEFRUIT Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Mims, FL USA, 2009 (White Marsh)	3	0.15	21	700	7	1	peel pulp whole	0.35, 0.34 0.022, 0.019 0.14, 0.14	0.35 0.021 0.14		DP-27554 Test 11
Oviedo, FL USA, 2009 (Flame)	3	0.15	11	1400	7	1	peel pulp whole	0.41, 0.43 0.028, 0.037 0.18, 0.2	0.42 0.032 0.19		DP-27554 Test 12
Holopaw, FL USA, 2009 (White)	3	0.15	10	1500	7	1	peel pulp whole	0.77, 0.67 0.043, 0.055 0.33, 0.3	0.72 0.049 0.31		DP-27554 Test 13
Alamo, TX USA, 2009 (Rio Red)	3	0.15	6	2400	7	1	peel pulp whole	0.45, 0.28 0.032, 0.019 0.11, 0.21	0.36 0.026 0.16	M1 = 0.015	DP-27554 Test 15
Elderwood, CA USA, 2009 Duncan	3	0.15	32	470	7	1	peel pulp whole	0.26, 0.18 0.035, 0.03 0.11, 0.076	0.22 0.033 0.091		DP-27554 Test 16
Sanger, CA USA, 2009 (Rio Red)	3	0.15	0.025	620	7	1	peel pulp whole	0.32, 0.29 0.02, 0.039 0.12, 0.12	0.3 0.029 0.12		DP-27554 Test 17 2009/03/11
Sanger, CA USA, 2009 (Marsh White)	3	0.15	0.01	1560	7	1	peel pulp whole	0.31, 0.34 0.012, 0.016 0.11, 0.13	0.33 0.014 0.12		DP-27554 Test 18 2009/04/02

M1: Average residues of metabolite IN-J9Z38 reported in peel

Table 5 Residues in lemons from supervised trials in the USA following soil band applications of cyantraniliprole, 200 SC formulation, (data previously reviewed by the 2013 JMPR)

LEMON Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	water L/tree			cyantraniliprole	mean	metabolites	
Sanger, CA USA, 2009/2010 (Eureka)	1	0.45	117	390	0.95	1 7 14 1 7 14 1 7 14	peel pulp whole 	< 0.01 ND ND ND ND ND < 0.01 ND ND			DP-27554 Test 22
Sanger, CA USA, 2009 (Lizbon 8A)	1	0.45	0.16	280	0.95	1 7 14 1 7 14 1 7 14	peel pulp whole 	ND < 0.01 ND ND ND ND ND < 0.01 ND			DP-27554 Test 23

LEMON Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	water L/tree			cyantraniliprole	mean	metabolites	
Elderwood, CA USA, 2009 (Lizbon)	1	0.45	0.17	260	0.95	1 7 14 1 7 14 1 7 14	peel pulp whole 	ND ND ND ND ND ND ND ND ND			DP-27554 Test 24

Table 6 Residues in oranges from supervised trials in Europe following foliar two/three applications of cyantraniliprole, 100 SE formulation

ORANGE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	No	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Kostaki Greece, 2009 (Salustiana)	3	0.15	0.01	1500	7	–0 1	peel pulp whole peel pulp whole	0.65 0.043 0.23 0.79 0.041 0.26			DP-27716 Test 01
Sicily Italy, 2009 (Tarocco)	3	0.15	0.01	1500	7	–0 1	peel pulp whole peel pulp whole	0.85 0.004 0.2 0.9 0.007 0.23			DP-27716 Test 02

Table 7 Residues in mandarins from supervised trials in Europe following two/three foliar applications of cyantraniliprole, 100 SE formulation, (data previously reviewed by the 2013 JMPR)

MANDARIN Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg			Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)	RTI (days)			cyantraniliprole	mean	metabolites	
Kostaki Greece, 2009 (Clementine)	3	0.15	0.01	1500	7	–0 1	peel pulp whole peel pulp whole	1.1 0.08 0.38 1.1 0.2 0.47		M1 = 0.01 M1 = 0.014	DP-27716 Test 03

M1: Average residues of metabolite IN-J9Z38 reported in peel

Berries and other small fruits

Strawberry

In trials on strawberries conducted in Europe, two to four foliar applications of 0.075 kg ai/ha cyantraniliprole (OD formulation) were applied at 6–7 day intervals, using 500–800 L/ha, with adjuvant added, or 2–4 drip irrigation of 0.075 kg ai/ha cyantraniliprole (SC formulation) were applied at 7 day intervals, using 3× vol tubing, with no adjuvant added.

Samples were stored at –18 °C for up to 9 months before analysis (within 5 days of extraction) for cyantraniliprole and six metabolites using analytical method DP15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 98–101% (cyantraniliprole) and 96–106% (metabolites) in samples spiked with 0.01 and 0.1 mg/kg.

Table 8 Residues in protected strawberries from supervised trials in EU following four foliar applications of cyantraniliprole, 100 OD formulation

Strawberry Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Horst-Meterik, Limburg, Netherlands, 2011 (Elsanta)	4	0.075	9.38	800	7	1	mature fruit	0.16		0.004(J9Z38)	DP29223 Test 01
Wellerlooi, Limburg, Netherlands, 2011 (Elsanta)	4	0.075	9.38	800	7	–0 0 1 3 5	mature fruit	0.19 0.22 0.22 0.23 0.19		0.012 0.007(J9Z38) 0.012(J9Z38) 0.010(J9Z38)	DP29223 Test 02
Svoronos, Central Macedonia, Greece, 2011 (Kamaroza)	4	0.075	9.38	800	7	1	mature fruit	0.26		0.011(J9Z38)	DP29223 Test 03
Contrada Spinagallo, Siracusa, Sicily, 2011 (Carmela)	4	0.075	9.38	800	7	1	mature fruit	0.23		0.009(J9Z38)	DP29223 Test 04
La Rive Haute, Aquitaine, South France, 2011 (Darselect)	4	0.075	9.38	800	7	1	mature fruit	0.050			DP29223 Test 05
Pact, Rhone-Alpes, South France, 2011 (Darselect)	4	0.075	9.38	800	7	–0 0 1 3 5	mature fruit	0.086 0.14 0.13 0.089 0.080		0.008(J9Z38) 0.008(J9Z38) 0.008(J9Z38) 0.005(J9Z38) 0.005(J9Z38)	DP29223 Test 06

Strawberry Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Bonares, Andalucia, South Spain, 2011 (Candonga)	4	0.075	9.38	800	7	–0 0 1 3 5	mature fruit	0.12 0.14 0.13 0.10 0.10		0.009(J9Z38) 0.006(J9Z38) 0.005(J9Z38) 0.006(J9Z38) 0.005(J9Z38)	DP29223 Test 07
Puerto Serrano, Andalucia, South Spain, 2011 (Camarosa)	4	0.075	9.38	800	7	–0 0 1 3 5	mature fruit	0.076 0.16 0.17 0.16 0.088		0.007(J9Z38) 0.010(J9Z38) 0.010(J9Z38) 0.012(J9Z38) 0.004(J9Z38)	DP29223 Test 08
Lucena del Puerto, Andalucia, South Spain, 2011 (Splendor)	4	0.075	9.38	800	7	–0 0 1 3 5	mature fruit	0.10 0.17 0.13 0.14 0.12		0.010(J9Z38) 0.009(J9Z38) 0.007(J9Z38) 0.009(J9Z38) 0.007(J9Z38)	DP29223 Test 09

Table 9 Residues in field strawberries from supervised trials in EU following two foliar applications of cyantraniliprole, 100 OD formulation

Strawberry Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Leisnig Saxony, Gemany, 2011 (Sonata)	2	0.075	30.84	15.42	8	1	mature fruit	0.10			DP29223 Test 10
Gerpinnes, Hainaut, Belgium, 2011 (Darselect)	2	0.075	9.38	800	7	1	mature fruit	0.054			DP29223 Test 11
Beugny, Nord-Pas de Calais, North France, 2011 (Darselect)	2	0.075	9.38	800	7	–0 0 1 3 5	mature fruit	0.027 0.071 0.040 0.030 0.033			DP29223 Test 12

Dairsie, Fife, UK North, 2011 (Elsanta)	2	0.075	13.63	560	7	–0 0 1 3 5	mature fruit	0.020 0.049 0.043 0.037 0.034			DP29223 Test 13
Leisnig Saxony, Gemany, 2012 (Sonata)	2	0.075	15.44	500	7	1	mature fruit	0.045			DP29223 Test 15
Mortemer, Picardie, North France, 2012 (Darselect)	2	0.075	9.38	800	6	1	mature fruit	0.12		0.005(J9Z38	DP29223 Test 16
Marbais, Brabant Wallon, Belgium, 2012 (Sonata)	2	0.075	9.37	770	7	–0 0 1 3 5	mature fruit	0.021 0.082 0.045 0.051 0.030		0.004(J9Z38	DP29223 Test 17
Fotheringhay, Cambs, UK South, 2012 (Elsanta)	2	0.075	9.38	800	8	–0 0 1 3 5	mature fruit	0.035 0.071 0.051 0.054 0.043		0.003(J9Z38 0.004(J9Z38	DP29223 Test 18

Table 10 Residues in protected strawberries from supervised trials in EU following four drip irrigations of cyantraniliprole, 200 SC formulation

Strawberry Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Wellerlooi, Limburg, Netherlands, 2012 (Elsanta)	4	0.075	3.75	2000	7	–0 0 1 5 10	mature fruit	0.004 ND 0.005 0.003 0.004		< 0.003	DP34085 Test 01
Horst-Meterik, Limburg, Netherlands, 2012 (Lambada)	4	0.075	3.75	2000	7	–0 0 1 5 10	mature fruit	< 0.003 < 0.003 < 0.003 < 0.003 < 0.003			DP34085 Test 02
Pact, Rhone-Alpes, South France, 2012 (Darselect)	4	0.075	3.75	2000	7	–0 0 1 5 10	mature fruit	< 0.003 < 0.003 < 0.003 < 0.003 < 0.003			DP34085 Test 03
Svoronos,Pieria, Central Macedonia, Greece, 2012 (Kamaroza)	4	0.075	3.75	2000	7	–0 0 1 5 10	mature fruit	0.006 0.007 0.007 0.006 0.006		0.003(J9Z38	DP34085 Test 04
Lucena del Puerto,Andalucia, Spain, 2012 (Splendor)	4	0.075	3.75	2000	7	–0 0 1 5 10	mature fruit	0.025 0.029 0.030 0.025 0.022		0.012(J9Z38) 0.013(J9Z38) 0.012(J9Z38)0.009(J9Z38) 0.008(J9Z38)	DP34085 Test 05

Table 11 Residues in field strawberries from supervised trials in EU following two drip irrigations of cyantraniliprole, 200 SC formulation

Strawberry Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Gembloux, Namur, Belgium, 2012 (Elsanta)	2	0.075	3.75	2000	7	–0 0 1 5 10	mature fruit	< 0.003 0.004 0.004 < 0.003 < 0.003			DP34085 Test 06
Beugny, Nord-Pas de Calais, North France, 2012 (Darselect)	2	0.075	3.75	2000	7	–0 0 1 5 10	mature fruit	< 0.003 0.012 0.003 < 0.003 < 0.003			DP34085 Test 07
Goch-Kessel, Nordrhein-Westfalen, Germany, 2012 (Sonata)	2	0.075	3.75	2000	7	–0 0 1 5 10	mature fruit	< 0.003 0.011 0.017 0.004 < 0.003			DP34085 Test 08
Fotheringhay, Cambridgeshire, UK South, 2012 (Elsanta)	2	0.075	3.75	2000	7	–0 0 1 5 10	mature fruit	< 0.003 < 0.003 < 0.003 < 0.003 < 0.003			DP34085 Test 09
Tattenhall, Cheshire, UK South, 2012 (Flamenco)	2	0.075	3.75	2000	7	–0 0 1 5 10	mature fruit	< 0.003 < 0.003 < 0.003 < 0.003 < 0.003			DP34085 Test 10

Pomegranate

Table 12 Residues in pomegranates from supervised trials in India following foliar two to five applications of cyantraniliprole, 100 OD formulation, (data previously reviewed by the 2013 JMPR)

POMEGRANATE Location Country, year (variety)	Application					DAT (days)	Cyantraniliprole residues (mg/kg)				Reference & Comments
	No	kg ai/ha	g ai/hL	water L/ha	RTI (days)		Rind (parent)	Rind M1	Seed	Juice	
Raichur India, 2011	2	0.075	12.5–19	400-600	10	0 1 3 5	0.05 0.03 0.006 < 0.003	0.03	< 0.003 < 0.003 < 0.003 < 0.003	< 0.003 < 0.003 < 0.003 < 0.003	IIBAT-1104829 Trial 1
Raichur India, 2011	2	0.09	15–23	400-600	10	0 1 3 5	0.07 0.03 0.008 < 0.003	M1=0.035	< 0.003 < 0.003 < 0.003 < 0.003	< 0.003 < 0.003 < 0.003 < 0.003	IIBAT-1104829 Trial 1
Raichur India, 2011	2	0.18	30–45	400-600	10	0 1 3 5	0.14 0.07 0.01 < 0.003	M1=0.065	< 0.003 < 0.003 < 0.003 < 0.003	< 0.003 < 0.003 < 0.003 < 0.003	IIBAT-1104829 Trial 1
Rahuri India, 2011	5	0.075	15	500	10	0 1 3 5	0.07 0.05 0.01 < 0.003	M1=0.02	< 0.003 < 0.003 < 0.003 < 0.003	< 0.003 < 0.003 < 0.003 < 0.003	IIBAT-1104829 Trial 2

POMEGRANATE Location Country, year (variety)	Application					DAT (days)	Cyantraniliprole residues (mg/kg)				Reference & Comments
	No	kg ai/ha	g ai/hL	water L/ha	RTI (days)		Rind (parent)	Rind M1	Seed	Juice	
Rahuri India, 2011	5	0.09	18	500	10	0 1 3 5	0.08 0.06 0.01 < 0.003	M1=0.03	< 0.003 < 0.003 < 0.003 < 0.003	< 0.003 < 0.003 < 0.003 < 0.003	IIBAT-1104829 Trial 2
Rahuri India, 2011	5	0.18	36	500	10	0 1 3 5	0.17 0.12 0.03 < 0.003	M1 = 0.05 M1 = 0.02	< 0.003 < 0.003 < 0.003 < 0.003	< 0.003 < 0.003 < 0.003 < 0.003	IIBAT-1104829 Trial 2
Medhak India, 2011	3	0.075	7.5	1000	10	0 1 3 5	0.04 0.03 0.005 < 0.003	M1 = 0.02	< 0.003 < 0.003 < 0.003 < 0.003	< 0.003 < 0.003 < 0.003 < 0.003	IIBAT-1104829 Trial 3
Medhak India, 2011	3	0.09	9	1000	10	0 1 3 5	0.05 0.02 0.006 < 0.003	M1 = 0.02	< 0.003 < 0.003 < 0.003 < 0.003	< 0.003 < 0.003 < 0.003 < 0.003	IIBAT-1104829 Trial 3
Medhak India, 2011	3	0.18	18	1000	10	0 1 3 5	0.09 0.04 0.009 < 0.003	M1 = 0.04	< 0.003 < 0.003 < 0.003 < 0.003	< 0.003 < 0.003 < 0.003 < 0.003	IIBAT-1104829 Trial 3
Trichy India 2011	5	0.075	12.5	600	10	0 1 3 5	0.06 0.03 0.01 < 0.003		< 0.003 < 0.003 < 0.003 < 0.003	< 0.003 < 0.003 < 0.003 < 0.003	IIBAT-1104829 Trial 4
Trichy India 2011	5	0.09	15	600	10	0 1 3 5	0.08 0.03 0.01 < 0.003		< 0.003 < 0.003 < 0.003 < 0.003	< 0.003 < 0.003 < 0.003 < 0.003	IIBAT-1104829 Trial 4
Trichy India 2011	5	0.18	30	600	10	0 1 3 5	0.16 0.06 0.03 < 0.003	M1 = 0.02 M1 = 0.01	< 0.003 < 0.003 < 0.003 < 0.003	< 0.003 < 0.003 < 0.003 < 0.003	IIBAT-1104829 Trial 4

M1: Residues of metabolite IN-J9Z38

Cucurbit vegetables

Cucumber

In trials conducted in North America on greenhouse cucumbers, three foliar applications of 0.15 kg ai/ha cyantraniliprole (SE formulation) were applied at 5 day intervals, using 300–1200 L/ha with adjuvant added.

Duplicate samples were stored at –20 °C for up to 11 months before analysis of whole fruit or pulp and peel for cyantraniliprole and six metabolites using an adaptation of method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 92–98% (cyantraniliprole) and 86–114% (metabolites) in samples spiked with 0.01, 0.1, 0.2 and 0.6 mg/kg.

Table 13 Residues in greenhouse cucumber from supervised trials in North America following three foliar applications of cyantraniliprole, 100 SE formulation

Cucumber Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Parlier, CA, USA 2010 (Manar F1)	3	0.15	40.0	400	5–6	0	mature fruit	0.20, 0.19	0.19	< 0.01(J9Z38) < 0.01(MLA84) < 0.01(MYX98) < 0.01(N7B69) < 0.01(JCZ38) < 0.01(K7H19)	IR4 Study No.10313 Test CA67
Citra, FLA, USA 2010 (Jawell)	3	0.15	50	300	4–5	0	mature fruit	0.33, 0.32	0.33	< 0.01(J9Z38) < 0.01(MLA84) < 0.01(MYX98) < 0.01(N7B69) < 0.01(JCZ38) < 0.01(K7H19)	IR4 Study No.10313 Test FL14
Salisbury, MD, USA 2010 (Danito)	3	0.15	32	460	4	0	mature fruit	0.039, 0.047	0.043	< 0.01(J9Z38) < 0.01(MLA84) < 0.01(MYX98) < 0.01(N7B69) < 0.01(JCZ38) < 0.01(K7H19)	IR4 Study No.10313 Test MD10
Raleigh, NC, USA 2010 (Jawell)	3	0.15	36	430	5	0	mature fruit	0.18, 0.18	0.18	< 0.01(J9Z38) < 0.01(MLA84) < 0.01(MYX98) < 0.01(N7B69) < 0.01(JCZ38) < 0.01(K7H19)	IR4 Study No.10313 Test NC12
Harrow, ON, Canada, 2010 (Camaro)	3	0.15	13	1200	5	0	mature fruit	0.027, 0.036	0.032	< 0.01(J9Z38) < 0.01(MLA84) < 0.01(MYX98) < 0.01(N7B69) < 0.01(JCZ38) < 0.01(K7H19)	IR4 Study No.10313 Test ON12

Legume vegetables (Group 014)

Pea—Europe

In trials conducted in Europe on peas (without pods, fresh) in the field, two applications of 0.075 kg ai/ha cyantraniliprole (WG formulation) were applied 7 days interval, using 200–1000 L spray mix/ha with added surfactants.

Samples of pods (with seeds) and foliage (leaves and stems) were stored at –18 °C for up to 10 months before extraction and analysis for cyantraniliprole and six metabolites (same day of extraction) using method DP15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 81–104% (cyantraniliprole) and 80–101% (metabolites) in samples spiked with 0.01, 0.1, 0.2, 1.0, 3.4 mg/kg and also 5 mg/kg cyantraniliprole.

Table 14 Residues in field peas without pods(fresh) from supervised trials in Europe following two foliar applications of cyantraniliprole, 400 g/kg WG formulation

Peas without pods(fresh) Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	Water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Market Weighton, East Yorkshire, United Kingdom, 2011 (Fresh)	2	0.075		200–1000	7	0 1 3(NCH) 7 14	Peas	0.11 0.09 0.05 0.05 0.01			Syngenta TK0057194 Test 01
Driffield, East Yorkshire, United Kingdom, 2011 (Fresh)	2	0.075			7	0 1 3(NCH) 7 14	Peas	0.38 0.13 0.08 0.04 0.02		0.01(J9Z38) 0.02(J9Z38)0.01(J9Z38)0.02(J9Z38) 0.02(J9Z38)	Syngenta TK0057194 Test 02
Sulniac, Bretagne, N. France, 2011 (Fresh)	2	0.075			7	0 1 3(NCH) 7 14	Peas	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01			Syngenta TK0057194 Test 03
Oinville Saint Liphard, Eure et Loire, N. France, 2011 (Fresh)	2	0.075			7	0 1 3(NCH) 7 14	Peas	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01			Syngenta TK0057194 Test 04
Behagnies, 62121, N. France, 2012 (Fresh)	2	0.075			7	1 3(NCH) 6	Peas	0.02 < 0.01 < 0.01			Syngenta TK0112971 Test 05
Mulfingen, 74673, Germany, 2012 (Fresh)	2	0.075			7	1 3(NCH) 7	Peas	< 0.01 < 0.01 < 0.01			Syngenta TK0112971 Test 06
Bretzfeld-Schwabbach, 74626, Germany, 2012 (Fresh)	2	0.075			7	1 3(NCH) 7	Peas	< 0.01 < 0.01 < 0.01			Syngenta TK0112971 Test 07
Cagnicourt, 62182, N. France, 2012 (Fresh)	2	0.075			7	1 3(NCH) 7	Peas	0.06 0.04 0.02			Syngenta TK0112971 Test 08
Houeilles, Lot et Garonne, Aquitaine, S France, 2011 (Fresh)	2	0.075			7	0 1 2(NCH) 7 14	Peas	0.03 0.04 0.04 0.02 0.02			Syngenta TK0057193 Test 09

Peas without pods(fresh) Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	Water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Elne, Pyrenees Orientales, Elne, S France, 2011 (Fresh)	2	0.075			7	0 1 2(NCH) 6 14	Peas	0.61 0.51 0.05 0.03 0.01			Syngenta TK0057193 Test 10
Granarolo, Emilia Romagna, Bologna, Italy, 2011 (Fresh)	2	0.075			7	0 1 3(NCH) 6 14	Peas	0.07 < 0.01 < 0.01 < 0.01 < 0.01			Syngenta TK0057193 Test 11
Villar de Chinchilla, Albacete, Spain, 2011 (Fresh)	2	0.075			7	0 1 3(NCH) 6 14	Peas	0.02 0.02 < 0.01 < 0.01 0.01			Syngenta TK0057193 Test 12
Montpouillan, 47200, S France, 2012 (Fresh)	2	0.075			7	1 3(NCH)	Peas	< 0.01 < 0.01			Syngenta TK0112985 Test 13
Saint Agnet, 40800, S. France, 2012 (Fresh)	2	0.075			7	1 3(NCH) 7	Peas	< 0.01 0.01 < 0.01			Syngenta TK0112985 Test 14
La Gineta, 02110, Spain, 2012 (Fresh)	2	0.075			7	1 3(NCH) 7	Peas	< 0.01 < 0.01 < 0.01			Syngenta TK0112985 Test 15
Papiano Marsciano, 06055, Italy, 2012 (Fresh)	2	0.075			7	1 3(NCH) 7	Peas	< 0.01 0.01 0.01			Syngenta TK0112985 Test 16

Bean/Pea—North America

In trials conducted in Northern America on bean/peas (edible-podded, succulent shelled, dry shelled) in the field, three foliar applications of 0.15 kg ai/ha cyantraniliprole (SE/OD formulation) were applied at 5 day intervals, using 200–500 L spray mix/ha with added surfactants.

Samples of pods (with seeds) and foliage (leaves and stems) were stored at –20 °C for up to 14 months before extraction and analysis for cyantraniliprole and six metabolites using method DP15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 81–104% (cyantraniliprole) and 75–102% (metabolites) in samples spiked with 0.01, 0.1, 1.0, 2.0, and 4.0 mg/kg cyantraniliprole.

Table 15 Residues in beans with pod (edible-podded bean) from supervised trials in the USA following three foliar applications of cyantraniliprole, 100 SE formulation

Bean with pod Location Country, year (variety)	Application						DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)	Growth stage			cyantraniliprole	mean	metabolites	
Germansville, PA, USA, 2011 (Savannah)	3	0.15	50	300	5		1	seed	0.41 0.46	0.43	0.016 (J9Z38) 0.007(MLA84)	DP 31668 Test 01 100 SE
Athens, GA, USA, 2011 (Blue Lake 274)	3	0.15	64	235	5		1	seed	0.42 0.30	0.36	0.013 (J9Z38) 0.006(MLA84)	DP 31668 Test 02 100 SE
Oviedo, FL, USA, 2011 (Provider Snap Bean)	3	0.15	54	280	5		1	seed	0.76 0.70	0.73	0.044 (J9Z38) 0.006(MLA84)	DP 31668 Test 03 100 SE
Geneva, MN, USA, 2011 (Top Crop)	3	0.15	80	190	4		1	seed	0.11 0.11	0.11	0.011 (J9Z38) 0.005(MLA84)	DP 31668 Test 04 100 SE
Northwood, ND, USA, 2011 (Top Crop)	3	0.15	54	280	4–5		1	seed	0.29 0.28	0.29	0.021 (J9Z38)	DP 31668 Test 05 100 SE
Richland, IA, USA, 2011 (Top Crop)	3	0.15	68	215	5		1	seed	0.21 0.25	0.23	0.016 (J9Z38)	DP 31668 Test 06 100 SE
Ephrata, WA, USA, 2011 (OSU 5630)	3	0.15	54	281	5		1	seed	0.10 0.11	0.11	0.009 (J9Z38)	DP 31668 Test 07 100 SE

Table 16 Residues in bean without pod (succulent shelled beans) from supervised trials in the USA following three foliar applications of cyantraniliprole, 100 SE formulation

Beans without pod Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Kerman, CA, USA, 2011 (Blue Lake 274)	3	0.15	55	280	5	1	seed	0.019 0.028	0.023		DP 31668 Test 31 100 SE
Payette, ID, USA, 2011 (Fordhook 242)	3	0.15	65	234	5–6	1	seed	0.010 0.008	0.009		DP 31668 Test 32 100 SE
Payette, ID, USA, 2011 (Fordhook 242)	3	0.15	64	235	4–6	1	seed	0.050 0.065	0.057	0.006 (J9Z38)	DP 31668 Test 33 100 SE

Table 17 Residues in pea with pod (edible-podded peas) from supervised trials in the USA following three foliar applications of cyantraniliprole, 100 SE and 100 OD formulation

Peas with pod Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Lenexa, KS, USA, 2011 (Melting Mammoth Sugar)	3	0.15	75	200	4–5	1	seed	0.81 0.72	0.76	0.009 (J9Z38) 0.007(MLA84)	DP 31668 Test 08 100 SE
								0.77 0.81	0.79	0.008 (J9Z38) 0.005(MLA84)	DP 31668 Test 08 100 OD
Geneva, MN, USA, 2011 (Cascadia)	3	0.15	78	190	4	1	seed	0.53	0.53	0.012 (J9Z38)	DP 31668 Test 09 100 SE
								0.63 0.58	0.61	0.013 (J9Z38)	DP 31668 Test 09 100 OD
Northwood, ND, USA, 2011 (Maestro)	3	0.15	54	280	5	1	seed	0.70 0.71	0.70	0.016 (J9Z38) 0.004(MLA84)	DP 31668 Test 10 100 SE
								0.83 0.74	0.78	0.014 (J9Z38)	DP 31668 Test 10 100 OD
Ephrata, WA, USA, 2011 (Sugar Bro)	3	0.15	53	281	5	1	seed	0.25 0.26	0.25		DP 31668 Test 11 100 SE
								0.29 0.30	0.29		DP 31668 Test 11 100 OD

Table 18 Residues in pea without pod (succulent shelled pea) from supervised trials in the USA following foliar applications of cyantraniliprole, 100 SE formulation

Pea without pod Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Germansville, PA, USA, 2011 (Strike)	3	0.15	65	234	5	1	seed	0.071 0.094	0.082	0.007 (J9Z38)	DP 31668 Test 12 100 SE
Geneva, MN, USA, 2011 (Green Arrow)	3	0.15	80	200	4–6	1	seed	0.052 0.040	0.046	0.006 (J9Z38)	DP 31668 Test 13 100 SE
Gardner, ND, USA, 2011 (Knight Peas)	3	0.15	65	234	4–5	1	seed	0.066 0.064	0.065	0.011 (J9Z38)	DP 31668 Test 14 100 SE
Marysville, OH, USA, 2011 (Knight Peas)	3	0.15	77	195	6	1	seed	0.020 0.018	0.019		DP 31668 Test 15 100 SE

Pea without pod Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Richland, IA, USA, 2011 (Laxton's Progress #9)	3	0.15	95	160	4–6	1	seed	0.099 0.10	0.10		DP 31668 Test 16 100 SE
Mt. Hood-Parkdale, OR, USA, 2011 (Progress #9)	3	0.15	65	236	5	1	seed	0.082 0.069	0.076		DP 31668 Test 17 100 SE

Soya bean

In trials conducted in Northern America on soya beans (edible-podded, succulent shelled, dry shelled) in the field, three foliar applications of 0.15 kg ai/ha cyantraniliprole (OD formulation) were applied at 5 day intervals, using 150–300 L spray mix/ha with added surfactants, and 0.04–0.08 g ai/ha of seed treatment

Table 19 Residues in soya beans from supervised trials in the USA following three foliar applications of cyantraniliprole, 100 OD formulation

Soya beans Location Country, year (variety)	Application						DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	Growth stage	RTI (days)			cyantraniliprole	mean	metabolites	
Frenchtown, NJ USA, 2011 (Pioneer 93M14)	3	0.15	50	304	R5 R5 R5-R6	4–6	6	Immature Seed	0.035 0.036	0.035	0.009(J9Z38) 0.005(MLA84)	DP29956 Trial 01 100 OD
Athens, GA, USA, 2011 (Pioneer 95Y20))	3	0.15	48	314	R5-R6 R5-R6 R6	5	7	Immature Seed	0.047 0.038	0.042		DP29956 Trial 02 100 OD
Blackville, SC, USA, 2011 (Pioneer 95Y20)	3	0.15	72	209	R5 R5 R6	5	7	Immature Seed	0.018 0.019	0.019		DP29956 Trial 03 100 OD
Ellendale, MN, USA, 2011 (92Y30)	3	0.15	80	192	R5 R5.5 R6	4–6	7	Immature Seed	0.038 0.033	0.036	0.008(J9Z38)	DP29956 Trial 07 100 OD
Gardner, ND, USA, 2011 (NK Seeds: Variety S02-M9)	3	0.15	64	234	R5 R5 R5	5	7	Immature Seed	0.12 0.16	0.14	0.006(J9Z38)	DP29956 Trial 08 100 OD

Pulses

Dry Bean/Pea

Table 20 Residues in bean, dry (dry shelled bean) from supervised trials in the USA following three foliar applications of cyantraniliprole, 100 SE and 100 OD formulations

Bean, Dry Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Northwood, ND, USA, 2011 (Ensign - ADM)	3	0.15	54	281	6–4	7	seed	0.004 0.004	0.004		DP 31668 Test 18 100 OD
Carrington, ND, USA, 2011 (Ensign)	3	0.15	54	281	5–6	7	seed	< 0.003 0.005	0.003		DP 31668 Test 19 100 OD
Larned, KS, USA, 2011 (Poncho Pinto)	3	0.15	73	205	5	8	seed	0.039 0.056	0.048		DP 31668 Test 20 100 OD
Jerome, ID, USA, 2011 (Small Reds)	3	0.15	61	220	6–4	8	seed	0.009 0.009	0.009		DP 31668 Test 21 100 OD
Jerome, ID, USA, 2011 (Seminis SNO-112-0490-N14)	3	0.15	75	205	4–5	7	seed	0.050 0.048	0.049		DP 31668 Test 22 100 OD
Marysville, OH, USA, 2011 (Espada)	3	0.15	75	200	5	7	seed	< 0.003, < 0.003	< 0.003		DP 31668 Test 23 100 SE
							seed	< 0.003 < 0.003	< 0.003		DP 31668 Test 23 100 OD
Lenexa, KS, USA, 2011 (Pinkeye-Purple Hull) Lenex	3	0.15	70	215	4–5	7	seed	0.005 0.007	0.006	0.004 (J9Z38)	DP 31668 Test 24 100 SE
					4–5	7	seed	0.004 0.004	0.004	0.005 (J9Z38)	DP 31668 Test 24 100 OD
Stafford, KS, USA, 2011 (Cow Pea)	3	0.15	70	210	5	7	seed	0.24 0.19	0.22	0.06 (J9Z38)	DP 31668 Test 25 100 SE
							seed	0.085 0.13	0.11	0.030 (J9Z38)	DP 31668 Test 25 100 OD
York, NE, USA, 2011 (California Blackeye #5)	3	0.15	80	190	5	8	seed	0.060 0.056	0.058	0.007 (J9Z38)	DP 31668 Test 26 100 SE
					5	8	seed	0.072 0.10	0.088	0.009 (J9Z38)	DP 31668 Test 26 100 OD

Table 21. Residues in beans, dry (dry shelled beans) from supervised trials in the USA following three foliar applications of cyantraniliprole, 100 OD formulation

Beans, dry Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Kerman, CA, USA, 2011 (Blue Lake 274)	3	0.15	55	280	5	7	seed	0.022 0.019	0.021		DP 31668 Test 31 100 OD
Payette, ID, USA, 2011 (Fordhook 242)	3	0.15	65	234	5–6	8	seed	< 0.003 0.004	< 0.003		DP 31668 Test 32 100 OD
Payette, ID, USA, 2011 (Fordhook 242)	3	0.15	64	235	4–5	6	seed	0.018 0.011	0.015		DP 31668 Test 33 100 OD

Table 22 Residues in pea, dry (dry shelled pea) from supervised trials in the USA following three foliar applications of cyantraniliprole, 100 SE and 100 OD formulations

Pea, Dry Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Payette, ID, USA, 2011 (Austrian Winter)	3	0.15	65	235	4–6	0	Seed	0.48 0.50	0.49	0.012(J9Z38)	DP 31668 Test 27 100 OD
					4–6	1		0.31 0.80	0.56	0.009 (J9Z38)	DP 31668 Test 27 100 OD
					4–6	3		0.29 0.42	0.35	0.019(J9Z38)	DP 31668 Test 27 100 OD
					4–6	5		1.4 0.46	0.93	0.014 (J9Z38) 0.004(MYX98)	DP 31668 Test 27 100 OD
					4–6	7		0.34 0.67	0.51	0.006 (J9Z38)	DP 31668 Test 27 100 OD
Jerome, ID, USA, 2011 (Austrian)	3	0.15	75	200	4–5	0	Seed	0.14 0.13	0.13		DP 31668 Test 28 100 OD
					4–5	1		0.13 0.11	0.12		DP 31668 Test 28 100 OD
					4–5	4		0.10 0.12	0.11		DP 31668 Test 28 100 OD
					4–5	5		0.063 0.080	0.071		DP 31668 Test 28 100 OD
					4–5	7		0.073 0.081	0.077		DP 31668 Test 28 100 OD

Pea, Dry Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Ephrata, WA, USA, 2011 (Austrian Winter)	3	0.15	53	281	5	7	Seed	0.017 0.020	0.019		DP 31668 Test 29 100 OD
Jerome, ID, USA, 2011 (Austrian Winter)	3	0.15	75	200	6	7	Seed	0.083 0.088	0.086		DP 31668 Test 30 100 OD

Soya bean, dry

Table 23 Residues in soya beans from supervised trials in the USA following three foliar applications of cyantraniliprole, 100 OD formulation

Soya beans Location Country, year (variety)	Application						DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	Growth stage	RTI (days)			cyantraniliprole	mean	metabolites	
Frenchtown, NJ USA, 2011 (Pioneer 93M14)	3	0.15	55	281	R7 R8 R8	5	7	Mature Seed	0.026 0.028	0.027		DP29956 Trial 01 100 OD
Athens, GA, USA, 2011 (Pioneer 95Y20)	3	0.15	49	315	R6-R7 R6-R7 R7-R8	5	7	Mature Seed	0.26 0.23	0.25	0.027(J9Z38)	DP29956 Trial 02 100 OD
Blackville, SC, USA, 2011 (Pioneer 95Y20)	3	0.15	74	205	R7 R7 R8	5–6	8	Mature Seed	0.009 0.014	0.011		DP29956 Trial 03 100 OD
Cheneyville, LA, USA, 2011 (Pioneer 95Y20)	3	0.15	63	246	BBCH80–81 BBCH85 BBCH88–89	4–5	7	Mature Seed	0.021 0.041	0.031		DP29956 Trial 04 100 OD
Fisk, MO, USA, 2011 (95Y50)	3	0.15	80	188	BBCH87 BBCH89 BBCH89	5	6	Mature Seed	0.028 0.026	0.027		DP29956 Trial 05 100 OD
Pollard, AR, USA, 2011 (Pioneer 95M50)	3	0.15	80	188	BBCH87 BBCH89 BBCH89	5	6	Mature Seed	0.012 0.013	0.012		DP29956 Trial 06 100 OD
Ellendale, MN, USA, 2011 (92Y30)	3	0.15	76	202	R7 R7 R8	4–5	7	Mature Seed	0.034 0.028	0.031		DP29956 Trial 07 100 OD
Gardner, ND, USA, 2011 (NK Seeds: Variety S02-M9)	3	0.15	65	234	R6 R7 R8	5	6	Mature Seed	0.10 0.10	0.10	0.003(J9Z38)	DP29956 Trial 08 100 OD
Northwood, ND, USA, 2011 (Pioneer 90M80)	3	0.15	53	281	BBCH89 BBCH89 BBCH89	5–6	5	Mature Seed	0.015 0.019	0.017		DP29956 Trial 09 100 OD

Soya beans Location Country, year (variety)	Application						DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	Growth stage	RTI (days)			cyantraniliprole	mean	metabolites	
Marysville, OH, USA, 2011 (93Y70)	3	0.15	75	203	BBCH80 BBCH85 BBCH87	5	7	Mature Seed	< 0.003 < 0.003	< 0.003		DP29956 Trial 10 100 OD
Rochelle, IL, USA, 2011 (Pioneer 93Y70)	3	0.15	51	295	R7 R7–R8 R8	4–5	7	Mature Seed	0.021 0.023	0.022		DP29956 Trial 11 100 OD
Richland, IA, USA, 2011 (93Y70)	3	0.15	70	215	R7 12 PHI 7 PHI	5	7	Mature Seed	0.088 0.077	0.083		DP29956 Trial 12 100 OD
			70	215		5	7	Seed (from field site for AGF grain dust)	0.084 0.079	0.081	0.006(J9Z38)	
			70	215		5	7	Seed (from processor for AGF grain dust)	0.069 0.073	0.071		
			70	215		5	7	AGF (grain dust)	46 47	46	0.12(J9Z38) 0.13(MYX98) 0.021(JCZ38) 0.028(N7B69)	
Tipton, MO, USA, 2011 (93Y70)	3	0.15	50	291	R7 R7 R8	4–6	8	Mature Seed	0.049 0.038	0.044		DP29956 Trial 13 100 OD
Fisk, MO, USA, 2011 (95M50)	3	0.15	79	188	BBCH87 BBCH88 BBCH89	5	6	Mature Seed	0.031 0.034	0.033		DP29956 Trial 14 100 OD
Gardner, KS, USA, 2011 (93Y70)	3	0.15	69	218	BBCH79 BBCH80 BBCH86	5	7	Mature Seed	0.15 0.16	0.16		DP29956 Trial 15 100 OD
Staffford, KS, USA, 2011 (Pioneer 93Y70)	3	0.15	72	212	BBCH79 BBCH81 BBCH82	5	8	Mature Seed	0.11 0.15	0.13	0.008(J9Z38)	DP29956 Trial 16 100 OD
York, NE, USA, 2011 (93Y12)	3	0.15	78	192	R7 R8 R8	4–5	8	Mature Seed	0.021 0.024	0.023		DP29956 Trial 17 100 OD
Springfield, NE, USA, 2011 (93Y70)	3	0.15	79	193	BBCH79–81 BBCH81 BBCH86	4–5	7	Mature Seed	0.13 0.12	0.12		DP29956 Trial 18 100 OD
Enid, OK, USA, 2011 (554-T5)	3	0.15	72	216	BBCH93 BBCH95 BBCH97	4–6	8	Mature Seed	0.14 0.15	0.15		DP29956 Trial 19 100 OD
Saginaw, MI, USA, 2012 (R54219R)	3	148	71	209	BBCH80 BBCH85 BBCH87	5–7	7	Mature Seed	0.065 0.056	0.061		DP29956 Trial 20 100 OD

Soya beans Location Country, year (variety)	Application						DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	Growth stage	RTI (days)			cyantraniliprole	mean	metabolites	
Hedrick, IA, USA, 2011 (93Y70)	3	151	67	224	BBCH85 BBCH85 BBCH85	5	7	Mature Seed	0.059 0.053	0.056		DP29956 Trial 21 100 OD

Table 24 Residues in soya beans from supervised trials in the USA following seed treatment of cyantraniliprole, 625 FS formulation

Soya beans Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Frenchtown, NJ USA, 2011 (Pioneer 93Y70)	1	0.388				161	Mature Seed	< 0.003 < 0.003	< 0.003		DP29956 Trial 01 625 FS
	1	0.777				161	Mature Seed	< 0.003,< 0.003	< 0.003		
Athens, GA, USA, 2011 (Pioneer 95Y20)	1	0.386				131	Mature Seed	0.006	0.006	0.006(J9Z38) 0.006(MLA84) 0.006(JCZ38) 0.006(N7B69)	DP29956 Trial 02 625 FS
Blackville, SC, USA, 2011 (Pioneer 95Y20)	1	0.386				136	Mature Seed	< 0.003 < 0.003	< 0.003		DP29956 Trial 03 625 FS
	1	0.773				136	Mature Seed	< 0.003 < 0.003	< 0.003		
Cheneyville, LA, USA, 2011 (Pioneer 95Y20)	1	0.291–0.364				120	Mature Seed	< 0.003 < 0.003	< 0.003		DP29956 Trial 04 625 FS
	1	0.583–0.729				120	Mature Seed	< 0.003 < 0.003	< 0.003		
Fisk, MO, USA, 2011 (95Y20)	1	0.386				131	Mature Seed	< 0.003 < 0.003	< 0.003		DP29956 Trial 05 625 FS
	1	0.773				131	Mature Seed	< 0.003 < 0.003	< 0.003		
Pollard, AR, USA, 2011 (Pioneer 95Y20)	1	0.386				117	Mature Seed	< 0.003 < 0.003	< 0.003		DP29956 Trial 06 625 FS
	1	0.773				117	Mature Seed	< 0.003 < 0.003	< 0.003		
Ellendale, MN, USA, 2011 (92Y30)	1	0.383				139	Mature Seed	< 0.003 < 0.003	< 0.003		DP29956 Trial 07 625 FS
	1	0.786				139	Mature Seed	< 0.003 < 0.003	< 0.003		
Gardner, ND, USA, 2011 (Pioneer 90M80)	1	0.403				123	Mature Seed	< 0.003, < 0.003	< 0.003		DP29956 Trial 08 625 FS
	1	0.806				123	Mature Seed	< 0.003, < 0.003	< 0.003		
Northwood, ND, USA, 2011 (Pioneer 90M80)	1	0.387				149	Mature Seed	< 0.003, < 0.003	< 0.003		DP29956 Trial 09 625 FS
	1	0.774				149	Mature Seed	< 0.003, < 0.003	< 0.003		
Marysville, OH, USA, 2011 (93Y70)	1	0.384				105	Mature Seed	< 0.003, < 0.003	< 0.003		DP29956 Trial 10 625 FS
	1	0.768				105	Mature	< 0.003,	< 0.003		

Soya beans Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
							Seed	< 0.003			
Rochelle, IL, USA, 2011 (Pioneer 93Y70)	1	0.386				164	Mature Seed	< 0.003, < 0.003	< 0.003		DP29956 Trial 11 625 FS
	1	0.773				164	Mature Seed	< 0.003, < 0.003	< 0.003		
Richland, IA, USA, 2011 (93Y70)	1	0.475				136	Mature Seed	0.007 < 0.003	0.004		DP29956 Trial 12 625 FS
Richland, IA, USA, 2011 (93Y70)	1	0.949				136	Mature Seed	< 0.003 < 0.003	< 0.003		DP29956 Trial 12 625 FS
Tipton, MO, USA, 2011 (93Y70)	1	0.409				132	Mature Seed	< 0.003, < 0.003	< 0.003		DP29956 Trial 13 625 FS
	1	0.817				132	Mature Seed	0.003, 0.004	0.004		
Fisk, MO, USA, 2011 (95M50)	1	0.386				117	Mature Seed	0.006, < 0.003	0.004		DP29956 Trial 14 625 FS
	1	0.773				117	Mature Seed	< 0.003, < 0.003	< 0.003		
Gardner, KS, USA, 2011 (93Y70)	1	0.386				128	Mature Seed	< 0.003, < 0.003	< 0.003		DP29956 Trial 15 625 FS
	1	0.771				128	Mature Seed	< 0.003, < 0.003	< 0.003		
Staffford, KS, USA, 2011 (Pioneer 93Y70)	1	0.386				124	Mature Seed	< 0.003, < 0.003	< 0.003		DP29956 Trial 16 625 FS
	1	0.773				124	Mature Seed	< 0.003, < 0.003	< 0.003		
York, NE, USA, 2011 (93Y12)	1	0.372				133	Mature Seed	< 0.003, < 0.003	< 0.003		DP29956 Trial 17 625 FS
	1	0.743				133	Mature Seed	< 0.003, < 0.003	< 0.003		
Springfield, NE, USA, 2011 (93Y70)	1	0.392				147	Mature Seed	< 0.003, < 0.003	< 0.003		DP29956 Trial 18 625 FS
	1	0.783				147	Mature Seed	< 0.003, < 0.003	< 0.003		
Enid, OK, USA, 2011 (93Y20)	1	0.378				167	Mature Seed	< 0.003, < 0.003	< 0.003		DP29956 Trial 19 625 FS
	1	0.755				167	Mature Seed	< 0.003, < 0.003	< 0.003		
Hedrick, IA, USA, 2011 (93Y70)	1	0.387				120	Mature Seed	< 0.003, < 0.003	< 0.003		DP29956 Trial 21 625 FS
Hedrick, IA, USA, 2011 (93Y70)	1	0.773				120	Mature Seed	< 0.003, < 0.003	< 0.003		DP29956 Trial 21 625 FS

Stalk and stem vegetables

Artichoke

In trials conducted on artichokes in Europe, two foliar applications of 0.05 kg ai/ha cyantraniliprole (OD formulation) were applied at 10–13 day intervals, using 800–1000 L/ha with no adjuvant.

Samples of artichoke were stored at –20 °C for up to 12 months before extraction and analysis for cyantraniliprole and six metabolites using method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 97–108% (cyantraniliprole) and 83–106% (metabolites) in samples spiked with 0.01, 0.1 and 0.2 mg/kg.

Table 25 Residues in artichokes (stem vegetables) from supervised trials in Southern Europe following two foliar applications of cyantraniliprole, 100 OD formulation

Artichokes Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Kato Souli, Central Greece Greece,2011 (Wild artichoke)	2	0.05	5	1000	11	7	Mature flower heads	0.033			DP29224 Test 01
Bussana, Liguria Italy,2011 (Spinosa)	2	0.05	5	1000	10	–0 0 1 3 7	Mature flower heads	0.05 0.14 0.086 0.076 0.038			DP29224 Test 02
Ventas de Zafarraya, Andalucia, South Spain,2011 (Blanca de Tudela)	2	0.05	5	1000	10	–0 0 1 3 7	Mature flower heads	0.009 0.11 0.044 0.041 0.019			DP29224 Test 03
Bastia D'Albenga, Liguria, Italy, 2012 (Spinoso)	2	0.05	5	1000	13	–0 0 1 3 7	Mature flower heads	0.004 0.092 0.054 0.046 0.050			DP29224 Test 05
Aguadulce, Andalucía, South Spain 2012 (Blanca de Tudela)	2	0.05	5.01	1000	10	7	Mature flower heads	0.016			DP29224 Test 06

Cereals

Maize

In twenty-three trials conducted on field or pop maize in the USA, seed treatment of 0.5 mg ai/seed of cyantraniliprole (FS formulation) or seed treatment of 0.5 mg ai/seed plus two foliar applications of 0.15 kg ai/ha of cyantraniliprole(WG formulation) were applied, with adjuvants added in foliar applications.

Samples were stored at –20 °C up to 16 month until analysed for cyantraniliprole and the metabolite using analysis method DP-15736. The reported LOQs for cyantraniliprole were 0.01 mg/kg. Average concurrent recoveries were 79–94% (cyantraniliprole) and 73–97% (metabolites) in samples spiked with 0.01, 0.1, 1.0, 5, 14–80 mg/kg.

Table 26 Residues in field maize from supervised trials in the USA following seed treatment of cyantraniliprole, FS formulation

Field Maize Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	mg ai seed	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
North Rose,NY USA,2011 (101 RM)	1	0.04	0.5				Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 01
Seven Springs, NC USA,2011 (114 RM)	1	0.05	0.5				Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 02
Wyoming, IL USA,2011 (109 RM))	1	0.048	0.5				Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 03
Carlyle, IL USA,2011 (109 RM)	1	0.04	0.5				Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test04
Fitchburg, WI USA,2011 (94 RM)	1	0.041	0.5				Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test05
Rice, MN USA,2011 (94 RM)	1	0.043	0.5				Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Tesst 06
Stafford, KS USA,2011 (105 RM)	1	0.04	0.5				Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 07
Campbell, MN USA,2011 (94 RM)	1	0.043	0.5				Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test08
TestTK0029740-09 Seymour, IL USA,2011 (114 RM)	1	0.041	0.5				Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 09
Perley, MN USA,2011 (85 RM)	1	0.037	0.5				Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 10
Geneva, MN USA,2011 (94 RM)	1	0.04	0.5				Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 11
Northwood, KS USA,2011 (85 RM)	1	0.037	0.5				Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test12
TStafford, KS USA,2011 (109 RM5)	1	0.039	0.5				Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 13
McVille, ND USA,2011 (85 RM)	1	0.037	0.5				Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test14
Jefferson, IA USA,2011 (114 RM)	1	0.043	0.5				Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 15
York,NE USA,2011 (114 RM)	1	0.04	0.5				Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test16
Fitchburg, WI USA,2011 (94 RM)	1	0.039	0.5				Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 17

Field Maize Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	mg ai seed	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Richland, IA USA,2011 (109 RM)	1	0.044	0.5				Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 18
Bagley, IA /USA,2011 (114 RM)	1	0.039	0.5				Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 19
Wall, TX USA,2011 (114 RM)	1	0.041					Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test20
York, NE, USA,2011 (Hybrid A3035)	1	0.045					Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 21
Geneva, MN USA,2011 (Hybrid A3035)	1	0.048					Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test22
Gardner, ND USA,2011 (Hybrid A3035)	1	0.052					Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 23

Table 27 Residues in field maize from supervised trials in the USA following one seed treatment, FS formulation, plus two foliar applications of cyantraniliprole, WG formulation

Field Maize Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	mg ai seed	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
North Rose, NY, USA, 2011, (101 RM)	1 + 2	0.04 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 01
Seven Springs, NC, USA,2011 (114 RM)	1 + 2	0.05 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 02
Wyoming, IL USA,2011 (109 RM))	1 + 2	0.048 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 03
Carlyle, IL USA,2011 (109 RM)	1 + 2	0.04 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test04
Fitchburg, WI USA,2011 (94 RM)	1 + 2	0.041 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test05
Rice, MN USA,2011 (94 RM)	1 + 2	0.043 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Tesst 06
Stafford, KS USA,2011 (105 RM)	1 + 2	0.04 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 07
Campbell, MN USA,2011 (94 RM)	1 + 2	0.043 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test08
Seymour, IL USA,2011 (114 RM)	1 + 2	0.041 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 09

Field Maize Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	mg ai seed	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Perley, MN USA,2011 (85 RM)	1 + 2	0.037 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 10
Geneva, MN USA,2011 (94 RM)	1 + 2	0.04 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TestTK0029740-REG Test 11
Northwood, KS USA,2011 (85 RM)	1 + 2	0.037 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test12
Stafford, KS USA,2011 (109 RM5)	1 + 2	0.039 + 0.15	0.5	2–200		14	Grain	0.02, 0.02	0.02		TK0029740-REG Test 13
McVille, ND USA,2011 (85 RM)	1 + 2	0.037 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test14
Jefferson, IA USA,2011 (114 RM)	1 + 2	0.043 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 15
York,NE USA,2011 (114 RM)	1 + 2	0.04 + 0.15	0.5	2–200		0 7 14 21 28	Grain	< 0.010, < 0.010 < 0.010, < 0.010, < 0.010 < 0.010, < 0.010	< 0.010 < 0.010 < 0.010 < 0.010 < 0.010		TK0029740-REG Test16
Fitchburg, WI USA,2011 (94 RM)	1 + 2	0.039 + 0.15	0.5	2–200		0 7 14 21 28	Grain	< 0.010 < 0.010 < 0.010, < 0.010 < 0.010, < 0.010	< 0.010 < 0.010 < 0.010 < 0.010 < 0.010		TK0029740-REG Test 17
Richland, IA USA,2011 (109 RM)	1 + 2	0.044 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 18
Bagley, IA /USA,2011 (114 RM)	1 + 2	0.039 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 19
Wall, TX USA,2011 (114 RM)	1 + 2	0.041 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test20
York, NE, USA, 2011 (Hybrid A3035)	1 + 2	0.045 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 21
Geneva, MN USA,2011 (Hybrid A3035)	1 + 2	0.048 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test22
Gardner, ND USA,2011 (Hybrid A3035)	1 + 2	0.052 + 0.15	0.5	2–200		14	Grain	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 23

Tree nuts

Almond

Table 28 Residues in almonds from supervised trials in the USA following three foliar applications of cyantraniliprole, 100 SE formulation

ALMOND Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Dinuba, CA, USA, 2011) (Non Pareil)	1 + 1 + 1 +	0.15	284 291 290	52	7	5	nutmeat	0.008, 0.08	0.008		DP-32057 Trial 01
Terra Bella, CA, USA, 2011 (Carmell)	1 + 1 + 1 +	0.15	32 32 34	474 477 439	7	5	nutmeat	0.011, 0.009	0.01		DP-32057 Trial 02
Strathmore, CA, USA, 2011 (Fritz)	1 + 1 + 1	0.15	34 35 34	444 443 440	7	4	nutmeat	0.004, 0.006	0.005		DP-32057 Trial 03
Sanger, CA, USA, 2011 (Non-Pareil)	1 + 1 + 1 +	0.15	32 34 32	478 446 473	7	5	nutmeat	0.013, 0.014	0.014		DP-32057 Trial 04

Table 29 Residues in almonds from supervised trials in the USA following foliar applications of cyantraniliprole, 100 OD or SE formulations,) data previously reviewed by the 2013 JMPR

ALMOND Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Turlock, CA USA, 2009 (Butte)	3	0.15	26	580	7, 6	5	nutmeat	0.012, 0.012	0.012		DP-27446 Trial 01
Kerman, CA USA, 2009 (Non-Pareil)	3	0.15	32	470	7	5	nutmeat	0.009, 0.01	0.009		DP-27446 Trial 02
Sanger, CA USA, 2009 (Neplus)	1 + 1 + 1	0.15	23 27	650 600 540	6 7	5	nutmeat	0.006, 0.007	0.007		DP-27446 Trial 03
Sutter, CA USA, 2009 (Non-Pareil)	3	0.15	310	50	7	5	nutmeat	0.024, 0.023	0.023		DP-27446 Trial 04
Sanger, CA USA, 2009 (Neplus)	1 + 2	0.15 0.15	6 12	2400 1300	7 8	5	nutmeat	0.005, 0.005	0.005		DP-27446 Trial 05
	1 + 2	0.15 0.15	6 12	2400 1300	7 8	5	nutmeat	0.008, 0.006	0.007		DP-27446 Trial 05 [100 SE]
Madera, CA USA, 2009 (Non-Pareil)	3	0.15	330	50	6, 7	5	nutmeat	0.006, 0.007	0.007		DP-27446 Trial 06
	3	0.15	11	1400	6, 7	5	nutmeat	0.016, 0.019	0.018		DP-27446 Trial 06 [100 SE]

M1: Average residues of metabolite IN-J9Z38

M2: Average residues of metabolite IN-MYX98

Pecan

Table 30 Residues in pecans from supervised trials in the USA following three foliar applications of cyantraniliprole, 100 SE formulation

Pecan Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Hawkinsville, GA, USA, 2011 (Desirable)	1 + 1 + 1 +	0.15	14	1065 1067 1087	7	5	nutmeat	0.004, 0.007	0.006		DP-32057 Trial 05
Girard, GA, USA, 2011 (Desirable)	1 + 1 + 1 +	0.15	15	998 1013 1022	 8 6	4	nutmeat	0.006, 0.006	0.006		DP-32057 Trial 06
Ocilla, GA, USA, 2011 (Sumner)	1 + 1 + 1 +	0.15	14 15 14	1044 1030 1036	 7 6	6	nutmeat	0.005, 0.009	0.007		DP-32057 Trial 07
Alexandria, LA, USA, 2011 (Creek)	1 + 1 + 1 +	0.15	292 308 306	51 48 49	7	4	nutmeat	0.005, 0.005	0.005		DP-32057 Trial 08
Pearsall, TX, USA, 2011 (Cheyenne)	1 + 1 + 1 +	0.15	26 24 23	593 617 644	7	5	nutmeat	< 0.003, 0.004	< 0.01		DP-32057 Trial 09
San Angelo, TX, USA, 2011 (Indian)	1 + 1 + 1 +	0.15	384 378 382	39	7	5	nutmeat	0.006, 0.008	0.007		DP-32057 Trial 10

Table 31 Residues in pecans from supervised trials in the USA following foliar three applications of cyantraniliprole, 100 OD or SE formulations, or soil (shank) injection, SC formulation, (data previously reviewed by the 2013 JMPR)

PECAN Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Girard, GA USA, 2009 (Desirables)	3	0.15	12	1200	7	5	nutmeat	< 0.003, < 0.003	< 0.003		DP-27446 Trial 07
Union Springs, AL USA, 2009 (Stewart)	3	0.15	12	1200	7	5	nutmeat	< 0.003, < 0.003	< 0.003		DP-27446 Trial 08
Bailey, NC USA, 2009 (Stuart)	3	0.15	12	1200	7	4	nutmeat	< 0.003, < 0.003	< 0.003		DP-27446 Trial 09
Alexandria, LA USA, 2009 (Creek)	1 + 1 + 1	0.16 0.16 0.15	24 21 23	660 780 630	7	5	nutmeat	0.008, 0.010	0.009		DP-27446 Trial 10
Eagle Lake, TX USA, 2009 (Pawnee)	3	0.15	360	40	7, 8	5	nutmeat	0.006, 0.004	0.005		DP-27446 Trial 11

PECAN Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Pearsall, TX USA, 2009 (Wichita	3	0.15 0.14 0.15	27 24 29	570 590 530	7	5	nutmeat	< 0.003, < 0.003	< 0.003		DP-27446 Trial 12
Pearsall, TX USA, 2009 (Wichita)	1	0.46	490	90		57	nutmeat	< 0.003, < 0.003	< 0.003		DP-27446 Trial 12 [200 SC soil injection]

Oilseeds

Cotton

Table 32 Residues in cotton from supervised trials in the USA following three foliar applications of cyantraniliprole, 100 OD formulation, (data previously reviewed by the 2013 JMPR)

COTTON SEED Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Seven Springs, NC USA, 2009 (ST 4554B2RF)	3	0.15	63	240	7	8	seed	0.011, 0.013	0.012		DP-27565 Trial 01
Cheneyville, LA USA, 2009 (Phytogen 485WRF)	3	0.15	75	200	7, 6	–0 0 5 7	seed	0.28, 0.2 0.63, 0.58 0.2, 0.14 0.17, 0.2	0.24 0.6 0.17 0.18	M1 = 0.02 M1 = 0.02 M1 = 0.02 M1 = 0.03	DP-27565 Trial 02
Fisk, MO USA, 2009 (DP 164 B2RF)	3	0.15	80	190	8	8	seed	0.023, 0.027	0.025		DP-27565 Trial 03
Newport, AR USA, 2009 (DP 164 B2RF)	3	0.15	80	190	7	7	seed	0.045, 0.025	0.035		DP-27565 Trial 04
East Bernard, TX USA, 2009 (DP0924 B2F)	3	0.15	75	200	8, 6	–0 0 1 5 7	seed	0.27, 0.33 0.94, 0.66 0.63, 0.89 0.56, 0.82 0.26, 0.26	0.3 0.8 0.76 0.69 0.26	M1 = 0.05 M1 = 0.05 M1 = 0.05 M1 = 0.07 M1 = 0.06	DP-27565 Trial 05
Larned, KS USA, 2009 (Delta Pine)	3	0.15	71	210	7	8	seed	0.27, 0.32	0.29	M1 = 0.01	DP-27565 Trial 06
Larned, KS USA, 2009 (Delta Pine)	1 + 1 + 1	0.19 0.1 0.15	109 48 72	180 210 210	146 7	8	seed	0.16, 0.14	0.15		DP-27565 Trial 06 [soil inject+ 2 foliar]
Hinton, OK USA, 2009 (FM1740B2F)	3	0.15	75	200	8, 9	9	seed gin trash	0.18, 0.13 2.6, 2.6	0.16 2.6	 M1 = 0.03 M2 = 0.01	DP-27565 Trial 07
Edmonson, TX USA, 2009 (DP 924)	3	0.15	97	160	8, 6	7	seed gin trash	0.83, 1.2 4.3, 5.7	0.99 5	 M1 = 0.02 M2 = 0.03	DP-27565 Trial 08

COTTON SEED Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Levelland, TX USA, 2009 (9063 B2F)	3	0.15	63	234	7	8	seed gin trash	0.11, 0.12 3.5, 3.5	0.12 3.5	 M1 = 0.07 M2 = 0.02	DP-27565 Trial 09
Uvalde, TX USA, 2009 (DP6167 B2RF)	3	0.15	65	234	7	6	seed gin trash	0.1, 0.14 2.8, 2.6	0.12 2.7	 M1 = 0.07 M2 = 0.01	DP-27565 Trial 10
Hickman, CA USA, 2009 (Pima)	3	0.15	40	374	7	8	seed	0.2, 0.2	0.2		DP-27565 Trial 11
Madera, CA USA, 2009 (Acala Riata RR)	3	0.15	64	234	7, 6	7	seed	0.15, 0.12	0.14		DP-27565 Trial 12
Sanger, CA USA, 2009 (PHY 725 RF Acala)	3	0.15	54 37	290 400	 6 8	7	seed	0.24, 0.21	0.22		DP-27565 Trial 13

M1: Average residues of metabolite IN-J9Z38

M2: Average residues of metabolite IN-MYX98

Rape seed (canola)

Table 33 Residues in oil-seed rape from supervised trials in the USA following foliar applications of cyantraniliprole, 100 OD formulation, with and without the use of cyantraniliprole-treated seed (data previously reviewed by the 2013 JMPR)

OILSEED RAPE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Stephens, GA USA, 2009 (Sumner)	1 + 1 + 1	0.15 0.15 0.14	51 62 70	290 240 210	7	7	seed	0.017, 0.022	0.019		DP27582 Test 01
Geneva, MN USA, 2009 (Pioneer 45H21)	3	0.15	79	190	6, 7	8	seed	0.027, 0.016	0.021		DP27582 Test 02
St. Marcsur Richelieu, QC CAN, 2009 (Pioneer D3150)	3	0.15	50	300	6, 9	1	seed	0.17, 0.16	0.16		DP27582 Trial 03
St. Marcsur Richelieu, QC CAN, 2009 (Pioneer D3150)	1 + 1 + 2	0.08 + 0.07 0.15	 24 50	 310 300	 6 9	1	seed	0.11, 0.13	0.12		DP27582 Trial 03 [with treated seed]
Carrington, ND USA, 2009 (Pioneer D3151)	3	0.15	54	280	7	7	seed	0.017, 0.017	0.017		DP27582 Test 04

OILSEED RAPE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Carrington, ND USA, 2009 (Pioneer D3151)	1 + 1 + 2	0.08 + 0.07 0.15	 25 54	 280 280	 7 7	7	seed	0.015, 0.016	0.015		DP27582 Test 04 [with treated seed]
Ephrata, WA USA, 2009 (7145 RR)	3	0.15	73	210	7	7	seed	0.087, 0.08	0.084	M1 = 0.01	DP27582 Test 05
Jerome, ID USA, 2009 (D3151)	2 + 1	0.15	76 80	200 190	6 8	7	seed	0.29, 0.34	0.32		DP27582 Test 06
Jerome, ID USA, 2009 (D3151)	1 + 1 + 1 + 1	0.08 + 0.07 0.15 0.16	37 74 80	200 200 190	6 8	7	seed	0.21, 0.22	0.21		DP27582 Test 06 [with treated seed]
Carberry, MB CAN, 2009 (D3151)	3	0.15	60	250	7, 6	6	seed	0.054, 0.065	0.059		DP27582 Test 07
Carberry, MB CAN, 2009 (D3151)	1 + 1 + 2	0.08 + 0.07 0.15	 29 60	 250 250	 7 6	6	seed	0.029, 0.032	0.031		DP27582 Test 07 [with treated seed]
Justice, MB CAN, 2009 (D3151)	3	0.15	60	250	7, 6	6	seed	0.022, 0.023	0.022		DP27582 Test 08
Justice, MB CAN, 2009 (D3151)	1 + 1 + 2	0.08 + 0.07 0.15	 29 60	 250 250	 7 6	6	seed	0.048, 0.047	0.047		DP27582 Test 08 [with treated seed]
Brandon, MB CAN, 2009 (Invigor 5030)	3	0.15	60	250	7	7	seed	0.18, 0.16	0.17		DP27582 Test 09
Alvena, SK CAN, 2009 (RR 7145)	3	0.15	75	200		7	seed	0.24, 0.3	0.27	M1 = 0.02	DP27582 Test 10
Ft. Saskatchewan, AB CAN, 2009 (Liberty 1141)	3	0.15	50	300	7, 6	7	seed	0.057, 0.066	0.061		DP27582 Trial 11
Ft. Saskatchewan, AB CAN, 2009 (1818 Roundup Ready)	3	0.15	50	300	7	7	seed	0.13, 0.12	0.12		DP27582 Trial 12
Lamont, AB CAN, 2009 (Invigor 8440)	3	0.15	50	300	6, 7	7	seed	0.14, 0.21	0.18		DP27582 Trial 13
Westlock, AB CAN, 2009 (Roundup Ready 1818)	3	0.15	50	300	7, 6	7	seed	0.07, 0.07	0.07	M1 = 0.01	DP27582 Trial 14
Waldheim, SK CAN, 2009 (Dekalb 7145 RR)	3	0.15	75	200	7	7	seed	0.57, 0.65	0.61	M1 = 0.02	DP27582 Trial 15

OILSEED RAPE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Blaine Lake, SK CAN, 2009 (Dekalb 7145 RR)		0.15	74	200	7	7	seed	0.25, 0.33	0.29	M1 = 0.01	DP27582 Trial 16
Wakaw, SK CAN, 2009 (RR 7145)	3	0.15	75	200	7	7	seed	0.066, 0.047	0.057		DP27582 Trial 17

M1: Average residues of metabolite INJ9Z38

Sunflower

Table 34 Residues in sunflower seed from supervised trials in the USA following foliar applications of cyantraniliprole, 100 OD formulation, (data previously reviewed by the 2013 JMPR)

SUNFLOWER Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Stafford, KS USA, 2009 (Sunflower/ Pioneer 63M61)	2 + 1	0.15 0.16	7175	210	7	7	seed	0.045, 0.082	0.064		DP27582 Trial 18
Atlantic, IA USA, 2009 (Sunflower/ 8007 Millborn)	3	0.15	80	190	7	7	seed	0.069, 0.065	0.067		DP27582 Trial 19
Carrington, ND USA, 2009 (Sunflower/ Pioneer)	3	0.15	54	280	5, 7	7	seed	0.068, 0.1	0.085		DP27582 Trial 20
Velda, ND USA, 2009 (Sunflower/ 8N835CL)	3	0.15	80	190	8, 7	7	seed	0.14, 0.15	0.14		DP27582@Trial 21
Jamestown, ND USA, 2009 (Sunflower/ IS 8048)	3	0.15	80	190	7	7	seed	0.03, 0.049	0.039		DP27582 Trial 22
Montpelier, ND USA, 2009 (Sunflower/ IS 8048)	1 + 2	0.16 0.15	8380	190 190	7	7	seed	0.026, 0.031	0.028		DP27582 Trial 23
Hinton, OK USA, 2009 (Sunflower/ 8N453DM)	3	0.15	63	240	8, 9	5	seed	0.06, 0.059	0.059		DP27582 Trial 24
Brookdale, MB CAN, 2009 (Sunflower/ 6946)	3	0.15	60	250	7, 6	6	seed	0.36, 0.28	0.32		DP27582 Trial 25
Neepawa, MB CAN, 2009 (Sunflower/ Jaguar)	3	0.15	60	250	7, 6	6	seed	0.092, 0.093	0.092		DP27582 Trial 26

Seeds for beverage and sweets

Coffee

Table 35 Residues in coffee beans from supervised trials in Brazil following soil drench, SC formulation, and foliar applications of cyantraniliprole, OD formulation,) (data previously reviewed by the 2013 JMPR)

COFFEE Country, year Location (variety)	Application				RTI (days)	DAT, (days)	Portion analysed	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	g ai/hL	water (L/ha)				cyantraniliprole	metabolites	
Campinas SP Brazil, 2011	2 + 2	0.2 (soil) 0.175	0.5 35	0.1 L/plant 500	30	7 14 28 35 45 60	beans	0.02 0.01 < 0.01 < 0.01 < 0.01 < 0.003		BRI- 10/11-008 Test A
Campinas SP Brazil, 2011	2	0.175	35	500		7 28	beans	< 0.01 < 0.01		BRI- 10/11-008 Test A
Espirito Santo do Pinhal SP Brazil, 2011	2 + 2	0.2 (soil) 0.175	0.6 35	0.1 L/plant 500	30	7 14 28 35	beans	0.01 < 0.01 < 0.01 < 0.01		BRI- 10/11-008 Test B
Cabo Verde Brazil, 2011	2 + 2	0.2 (soil) 0.175	0.5 35	0.1 L/plant 500	30	7 28	beans	0.03 0.01		BRI- 10/11-008 Test C
Pardinho – SP Brazil, 2011	2 + 2	0.2 (soil) 0.175	0.6 30	0.1 L/plant 580	30	7 28	beans	0.02 < 0.01		BRI- 10/11-008 Test D
Restinga – SP Brazil, 2011	2 + 2	0.2 (soil) 0.175	0.2 29	0.1 L/plant 600	30	7 14 28 35	beans	< 0.01 < 0.01 < 0.01 < 0.01		BRI- 10/11-008 Test E
Monte Santo de Minas Brazil, 2011	2 + 2	0.2 (soil) 0.175	0.4 29	0.1 L/plant 600	30	7 28	beans	0.01 0.01		BRI- 10/11-008 Test F
Monte Santo de Minas Brazil, 2011	2	0.175	29	600		7 28	beans	0.02 0.02		BRI- 10/11-008 Test F
Indianopolis Brazil, 2011	2 + 2	0.2 (soil) 0.175	0.7 29	0.1 L/plant 600	30	7 28 45 60	beans	< 0.01 < 0.01 < 0.01 < 0.01		BRI- 10/11-008 Test G
Lohdrina Brazil 2011	2 + 2	0.2 (soil) 0.175	0.5 44	0.1 L/plant 400	30	7 14 28 35	beans	< 0.01 < 0.01 < 0.003 < 0.003		BRI- 10/11-008 Test I

Tea, green

In field trials conducted in Japan and reported by Higuchil, 2013 [Ref: DP-37521], one foliar application of 0.20 kg ai/ha cyantraniliprole (OD formulation) was applied with no adjuvant added.

Samples of raw tea leaves were processed within one day, the processed tea samples were stored at –20 °C up to 2 months until analysis for cyantraniliprole and the metabolite using method DP-15736, with reported LOQs of 0.04 mg/kg. Average concurrent recoveries were 73–88% cyantraniliprole and 82–88% IN-J9Z38 in samples spiked with 0.04, 2.0 and 25 mg/kg.

Table 36 Residues in tea from supervised trials in Japan following a single foliar application of cyantraniliprole, OD formulation

Tea, green Country, year Location (variety)	Application				RTI (days)	DAT, (days)	Commodity or Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water (L/ha)				cyantraniliprole	mean	metabolites	
Kochi, Japan 2010 (Yabukita)	1	0.2	5	4000		7 14 21	Processed leaves	20.4, 20.7 1.07, 1.05 < 0.04, < 0.04	20.6 1.06 < 0.04	0.73(J9Z38) 0.21 (J9Z38) 0.07(NXX70)	DP-37521 Test 1
Miyazaki, Japan 2010 (Fuushun)	1	0.2	5	4000		7 14 21	Processed leaves	4.19, 4.18 1.91, 1.81 < 0.04, < 0.04	4.19 1.86 < 0.04	0.74(J9Z38) 0.11(NXX70) 0.47(J9Z38) 0.23(NXX70)	DP-37521 Test 2-

Animal feed

Pea remaining plant and empty pod

Table 37 Residues in field remaining plant and empty pod of peas (fresh) from supervised trials in Europe following two foliar applications of cyantraniliprole, 400 g/kg WG formulation

Peas remaining plant and empty pod Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Market Weighton, East Yorkshire, United Kingdom, 2011 (Fresh)	2	0.075		200–1000	7	0 1 3(NCH) 7 14	Remaining Plant and Empty Pod	2.98 2.76 2.34 2.98 0.14			Syngenta TK0057194 Test 01
Driffield, East Yorkshire, United Kingdom, 2011 (Fresh)	2	0.075			7	0 1 3(NCH) 7 14	Remaining Plant and Empty Pods	2.55 1.93 1.85 1.01 0.96		0.01(J9Z38 0.02 (J9Z38 0.01 (J9Z38 0.02 (J9Z38 0.02 (J9Z38	Syngenta TK0057194 Test 02
Sulniac, Bretagne, N. France, 2011 (Fresh)	2	0.075			7	0 1 3(NCH) 7 14	Remaining Plant and Empty Pods	0.54 0.08 0.14 0.07 0.03		0.02 (J9Z38) 0.03 (J9Z38) 0.04 (J9Z38) 0.04 (J9Z38) 0.01 (MLA84) 0.08 (J9Z38) 0.01 (MLA84)	Syngenta TK0057194 Test 03
Oinville Saint Liphard, Eure et Loire, N. France, 2011 (Fresh)	2	0.075			7	0 1 3(NCH) 7 14	Remaining Plant and Empty Pods	2 2.03 1.96 1.88 0.97		0.02 (J9Z38) 0.03 (J9Z38) 0.04 (J9Z38) 0.04 (J9Z38) 0.01 (MLA84) 0.08 (J9Z38) 0.01 (MLA84)	Syngenta TK0057194 Test 04

Peas remaining plant and empty pod Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Behagnies, 62121, N. France, 2012 (Fresh)	2	0.075			7	1 3(NCH) 6	Remaining Plant and Empty Pod	2.14 1.15 0.98		0.02 (J9Z38) 0.04 (J9Z38) 0.05 (J9Z38)	Syngenta TK0112971 Test 05
Mulfingen, 74673, Germany, 2012 (Fresh)	2	0.075			7	1 1 3(NCH) 7	Remaining Plant and Empty Pods	1.66 1.33 0.72		0.02 (J9Z38) 0.02 (J9Z38) 0.03 (J9Z38)	Syngenta TK0112971 Test 06
Bretzfeld-Schwabbach, 74626, Germany, 2012 (Fresh)	2	0.075			7	1 3(NCH) 7	Remaining Plant and Empty Pods	0.67 0.5 0.09		0.01 (J9Z38) 0.02 (J9Z38)	Syngenta TK0112971 Test 07
Cagnicourt, 62182, N. France, 2012 (Fresh)	2	0.075			7	1 3(NCH) 7	Remaining Plant and Empty Pods	1.75 1.8 0.93		0.03 (J9Z38) 0.03 (J9Z38) 0.06 (J9Z38)	Syngenta TK0112971 Test 08
Houeilles, Lot et Garonne, Aquitaine, S France, 2011 (Fresh)	2	0.075			7	0 1 2(NCH) 7 14	Remaining Plant and Empty Pods	.92 1.43 1.24 1.89 1.15		0.01 (J9Z38) 0.05 (J9Z38)	Syngenta TK0057193 Test 09
Elne, Pyrenees Orientales, Elne, S France, 2011 (Fresh)	2	0.075			7	0 1 2(NCH) 6 14	Remaining Plant and Empty Pods	3.98 3.48 2.69 2.2 2.31		0.01 (J9Z38) 0.01 (J9Z38) 0.01 (J9Z38) 0.04 (J9Z38) 0.09 (J9Z38)	Syngenta TK0057193 Test 10
Granarolo, Emilia Romagna, Bologna, Italy, 2011 (Fresh)	2	0.075			7	0 1 3(NCH) 6 14	Remaining Plant and Empty Pods	0.72 0.15 0.1 0.11 0.08			Syngenta TK0057193 Test 11
Villar de Chinchilla, Albacete, Spain, 2011 (Fresh)	2	0.075			7	0 1 3(NCH) 6 14	Remaining Plant and Empty Pods	3.83 3.2 1.53 2.65 3.15		0.05 (J9Z38) 0.01 (J9Z38) 0.03 (J9Z38) 0.04 (J9Z38)	Syngenta TK0057193 Test 12
Montpouillan, 47200, S France, 2012 (Fresh)	2	0.075			7	1 3(NCH)	Remaining Plant and Empty Pods	0.83 0.89		0.02 (J9Z38) 0.03 (J9Z38)	Syngenta TK0112985 Test 13
Saint Agnet, 40800, S. France, 2012 (Fresh)	2	0.075			7	1 3(NCH) 7	Remaining Plant and Empty Pods	0.45 0.15 0.08			Syngenta TK0112985 Test 14

Peas remaining plant and empty pod Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
La Gineta, 02110, Spain, 2012 (Fresh)	2	0.075			7	1 3(NCH) 7	Remaining Plant and Empty Pods	0.37 0.15 0.17		0.02 (J9Z38) 0.01 (J9Z38)	Syngenta TK0112985 Test 15
Papiano Marsciano, 06055, Italy, 2012 (Fresh)	2	0.075			7	1 3(NCH) 7	Remaining Plant and Empty Pods	1.46 1.41 1.46		0.03 (J9Z38) 0.03 (J9Z38) 0.06 (J9Z38)	Syngenta TK0112985 Test 16

Bean, forage and hay

Table 38 Residues in bean forage and hay (dry shelled bean) from supervised trials in the USA following three foliar applications of cyantraniliprole, 100 SE and 100 OD formulation

Bean forage and hay Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Marysville, OH, USA, 2011 (Espada)	3	0.15	75	200	5	7	Forage	0.96 0.74	0.85	0.051 (J9Z38) 0.013(MLA84) 0.003(MYX98)	DP 31668 Test 23 100 SE
							Forage	1.1 1.6	1.4	0.079 (J9Z38) 0.022(MLA84) 0.005(MYX98) 0.004(JCZ38)	DP 31668 Test 23 100 OD
							Hay	2.1 2.6	2.4	0.20 (J9Z38) 0.053(MLA84) 0.014(MYX98) 0.010(JCZ38) 0.005(N7B69)	DP 31668 Test 23 100 SE
							Hay	2.1 3.4	2.8	0.23 (J9Z38) 0.073(MLA84) 0.013(MYX98) 0.013(JCZ38) 0.005(N7B69)	DP 31668 Test 23 100 OD
Lenexa, KS, USA, 2011 (Pinkeye-Purple Hull) Lenex	3	0.15	70	215	5	6	Forage	3.0 3.0	3.0	0.18 (J9Z38) 0.010(MLA84) 0.010(MYX98) 0.012(JCZ38) 0.005(N7B69)	DP 31668 Test 24 100 SE
					5	6	Forage	2.3 2.2	2.3	0.24 (J9Z38) 0.014(MLA84) 0.008(MYX98) 0.015(JCZ38) 0.004(N7B69)	DP 31668 Test 24 100 OD
					5	6	Hay	9.8 9.7	9.8	0.76 (J9Z38) 0.037(MLA84) 0.042(MYX98) 0.040(JCZ38) 0.015(N7B69)	DP 31668 Test 24 100 SE

Bean forage and hay Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
					5	6	Hay	6.2 6.1	6.2	0.85 (J9Z38) 0.050(MLA84) 0.028(MYX98) 0.038(JCZ38) 0.011(N7B69)	DP 31668 Test 24 100 OD
Stafford, KS, USA, 2011 (Cow Pea)	3	0.15	70	210	5	7	Forage	1.1 1.5	1.3	0.20 (J9Z38) 0.009(MLA84) 0.004(MYX98) 0.010(JCZ38)	DP 31668 Test 25 100 SE
							Forage	0.53 0.51	0.52	0.19 (J9Z38) 0.010(MLA84) 0.009JCZ38)	DP 31668 Test 25 100 OD
							Hay	3.0 2.5	2.8	0.49 (J9Z38) 0.023(MLA84) 0.014(MYX98) 0.025(JCZ38) 0.006(N7B69)	DP 31668 Test 25 100 SE
							Hay	1.4 1.2	1.3	0.43 (J9Z38) 0.021(MLA84) 0.004(MYX98) 0.021(JCZ38) 0.004(N7B69)	DP 31668 Test 25 100 OD
York, NE, USA, 2011 (California Blackeye #5)	3	0.15	80	190	4-5	7	Forage	0.78 0.78	0.78	0.24 (J9Z38) 0.013(MLA84) 0.007(JCZ38)	DP 31668 Test 26 100 SE
					4	7	Forage	0.88 0.39	0.64	0.19 (J9Z38) 0.012(MLA84) 0.005(JCZ38)	DP 31668 Test 26 100 OD
					4-5	7	Hay	2.6 3.6	3.1	0.90 (J9Z38) 0.064(MLA84) 0.013(MYX98) 0.036(JCZ38) 0.011(N7B69)	DP 31668 Test 26 100 SE
					4	7	Hay	3.7 2.4	3.0	0.88 (J9Z38) 0.062(MLA84) 0.011(MYX98) 0.032(JCZ38) 0.010(N7B69)	DP 31668 Test 26 100 OD

Table 39 Residues in pea vine and hay (dry shelled pea) from supervised trials in the USA following three foliar applications of cyantraniliprole, 100 SE and 100 OD formulation

Pea vine and hay Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Payette, ID, USA, 2011 (Austrian Winter)	3	0.15	65	235							
					5	0	Vine	9.1 8.9	9.0	0.15 (J9Z38) 0.045(MLA84) 0.017(MYX98) 0.013(JCZ38) 0.005(N7B69)	DP 31668 Test 27 100 OD

Pea vine and hay Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
					5	1		8.3 9.5	8.9	0.10 (J9Z38) 0.048(MLA84) 0.019(MYX98) 0.014(JCZ38) 0.006(N7B69)	DP 31668 Test 27 100 OD
					5	3		8.3 8.5	8.4	0.14(J9Z38) 0.059(MLA84) 0.022(MYX98) 0.017(JCZ38) 0.006(N7B69)	DP 31668 Test 27 100 OD
					5	5		8.6 8.6	8.6	0.14 (J9Z38) 0.065(MLA84) 0.024(MYX98) 0.019(JCZ38) 0.006(N7B69)	DP 31668 Test 27 100 OD
					5	7		8.2 8.7	8.5	0.12 (J9Z38) 0.060(MLA84) 0.028(MYX98) 0.020(JCZ38) 0.008(N7B69)	DP 31668 Test 27 100 OD
					5	0	Hay	29 34	31	1.7 (J9Z38) 0.22(MLA84) 0.077(MYX98) 0.049(JCZ38) 0.025(N7B69)	DP 31668 Test 27 100 OD
					5	1		28 34	31	1.7 (J9Z38) 0.24(MLA84) 0.090(MYX98) 0.050(JCZ38) 0.027(N7B69)	DP 31668 Test 27 100 OD
					5	3		24 25	24	1.3(J9Z38) 0.23(MLA84) 0.075(MYX98) 0.051(JCZ38) 0.023(N7B69)	DP 31668 Test 27 100 OD
					5	5		27 26	26	1.5 (J9Z38) 0.26(MLA84) 0.090(MYX98) 0.057(JCZ38) 0.027(N7B69)	DP 31668 Test 27 100 OD
					5	7		16 20	18	1.0 (J9Z38) 0.22(MLA84) 0.072(MYX98) 0.050(JCZ38) 0.021(N7B69)	DP 31668 Test 27 100 OD
Jerome, ID, USA, 2011 (Austrian)	3	0.15	75	200							
					4–6	0	Vine	3.5 3.6	3.5	0.060(J9Z38) 0.033(MLA84) 0.004(MYX98) 0.007(JCZ38)	DP 31668 Test 28 100 OD
					4–6	1		4.3 3.6	3.9	0.061(J9Z38) 0.041(MLA84) 0.007(MYX98) 0.010(JCZ38) 0.004(N7B69)	DP 31668 Test 28 100 OD

Pea vine and hay Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
					4–6	4		1.4 1.5	1.4	0.063(J9Z38) 0.042(MLA84) 0.004(MYX98) 0.009(JCZ38)	DP 31668 Test 28 100 OD
					4–6	5		1.3 1.5	1.4	0.086 (J9Z38) 0.051(MLA84) 0.011(JCZ38) 0.003(N7B69)	DP 31668 Test 28 100 OD
					4–6	7		1.2 1.2	1.2	0.065 (J9Z38) 0.048(MLA84) 0.003(MYX98) 0.012(JCZ38)	DP 31668 Test 28 100 OD
					4–6	0	Hay	35 33	34	0.59 (J9Z38) 0.49(MLA84) 0.045(MYX98) 0.076(JCZ38) 0.017(N7B69)	DP 31668 Test 28 100 OD
					4–6	1		32 34	33	0.64 (J9Z38) 0.49(MLA84) 0.070(MYX98) 0.079(JCZ38) 0.021(N7B69)	DP 31668 Test 28 100 OD
					4–6	4		9.2 9.9	9.5	0.74 (J9Z38) 0.43(MLA84) 0.032(MYX98) 0.073(JCZ38) 0.013(N7B69)	DP 31668 Test 28 100 OD
					4–6	5		11 7.3	9.2	0.76 (J9Z38) 0.47 (MLA84) 0.033(MYX98) 0.081(JCZ38) 0.014(N7B69)	DP 31668 Test 28 100 OD
					4–6	7		12 8.4	10	0.91 (J9Z38) 0.43(MLA84) 0.030(MYX98) 0.074(JCZ38) 0.015(N7B69)	DP 31668 Test 28 100 OD
Ephrata, WA, USA, 2011 (Austrian Winter)	3	0.15	53	281	5	7	Vine	0.72 0.65	0.69	0.023(J9Z38) 0.028(MLA84) 0.011(JCZ38)	DP 31668 Test 29 100 OD
							Hay	1.8 1.9	1.9	0.24 (J9Z38) 0.17(MLA84) 0.009(MYX98) 0.039(JCZ38) 0.005(N7B69)	DP 31668 Test 29 100 OD
Jerome, ID, USA, 2011 (Austrian Winter)	3	0.15	75	200	4–5	7	Vine	1.6 1.2	1.4	0.13 (J9Z38) 0.059(MLA84) 0.004(MYX98) 0.012(JCZ38)	DP 31668 Test 30 100 OD
					4–5	7	Hay	4.8 4.5	4.7	0.42 (J9Z38) 0.23(MLA84) 0.020(MYX98) 0.045(JCZ38)	DP 31668 Test 30 100 OD

Soya bean forage and hay

Table 40 Residues in forage and hay of soya beans from supervised trials in the USA following three foliar applications of cyantraniliprole, 100 OD formulation

Soya bean forage and hay Location Country, year (variety)	Application						DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	Growth stage	RTI (days)			cyantraniliprole	mean	metabolites	
Frenchtown, NJ USA, 2011 (Pioneer 93M14))	3	0.15	56	281	V5–V6 V5–R1 R1–R2	5	7	Forage	1.2 1.2	1.2	0.16(J9Z38) 0.17(MLA84) 0.004(MYX98) 0.005(JCZ38)	DP29956 Trial 01 100 OD
			56	281	V5–V6 V5–R1 R1–R2	5	7	Hay	6.3 5.9	6.1	1.0(J9Z38) 0.88(MLA84) 0.017(MYX98) 0.027(JCZ38) 0.010(N7B69)	
Athens, GA, USA, 2011 (Pioneer 95Y20))	3	0.15	48	313	R1 R2 R2	5	7	Forage	2.0 2.1	2.1	0.14(J9Z38) 0.071(MLA84) 0.012(MYX98) 0.006(JCZ38)	DP29956 Trial 02 100 OD
			48	313	R1 R2 R2	5	7	Hay	6.1 6.6	6.3	0.39(J9Z38) 0.21(MLA84) 0.040(MYX98) 0.021(JCZ38) 0.011(N7B69)	
Cheneyville, LA, USA, 2011 (Pioneer 95Y20)	3	0.15	98	152	BBCH61–62 BBCH63–64 BBCH64–65	5–6	7	Forage	3.0 2.7	2.9	0.16(J9Z38) 0.097(MLA84) 0.009MYX98) 0.008(JCZ38)	DP29956 Trial 04 100 OD
			98	152	BBCH61–62 BBCH63–64 BBCH64–65	5	7	Hay	8.3 7.3	7.8	0.87(J9Z38) 0.37(MLA84) 0.036(MYX98) 0.023(JCZ38) 0.009(N7B69)	
Fisk, MO, USA, 2011 (95Y50)	3	0.15	79	188	BBCH61/R1 BBCH67–69 BBCH69	4–5	7	Forage	2.7 2.9	2.8	0.21(J9Z38) 0.082(MLA84) 0.009(MYX98) 0.005(JCZ38)	DP29956 Trial 05 100 OD
			79	188	BBCH61/R1 BBCH67–69 BBCH69	4–5	7	Hay	8.6 10	9.4	0.83(J9Z38) 0.35(MLA84) 0.030(MYX98) 0.021(JCZ38) 0.010(N7B69)	
Pollard, AR, USA, 2011 (Pioneer 95M50)	3	0.15	80	193	V5–R1 R1–R2 R2	4–5	7	Forage	3.1 3.1	3.1	0.29(J9Z38) 0.15(MLA84) 0.010(MYX98) 0.014(JCZ38) 0.004(N7B69)	DP29956 Trial 06 100 OD
			80	193	V5–R1 R1–R2 R2	4–5	7	Hay	13 15	14	1.3(J9Z38) 0.61(MLA84) 0.056(MYX98) 0.056(JCZ38) 0.030(N7B69)	
Ellendale, MN, USA, 2011 (92Y30)	3	0.15	80	193	V5–R1 R1–R2 R2	4–5	7	Forage	3.1 3.1	3.1	0.29(J9Z38) 0.15(MLA84) 0.010(MYX98) 0.014(JCZ38) 0.004(N7B69)	DP29956 Trial 07 100 OD

Soya bean forage and hay Location Country, year (variety)	Application						DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	Growth stage	RTI (days)			cyantraniliprole	mean	metabolites	
			80	193	V5–R1 R1–R2 R2	4–5	7	Hay	13 15	14	1.3(J9Z38) 0.61(MLA84) 0.056(MYX98) 0.056(JCZ38) 0.030(N7B69)	
Gardner, ND, USA, 2011 (NK Seeds: Variety S02-M9)	3	0.15	66	234	BBCH51 BBCH60 BBCH64	4–5	7	Forage	1.9 1.9	1.9	0.13(J9Z38) 0.14(MLA84) 0.007(MYX98) 0.019(JCZ38)	DP29956 Trial 08 100 OD
			66	234	BBCH51 BBCH60 BBCH64	4–5	7	Hay	8.9 8.3	8.6	0.63(J9Z38) 0.50(MLA84) 0.035(MYX98) 0.067(JCZ38) 0.037(N7B69)	
Northwood, ND, USA, 2011 (Pioneer 90M80)	3	0.15	53	281	R3 R3 R3	5–6	6	Forage	4.5 5.0	4.8	0.094(J9Z38) 0.11(MLA84) 0.020(MYX98) 0.026(JCZ38) 0.010(N7B69)	DP29956 Trial 09 100 OD
			53	281	R3 R3 R3	5–6	6	Hay	19 20	20	0.26(J9Z38) 0.31(MLA84) 0.067(MYX98) 0.069(JCZ38) 0.029(N7B69)	
Marysville, OH, USA, 2011 (93Y70)	3	0.15	75	202	BBCH60 BBCH62 BBCH65	5	7	Forage	0.27 0.19	0.23	0.018(J9Z38) 0.031(MLA84)	DP29956 Trial 10 100 OD
			75	202	BBCH60 BBCH62 BBCH65	5	7	Hay	1.1 1.1	1.1	0.077(J9Z38) 0.17(MLA84) 0.003(MYX98) 0.011(JCZ38)	
Rochelle, IL, USA, 2011 (Pioneer 93Y70)	3	0.15	52	288	R1 R1 R2	4–6	7	Forage	0.29 0.36	0.33	0.040(J9Z38) 0.086(MLA84) 0.005(JCZ38)	DP29956 Trial 11 100 OD
			52	288	R1 R1 R2	4–6	67	Hay	1.3 1.1	1.2	0.17(J9Z38) 0.32(MLA84) 0.005(MYX98) 0.017(JCZ38) 0.008(N7B69)	
Richland, IA, USA, 2011 (93Y70)	3	0.15	73	206	BBCH63 BBCH65 BBCH67	5	7	Forage	2.6 3.4	3.0	0.22(J9Z38) 0.10(MLA84) 0.014(MYX98) 0.013(JCZ38) 0.005(N7B69)	DP29956 Trial 12 100 OD
			73	206	BBCH63 BBCH65 BBCH67	5	7	Hay	4.8 7.1	5.9	0.44(J9Z38) 0.22(MLA84) 0.028(MYX98) 0.022(JCZ38) 0.016(N7B69)	
Tipton, MO, USA, 2011 (93Y70)	3	0.15	53	290	R1 R1 R2	4–5	8	Forage	1.4 1.2	1.3	0.069(J9Z38) 0.063(MLA84) 0.005(MYX98) 0.007(JCZ38)	DP29956 Trial 13 100 OD
			53	290	R1 R1 R2	4–5	8	Hay	4.0 3.6	3.8	0.32(J9Z38) 0.22(MLA84) 0.014(MYX98) 0.018(JCZ38) 0.007(N7B69)	

Soya bean forage and hay Location Country, year (variety)	Application						DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	Growth stage	RTI (days)			cyantraniliprole	mean	metabolites	
Fisk, MO, USA, 2011 (95M50)	3	0.15	80	187	BBCH61 BBCH65 BBCH67–69	5–6	8	Forage	10 11	11	0.14(J9Z38) 0.12(MLA84) 0.046(MYX98) 0.019(JCZ38) 0.010(N7B69)	DP29956 Trial 14 100 OD
			80	187	BBCH61 BBCH65 BBCH67–69	5–6	8	Hay	35 25	30	0.37(J9Z38) 0.34(MLA84) 0.14(MYX98) 0.052(JCZ38) 0.030(N7B69)	
Gardner, KS, USA, 2011 (93Y70)	3	0.15	70	217	BBCH61 BBCH62 BBCH64	5	7	Forage	6.2 7.2	6.7	0.047(J9Z38) 0.072(MLA84) 0.038(MYX98) 0.014(JCZ38) 0.007(N7B69)	DP29956 Trial 15 100 OD
			70	217	BBCH61 BBCH62 BBCH64	5	7	Hay	29 27	28	0.54(J9Z38) 0.24(MLA84) 0.14(MYX98) 0.042(JCZ38) 0.033(N7B69)	
Staffford, KS, USA, 2011 (Pioneer 93Y70)	3	0.15	74	209	BBCH60 BBCH64 BBCH65	4–5	6	Forage	4.8 5.9	5.3	0.26(J9Z38) 0.18(MLA84) 0.021(MYX98) 0.025(JCZ38) 0.007(N7B69)	DP29956 Trial 16 100 OD
			74	209	BBCH60 BBCH64 BBCH65	4–5	6	Hay	20 21	20	0.32(J9Z38) 0.57(MLA84) 0.074(MYX98) 0.073(JCZ38) 0.022(N7B69)	
York, NE, USA, 2011 (93Y12)	3	0.15	81	188	BBCH51 R1 R2	5–6	7	Forage	3.3 3.0	3.2	0.33(J9Z38) 0.30(MLA84) 0.009(MYX98) 0.016(JCZ38) 0.003(N7B69)	DP29956 Trial 17 100 OD
			81	188	BBCH51 R1 R2	5–6	7	Hay	14 14	14	1.0(J9Z38) 1.3(MLA84) 0.036(MYX98) 0.060(JCZ38) 0.015(N7B69)	
Springfield, NE, USA, 2011 (93Y70)	3	0.15	76	192	R1 R1 R1	4–5	6	Forage	7.4 5.5	6.4	0.39(J9Z38) 0.26(MLA84) 0.020(MYX98) 0.018(JCZ38) 0.006(N7B69)	DP29956 Trial 18 100 OD
			76	192	R1 R1 R1	4–5	6	Hay	21 20	21	1.4(J9Z38) 0.76(MLA84) 0.058(MYX98) 0.044(JCZ38) 0.026(N7B69)	

Table 41 Residues in soya bean forage and hay from supervised trials in the USA following seed treatment of cyantraniliprole, 625 FS formulation

Soya bean forage and hay Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Frenchtown, NJ USA, 2011 (Pioneer 93Y70)	1	0.388				64	Forage	< 0.003, 0.004	< 0.003		DP29956 Trial 01 625 FS
						64	Hay	0.014, 0.012	0.013	0.004(J9Z38) 0.006(MLA84)	
		0.777				64	Forage	0.005, 0.004	0.005		
						64	Hay	0.025 0.02	0.022	0.007(J9Z38) 0.010(MLA84)	
Athens, GA, USA, 2011 (Pioneer 95Y20)	1	0.386				75	Forage	< 0.003, < 0.003	< 0.003		DP29956 Trial 02 625 FS
						75	Hay	0.007, 0.004	0.006		
		0.773				75	Forage	0.004, < 0.003	< 0.003		
						75	Hay	0.007, 0.007	0.007		
Cheneyville, LA, USA, 2011 (Pioneer 95Y20)	1	0.291–0.364				61	Forage	0.003, 0.003	0.003		DP29956 Trial 04 625 FS
	1					61	Hay	0.013, 0.010	0.012	0.005(MLA84)	
	1	0.583–0.729				61	Forage	0.006, 0.008	0.007		
	1					61	Hay	0.026, 0.019	0.023	0.006(J9Z38) 0.010(MLA84)	
Fisk, MO, USA, 2011 (95Y20)	1	0.386				59	Forage	0.007, 0.008	0.007		DP29956 Trial 05 625 FS
	1					59	Hay	0.026, 0.031	0.028	0.004(J9Z38) 0.007(MLA84)	
	1	0.773				59	Forage	0.015, 0.012	0.013		
	1					59	Hay	0.062, 0.050	0.056	0.007(J9Z38) 0.016(MLA84)	
Ellendale, MN, USA, 2011 (92Y30)	1	0.383				62	Forage	< 0.003, < 0.003	< 0.003		DP29956 Trial 07 625 FS
	1					62	Hay	0.004, 0.004	0.004		
	1	0.786				62	Forage	< 0.003, < 0.003	< 0.003		
	1					62	Hay	0.011, 0.010	0.010		
Gardner, ND, USA, 2011 (Pioneer 90M80)	1	0.403				63	Forage	< 0.003, < 0.003	< 0.003		DP29956 Trial 08 625 FS
	1					63	Hay	0.007, 0.008	0.008		
	1	0.806				63	Forage	0.006, 0.004	0.005		
	1					63	Hay	0.014, 0.014	0.014		
Northwood, ND, USA, 2011 (Pioneer 90M80)	1	0.387				95	Forage	< 0.003, < 0.003	< 0.003		DP29956 Trial 09 625 FS
	1					95	Hay	0.004, 0.004	0.004		
	1	0.774				95	Forage	< 0.003, < 0.003	< 0.003		
	1					95	Hay	0.007, 0.007	0.007		
Marysville, OH, USA, 2011 (93Y70)	1	0.384				72	Forage	< 0.003, < 0.003	< 0.003		DP29956 Trial 10 625 FS
	1					72	Hay	0.008, 0.008	0.008		
	1	0.768				105	Mature Seed	< 0.003, < 0.003	< 0.003		
	1					72	Forage	< 0.003, < 0.003	< 0.003		
	1					72	Hay	0.010, 0.010	0.010		
Rochelle, IL, USA, 2011	1	0.386				73	Forage	< 0.003, < 0.003	< 0.003		DP29956 Trial 11

Soya bean forage and hay Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
(Pioneer 93Y70)	1					73	Hay	0.006, 0.006	0.006		625 FS
	1	0.773				73	Forage	< 0.003, < 0.003	< 0.003		
	1					73	Hay	0.012, 0.010	0.011	0.004(J9Z38)	
Tipton, MO, USA, 2011 (93Y70)	1	0.409				80	Forage	0.005, 0.004	0.005		DP29956 Trial 13 625 FS
	1					80	Hay	0.012, 0.011	0.012	0.005(MLA84) 0.005(JCZ38)	
	1	0.817				80	Forage	0.009, 0.010	0.010		
	1					80	Hay	0.017, 0.019	0.018	0.004(J9Z38) 0.006(JCZ38)	
Fisk, MO, USA, 2011 (95M50)	1	0.386				56	Forage	0.008, 0.009	0.009		DP29956 Trial 14 625 FS
	1					56	Hay	0.024 0.020	0.022	0.005(MLA84)	
	1	0.773				56	Forage	0.006, 0.011	0.009		
	1					56	Hay	0.030, 0.031	0.031	0.004(J9Z38) 0.010(MLA8)	
Gardner, KS, USA, 2011 (93Y70)	1	0.386				60	Forage	< 0.003, 0.054	0.028		DP29956 Trial 15 625 FS
	1					60	Hay	0.008, < 0.003	0.005		
	1	0.771				60	Forage	< 0.003, < 0.003	< 0.003		
	1					60	Hay	0.004, < 0.003	< 0.003		
Staffford, KS, USA, 2011 (Pioneer 93Y70)	1	0.386				56	Forage	0.009, 0.011	0.010		DP29956 Trial 16 625 FS
	1					56	Hay	0.034, 0.037	0.036	0.013(MLA84) 0.004(JCZ38)	
	1	0.773				56	Forage	0.018, 0.022	0.020	0.007(MLA84)	
	1					56	Hay	0.061 0.057,	0.059	0.005(J9Z38) 0.022(MLA84) 0.007(JCZ38)	
York, NE, USA, 2011 (93Y12)	1	0.372				53	Forage	0.004, < 0.003	< 0.003		DP29956 Trial 17 625 FS
	1					53	Hay	0.008, 0.009	0.008		
	1	0.743				53	Forage	0.003, 0.003	0.003		
	1					53	Hay	0.013, 0.012	0.012	0.003(MLA84)	
Springfield, NE, USA, 2011 (93Y70)	1	0.392				72	Forage	< 0.003, < 0.003	< 0.003		DP29956 Trial 18 625 FS
	1					72	Hay	0.004, 0.005	0.005		
	1	0.783				72	Forage	< 0.003, < 0.003	< 0.003		
	1					72	Hay	0.005, 0.005	0.005		

Maize

Table 42 Residues in field maize forage and stover from supervised trials in the USA following seed treatment of cyantraniliprole, FS formulation

Maize forage and hay Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	mg ai seed	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
North Rose, NY USA,2011 (101 RM)	1	0.04	0.5				Forage	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 01
							Stover	< 0.010, < 0.010	< 0.010		

Maize forage and hay Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	mg ai seed	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Seven Springs, NC USA,2011 (114 RM)	1	0.05	0.5				Forage	< 0.010, 0.0113	0.06		TK0029740-REG Test 02
							Stover	< 0.010, < 0.010	< 0.010		
Wyoming, IL USA,2011 (109 RM))	1	0.048	0.5				Forage	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 03
							Stover	< 0.010, < 0.010	< 0.010		
Carlyle, IL USA,2011 (109 RM)	1	0.04	0.5				Forage	0.0158, 0.0165	0.018		TK0029740-REG Test04
							Stover	< 0.010, < 0.010	< 0.010		
Fitchburg, WI USA,2011 (94 RM)	1	0.041	0.5				Forage	< 0.010, < 0.010	< 0.010		TK0029740-REG Test05
							Stover	< 0.010, < 0.010	< 0.010		
Rice, MN USA,2011 (94 RM)	1	0.043	0.5				Forage	0.0135, 0.0130	0.013		TK0029740-REG Tesst 06
							Stover	< 0.010, < 0.010	< 0.010		
Stafford, KS USA,2011 (105 RM)	1	0.04	0.5				Forage	0.0262, 0.0266	0.026		TK0029740-REG Test 07
							Stover	< 0.010, < 0.010	< 0.010		
Campbell, MN USA,2011 (94 RM)	1	0.043	0.5				Forage	< 0.010, < 0.010	< 0.010		TK0029740-REG Test08
							Stover	< 0.010, < 0.010	< 0.010		
Seymour, IL USA,2011 (114 RM)	1	0.041	0.5				Forage	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 09
							Stover	< 0.010, < 0.010	< 0.010		
Perley, MN USA,2011 (85 RM)	1	0.037	0.5				Forage	0.0203, 0.0222	0.021		TK0029740-REG Test 10
							Stover	< 0.010, < 0.010	< 0.010		
Geneva, MN USA,2011 (94 RM)	1	0.04	0.5				Forage	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 11
							Stover	< 0.010, < 0.010	< 0.010		
Northwood, KS USA,2011 (85 RM)	1	0.037	0.5				Forage	< 0.010, < 0.010	< 0.010		TK0029740-REG Test12
							Stover	< 0.010, < 0.010	< 0.010		
Stafford, KS USA,2011 (109 RM5)	1	0.039	0.5				Forage	0.0122, 0.0112	0.0117		TK0029740-REG Test 13
							Stover	< 0.010, < 0.010	< 0.010		
McVille, ND USA,2011 (85 RM)	1	0.037	0.5				Forage	< 0.010, < 0.010	< 0.010		TK0029740-REG Test14
							Stover	< 0.010, < 0.010	< 0.010		
Jefferson, IA USA,2011 (114 RM)	1	0.043	0.5				Forage	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 15
							Stover	< 0.010, < 0.010	< 0.010		

Maize forage and hay Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	mg ai seed	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
York,NE USA,2011 (114 RM)	1	0.04	0.5				Forage	< 0.010, < 0.010	< 0.010		TK0029740-REG Test16
							Stover	< 0.010, < 0.010	< 0.010		
Fitchburg, WI USA,2011 (94 RM)	1	0.039	0.5				Forage	0.0202, 0.0269	0.024		TK0029740-REG Test 17
							Stover	< 0.010, < 0.010	< 0.010		
Richland, IA USA,2011 (109 RM)	1	0.044	0.5				Forage	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 18
							Stover	< 0.010, < 0.010	< 0.010		
Bagley, IA /USA,2011 (114 RM)	1	0.039	0.5				Forage	0.0161, 0.0180	0.0170		TK0029740-REG Test 19
							Stover	< 0.010, 0.0131	< 0.010		
Wall, TX USA,2011 (114 RM)	1	0.041					Forage	0.0147, < 0.010	< 0.010		TK0029740-REG Test20
							Stover	< 0.010, < 0.010	< 0.010		
York, NE, USA,2011 (Hybrid A3035)	1	0.045					Forage	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 21
							Stover	< 0.010, < 0.010	< 0.010		
Geneva, MN USA,2011 (Hybrid A3035)	1	0.048					Forage	< 0.010, < 0.010	< 0.010		TK0029740-REG Test22
							Stover	< 0.010, < 0.010	< 0.010		
Gardner, ND USA,2011 (Hybrid A3035)	1	0.052					Forage	< 0.010, < 0.010	< 0.010		TK0029740-REG Test 23
							Stover	< 0.010, < 0.010	< 0.010		

Table 43 Residues in field maize forage and stover from supervised trials in the USA following one seed treatment, FS formulation, plus two foliar applications of cyantraniliprole, WG formulation

Maize forage and hay Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	mg ai seed	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
North Rose, NY, USA, 2011 (101 RM)	1 + 2	0.04 + 0.15	0.5	2–200		1	Forage	3.6, 3.62	3.61	0.02(J9Z38)	TestTK0029740-REG Test 01
						14	Stover	0.75, 0.91	0.83	0.16(J9Z38)	
Seven Springs, NCUSA, 2011 (114 RM)	1 + 2	0.05 + 0.15	0.5	2–200		1	Forage	6.5, 5.78	6.14	0.02(J9Z38)	TK0029740-REG Test 02
						14	Stover	0.57, 0.54	0.56	0.07(J9Z38)	
Wyoming, IL USA,2011 (109 RM))	1 + 2	0.048 + 0.15	0.5	2–200		1	Forage	4.84, 4.87	4.86	0.02(J9Z38) 0.01(MYX98	TK0029740-REG Test 03
						14	Stover	3.75, 2.79	3.27	0.03(J9Z38) 0.01(MYX98)	

Maize forage and hay Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	mg ai seed	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Carlyle, IL USA,2011 (109 RM)	1 + 2	0.04 + 0.15	0.5	2–200		1	Forage	7.0, 7.57	7.29	0.01(J9Z38) 0.02(MYX98)	TK0029740-REG Test04
						14	Stover	4.38, 5.46	4.92	0.07(J9Z38) 0.03(MYX98) 0.01(N7B69)	
Fitchburg, WI USA,2011 (94 RM)	1 + 2	0.041 + 0.15	0.5	2–200		1	Forage	4.67, 4.51	4.59	0.02(J9Z38)	TK0029740-REG Test05
						14	Stover	< 0.010, < 0.010	< 0.010	0.04(J9Z38)	
Rice, MN USA,2011 (94 RM)	1 + 2	0.043 + 0.15	0.5	2–200		1	Forage	1.99, 1.32	1.67	0.03(J9Z38)	TK0029740-REG Tesst 06
						14	Stover	5.72, 8.44	7.08	0.03(J9Z38) 0.04(MYX98) 0.01(N7B69)	
Stafford, KS USA,2011 (105 RM)	1 + 2	0.04 + 0.15	0.5	2–200		1	Forage	4.43, 4.19	4.31	0.01(J9Z38) 0.02(MYX98)	TK0029740-REG Test 07
						14	Stover	< 0.010, < 0.010	< 0.010	0.01(J9Z38)	
Campbell, MN USA,2011 (94 RM)	1 + 2	0.043 + 0.15	0.5	2–200		1	Forage	4.64, 4.41	4.53	0.02(J9Z38) 0.02(MYX98)	TK0029740-REG Test08
						14	Stover	2.48, 3.10	2.97	0.02(J9Z38) 0.01(MYX98)	
TestTK0029740-09 Seymour, IL USA,2011 (114 RM)	1 + 2	0.041 + 0.15	0.5	2–200		1	Forage	3.12, 3.63	3.37	0.01(J9Z38) 0.01(MYX98)	TK0029740-REG Test 09
						14	Stover	3.60, 3.62	3.61	0.03(J9Z38)	
Perley, MN USA,2011 (85 RM)	1 + 2	0.037 + 0.15	0.5	2–200		1	Forage	6.35, 4.36	5.36	0.02(J9Z38) 0.03(MYX98)	TK0029740-REG Test 10
						14	Stover	7.44, 9.64	8.54	0.01(J9Z38)	
Geneva, MN USA,2011 (94 RM)	1 + 2	0.04 + 0.15	0.5	2–200		1	Forage	0.69, 0.66	0.68	0.01(J9Z38)	TestTK0029740-REG Test 11
						14	Stover	9.49, 9.17	9.33	0.04(J9Z38) 0.01(MYX98)	
Northwood, KS USA,2011 (85 RM)	1 + 2	0.037 + 0.15	0.5	2–200		1	Forage	6.57, 5.10	5.84	0.02(J9Z38) 0.01(MYX98)	TK0029740-REG Test12
						14	Stover	8.70, 16.2	12.45	0.03(J9Z38) 0.04(MYX98) 0.01(N7B69)	
TestTK0029740-13 Stafford, KS USA,2011 (109 RM5)	1 + 2	0.039 + 0.15	0.5	2–200		1	Forage	7.58, 8.75	8.17	0.02(J9Z38) 0.02(MYX98)	TK0029740-REG Test 13
						14	Stover	1.47, 1.38	1.43	0.01(J9Z38)	
McVille, ND USA,2011 (85 RM)	1 + 2	0.037 + 0.15	0.5	2–200		1	Forage	0.46, 0.35	0.41		TK0029740-REG Test14
						14	Stover	2.70, 2.0	2.35	0.01(J9Z38) 0.01(MYX98)	
Jefferson, IA USA,2011 (114 RM)	1 + 2	0.043 + 0.15	0.5	2–200		1	Forage	8.44, 12.4	10.42	0.03(J9Z38) 0.02(MYX98)	TK0029740-REG Test 15
						14	Stover	2.51, 1.85	2.18	0.01(J9Z38) 0.01(MYX98)	
York,NE USA,2011 (114 RM)	1 + 2	0.04 + 0.15	0.5	2–200		1 3 7	Forage	0.33, 0.37 0.08 0.02	0.35 0.08 0.02	0.01(J9Z38) 0.01(J9Z38)	TK0029740-REG Test16

Maize forage and hay Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	mg ai seed	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
						0 7 14 21 28	Stover	8.93 3.92 3.41, 3.02 3.2 0.32	8.93 3.92 3.22 3.20 0.32	 0.01(J9Z38) 0.01(J9Z38) 0.03(J9Z38) 0.03(J9Z38)	
Fitchburg, WI USA,2011 (94 RM)	1 + 2	0.039 + 0.15	0.5	2–200		1 3 3 7	Forage	2.21, 4.06 1.76 0.33	03.14 1.76 0.33	0.02(J9Z38) 0.04(J9Z38) 0.01(MYX98) 0.02(J9Z38)	TK0029740-REG Test 17
						0 7 14 21 28	Stover	3.87 6.19 4.88, 6.90 6.76 4.14	3.87 6.19 5.89 6.76 4.14	0.01(J9Z38) 0.03(J9Z38) 0.03(J9Z38) 0.05(J9Z38) 0.05(J9Z38)	
Richland, IA USA,2011 (109 RM)	1 + 2	0.044 + 0.15	0.5	2–200		1	Forage	5.17, 4.49	4.83	0.03(J9Z38)	TK0029740-REG Test 18
						14	Stover	3.64, 2.47	3.06	0.03(J9Z38)	
Bagley, IA /USA,2011 (114 RM)	1 + 2	0.039 + 0.15	0.5	2–200		1	Forage	5.75, 8.81	7.19	0.04(J9Z38) 0.01(MYX98)	TK0029740-REG Test 19
						14	Stover	< 0.010, < 0.01	< 0.01	0.02(J9Z38) 0.02(MYX98)	
Wall, TX USA,2011 (114 RM)	1 + 2	0.041 + 0.15	0.5	2–200		1	Forage	1.17, 0.86	1.02	0.01(J9Z38)	TK0029740-REG Test20
						14	Stover	2.43, 4.11	3.27	0.03(J9Z38) 0.03(MYX98)	
York, NE, USA, 2011 (Hybrid A3035)	1 + 2	0.045 + 0.15	0.5	2–200		14	Stover	1.44, 2.24	1.84	0.21(J9Z38) 0.01(MYX98)	TK0029740-REG Test 21
Geneva, MN USA,2011 (Hybrid A3035)	1 + 2	0.048 + 0.15	0.5	2–200		14	Stover	3.13, 2.55	2.84	0.11(J9Z38) 0.02(MYX98)	TK0029740-REG Test22
Gardner, ND USA,2011 (Hybrid A3035)	1 + 2	0.052 + 0.15	0.5	2–200		14	Stover	0.82, 1.15	0.99	0.11(J9Z38)	TK0029740-REG Test 23

Almond hulls

Table 44 Residues in almond hulls from supervised trials in the USA following three foliar applications of cyantraniliprole, 100 SE formulation

ALMOND hull Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Dinuba, CA, USA, 2011) (Non Pareil)	1 + 1 + 1 +	0.15	284 291 290	52	7	5	hull	0.68, 0.77	0.72	M1 = 0.01	DP-32057 Trial 01
Terra Bella, CA, USA, 2011 (Carmell)	1 + 1 + 1 +	0.15	32 32 34	474 477 439	7	5	hull	1.4, 1.4	1.4	M1 = 0.01	DP-32057 Trial 02
Strathmore, CA, USA, 2011 (Fritz)	1 + 1 + 1	0.15	34 35 34	444 443 440	7	4	hull	1.1, 0.72	0.93		DP-32057 Trial 03

ALMOND hull Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Sanger, CA, USA, 2011 (Non-Pareil)	1 + 1 + 1 +	0.15	32 34 32	478 446 473	7	5	hull	1.6, 2.2	1.9		DP-32057 Trial 04

Table 45 Residues in almonds hull from supervised trials in the USA following foliar applications of cyantraniliprole, 100 OD or SE formulations, (data previously reviewed by the 2013 JMPR)

ALMOND hull Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Turlock, CA USA, 2009 (Butte)	3	0.15	26	580	7, 6	5	hull	4.5, 4.6	4.6	M1 = 0.03 M2 = 0.01	DP-27446 Trial 01
Kerman, CA USA, 2009 (Non-Pareil)	3	0.15	32	470	7	5	hull	2.0, 1.7	1.9	M1 = 0.01	DP-27446 Trial 02
Sanger, CA USA, 2009 (Neplus)	1 + 1 + 1	0.15	23 27	650 600 540	6 7	5	hull	0.78, 0.98	0.88		DP-27446 Trial 03
Sutter, CA USA, 2009 (Non-Pareil)	3	0.15	310	50	7	5	hull	3.0, 2.8	2.9	M1 = 0.02	DP-27446 Trial 04
Sanger, CA USA, 2009 (Neplus)	1 + 2	0.15 0.15	6 12	2400 1300	7 8	5	hull	1.2, 1.4	1.3	M1 = 0.01	DP-27446 Trial 05
	1 + 2	0.15 0.15	6 12	2400 1300	7 8	5	hull	2.3, 2.7	2.5	M1 = 0.01	DP-27446 Trial 05 [100 SE]
Madera, CA USA, 2009 (Non-Pareil)	3	0.15	330	50	6, 7	5	hull	0.88, 0.94	0.91		DP-27446 Trial 06
	3	0.15	11	1400	6, 7	5	hull	3.7, 3.5	3.6	M1 = 0.04	DP-27446 Trial 06 [100 SE]

M1: Average residues of metabolite IN-J9Z38

M2: Average residues of metabolite IN-MYX98

Cotton, gin trash

Table 46 Residues in cotton, gin trash from supervised trials in the USA following three foliar applications of cyantraniliprole, 100 OD formulation, (data previously reviewed by the 2013 JMPR)

COTTON, gin trash Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Hinton, OK USA, 2009 (FM1740B2F)	3	0.15	75	200	8, 9	9	gin trash	2.6, 2.6	2.6	M1 = 0.03 M2 = 0.01	DP-27565 Trial 07
Edmonson, TX USA, 2009 (DP 924)	3	0.15	97	160	8, 6	7	gin trash	4.3, 5.7	5	M1 = 0.02 M2 = 0.03	DP-27565 Trial 08

COTTON, gin trash Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Levelland, TX USA, 2009 (9063 B2F)	3	0.15	63	234	7	8	gin trash	3.5, 3.5	3.5	M1 = 0.07 M2 = 0.02	DP-27565 Trial 09
Uvalde, TX USA, 2009 (DP6167 B2RF)	3	0.15	65	234	7	6	gin trash	2.8, 2.6	2.7	M1 = 0.07 M2 = 0.01	DP-27565 Trial 10

M1: Average residues of metabolite IN-J9Z38

M2: Average residues of metabolite IN-MYX98

Fate of residues in storage and processing

Maize (corn)

In two field trials on maize conducted in the USA and reported by Thomas J. Mäyer, 2013 [Ref: TK0029740], plots were treated with one seed treatment of 0.5 mg ai/seed (FS formulation) plus two late season foliar applications at an exaggerated rate of 0.75 kg ai/ha (5×) cyantraniliprole (WG formulation) with added surfactant; samples of were taken 14 day before harvest for processing.

Bulk samples were composited and shipped at ambient temperature directly to the processing facility where samples were processed into aspirated grain fraction, meal, flour, grits, refined oil (dry and wet milling) and starch.

Table 47 Residues in fresh and processed maize from supervised trials in North America following three foliar applications of cyantraniliprole, 100 OD formulation

Maize Study ID	Matrix	Cyantraniliprole	IN-J9Z38	Total		Other metabolites (mg/kg)					
		mg/kg	mg/kg	mg/kg	PF	M2	M3	M4	M5	M6	M7
TK0029740 Trial 18	Grain	0.08	< 0.01	0.09							
	AGF	15.9	0.07	15.97	177.4	0.05	0.01			N/A	N/A
	Meal	0.03	< 0.01	0.04	0.44						
	Flour	0.02	< 0.01	0.03	0.33						
	Grits	0.01	< 0.01	0.02	0.22						
	Oil—dry	< 0.01	< 0.01	< 0.02	< 0.22						
	Oil—wet	< 0.01	< 0.01	< 0.02	< 0.22				0.03		
	Starch	< 0.01	< 0.01	< 0.02	< 0.22						
TK0029740 Trial 19	Grain	0.08	< 0.01	0.09							
	AGF	15.7	0.05	15.75	175	0.07	0.02			N/A	N/A
	Meal	0.01	< 0.01	0.02	0.22						
	Flour	0.01	< 0.01	0.02	0.22						
	Grits	< 0.01	< 0.01	< 0.02	< 0.22						
	Oil—dry	< 0.01	< 0.01	< 0.02	< 0.22						
	Oil—wet	< 0.01	0.03	0.04	0.44						
	Starch	< 0.01	< 0.01	< 0.02	< 0.22						

AGF: Aspirated Grain Faction, N/A: Not Applicable

M2: Residues of metabolite IN-MYX98 M3: Residues of metabolite IN-N7B69

M4: Residues of metabolite IN-MLA84 M5: Residues of metabolite IN-JCZ38

M6: Residues of metabolite IN-N5M09 M7: Residues of metabolite IN-F6L99

For calculation purposes, where the residue in the processed commodity was below the LOQ, a value of 0.01 mg/kg was used. Where residues of IN-J9Z38 are below the LOQ in the RAC, a value of 0.01 has been used to calculate 'total' residues.

Cotton seed

Table 48 Residues in raw and processed cotton seed from supervised trials in the USA following three foliar applications of cyantraniliprole, 100 OD formulation, (data previously reviewed by the 2013 JMPR)

COTTON SEED Study ID	Matrix	Cyantraniliprole	IN-J9Z38	Total		Other metabolites (mg/kg)					
		mg/kg	mg/kg	mg/kg	PF	M2	M3	M4	M5	M6	M7
DP-27565 Trial 4	cottonseed	0.52	< 0.01	0.53						< 0.003	< 0.003
	raw oil (solvent extr)	0.02	< 0.01	0.03	0.06					< 0.003	< 0.003
	refined oil (solvent extr)	< 0.003	0.02	0.02	0.04						
	meal (solvent extr)	0.05	< 0.003	0.05	0.09						
	hulls	0.17	< 0.01	0.18	0.34						
	raw oil (cold press)	0.16	< 0.003	0.16	0.3						
	refined oil (cold press)	< 0.003	0.02	0.02	0.04						
	meal (cold press)	0.06	< 0.003	0.06	0.11						
DP-27565 Trial 10	cottonseed	0.71	0.02	0.73						< 0.003	< 0.003
	raw oil (solvent extr)	0.017	0.016	0.03	0.04					< 0.01	< 0.01
	refined oil (solvent extr)	< 0.003	0.02	0.02	0.03						
	meal (solvent extr)	0.01	< 0.01	< 0.02	< 0.03						
	hulls	0.25	0.02	0.27	0.37						
DP-27565 Trial 13	cottonseed	1.6	0.01	1.6						< 0.003	< 0.003
	raw oil (solvent extr)	0.05	0.07	0.12	0.08					< 0.003	< 0.003
	refined oil (solvent extr)	< 0.003	0.08	0.08	0.05						
	meal (solvent extr)	0.06	0.02	0.08	0.05						
	hulls	0.42	< 0.01	< 0.43	< 0.27						
	raw oil (cold press)	0.34	< 0.01	< 0.34	< 0.21						
	refined oil (cold press)	< 0.003	0.07	0.07	0.04						
	meal (cold press)	0.11	< 0.003	0.11	0.07						

M2: Residues of metabolite IN-MYX98 M3: Residues of metabolite IN-N7B69

M4: Residues of metabolite IN-MLA84 M5: Residues of metabolite IN-JCZ38

M6: Residues of metabolite IN-N5M09 M7: Residues of metabolite IN-F6L99

For calculation purposes, where the residue in the processed commodity was reported as ND (< LOD), a value of 0.003 mg/kg was used and where residues were above the LOD but below the LOQ, a value of 0.01 mg/kg was used. In both cases, the PF was expressed as "less than" (e.g. < 0.01). Where residues of IN-J9Z38 are below the LOQ in the RAC, a value of 0.01 has been used to calculate 'total' residues.

Table 49 Summary of processing factors for cyantraniliprole and cyantraniliprole + IN-J9Z38

RAC	Matrix	Cyantraniliprole [a]		Cyantraniliprole + IN-J9Z38 [b]	
		Calculated processing factors	PF Median	Calculated processing factors	PF median
Corn	Grain				
	AGF			175, 177.4	176
	Meal			0.22, 0.44	0.33
	Flour			0.22, 0.33	0.27
	Grits			< 0.22, 0.22	0.22
	Oil—dry			< 0.22, < 0.22	< 0.22
	Oil—wet			0.44, < 0.22	0.33
	Starch			< 0.22, < 0.22	< 0.22
Cottonseed	Seed				
	raw oil (solvent extr)	0.04, 0.02, 0.03	0.03	0.06, 0.04, 0.08	0.06
	refined oil (solvent extr)	< 0.006, < 0.005, < 0.002	< 0.005	0.04, 0.03, 0.05	0.04
	meal (solvent extr)	0.1, 0.01, 0.04	0.04	0.09, < 0.03, 0.05	0.05
	Hulls	0.33, 0.35, 0.29	0.33	0.34, 0.37, < 0.27	0.34
	raw oil (cold press)	0.31, 0.21	0.26	0.3, < 0.21	0.25
	refined oil (cold press)	< 0.01, < 0.002	< 0.006	0.04, 0.04	0.04
	meal (cold press)	0.12, 0.07	0.1	0.11, 0.07	0.09

AGF: Aspirated Grain Faction

[a] Each value represents a separate study where residues were above the LOQ in the RAC. The factor is the ratio of the cyantraniliprole residues in the processed item divided by the residue of cyantraniliprole in the RAC.

[b] Each value represents a separate study where residues were above the LOQ in the RAC. The factor is the ratio of the combined cyantraniliprole plus IN-J9Z38 metabolite residues in the processed item divided by the residue of cyantraniliprole in the RAC.

APPRAISAL

Cyantraniliprole is a diamide insecticide with a mode of action (ryanodine receptor activation) similar to chlorantraniliprole and flubendiamide, with foliar and systemic activity. It is effective against the larval stages of lepidopteran insects and also on thrips, aphids and other chewing and sucking insects.

Cyantraniliprole was initially evaluated for toxicology and residues by JMPR in 2013 and a ADI of 0–0.03mg/kg bw/day was estastablished. An ARfD was deemed to be unnecessary. The residue definitions were also established:

Definition of residue for compliance with MRL for both animal and plant commodities: cyantraniliprole.

Definiton of residue for estimation of dietary intake for unprocessed plant commodities: cyantraniliprole.

Definition of residue for estimation of dietary intake for processed plant commodities: sum of cyantraniliprole and IN –J9Z38, expressed as cyantraniliprole.

Definition of residue for estimation of dietary intake for animal commodities:

sum of cyantraniliprole, *2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]-3,4-dihydro-3,8-dimethyl-4-oxo-6-quinazolinecarbonitrile [IN-J9Z38], 2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]-1,4-dihydro-8-methyl-4-oxo-6-quinazolinecarbonitrile [IN-MLA84], 3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2-(hydroxymethyl)-6-[(methylamino)carbonyl]phenyl]-1H-pyrazole-5-carboxamide [IN- N7B69] and 3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2[[(hydroxymethyl)amino]carbonyl]-6-methylphenyl]-1H-pyrazole-5-carboxamide [IN-MYX98],* expressed a cyantraniliprole.

The residue is not fat soluble.

At the Forty-sixth Session of the CCPR(2014), cyantraniliprole was scheduled for evaluation of additional use patterns by 2015 JMPR.

The Meeting received supervised residue trial data for foliar and soil applications of cyantraniliprole on a range of fruit and vegetable crops, cereals, tree nuts and tea, and information on registered uses of cyantraniliprole on corresponding crops. The processing studies on corn were also submitted to the Meeting.

Methods of analysis

The analytical methods were previously evaluated (2013 Meeting). The same methods were used in the trials submitted to the current Meeting, and are considered valid for the commodities evaluated.

Stability of residues in stored analytical samples

The stability of residues of cyantraniliprole and metabolites in stored samples was covered by the freezer stability studies evaluated by the 2013 JMPR, and is considered adequate for the trials submitted to the current Meeting.

Results of Supervised residue trials on crops

The Meeting received the residue trials for strawberry, greenhouse cucumber, bean, pea, soya bean, artichoke, maize, and tea.

Where residues have been reported as not detected (ND), i.e., <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 cyantraniliprole and the product labels were available from Canada, Columbia, India, Japan, Vietnam and USA.

Citrus fruits

The critical GAP for cyantraniliprole on citrus fruits is in USA: 3 foliar applications of 0.15 kg ai/ha with a total of 0.45 kg ai/ha/season, applied at least 7 days intervals with a PHI of 1 day. The 2013 Meeting received the supervised residue trials for cyantaniliprole on citrus fruit (orange, lemon, grapefruit and mandarin). The current Meeting evaluated the data against the GAP for citrus fruits from the USA. Cyantraniliprole was also registered for soil application in citrus, however, the residue trials with soil application showed that the soil application did not contribute significant residues in citrus fruits.

Orange

In trials conducted in USA and Europe matching the USA GAP (with 3 applications of 0.15 kg ai/ha, PHI of 1 day), cyantraniliprole residues in whole fruit were: 0.1(2), 0.12, 0.17, 0.2, 0.21, 0.22, 0.23, 0.26, 0.28, 0.3, 0.35 and 0.39 mg/kg (n=13). The cyantraniliprole residues in pulp were: 0.01, 0.013, 0.018, 0.021, 0.036, 0.04, 0.041, 0.043, 0.046, 0.064, 0.069, and 0.086(2) mg/kg (n=13).

Lemon

In trials conducted in USA matching the USA GAP (with 3 applications of 0.15 kg ai/ha, PHI of 1 day), cyantraniliprole residues in whole fruit were: 0.16(2), 0.19, **0.21 and 0.3 mg/kg (**n=5). Cyantraniliprole residues in pulp were: 0.023, 0.057, 0.063, 0.07 and 0.11 mg/kg (n=5).

Grapefruit

In trials conducted in USA matching the USA GAP (with 3 applications of 0.15 kg ai/ha, PHI of 1 day), cyantraniliprole residues in whole fruit were: 0.091, 0.12(2), 0.14, 0.16, 0.19, and 0.31**mg/kg (**n=7). Cyantraniliprole residues in pulp were: 0.014, 0.021, 0.026, 0.029, 0.032, 0.033 and 0.049 mg/kg (n=7).

Mandarins

In trials conducted in Europe matching the USA GAP (with 3 applications of 0.15 kg ai/ha, PHI of 1 day), cyantraniliprole residues in whole fruit were: 0.47 **mg/kg (**n=1). Cyantraniliprole residues in pulp were: 0.2 mg/kg (n=1).

The Meeting noted that the GAP in USA was for citrus and the medians of the data sets for oranges, lemons, grapefruits and mandarins differed by less than 5-fold, and agreed to consider a group maximum residue level. In deciding on the data set to use for estimating a group maximum residue level (the Kruskal-Wallis H-test indicated that the residue populations for oranges, lemons, grapefruits and mandarins were not different) it was agreed to combine the results to give a data set of: 0.091, 0.1(2), 0.12(3), 0.14, 0.16(3), 0.17, 0.19(2), 0.2, 0.21(2)**,** 0.22, 0.23, 0.26, 0.28, 0.3(2), 0.31, 0.35, 0.39 and 0.47 mg/kg (n=26) to recommend a maximum residue level for the citrus fruit group. It was agreed to combine the results in pulp to give a data set of: 0.01, 0.013, 0.014, 0.018, 0.021(2), 0.023, 0.026, 0.029, 0.032, 0.033, 0.036, 0.04, 0.041, 0.043, 0.046, 0.049, 0.057, 0.063, 0.064, 0.069, 0.07, 0.086(2), 0.11 and 0.2 mg/kg (n=26).

The Meeting estimated an STMR of 0.041 mg/kg and an HR of 0.2 mg/kg based on residues in pulp, and recommended a group maximum residue level of 0.7 mg/kg for cyantraniliprole on citrus fruit. The Meeting estimated an STMR of 0.20 mg/kg in orange fruit for calculation of STMR-P.

Pomegranate

The approved GAP for cyantraniliprole on pomegranate is available from India, up to 3 foliar applications of 0.09 kg ai/ha, applied at least 7-10 day intervals with a PHI of 5 days. The assessment was undertaken using the supervised residue trials for cyantaniliprole on pomeganate received by the 2013 Meeting. The current Meeting evaluated the data against the new GAP for pomegranate from India.

In one trial conducted on pomegranate in India matching the Indian GAP cyantraniliprole residues in rind, seed and juice were < 0.01 mg/kg (n=1). In other trials conducted in four locations in India with 2, 3, 5 applications at rate of 0.075-0.18 kg ai/ha and PHI of 5 days, the cyantraniliprole residues in rind, seed and juice were all < 0.01 mg/kg (n=11).

The Meeting noted that since different times and rates of application resulted in the same residues in pomegranate, the Meeting agreed to combine the data together to estimate a maximum residue level of 0.01* mg/kg, and an STMR of 0.01 mg/kg.

Fruiting vegetables, Cucurbits

The critical GAP for cyantraniliprole on cucurbit vegetables is in Canada, up to 4 foliar applications of 0.15 kg ai/ha, applied at least 5–7 day intervals with a PHI of 1 day.

The new trials conducted on protected cucumber in North America (with 3 applications of 0.15 kg ai/ha, a PHI of 0 day) did not match the critical GAP. The meeting confirmed the previous recommendation.

Legume vegetables

The critical GAP for cyantraniliprole on legume vegetables in Canada is up to 4 foliar applications of 0.15 kg ai/ha, applied at least 5 day intervals with a PHI of 1 day for succulent seed. The 2013 Meeting received the supervised residue trials for cyantaniliprole on bean and pea from Europe. The current Meeting received new trials on bean, pea and soya bean, and evaluated all trials available to the Meeting against the new GAP for legume vegetables from Canada.

Pea with pod

In trials conducted on pea with pod (edible-podded peas) in USA matching the Canadian GAP (4 foliar applications of 0.15kg ai/ha, 1 day PHI), cyantraniliprole residues in pea with pod were: 0.29, 0.61, 0.78 and 0.79 mg/kg (n=4).

The Meeting estimated an STMR of 0.7mg/kg, and the maximum residue level of 2.0 mg/kg for cyantraniliprole in pea with pod.

Pea without pod

In trials conducted on pea without pod (succulent shelled pea) in USA matching the Canadian GAP (4 foliar applications of 0.15 kg ai/ha, 1 day PHI), cyantraniliprole residues in seed of pea without pod were: 0.019, 0.046, 0.065, 0.076, 0.082 and 0.10 mg/kg (n=6).

The Meeting estimated an STMR of 0.07 mg/kg, and maximum residue level of 0.3 mg/kg in pea without pod.

Bean with pod

In trials conducted on bean with pod (edible-podded beans) in USA matching the Canadian GAP (4 foliar applications of 0.15kg ai/ha, PHI of 1 day), cyantraniliprole residues in bean were: 0.11, 0.11, 0.23, 0.29, 0.36, 0.43, and 0.73 mg/kg (n=7).

The Meeting estimated an STMR of 0.29 mg/kg and recommended the maximum residue level of 1.5 mg/kg for cyantraniliprole in bean with pod.

Bean without pod

In trials conducted on bean without pod (succulent shelled beans) in the USA matching the Canadian GAP (4 foliar applications at 0.15kg ai/ha, PHI of 1 day), cyantraniliprole residues in seed of succulent shelled bean were: 0.01, 0.023 and 0.057 mg/kg (n=3).

Since three trials were insufficient to estimate the STMR and maximum residue level, the Meeting agreed to extrapolate the STMR and maximum residue level from pea without pods. The Meeting estimated an STMR of 0.07 mg/kg, and a maximum residue level of 0.3 mg/kg in bean without pod.

Soya bean, immature seed

In trials conducted on soya bean in the USA matching Canadian GAP (4 applications at 0.15 kg ai/ha, PHI of 1 day). The cyantraniliprole residues in immature seed were: 0.019, 0.035, 0.036, 0.042 and 0.14 mg/kg (n=5)

The Meeting estimated an STMR of 0.036 mg/kg and recommended the maximum residue level of 0.3 mg/kg for cyantraniliprole in soya bean, immature seed.

Pulses

The critical GAP for cyantraniliprole on pulses in Canada is up to 4 foliar applications of 0.15 kg ai/ha, applied at 5 day intervals with a PHI of 7 days.

Beans (dry)

In new trials conducted on bean, dry (dry shelled beans) in USA matching the Canadian GAP (4 foliar applications of 0.15kg ai/ha, PHI of 7 day), cyantraniliprole residues in bean, dry were: < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, 0.01, 0.015, 0.021, 0.048, 0.049, 0.088 and 0.22 mg/kg (n=12).

The Meeting estimated a STMR of 0.01 mg/kg and recommended the maximum residue level of 0.3 mg/kg for cyantraniliprole in bean (dry).

Peas (dry)

In new trials conducted on pea, dry (dry shelled peas) in the USA matching Canadian GAP (4 foliar applications of 0.15kg ai/ha, PHI of 7 day), cyantraniliprole residues in peas (dry) were: 0.019, 0.077, 0.086 and 0.51 mg/kg (n=4).

The Meeting agreed that four trials were insufficient for the estimation of a STMR and maximum residue level recommendation.

Soya bean (dry)

In new trials, conducted on soya bean in the USA, matching the Canadian GAP (4 applications of 0.15 kg ai/ha, PHI of 7 days), cyantraniliprole residues in soya bean (dry) were: < 0.01, 0.011, 0.012, 0.017, 0.022, 0.023, 0.027, 0.027, 0.031, 0.031, 0.033, 0.044, 0.056, 0.061, 0.083, 0.1, 0.12, 0.13, 0.15, 0.16 and 0.25 mg/kg (n=21).

The meeting estimated an STMR of 0.033 mg/kg and recommended the maximum residue level of 0.4 mg/kg for cyantraniliprole in soya bean (dry).

Artichoke

The GAP for cyantraniliprole on artichoke in Canada is up to 4 foliar applications of 0.025–0.15 kg ai/ha with a total of 0.45 kg ai/ha/season, applied at least 5–7 day intervals with a PHI of 7 days.

The new trials conducted on artichoke in Europe (2 foliar applications of 0.05kg/ha) did not match the Canadian GAP.

Maize

The GAP for cyantraniliprole on maize is available from Canada, for seed treatment at 0.012–0.024 kg ai/ha (up to 0.25 mg ai/ seed, or 100 g ai/100 kg seeds).

There were no trials matching the Canadian GAP, however, the Meeting noted that in 23 trials conducted on maize in North America, seed treatment of 0.5 mg ai/ seed, i.e., 2× GAP rate, the residues of cyantraniliprole in maize grain were all < 0.01 mg/kg. The Meeting agreed to estimate a STMR of 0 mg/kg and recommend a maximum residue level of 0.01 mg/kg for cyantraniliprole in maize grain.

Tree nuts

The critical GAP for cyantraniliprole on tree nuts is from the USA, 3 foliar applications of 0.15 kg ai/ha, with a seasonal total of 0.45 kg ai/ha, applied at 7 day intervals with a PHI of 5 days. The Meeting received four new trials on almond and six new trials on pecan. In addition, the 2013 Meeting received supervised residue trials for cyantaniliprole on almond (6) and pecan (6). The current Meeting evaluated all available trials together against the GAP of the USA.

Almond

In trials conducted on almonds in the USA, matching US GAP (3 foliar application of 0.15 kg ai/ha, 0.45 kg ai/ha/season, PHI of 5 days), cyantraniliprole residues in nutmeat were < 0.01 (5), 0.01, 0.012, 0.014, 0.018 and 0.023 mg/kg (n=10).

Pecan

In trials conducted on pecans in the USA, matching US GAP (3 foliar application of 0.15 kg ai/ha, 0.45 kg ai/ha/season, PHI of 5 days), cyantraniliprole residues in nutmeat were all < 0.01 mg/kg (n=12).

The Meeting noted that the GAP in the USA was for tree nuts and the medians of the data sets for almond and pecan differed by less than 5-fold and agreed to consider a group maximum residue level. In deciding on the data set to use for estimating a group maximum residue level (the Kruskal-Wallis H-test indicated that the residue populations for almond and pecan were not different) it was agreed to combine the results to give a data set of: < 0.01(16), 0.01(2), 0.012, 0.014, 0.018 and 0.023 mg/kg (n=22) to recommend a maximum residue level for the tree nut group.

The Meeting estimated an STMR of 0.01 mg/kg, and recommended a group maximum residue level of 0.04 mg/kg for cyantraniliprole on tree nuts.

Oilseeds

The 2013 Meeting received supervised residue trials for cyantraniliprole on cotton, rapeseed and sunflower. The current Meeting evaluated the data against the GAP of the USA.

Cotton

The critical GAP for cyantraniliprole on cotton in the USA is for up to 3 foliar applications of 0.15 kg ai/ha with a total of 0.45 kg ai/ha/season, applied at 7 day intervals with a PHI of 7 days.

In trials conducted on cotton in the USA matching GAP, cyantraniliprole residues in cotton seed were: 0.012, 0.025, 0.035, 0.12, 0.12, 0.14, 0.16, 0.18, 0.2, 0.22, 0.26, 0.29 and 0.99 mg/kg (n=13).

The Meeting estimated an STMR of 0.16 mg/kg, and recommended the maximum residue level of 1.5 mg/kg for cyantraniliprole in cotton seed.

Rape seed (canola)

The critical GAP for cyantraniliprole on rape seed (canola) in the USA is up to 3 foliar applications of 0.15 kg ai/ha with a total of 0.45 kg ai/ha/season, applied at 7 day intervals with a PHI of 7 days.

In trials conducted on canola in the USA matching GAP, cyantraniliprole residues in rapeseed were: 0.019, 0.021, 0.022, 0.05, 0.059, 0.061, 0.07, 0.07, 0.084, 0.12, 0.17, 0.18, 0.27, 0.29, 0.32 and 0.61 mg/kg (n=16).

The Meeting estimated the maximum residue level of 0.8 mg/kg and an STMR of 0.077 mg/kg for cyantraniliprole in rapeseed.

Sunflower

The critical GAP for cyantraniliprole on sunflower in the USA is for up to 3 foliar applications of 0.15 kg ai/ha with a total of 0.45 kg ai/ha/season, applied at 7 day intervals with a PHI of 7 days.

In trials conducted on sunflower in the USA matching the USA GAP, cyantraniliprole residues in sunflower seed were: 0.028, 0.039, 0.059, 0.064, 0.067, 0.085, 0.092, 0.14 and 0.32 mg/kg (n=9).

The Meeting estimated the maximum residue level of 0.5 mg/kg and a STMR of 0.067 mg/kg for cyantraniliprole in sunflower.

Seed for beverages and sweets

Coffee

The 2013 Meeting received supervised residue trials for cyantraniliprole on coffee. The current Meeting evaluated the data against the new GAP from Columbia.

The new approved GAP for cyantraniliprole on coffee from Columbia is for up to 2 foliar application of 2.5–3.5 g ai/5 litres/100 trees, equivalent to 0.06–0.175 kg ai/ha with a total of 0.3 kg ai/ha/season, with a PHI of 7 days.

In two Brazilian trials matching the Columbian GAP, cyantraniliprole residues in green coffee beans were: < 0.01 and 0.02 mg/kg.

The Meeting noted that in a further eight trials from Brazil involved 2 foliar applications that matched the Columbian GAP but in which two soil drenches (0.01–0.06 g ai/100 mL/plant to achieve the equivalent of 0.2 kg ai/ha/treatment) were also applied 90 and 120 days before harvest, cyantraniliprole residues in green bean were: < 0.01 (3), 0.01(2), 0.02, 0.02, and 0.03 mg/kg (n=8).

The Meeting agreed that since the early season soil drench treatments did not appear to contribute to the final residue in coffee beans, the data from these two sets of results could be combined, giving a data set of: < 0.01(4), 0.01(2), 0.02(3) and 0.03 mg/kg (n=10).

The Meeting estimated a STMR of 0.01 mg/kg, and recommended a maximum residue level of 0.05 mg/kg for cyantraniliprole on coffee bean, with the withdrawal of the previous maximum residue level recommendation of 0.03 mg/kg.

Tea, green, dry

The approved GAP for cyantraniliprole on tea is from Japan, with 1 foliar application of 0.1–0.2 kg ai/ha and a PHI of 7 days.

In trials conducted in Japan matching the Japanese GAP, cyantraniliprole residues in tea, green(dry) were 4.19 and 20.6 mg/kg (n=2). The Meeting agreed that two trials were insufficient for the estimation of a STMR and a maximum residue level recommendation.

Animal feed

Bean forage and bean hay

The 2013 Meeting received supervised residue trials for cyantaniliprole on beans from Europe. The current Meeting received new trials on beans from the USA, and evaluated all available trials against the new GAP for pulses from Canada.

In new trials conducted on bean forage and hay (dry shelled beans) in the USA, matching the Canadian GAP (3 foliar applications of 0.15kg ai/ha, PHI of 7 day), cyantraniliprole residues in bean forage (dry matter) were: 6.3, 7.6, 11.6, and 16.9 mg/kg (n=4); cyantraniliprole residues in bean hay (dry matter) were: 5.2, 7.7, 9.2 and 19.1 mg/kg (n=4).

The Meeting estimated a median residue of 9.6 mg/kg and a highest residue of 16.9 mg/kg for cyantraniliprole in bean forage (dry matter) for the calculation of livestock dietary burdens.

The Meeting estimated a median residue of 8.5 mg/kg and a high residue of 19.1 mg/kg for cyantraniliprole in bean hay (dry matter), and recommended a maximum residue level of 40 mg/kg (DM).

Pea vine and pea hay

The 2013 Meeting received supervised residue trials for cyantaniliprole on peas from Europe. The current Meeting received new trials on peas from the USA, and evaluated all available trials against the new GAP for pulses from Canada.

In new trials conducted on pea vine and hay in USA matching the Canadian GAP (3 foliar applications of 0.15kg ai/ha, 7 day PHI), cyantraniliprole residues in pea vine (dry matter basis) were: 4.1, 6.6, 11.4 and 47.1 mg/kg (n=4); cyantraniliprole residues in pea hay (dry matter) were: 3.5, 6.6, 12.8 and 28.5 mg/kg (n=4).

The Meeting estimated a median residue of 9.0 mg/kg and a highest residue of 47.1 mg/kg (DM) for cyantraniliprole in pea vine (dry matter) for calculation of livestock dietary burdens.

The Meeting estimated a median residue of 9.7 mg/kg and a highest residue of 28.5 mg/kg for cyantraniliprole in pea hay, and recommended a maximum residue level of 60 mg/kg (DM) for cyantraniliprole in pea hay,

Soya bean forage and hay

The Meeting received new trials conducted on soya bean forage and hay from the USA, matching Canadian GAP (3 applications of 0.15 kg ai/ha, PHI of 7 days).

The cyantraniliprole residues in soya bean forage, on dry matter basis, were: 1.2, 2.7, 4.5, 4.9, 6.1, 12.0, 12.5 14.2, 16.9, 17.8, 21.6, 27.1, 27.2, 30.6, 39.5 and 45.3 mg/kg (n=16).

The cyantraniliprole residues in soya bean hay in dry matter were: 1.6, 2.5, 6.0, 10.0, 10.8, 10.9, 13.1, 13.2, 14.3, 22.5, 27.3, 28.4, 28.9, 32.8, 42.7 and 46.4 mg/kg (n=16)

The Meeting estimated a median residue of 15.5 mg/kg and a highest residue of 45.3 mg/kg for cyantraniliprole in soya bean forage (dry matter) for calculation of animal dietary burdens.

The Meeting estimated a median residue of 13.7 mg/kg and a highest residue of 46.4 mg/kg for cyantraniliprole in soya bean hay (dry matter), and recommended a maximum residue level of 80 mg/kg (DM) for cyantraniliprole in soya bean hay.

Almond hull

The 2013 Meeting received supervised residue trials for cyantaniliprole on almond hulls. The current Meeting evaluated the data against the new GAP from the USA.

In trials conducted on almonds hulls in the USA, matching US GAP (3 foliar application of 0.15 kg ai/ha, 0.45 kg ai/ha/season, PHI of 5 days), cyantraniliprole residues in almond hulls were: 0.72, 0.88, 0.93, 1.4, 1.9, 1.9, 2.5, 2.9, 3.6 and 4.6 mg/kg (n=10).

The Meeting estimated a mean residue of 1.9 mg/kg, a highest residue of 4.6 mg/kg on almond hulls for the purpose of estimating livestock dietary burdens.

Cotton gin trash

The 2013 Meeting received supervised residue trials for cyantraniliprole on cotton gin trash. The current Meeting evaluated the data against the GAP of the USA (3 applications of 0.15 kg ai/ha, with interval of 7 days and a PHI of 7 days).

In trials conducted on cotton in the USA, matching US GAP, residues in cotton gin trash were: 2.6, 2.7, 3.5 and 5 mg/kg (n=4)

The Meeting estimated the median residue of 3.1 mg/kg and the highest residue of 5 mg/kg in cotton gin trash for estimating livestock dietary burden.

Fate of residues during processing

The Meeting received processing studies on cyantranilipole residues in maize, cottonseed and oranges. The Meeting agreed that for commodities not being considered for maximum residue levels at this Meeting, the relevant processing studies would not be reviewed and processing factors would not be estimated. Estimated processing factors and STMR-Ps for the commodities considered at this Meeting are summarized below.

Summary of processing factors and STMR-P for cyantraniliprole+IN-J9Z38

RAC	Commodity	Cyantraniliprole+IN-J9Z38 [a]		RAC STMR (mg/kg) [b]	STMP-P (mg/kg)[d]
		Calculated processing factors	PF best estimate		
Maize	Grain			0.01	
	Asp gr fn [f]	175, 177.4	176		1.76
	Meal	0.22, 0.44	0.33		0.0033
	Flour	0.22, 0.33	0.27		0.0027
	Grits	<0.22, 0.22	0.22		0.0022
	Oil-dry	<0.22, <0.22	<0.22		<0.0022
	Oil-wet	0.44, <0.22	0.33		0.0033
	Starch	<0.22, <0.22	<0.22		<0.0022
Cottonseed [c]	RAC: seed			0.16	
	raw oil (solvent extr)		0.06		0.0096
	refined oil (solvent extr)		0.04		0.0064
	meal (solvent extr)		0.05		0.008
	hulls		0.34		0.054
	raw oil (cold press)		0.25		0.04
	refined oil (cold press)		0.04		0.0064
	meal (cold press)		0.09		0.014
Orange[(c)]	RAC: fruit			0.20	
	juice		<0.03		<0.006
	wet pulp		0.24		0.048
	dry pulp		<0.33		0.066
	meal		0.47		0.094
	molasses		0.59		0.12
	marmalade		<0.06		0.012
	oil		8.5		1.7
	canned		<0.03		<0.006
Orange	Oil	2.3, 8.2, 6.2[e]	6.2[e]		

[a] Each PF value represents a separate study where residues were above the LOQ in the RAC. The factor is the ratio of the combined cyantraniliprole plus IN-J9Z38 metabolite residues in the processed item divided by the residue of cyantraniliprole in the RAC.

[b] Residues in the RAC is cyantraniliprole.

[c] The processing factor was estimated in 2013 JMPR, the STMR-P was calculated in this Meeting.

[d] Residues in processed commodities is cyantraniliprole plus IN-J9Z38

[e] The processing factor based on residues of cyantraniliprole only for estimation of maximum residue level.

[f] Aspirated grain faction

The Meeting noted that in the studies available, cyantraniliprole residues did not concentrate in food commodities during processing except for orange oil. The Meeting estimated a maximum level of 4.5 (0.7×6.2) mg/kg for citrus oil, the processing factor was based on residues of parent only.

Residues in animal commodities

Farm animal dietary burden

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: pea, soya bean, cotton gin trash, maize and potatoes (including by-products). Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are presented Annex 6 to the Report and are summarized below.

Estimated maximum and mean dietary burden of farm animal (ppm of dry matter diet)

	Animal dietary burden, cyantraniliprole							
	US-Canada		EU		Australia		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	0.69	0.37	12.6	3.38	46.8 [a]	15.59 [c,]	0.14	0.009
Dairy cattle	9.86	3.42	14	3.82	35.95 [b]	12.05 [d]	0.29	0.024
Poultry-broiler	0.00	0.00	0.05	0.02	0.00	0.00	0.00	0.00
Poultry-layer	0.00	0.00	4.71 [e, g]	1.56 [f, h]	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 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

Animal commodity maximum residue level

For beef and dairy cattle, the calculated maximum dietary burden suitable for estimating maximum residue levels in mammalian tissues and milk are 47 ppm and 36 ppm dry weight of feed, and the calculated mean dietary burdens suitable for estimating STMRs in mammalian tissues and in milk are 16 ppm and 12 ppm dry weight of feed respectively. The residue levels of cyantraniliprole and metabolites included in the residue definition in milk and tissue were calculated by estimation based on 10ppm, 30ppm and 100ppm feeding level in the feeding studies.

Cyantraniliprole feeding study	Feed level, ppm, for		Residue [a], mg/kg				
	Tissue residue	Milk residue	Milk	Muscle	Liver	Kidney	Fat
MRL, beef or dairy cattle							
Feeding study [b]	30	30	0.445	0.11	0.936	0.427	0.27
	100	100	1.109	0.373	2.3	1.351	1.03
Dietary burden and high residue	47	36	0.50	0.17	1.26	0.65	0.45
STMR, beef or dairy cattle							
Feeding study [c]	10	10	0.11	0.026	0.246	0.128	0.065
	30	30	0.445	0.081	0.722	0.356	0.202
Dietary burden mean residue estimate	16	12	0.21	0.041	0.38	0.19	0.10

[a] Residue values used in estimating STMR are the sum of cyantraniliprole and metabolites IN-N7B69, IN-J9Z38, IN-MLA84 and IN-MYX98

[b] high residues for tissues and mean residues for milk

[c] mean residues for tissues and mean residues for milk

Residues of cyantraniliprole expected in cattle milk and tissues for use in estimating maximum residue levels are: 0.45 mg/kg (fat), 0.17 mg/kg (muscle), 1.26 mg/kg (liver) and 0.65 mg/kg (kidney) and the mean residue for milk is 0.50 mg/kg.

The Meeting estimated maximum residue levels of 0.2 mg/kg for cyantraniliprole in meat (from mammals other than marine mammals), 1.5 mg/kg for edible offal (mammalian), 0.5 mg/kg for mammalian fat and 0.6 mg/kg for milks. The Meeting estimated STMRs (parent plus metabolites) for dietary intake estimation are 0.041 mg/kg for meat, 0.38 mg/kg for edible offal, 0.1 mg/kg for fat and 0.21 mg/kg for milk. The previous recommendations should be replaced.

For poultry, noting that in some countries, laying hens may also be consumed; the calculated maximum dietary burden suitable for estimating maximum residue levels in poultry tissues and eggs is 4.7 ppm and the calculated mean dietary burden suitable for estimating STMRs in poultry tissues and in eggs is 1.6 ppm. The residue levels of cyantraniliprole and metabolites included in the residue definition in eggs and tissue were calculated by estimation based on 3.0 ppm and 10 ppm feeding level, or extrapolation below the 3.0 ppm feeding level in the feeding studies.

Residues in kidney and liver at the expected dietary burden

	Feed level, ppm, for		Residue [a], mg/kg			
	Tissues residues	Eggs residues	Eggs	Muscle	Liver	Fat
Highest residue level, hens						
Feeding study [b]	3	3	0.151	0.009	0.098	0.014
	10	10	0.32	0.028	0.225	0.084
Calculated burden	4.7	4.7	0.13	0.014	0.13	0.031
STMR, hens						
Feeding study [c]	3	3	0.082	0.0075	0.0617	0.0159
Calculated burden	1.6	1.6	0.0426	0.0039	0.0321	0.0083

[a] Residue values used in estimating STMR are the sum of cyantraniliprole and metabolites IN-N7B69, IN-J9Z38, IN-MLA84 and IN-MYX98

[b] high residues for tissues and mean residues for egg

[c] mean residues for tissues and mean residues for egg

Residues of cyantraniliprole expected in poultry egg and tissues for use in estimating maximum residue levels are: 0.031 mg/kg (fat), 0.014mg/kg (muscle), and 0.13mg/kg (liver) and the mean residue for egg is 0.13 mg/kg.

The Meeting estimated maximum residue levels of 0.02 mg/kg for cyantraniliprole in poultry meat, 0.15 mg/kg for poultry offal, 0.04 mg/kg for poultry fat and 0.15 mg/kg for eggs. The Meeting estimated STMRs (parent plus metabolites) for dietary intake estimation are 0.004 mg/kg for meat, 0.032 mg/kg for edible offal, 0.008 mg/kg for fat and 0.043 mg/kg for egg. The Meeting withdrew its previous recommendations.

RECOMMENDATIONS

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

CCN	**Commodity**	**Recommended Maximum residue level (mg/kg)**		**STMR or STMR-P mg/kg**	**HR or HR-P mg/kg**
		New	**Previous**		
VP 0526	Common bean (pods and/or immature seeds)	1.5		0.29	
VP 0062	Beans, shelled	0.3		0.07	
VD 0071	Bean(dry)	0.3		0.01	
AL 0061	Bean fodder	40(DM)[a]		8.5(DM)	19.1(DM)
FC 0001	Citrus	0.7		0.041	

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
OR 0001	Citrus oil, edible	4.5			
SB 0716	Coffee beans	0.05	0.03	0.01	
SO 0691	Cotton, seed	1.5		0.16	
MO 0105	Edible offal(Mammalian)	1.5	0.05	0.38	
PE 0112	Eggs	0.15	0.015	0.0426	
GC 0645	Maize	0.01		0	
MM 0069	Mammalian fat (except milk fats)	0.5	0.01	0.1	
MM 0095	Meat (from mammals other than marine mammals)	0.2	0.01	0.041	
ML 0106	Milks	0.6	0.02	0.21	
VP 0063	Peas (pods and succulent = immature seeds)	2.0		0.7	
VP 0063	Peas, shelled (succulent seeds)	0.3		0.07	
AL 0072	Pea hay or pea fodder (dry)	60(DM)		9.7(DM)	28.5(DM)
FI 0355	Pomegranate	0.01*		0.01	
PO 0111	Poultry, edible offal of	0.15	0.01	0.0321	
PF 0111	Poultry fat	0.04	0.01	0.0083	
PM 0110	Poultry meat	0.02	0.01	0.0039	
SO 0495	Rape seed	0.8		0.077	
VP 0541	Soya bean, immature seed	0.3		0.036	
VD 4521	Soya bean (dry)	0.4		0.033	
AL 0541	Soya bean fodder	80(DM)		13.7(DM)	46.4(DM)
SO 0702	Sunflower seed	0.5		0.067	
TN 0085	Tree nuts	0.04		0.01	
AN 0660	Almond hulls			1.9	4.6
AL 1030	Bean forage (green)			9.6(DM)	16.9(DM)
AB 0001	Citrus pulp, dry			0.066	
OR 0691	Cotton seed meal(cold press)			0.014	
AB 1204	Cotton gin trash			3.1	5.0
AL 0528	Pea vines (green)			9.0(DM)	47.1(DM)
AL 1265	Soya bean, forage (green)			15.5(DM)	45.3(DM)

[a] DM – Dry matter

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intake (IEDI) for cyantraniliprole 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 to the 2015 Report.

The International Estimated Daily Intakes of cyantraniliprole for the 17 GEMS/Food regional diets, based on estimated STMRs were 2–20% of the maximum ADI of 0.03 mg/kg bw. The Meeting concluded that the long-term intake of residues of cyantraniliprole from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The 2013 JMPR decided that an ARfD was unnecessary and concluded that the short-term intake of cyantraniliprole residues is unlikely to present a public health concern.

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Author	Year	Title, institute, report reference
Kingston, CK	2014a	Cyantraniliprole formulated product residue bridging. DuPont Stine-Haskell Research Center. DuPont Report No. DuPont-30877, Revision No. 1. Unpublished
Kingston, CK	2014b	Cyantraniliprole: Relative residues for different application method treatment regimes. DuPont Stine-Haskell Research Center. DuPont Report No. DuPont-30876, Revision No. 1. Unpublished
Aitken, A, Just, G, Haigh, I	2013	Magnitude and decline of cyantraniliprole (DPX-HGW86) and metabolite residues in strawberries (berries and small fruit) following foliar application of DPX-HGW86 100 g/L OD—Europe—2011 Initiation. Charles River Laboratories. DuPont Report No. DuPont-29223. Unpublished
McConnell, K., Haigh, I., Just, G	2013	Magnitude and decline of cyantraniliprole (DPX-HGW86) and metabolite residues in protected and field-grown strawberries (berries and small fruit) following soil application of DPX-HGW86 200 g/L SC—Europe—2012 initiation. Charles River Laboratories. DuPont Report No. DuPont-34085. Unpublished.
Dorschner, KW	2012	Cyantraniliprole: Magnitude of the residue on cucumber (greenhouse). IR-4 Project, Rutgers, the College of New Jersey. Report No. IR-4 PR No.10313. Unpublished
Thiel, A	2012	Magnitude and decline of DPX-HGW86 and metabolite residues in legume vegetables (edible-podded beans/peas, succulent shelled beans/peas, and dry shelled beans/peas) following foliar applications of DPX-HGW86 100 g/L SE and DPX-HGW86 100 g/L OD—USA, 2011. ABC Laboratories, Inc. DuPont Report No. DuPont-31668. Unpublished
Seck, C	2012	Cyantraniliprole—Residue study on peas without pods (fresh) in southern France, Italy and Spain in 2011. Battelle UK Ltd. Report No. TK0057193-REG. Unpublished
Seck, C	2012	Cyantraniliprole—Residue study on peas without pods (fresh) in the United Kingdom and nothern France. Battelle UK Ltd. Report No. TK0057194-REG. Unpublished
Seck, C	2013	Cyantraniliprole—Residue study on peas without pods (fresh) in northern France and Germany in 2012. Battelle UK Ltd. Report No. TK0112971-REG. Unpublished
Seck, C	2013	Cyantraniliprole—Residue study on peas without pods (fresh) in southern France, Spain and Italy in 2012. Battelle UK Ltd. Report No. TK0112985-REG. Unpublished
Thiel, A	2013	Magnitude of DPX-HGW86 and metabolite residues in legume vegetables (soya beans) following foliar applications of DPX-HGW86 100 g/L OD—USA, 2011/2012. ABC Laboratories, Inc. DuPont Report No. DuPont-29956. Unpublished
Samoil, KS	2013	Cyantraniliprole (HGW86): Magnitude of the residue on carrot. IR-4 Project, Rutgers the State Universtiy of New Jersey. Report No. IR-4 PR No. 10364. Unpublished
Ure, GB	2013	Cyantraniliprole: Magnitude of the residue on radish. Pest Management Centre; Agriculture and Agri-Food Canada. Report No. AAFC 10-002R. Unpublished
Samoil, KS	2013	Cyantraniliprole: Magnitude of the residue on radish. IR-4 Project, Rutgers, the State University of New Jersey. Report No. IR-4 PR No. 10641. Unpublished
McConnell, K, Haigh, I, Just, G	2013	Magnitude and decline of cyantraniliprole (DPX-HGW86) and metabolite residues in artichokes (stem vegetables) following foliar application of DPX-HGW86 100 g/L OD—Southern Europe—2011 initiation. Charles River Laboratories. DuPont Report No. DuPont-29224. Unpublished
Thiel, A	2013	Magnitude of DPX-HGW86 and metabolite residues in legume vegetables (soya beans) following foliar applications of DPX-HGW86 100 g/L OD—USA, 2011/2012. ABC Laboratories, Inc. DuPont Report No. DuPont-29956. Unpublished
Mäyer, TJ	2013	Cyantraniliprole FS and WG (A17960B and A16971B)—Magnitude of the residues in or on field and popcorn resulting from seed treatment only and from seed treatment + foliar applications USA 2011. Sygenta Crop Protection.

Author	Year	Title, institute, report reference
		Report No. TK0029740. Unpublished
Qing, HX	2011	Residue test report of cyantraniliprole on the rice field. Institute of Quality and Standard for Agro-products, Zhejiang Academy of Agricultural Scienices. Report No. CL-2010-026. Unpublished
Thiel, A	2012	Magnitude of cyantraniliprole and metabolite residues in tree nuts (almonds and pecans) following foliar applications (DPX-HGW86 100 g/L SE)—NAFTA, 2011. ABC Laboratories, Inc. DuPont Report No. DuPont-32057. Unpublished
Higuchi, S	2013	Magnitude of residue of cyantraniliprole and it metabolites on tea. Japan Analytical Chemistry Consultants Co., Ltd. DuPont Report No. DuPont-37521, Revision No. 1. Unpublished

CYAZOFAMID (281)

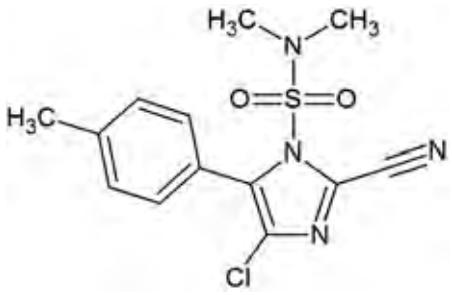
The first draft was prepared by Dr Michael Doherty, United States Environmental Protection Agency, Washington, DC, USA

EXPLANATION

Cyazofamid (ISO common name, published) is a fungicide belonging to both the cyano-imidazole and sulphonamide classes of compounds. The biochemical mode of action is inhibition of all stages of fungal development. It is registered for control of and protection against Oomycete fungi. Cyazofamid was considered for the first time for toxicology and residues by the 2015 JMPR.

Note that throughout this document, values are listed to the precision provided in the submitted reports, except for method recoveries (whole number) and values calculated by the JMPR (two significant figures for processing factors and for combined residues of cyazofamid and CCIM). All rounding was in accordance with ISO standards.

IDENTITY

ISO common name	Cyazofamid (published)
Chemical Name	
IUPAC	4-chloro-2-cyano-N,N-dimethyl-5-*p*-tolylimidazole-1-sulfonamide
CAS	4-chloro-2-cyano-N,N-dimethyl-5-(4-methylphenyl)-1H-imidazole-1-sulfonamide
CIPAC No.	653
CAS No.	120116-88-3
Structural Formula	
Molecular formula	$C_{13}H_{13}ClN_4O_2S$
Molecular mass	324.8

Physical and chemical properties

Table 1 Physical and chemical properties of cyazofamid

Property	Guideline and method	Test material specification and purity	Findings	Reference/ Remarks
Technical Grade Active Ingredient				
Physical state and colour	EC Annex II Section 2.4 and 40 CFR 158.190 Pesticide Assessment Guidelines Subdivision D: Product Chemistry Guidelines	Cyazofamid TGAI Lot 9506 96.4%	Munsell colour (24.5°C) = 5Y 9/1 “ivory” Physical state (25.3°C) = “solid powder”	RA-1006

Property	Guideline and method	Test material specification and purity	Findings	Reference/ Remarks
	63-2, 3, 4			
Solubility in organic solvents	40 CFR 158.190 Pesticide Assessment Guidelines, Subdivision D: Product Chemistry Guidelines 63-8 and EC Annex II Section 2.7	Cyazofamid TGAI Lot 9506, 95.5%	Solubility (g/L;21.2 ± 1 °C) Acetone 45.64 Ethyl Acetate 16.49 Dichloromethane 102.12 Acetonitrile 30.59 Methanol 1.74 Toluene 6.00 Hexane 0.03 n-Octanol 0.04 2-Propanol 0.43	RA-1033 Test material unstable in methanol, toluene, and n-octanol. Equilibrium achieved and maintained; did not impact study results.
Flammability	OPPTS 830.7000 and EC Annex II Section 2.11.1	Cyazofamid TGAI Lot 9506 95.5%	Test substance was "non-flammable"	RA-1029
Auto-flammability	OPPTS 830.7000 and EC Annex II Section 2.11.2	Cyazofamid TGAI Lot 9506 95.5%	No auto flammable behaviour was observed	RA-1029
Explosive Properties	EEC Directive 92/69/EEC, Part A.14	Cyazofamid TGAI Lot 9506 95.5%	The substance was not considered to be explosive: 1) not thermally sensitive 2) not shock sensitive 3) not sensitive to friction	RA-1049
Pure Active Ingredient				
Melting, freezing or solidification point	EC Annex II Section 2.1.1 and 40 CFR 158.190 Pesticide Assessment Guidelines, Subdivision D: Product Chemistry Guidelines 63-2, 3, 4	Cyazofamid PAI Lot 9505 99.1%	Mean melting point 152.7 °C	RA-1005
Boiling point			Not relevant. Material is a solid and does not have a low melting point	RA-1005
Relative density of purified active substance	EC Annex II Section 2.2 and 40 CFR 158.190 Pesticide Assessment Guidelines, Subdivision D: Product Chemistry Guidelines 63-2, 3, 4	Cyazofamid PAI Lot 9505 99.1%	$D_{20/4}$ = 1.446 ± 0.0009	RA-1005
Vapour pressure of purified active substance	OPPTS 830.7950 and EEC Method A.4 Vapour Pressure	Cyazofamid PAI Lot 9704-1 99.1%	<1 x 10-7 torr (1.33 x 10-5 Pascal)	RA-1030
Physical state and colour	EC Annex II Section 2.4 and 40 CFR 158.190 Pesticide Assessment Guidelines Subdivision D: Product Chemistry Guidelines 63-2, 3, 4	Cyazofamid PAI Lot 9505 99.1%	Munsell color at 24.3 °C = N 9.5/90.0%R "white" Physical state at 25.4 °C = "solid powder"	RA-1005

Property	Guideline and method	Test material specification and purity	Findings	Reference/ Remarks
Dissociation Constant	EU Guideline 2.9.4 and OECD Guideline for Testing of Chemicals 112	Cyazofamid PAI Lot 9505 99%	Because no quantifiable spectral differences were observed from 200-750 nm, it was concluded that no pKa was evident in the pH range of 2-12 (20 ± 1 °C) using this method	RA-1007
Solubility in organic solvents	Pesticide Assessment Guidelines, Subdivision D: Product Chemistry Guidelines 63-8 and Japan MAFF 9 Nousan, Notification No. 5089 Product Chemistry Guidelines	Cyazofamid PAI Lot 9704-1 99.1%	Solubility (g/L; 20 ± 1 °C) Acetone 41.92 Ethyl Acetate 15.63 Dichloromethane 101.84 Acetonitrile 29.42 Methanol 1.54 Toluene 5.28 Hexane 0.03 n-Octanol 0.25 2-Propanol 0.39	RA-1044
Solubility in water	OPPTS 830.7840 and EC Annex II Section 2.6	Cyazofamid PAI, Lot 9505 99.0%	Mean solubility at 20 ± 1 °C pH 5 – 121 ppb pH 7 – 107 ppb pH 9 – 109 ppb	RA-1010 Test material was unstable in water; however, equilibrium was achieved and study results were not impacted.
n-octanol/ water partition coefficient	OPPTS 830.7570 and EC Annex II Section 2.8	Cyazofamid PAI Lot 9505, 99.0%	At 24-25 °C, the octanol/water partition coefficient was 1585 (Log Kow = 3.2)	RA-1037
Direct phototrans-formation of purified active substance in water	United States EPA Guideline 161-2 EC Directive, Annex II, Sections 2.9.2 and 7.2.1.2	[^{14}C-Bz]Cyazofamid, Lot CP-1863-2, purity ≥99.5%; [^{14}C-Im]Cyazofamid, Lot CP-1864, purity ≥99.5%; Cyazofamid PAI Lot 9505, 99.0%	[14C]Cyazofamid and product half-lives **Cyazofamid(Bz)** 28 minutes **Cyazofamid(Im)** 34 minutes **Cyazofamid(Bz)** CCIM 20.7 days CCTS 2.3 days HTID 46.1 days **Cyazofamid(Im)** CCIM 25.6 days CCTS 2.1 days HTID 41.6 days	RA-4013
Hydrolysis at pH 4, 7, and 9	United States EPA Guideline 161-1 EC Directive, Annex II, Sections 2.9.1 and 7.2.1.1	[^{14}C-Bz]Cyazofamid, Lot CP-1863-2, purity ≥99.5%; [^{14}C-Im]Cyazofamid, Lot CP-1864, purity ≥99.5%;	At pH 4, 5, 7 and 9 at 25ºC, the main product of hydrolysis was CCIM. After 30 days, CCIM represented 82-83% of the radioactivity in the pH 4, 5 and 7 samples and 74-77% of the radioactivity in the pH 9 samples. At pH 9, further	RA-4003

<table>
<tr><th>Property</th><th>Guideline and method</th><th>Test material specification and purity</th><th>Findings</th><th>Reference/ Remarks</th></tr>
<tr><td></td><td></td><td>Cyazofamid PAI Lot 9505, 99.0%</td><td>reaction formed CCIM-AM. At the end of the study, CCIM-AM represented 9-10% of the radioactivity in the pH 9 samples. The course of the hydrolysis was the same at 50 ºC.
Half-lives in days (20 °C is an estimate)
<table>
<tr><th></th><th colspan="2">25 ºC</th><th>20 ºC</th></tr>
<tr><th>pH</th><th>Bz</th><th>Im</th><th>Bz</th></tr>
<tr><td>4</td><td>12.4</td><td>12.3</td><td>24.6</td></tr>
<tr><td>5</td><td>13.3</td><td>12.6</td><td>27.2</td></tr>
<tr><td>7</td><td>12.1</td><td>12.3</td><td>24.8</td></tr>
<tr><td>9</td><td>11.8</td><td>10.6</td><td>24.8</td></tr>
</table></td><td></td></tr>
</table>

Cyazofamid is registered as a suspension concentrate (SC) formulation containing 400 g ai/L.

METABOLISM AND ENVIRONMENTAL FATE

Metabolism and environmental fate studies were conducted with cyazofamid labelled in either the imidazole (Im) or benzene (Bz) rings (Figure 1 and Figure 2, respective).

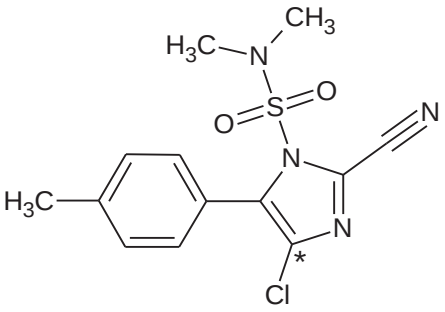


Figure 1. [imidazole-14C]cyazofamid

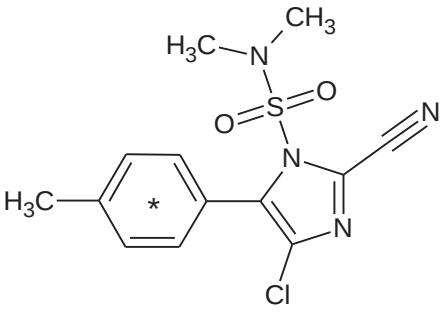
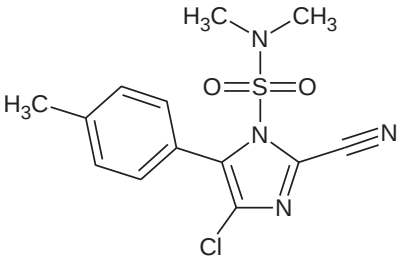
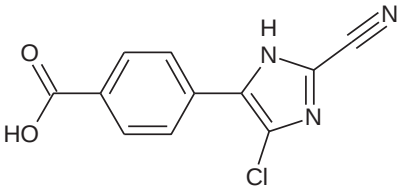
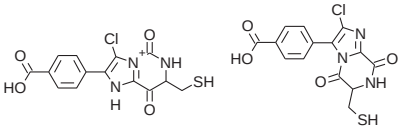
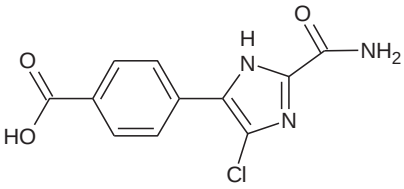
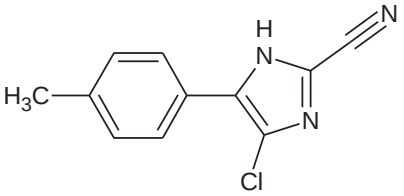
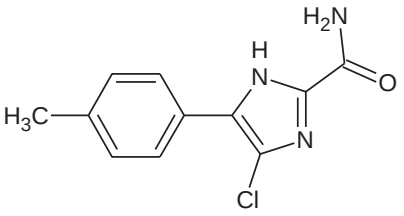
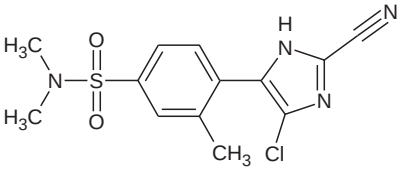
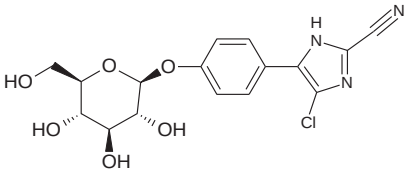


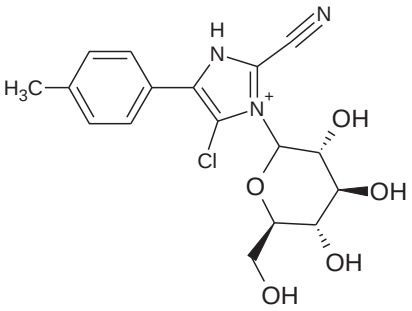
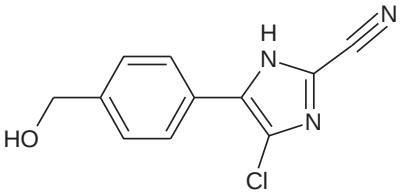
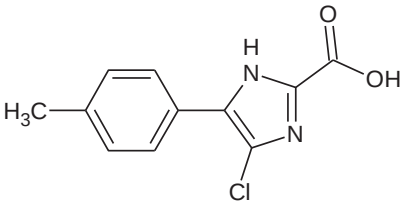
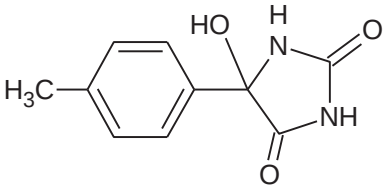
Figure 2. [benzene-14C]cyazofamid

Chemical names, structures, and code names of metabolites and degradation products of cyazofamid are shown below (Table 2). All of the compounds in Table 2 were identified in at least one matrix in studies with radiolabelled cyazofamid.

Table 2 Known metabolites and degradation products of cyazofamid

Code Names	Chemical name, molecular formula, molar mass	Structure	Where found ≥ 10% TRR
Cyazofamid	4-chloro-2-cyano-N,N-dimethyl-5-*p*-tolylimidazole-1-sulfonamide		Tomato Lettuce Potato (rinsate only) Confined rotational lettuce tops

Code Names	Chemical name, molecular formula, molar mass	Structure	Where found ≥ 10% TRR
CCBA	4-(4-chloro-2-cyanoimidazol-5-yl)benzoic acid		Hen kidney Hen liver
CCBA (cysteine conjugates)			Goat kidney Goat fat Goat muscle Milk
CCBA-AM	4-(4-chloro-2-amidoimidazol-5-yl)benzoic acid		Milk
CCIM	4-chloro-5-p-tolylimidazole-2-carbonitrile		Goat fat Goat muscle Hydrolysis Photolysis Aerobic soil metabolism Confined rotational lettuce tops
CCIM-AM	4-chloro-5-p-tolylimidazole-2-carboxamide		Goat liver Goat fat Hydrolysis Aerobic soil metabolism Confined rotational lettuce tops
CCTS	6-(4-chloro-2-cyanoimidazol-5-yl)-N,N-dimethyl-m-toluenesulfonamide		Photolysis
CGCN	4-chloro-5-(4-{[(2S,3R,4S,5S,6R)-3,4,5-trihydroxy-6-(hydroxymethyl)tetrahydro-2H-pyran-2-yl]oxy}phenyl)-1H-imidazole-2-carbonitrile		Rotational crops

Code Names	Chemical name, molecular formula, molar mass	Structure	Where found ≥ 10% TRR
5CGTC	4-chloro-2-cyano-5-(4-methylphenyl)-3-[(3R,4S,5S,6R)-3,4,5-trihydroxy-6-(hydroxymethyl)tetrahydro-2H-pyran-2-yl]-1H-imidazol-3-ium		Rotational crops
CHCN	4-chloro-5-(4-hydroxymethylphenyl)imidazole-2-carbonitrile		Hen kidney (conjugated) Hen liver (conjugated)
CTCA	4-chloro-5-p-tolylimidazole-2-carboxylic acid		Aerobic soil metabolism
HTID	5-hydroxy-5-(4-methylphenyl)imidazolidine-2,4-dione		Rotational crops

Plant metabolism

The Meeting received studies depicting the metabolism of cyazofamid in grape, tomato, lettuce, and potato. All of the studies were conducted with cyazofamid which was radiolabelled, separately in the imidazole (Im) and the benzene (Bz) ring (see Figs. 1 and 2).

Grape

The nature of the residues of cyazaofamid in Pinot Noir grapes were investigated by Mamouni (1997, Report RA-3002). For each radiolabel position, five applications of cyazofamid, formulated as a suspension concentrate, were made to grapevines growing in the field. All five applications were at a rate of ca. 100 g ai/ha and were made at intervals of 21–25 days. Grapes were harvested 44 days after the last treatment (DAT). Harvested grapes were crushed and assayed for total radioactivity. The harvested grapes were also processed into wine, yielding vin de goutte, vin de presse, and marc, and into juice, yielding juice and pulp. Both types of wine were clarified using bentonite and/or centrifugation. Radioactivity was determined by combustion/LSC (grapes and solid material after preparation of wine and juice) or direct LSC [wine (raw and clarified) and juice]. Neither characterization nor identification of residues was reported in the study.

Mean (n=5) TRR in grapes was 0.31 mg eq./kg (0.62 % of applied) for the Im label and 0.53 mg eq./kg (0.89% of applied) for the Bz label. When the grapes were processed into wine, TRR (both labels) distributed as approximately 15% into vin de goutte (0.21 mg eq./L), 10% into

vin de presse (0.32 mg eq./L), and 70% into marc (3.7 mg eq./kg). A small amount (<6% TRR) of radioactivity was associated with the nylon bag used to press the grapes. Following clarification, 74 to 90% of the initial radioactivity remained in the vin de goutte and 50 to 60% remained in the vin de presse. When the grapes were processed into juice, TRR (both labels) distributed as 33% (0.30 mg eq./L) into juice and 54% (1.4 mg eq./kg) into marc. More radioactivity was retained by the nylon bag (12% TRR) used for juicing than the bag used for wine making.

Tomato

The metabolism of cyazofamid in tomato was investigated by Neal and Gupta (1999, Report RA-3009). For each radiolabel position, four applications of cyazofamid, formulated as a suspension concentrate (10% ai) and at rates of approximately 60, 95, 95, and 95 g ai/ha, were made at 7-day intervals to foliage of tomato plants grown in the field. At additional test plots, applications were made at exaggerated rates (nominally 4×). Samples from the exaggerate-rate trials were not analysed.

Mature tomato fruits and foliage were harvested 1 DAT. The harvested fruits were rinsed with ACN, homogenized, and centrifuged to separate juice from pulp. Radioactivity was determined by LSC for the rinsate and juice, and by combustion/LSC for the pulp. Residues in pulp were extracted with hexane, ethyl acetate, and H_2O, separately. Tomato rinsate, juice, and pulp extracts were subjected to various levels of clean-up and HPLC analysis. For juice, the TM-1 fractions from the HPLC analysis underwent ion exchange chromatography, hydrolysis of oligosaccharides, oxidation and reduction of sugars, dimedone adduction, esterification, and acetylation treatments to characterize the residues. Pulp samples were subjected to chemical treatments in order to fractionate the material into its cell wall components for determination of radioactivity in natural plant constituents.

The TRR in fruits, measured as a sum of residues in surface rinses, juice, and pulp were 0.080 mg eq./kg for the Im label and 0.290 mg eq./kg for the Bz label. Of the total residue, the majority was contained in the surface rinse (54% and 83% for the Im and Bz labels, respectively). Of the radioactivity remaining in the fruits, approximately 71-87% was in the pulp fraction. HPLC analysis indicated the presence of 18 metabolites. Across all sample components, cyazofamid was the only major residue, accounting for ca. 78% TRR (0.064 and 0.22 mg/kg). Other residues were < 6% TRR and < 0.02 mg eq./kg (Table 3).

Table 3 Characterization and identification of radioactive residues in tomato

	[Bz-^{14}C]		[Im-^{14}C]	
Metabolite	mg eq./kg	% TRR	mg eq./kg	% TRR
Total	0.2896		0.0801	
Cyazofamid	0.2212	76.4	0.064	79.9
CCBA	0.0015	0.5	0.0004	0.5
CCBA-AM	0.0001	0.03	n.d.	n.d.
Ester of CCBA	0.0008	0.3	0.0003	0.4
CCBG [a]	0.0009	0.3	0.0004	0.5
CCIM	0.0128	4.4	0.004	5.0
CCIM-AM	0.0028	1.0	0.0003	0.4
CCTS	0.0049	1.7	0.0012	1.5
CDTS	0.0028	1.0	0.0005	0.6
CHCN	0.0011	0.4	0.0001	0.1
CHCN conjugate	0.0007	0.2	0.0001	0.1
HTID	0.0011	0.4	0.0001	0.1
TM-1	0.0157	5.4	0.002	2.5
TM-1a	0.0013	0.4	0.0001	0.1
TM-3	0.0008	0.3	0.0002	0.2
TM-4	0.0007	0.2	0.0001	0.1
TM-5	0.0006	0.2	0.0001	0.1
TM-12	0.0005	0.2	n.d.	n.d.
Unextracted	0.0091	3.1	0.0021	2.6

[a] TRR and % TRR values are reported for fraction TM-6, which was found to contain, in part, CCBG.

Analysis of the TM-1 fraction showed incorporation of radioactivity into sugars (sucrose, glucose, and fructose) and citric acid (specific amounts not reported. Analysis of the post-extraction solids (PES) from the tomato pulp demonstrated incorporation of radioactivity in structural components (Table 4).

Table 4 Distribution of radioactivity into structural components of tomato pulp PES

	[Im-^{14}C]		[Bz-^{14}C]	
Matrix/Substance	mg eq./kg	%TRR of PES	mg eq./kg	%TRR of PES
PES pulp	0.0021	100	0.0091	100
Cellulose	0.0001	4.1	0.0008	8.4
Hemicellulose	0.0001	6.5	0.0009	10.0
Lignin	0.0002	8.6	0.0020	21.7
Pectin	0.0002	10.0	0.0010	10.7
Protein	0.0002	9.7	0.0009	9.8
Starch	0.0002	9.4	0.0007	8.2
Water-soluble polysaccharides	0.0003	14.6	0.0011	12.1
Solid residue	0.0001	2.4	0.0003	2.8
Total	0.0014	65.5	0.0076	83.7

Results from foliage were similar to those from fruits and are not further evaluated herein.

Lettuce

The metabolism of cyazofamid in glasshouse-grown lettuce was investigated by Gupta and Song (2002, Report RA-3092). Three applications of cyazofamid, formulated as a suspension concentrate (10% ai) containing both the Bz and Im radiolabel in a 1:1 ratio, were made to lettuce at a nominal rate of 100 g ai/ha. Applications were made on a 14-day interval and the final application was two weeks prior to harvest of mature leaves.

Harvested lettuce samples were homogenized and the TRRs in each sample were determined by combustion analysis and LSC. Samples were extracted three times with ACN:H_2O (60:40, 0.1% acetic acid; v/v). Radioactivity in the extracts was determined by LSC and the extracted residues were analysed by HPLC. Metabolites were isolated from the extract by HPLC fraction collection. Fractions were subjected to acid and base hydrolysis, and ion-exchange chromatography. Radioactivity of PES was determined by combustion/LSC, followed by chemical treatments to isolate radioactivity in cell wall components.

Total radioactive residues were 0.85 mg eq./kg, of which 97% was extracted (Table 5). Analysis of the extracts showed parent to be the predominant residue. Four metabolites were identified, of which CCIM was the most abundant (0.31 mg/kg, 3.7% TRR). Other identified metabolites were < 0.01 mg/kg (<1% TRR). Analysis of the extract and the PES showed incorporation of radioactivity into natural products (0.028 mg eq./kg, 3.3% TRR and 0.022 mg eq./kg, 2.6% TRR, respectively).

Table 5 Characterization and identification of residues in lettuce following application of cyazofamid

	Combined [Im-^{14}C] and [Bz-^{14}C]	
Matrix/fraction/metabolite	mg eq./kg	%TRR
Mature lettuce	0.85	100
Extracted residue	0.83	97.4
Cyazofamid	0.76	89.3
CCIM	0.031	3.7
CCTS	0.0041	0.5
CDTS	0.0052	0.6
Natural products	0.028	3.3
PES	0.022	2.6
Water-soluble polysaccharides	0.0051	0.60
Starch	0.0063	0.74

	Combined [Im-^{14}C] and [Bz-^{14}C]	
Matrix/fraction/metabolite	mg eq./kg	%TRR
Protein	0.0043	0.50
Cellulose, hemicellulose, pectin	0.0029	0.34
Lignin	0.0022	0.26

Potato

The metabolism of cyazofamid in both field-grown and greenhouse-grown potato was investigated by Gupta (1999, Report RA-3008). For each radiolabel position, cyazofamid was formulated as a soluble concentrate (10% ai) and applications were made to the foliage of growing plants on a one-week interval, with the final application being 7 days before harvest. For the field study, three applications were made at rates of either 100 (1 plant) or 400 (3 plants) g ai/ha. For the greenhouse study, five applications were made at a rate of 400 (3 plants) g ai/ha.

Samples of potato tuber were harvested, washed gently with water to remove soil, and then air dried. Tubers were rinsed with ACN:H_2O (80:20, v/v) prior to homogenization and the radioactivity in the rinsate was determined by LSC. One subsample from the field study and two samples from the greenhouse study were peeled prior to homogenization to determine residue levels in peel. Tuber samples and one separated peel/pulp sample were extracted sequentially with ACN, ACN:H_2O (80:20, v/v), and ACN:H_2O (50:50, v/v). Radioactivity in each extract was assayed. Extractable residues were analysed by HPLC. PES were analysed for incorporation of radioactivity into natural products. Potato foliage samples were also harvested and analysed. Residue in foliage was ca. 97% parent compound; therefore, detailed evaluation is not presented herein.

The majority of the radioactivity remained with the tuber after the ACN:H_2O rinse (Table 6). Based on the samples from the greenhouse study, approximately 20% of the residue in the rinsed potato was associated with the peel. The result from the field study indicates 50%, but that figure may be less robust due to the low residue levels and the results reflecting only one plant.

Table 6 Average (n=3) total radioactive residues (mg eq./kg) in and on potato tubers following treatment with cyazofamid

	[Im-^{14}C]		[Bz-^{14}C]	
Treatment	Rinsate	Tuber	Rinsate	Tuber
Field, 3×100 g ai/ha	0.00027	0.0019	0.00005[a]	0.0008[a]
Peel (% of TRR in tuber)	--	--	--	50
Pulp (% of TRR in tuber)	--	--	--	50
Field, 3×400 g ai/ha	0.00040	0.0051	0.00030	0.0053
Greenhouse, 5×400 g ai/ha	0.0014	0.022	0.0023	0.016
Peel (% of TRR in tuber)	--	21	--	22
Pulp (% of TRR in tuber)	--	79	--	78

[a] Results from one plant

The majority of the residue in the rinsate from the greenhouse 5×400 g ai/ha treatment consisted of cyazofamid (0.0009-0.0018 mg/kg, 67–80% TRR), with lesser amounts of CCIM (0.0003 mg/kg, 14–20% TRR; Table 7). For the tubers, three metabolite fractions were found in the extracts of peel and pulp. The majority of the radioactivity (0.005 mg/kg, ca. 30% TRR) was associated with a fraction that was shown to consist primarily of starch. The remaining two fraction were determined to be cyazofamid (0.001 mg/kg, 4.8% TRR) and CCIM (0.0002 mg/kg, 1% TRR). The balance of the radioactivity was not extracted.

Table 7 Identification of radioactive residues in rinsate and tuber extracts following treatment with cyazofamid

	[Im-^{14}C]		[Bz-^{14}C]	
Treatment/Matrix	mg/kg	% TRR	mg/kg	% TRR
Field, 3×100 g ai/ha				
Rinsate	0.00027 (eq)	--	0.00005 (eq)	--
Cyazofamid	--	--	--	--
CCIM	--	--	--	--
Starch	--	--	--	--
Tuber	0.0019 (eq)	--	0.0008 (eq)	--
Cyazofamid	0.00006	1.5	0.0	0.0
CCIM	0.00011	2.8	0.0	0.0
Starch	0.0012	30	0.0004	52.8
Greenhouse, 5×400 g ai/ha				
Rinsate	0.0014 (eq)	--	0.0023 (eq)	--
Cyazofamid	0.0009	67	0.0018	80
CCIM	0.0003	20	0.0003	14
Starch	0.0001	6.2	0.0001	2.6
Tuber	0.022 (eq)	--	0.016 (eq)	--
Cyazofamid	0.00083	4.7	0.0011	4.8
CCIM	0.0002	1.5	0.00017	0.6
Starch	0.0050	30	0.0049	23

Overall, the metabolism of cyazofamid in grape, tomato, lettuce, and potato is similar. The metabolic pathway proposed for grapes in the industry submission is provided as an illustrative example in Figure 3.

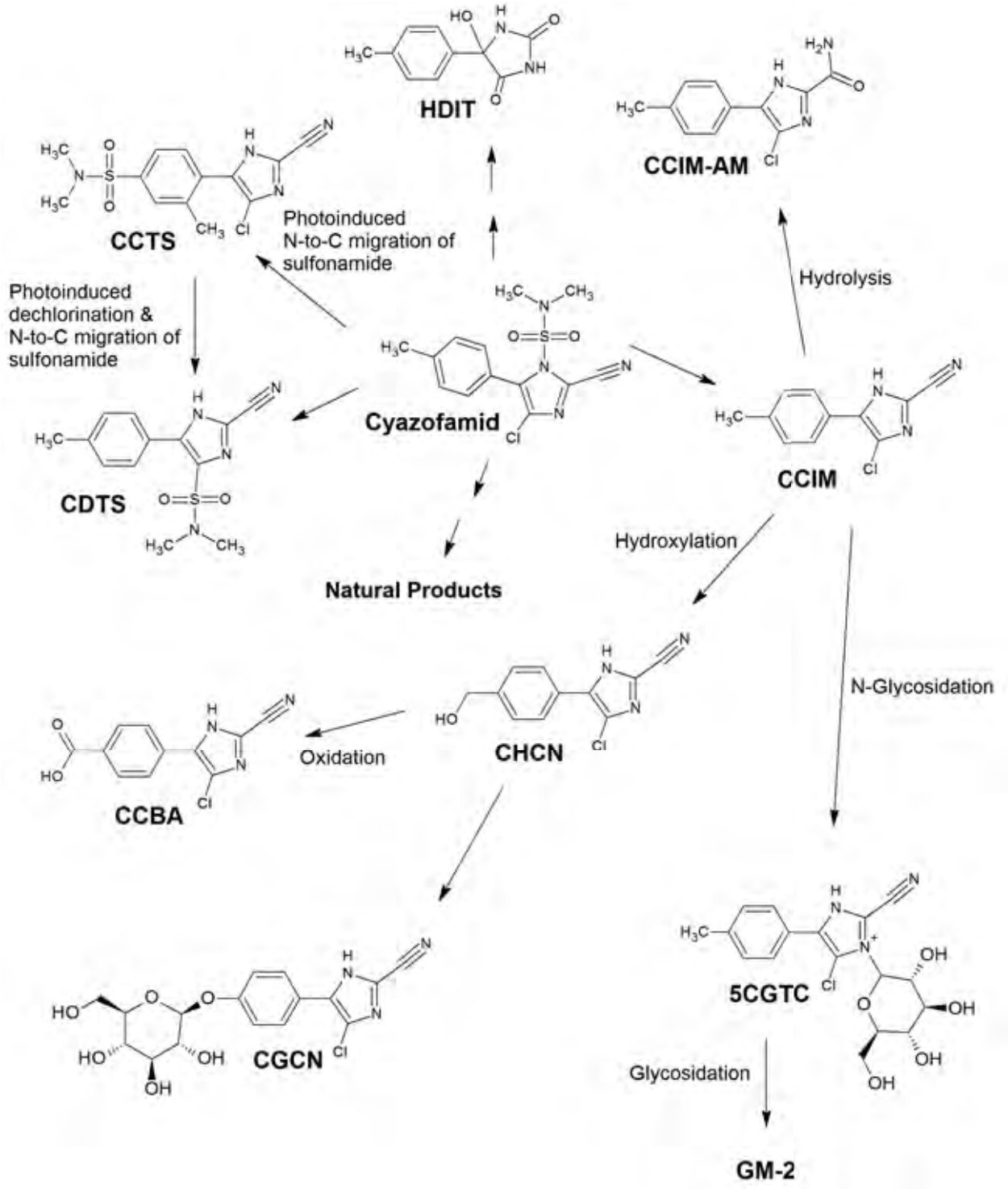


Figure 3 Proposed metabolic pathway of cyazofamid in target and rotational crops.

Animal Metabolism

The Meeting received metabolism studies on laboratory animals, lactating goats, and laying hens. All of the studies with laboratory animals, lactating goats, and laying hens were conducted with cyazofamid which was radiolabelled, separately, in the benzene ring [(u)Benzene-^{14}C; Bz] or the number 4 carbon of the imidazole ring [Imidazole-4-^{14}C; Im].

Laboratory animals

Examination of radioactivity following gavage dosing of Bz- and Im-radiolabelled cyazofamid to rats indicated that cyazofamid is well absorbed, with the majority of excretion occurring via urine. In a biotransformation study where blood, liver, and stomach (with contents) were analysed 0.5 hours after a dosing of [^{14}C-Bz] cyazofamid, most (97.2%) of the radiolabel in the stomach contents was the parent compound and a small fraction was CCIM. Analysis of the liver from this group showed only 6.1% of the radiolabel was cyazofamid, while 24.2% was CCIM and 41.9% was CCBA. In the plasma, there was no cyazofamid and the majority of radiolabel was CCIM. These data demonstrate that a dose of cyazofamid is rapidly metabolized, and that CCIM is a major metabolite in the initial metabolism of cyazofamid. At 0.5 hours after a dose of [^{14}C-Bz]-CCIM, all of the radiolabel in the stomach contents was CCIM, and most of the radiolabel in liver (76.5%) and plasma (67.9%) was CCIM. CCBA, the main metabolite seen in these tissues 0.5 hours after dosing with CCIM, was also found in the blood and liver from the animals dosed with cyazofamid. Concentrations in blood and liver were greater in the CCIM-dosed animals than that in cyazofamid treated animals, suggesting that CCIM was much more rapidly absorbed than cyazofamid.

Lactating goats

The metabolism of cyazofamid in lactating goats was investigated by Hatzenbeler and Savides (1998, Study RA-3001). For each radiolabel position, two goats were dosed for five days at 32.5 mg/day (Im) or 25.4 mg/day (Bz; both equivalent to ca. 10 ppm in the diet). Milk was collected twice daily, and excreta and stanchion washes were collected once daily throughout the study. Goats were terminated ca. 8 hours after the final dosing, at which point tissues and blood were collected for analysis.

Total radioactive residues (TRR) in urine stanchion wash, and milk were determined by direct liquid scintillation counting (LSC). The TRR in faeces, blood, and tissues was determined by combustion followed by LSC. Samples were processed differently, depending on the matrix. Milk from the Day 5 sampling was extracted twice with acetonitrile (ACN). The extract was then concentrated and the residues partitioned against hexane; the aqueous partition was concentrated prior to analysis by HPLC. Kidney, liver, and faeces were extracted once with ACN followed by two extractions with ACN:H_2O (50:50, v/v). The ACN and ACN:H_2O were concentrated, separately, and analysed by HPLC. The post-extraction solids (PES) were dried, combusted, and analysed by LSC to determine unextracted radioactivity. Liver PES were further processed by acid (1.0 M HCl) and base (1.0 M NaOH) hydrolysis and enzyme (protease) digestion, followed by LSC analysis and, in the case of the enzyme digest samples, HPLC. Muscle and fat samples were extracted two times with ACN:H_2O (75:25, v/v). The extracts were combined, concentrated, and analysed by HPLC. The PES were dried, combusted or solubilized, and analysed by LSC to determine unextracted radioactivity. The limit of detection for the LSC analysis was defined to be twice the disintegrations per minute of control samples, which translated to 0.001 to 0.005 mg eq./kg.

Four HPLC systems were used to analyse samples. All were reverse-phase systems using UV and radioactive flow detectors. The systems differed in the mobile phase gradients that were used, the specific column (though all were C-18), and the mobile phase modifiers (acetic acid or tetrabutylammonium bromide).

The total recovery of radioactivity was 58 and 60% of the administered dose (AD) for the Im and Bz labels, respectively (Table 8). The totals do not include the GI tract or its contents. Nearly 100% of the radioactivity was recovered in the excreta (99+%), with the principal residues being unchanged parent compound in faeces (ca. 85% TRR, ca. 7 mg eq./kg) and CCBA in urine (86-92% TRR, 1.6–2.2 mg eq./kg). Radioactivity in milk was low, ranging from 0.002 mg eq./kg to 0.010 mg eq./kg. The levels of TRR appeared to plateau for the Bz label by Day 2; however, the Im label showed a consistent increase over the five-day treatment period. Major metabolites (>10% TRR) identified in milk were CCBA (42% TRR, 0.004 mg eq./kg maximum on Day 5) and CCBA-AM (29% TRR, 0.002 mg eq./kg maximum on Day 5); all other identified residues in milk were <5% TRR (< 0.001 mg eq./kg). Radioactivity in liver was ca.

0.12 mg eq./kg, with CCIM-AM being the only major residue (12% TRR, 0.014 mg eq./kg). In kidney, the major residue was the cysteine conjugate of CCBA (70% TRR, 0.073 mg eq./kg). In muscle and fat, the major residues were CCBA and CCIM. In muscle, the two residues occurred at similar TTR levels (ca. 25%) and were low (≤ 0.002 mg eq./kg). In fat, CCBA occurred at higher levels than CCIM in terms of both relative (38–58% TRR vs. 26–33% TRR) and absolute (0.003–0.006 mg eq./kg vs. 0.002-0.003 mg eq./kg) amounts.

Table 8 Total radioactive residues (TRRs) of [^{14}C]cyazofamid in tissues, body fluids and excreta of lactating goats following exposure equivalent to 10 ppm in the diet

	[Im-^{14}C]Cyazofamid		[Bz-^{14}C]Cyazofamid	
Matrix	mg eq./kg	% of admin. dose	mg eq./kg	% of admin. dose
Tissues and milk				
Fat (omental)	0.010	0.01	0.006	< 0.01
Fat (perirenal)	0.010	< 0.01	0.010	< 0.01
Liver	0.12	0.13	0.11	0.10
Kidney	0.11	0.02	0.070	0.01
Milk (Day 5)	0.01	0.01	0.006	< 0.01
Muscle (loin)	0.006	0.03	0.004	0.03
Muscle (rear leg)	≤0.01	≤0.01	≤0.01	≤0.01
Blood and Excreta (average)				
Blood	0.059	0.13	0.053	0.15
Feces	6.7	8.7	6.5	9.2
Urine	2.3	2.9	1.8	2.7
Stanchion Wash	0.058	0.050	0.050	0.058
Total Recovery	--	58.2	--	60.0

Table 9 Time course of total radioactive residues (TRRs) of [Im-^{14}C]cyazofamid in milk and excreta

	Milk [a]				Urine				FAeces				Wash			
	mg eq./kg		% of AD		mg eq./kg		% of AD		mg eq./kg		% of AD		mg eq./kg		% of AD	
Samp. Day	Im	Bz	Im	Bz	Im	Bz	Im	Bz	Im	Bz	Im	Bz	Im	Bz	Im	Bz
1	0.005	0.005	0.01	0.00	2.2	1.9	2.6	3.0	3.6	3.5	5.2	5.3	0.11	0.069	0.04	0.05
2	0.006	0.005	0.01	0.00	2.2	1.7	3.4	3.2	6.7	7.2	9.9	11	0.027	0.031	0.03	0.04
3	0.007	0.005	0.01	0.01	2.2	1.9	3.3	3.2	7.4	7.7	12	13	0.035	0.037	0.04	0.05
4	0.008	0.005	0.01	0.00	2.4	1.7	3.5	2.7	8.5	7.5	13	12	0.029	0.037	0.03	0.05
5	0.010	0.006	0.01	0.00	2.5	2.0	1.6	1.5	7.4	6.8	3.5	4.5	0.095	0.074	0.11	0.10

[a] Weighted average of the morning and evening collections.

Table 10 Summary of extraction of radioactive residues from the cyazofamid goat metabolism study

			% TRR					
	TRR (mg eq./kg)		ACN		ACN:H_2O [a]		PES	
Matrix	[Im-^{14}C]	[Bz-^{14}C]	[Im-^{14}C]	[Bz-^{14}C]	[Im-^{14}C]	[Bz-^{14}C]	[Im-^{14}C]	[Bz-^{14}C]
Fat (omental)	0.010	0.006	--	--	100	100	0	0
Fat (perirenal)	0.010	0.010	--	--	100	100	0	0
Kidney	0.106	0.070	43	41	56	60	7	8
Liver	0.125	0.111	19	17	22	24	51	53
1.0 M HCl	--	--	--	--	--	--	12	--
1.0 M NaOH	--	--	--	--	--	--	18	--
Protease	--	--	--	--	--	--	15	--
Milk [b]	0.010	0.006	89	91	--	--	6	8
Muscle	0.006	0.004	--	--	73	74	27	26

[a] For kidney and liver, ACN:H_2O was 50:50 (v/v) and was subsequent to ACN extraction. For fat and muscle, ACN:H_2O was 75:25 (v/v) and was the only extraction solvent.

[b] Milk from Day 5.

Table 11 Characterization of radioactive residues in kidney

	Kidney			
	[Im-^{14}C]		[Bz-^{14}C]	
Fraction	mg eq./kg	%TRR	mg eq./kg	%TRR
TRR	0.106	100.0	0.070	100.0
Solvent Extracted	0.104	99.0	0.072	101.2
Cyazofamid	< 0.001	0.1	< 0.001	0.2
CCBA	0.010	8.4	0.003	4.2
CCBA-AM	0.007	7.0	0.005	6.2
CCBA (Cysteine conjugate)	0.073	69.7	0.050	70.0
CCIM	0.001	0.3	< 0.001	0.3
CCIM-AM	0.005	5.0	0.006	7.6
CSBA	0.004	3.6	0.003	4.5
Bound	0.008	7.2	0.006	7.6
Recovered	0.012	106.2	0.077	108.8

Table 12 Characterization of radioactive residues in liver

	Liver			
	[Im-^{14}C]		[Bz-^{14}C]	
Fraction	mg eq./kg	%TRR	mg eq./kg	%TRR
TRR	0.125	100.0	0.111	100.0
Solvent Extracted	0.052	41.1	0.046	40.9
Cyazofamid	< 0.001	0.3	< 0.001	0.2
CCBA (incl. Cysteine conjugate)	0.006	4.7	0.006	5.1
CCBA-AM	0.014	9.9	0.010	8.5
CCIM	0.002	1.4	0.002	1.6
CCIM-AM	0.014	11.1	0.014	12.2
Polar Region	0.007	5.0	0.004	3.4
Bound	0.064	51.4	0.057	52.9
Exhaustive extraction	--	--	0.058	46.4
1.0 M HCl				
Released	--	--	0.030	23.8
Organic soluble	--	--	0.008	6.2
Aqueous soluble	--	--	0.020	15.7
Bound	--	--	0.028	22.6
1.0 M NaOH				
Released	--	--	0.045	36.2
Organic soluble	--	--	0.010	8.2
Aqueous soluble	--	--	0.012	9.8
Emulsion layer	--	--	0.018	14.2
Bound	--	--	0.013	10.2
Protease				
Released	--	--	0.036	29.1
Organic soluble	--	--	--	--
Aqueous soluble	--	--	--	--
Bound	--	--	0.022	17.3
Recovered	0.116	92.5	0.104	93.8

Table 13 Characterization of radioactive residues in omental fat

	Omental Fat			
	[Im-^{14}C]		[Bz-^{14}C]	
Fraction	mg eq./kg	%TRR	mg eq./kg	%TRR
TRR	0.010	100.0	0.006	100.0
Solvent Extracted	0.010	100.0	0.006	100.0
Cyazofamid	< 0.001	3.7	< 0.001	1.9
CCBA (incl. Cysteine conjugate)	0.004	38.3	0.003	43.8
CCBA-AM	< 0.001	1.8	< 0.001	5.0
CCIM	0.003	30.5	0.002	33.4
CCIM-AM	< 0.001	4.1	< 0.001	5.4

	Omental Fat			
	[Im-^{14}C]		[Bz-^{14}C]	
Fraction	mg eq./kg	%TRR	mg eq./kg	%TRR
Polar Region	0.001	9.3	< 0.001	2.3
Bound	< 0.002	-	< 0.002	-
Recovered	0.010	100.0	0.006	100.0

Table 14 Characterization of radioactive residues in perirenal fat

	Perirenal Fat			
	[Im-^{14}C]		[Bz-^{14}C]	
Fraction	mg eq./kg	%TRR	mg eq./kg	%TRR
TRR	0.010	100.0	0.010	100.0
Solvent Extracted	0.010	100.0	0.010	100.0
Cyazofamid	< 0.001	1.2	< 0.001	0.6
CCBA (incl. Cysteine conjugate)	0.005	48.8	0.006	57.6
CCBA-AM	< 0.001	0.6	< 0.001	0.7
CCIM	0.003	28.6	0.003	26.1
CCIM-AM	0.001	5.2	0.001	10.7
Polar Region	0.001	5.5	< 0.001	1.5
Bound	< 0.002	-	< 0.002	-
Recovered	0.010	100.0	0.010	100.0

Table 14 Characterization of radioactive residues in milk

	Milk (Day 5)			
	[Im-^{14}C]		[Bz-^{14}C]	
Fraction	mg eq./kg	%TRR	mg eq./kg	%TRR
TRR	0.010	100.0	0.006	100.0
Solvent Extracted	0.009	89.0	0.005	91.4
Cyazofamid	< 0.001	1.0	< 0.001	1.2
CCBA (incl. Cysteine conjugate)	0.004	41.3	0.003	42.3
CCBA-AM	< 0.001	3.3	0.002	28.6
CCIM	< 0.001	0.4	< 0.001	0.7
CCIM-AM	< 0.001	1.9	< 0.001	2.6
Polar Region	0.003	30.6	< 0.001	1.6
Bound	0.001	6.4	< 0.001	7.5
Recovered	0.009	95.4	0.006	98.9

Table 16 Characterization of radioactive residues in muscle

	Muscle (loin)			
	[Im-^{14}C]		[Bz-^{14}C]	
Fraction	mg eq./kg	%TRR	mg eq./kg	%TRR
TRR	0.006	100.0	0.004	100.0
Solvent Extracted	0.004	73.2	0.003	73.6
Cyazofamid	< 0.001	1.0	< 0.001	0.7
CCBA (incl. Cysteine conjugate)	0.001	22.4	0.001	24.0
CCBA-AM	< 0.001	2.0	< 0.001	3.8
CCIM	0.002	22.6	0.001	26.8
CCIM-AM	< 0.001	3.6	< 0.001	6.9
Polar Region	0.001	11.9	< 0.001	2.6
Bound	0.002	26.8	0.001	26.4
Recovered	0.006	100.0	0.004	100.0

In summary, cyazofamid was not a significant residue in goat tissues or milk. The principal residues in were CCBA (free or cysteine-conjugated), CCIM, and their amide analogues. Although these metabolites are considered major residues based on percent of TRR, the absolute levels in mg eq./kg were generally low. The HPLC system used for most matrices

did not separate CCBA from its cysteine conjugate; however, the results from the analysis of kidney samples indicates that the cysteine conjugate likely makes up the majority of the CCBA-related residues. Extraction with ACN and/or ACN:H_2O extracted 73-101% of the radioactive residues from all matrices except liver (ca. 41%). Treatment of liver PES with acid, base, or enzymatic extraction released an additional 24-36% of the residue, of which a greater proportion partitioned into the aqueous or aqueous+emulsion fractions. Further identification was not possible due to low levels of radioactivity and matrix interferences. Analysis of the enzymatic extraction showed a number of radiolabelled components, none greater than 0.005 mg eq./kg. The proposed metabolic pathway in goats is summarized in Figure 4.

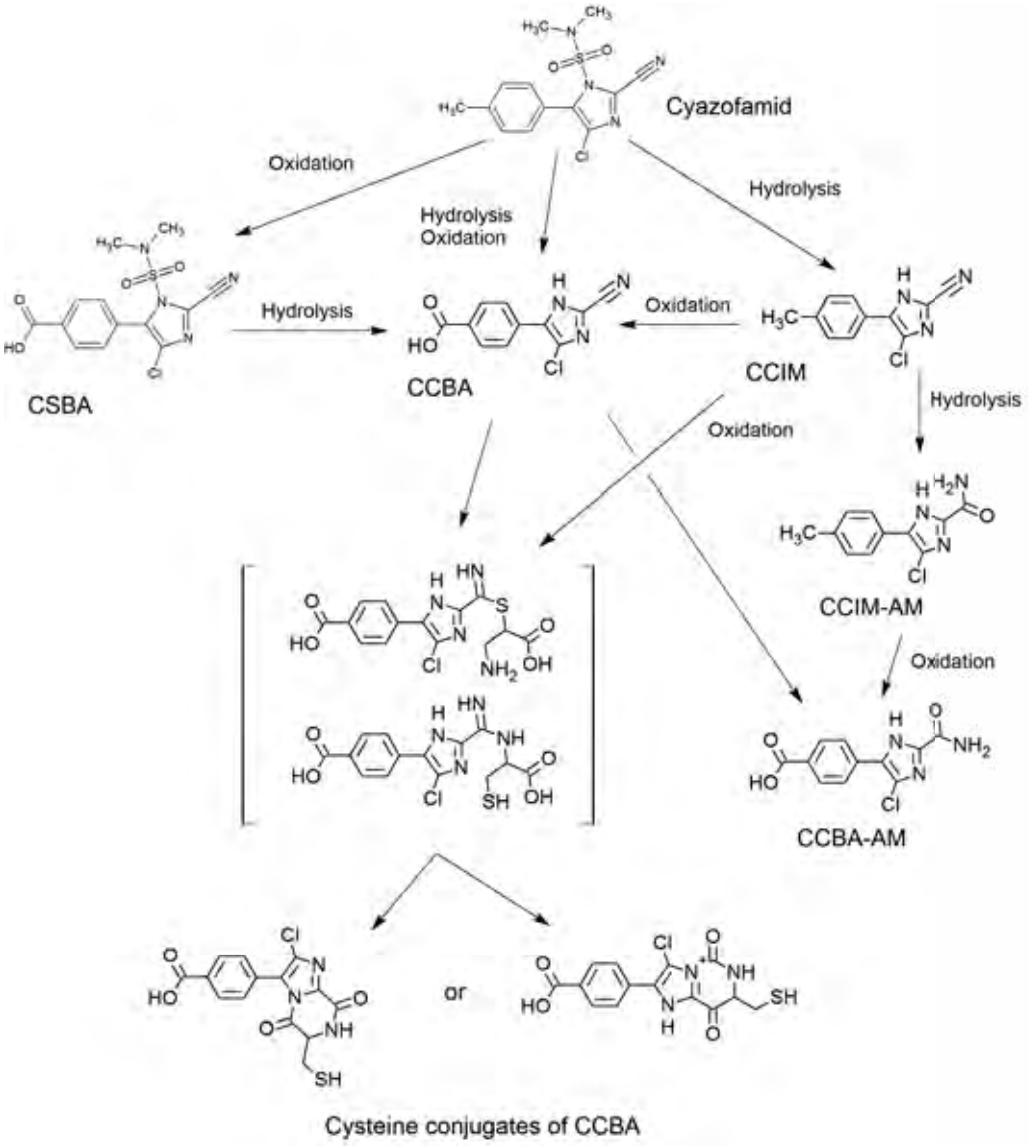


Figure 4 Proposed metabolic pathway of cyazofamid in lactating goat

Laying hens

The metabolism of cyazofamid in laying hens was investigated by Gupta and Bassett (1999, Study RA-3011). Cyazofamid was radiolabelled in the imidazole (Im) or benzene (bz) rings. For each radiolabel position, a group of ten hens was dosed for five consecutive days at ca. 1.1 mg/bird/day (equivalent to ca. 10 ppm in the diet). Eggs were collected twice daily, pooled based on test group from the evening and morning collections, and separated into yolks and whites. Excreta were

collected once daily throughout the study. Hens were sacrificed 9 hours after the final dosing, at which point tissues and blood were collected for analysis.

All samples were mixed or homogenized prior to subsampling for analysis. Tissues were homogenized in the presence of dry ice. Total radioactive residues were determined by combustion and LSC for all tissues and for egg white. The TRR of egg yolk was determined by direct LSC of solubilized sample. Excreta were extracted with ACN, and the TRR was determined by LSC of the extract and combustion/LSC of the PES. Samples of kidney and liver were each extracted with ACN (2×) followed by ACN:H_2O (50:50, v/v + 0.2-1% acetic acid; 2×). The PES from kidney, liver, and excreta were treated with 1.0 M HCl, protease, amylase, collagenase, 6.0 M HCl, and 1.0 M NaOH, in that order. The metabolic profiles of solvent-extracted liver, kidney, and excreta residues and 1.0 M HCl-extracted excreta residues were determined by HPLC. Samples of egg, breast muscle, thigh muscle, blood, fat, skin, and cage wash were not assayed for metabolite profiles due to the low level of radioactivity in those matrices. The limit of detection for the LSC analysis was defined to be twice the disintegrations per minute of control samples, which translated to 0.006 mg eq./kg. Approximately 30% of the TRR in liver and approximately 50% of the TRR in kidney was extracted. Further treatments of the PES quantitatively released the unextracted residues remaining from the solvent extraction.

Five HPLC systems were used to analyse samples. All were reverse-phase systems using UV and radioactive flow detectors. The systems differed in the mobile phase gradients that were used and the specific column (four C-18, one phenyl).

Approximately 90% of the administered dose was excreted and < 0.1% was accounted for in tissues (liver and kidney; Table 17). Residues identified in excreta were cyazofamid, CCBA, CCIM, CCTS, CHCN, and unidentified conjugates of CHCN. Total radioactive residues were < 0.006 mg eq./kg in all samples of eggs, muscle, blood, fat, and skin; as such, residue plateau in eggs could not be assessed. In kidney (

Table 18), the only major residues for both label positions were CHCN conjugates (15% TRR, 0.0044-0.0086 mg eq./kg), CCBA (12% TRR, 0.0035-0.0064 mg eq./kg), and unextracted residues (56% TRR, 0.017–0.031 mg eq./kg). In liver (Table 19), the only major residues were unextracted residues (75% TRR, 0.033–0.066 mg eq./kg). Further workup of the post-extraction solids in kidney and liver released the entire unextracted residue (104 and 109%, respectively) from the Bz label and nearly all from the Im label (95 and 87%, respectively). The majority of the residue was extracted with the 1 M HCl, protease, and amylase treatments (Table 20). In the 1 M HCl hydrolysate of kidney and liver PES, the major identified residues (Table 21) were CHCN conjugate (30-67%TRR) and CCBA (14% TRR; liver from Im label only). Residues of all fractions in the hydrolysate were ≤ 0.01 mg eq./kg and most were < 0.001 mg eq./kg.

Table 17 Total radioactive residues (TRRs) in tissues and excreta of hens following exposure equivalent to 10 ppm in the diet

Matrix	[Im-^{14}C]		[Bz-^{14}C]	
	mg eq./kg	% of dose	mg eq./kg	% of dose
Eggs and Tissues				
Egg	< 0.006	--	< 0.006	--
Fat	< 0.006	--	< 0.006	--
Kidney	0.058	0.01	0.029	0.01
Liver	0.088	0.05	0.044	0.03
Muscle (breast)	< 0.006	--	< 0.006	--
Muscle (thigh)	< 0.006	--	< 0.006	--
Skin	< 0.006	--	< 0.006	--
Blood and Excreta				
Blood	< 0.006	--	< 0.006	--
Cage wash	2.37	1.92	1.09	1.31
Excreta	51.2	90.3	41.1	84.9

Table 18 Characterization of radioactive residues in kidney

	Kidney			
	[Im-^{14}C]		[Bz-^{14}C]	
Fraction	mg eq/kg	%TRR	mg eq/kg	%TRR
TRR	0.0288	100	0.0578	100
Cyazofamid	0.0001	0.4	0.0002	0.3
CCBA	0.0035	12.3	0.0064	11.1
CCIM	0.0002	0.8	0.0005	0.8
CCTS	0.0002	0.6	0.0003	0.6
CHCN	0.0004	1.2	0.0009	1.6
CHCN Conjugates [a]	0.0049	17.2	0.0096	16.8
CM-2	0.0002	0.6	0.0003	0.4
CM-3	0.0002	0.6	0.0003	0.6
CM-6	0.0003	1.0	0.0005	0.8
CM-7	0.0007	2.3	0.0005	0.9
CM-10	0.0007	2.4	0.0012	2.1
CM-11	0.0004	1.3	0.0006	1.0
CM-12	0.0002	0.6	0.0003	0.6
Solvent Extractable	0.0120	41.3	0.0216	37.6
PES	0.0171	59.4	0.0312	54.0

[a] Combination of fractions CM-1, CM-4, and CM-5

Table 19 Characterization of radioactive residues in liver

	Liver			
	[Im-^{14}C]		[Bz-^{14}C]	
Fraction	mg eq/kg	%TRR	mg eq/kg	%TRR
TRR	0.088	100	0.044	100
Cyazofamid	< 0.006	--	0.0004	0.4
CCBA	0.0002	0.5	0.0015	1.7
CCIM	0.0002	0.4	0.0013	1.5
CCTS	0.0002	0.4	0.0007	0.8
CHCN	0.0001	0.3	0.0013	1.5
CHCN Conjugates [a]	0.0018	4.1	0.0111	12.5
CM-2	0.0002	0.5	0.0002	0.3
CM-3	0.0001	0.2	0.0002	0.3
CM-6	0.0001	0.3	0.0006	0.7
CM-7	0.0001	0.2	0.0004	0.4
CM-10	0.0008	1.8	0.0007	0.8
CM-11	0.0010	2.2	0.0009	1.1
CM-12	0.0005	1.1	0.0003	0.3
Solvent Extractable	0.0053	12.0	0.0192	21.9
PES	0.0327	74.5	0.0660	75.2

[a] Combination of fractions CM-1, CM-4, and CM-5.

Table 20 Exhaustive extraction of kidney and liver post-extraction solids

	[Im-^{14}C]			[Bz-^{14}C]		
Fraction	mg eq./kg	%TRR	% unextracted residue	mg eq./kg	%TRR	% unextracted residue
Kidney						
PES	0.0312	54.0	--	0.0171	59.5	--
Acid (1 M HCl)	0.0150	26.7	49.4	0.0086	30.0	50.5
Protease	0.0060	9.8	18.2	0.0032	11.0	18.6
Amylase	0.0060	9.5	17.6	0.0043	14.8	24.9
Collagenase	0.0010	1.7	3.2	0.0004	1.5	2.5
Acid (6 M HCl)	0.0010	1.0	1.9	0.0003	0.9	1.6
Base (1 M NaOH)	0.0020	2.8	5.1	0.0011	3.8	6.3

Fraction	[Im-^{14}C]			[Bz-^{14}C]		
	mg eq./kg	%TRR	% unextracted residue	mg eq./kg	%TRR	% unextracted residue
Total	0.0310	51.5	95.4	0.079	62.0	104.4
Liver						
PES	0.0660	75.2	--	0.0327	74.6	--
Acid (1 M HCl)	0.0156	17.7	23.6	0.0078	17.8	23.8
Protease	0.0017	1.9	2.5	0.0108	24.7	33.1
Amylase	0.0116	13.2	17.6	0.0130	29.6	39.7
Collagenase	0.0072	8.2	10.9	0.0012	2.8	3.7
Acid (6 M HCl)	0.0026	2.9	3.9	0.0004	0.9	1.2
Base (1 M NaOH)	0.0185	21.1	28.1	00.026	5.8	7.8
Total	0.0572	65.0	86.6	0.0592	81.6	109.3

Table 21 Distribution of radiolabelled residues in the acid hydrolysate from hen liver/kidney post-extraction solids

Fraction	mg eq./kg			% TRR		
	Liver [Im-^{14}C]	Kidney [Bz-^{14}C]	Kidney [Im-^{14}C]	Liver [Im-^{14}C]	Kidney [Bz-^{14}C]	Kidney [Im-^{14}C]
TRR	0.0156	0.0086	0.015	100.0	100.0	100.0
Cyazofamid	0.0001	0.0001	0.0001	0.6	1.2	0.7
CHCN Conjugate	0.0073	0.0026	0.0101	46.8	30.2	67.3
CHCN	0.0001	0.0001	0.0001	0.6	1.2	0.7
CCBA	0.0022	0.0002	0.0003	14.1	2.3	2.0
CCTS	0.0002	0	0.0001	1.3	0.0	0.7
CCIM	0.0006	0	0.0001	3.8	0.0	0.7
CM-2	0.002	0.001	0.0001	12.8	11.6	0.7
CM-3	0.002	0.0016	0.0012	12.8	18.6	8.0
CM-6	0.0001	0.0003	0.0002	0.6	3.5	1.3
CM-7	0.0001	0.0001	0.0001	0.6	1.2	0.7
CM-10	0.0004	0.0004	0.0008	2.6	4.7	5.3
CM-11	0.0003	0.0002	0.0004	1.9	2.3	2.7
CM-12	0.0001	0	0.0001	0.6	0.0	0.7

In summary, the poultry metabolism study shows essentially no transfer of cyazofamid residues into poultry eggs, meat, fat, and skin, and only very little transfer of residues into poultry offal. CCBA and conjugates of CHCN were the only identified residues occurring at greater than 10% TRR in any matrix; even so, the absolute levels of these metabolites were low. The proposed metabolic pathway of cyazofamid in laying hens is portrayed in Figure 5.

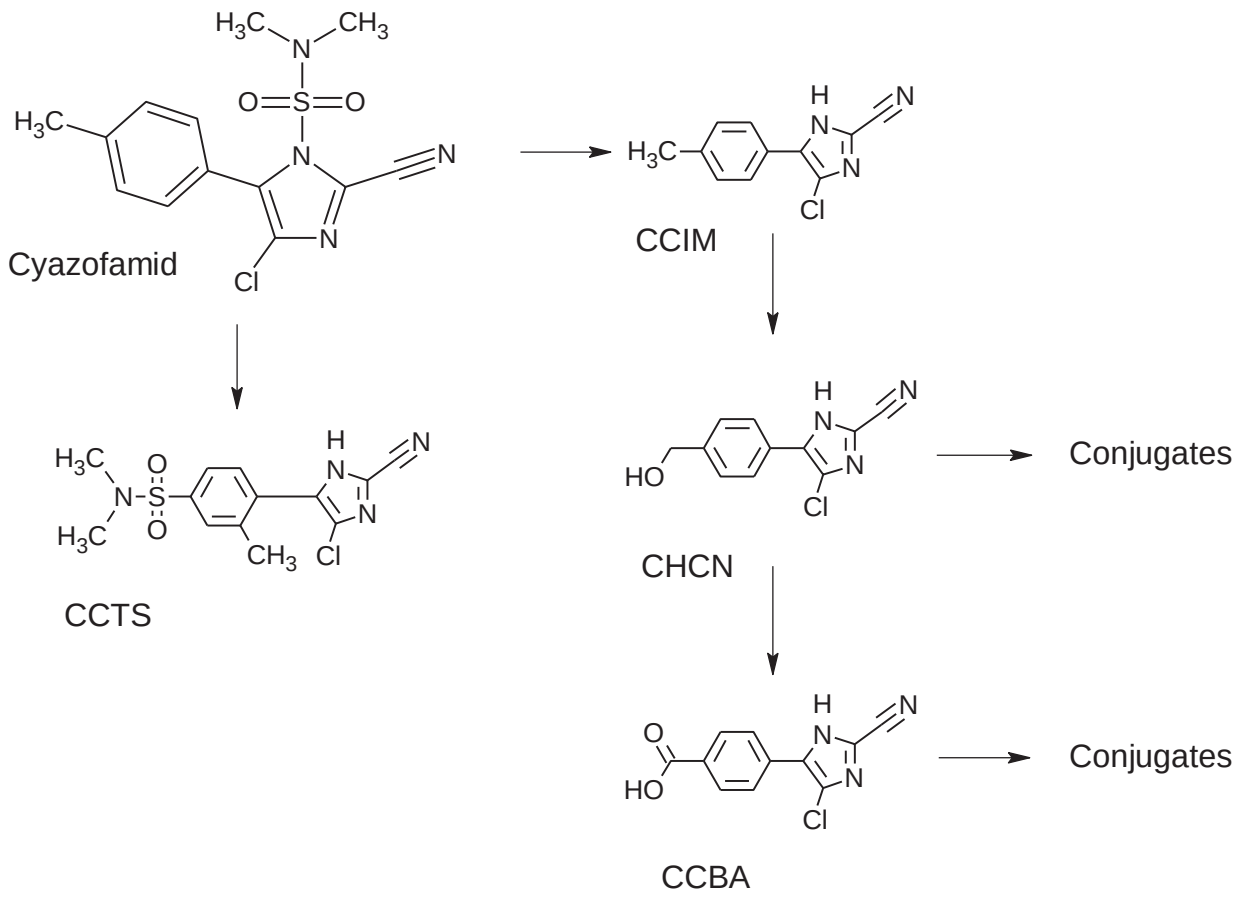


Figure 5 Proposed metabolic pathway of cyazofamid in laying hens

Environmental fate

The Meeting received studies for cyazofamid depicting the aqueous hydrolysis, aqueous and soil photolysis, aerobic soil metabolism, and a confined rotational crop study with carrot, lettuce, and wheat.

Hydrolysis

Hydrolysis of cyazofamid was investigated by I. S. Hendrix and T.R. Neal (1997, RA-4003; see Table 1).

The test material was hydrolysed, with half-lives ranging from 10.6 days to 13.3 days at 25 °C and from 0.39 to 0.55 days at 50 °C.

Hydrolysis of the cyazofamid metabolites CCIM, CCIM-AM, and CTCA was investigated in a separate study by T. Repko (1999, RA-4205). Each of the three compounds, radiolabelled in the benzene ring, was dissolved in sterile buffered solutions (pH 4, 7, and 9) with acetonitrile (< 1%) as a co-solvent. The solutions were maintained in darkness at 50±0.1 °C and sampled after 0 and 5 days. Hydrolysis of each test substance was minimal, as summarized in Table 22.

Table 22 Hydrolysis of cyazofamid metabolites at 50 °C

	Concentration of test material (mg/L)					
	CCIM		CCIM-AM		CTCA	
pH	Day 0	Day 5	Day 0	Day 5	Day 0	Day 5
4	0.062	0.062	0.067	0.067	0.062	0.057
7	0.062	0.061	0.067	0.066	0.062	0.060

	Concentration of test material (mg/L)					
	CCIM		CCIM-AM		CTCA	
pH	Day 0	Day 5	Day 0	Day 5	Day 0	Day 5
9	0.092	0.060	0.067	0.064	0.062	0.060

Photolysis

Photolysis of cyazofamid in **aqueous buffer** was investigated by Hendrix (1999, Report RA-4013; see Table 1). Cyazofamid degraded very rapidly, with a half-life of approximately 30 minutes. Cyazofamid also dissipated in the dark-control samples, with recovery falling to 21% by Day 26.

Photolysis of cyazofamid on the surface of a loamy sand **soil** was investigated by Shelby (1999, Report RA-4018). Cyazofamid, radiolabelled in either the benzene (Bz) ring or the imidazole (Im) ring, was applied to soil and exposed to simulated sunlight for 30 days (12-hr light/dark cycle). At intervals throughout the study, samples were extracted with ACN and water and analysed by radio-HPLC. Reference standards included in the study design were cyazofamid, CCIM, CCIM-AM, CHCN, CHCA, CCBA, CTCA, CTI, and CCIS

Mass balance recovery in the study was acceptable, ranging from 75 to 102% (Table 23). The proportion of unextracted residues increased during the 30-day exposure period. In the study, CCIM was formed from parent cyazofamid. The CCIM was then reduced to CCIM-AM and then oxidized to CCBA. CCBA underwent further degradation to unidentified products. As with the aqueous photolysis study, residues of cyazofamid in dark-control samples declined during the exposure period. Calculated first-order $DT_{50}$and DT_{90} times for cyazofamid were similar between the light-irradiated samples and their dark-control counterparts. Although the initial degradation of cyazofamid is not significantly impacted by photolytic processes, time-course data indicate that photolysis does impact the amount of CCBA metabolite present in the samples (

Table 24). Formation of CO_2 was minimal (12% Im, 3% Bz).

Table 23 Percent of applied radioactivity from the soil photolysis study with cyazofamid

	Extracted		Unextracted		Total	
Time, days	Dark	Light	Dark	Light	Dark	Light
[Im-^{14}C]						
0	94	94	2	2	96	96
3	97	87	5	5	102	92
7	89	82	9	10	98	92
14	81	70	14	23	95	93
21	58	72	17	30	75	102
30	81	54	20	29	101	82
[Bz-^{14}C]						
0	94	94	2	2	96	96
3	87	93	4	8	91	101
7	84	83	11	14	95	97
14	77	84	20	13	97	97
21	70	93	15	27	85	90
30	76	59	22	37	98	96

Table 24 Percent TRR of residues in soil from the soil photolysis study

	Cyazofamid		CCIM		CCIM-AM		CCBA	
Time, days	Dark	Light	Dark	Light	Dark	Light	Dark	Light
[Im-^{14}C]								
0	91	91	3	3	0	0	0	0
3	67	53	25	24	2	3	0	0
7	42	33	40	33	3	5	1	1
14	19	19	12	34	3	4	45	2
21	8	12	18	16	3	2	19	28
30	8	5	12	19	2	3	54	13
[Bz-^{14}C]								

Time, days	Cyazofamid		CCIM		CCIM-AM		CCBA	
	Dark	Light	Dark	Light	Dark	Light	Dark	Light
0	90	90	3	3	0	0	0	0
3	61	57	22	25	2	2	0	1
7	39	27	36	34	1	4	0	3
14	16	29	11	39	2	4	45	3
21	17	17	17	24	3	4	31	9
30	9	5	15	19	2	3	44	17

Aerobic soil metabolism

Two studies depicting the metabolism and degradation kinetics of cyazofamid in aerobic soils were submitted. In the first, Hartman (1997, Report RA-4004) treated a loamy sand soil with benzene- (Bz) or imidazole- (Im) radiolabelled cyazofamid to a level of 0.1 mg/kg. After thorough mixing, the soils samples were placed into a metabolism apparatus (dark conditions, 20 °C) and analysed 0, 1, 3, 5, 10, 15, 20, 26, 30, 44, and 59 days after treatment. In the second study (Hartman, Korsch, and Lentz, 1999, Report RA-4012), a sandy loam soil (20 °C), a sandy soil (20 °C and 10 °C), and a loamy sand soil (20 °C) were treated in the same manner as the first study and incubated under aerobic conditions for 30 or 45 days (20 °C) or 110 days (10 °C), with samples taken intermittently for analysis.

In both studies, soil samples were extracted twice with ACN/H_2O (80/20, v/v), and the extracts were analysed for total radioactivity by LSC. At later sampling times (first study ≥ Day 20, second study ≥ Day 7), additional extractions were done with ACN/H_2O (50/50, v/v) followed by 0.1N NaCl. All extracts were analysed by HPLC. Radioactivity in the PES was determined by combustion/LSC. In addition, PES from the 10- and 59-Day samples in the first study were fractionated into their various organic matter constituentsand the radioactivity in those fractions was assayed combustion/LSC or direct LSC.

Mass balance from both studies was acceptable. Average recovery of radioactivity across extracted, unextracted, and CO2 fractions and across all soils, temperatures, and sampling times ranged from 96% to 100% of applied. Over the time course of the studies, unextracted residues increased from < 5% of applied material at Hour 0 to 35–64% at termination. Similarly, 14CO2 increased from 0% of applied radioactivity at the onset of the incubation period to 14% at study termination. Results from the first and second studies from incubations at 20 °C gave similar DT50 estimates of approximately 5 days. Estimated DT90 values were more diverse, ranging in the first study from 16 to 25 days and in the second study from 35 to 39 days. Dissipation times were longer at the 10 °C incubation temperature, averaging 16 days for DT50 and > 110 days for DT90. Degradates identified in the two studies were CCIM, CCIM-AM, and CTCA. All occurred at ≥ 10% of the applied radioactivity at some point in the study; as such, they would be ≥ 10% TRR and are considered to be major degradates. In terms of relative kinetics, parent compound appears to first degrade to CCIM, which peaks early in the temporal profile, followed by CCIM-AM. Degradate CTCA forms last; data are inconclusive of whether CTCA has peaked by study termination. Characterization of the PES from the first study showed association of radioactivity predominantly with fulvic acid and to a lesser extent humin and humic acid.

Confined rotational crop studies

The fate of cyazofamid as it relates to rotational crops was investigated by McFadden (1999, Report OR-4019). In that study, carrot, lettuce, and wheat were planted into a loamy sand soil that had been treated with cyazofamid, radiolabelled in either the benzene (Bz) or imidazole (Im) ring, at a rate of ca. 500 g/ha (100 g/ha×5 applications at 7-day interval). The crops were planted into the soil at 31, 120, and 360 days after treatment. At each plant-back interval (PBI), samples of immature and mature crop were collected.

Harvested samples were homogenized in the presence of dry ice and stored frozen (≤ 7 days) prior to analysis. Total radioactive residues in each sample were determined by combustion. Further analysis of the samples was determined by their TRR levels: <

0.01 mg eq./kg, no further analyses were attempted; between 0.01 and 0.05 mg eq./kg, samples underwent limited solvent extraction; and > 0.05 mg eq./kg, samples underwent a more exhaustive extraction. Limited extraction consisted of three extractions with ACN/0.1% formic acid (9/1, v/v) followed by partitioning against methylene chloride. Extracts and PES were analysed for radioactivity by LSC (combustion/LSC for PES). The aqueous fraction was neutralized with 0.2 M potassium carbonate, concentrated, and filtered prior to carbohydrate analysis by HPLC. The more exhaustive extraction was performed on the organic fraction by diluting it with 0.1 N formic acid to make a 1:4 solvent:formic acid solution, and then partitioning it twice with ethyl acetate and cleaned up by solid-phase extraction. All eluents from solid-phase extraction were assayed by LSC and ACN/H_2O eluates were analysed by HPLC. Residues in PES from wheat tissues were characterized by acid hydrolysis in addition to combustion analysis.

Total radioactive residues in the rotational crops are shown in Table 25. In all samples, residues resulting from the Im treatment were greater than those from the Bz treatment. Decreases in TRR were generally modest between the 31- and 120-day PBIs and then more pronounced between the 120- and 360-day intervals. Characterization of residues is based on residues in the 31- and 120-day PBI samples due to the low levels of radioactivity in the 36-day PBI samples.

Mass balance of radioactivity was generally adequate, with recovery of radioactivity from the aqueous fraction, the organic fraction, and PES, combined, ranging from 61 to 123% of the TRR. Low recoveries were associated with highly pigmented extracts and may be due to quenching during LSC rather than actual low recovery.

Table 25 Summary of TRRs in rotational crops at each plant-back interval following application of cyazofamid

Crop	Harvest (DAT)			TRR (mg eq./kg) [Im-^{14}C]			[Bz-^{14}C]		
PBI (DAT)	31	120	360	31	120	360	31	120	360
Carrot (immature)	94	202	460	0.059	0.025	0.011	0.017	0.020	0.005
Carrot (foliage)	150	228	511	0.074	0.045	0.018	0.019	0.019	0.005
Carrot (root)	150	228	511	0.018	0.010	0.003	0.009	0.006	0.002
Lettuce (immature)	86	145	448	0.037	0.022	0.004	0.016	0.009	0.002
Lettuce (mature)	108	179	479	0.015	0.008	0.006	0.005	0.004	0.007
Wheat (forage)	66	157	405	0.510	0.097	0.015	0.108	0.029	0.006
Wheat (chaff)	251	291	571	0.269	0.289	0.017	0.046	0.027	0.012
Wheat (straw)	251	291	571	0.498	0.209	0.031	0.126	0.085	0.015
Wheat (grain)	251	291	571	0.090	0.062	0.002	0.024	0.014	0.001

DAT = Days after last treatment.

Total radioactivity in extracts from lettuce (all PBIs), carrot (120- and 360-day PBIs), and wheat grain (all PBIs) was low, and samples were not further analysed to determine the nature of the residues. HPLC analysis of carrot tops (Im label only) from the 31-day PBI showed residues of CCBA (2.2% TRR), CCIM (10.4% TRR), CCIM-AM (39.5% TRR, 0.001 mg/kg), and cyazofamid (20.1% TRR, 0.003 mg/kg). Radioactivity in wheat forage and chaff was associated primarily with carbohydrates (0.01-0.195 mg eq./kg); levels of cyazofamid and metabolites were ≤ 0.003 mg eq./kg. Similar results occurred for wheat straw. The proposed metabolic pathway for cyazofamid in rotational crops was included previously in Figure 3.

RESIDUE ANALYSIS

Summary of analytical methods

Methods for the analysis of cyazofamid and CCIM used in the residue trials are generally the same, consisting of solvent extraction (usually acetonitrile) followed by partitioning and solid-phase extraction clean-up steps. Analysis of the residue was most frequently accomplished using LC-

MS/MS, although some studies used HPLC-UV or GC-NPD. The methods are summarized in Table 26. The LC-MS/MS and HPLC-UV methods underwent independent laboratory validation and appear to be suitable for enforcement purposes.

Table 26 Overview of the analytical methods submitted for cyazofamid and CCIM

Report ID	Matrix	Extraction	Clean-up	Separation/ Analysis/LOQ	Concurrent Recovery (%) (n) Mean ± Std. Dev.	
					Cyazofamid	CCIM
RA-3058, RA-3091	Grapes & processed commodities	Acetonitrile (2X)	Partitioning (hexane followed by aqueous sodium sulfate/methylene chloride)	HPLC-UV, C-18 column LOQ[a] = 0.01 mg/kg	(13) 92±20	(13) 105±25
RA-3067, RA-3090	Cucumber, squash, melon		Florisil® solid-phase extraction		(16)91±14 (16)86±12 (8)83±15	(16)94±21 (16)90±14 (8)102±19
RA-3065, RA-3077, RA-3089	Tomato				(14)98±15	(15)94±22
RA-3066, RA-3075, RA-3093	Potato & processed commodities				(22)79±15 (16)90±17	(25)92±19 (18)90±8
RA-3202A, RA-3203A, RA-3204A	Potato	Acetone	Partitioning (methylene chloride) Gel-permeation chromatography	GC-NPD LOQ[a] = 0.05 mg/kg	(16)96±4	--
RA-3082, RA-3083, RA-3084, RA-3085, RA-3086, RA-3095	Grapes	Acetonitrile/ acetone (8/2, v/v) (2X)	Partitioning (hexane followed by aqueous sodium sulfate/methylene chloride) Florisil® solid-phase extraction	HPLC-UV, C-18 column LOQ[a] = 0.01 mg/kg	(77)86±10	(75)90±9
RA-3123	Broccoli	Acetonitrile (2X)	Partitioning (hexane) Polymeric solid-phase extraction	LC-MS/MS, propyl column LOQ[a] = 0.01 mg/kg	(10)104±19	(10)98±23
RA-3124	Cabbage				(9)97±18	(9)91±19
RA-3096, RA-3199	Lettuce				(13)90±7	(13)88±9
RA-3125	Mustard greens				(10)95±21	(10)98±31
RA-3126	Spinach				(9)93±24	(9)91±27
RA-3195, RA-3198	Beans				(12)87±7	(12)88±5
RA-3107	Carrot				(26)83±8	(26)69±7
RA-3197	Basil				(17)88±8	(17)90±6
RA-3127	Hops	Acetonitrile	Partitioning (hexane) Extract split Cyazofamid: NH_2 SPE CCIM: Polymeric SPE	LC-MS/MS, propyl column LOQ[a] = 0.05 mg/kg	(20)82±17	(20)86±18
RA-3169, RA-3188, RA-3190 RA-1166	Hops	Acetonitrile/ acetone (8/2, v/v)	C-18 SPE	LC-MS/MS, C-18 column LOQ[a] = 0.01 mg/kg	(10)89±4	(10)92±3
RA-1166	Onion				(3)99±3	(3)99±4
RA-3101	Pepper	Acetonitrile/H_2O with 2% acetic acid (1/1, v/v)	Partitioning (methylene chloride) Florisil® solid-phase extraction	LC-MS/MS, C-18 column LOQ[a] = 0.01 mg/kg	(12)92±6	(12)95±4

[a] Defined as the lowest limit of method validation.

Plant materials

Methods used for the analysis of residues of cyazofamid and CCIM in plant materials in residue trials are all very similar (see Table 26). Extraction of homogenized sample is by a relatively polar solvent followed, in most cases, by partitioning of the residue into a non-polar solvent. Further clean-up is by solid-phase extraction using various sorbents. Most of the methods use either LC-MS/MS or HPLC-UV for separation and detection of the analytes. Method validation recoveries across all matrices and fortification levels (0.01–100 mg/kg) ranged from 63 to 128%, with a weighted average and relative standard deviation of 90±8% (Table 27).

Three methods underwent independent laboratory validation and were determined to be suitable for compliance purposes. In the first method, validated using bulb onion, lettuce, and green hops (Study RA-1177), cyazofamid and CCA are extracted by homogenizing the sample in 120 mL of acetonitrile/acetone (8/2, v/v), isolating the extract by vacuum filtration, and reducing the volume of the extract to 5 mL by rotary evaporation. Clean-up of the extract is by C-18 solid-phase extraction, and analysis of the residues is by LC-MS/MS on a C-18 column with an isocratic mobile phase consisting of acetonitrile (80%) and 0.2% acetic acid in water (20%). Mass transitions [$M+H^+$] of 325.1 m/z→108.0 m/z for cyazofamid and 218.3 m/z→183.2 m/z for CCIM are used for quantification. Confirmation of cyazofamid is made using the same ion transitions but with a cyano column on a gradient mobile phase. Confirmation of CCIM is based on a mass transition of 218.3 m/z→139.2 m/z. A confirmatory transition for cyazofamid is available (325.1 m/z→261.2 m/z).

In the second method, validated using barley grain and olive (Study RA-1177), cyazofamid and CCIM are extracted by shaking samples in 10 mL water followed by 10 mL acetonitrile (barley), or 10 mL acetonitrile only (olive). The extracts are then cleaned up using dispersive solid-phase extraction (onto magnesium sulfate, sodium chloride, sodium citrate dibasic sesquihydrate, and sodium citrate tribasic dihydrate). Analysis of the residues is the same as described in the first method.

In the third method, validated using tomato (Study RA-3062), cyazofamid and CCIM are extracted with acetonitrile. Co-extracted materials are then partitioned into hexane, which is discarded. Residues in the acetonitrile portion are then concentrated by rotary evaporation. A second partitioning is then done using sodium sulfate (2%) and methylene chloride. The methylene chloride phase is retained and evaporated to dryness. Residues of cyazofamid and CCIM are dissolved in ethyl ether and cleaned up by passing over a Florisil® column. After elution from the column, the ethyl ether is evaporated and the residues dissolved in acetonitrile/0.5% ascorbic acid in water (1/1, v/v) for analysis by HPLC-UV. Separation is achieved on a C18 column using a mobile phase of acetonitrile/0.5% ascorbic acid in water (1/1, v/v); detection is a 280 nm.

Table 27 Summary of analyte recoveries from method validations of methods for cyazofamid and CCIM

Report	Method Summary	Matrix	Analyte	Fortification, mg/kg	n	Recovery, % Mean ± Std. Dev.
RA-1177	Solvent: ACN:Acetone Cleanup: C18 (onion, lettuce, hops) Dispersive SPE (olive, barley) Analysis: LC-MS/MS	Onion	Cyazofamid	0.01-0.1	10	85 ± 15
			CCIM	0.01-0.1	10	86 ± 10
		Lettuce	Cyazofamid	0.01-0.1	10	70 ± 9
			CCIM	0.01-0.1	10	86 ± 10
		Olive	Cyazofamid	0.01-0.1	10	98 ± 8
			CCIM	0.01-0.1	10	94 ± 9
		Barley grain	Cyazofamid	0.01-0.1	10	94 ± 12
			CCIM	0.01-0.1	10	86 ± 20
		Hops (fresh)	Cyazofamid	0.01-0.1	10	104 ± 10
			CCIM	0.01-0.1	10	109 ± 14

Report	Method Summary	Matrix	Analyte	Fortification, mg/kg	n	Recovery, % Mean ± Std. Dev.
RA-3062	Solvent: ACN Cleanup: Hexane, $MeCl_2$, Florisil Analysis:HPLC-UV	Tomato	Cyazofamid	0.01-1.0	4	90.6 ± 8.2
			CCIM	0.01-1.0	4	87.9 ± 3.1
RA-1172	Solvent: ACN:Acetone Cleanup: C18 Analysis: LC-MS/MS	Onions	Cyazofamid	0.01-0.1	10	86 ± 2
			CCIM	0.01-0.1	10	91 ± 3
		Hops (fresh)	Cyazofamid	0.01-0.1	10	85 ± 3
			CCIM	0.01-0.1	10	88 ± 3
		Hops (dried cones)	Cyazofamid	0.01-0.1	10	99 ± 3
			CCIM	0.01-0.1	10	100 ± 7
RA-1101	Solvent: ACN Cleanup: Hexane, $MeCl_2$, Florisil Analysis:HPLC-UV	Grapes	Cyazofamid	0.01-0.6	14	83 ± 12
			CCIM	0.01-0.6	14	83 ± 12
		Potatoes	Cyazofamid	0.01-0.1	18	86 ± 8
			CCIM	0.01-0.1	18	82 ± 7
		Tomatoes	Cyazofamid	0.01-1.0	31	94 ± 14
			CCIM	0.01-1.0	31	103 ± 18
		Cucumber	Cyazofamid	0.01-0.1	23	92 ± 15
			CCIM	0.01-0.1	23	93 ± 18
		Cantaloupe	Cyazofamid	0.01-0.1	20	90 ± 15
			CCIM	0.01-0.1	20	92 ± 14
		Summer squash	Cyazofamid	0.01-0.1	15	85 ± 14
			CCIM	0.01-0.1	15	103 ± 15
		Potato (wet peel)	Cyazofamid	0.01-0.5	6	92 ± 7
			CCIM	0.01-0.5	6	106 ± 11
		Potato (flakes)	Cyazofamid	0.01-0.5	6	87 ± 10
			CCIM	0.01-0.5	6	89 ± 3
		Potato (chips)	Cyazofamid	0.01-0.5	6	92 ± 7
			CCIM	0.01-0.5	6	68 ± 4
		Tomato (paste)	Cyazofamid	0.01-0.5	7	86 ± 9
			CCIM	0.01-0.5	7	88 ± 6
		Tomato (puree)	Cyazofamid	0.01-0.2	6	89 ± 12
			CCIM	0.01-0.2	6	88 ± 4
		Raisins	Cyazofamid	0.01-0.5	6	67 ± 10
			CCIM	0.01-0.5	6	83 ± 11
		Grape (juice)	Cyazofamid	0.2	8	77 ± 5
			CCIM	0.2	8	80 ± 4
RA-3003	Solvent: ACN Cleanup: Hexane, $MeCl_2$, Florisil Analysis:HPLC-UV	Potato	Cyazofamid	0.01-1.0	6	97 ± 12
			CCIM	0.01-1.0	9	80 ± 13
		Tomato	Cyazofamid	0.01-1.0	6	84 ± 13
			CCIM	0.01-1.0	6	86 ± 5
		Grape	Cyazofamid	0.01-1.0	10	71 ± 9
			CCIM	0.01-1.0	6	74 ± 7
		Must	Cyazofamid	0.01-1.0	6	97 ± 18
			CCIM	0.01-1.0	6	90 ± 3
		Wine	Cyazofamid	0.01-1.0	9	78 ± 9
			CCIM	0.01-1.0	4	81 ± 1

Stability of residues in stored samples

The stability of cyazofamid and CCIM in frozen storage has been investigated in bean, grape (homogenized and unhomogenized; cyazofamid only), oilseed rape, potato, and tomato. For all matrices except grape, samples were spiked, separately, with cyazofamid and CCIM. Samples were placed into frozen storage and analysed after varying durations in frozen storage to determine the amounts of analyte remaining in the sample. For grape, a large sample of was collected from a field trial location and split into two subsamples. One subsample was homogenized and the other was maintained as whole, unhomogenized grapes. Both subsamples were placed into stored frozen.

Incurred residues of cyazofamid were analysed at various storage durations to determine the amount of compound remaining.

Residues of both analytes were stable (≥ 70% remaining) for at least 400 days in beans and oilseed rape, and for up to 181 days in potato. In tomato, cyazofamid was stable for up to 365 days and CCIM was stable for at least 1093 days. In grape, residues of cyazofamid appeared to be more stable in unhomogenized matrix, generally showing > 70% remaining for the 365-day duration of the study versus homogenized matrix, in which the percent remaining was generally <70% at sampling times greater than 8 days. Interpretation of the cyazofamid stability data in grape is complicated by the experimental design and the variability in residue levels, especially for the unhomogenized grape subsample.

In addition to the specific storage stability studies summarized above, a storage stability component was included in the experimental designs of studies conducted by IR-4. The storage stability data from these studies do not include analysis of residues at 0 days. If fortifications were made correctly, the data indicate that under frozen storage conditions, cyazofamid and CCIM are stable for at least 860 days in cabbage; for at least 634 days in lettuce; for at least 977 days in mustard greens; for at least 949 days in spinach; at least 887 days in bean pods with seeds, at least 889 days in bean plants with pods, and at least 140 days in ben seeds without pods; and at least 509 days in hops cones. Cyazofamid was stable for at least 284 days in fresh basil and 297 days in dried basil; however, CCIM was not stable in either commodity (47% remaining in fresh basil and 59% remaining in dried basil). Neither cyazofamid nor CCIM were shown to be stable in carrot, with 58% cyazofamid and 38% CCIM remaining after 374 days in storage.

Table 28 Storage Stability of cyazofamid and CCIM in dry beans (Report RA-3171)

Analyte	Fortification, mg/kg	Storage Time, days	n	Avg. Conc., mg/kg	Avg. % Remaining	Std. Dev.	Concurrent Recovery
Cyazofamid	0.1	1	3	0.10	100	0	100
		29	2	0.090	90	0	92
		95	2	0.090	90	0	94
		209	2	0.075	75	7	84
		400	2	0.095	95	7	88
CCIM	0.1	1	3	0.090	90	0	92
		29	2	0.090	90	0	91
		95	2	0.090	90	0	94
		209	2	0.085	85	7	87
		400	2	0.10	100	0	90

Table 29 Storage Stability of cyazofamid in grape berries (Report RA-3088)

Matrix State	Fortification, mg/kg [a]	Storage Time, days	n	Avg. Conc., mg/kg	Avg. % Remaining	Std. Dev.	Concurrent Recovery
Homogenized	0.74	0	3	0.74	100	5	93
		8	3	0.73	99	14	100
		15	3	0.47	63	4	87
		28	3	0.39	53	6	89
		64	3	0.56	76	9	85
		125	3	0.51	69	8	97
		244	3	0.47	63	6	80
		365	3	0.49	66	9	82
Unhomogenized	0.70	0	3	0.70	100	54	90
		8	3	0.59	84	25	97
		15	3	0.50	71	6	93
		28	3	0.58	83	24	86
		64	3	0.68	97	19	84
		125	3	0.63	90	7	94
		244	3	0.81	120	17	83
		365	3	0.76	110	29	94

[a] Samples were not fortified. The value specified is the average concentration from the samples at the 0-Day sampling.

Table 30 Storage Stability of cyazofamid and CCIM in oilseed rape seed (Report RA-3171)

Analyte	Fortification, mg/kg	Storage Time, days	n	Avg. Conc., mg/kg	Avg. % Remaining	Std. Dev.	Concurrent Recovery
Cyazofamid	0.1	1	3	0.097	97	6	100
		29	2	0.090	90	0	92
		95	2	0.090	90	0	91
		209	2	0.090	90	0	92
		400	2	0.085	85	7	96
CCIM	0.1	1	3	0.090	90	0	96
		29	2	0.085	85	7	88
		95	2	0.090	90	0	92
		209	2	0.090	90	0	94
		400	2	0.090	90	0	93

Table 31 Storage Stability of cyazofamid and CCIM in potato tuber (Report RA-3064)

Analyte	Fortification, mg/kg	Storage Time, days	n	Avg. Conc., mg/kg	Avg. % Remaining	Std. Dev.	Concurrent Recovery
Cyazofamid	0.5	0	4	0.54	110	10	110
		1	4	0.49	98	4	100
		3	4	0.46	92	4	92
		7	4	0.56	110	18	120
		14	4	0.44	88	9	88
		29	4	0.43	86	4	92
		91	4	0.44	88	2	98
		181	4	0.37	74	4	84
		367	4	0.32	64	9	100
		793	4	0.29	58	3	90
		1099	4	0.30	60	13	94
CCIM	0.5	0	4	0.49	98	5	88
		1	4	0.41	82	4	96
		14	4	0.44	88	2	100
		29	4	0.38	76	7	92
		104	4	0.33	66	4	80
		181	4	0.46	92	5	92
		469	4	0.31	62	12	82
		784	4	0.26	52	6	74
		1091	4	0.31	62	9	110

Table 32 Storage Stability of cyazofamid and CCIM in tomato fruit (Report RA-3063)

Analyte	Fortification, mg/kg	Storage Time, days	n	Avg. Conc., mg/kg	Avg. % Remaining	Std. Dev.	Concurrent Recovery
Cyazofamid	0.5	0	4	0.49	98	15	96
		1	4	0.46	92	10	90
		7	4	0.42	84	7	90
		14	4	0.45	90	7	88
		29	4	0.45	90	2	100
		91	4	0.45	90	3	86
		179	4	0.38	76	15	98
		365	4	0.45	90	5	110
		798	4	0.33	66	8	84
		1099	4	0.31	62	7	76
CCIM	0.5	0	4	0.51	100	9	100
		1	4	0.48	96	10	100
		29	4	0.39	78	4	78

Analyte	Fortification, mg/kg	Storage Time, days	n	Avg. Conc., mg/kg	Avg. % Remaining	Std. Dev.	Concurrent Recovery
		90	4	0.42	84	5	78
		180	4	0.50	100	6	88
		467	4	0.43	86	5	96
		788	4	0.38	76	1	88
		1093	4	0.38	76	5	110

Table 33 Storage Stability of cyazofamid and CCIM in IR-4 studies

Crop	Analyte	Storage Time, days	n	Avg. % of Nominal Remaining	Std. Dev.	Concurrent Recovery	Reference
Cabbage	Cyazofamid	860	3	112	4	115	RA-3124
	CCIM	860	3	100	1	100	
Lettuce	Cyazofamid	634	3	72	13	83	RA-3196
	CCIM	634	3	78	12	80	
Mustard greens	Cyazofamid	977	3	112	0	115	RA-3125
	CCIM	977	3	108	6	117	
Spinach	Cyazofamid	949	3	102	2	120	RA-3126
	CCIM	949	3	118	5	117	
Beans (plants with pods)	Cyazofamid	889	3	80	5	94	RA-3198
	CCIM	889	3	76	5	88	
Beans (pods with seeds)	Cyazofamid	887	3	78	15	86	
	CCIM	887	3	88	12	88	
Beans (seeds without pods)	Cyazofamid	140	3	80	4	85	
	CCIM	140	3	76	2	93	
Carrot	Cyazofamid	374	3	58	5	75	RA-3107
	CCIM	374	3	38	2	91	
Basil (fresh)	Cyazofamid	284	3	80	5	83	RA-3197
	CCIM	284	3	47	1	88	
Basil (dried)	Cyazofamid	297	3	78	1	88	
	CCIM	297	3	59	1	82	
Hops (dry cones)	Cyazofamid	509	3	86	7	79	RA-3127
	CCIM	509	3	78	2	87	

USE PATTERN

Table 34 Good agricultural practices (GAPs) authorized for cyazofamid

Crop	Country	Application method(s)	Growth stage	Rate, kg (max), ai/ha	No.	Retreatment interval (min), days	PHI, days
Grape	Germany	Broadcast spray (incl. chemigation)	BBCH 15-61	0.025	8	12-14	21
			BBCH 61-71	0.05			
			BBCH 71-75	0.075			
			BBCH 75-85	0.1			
	USA[a]	Broadcast spray (incl. chemigation)	n.s.	0.08	6	10-14	30
Brassica (cole) leafy vegetables [Crop Group 5][h]	USA[c]	Transplant soil drench	n.s.	0.753	6	7-10	0
		soil incorporation		0.58			
		broadcast spray (incl. chemigation)		0.08			
Cucurbit vegetables [Crop Group 9][i]	USA[a]	Broadcast spray (incl. chemigation)	n.s.	0.08	6	7-10	0
Fruiting vegetables [Crop Group 8-10][j]	USA[a]	Broadcast spray (incl. chemigation)	n.s.	0.08	6	7-10	0

Crop	Country	Application method(s)	Growth stage	Rate, kg (max), ai/ha	No.	Retreatment interval (min), days	PHI, days
Tomato (glasshouse)	USA	Transplant soil drench	At planting and up to 1 week before transplanting	0.01 kg ai/hL	1	--	--
Leafy greens [Crop Subgroup 4A] [k]	USA[a]	Broadcast spray (incl. chemigation)	n.s.	0.08	6	7-10	0
Lettuce	Canada	Broadcast spray (incl. chemigation)	n.s.	0.08	6	7-14	0
Mustard greens	USA[c]	Transplant soil drench	n.s.	0.753	6	7-10	0
		soil incorporation		0.58			
		broadcast spray (incl. chemigation)		0.08			
Beans (succulent podded and succulent shelled)	USA[d]	Broadcast spray (incl. chemigation)	n.s.	0.08	6	7-14	0
Carrot	USA[e]	Broadcast spray (incl. chemigation)	n.s.	0.175	5	14-21	14
Potato	Brazil	Broadcast spray (incl. chemigation)	n.s.	0.10	6	7-10	7
	Canada[f]	Broadcast spray (incl. chemigation)	n.s.	0.08	6	7	7
Tuberous and corm vegetables [Crop Subgroup 1C] [l]	USA[g]	In-furrow	In-furrow application at planting	0.178	10	7-10	7
		broadcast spray (incl. chemigation)	Lay-by/hilling	0.08			
Basil	USA[b]	Broadcast spray (incl. chemigation)	n.s.	0.088	9	7-10	0
Hops	USA[a]	Broadcast spray (incl. chemigation)	n.s.	0.08	6	7-10	3

[a] Do not apply more than 480 g ai/ha/season

[b] Do not apply more than 790 g ai/ha/season. Can be applied to basil grown in a glasshouse

[c] Make a single soil application followed by 5 foliar applications. Do not apply more than 1.15 kg ai/ha/season

[d] Do not apply more than 480 g ai/ha/season. Do not apply to cowpeas used for livestock feed

[e] Do not apply more than 877 g ai/ha/season

[f] Last 2 applications to be made at maximum rate, plant-back interval 30 days

[g] Do not apply more than 800 g ai/ha/season; Last 2-3 applications to be made at maximum rate

[h] Crop Group 5 = Broccoli; broccoli, Chinese (gai lon); broccoli raab (rapini); Brussels sprouts; cabbage; cabbage, Chinese (bok choy); cabbage, Chinese (napa); cabbage, Chinese mustard (gai choy); cauliflower; cavalo broccolo; collards; kale; kohlrabi; mizuna; mustard greens; mustard spinach; and rape greens

[I] Crop Group 9 = Chayote (fruit) ; Chinese waxgourd (Chinese preserving melon) ; citron melon ; cucumber ; gherkin ; gourd, edible (includes hyotan, cucuzza, hechima, Chinese okra); Momordica spp. (includes balsam apple, balsam pear, bitter melon, Chinese cucumber); muskmelon (hybrids and/or cultivars of Cucumis melo; includes true cantaloupe, cantaloupe, casaba, crenshaw melon, golden pershaw melon, honeydew melon, honey balls, mango melon, Persian melon, pineapple melon, Santa Claus melon, and snake melon); pumpkin ; squash, summer (includes crookneck squash, scallop squash, straightneck squash, vegetable marrow, zucchini); squash, winter (includes butternut squash, calabaza, hubbard squash, acorn squash, spaghetti squash); and watermelon

[j] Crop Group 8-10 = African eggplant; bush tomato; cocona; currant tomato; eggplant; garden huckleberry; goji berry; groundcherry; martynia; naranjilla; okra; pea eggplant; pepino; pepper, bell; pepper, nonbell; roselle; scarlet eggplant; sunberry; tomatillo; tomato; tree tomato

[k] Crop Subgroup 4A = Amaranth; arugula; chervil; chrysanthemum, edible-leaved; chrysanthemum, garland; corn salad; cress, garden; cress, upland; dandelion; dock; endive; lettuce; orach; parsley; purslane, garden; purslane, winter; radicchio (red chicory); spinach; spinach, New Zealand; spinach, vine

[l] Crop Subgroup 1C = Arracacha; arrowroot; artichoke, Chinese; artichoke, Jerusalem; canna, edible; cassava, bitter and sweet; chayote (root); chufa; dasheen; ginger; leren; potato; sweet potato; tanier; turmeric; yam bean; yam, true

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received data from supervised residue trials conducted on grape, basil, hops, broccoli, cabbage, cucumber, summer squash, muskmelon, peppers, tomato, head and leaf lettuce, mustard greens, spinach, snap bean, lima bean, carrot, and potato. In all trials, a soluble concentrate (SC) formulation was applied as a tank mixture prepared uniquely for that trial site. Trials were conducted in the USA for all crops. In addition, trials on grapes were conducted in Argentina, Mexico, Northern Europe, and Southern Europe; trials on lettuces were conducted in Canada; trials on potato were conducted in Canada and Brazil; and trials on hops were conducted in Germany and the USA. Trials on basil included field-grown and glasshouse-grown crops.

The field trial 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 the methods described above. The results are supported by concurrent recoveries ranging, across all commodities, 77–95% ± 4–21% (mean ± RSD) for cyazofamid and 86–101% ± 5–19% for CCIM. The maximum durations for samples in frozen storage were:

- Grape = 295 days,
- Broccoli = 773 days,
- Cabbage = 860 days,
- Cucumber = 552 days,
- Summer squash = 535 days,
- Muskmelon = 278 days,
- Peppers = 272 days (bell) and 268 days (non-bell),
- Tomato = 455 days,
- Lettuce = 634 days (head) and 624 days (leaf),
- Mustard greens = 965 days,
- Spinach = 927 days,
- Snap bean = 945 days,
- Lima bean = 147 days,
- Carrot = 443 days,
- Potato = 535 days,
- Basil = 284 days (fresh) and 297 days (dried), and
- Hops = 552 days.

Except for carrot (cyazofamid and CCIM) and basil (CCIM only), the storage durations are less than or equal to those for which residues have been demonstrated to be stable. Unless otherwise noted in the tables below, harvested commodities were maintained whole in the field and not cut or homogenized until they reached the analytical laboratory.

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. In the summary tables, values used for making maximum residue level recommendations are underlined, values used for dietary intake estimates are italicized, and highest individual values for estimating dietary intake are bolded. Trial locations that appear to be dependent are grouped by a heavy cell border in the tables (e.g., Table 36).

Supervised trials for cyazofamid:

Category	Crop	Table
Berries and other small fruits	Grape (FB 0269)	35
Brassica (cole or cabbage) vegetables, head cabbage, flowerhead Brassicas	Broccoli (VB 0400)	36
	Cabbage (VB 4175)	37
Fruiting vegetables, cucurbits	Cucumber (VC 0424)	38
	Summer squash (VC 0431)	39
	Muskmelon (VC 4239)	40
Fruiting vegetables, other than cucurbits	Peppers (VO 0051)	41
	Tomato (VO 0448)	42
Leafy vegetables (including Brassica leafy vegetables)	Lettuce, head/leaf (VL 0482/VL0483)	43
	Mustard greens (VL 0485)	44
	Spinach (VL 0502)	45
Legume vegetables	Lima bean, young pods and/or immature beans(VP 0534)	46
	Snap bean, young pods (VP 4453)	47
Root and tuber vegetables	Carrot (VR 0577)	48
	Potato (VR 0589)	49
Herbs	Basil (HH 0722)	50
Dried herbs	Hops, dry (DH 1100)	51

Table 35 Residues of cyazofamid and CCIM in **grape** following foliar application.

Location (Year) Country *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
GAP: Germany	8×(100)	12-14	21	--	--	--	--
GAP: USA	6×(80)	10-14	30	--	--	--	--
Kerman, CA (1999) USA *Thompson Seedless* 010222-C	8×(~100)	11-14	0	0.29, 0.31 (0.30)	0.010, 0.020 (0.015)	0.30, 0.34 (0.32)	RA-3058
			7	0.44, 0.42 (0.43)	0.030, 0.020 (0.025)	0.48, 0.45 (0.47)	
			14	0.36, 0.34 (0.35)	0.020, 0.020 (0.020)	0.39, 0.37 (0.38)	
			21	0.32, 0.16 (0.24)	0.020, 0.010 (0.015)	0.35, 0.17 (0.26)	
			28	0.16, 0.30 (0.23)	0.010, 0.020 (0.015)	0.17, 0.33 (0.25)	
Fresno, CA (1999) USA *Thompson Seedless* 010222-D	8×(~100)	10-14	21	0.080, 0.070 (0.075)	0.010, 0.010 (0.010)	0.095, 0.085 (0.090)	
Madera, CA (1999) USA *Thompson Seedless* 010222-E	8×(~100)	10-14	21	0.19, 0.16 (0.18)	0.010, 0.010 (0.010)	0.20, 0.17 (0.19)	
St. Gilles, Languedoc (1999) France *Carignan* PRE 99081 A06	8×(100)	11-13	0	0.12, 0.12 (0.12)	< 0.01, < 0.01 (< 0.010)	< 0.13, < 0.13 (< 0.13)	RA-3082
			7	0.10, 0.12 (0.11)	< 0.01, < 0.01 (< 0.010)	< 0.11, < 0.13 (< 0.12)	

Location (Year) Country *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
			14	0.020, 0.030 (0.025)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.045 (< 0.040)	
			21	0.040, 0.040 (0.040)	< 0.01, < 0.01 (< 0.010)	< 0.055, < 0.055 (*< 0.055*)	
			28	0.010, 0.010 (0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
Rudesheim (1999) Germany *Riesling* 99/025-0	8×(100)	11-14	0	0.14, 0.13 (0.14)	< 0.01, < 0.01 (< 0.010)	< 0.15, < 0.14 (< 0.15)	RA-3083
			7	0.070, 0.080 (0.075)	< 0.01, < 0.01 (< 0.010)	< 0.085, < 0.095 (< 0.090)	
			14	0.040, 0.050 (0.045)	< 0.01, < 0.01 (< 0.010)	< 0.055, < 0.065 (< 0.060)	
			21	0.030, 0.040 (0.035)	< 0.01, < 0.01 (< 0.010)	< 0.045, < 0.055 (*< 0.050*)	
			28	0.020, 0.020 (0.020)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.035 (< 0.035)	
Rudesheim (1999) Germany *Riesling* 99/026-0	8×(100)	11-14	21	0.030, 0.040 (0.035)	< 0.01, < 0.01 (< 0.010)	< 0.045, < 0.055 (< 0.050)	
Nogent L'Abbesse, Champagne-Ardenne (1999) France *Chardonnay* EA990162FR01	9×(100)	11-13	0	0.22, 0.27 (0.25)	< 0.01, < 0.01 (< 0.010)	< 0.23, < 0.28 (< 0.26)	RA-3086
			7	0.11, 0.13 (0.12)	< 0.01, < 0.01 (< 0.010)	< 0.12, < 0.14 (< 0.13)	
			14	0.090, 0.11 (0.10)	< 0.01, < 0.01 (< 0.010)	< 0.10, < 0.12 (< 0.11)	
			21	0.090, 0.090 (0.090)	< 0.01, < 0.01 (< 0.010)	< 0.10, < 0.10 (*< 0.10*)	
			28	0.060, 0.050 (0.055)	< 0.01, < 0.01 (< 0.010)	< 0.075, < 0.065 (< 0.070)	
Fumane, Verona (1999) Italy *Rondinella* EA990162IT01	8×(100)	11-13	0	0.69, 0.78 (0.74)	0.010, 0.010 (0.010)	0.70, 0.79 (0.75)	
			7	0.71, 0.93 (0.82)	< 0.01, < 0.01 (< 0.010)	< 0.72, < 0.94 (< 0.83)	
			14	0.41, 0.41 (0.41)	0.010, 0.010 (0.010)	0.42, 0.42 (0.42)	
			22	0.69, 0.62 (0.66)	0.010, 0.010 (0.010)	**0.70**, 0.63 (*0.67*)	
			28	0.43, 0.51 (0.47)	< 0.01, < 0.01 (< 0.010)	< 0.44, < 0.52 (< 0.48)	
San Maria della Versa (1999) Italy *Barbera* EA990162IT02	9×(100)	11-14	21	0.02, 0.03, 0.03, 0.03 (0.03)	< 0.01, < 0.01, < 0.01, < 0.01 (< 0.01)	< 0.035, < 0.045, < 0.045, < 0.045 (*< 0.042*)	
Los Ruices, Valencia (1999) Spain *Bobal* 99069-F/G	8×(87.5)	11-13	21	0.010, 0.010 (0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (*< 0.025*)	RA-3084
Sobreiros-Alenquer, Estremadura (1999) Portugal *Santarem* P99004R	8×(75)	11-12	21	0.050, 0.050, 0.070, 0.070 (0.060)	< 0.01, < 0.01, < 0.01, < 0.01 (< 0.010)	< 0.065, < 0.065, < 0.085, < 0.085 (*< 0.075*)	RA-3085

Location (Year) Country *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
Lujan de Cuyo, Mendoza (2001) Argentina *Emperor* MDG-011-01	8×(~100)	10-16	21	0.33, 0.35 (0.34)	0.020, 0.020 (0.020)	0.36, 0.38 (0.37)	RA-3091
Los Mochis, Sinaloa (2001) Mexico *Superior* MDG-011-02	8×(~100)	11-13	21	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	

[a] Molecular weight ratio cyazofamid:CCIM = 1.49. Combined = Cyazofamid residue + (CCIM residue × 1.49)

Table 36 Residues of cyazofamid and CCIM in broccoli following foliar application in the USA (Study RA-3123)

Location (Year) *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)
GAP: USA [Brassica (cole) leafy vegetables]	1 soil at plant (753) + 5 foliar (80)	7-10	0	--	--	--
Salinas, CA (2006) *Everest* 09717.06-CA*38 [b c]	132 soil + 5×(~82) foliar	55, 6-7	0	0.91, 0.76 (0.84)	< 0.01, < 0.01 (< 0.010)	< 0.**92**, < 0.77 (< *0.85*)
Salinas, CA (2006) *Marathon* 09717.06-CA*39 [b c]	132 soil + 5×(~80) foliar	48, 7-8	0	0.41, 0.33 (0.37)	< 0.01, < 0.01 (< 0.010)	< 0.42, < 0.34 (< *0.38*)
Holtville, CA (2006) *Heritage* 09717.06-CA40 [d]	130 soil + 6×(~81) foliar	58, 6-8	0	0.26, 0.19 (0.23)	< 0.01, < 0.01 (< 0.010)	< 0.27, < 0.20 (< *0.24*)
Holtville, CA (2006) *Triathalon* 09717.06-CA41 [d]	128 soil + 5×(~83) foliar	65, 7-8	0	0.28, 0.39 (0.34)	< 0.01, < 0.01 (< 0.010)	< 0.29, < 0.40 (< *0.35*)
Aurora, OR (2006) *General* 09717.06-OR27 [b]	132 soil + 6×(~81) foliar	19, 6-8	0	0.47, 0.45 (0.46)	< 0.01, < 0.01 (< 0.010)	< 0.48, < 0.46 (< *0.47*)
Weslaco, TX (2006) *Gypsy* 09717.06-TX*13 [b]	132 soil + 5×(~83) foliar	64, 6-8	0	0.18, 0.27 (0.23)	< 0.01, < 0.01 (< 0.010)	< 0.19, < 0.28 (< *0.24*)

[a] Molecular weight ratio cyazofamid:CCIM = 1.49. Combined = Cyazofamid residue + (CCIM residue × 1.49)

[b] Samples were cut in the field.

[c] Final application and harvest differed between these trials by 53 days.

[d] Final application and harvest differed between these trials by 0 days.

Table 37 Residues of cyazofamid and CCIM in **cabbage** (with wrapper leaves) following foliar application in the USA (Study RA-3124)

Location (Year) *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)
GAP: USA [Brassica (cole) leafy vegetables]	1 soil at plant (753) + 5 foliar (80)	7-10	0	--	--	--
Salinas, CA (2006) *Charmant* 09082.06-CA*42 [b]	132 soil + 5×(~82) foliar	62, 6-8	0	0.25, 0.25 (0.25)	< 0.01, < 0.01 (< 0.010)	< 0.26, < 0.26 (< *0.26*)
Brighton, CO (2006) *Rocket*	132 soil + 5×(~82) foliar	36, 6-7	0	0.30, 0.29 (0.30)	< 0.01, < 0.01 (< 0.010)	< 0.31, < 0.30 (< *0.31*)

Location (Year) *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)
09082.06-CO05 [b]						
Citra, FL (2006) *Bravo* 09082.06-FL17 [b]	132 soil + 5×(~85) foliar	42, 7	0	0.61, 0.50 (0.56)	0.016, 0.012 (0.014)	0.63, 0.52 (*0.58*)
Salisbury, MD (2006) *Prima* 09082.06-MD21 [b]	132 soil + 5×(~81) foliar	31, 6-8	0	0.87, 0.63 (0.75)	0.025, 0.020 (0.023)	**0.91**, 0.66 (*0.78*)
Bridgeton, NJ (2006) *Wisconsin Golden Acre* 09082.06-NJ08 [b]	132 soil + 5×(~78) foliar	31, 6-8	0	0.40, 0.16 (0.28)	0.013, < 0.01 (0.012)	0.42, < 0.17 (< *0.30*)
Freeville, NY (2006) *Bobcat* 09082.06-NY07 [b]	132 soil + 6×(~77) foliar	77, 6-7	0	0.22, 0.17 (0.20)	< 0.01, < 0.01 (< 0.010)	< 0.23, < 0.18 (< *0.21*)
Charleston, SC (2006) *Copenhagen* 09082.06-SC*05 [b]	133 soil + 5×(~80) foliar	63, 7-8	0	0.16, 0.14 (0.15)	< 0.01, < 0.01 (< 0.010)	< 0.17, < 0.15 (< *0.16*)
Weslaco, TX (2006) *Blue Vantage* 09082.06-TX*14 [b]	132 soil + 5×(~82) foliar	78, 6-7	0	0.33, 0.31 (0.32)	< 0.01, < 0.01 (< 0.010)	< 0.34, < 0.32 (< *0.33*)
Arlington, WI (2006) *Blue Vantage* 09082.06-WI10 [b]	135 soil + 5×(~79) foliar	60, 6-8	0	0.12, 0.13 (0.13)	< 0.01, < 0.01 (< 0.010)	< 0.13, < 0.14 (< *0.14*)

[a] Molecular weight ratio cyazofamid:CCIM = 1.49. Combined = Cyazofamid residue + (CCIM residue × 1.49)

[b] Samples were cut in the field.

Table 38 Residues of cyazofamid and CCIM in **cucumber** following foliar application in the USA

Location (Year) *Variety* Site ID [Study ID]	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
GAP: USA [Cucurbit vegetables]	6×(80)	7-10	0	--	--	--	--
Cary, NC (1999) *Poinsett* B	6×(~81)	2-12	0	0.020, 0.010 (0.015)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.025 (< 0.030)	RA-3067
			1	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
			3	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
			7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
Pelham, GA (1999) *Thunder* E	6×(~81)	7	7	0.030, 0.040 (0.035)	< 0.01, < 0.01 (< 0.010)	< 0.045, < 0.055 (< 0.050)	
Jupiter, FL (1999) *Meteor* G	6×(~80)	7	7	0.020, < 0.01 (0.015)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.025 (< 0.030)	
Macon, MO (1999) *Long Green* H	6×(~82)	7	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
Arkansaw, WI (1999) *Lucky Strike Hybrid* J	6×(~80)	7	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
Eakly, OK	6×(~81)	7	7	< 0.01, < 0.01	< 0.01, < 0.01	< 0.025, < 0.025	

Location (Year) *Variety* Site ID [Study ID]	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
(1999) *Straight Eight* L				(< 0.010)	(< 0.010)	(< 0.025)	
Cotton, GA (2000) *Cross Country (Pickling)* 3	6×(~45)	6-8	0	0.020, 0.020 (0.020)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.035 (< 0.035)	RA-3090
Hobe Sound, FL (2000) *Speedway* 5	6×(~82)	7	0	0.020, 0.020 (0.020)	0.010, 0.010 (0.010)	0.035, 0.035 (*0.035*)	
Arkansaw, WI (2001) *Hybrid Eureka* 7	6×(~80)	6-8	0	0.010, < 0.01 (0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Clarence, MO (2001) *Bush Champion* 9	6×(~79)	6-8	0	0.030, 0.020 (0.025)	< 0.01, < 0.01 (< 0.010)	**< 0.045**, < 0.035 (< *0.040*)	
Eakly, OK (2001) *Boston Pickling* 10	6×(~78)	6-7	0	0.020, 0.020 (0.020)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.035 (< *0.035*)	

[a] Molecular weight ratio cyazofamid:CCIM = 1.49. Combined = Cyazofamid residue + (CCIM residue × 1.49)

Table 39 Residues of cyazofamid and CCIM in **summer squash** following foliar application in the USA

Location (Year) *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
GAP: USA [Cucurbit vegetables]	6×(80)	7-10	0	--	--	--	--
North Rose, NY (1999) *Zucchini Select* A	6×(~80)	7	0	0.020, 0.030 (0.025)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.045 (< *0.040*)	RA-3067
			1	0.020, 0.020 (0.020)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.035 (< 0.035)	
			3	0.010, 0.010 (0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
			7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
Cary, NC (1999) *Early Prolific Straightneck* D	6×(~80)	7	7	0.010, < 0.01 (0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
Chipley, FL (1999) *Prelude II Hybrid* F	6×(~78)	7	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
Theilman, MN (1999) *Monet, Yellow Straightneck* I	6×(~79)	7	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
Porterville, CA	6×(~79)	7	7	< 0.01, 0.010 (0.010)	< 0.01, < 0.01	< 0.025,	

Location (Year) *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
(1999) *Peter Pan* P					(< 0.010)	< 0.025 (< 0.025)	
Rose Hill, NC (2001) *Early Prolific Straightneck* 1	6×(~79)	6-8	0	0.030, 0.020 (0.025)	< 0.01, < 0.01 (< 0.010)	< 0.045, < 0.035 (< *0.040*)	RA-3090
Quincy, FL (2000) *Yellow Crook Neck* 4	78 + 5×(~45)	6-7	0	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
Arkansaw, WI (2001) *Hybrid Monet* 6	6×(~80)	6-8	0	0.040, 0.040 (0.040)	< 0.01, < 0.01 (< 0.010)	< 0.055, < 0.055 (< *0.055*)	
Porterville, CA (2001) *Peter Pan* 13	6×(~81)	6-8	0	0.050, 0.030 (0.040)	< 0.01, < 0.01 (< 0.010)	**< 0.065**, < 0.045 (< *0.055*)	

[a] Molecular weight ratio cyazofamid:CCIM = 1.49. Combined = Cyazofamid residue + (CCIM residue × 1.49)

Table 40 Residues of cyazofamid and CCIM in **muskmelon** following foliar application in the USA

Location (Year) *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
GAP: USA [Cucurbit vegetables]	6×(80)	7-10	0	--	--	--	--
Cary, NC (1999) *Hales Best Jumbo* C [b]	6×(~81)	6-8	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	RA-3067
Arkansaw, WI (1999) *Cantaloupe Hybrid Pulsar* K [b]	6×(~80)	6-8	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
Eakly, OK (1999) *Tesoro* M [b]	6×(~79)	7	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
San Luis Obispo, CA (1999) *Gold Master* N	6×(~81)	7-8	7	0.010, 0.020 (0.015)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.035 (< 0.030)	
Kerman, CA (1999) *Hales Best Jumbo* O	6×(~80)	7	7	0.020, 0.020 (0.020)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.035 (< 0.035)	
Visalia, CA (1999) *Hales Best Jumbo* Q	6×(~78)	7	0	0.030, 0.030 (0.030)	< 0.01, < 0.01 (< 0.010)	< 0.045, < 0.045 (< *0.045*)	
			1	0.020, 0.020 (0.020)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.035 (< 0.035)	
			3	0.010, < 0.01 (0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
			7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
Rose Hill, NC (2001) *Hales Best Jumbo*	6×(~81)	7	0	0.060, 0.070 (0.065)	< 0.01, 0.010 (0.010)	< 0.075, **0.085** (< *0.080*)	RA-3090

Location (Year) *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
2							
Arkansaw, WI (2001) *Hybrid Primo* 8 [b]	6×(~81)	7	0	0.030, 0.030 (0.030)	< 0.01, < 0.01 (< 0.010)	< 0.045, < 0.045 (*< 0.045*)	
Eakly, OK (2001) *Tesoro* 11 [b]	6×(~79)	6-7	0	0.030, 0.010 (0.020)	< 0.01, < 0.01 (< 0.010)	< 0.045, < 0.025 (*< 0.035*)	
Fresno, CA (2001) *Top Mark Cantaloupe* 12	6×(~80)	7	0	0.010, 0.020 (0.015)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.035 (*< 0.030*)	
Holtville, CA (2000) *IMPAC Cantaloupe* 14	6×(~81)	5-8	0	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (*< 0.025*)	

[a] Molecular weight ratio cyazofamid:CCIM = 1.49. Combined = Cyazofamid residue + (CCIM residue × 1.49)

[b] Samples were cut in the field.

Table 41 Residues of cyazofamid and CCIM in **peppers** following foliar application in the USA (Study RA-3101)

Location (Year) *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)
GAP: USA [Fruiting vegetables]	6×(80)	7-10	0	--	--	--
Sweet Peppers						
Goldsboro, NC (2006) *Heritage* 01	6×(~79)	7	0	0.037, 0.038 (0.038)	< 0.01, < 0.01 (< 0.010)	< 0.052, < 0.053 (*< 0.052*)
Jennings, FL (2006) *Aristotle* 02	6×(~79)	7	0	0.055, 0.060 (0.058)	< 0.01, < 0.01 (< 0.010)	< 0.070, < 0.075 (*< 0.072*)
Northwood, ND (2006) *Lady Bell* 03	6×(~78)	6-8	0	0.079, 0.065 (0.072)	< 0.01, < 0.01 (< 0.010)	< 0.094, < 0.080 (*< 0.087*)
			1	0.073, 0.030 (0.052)	< 0.01, < 0.01 (< 0.010)	< 0.088, < 0.045 (< 0.066)
			3	0.033, 0.027 (0.030)	< 0.01, < 0.01 (< 0.010)	< 0.048, < 0.042 (< 0.045)
			7	0.020, 0.020 (0.020)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.035 (< 0.035)
Hinton, OK (2006) *California Wonder* 05	6×(~79)	7-8	0	0.071, 0.12 (0.098)	< 0.01, < 0.01 (< 0.010)	< 0.086, < 0.13 (*< 0.11*)
Fresno, CA (2006) *Taurus* 07	6×(~80)	7	0	0.048, 0.062 (0.055)	< 0.01, < 0.01 (< 0.010)	< 0.063, < 0.077 (*< 0.070*)
Madera, CA (2006) *Macabbi* 09	6×(~80)	6-8	0	0.16, 0.28 (0.22)	< 0.01, 0.014 (0.012)	< 0.17, **0.30** (*< 0.24*)
Chili Peppers						
Northwood, ND (2006) *Long Red Cayenne* 04	6×(~78)	6-8	0	0.28, 0.21 (0.24)	0.017, < 0.01 (0.014)	0.31, < 0.22 (*< 0.27*)
Dill City, OK (2006) *Anaheim* 06	6×(~78)	6-8	0	0.32, 0.30 (0.31)	0.014, 0.014 (0.014)	**0.34**, 0.32 (*0.33*)
Fresno, CA (2006) *Anaheim (Sonora)* 08	6×(~80)	7	0	0.25, 0.25 (0.25)	0.013, < 0.01 (0.012)	0.27, < 0.26 (*< 0.27*)

[a] Molecular weight ratio cyazofamid:CCIM = 1.49. Combined = Cyazofamid residue + (CCIM residue × 1.49)

Table 42 Residues of cyazofamid and CCIM in **tomato** following foliar application in the USA

Location (Year) *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
GAP: USA [Fruiting vegetables]	6×(80)	7-10	0	--	--	--	--
North Rose, NY (1999) *Mountain Pride* A	6×(~79)	7	7	0.020, 0.030 (0.025)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.045 (< 0.040)	RA-3065
Cary, NC (1999) *Better Boy* B	6×(~80)	2-12	0	0.050, 0.060 (0.055)	< 0.01, < 0.01 (< 0.010)	< 0.065, < 0.075 (< 0.070)	
			1	0.040, 0.030 (0.035)	< 0.01, < 0.01 (< 0.010)	< 0.055, < 0.045 (< 0.050)	
			3	0.030, 0.020 (0.025)	< 0.01, < 0.01 (< 0.010)	< 0.045, < 0.035 (< 0.040)	
			7	0.020, < 0.01 (0.015)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.025 (< 0.030)	
Chipley, FL (1999) *Florida-47* C	6×(~81)	7	7	< 0.01, 0.010 (0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
Jupiter, FL (1999) *Sanibel* D	6×(~82)	7	7	< 0.01, 0.010 (0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
Orlando, FL (1999) *Florida-47* E	6×(~79)	7	7	0.020, 0.020 (0.020)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.035 (< 0.035)	
Proctor, AR (1999) *Better Boy* F	6×(~81)	7	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
Visalia, CA (1999) *Rio Grande* G	6×(~79)	7	0	0.020, 0.030 (0.025)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.045 (< *0.040*)	
			1	0.060, 0.060 (0.060)	< 0.01, < 0.01 (< 0.010)	< 0.075, < 0.075 (< 0.075)	
			3	0.060, 0.070 (0.065)	< 0.01, < 0.01 (< 0.010)	< 0.075, < 0.085 (< 0.080)	
			7	0.020, 0.010 (0.015)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.025 (< 0.030)	
Fresno, CA (1999) *Celebrity* H	6×(~80)	7	7	0.020, 0.020 (0.020)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.035 (< 0.035)	
Suisun, CA (1999) *Heinz 9281* I	6×(~79)	7	7	0.030, 0.020 (0.025)	< 0.01, < 0.01 (< 0.010)	< 0.045, < 0.035 (< 0.040)	
Manteca, CA (1999) *HP-108* J	6×(~79)	7	7	0.020, 0.030 (0.025)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.045 (< 0.040)	
Woodland, CA (1999) *Rio Grande* K	6×(~76)	7	7	0.040, 0.060 (0.050)	< 0.01, < 0.01 (< 0.010)	< 0.055, < 0.075 (< 0.065)	
San Luis Obispo, CA (1999) *Shady Lady* L	6×(~79)	6-8	7	0.020, 0.020 (0.020)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.035 (< 0.035)	
King City, CA (1999) *Mountain Fresh* M	6×(~80)	7-8	7	0.020, 0.020 (0.020)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.035 (< 0.035)	
Huron, CA (1999) *Roma* N	6×(~80)	7	7	0.030, 0.030 (0.030)	< 0.01, < 0.01 (< 0.010)	< 0.045, < 0.045 (< 0.045)	
Kerman, CA (1999) *Roma*	6×(~80)	7	7	0.020, 0.020 (0.020)	< 0.01, < 0.01 (< 0.010)	< 0.035, < 0.035 (< 0.035)	

Location (Year) *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
O-1							
Porterville, CA (1999) *ACC* 55 *VF* P	6×(~79)	7	7	0.030, 0.030 (0.030)	< 0.01, < 0.01 (< 0.010)	< 0.045, < 0.045 (< 0.045)	
Fresno, CA (1999) *Heinz 9382* Q	6×(~81)	6-8	7	0.040, 0.030 (0.035)	< 0.01, < 0.01 (< 0.010)	< 0.055, < 0.045 (< 0.050)	
Hughson, CA (1999) *Cannery Row* R	6×(~80)	7	7	0.060, 0.060 (0.060)	< 0.01, < 0.01 (< 0.010)	< 0.075, < 0.075 (< 0.075)	
San Luis Obispo, CA (2000) *Shady Lady* Plot 2	6×(~79)	6-8	0	0.050, 0.020 (0.035)	< 0.01, < 0.01 (< 0.010)	< 0.065, < 0.035 (< 0.050)	RA-3077
			7	0.030, 0.020 (0.025)	< 0.01, < 0.01 (< 0.010)	< 0.045, < 0.035 (< 0.040)	
Plot 3	6×(~80)	6-7	0	0.050, 0.030 (0.040)	< 0.01, < 0.01 (< 0.010)	< 0.065, < 0.045 (< 0.055)	
			7	0.040, 0.060 (0.050)	< 0.01, < 0.01 (< 0.010)	< 0.055, < 0.075 (< *0.065*)	
North Rose, NY (2001) *Floradade* 1	6×(~80)	7	0	0.060, 0.040 (0.050)	0.010, 0.010 (0.010)	0.075, 0.055 (*0.065*)	RA-3089
Quincy, FL (2001) *Solo Set* 2	80 + 5×(~45)	6-7	0	0.010, 0.010 (0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Hobe Sound, FL (2001) *Sanibel* 3	6×(~82)	6-8	0	0.060, 0.090 (0.075)	< 0.01, < 0.01 (< 0.010)	< 0.075, < 0.10 (< *0.090*)	
Winter Garden, FL (2001) *Better Boy* 4	6×(~80)	6-7	0	0.070, 0.060 (0.065)	< 0.01, < 0.01 (< 0.010)	< 0.085, < 0.075 (< *0.080*)	
Proctor, AR (2001) *Better Bush* 5	6×(~80)	7	0	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Madera, CA (2001) *Celebrity* 6	6×(~81)	7	0	0.030, 0.030 (0.030)	< 0.01, < 0.01 (< 0.010)	< 0.045, < 0.045 (< *0.045*)	
Fresno, CA (2001) *Super Roma* 7	6×(~81)	7	0	0.050, 0.050 (0.050)	< 0.01, < 0.01 (< 0.010)	< 0.065, < 0.065 (< *0.065*)	
Hickman, CA (2001) *9775* 8	6×(~80)	7	0	0.16, 0.13 (0.15)	< 0.01, < 0.01 (< 0.010)	**< 0.17**, < 0.14 (< *0.16*)	
Dixon, CA (2001) *Brigade* 9	6×(~80)	6-8	0	0.050, 0.030 (0.040)	< 0.01, < 0.01 (< 0.010)	< 0.065, < 0.045 (< *0.055*)	
Fresno, CA (2001) *Shady Lady* 10	6×(~80)	7	0	0.13, 0.080 (0.11)	0.020, 0.010 (0.015)	0.16, 0.095 (*0.13*)	
Watsonville, CA (2001) *Sunbolt* 11	6×(~77)	6-8	0	0.050, 0.050 (0.050)	< 0.01, < 0.01 (< 0.010)	< 0.065, < 0.065 (< *0.065*)	
San Luis Obispo, CA (2001) *Shady Lady* 12	6×(~82)	7	0	0.030, 0.040 (0.035)	0.010, < 0.01 (0.010)	0.045, < 0.055 (< *0.050*)	
Porterville, CA (2001) *Ace 55* 13	6×(~80)	7	0	0.020, 0.040 (0.030)	< 0.01, 0.010 (0.010)	< 0.035, 0.055 (< *0.045*)	

[a] Molecular weight ratio cyazofamid:CCIM = 1.49. Combined = Cyazofamid residue + (CCIM residue × 1.49)

Table 43 Residues of cyazofamid and CCIM in **lettuce** following foliar application. In non-independent trial sets, final application and harvest occurred on the same day within a set

Location (Year) Country *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
GAP: USA [Leafy greens]	6×(80)	7-10	0	--	--	--	
GAP: Canada [Lettuce]	6×(80)	7-14	0	--	--	--	
Head Lettuce (without wrapper leaves)							
Delhi, ON (2009) Canada *Great Lakes 659* AAFC08-053RA-690 [b]	7×(~80)	21, 15, 6-8	0	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	RA-3199
St. Jean-sir-Richelieu, QC (2009) Canada *Ithica* AAFC08-053RA-691 [b]	6×(~79)	19, 7-8	0	0.050, 0.070 (0.060)	< 0.01, < 0.01 (< 0.010)	< 0.065, < 0.085 (< 0.075)	
Agassiz, BC (2009) Canada *Mighty Joe MI* AAFC08-053RA-692	6×(~81)	18, 6-8	0	0.57, 0.54 (0.56)	0.013, 0.012 (0.013)	0.59, 0.56 (0.57)	
Head Lettuce (with wrapper leaves)							
Delhi, ON (2009) Canada *Great Lakes 659* AAFC08-053RA-690 [b]	7×(~80)	21, 15, 6-8	0	0.050,0.090 (0.070)	< 0.01, < 0.01 (< 0.010)	< 0.065, < 0.10 (*< 0.085*)	RA-3199
St. Jean-sir-Richelieu, QC (2009) Canada *Ithica* AAFC08-053RA-691 [b]	6×(~79)	19, 7-8	0	0.41, 0.50 (0.46)	0.010, 0.010 (0.010)	0.42, 0.51 (*0.47*)	
Agassiz, BC (2009) Canada *Mighty Joe MI* AAFC08-053RA-692	6×(~81)	18, 6-8	0	1.3, 1.1 (1.2)	0.022, 0.021 (0.022)	1.3, 1.1 (*1.2*)	
Freeville, NY (2008) USA *Ponderosa* 10037.08-NY31	6×(~80)	6-7	0	0.78, 0.67 (0.73)	0.013, 0.012 (0.013)	0.80, 0.69 (*0.74*)	RA-3196
Citra, FL (2008) USA *Optima* 10037.08-FL49 [b]	6×(~81)	7	0	1.7, 1.3 (1.5)	0.029, 0.022 (0.026)	1.7, 1.3 (*1.5*)	
Salinas, CA (2008) USA *Samurai* 10037.08-CA*05 [b]	7×(~81)	42, 6-8	0	1.3, 1.4 (1.4)	0.015, 0.014 (0.015)	1.3, 1.4 (*1.4*)	
Salinas, CA (2008) USA *Gabilan* 10037.08-CA*04 [b]	7×(~81)	42, 6-8	0	1.7, 1.6 (1.7)	0.018, 0.015 (0.017)	1.7, 1.6 (*1.7*)	
Parlier, CA (2008) USA *Great Lakes 659* 10037.08-CA02 [b]	6×(~82)	72, 5-8	0	2.0, 1.6 (1.8)	0.011, < 0.01 (0.011)	**2.0**, <1.6 (*<1.8*)	
Las Cruces, NM (2008) USA *Salinas* 10037.08-NM11	7×(~80)	70, 6-8	0	0.24, 0.28 (0.26)	< 0.01, < 0.01 (< 0.010)	< 0.25, < 0.29 (*< 0.27*)	
Holtville, CA (2008) USA *Deuce* 10037.08-CA03 [b]	6×(~80)	78, 6-8	0	0.60, 0.65 (0.63)	< 0.01, < 0.01 (< 0.010)	< 0.61, < 0.66 (*< 0.64*)	
Holtville, CA (2008)	6×(~79)	78,	0	0.29, 0.43	< 0.01,	< 0.30, < 0.44	

Location (Year) Country *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
USA *Quest* 10037.08-CA19 [b]		6-8		(0.36)	< 0.01 (< 0.010)	(*< 0.37*)	
Ste-Clotilde, QC (2008) Canada *Estival* 10037.08-QC08 [b]	6×(~82)	14, 6-8	0	0.63, 0.62 (0.63)	0.017, 0.016 (0.017)	0.66, 0.64 (*0.65*)	
Harrow, ON (2008) Canada *Mighty Joe* 10037.08-ON19 [b]	6×(~80)	28, 6-7	0	0.19, 0.20 (0.20)	< 0.01, < 0.01 (< 0.010)	< 0.20, < 0.21 (*< 0.21*)	
Leaf Lettuce							
Delhi, ON (2009) Canada *Simpson Elite* AAFC08-053RA-693	6×(~80)	6-8	0	3.0, 2.6 (2.8)	0.050, 0.037 (0.044)	3.1, 2.7 (*2.9*)	RA-3199
St. Jean-sir-Richelieu, QC (2009) Canada *Panther* (*Romaine*) AAFC08-053RA-694	6×(~81)	19, 7-8	0	0.48, 0.58 (0.53)	< 0.01, 0.011 (0.011)	< 0.49, 0.60 (*< 0.55*)	
Salisbury, MD (2008) USA *Tropicana* 10037.08-MD23 [b]	6×(~80)	15, 7-8	0	0.83, 0.69 (0.76)	0.031, 0.022 (0.027)	0.88, 0.72 (*0.80*)	RA-3196
Citra, FL (2008) USA *Two Star* 10037.08-FL50 [b]	6×(~80)	7	0	1.9, 1.6 (1.8)	0.021, 0.021 (0.021)	1.9, 1.6 (*1.8*)	
Las Cruces, NM (2008) USA *Oakleaf* 10037.08-NM14	7×(~82)	36, 7-8	0	2.9, 3.3 (3.1)	0.036, 0.043 (0.040)	3.0, 3.4 (*3.2*)	
Las Cruces, NM (2008) USA *Salad Bowl* 10037.08-NM12	6×(~82)	51, 7-8	0	4.5, 3.5 (4.0)	0.045, 0.037 (0.041)	4.6, 3.6 (*4.1*)	
Holtville, CA (2008) USA *Greenleaf* 10037.08-CA07 [b]	6×(~80)	73, 7	0	1.2, 1.5 (1.4)	0.010, 0.013 (0.012)	1.2, 1.5 (*1.4*)	
Salinas, CA (2008) USA *Kremlin* 10037.08-CA*22	6×(~80)	26, 6-8	0	2.6, 2.5 (2.6)	0.039, 0.034 (0.037)	2.7, 2.6 (*2.6*)	
			4	1.5, 1.3 (1.4)	0.021, 0.019 (0.020)	1.5, 1.3 (1.4)	
			7	1.1, 1.2 (1.2)	0.015, 0.018 (0.017)	1.1, 1.2 (1.2)	
			15	0.42, 0.35 (0.39)	< 0.01, < 0.01 (< 0.010)	< 0.43, < 0.36 (< 0.40)	
			21	0.069, 0.15 (0.11)	< 0.01, < 0.01 (< 0.010)	< 0.084, < 0.16 (< 0.12)	
Salinas, CA (2008) USA *Pacifica* 10037.08-CA*21	6×(~82)	33, 6-8	0	2.9, 3.1 (3.0)	0.032, 0.034 (0.033)	2.9, 3.2 (*3.0*)	
Parlier, CA (2008) USA *Waldmann's Green* 10037.08-CA06	6×(~82)	27, 7	0	2.6, 2.8 (2.7)	0.040, 0.041 (0.041)	2.7, 2.9 (*2.8*)	

Location (Year) Country *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
Jordan Station, ON (2008) Canada *Green Tower* 10037.08-ON20 [b]	6×(~83)	20, 6-7	0	1.0, 0.73 (0.87)	0.018, 0.013 (0.016)	1.0, 0.75 (*0.89*)	
Ste-Clotilde, QC (2008) Canada *Green Tower* 10037.08-QC07 [b]	6×(~84)	14, 6-8	0	0.91, 0.87 (0.89)	0.024, 0.025 (0.025)	0.95, 0.91 (*0.93*)	
Agassiz, BC (2008) Canada *Lasting Green 1* 10037.08-BC06 [b]	6×(~84)	28, 6-7	0	4.4, 4.5 (4.4)	0.041, 0.043 (0.042)	4.5, **4.6** (*4.5*)	

[a] Molecular weight ratio cyazofamid:CCIM = 1.49. Combined = Cyazofamid residue + (CCIM residue × 1.49)

[b] Samples were cut in the field.

Table 44 Residues of cyazofamid and CCIM in **mustard greens** following soil + foliar application in the USA (Study RA-3125)

Location (Year) *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)
GAP: USA [Brassica (cole) leafy vegetables]	1 soil at plant (753) + 5 foliar (80)	7-10	0	--	--	--
Salinas, CA (2006) *Green Wave Mustard* 09083.06-CA*43	132 soil + 5×(~81) foliar	8, 6-7	0	2.9, 3.9 (3.4)	0.030, 0.040 (0.035)	2.9, 4.0 (*3.5*)
Riverside, CA (2006) *Florida Broadleaf* 09083.06-CA44	133 soil + 5×(~82) foliar	21, 6-8	0	3.0, 3.6 (3.3)	0.032, 0.032 (0.032)	3.0, 3.6 (*3.3*)
Citra, FL (2006) *Florida Broadleaf* 09083.06-FL18	131 soil + 5×(~87) foliar	7	0	6.0, 5.9 (6.0)	0.094, 0.090 (0.092)	6.1, 6.0 (*6.1*)
Tifton, GA (2006) *Green Wave* 09083.06-GA*06	132 soil + 5×(~84) foliar	15, 6-7	0	6.8, 5.8 (6.3)	0.056, 0.050 (0.053)	**6.9**, 5.9 (*6.4*)
Salisbury, MD (2006) *Green Wave* 09083.06-MD06	132 soil + 5×(~81) foliar	16, 6-8	0	5.5, 5.5 (5.5)	0.050, 0.050 (0.050)	5.6, 5.6 (*5.6*)
Bridgeton, NJ (2006) *Southern Curled* 09083.06-NJ09	132 soil + 5×(~79) foliar	7-9	0	3.0, 4.0 (3.5)	0.13, 0.17 (0.15)	3.2, 4.3 (*3.7*)
Jackson, TN (2006) *Florida Broadleaf* 09083.06-TN06	132 soil + 5×(~84) foliar	7-8	0	1.5, 1.3 (1.4)	0.11, 0.10 (0.11)	1.7, 1.4 (*1.6*)
Weslaco, TX (2006) *Florida Broadleaf* 09083.06-TX*15	132 soil + 5×(~81) foliar	20, 7	0	2.1, 1.7 (1.9)	0.038, 0.032 (0.035)	2.2, 1.7 (*2.0*)
			1	1.2, 1.4 (1.3)	0.019, 0.019 (0.019)	1.2, 1.4 (1.3)
			3	0.33, 0.38 (0.36)	< 0.01, 0.012 (0.011)	< 0.34, 0.40 (< 0.37)
			6	0.061, 0.066 (0.064)	< 0.01, < 0.01 (< 0.010)	< 0.076, < 0.081 (< 0.078)
			7	0.022, 0.024 (0.023)	< 0.01, < 0.01 (< 0.010)	< 0.037, < 0.039 (< 0.038)
Arlington, WI (2006) *Florida Broadleaf* 09083.06-WI11	137 soil + 5×(~80) foliar	6-8	0	3.8, 3.6 (3.7)	0.19, 0.17 (0.18)	4.1, 3.9 (*4.0*)

[a] Molecular weight ratio cyazofamid:CCIM = 1.49. Combined = Cyazofamid residue + (CCIM residue × 1.49)

Table 45 Residues of cyazofamid and CCIM in **spinach** following foliar application in the USA (Study RA-3126)

Location (Year) *Variety* Site ID [Study ID]	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)
GAP: USA [Leafy greens]	6×(80)	7-10	0	--	--	--
Salinas, CA (2006) *Whale* F1 *06*/*Smooth leaf* 09265.06-CA*36 [b]	5×(~80)	6-7	0	3.9, 3.3 (3.6)	0.044, 0.045 (0.045)	4.0, 3.4 (*3.7*)
Salinas, CA (2006) *Space F1*/*Smooth leaf* 09265.06-CA*37 [b]	5×(~78)	6-8	0	3.0, 3.6 (3.3)	0.036, 0.032 (0.034)	3.1, 3.6 (*3.4*)
Fort Collins, CO (2006) *Bloomsdale Savoy* 09265.06-CO04	5×(~79)	6-7	0	2.4, 1.9 (2.2)	0.036, 0.032 (0.049)	2.5, 1.9 (*2.2*)
			1	2.5, 1.6 (2.1)	0.031, 0.023 (0.027)	2.5, 1.6 (2.1)
			3	1.2, 1.2 (1.2)	0.015, 0.012 (0.014)	1.2, 1.2 (1.2)
			4	0.90, 1.0 (0.95)	0.013, 0.016 (0.015)	0.92, 1.0 (0.97)
			6	0.69, 0.82 (0.76)	< 0.01, 0.011 (0.011)	< 0.70, 0.84 (< 0.77)
Bridgeton, NJ (2006) *Melody* 09265.06-NJ07	5×(~80)	6-7	0	6.3, 6.5 (6.4)	0.12, 0.12 (0.12)	6.5, **6.7** (*6.6*)
Freeville, NY (2006) *Tyee F1* 09265.06-NY06	5×(~80)	6-8	0	2.1, 1.9 (2.0)	0.031, 0.027 (0.029)	2.1, 1.9 (*2.0*)
Charleston, SC (2006) *Skooku*, *Hybrid* 09265.06-SC*04	5×(~82)	7-8	0	2.6, 3.1 (2.9)	0.081, 0.094 (0.088)	2.7, 3.2 (*3.0*)
Crossville, TN (2006) *Bloomsdale* 09265.06-TN07	5×(~82)	6-8	0	3.6, 3.2 (3.4)	0.10, 0.086 (0.093)	3.7, 3.3 (*3.5*)
Jackson, TN (2006) *Bloomsdale* 09265.06-TN08	5×(~82)	7	0	1.8, 2.2 (2.0)	0.088, 0.011 (0.050)	1.9, 2.2 (*2.1*)
Weslaco, TX (2006) *Spargo F1* 09265.06-TX11 [c]	5×(~81)	6-7	0	1.7, 1.4 (1.6)	0.064, 0.054 (0.059)	1.8, 1.5 (*1.6*)
Weslaco, TX (2006) *Samish* 09265.06-TX*12 [c]	5×(~79)	6-8	0	4.1, 5.1 (4.6)	0.13, 0.15 (0.14)	4.3, 5.3 (*4.8*)

[a] Molecular weight ratio cyazofamid:CCIM = 1.49. Combined = Cyazofamid residue + (CCIM residue × 1.49)

[b] Final application and harvest differed between these trials by 20 days.

[c] Final application and harvest differed between these trials by 43 days.

Table 46 Residues of cyazofamid and CCIM in **lima bean** following foliar application in the USA (Study RA-3195)

Location (Year) Country *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)
GAP: USA [Beans (succulent podded and succulent shelled)	6×(80)	7-14	0	--	--	--
Irvine, CA (2009) *Fordhook 242* 09532.09-CA134	7×(~80)	6-7	0	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (*< 0.025*)
Parlier, CA (2009) *Fordhook 242*	6×(~81)	6-8	0	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025

Location (Year) Country *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)
09532.09-CA135						(< *0.025*)
Kimberly, ID (2009) *M15 Lima* 09532.09-ID20	6×(~81)	6-8	0	0.033, 0.047 (0.040)	< 0.01, < 0.01 (< 0.010)	< 0.048, **< 0.062** (< *0.055*)
Salisbury, MD (2009) *Eastland* 09532.09-MD15	6×(~80)	6-8	0	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)
Salisbury, MD (2009) *Burpee Improved* 09532.09-MD24	6×(~80)	6-8	0	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)
Clinton, NC (2009) *Ford Hook* 09532.09-NC30	7×(~80)	6-7	0	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)
Clinton, NC (2009) *Thorogreen* 09532.09-NC31	7×(~84)	6-7	1	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)
Arlington, WI (2009) *Cypress* 09532.09-WI20	6×(~80)	7-8	0	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)

[a] Molecular weight ratio cyazofamid:CCIM = 1.49. Combined = Cyazofamid residue + (CCIM residue × 1.49)

Table 47 Residues of cyazofamid and CCIM in **snap bean** following foliar application in the USA (Study RA-3198)

Location (Year) *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)
GAP: USA [Beans (succulent podded and succulent shelled)	6×(80)	7-14	0	--	--	--
Irvine, CA (2007) *Jade* 09094.07-CA10	7×(~82)	7-8	0	0.21, 0.19 (0.20)	< 0.01, < 0.01 (< 0.010)	< 0.22, < 0.20 (< *0.21*)
Salinas, CA (2007) *Tongue of Fire* 09094.07-CA*09	6×(~80)	6-8	0	0.056, 0.061 (0.059)	< 0.01, < 0.01 (< 0.010)	< 0.071, < 0.076 (< *0.073*)
Tifton, GA (2007) *Bluelake Bush 274* 09094.07-GA*11	6×(~80)	6-8	0	0.22, 0.17 (0.20)	< 0.01, < 0.01 (< 0.010)	**< 0.23**, < 0.18 (< *0.21*)
			2	0.22, 0.16 (0.19)	< 0.01, < 0.01 (< 0.010)	< 0.23, < 0.17 (< 0.20)
			7	0.16, 0.15 (0.16)	< 0.01, < 0.01 (< 0.010)	< 0.17, < 0.16 (< 0.17)
			12	0.15, 0.11 (0.13)	< 0.01, < 0.01 (< 0.010)	< 0.16, < 0.12 (< 0.14)
Salisbury, MD (2007) *Prorider* 09094.07-MD01	6×(~87)	6-9	0	0.094, 0.11 (0.10)	< 0.01, < 0.01 (< 0.010)	< 0.11, < 0.12 (< *0.12*)
Holt, MI (2007) *Bush Blue Lake 156* 09094.07-MI36	6×(~80)	7-8	0	0.10, 0.13 (0.12)	< 0.01, < 0.01 (< 0.010)	< 0.11, < 0.14 (< *0.13*)
Bridgeton, NJ (2007) *Strike* 09094.07-NJ05	6×(~80)	6-9	0	0.17, 0.20 (0.19)	< 0.01, < 0.01 (< 0.010)	< 0.18, < 0.21 (< *0.20*)
Moxee, WA (2007) *Jade* 09094.07-WA*01	6×(~80)	7	0	0.038, 0.053 (0.046)	< 0.01, < 0.01 (< 0.010)	< 0.053, < 0.068 (< *0.060*)
Arlington, WI (2007) *Hystyle* 09094.07-WI06	6×(~81)	6-8	0	0.012, 0.026 (0.019)	< 0.01, < 0.01 (< 0.010)	< 0.027, < 0.041 (< *0.034*)

[a] Molecular weight ratio cyazofamid:CCIM = 1.49. Combined = Cyazofamid residue + (CCIM residue × 1.49)

Table 48 Residues of cyazofamid and CCIM in **carrot** following foliar application (Study RA-3107)

Location (Year) Country *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Storage time (days)
GAP: USA	5×(175)	14-21	14	--	--		--
Laingsburg, MI (2004) USA *Paramount S7540* 08522.04-MI09	5×(~175)	14-22	15	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	244
Laingsburg, MI (2004) USA *Paramount S7540* 08522.04-MI09 (a)	5×(~175)	14-22	15	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	244
Citra, FL (2004) USA *Indiana F1* 08522.04-FL25	5×(~175)	12-21	14	0.021, 0.023 (0.022)	< 0.01, < 0.01 (< 0.010)	< 0.036, < 0.038 (< 0.037)	352
Weslaco, TX (2004) USA *Six Pence F1* 08522.04-TX24	5×(~178)	13-35	0	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	373
			7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	366
			15	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	358
			20	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	353
			29	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	344
Tifton, GA (2004) USA *Nelson F1* 08522.04-GA*09	5×(~175)	16-92	14	0.027, 0.026 (0.027)	< 0.01, < 0.01 (< 0.010)	< 0.042, < 0.041 (< 0.041)	443
Moxee, WA (2004) USA *Enterprise F1* 08522.04-WA*04	5×(~178)	15-33	16	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	272
Moxee, WA (2004) USA *Enterprise F1* 08522.04-WA*04 (a)	5×(~175)	15-33	16	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	272
Salinas, CA (2004) USA *Mokum* 08522.04-CA*55	5×(~177)	7-21	14	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	270
Holtville, CA (2004) USA *Choctaw* 08522.04-CA52	5×(~175)	14-64	13	0.040, 0.027 (0.034)	< 0.01, < 0.01 (< 0.010)	< 0.055, < 0.042 (< 0.048)	91
Holtville, CA (2004) USA *Choctaw* 08522.04-CA52 (a)	5×(~175)	14-64	13	0.023, 0.035 (0.029)	< 0.01, < 0.01 (< 0.010)	< 0.038, < 0.050 (< 0.044)	91
Parlier, CA (2004) USA *Danvers Half Long 126* 08522.04-CA53	5×(~179)	12-34	0	0.028, 0.012 (0.020)	< 0.01, < 0.01 (< 0.010)	< 0.043, < 0.027 (< 0.035)	300
			8	0.044, 0.014	< 0.01, < 0.01	< 0.059, < 0.029	292

Location (Year) Country *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Storage time (days)
				(0.029)	(< 0.010)	(< 0.044)	
			14	0.026, 0.018 (0.022)	< 0.01, < 0.01 (< 0.010)	< 0.041, < 0.033 (< 0.037)	286
			21	0.018, 0.021 (0.020)	< 0.01, < 0.01 (< 0.010)	< 0.033, < 0.036 (< 0.034)	279
			28	0.023, 0.020 (0.022)	< 0.01, < 0.01 (< 0.010)	< 0.038, < 0.035 (< 0.036)	272
Riverside, CA (2004) USA *SXC 3293* 08522.04-CA54	5×(~175)	13-73	14	0.033, **0.045** (0.039)	< 0.01, < 0.01 (< 0.010)	< 0.048, < 0.060 (< 0.054)	105
Riverside, CA (2004) USA *SXC 3293* 08522.04-CA54 (a)	5×(~177)	13-73	14	0.033, 0.032 (0.033)	< 0.01, < 0.01 (< 0.010)	< 0.048, < 0.047 (< 0.047)	105
Elm Creek, MB (2004) Canada *Kamanan* 08522.04-MB01	5×(~175)	14-50	14	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	241
Elm Creek, MB (2004) Canada *Cheyenne* 08522.04-MB02	5×(~175)	14-50	14	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	241
Hunter River, PE (2004) Canada *Sweetness* II 08522.04-PE01	5×(~175)	12-35	13	0.027, 0.030 (0.029)	< 0.01, < 0.01 (< 0.010)	< 0.042, < 0.045 (< 0.043)	237
Napierville, QC (2004) Canada *Sun 255* 08522.04-QC04	5×(~175)	13-55	15	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	239
Napierville, QC (2004) Canada *Sunrise* 08522.04-QC05	5×(~175)	13-55	15	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	239

[a] Molecular weight ratio cyazofamid:CCIM = 1.49. Combined = Cyazofamid residue + (CCIM residue × 1.49)

Table 49 Residues of cyazofamid and CCIM in potato following soil+foliar and/or foliar application

Location (Year) Country *Variety* Site ID [Study ID]	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
GAP: Brazil	6×(100)	7-10	7	--	--	--	--
GAP: Canada	6×(80)	7	7	--	--	--	--
GAP: USA [Tuberous and corm vegetables]	1 in-furrow at-planting (178) + 9×(80)	7-10	7	--	--	--	--
Eaton Township, PQ (2001) Canada *Shepody* 01	10×(~80)	6-8	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	RA-3093
New Glasgow, PE (2001) Canada *Russett Burbank EII* 02	10×(~80)	6-8	8	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	

Location (Year) Country *Variety* Site ID [Study ID]	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
New Glasgow, PE (2001) Canada *Yukon Gold E4* 04	8×(~80) + 154	6	0	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
			1	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
			3	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
			7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
Nictaux, NS (2001) Canada *Superior* 03	6×(~80) + 2×(~160)	6-8	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Sheffild Mills, NS (2001) Canada *Atlantic* 05	10×(~80)	6-8	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
St-Paul d'Abbotsford, PQ (2001) Canada *Chiefton* 06	10×(~80)	6-7	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Medicine Hat, AB (2001) Canada *Russett Burbank* 07	10×(~81)	6-8	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Abbotsford, BC (2001) Canada *Russett Burbank* 08	10×(~82)	6-9	7	< 0.01, 0.010 (0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Leduc, AB (2001) Canada *Yukon Gold* 09	10×(~80)	6-7	8	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Northwood, ND (2000) USA *Atlantic* Plot 2	10×(~82)	6-8	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	RA-3075
Northwood, ND (2000) USA *Atlantic* Plot 3	10×(~82)	6-8	7	< 0.01, 0.010 (0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
Ibipora, Parana (2000) Brazil *Bintje* Plot 1	9×(100)	7	0	< 0.05	n.r.	Not applicable	RA-3202A
			3	< 0.05	n.r.	Not applicable	
			7	< 0.05	n.r.	Not applicable	
			14	< 0.05	n.r.	Not applicable	
Ibipora, Parana (2000) Brazil *Bintje* Plot 2	9×(200)	7	0	< 0.05	n.r.	Not applicable	
			3	< 0.05	n.r.	Not	

Location (Year) Country *Variety* Site ID [Study ID]	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
						applicable	
			7	< 0.05	n.r.	Not applicable	
			14	< 0.05	n.r.	Not applicable	
Botucatu, Sao Paulo (2001) Brazil *Bintje* Plot 2	6×(100)	7	3	< 0.05	n.r.	Not applicable	
Botucatu, Sao Paulo (2001) Brazil *Bintje* Plot 3	6×(200)	7	3	< 0.05	n.r.	Not applicable	
Engenheiro Coelho, Sao Paulo (2000) Brazil *Bintje* Plot 2	6×(100)	5-8	3	< 0.05	n.r.	Not applicable	
Engenheiro Coelho, Sao Paulo (2000) Brazil Bintje Plot 3	6×(200)	5-8	3	< 0.05	n.r.	Not applicable	
North Rose, NY (1999) USA *Green Mountain* A	10×(~80)	7	7	< 0.01, < 0.01 (≤ 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	RA-3066
Orno, ME (1999) USA *FL-1533* B	10×(~80)	7	7	< 0.01, < 0.01 (≤ 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Cary, NC (1999) USA *Kennebec* C	10×(~80)	7-8	7	< 0.01, < 0.01 (≤ 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Live Oak, FL (1999) USA *Red Pontiac* D	10×(~81)	6-8	7	< 0.01, < 0.01 (≤ 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Northwood, ND (1999) USA *Atlantic* E	10×(~80)	6-8	0	0.010, < 0.01 (0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
			1	0.010, 0.010 (0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
			3	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
			7	< 0.01, < 0.01 (≤ 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Fisher, MN (1999) USA *Red Norland* F	8×(~80) + 159	6-8	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
Arkansaw, WI (1999) USA *Russett Burbank* G	10×(~80)	6-8	7	< 0.01, < 0.01 (≤ 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Macon, MO (1999)	4×(~83) + 3×(~167)	4-10	7	< 0.01,	< 0.01,	< 0.025,	

Location (Year) Country *Variety* Site ID [Study ID]	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
USA *Irish Cobbler* H				< 0.01 (< 0.010)	< 0.01 (< 0.010)	< 0.025 (< 0.025)	
New Holland, OH (1999) USA *Landsglad* I	10×(~80)	6-8	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Center, CO (1999) USA *Norkotah* J	10×(~80)	7	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Kerman, CA (1999) USA *White Rose* K	10×(~80)	7	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Rupert, ID (1999) USA *Russett Burbank* L	10×(~80)	6-8	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Minidoka, ID (1999) USA *Russett Burbank* M	10×(~80)	6-7	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
American Falls, ID (1999) USA *Russett Burbank* N	10×(~79)	7	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Payette, ID (1999) USA *Russett Burbank* O	10×(~80)	7	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Hillsboro, OR (1999) USA *Russett Burbank* P	10×(~80)	7	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Yakima, WA (1999) USA *Norkotah* Q	10×(~80)	5-8	0	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
			1	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
			3	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< 0.025)	
			7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Ephrata, WA (1999) USA *Russett Burbank* R2	10×(~80)	5-7	7	< 0.01, < 0.01 (< 0.010)	< 0.01, < 0.01 (< 0.010)	< 0.025, < 0.025 (< *0.025*)	
Ephrata, WA (1999) USA *Russett Burbank* R3	9×(~80) + 1×(~800)	5-7	3	< 0.01, 0.020 (0.011)	< 0.01, < 0.01 (0.010)	< 0.025, < 0.035 (< 0.030)	

[a] Molecular weight ratio cyazofamid:CCIM = 1.49. Combined = Cyazofamid residue + (CCIM residue × 1.49)

n.r. = Not Reported

Table 50 Residues of cyazofamid and CCIM in **sweet basil** following foliar application in the USA (Study RA-3197)

Location (Year) *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Fresh/ Dried	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)
GAP: USA	9×(88)	7-10	0	--	--	--	--
Salisbury, MD (2009) *Genovese Compact Improved* 10118.09-MD04 Field Grown	9×(~90)	6-8	0	Fresh	2.3, 2.7 (2.5)	0.041, 0.046 (0.044)	2.4, 2.8 (*2.6*)
			4		1.1, 0.82 (0.96)	0.013, 0.013 (0.013)	1.1, 0.84 (0.98)
			7		0.32, 0.69 (0.51)	< 0.01, 0.012 (0.011)	< 0.33, 0.71 (< 0.52)
			10		0.52, 0.89 (0.71)	< 0.01, < 0.01 (< 0.010)	< 0.53, < 0.90 (< 0.72)
			14		0.13, 0.51 (0.32)	< 0.01, 0.014 (0.012)	< 0.14, 0.53 (< 0.34)
Clinton, NC (2009) *Genovese* 10118.09-NC16 Field Grown	9×(~87)	6-7	0	Fresh	10, 8.7 (9.4)	0.19, 0.18 (0.18)	**10**, 9.0 (*9.6*)
Salinas, CA (2009) *Italian Large Leaf* 10118.09-CA*82 Field Grown	9×(~90)	6-8	0	Fresh	3.2, 2.6 (2.9)	0.029, 0.025 (0.027)	3.2, 2.6 (*2.9*)
Maricopa, AZ (2009) *Lemon* 10118.09-AZ*01 Field Grown	9×(~87)	6-8	0	Fresh	6.9, 7.6 (7.2)	0.062, 0.071 (0.066)	7.0, 7.7 (*7.3*)

[a] Molecular weight ratio cyazofamid:CCIM = 1.49. Combined = Cyazofamid residue + (CCIM residue × 1.49)

Table 51 Residues of cyazofamid and CCIM in fresh and dried **hops** cones following foliar application

Location (Year) Country *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
GAP: USA	6×(80)	7-10	3	--	--	--	--
Fresh Cones							
Wolnzach-Gebrontshausen, Pfaffenhofen (2011) Germany *Hallertauer Tradition* FHO11-RE01-DE01	6×(93-193)	8-14	0	0.47	< 0.01	< 0.48	RA-3190
			6	0.42	< 0.01	< 0.43	
			13	0.42	< 0.01	< 0.43	
Wolnzach-Gebrontshausen, Pfaffenhofen (2011) Germany *Perle* FHO11-RE01-DE02	6×(95-197)	9-14	0	0.49	< 0.01	< 0.50	
			7	0.11	< 0.01	< 0.12	
			14	0.050	< 0.01	< 0.065	
Wolnzach-Gebrontshausen, Pfaffenhofen (2011)	6×(88-204)	10-16	0	0.49	< 0.01	< 0.50	

Location (Year) Country *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
Germany *Spalter Select* FHO11-RE01-DE03							
			7	0.23	< 0.01	< 0.24	
			14	0.21	< 0.01	< 0.22	
Wolnzach-Gebrontshausen, Pfaffenhofen (2011) Germany *Hallertauer Magnum* FHO11-RE01-DE04	6×(95-188)	11-15	0	0.23	< 0.01	< 0.24	
			7	0.040	< 0.01	< 0.055	
			14	0.050	< 0.01	< 0.065	
Wolnzach-Gebrontshausen, Pfaffenhofen (2012) Germany *Hallertauer Tradition* FHO12-RE01-DE01	6×(89-198)	12-16	21	1.02	< 0.01	<1.0	RA-3169
Wolnzach-Gebrontshausen, Pfaffenhofen (2012) Germany *Perle* FHO12-RE01-DE02	6×(83-195)	11-16	21	0.48	< 0.01	< 0.49	
Wolnzach-Gebrontshausen, Pfaffenhofen (2012) Germany *Spalter Select* FHO12-RE01-DE03	6×(97-193)	11-16	21	0.47	< 0.01	< 0.48	
Wolnzach-Gebrontshausen, Pfaffenhofen (2012) Germany *Hallertauer Magnum* FHO12-RE01-DE04	6×(86-192)	11-16	21	0.20	< 0.01	< 0.21	
Sondershausen, Kyffhauser (2013) Germany *Northern Brewer* FHO13-RE01-DE01	6×(96-201)	14	21	0.67	< 0.01	< 0.68	RA-3188
Golzern, Leipzig (2013) Germany *Nugget* FHO13-RE01-DE02	6×(96-184)	11-17	21	0.32	< 0.01	< 0.33	
Dried Cones							
Parma, ID (2007) USA *Nugget* ID01	6×(~79)	7-8	4	5.7, 6.9 (6.3)	0.13, 0.13 (0.13)	5.9, 7.1 (*6.5*)	RA-3127
Hubbard, OR (2007) USA *Nugget* OR04	6×(~82)	6-8	2	2.8, 3.6 (3.2)	0.21, 0.28 (0.24)	3.1, 4.0 (*3.6*)	
Benton County, WA (2007) USA *Nugget* WA02	6×(~83)	6-8	3	2.5, 2.5 (2.5)	0.42, 0.45 (0.44)	3.1, 3.2 (*3.1*)	
Norfolk, ON (2013) Canada *Nugget* A9823.13-ON12	6×(~82)	6-7	0	14	0.30	14	A9823
			4	7.6, 5.8, 7.7, **7.9** (7.2)	0.17, 0.22, 0.18, 0.12 (0.17)	7.9, 6.1, 8.0, **8.1** (7.5)	
			7	7.4	0.10	7.5	
			14	4.9	0.073	5.0	
Prosser, WA (2013) USA	6×(~82)	6-8	3	3.5, 3.4, 1.9, 2.9	0.19, 0.20, 0.16, 0.17	3.8, 3.7, 2.1, 3.2 (*3.2*)	

Location (Year) Country *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)	Reference
Tomahawk A9823.13-WA03				(2.9)	(0.18)		
Wolnzach-Gebrontshausen, Pfaffenhofen (2011) Germany *Hallertauer Tradition* FHO11-RE01-DE01	6×(93-193)	8-14	20	0.32	0.09	0.45	RA-3190
			27	0.26	0.12	0.44	
Wolnzach-Gebrontshausen, Pfaffenhofen (2011) Germany *Perle* FHO11-RE01-DE02	6×(95-197)	9-14	21	0.20	0.17	0.45	
			28	0.5	0.28	0.92	
Wolnzach-Gebrontshausen, Pfaffenhofen (2011) Germany *Spalter Select* FHO11-RE01-DE03	6×(88-204)	10-16	21	4.6	1.3	6.5	
			28	3.1	0.75	4.2	
Wolnzach-Gebrontshausen, Pfaffenhofen (2011) Germany *Hallertauer Magnum* FHO11-RE01-DE04	6×(95-188)	11-15	21	1.0	0.40	1.6	
			28	1.1	0.37	1.7	
Wolnzach-Gebrontshausen, Pfaffenhofen (2012) Germany *Hallertauer Tradition* FHO12-RE01-DE01	6×(89-198)	12-16	21	4.7	0.40	5.3	RA-3169
Wolnzach-Gebrontshausen, Pfaffenhofen (2012) Germany *Perle* FHO12-RE01-DE02	6×(83-195)	11-16	21	4.5	0.36	5.0	
Wolnzach-Gebrontshausen, Pfaffenhofen (2012) Germany *Spalter Select* FHO12-RE01-DE03	6×(97-193)	11-16	21	3.6	0.22	3.9	
Wolnzach-Gebrontshausen, Pfaffenhofen (2012) Germany *Hallertauer Magnum* FHO12-RE01-DE04	6×(86-192)	11-16	21	2.1	0.33	2.6	
Sondershausen, Kyffhauser (2013) Germany *Northern Brewer* FHO13-RE01-DE01	6×(96-201)	14	21	9.3	0.95	11	RA-3188
Golzern, Leipzig (2013) Germany *Nugget* FHO13-RE01-DE02	6×(96-184)	11-17	21	4.8	1.0	6.3	

[a] Molecular weight ratio cyazofamid:CCIM = 1.49. Combined = Cyazofamid residue + (CCIM residue × 1.49)

FATE OF RESIDUES IN STORAGE AND PROCESSING

Nature of the residue during processing

High-temperature hydrolysis

High-temperature hydrolysis of cyazofamid was investigated by J. Bernal (2014, RA-3186). In the study, [^{14}C]cyazofamid (radiolabel position not specified) was spiked into buffered solutions, in triplicate, 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 analysed by LSC for total radioactivity and by radio-HPLC for determination of hydrolysis products. Mass balance of radioactivity after processing was 102, 107, and 116% for 90 °C/pH4, 100 °C/pH5, and 120 °C/pH6, respectively. The correlation between temperature and mass balance was surmised in the study report to be due to better solubilisation after heating.

Radio-HPLC analysis showed a single peak prior to processing and two peaks after processing. The second peak was shown to be the cyazofamid metabolite CCIM. Under pasteurisation conditions, most of the cyazofamid was converted to CCIM; under the other two conditions tested, 100% of the test material converted to CCIM (Table 52).

Table 52 High-temperature hydrolysis radio-HPLC results for cyazofamid

	% of Radiolabel			
	Start		End	
Conditions	Cyazofamid	CCIM	Cyazofamid	CCIM
90°C, 20 minutes, pH 4	100	0	21	79
	100	0	18	82
	100	0	16	84
100°C, 60 minutes, pH 5	100	0	0	100
	100	0	0	100
	100	0	0	100
120°C, 20 minutes, pH 6	100	0	0	100
	100	0	0	100
	100	0	0	100

Residues after processing

The Meeting received data depicting residues of cyazofamid and CCIM in raw and processed commodities of basil, hops, grape, tomato, and potato.

For basil, fresh leaves and stem from field trial samples (see Table 50[a] Molecular weight ratio cyazofamid:CCIM = 1.49. Combined = Cyazofamid residue + (CCIM residue × 1.49)

) were dried according to "local commercial practices," with a recommended procedure of placing the sample in a drier, at 43–49 °C, for 24 hours. Stability of CCIM has not been demonstrated for the storage durations used in the study.

Table 53 Residues of cyazofamid and CCIM in **dried basil** following foliar applications in the USA (Study RA-3197)

Location (Year) *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Fresh/ Dried	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)
GAP: USA	9×(88)	7-10	0	--	--	--	--
Salisbury, MD (2009) *Genovese Compact Improved* 10118.09-MD04 Field Grown	9×(~90)	6-8	0	Dried	9.3, 10 (9.7)	1.0, 1.1 (1.1)	11, 12 (*11*)
Clinton, NC (2009) *Genovese*	9×(~87)	6-7	0	Dried	14, 12 (13)	11, 11 (11)	30, 28

Location (Year) *Variety* Site ID	Applications # × (rate) (g ai/ha)	RTIs (days)	DAT	Fresh/ Dried	Cyazofamid (mg/kg)	CCIM (mg/kg)	Combined [a] (mg eq./kg)
10118.09-NC16 Field Grown							(*29*)
Salinas, CA (2009) *Italian Large Leaf* 10118.09-CA*82 Field Grown	9×(~90)	6-8	0	Dried	15, 14 (14)	10, 10 (10)	30, 29 (*29*)
Maricopa, AZ (2009) *Lemon* 10118.09-AZ*01 Field Grown	9×(~87)	6-8	0	Dried	36, 43 (40)	2.0, 2.2 (2.1)	39, **46** (*43*)
Citra, FL (2010) *Genova* 10118.09-FL29 Glasshouse Grown	9×(89)	7-8	0	Dried	14, 15 (14)	0.062, 0.069 (0.066)	14, 15 (*15*)
Parlier, CA (2009) *Aroma 2 OG* 10118.09-CA81 Glasshouse Grown	9×(~87)	7	0	Dried	15, 12 (14)	0.10, 0.072 (0.086)	15, 12 (*14*)

[a] Molecular weight ratio cyazofamid:CCIM = 1.49. Combined = Cyazofamid residue + (CCIM residue × 1.49)

Hop cones from plots treated six times at 96–201 g ai/ha were harvested, dried, and processed into beer. Dried cones were stored, frozen, for ca. 90 days prior to processing into beer. Residues of both cyazofamid and CCIM were reduced upon processing (Table 54).

In one of the grape studies conducted in the USA (RA-3058), grapes harvested from trials approximating the USA GAP were processed into raisins by sun drying for 37 days. Residues of cyazofamid and CCIM decreased during processing of grapes into raisins. Grapes/raisins from this study were stored frozen for a total of 249 days from sampling to analysis. For both cyazofamid and CCIM, the processing factor is <1, indicating that residues are reduced upon processing.

In grape trials conducted in France (RA-3082), Germany (RA-3083), and Italy (RA-3086), samples of treated grapes were processed into must and wine using simulated commercial practices suitable for each grape type and region. In the case of France, wine was divided into young wine and mature wine, and both must and wine were assayed before and after pasteurisation. In the trial from Germany, wine was divided into young and mature wine; processed products were not pasteurised. Frozen storage time for must and wine samples ranged from 237 to 412 days. For must, the processing factors for cyazofamid were rather variable, ranging from 0.21 to 2.3. There is a trend for cyazofamid to concentrate in must from red varieties but not in must from white varieties. NOTE: Method issue for raisins

Tomatoes for processing were obtained from a field trial which received five applications at the target rate and a final application at a 3X exaggerated rate (RA-3065). Samples were processed into paste and puree (RA-3065) using simulated commercial practices. Residues of cyazofamid did not show concentration in either paste or puree; however, residues of CCIM may concentrate in those commodities.

For potato, samples of tubers taken from a field-trial plot receiving nine applications at the target GAP rate and a tenth application at a 10× exaggerated rate were processed into wet peels, potato flakes, and potato chips (RA-3066). The processing followed simulated commercial practices. Samples were stored frozen from 95 to 422 days. Residues were <LOQ in all samples; therefore, meaningful processing factors could not be calculated.

Table 54 Residues of cyazofamid, CCIM, and combined (cyazofamid + CCIM as cyazofamid equivalents) in raw and processed commodities for estimation of long-term dietary exposure

	Residues (mg/kg)							
	Raw Commodity			Processed Commodity				
Processed Commodity	Cyaz.	CCIM	Combined	Cyaz.	CCIM	Combined	Proc. factor	Reference
Grape								
Dried (raisin)	0.44, 0.42	0.03, 0.02	0.48, 0.45 (0.47)	0.08, 0.07	0.02, 0.02	0.11, 0.10 (0.10)	0.22	RA-3058
Must	0.04, 0.05	< 0.01, < 0.01	0.06, 0.07 (0.06)	0.09, 0.09	< 0.01, < 0.01	0.10, 0.10 (0.10)	1.8	RA-3082
	0.03, 0.03	< 0.01, < 0.01	0.05, 0.05 (0.05)	0.07, 0.07	< 0.01, < 0.01	0.09, 0.09 (0.09)	1.9	
	0.11, 0.13	< 0.01, < 0.01	0.12, 0.14 (0.13)	0.04, 0.01	< 0.01, < 0.01	0.06, 0.03 (0.04)	0.30	
	0.03, 0.04	< 0.01, < 0.01	0.05, 0.06 (0.05)	0.01, 0.01	< 0.01, < 0.01	0.03, 0.03 (0.03)	0.50	RA-3083
	0.03, 0.04	< 0.01, < 0.01	0.05, 0.06 (0.05)	0.01, 0.01	0.01, 0.01	0.03, 0.03 (0.03)	0.50	RA-3083
	0.03, 0.04	< 0.01, < 0.01	0.05, 0.06 (0.05)	0.05, 0.05	< 0.01, < 0.01	0.07, 0.07 (0.07)	1.3	RA-3086
	0.03, 0.04	< 0.01, < 0.01	0.05, 0.06 (0.05)	0.02, 0.02	0.006, 0.007	0.03, 0.03 (0.03)	0.59	
Wine	0.04, 0.05	< 0.01, < 0.01	0.06, 0.07 (0.06)	< 0.01, < 0.01	0.02, 0.02	0.04, 0.04 (0.04)	0.66	RA-3082
	0.11, 0.13	< 0.01, < 0.01	0.12, 0.14 (0.13)	< 0.01, < 0.01	0.01, < 0.01	0.03, 0.03 (0.03)	0.18	
	0.03, 0.03	< 0.01, < 0.01	0.05, 0.05 (0.05)	< 0.01, < 0.01	0.01, 0.01	0.03, 0.03 (0.03)	0.55	
	0.03, 0.04	< 0.01, < 0.01	0.05, 0.06 (0.05)	< 0.01, < 0.01	0.01, 0.01	0.03, 0.03 (0.03)	0.50	RA-3086
	0.03, 0.04	< 0.01, < 0.01	0.05, 0.06 (0.05)	< 0.01, < 0.01	< 0.01, < 0.01	0.03, 0.03 (0.03)	0.50	RA-3083
	0.03, 0.04	< 0.01, < 0.01	0.05, 0.06 (0.05)	< 0.01, < 0.01	0.01, 0.01	0.03, 0.03 (0.03)	0.50	
	0.03, 0.04	< 0.01, < 0.01	0.05, 0.06 (0.05)	< 0.01, < 0.01	< 0.01, < 0.01	0.03, 0.03 (0.03)	0.50	
	0.03, 0.04	< 0.01, < 0.01	0.05, 0.06 (0.05)	< 0.01, < 0.01	< 0.01, < 0.01	0.03, 0.03 (0.03)	0.50	
	0.03, 0.04	< 0.01, < 0.01	0.05, 0.06 (0.05)	< 0.01, < 0.01	< 0.01, < 0.01	0.03, 0.03 (0.03)	0.50	
	0.03, 0.04	< 0.01, < 0.01	0.05, 0.06 (0.05)	< 0.01, < 0.01	< 0.01, < 0.01	0.03, 0.03 (0.03)	0.50	
Hops								
Beer	9.43, 7.35	1.7, 1.3	12, 9.3 (11)	< 0.01, < 0.01	< 0.01, < 0.01	0.03, 0.03 (0.03)	0.0023	RA-3188
Tomato								
Paste	0.04, 0.04	< 0.01, < 0.01	0.06, 0.06 (0.06)	< 0.01, < 0.01	0.02, 0.02	0.04, 0.04 (0.04)	0.72	RA-3065
Puree	0.04, 0.04	< 0.01, < 0.01	0.06, 0.06 (0.06)	< 0.01, < 0.01	0.01, 0.01	0.03, 0.03 (0.03)	0.45	

Table 55 Residues of cyazofamid, CCIM, and combined (cyazofamid as CCIM equivalents + CCIM) in raw and processed commodities for estimation of short-term dietary exposure

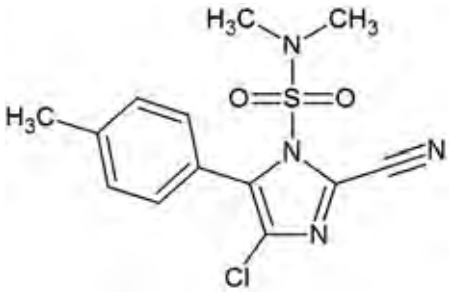
	Residues (mg/kg)					
	Raw Commodity			Processed Commodity		
Processed Commodity	Cyaz.	CCIM	Combined	CCIM	Processing factor	Reference
Grape						
Dried (raisin)	0.44, 0.42	0.03, 0.02	0.33, 0.30 (0.31)	0.02, 0.02 (0.02)	0.064	RA-3058
Must	0.04, 0.05	< 0.01, < 0.01	0.04, 0.04 (0.040)	< 0.01, < 0.01 (0.01)	0.25	RA-3082
	0.03, 0.03	< 0.01, < 0.01	0.03, 0.03 (0.030)	< 0.01, < 0.01 (0.01)	0.33	
	0.11, 0.13	< 0.01, < 0.01	0.08, 0.10 (0.091)	< 0.01, < 0.01 (0.01)	0.11	

	0.03, 0.04	< 0.01, < 0.01	0.03, 0.04 (0.033)	< 0.01, < 0.01 (0.01)	0.30	RA-3083
	0.03, 0.04	< 0.01, < 0.01	0.03, 0.04 (0.033)	0.01, 0.01 (0.01)	0.30	RA-3083
	0.03, 0.04	< 0.01, < 0.01	0.03, 0.04 (0.033)	< 0.01, < 0.01 (0.01)	0.30	RA-3086
	0.03, 0.04	< 0.01, < 0.01	0.03, 0.04 (0.033)	0.006, 0.007 (0.01)	0.19	
Wine	0.04, 0.05	< 0.01, < 0.01	0.04, 0.04 (0.040)	0.02, 0.02 (0.02)	0.50	RA-3082
	0.11, 0.13	< 0.01, < 0.01	0.08, 0.10 (0.091)	0.01, < 0.01 (0.01)	0.11	
	0.03, 0.03	< 0.01, < 0.01	0.03, 0.03 (0.030)	0.01, 0.01 (0.01)	0.33	
	0.03, 0.04	< 0.01, < 0.01	0.03, 0.04 (0.033)	0.01, 0.01 (0.01)	0.30	RA-3086
	0.03, 0.04	< 0.01, < 0.01	0.03, 0.04 (0.033)	< 0.01, < 0.01 (0.01)	0.30	RA-3083
	0.03, 0.04	< 0.01, < 0.01	0.03, 0.04 (0.033)	0.01, 0.01 (0.01)	0.30	
	0.03, 0.04	< 0.01, < 0.01	0.03, 0.04 (0.033)	< 0.01, < 0.01 (0.01)	0.30	
	0.03, 0.04	< 0.01, < 0.01	0.03, 0.04 (0.033)	< 0.01, < 0.01 (0.01)	0.30	
	0.03, 0.04	< 0.01, < 0.01	0.03, 0.04 (0.033)	< 0.01, < 0.01 (0.01)	0.30	
	0.03, 0.04	< 0.01, < 0.01	0.03, 0.04 (0.033)	< 0.01, < 0.01 (0.01)	0.30	
Hops						
Beer	9.43, 7.35	1.7, 1.3	8.0, 6.2 (7.1)	< 0.01, < 0.01 (0.01)	0.0014	RA-3188
Tomato						
Paste	0.04, 0.04	< 0.01, < 0.01	0.04, 0.04 (0.037)	0.02, 0.02 (0.02)	0.54	RA-3065
Puree	0.04, 0.04	< 0.01, < 0.01	0.04, 0.04 (0.037)	0.01, 0.01 (0.01)	0.27	

APPRAISAL

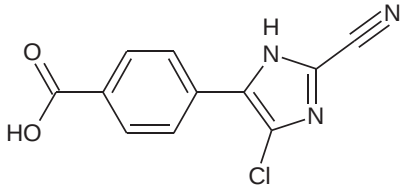
Cyazofamid (ISO common name, published) is a fungicide belonging to both the cyano-imidazole and sulphonamide classes of compounds. The biochemical mode of action is inhibition of all stages of fungal development. It was considered for the first time by the 2015 JMPR for toxicology and for residues.

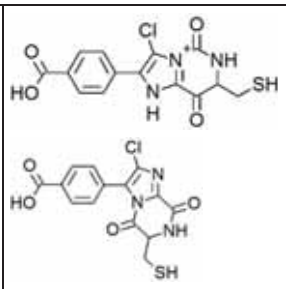
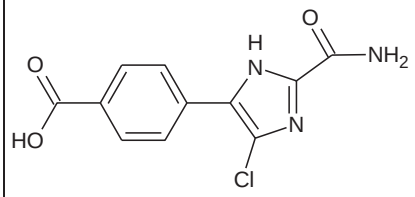
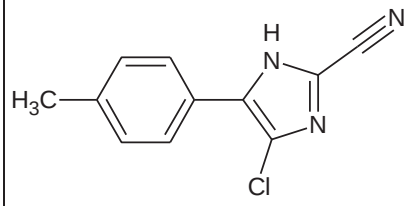
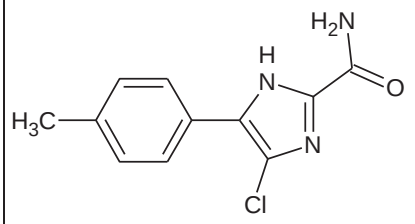
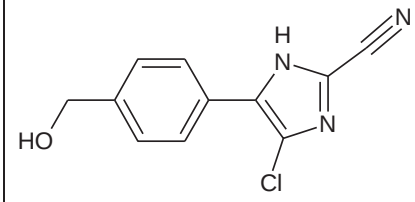
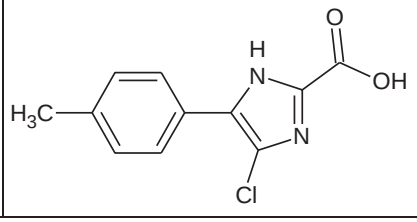
The IUPAC name for cyazofamid is 4-chloro-2-cyano-*N*,*N*-dimethyl-5-*p*-tolylimidazole-1-sulfonamide and the CA name is 4-chloro-2-cyano-N,N-dimethyl-5-(4-methylphenyl)-1H-imidazole-1-sulfonamide, with registry number 120116-88-3.



Cyazofamid with ^{14}C radiolabelling in the benzene ring or in the imidazole ring was used in the metabolism and environmental fate studies. In this appraisal, these positions are referred to as the Bz and Im labels, respectively.

The following abbreviations, along with IUPAC names and structures, are used for the metabolites discussed in this appraisal:

CCBA	4-(4-chloro-2-cyanoimidazol-5-yl)benzoic acid	

CCBA (cysteine conjugates)		
CCBA-AM	4-(4-chloro-2-amidoimidazol-5-yl)benzoic acid	
CCIM	4-chloro-5-p-tolylimidazole-2-carbonitrile	
CCIM-AM	4-chloro-5-p-tolylimidazole-2-carboxamide	
CHCN	4-chloro-5-(4-hydroxymethylphenyl) imidazole-2-carbonitrile	
CTCA	4-chloro-5-p-tolylimidazole-2-carboxylic acid	

Plant metabolism

The Meeting received studies depicting the metabolism of cyazofamid in grapes, tomatoes, lettuce, and potatoes. All of the studies were conducted with cyazofamid which was radiolabelled, separately, in the benzene and imidazole rings.

Cyazofamid was applied five times, at ca. 100 g ai/ha at 21–25-day intervals, to grapevines growing in the field. Grapes were harvested 44 days after the last application (DALA). TRR in grapes was greater following treatment with Bz-labelled material (0.53 mg eq/kg, 0.89% of applied) than with Im-labelled material (0.31 mg eq/kg, 0.62% of applied). When processed into wine, radioactivity distributed primarily into the marc (wet pomace; 70% TRR, 3.7 mg eq/kg), with significantly lesser amounts in the vin de goutte (juice prior to pressing; 15% TRR, 0.21 mg eq/L) and vin de presse (juice after pressing; 10% TRR,

0.32 mg eq/L), indicating radioactivity may have been associated with surface residues. For grapes processed into juice, a similar trend was observed: 54% TRR (1.4mg eq/kg) in the marc and 33% TRR (0.3 mg eq/L) in the juice. Neither characterization nor identification of residues was reported in the study.

Metabolism of cyazofamid on tomatoes was investigated following treatment of field-grown plants with four foliar applications of cyazofamid at approximately 60, 95, 95, and 95 g ai/ha at 7-day intervals. In fruits harvested 1 DALA, TRR (surface rinses + juice + pulp) was 0.08 mg eq/kg from the Im treatment and 0.29 mg eq/kg from the Bz treatment. Of the total residue, the majority was contained in the surface rinse (54% and 83% for the Im and Bz labels, respectively). Of the radioactivity remaining in the fruits after rinsing, 71–81% TRR (ca. 0.033 mg eq/kg) was associated with the pulp and 13–29% TRR (ca. 5.5 mg eq/kg) was associated with the juice. Extraction of the pulp with, sequentially, hexane, ethyl acetate, and water released 75% of the radioactivity from the Bz-labelled sample and 90% from the Im-labelled sample. The principal residue from both labels was parent cyazofamid (ca. 78% TRR; 0.064 mg eq/kg Im, 0.22 mg/kg Bz), which is not unexpected given the short interval between application and harvest. The next-highest identified residue was CCIM (ca. 4–5% TRR, 0.004–0.13 mg/kg). A chromatographic fraction which was shown to consist primarily of radiolabelled sugars and citric acid accounted for 2.5–5.4% TRR (0.002–0.16 mg eq/kg), indicating breakdown of cyazofamid and incorporation into natural plant constituents.

Metabolism in lettuce was investigated following foliar treatment of glasshouse-grown plants. Three applications were made at a nominal rate of 100 g ai/ha on 14-day intervals. The test material was a mixture of cyazofamid labelled, separately, in the Im and Bz positions (in a 1:1 ratio). Lettuce leaves were harvested 14 DALA. Total radioactive residues were 0.85 mg eq/kg in the harvested leaves and 97% of the residues were extracted with ACN:H_2O (60:40, v/v with 0.1% acetic acid). Cyazofamid made up 89% of the TRR (0.76 mg/kg). No other compounds occurred at > 10% TRR. CCIM occurred at 3.7% TRR (0.031 mg/kg). Radioactivity in natural plant constituents occurred at 3.3% TRR (0.028 mg eq/kg). Based on analysis of the post-extraction solids (PES), those plant constituents consisted of starch and other water-soluble polysaccharides, protein, cellulose, and lignin.

Metabolism of cyazofamid was investigated in both field-grown and glasshouse-grown potatoes. In the field study, three foliar applications were made at rates of 100 or 400 g ai/ha. In the glasshouse study, five foliar applications were made at a rate of 400 g ai/ha. In both cases, applications were made on a 7-day interval and harvesting was done 7 DALA. In foliage, nearly all of the residue was cyazofamid. In tubers, the majority of the radioactivity was associated with the pulp. Sequential extractions of the pulp with ACN, ACN:H_2O (80:20, v/v), and ACN:H_2O (50:50, v/v) released 43 to 70% of the radioactivity, with the Bz-labelled samples generally being at the higher end of that range. In rinses of the tubers, the majority of the residue was cyazofamid (67–80% TRR, 0.0009–0.0018 mg/kg) and CCIM (14–20% TRR, 0.003 mg/kg); whereas in the tuber itself, the majority of the radioactivity was associated with starch (23–30% TRR, 0.005 mg/kg). Cyazofamid and CCIM were both < 5% TRR in tubers.

In plant metabolism studies with identification of residues, cyazofamid was the major residue in aerial portions of the plants and there was consistent demonstration of incorporation of radioactivity into natural plant components. The available data indicate that cyazofamid is translocated. The metabolite CCIM was consistently identified in these studies but never occurred at greater than 10% TRR.

Animal metabolism

The Meeting received studies elucidating the metabolism of cyazofamid in laboratory animals, lactating goats, and laying hens.

In rats, cyazofamid is well absorbed at doses relevant to dietary exposure, and rapidly metabolised, with the majority of excretion occurring via urine. In the plasma, there was no cyazofamid and the majority of radiolabel was CCIM. At 0.5 hours after a dose of [^{14}C-Bz]-

CCIM, all of the radiolabel in the stomach contents was CCIM, and most of the radiolabel in liver (76.5%) and plasma (67.9%) was CCIM. CCBA, the main metabolite seen in these tissues 0.5 hours after dosing with CCIM, was also found in the blood and liver from the animals dosed with cyazofamid. Concentrations in blood and liver were greater in the CCIM-dosed animals than that in cyazofamid treated animals, suggesting that CCIM was much more rapidly absorbed than cyazofamid.

In goats dosed for five consecutive days at approximately 32 mg/animal/day (Im) or 25 mg/animal/day (Bz; both equivalent to 10 ppm in the diet), overall recovery of radioactivity was ca. 60% of the administered dose (AD). Most of the recovered radioactivity was in urine and faeces, with only 0.22% (Im) or 0.18% (Bz) of the AD accounted for in tissues. Despite the low retention of radioactivity, sufficient residues were present to characterize and identify specific compounds in all tissues. Total radioactive residues (TRR) in urine and faeces appeared to plateau by Day 3 of dosing. In milk from Bz-treated goats, TRR remained near the limit of quantification (LOQ, 0.005 mg eq/kg) for the duration of the dosing period. TRR did not plateau during the dosing period for the Im label, rising steadily from 0.005 mg eq/kg to 0.10 mg eq/kg. Aside from this difference in milk, there was little difference in the behaviour of cyazofamid based on the position of the radiolabel. Solvent (ACN or ACN:H_2O) extracted 74% TRR, 90% TRR, and 100% TRR in muscle, milk, and fat, respectively, and sequential extraction with ACN and ACN:H_2O extracted 92% TRR from kidney. For liver, the same sequential solvents used for kidney extracted only ca. 50% TRR. An additional 45% TRR was released from liver, in total, using HCl, NaOH, and protease treatments of the post-extraction solids (PES). Liver and kidney contained the highest levels of radioactivity (ca. 0.1 mg eq/kg). In other tissues and in milk, radioactivity was approximately an order of magnitude lower than in liver/kidney. Cyazofamid residues were < 0.001 mg/kg (0.1–0.3% TRR) in all tissues. The principal residues in tissues and milk were CCBA (free or cysteine-conjugated), CCIM, and their amide analogs. Total CCBA-related residues ranged from 12% TRR (< 0.002 mg/kg; muscle) to 85% TRR (0.090 mg/kg; kidney), and total CCIM-related residues ranged from 5.3% TRR (0.006 mg/kg; kidney) to 39% TRR (< 0.003 mg/kg; fat); the highest concentrations of CCIM-related residues was in liver, at 0.016 mg/kg (14% TRR). The chromatographic system used in the goat metabolism studies was generally not able to separate CCBA and its cysteine conjugate, and those residues were typically the main residues in all tissues.

In hens dosed for five consecutive days at 1.1 mg/bird/day (10 ppm in the diet), total radioactive residues (TRR) in excreta accounted for approximately 85–90% of the dosed material, and < 0.1% of the AD was retained in tissues/eggs. Total radioactive residues were < 0.006 mg eq/kg in all samples of eggs, muscle, blood, fat, and skin. Residue plateau in eggs could not be assessed. Acetonitrile + ACN:H_2O extraction was not efficient at solubilizing residues in kidney (ca. 50% TRR) and liver (ca. 30% TRR); however, chemical and enzymatic treatment of the resulting PES was able to release the unextracted residues, resulting in 100% recovery of TRR. In kidney, the only identified compounds occurring at > 10% TRR were CCBA (solvent-extracted; 12% TRR, 0.0035–0.0064 mg/kg), and CHCN conjugates (not further identified; solvent-extracted; 17% TRR, 0.005–0.010 mg/kg and PES acid hydrolysate; 30–67% TRR, 0.003–0.010 mg/kg). Two unidentified fractions from the acid-hydrolysate treatment, CM-2 and CM-3, accounted for ca. 15% TRR (0.001 mg eq/kg) each. Residue profiles in liver were similar to those in kidney, consisting of CCBA (acid hydrolysate only, 14% TRR, 0.002 mg/kg), CHCN conjugates (solvent extract, 12% TRR, 0.011 mg eq/kg; acid hydrolysate, 47% TRR, 0.0073 mg eq/kg), and CM-2/CM-3 (acid hydrolysate, 13% TRR, 0.002 mg eq/kg).

Overall, the animal metabolism studies show that the majority (99+%) of the dosed radioactivity is excreted. In goat, the principal terminal residues are CCBA, CCIM, and their related conjugates and amides. In hens, the principal terminal residues are CCBA, CHCN, and their conjugates. Although CCBA is common to both species, the formation of that compound appears to occur through different pathways.

Environmental fate

Cyazofamid is prone to hydrolysis (25 °C, pH 4, 7, 9). The main product of hydrolysis at 25 °C at all pH levels was CCIM, which represented ca. 82% of the radioactivity at pHs of 4, 5, and 7, and 77% at pH 9. At pH 9, CCIM-AM was found at level of ca. 10% of the radioactivity. CCIM itself is stable to hydrolysis. Cyazofamid is also prone to photolysis in aqueous systems [DT_{50} of 30 minutes], forming CCIM and CCTS; both of which undergo further photolysis. In soil, photolysis does not appear to be a significant pathway for degradation since dissipation was similar in both irradiated and dark samples.

In an aerobic soil metabolism study, cyazofamid had DT_{50} estimates of ca. five days and DT_{90} estimates ranging from 16 to 25 days. The major residues following treatment with cyazofamid were CCIM (peak on Day 3, ca. 20% AD, ca 0.025 mg eq/kg), CCIM-AM (peak on Day 7, 13% AD, 0.016 mg eq/kg), and CTCA (peak ca. Day 20 at ca. 20% of the applied dose, 0.025 mg eq/kg). The aerobic soil metabolism study also showed an increase in unextracted residues over time (up to 64% at study termination) as well as production of $^{14}CO_2$ (14% of applied material by study termination). In unextracted residues, radioactivity was associated predominantly with fulvic acid as well as humin and humic acid.

In a study with confined rotational crops, bare soil was treated with 5× 100 g/ha (for both radiolabel positions on a 7-day interval). Crops of lettuce, carrot, and wheat were put into the treated soil at plant-back intervals (PBIs) of 31, 120, and 360 days. For all PBIs, residues in lettuce, carrot root, carrot tops (Days 120 and 360), and wheat grain were too low to allow residue identification/characterization. In carrot tops (Day 31 only), residues of CCBA (2.2% TRR), CCIM (10.4% TRR), CCIM-AM (39.5% TRR, 0.001 mg/kg), and cyazofamid (20.1% TRR, 0.003 mg/kg) were identified. In wheat chaff, forage and straw, residues were associated primarily with carbohydrates (0.01–0.20 mg eq/kg). Residues of cyazofamid and metabolites were $\leq$ 0.003 mg eq/kg in those matrices. No field rotational crop or field dissipation studies were provided. The Meeting concluded that the confined rotational crop study adequately reflects critical gap conditions and that residues are not expected in rotational crops following treatments according to the GAPs under consideration.

Overall, there are no indications that cyazofamid or any of its degradation products are expected to accumulate in soils. Significant dissipation pathways in an agricultural system appear to be hydrolysis and potentially photolysis. The DT_{90} estimates for cyazofamid in the aerobic soil metabolism study indicate that applications made more than ca. 1 month prior to harvest will not contribute significantly to the residue levels in harvested crops.

Methods of residue analysis

The Meeting received analytical methods for the analysis of cyazofamid and CCIM in plant matrices. Method validation recoveries were reported for grapes, cucurbit vegetables, root crops, Brassica vegetables, leafy vegetables, beans, peppers, and hops. Three methods for plant matrices underwent independent laboratory validation. No methods were submitted for analysis of animal materials or soil (aside from the techniques used in the studies with radiolabelled material).

In summary, extraction of residues in field trial samples was accomplished with ACN, ACN:H_2O (80:20, v/v), ACN:H_2O w/ 2% acetic acid (50:50, v/v), ACN:acetone (80:20, v/v) or acetone. Extracted residues were then generally cleaned up by partitioning into a non-polar organic solvent, with additional clean-up by solid-phase extraction (or in one case gel-permeation chromatography). Analysis of residues was by LC-MS/MS, HPLC-UV, or GC-NPD. Three methods underwent independent laboratory validation. For those methods, extraction of cyazofamid and CCIM is by ACN:acetone, H_2O followed by acetonitrile, or acetonitrile only. Clean-up varies across the three methods, consisting of traditional solid-phase extraction (C_{-18}), dispersive solid-phase extraction (magnesium sulphate, sodium chloride, sodium citrate dibasic sesquihydrate, and sodium citrate tribasic dehydrate), or liquid/liquid partitioning (hexane and methylene chloride, sequentially) with Florisil® solid-phase extraction. For the validated methods, residue separation and quantitation is by LC-MS/MS in positive ionisation mode or by HPLC-UV (280 nm). For LC-MS/MS, evaluated ion transitions [M+H+] for quantification were

325.1 m/z→108.0 m/z for cyazofamid and 218.3 m/z→183.2 m/z for CCIM. Confirmation of cyazofamid is made using the same ion transitions, but with a cyano column on a gradient mobile phase. Confirmation of CCIM is based on a mass transition of 218.3 m/z→139.2 m/z. Based on results from other submitted studies, a confirmatory transition for cyazofamid is available (325.1 m/z→261.2 m/z). Method validation testing resulted in percent recoveries for cyazofamid ranging from 70 to 111% (except for raisins at 67%) and for CCIM ranging from 74 to 120% (except for potato chips at 68%). For both analytes in all matrices, relative standard deviations of recovery were less than 21%. An LOQ of 0.01 mg/kg was achieved for all matrices and analytes.

The solvent used for extraction is similar to that used in the metabolism studies with lettuce and potato (the first two extraction solvents in the tomato metabolism study were much less polar). On that basis, the methods are expected to have adequate extraction efficiency of incurred residues.

Testing of cyazofamid and CCIM through the FDA PAM multi-residue method protocols demonstrated that for most protocols, the test compounds showed poor sensitivity, poor recovery, and/or poor chromatography. An open-literature study[1] demonstrated good recovery of both cyazofamid (80% to 105%) and CCIM (75% to 99%) from fortified crop samples using the QuEChERS method, with relative standard deviations of ≤ 16%.

Analytical methods are available for analysis of cyazofamid and CCIM in plant commodities. Analytical methods for the analysis of cyazofamid residues in animal commodities were not provided.

Stability of residues in stored analytical samples

The Meeting received data indicating that residues of cyazofamid and CCIM are stable under frozen conditions as follows:

Matrix	Cyazofamid	CCIM
Grape (homogenized)	Up to 8 days	No Data
Grape (unhomogenized)	At least 365 days	No Data
Basil (fresh)	At least 284 days	Not stable [a] (less than 284 days)
Basil (dried)	At least 297 days	Not stable [a] (less than 297 days)
Hops cones	At least 509 days	At least 509 days
Cabbage	At least 860 days	At least 860 days
Tomato	Up to 365 days	At least 1093 days
Lettuce	At least 634 days	At least 634 days
Mustard greens	At least 977 days	At least 977 days
Spinach	At least 949 days	At least 949 days
Bean plants with pods	At least 889 days	At least 889 days
Bean pods with seeds	At least 887 days	At least 887 days
Been seeds without pods	At least 140 days	At least 140 days
Dry beans	At least 400 days	At least 400 days
Carrot	Not stable [a] (less than 374 days)	Not stable [a] (less than 374 days)
Potato	Up to 181 days	Up to 181 days

[a] Residues were measured only at the indicated storage period, and the amount remaining was < 70%. Basil and carrot samples were analysed on the same day as extraction.

Cyazofamid and CCIM were demonstrated to be stable in extracts of oilseed rape and dry beans for at least four days. Stability of these analytes in extracts from other matrices was not reported.

Definition of the residue

In plants, parent cyazofamid was the only compound to occur as a major residue in metabolism studies, and suitable methods are available for analysis. CCIM was consistently identified in

[1] Lee, H. Kim, E, Lee, JH. Sung, JH, Choi, H, and Kim, JH. 2014. Bull Environ Contam Toxicol 93(5):586-90. Analysis of cyazofamid and its metabolite in the environmental and crop samples using LC-MS/MS.

metabolism studies as a minor residue and occurred at levels that were typically at least five-fold lower than cyazofamid, and typically < 0.01 mg/kg, in supervised residue trials. The Meeting considered residues of cyazofamid in rotational crops and concluded that uptake of residues from soil into rotational crops will be insignificant. Cyazofamid is expected to degrade during the production of processed products; especially those in which heating and/or hydrolysis occurs, resulting in the formation of CCIM. Nevertheless, levels of CCIM in processed commodities are generally low.

Cyazofamid exhibited low acute oral toxicity, and there was an absence of developmental toxicity and any other toxicological effects that would be likely to be elicited by a single dose. The primary plant metabolite, CCIM, however, was more acutely toxic than the parent compound and resulted in clinical signs at all doses tested in acute toxicity studies. For long-term exposures, the toxicity of CCIM is adequately addressed by parent cyazofamid.

The Meeting concluded that the residue definition for enforcement of MRLs in plant commodities is the parent compound, cyazofamid, only. Furthermore, the Meeting concluded that the residue definition for assessing long-term dietary intake from plant commodities is the combined residues of cyazofamid and CCIM, expressed as cyazofamid. An ARfD is not necessary for cyazofamid; however, the current Meeting established an ARfD for CCIM, and the residue definition for assessing short-term dietary intake from plant commodities is CCIM.

Studies depicting the nature of the residues in animals show generally low transfer of residues to tissues, milk, and eggs. Metabolism studies indicate that of the amount retained, residues are expected to be highest in offal and lower by approximately an order of magnitude in other matrices. Cyazofamid was not detected in any livestock matrix. The metabolite CCBA (free and as cysteine conjugates) was consistently found as a major residue (> 10% TRR, ranging from 0.002 mg/kg to 0.09 mg/kg) in goat and hen commodities. Data from goat kidney indicate that the cysteine conjugates form the majority of the CCBA residues (separate free/conjugated residue data were not reported for other matrices). The Meeting was uncertain about the relative amounts of free and cysteine-conjugated CCBA in tissues other than liver and about the availability of reference standards for cysteine-conjugated CCBA. The Meeting agreed not to establish residue definitions for livestock commodities.

Definition of the residue for compliance with the MRLs for plant commodities: *Cyazofamid.*

Definition of the residue for long-term dietary intake from plant commodities: *Cyazofamid and CCIM, expressed as cyazofamid.*

Noting that the current meeting established an ARfD for CCIM (in the absence of an ARfD for cyazofamid), the definition of the residue for short-term dietary intake from plant commodities is *CCIM*.

Definition of the residue for compliance with the MRLs and for dietary intake for animal commodities: *Not defined.*

Results of supervised residue trials on crops

The Meeting received supervised residue trial data for grapes, basil, hops, broccoli, cabbage, cucumber, summer squash, muskmelon, peppers, tomatoes, head and leaf lettuce, mustard greens, spinach, snap beans, lima beans, carrots, and potatoes. The trials were conducted in the USA for all crops, as well as Argentina, Europe (north and south), and Mexico for grapes; Germany for hops; Canada for lettuces; and Brazil and Canada for potatoes. For basil, residue data reflect both field and glasshouse growing conditions. All residue results are supported by adequate method and storage stability data unless otherwise noted.

For field trials with cabbage, all cabbage heads were cut in the field in order to reduce the size/weight of the sample; for lettuce and muskmelon, some samples were cut in the field. A comparison of the residue levels in field-cut and uncut samples indicates that field-cutting did not compromise the quality of the residue data obtained from field-cut samples.

For estimating dietary intake, combined residues (cyazofamid + CCIM) were calculated by multiplying the individual sample results from field trials of CCIM by the molecular weight factor of 1.49 (cyazofamid molecular weight = 324.8, CCIM molecular weight = 217.7) and adding the result to the corresponding residue of cyazofamid. For residues below the LOQ, the residue was assumed to be at the LOQ for calculation purposes; the "less than" designation was retained only if both residues were below the LOQ. Examples are shown below:

Cyazofamid	CCIM	Combined (expressed to two significant figures)
0.5 mg/kg	0.06 mg/kg	0.5 mg/kg + (0.06 mg/kg × 1.49) = 0.59 mg/kg
0.5 mg/kg	< 0.01 mg/kg	0.5 mg/kg + (0.01 mg/kg × 1.49) = 0.51 mg/kg
< 0.01 mg/kg	0.06 mg/kg	0.01 mg/kg + (0.06 mg/kg × 1.49) = 0.099 mg/kg
< 0.01 mg/kg	< 0.01 mg/kg	< 0.01 mg/kg + (< 0.01 mg/kg × 1.49) = < 0.025 mg/kg

Grapes

In grapes, the critical GAP based on highest application rate and shortest PHI is from the registration in Germany (eight foliar applications at 0.1 kg ai/ha on a 12- to 14-day interval with a 21-day PHI). Only a single field trial is available from Germany; however, additional residue trials matching the critical GAP are available from France, Italy, Spain, and Portugal. The Meeting noted that in all of these trials, grapes were stored as whole berries and, therefore, the residue levels are supported by the available storage stability data.

Mean field trial residues of cyazofamid from independent field trials matching the critical GAP (n=7) were: 0.01, 0.03, 0.04, 0.04, 0.06, 0.09, and 0.66 mg/kg.

Based on those data, the Meeting estimated a maximum residue level for grapes of 1.5 mg/kg.

From the trials cited above, residues of CCIM were (n=7): < 0.01 (7) mg/kg. For assessing short-term dietary intake from grapes, the HR, from a single sample, is 0.01 mg/kg.

From the trials cited above, the combined residues of cyazofamid and CCIM, expressed as cyazofamid, were (n=7): 0.02, 0.04, 0.05, 0.06, 0.08, 0.1, and 0.67 mg/kg. For assessing long-term dietary intake from grapes, the STMR from that data set is 0.06 mg/kg.

Brassica (Cole or Cabbage) Vegetables, Head Cabbage, Flowerhead Brassicas

The critical GAP is from the registration of cyazofamid on the Brassica (Cole) leafy vegetables crop group in the USA (one soil application at 0.753 kg ai/ha followed by five foliar applications at 0.08 kg ai/ha on a 7-10-day interval with a zero-day PHI). Supervised residue trials matching this GAP are available from the USA.

Mean field trial residues of cyazofamid in broccoli from independent field trials matching the critical GAP (n=5) were: 0.23, 0.34, 0.37, 0.46, and 0.84 mg/kg.

Mean field trial residues of cyazofamid in cabbage (with wrapper leaves) from independent field trials matching the critical GAP (n=9) were: 0.13, 0.15, 0.20, 0.25, 0.28, 0.30, 0.32, 0.56, and 0.75 mg/kg.

Noting that the residue trials address crops in the Codex commodity designation Brassica (Cole or cabbage) vegetables, head cabbage, flowerhead Brassicas and that the median residues from each crop are within a 5-fold range, the Meeting determined that a group MRL is appropriate. The cyazofamid residue data across the test crops are not significantly different by the Kruskal-Wallis test; therefore, the Meeting grouped the data together and is estimating a group maximum residue level for Brassica (Cole or cabbage) vegetables, head cabbage, flowerhead Brassicas based on the following cyazofamid residue data set (n=14): 0.13, 0.15, 0.20, 0.23, 0.25, 0.28, 0.30, 0.32, 0.34, 0.37, 0.46, 0.56, 0.75, and 0.84 mg/kg.

Based on those data, the Meeting estimated a maximum residue level for Brassica (Cole or cabbage) vegetables, head cabbage, and flowerhead Brassicas of 1.5 mg/kg.

From the trials cited above, residues of CCIM were (n=14): < 0.01 (11), 0.012, 0.014, and 0.023 mg/kg. For assessing short-term dietary intake from Brassica (Cole or cabbage) vegetables, head cabbage, flowerhead Brassicas, the HR, from a single sample, is 0.025 mg/kg.

From the trials cited above, the combined residues of cyazofamid and CCIM, expressed as cyazofamid, were (n=14): 0.14, 0.16, 0.21, 0.24, 0.26, 0.3, 0.31, 0.33, 0.35, 0.38, 0.47, 0.58, 0.78, and 0.85 mg/kg. For assessing long-term dietary intake from Brassica (Cole or cabbage) vegetables, head cabbage, flowerhead Brassicas, the STMR from that data set is 0.31 mg/kg.

Fruiting vegetables, Cucurbits

The critical GAP is from the registration of cyazofamid on the cucurbit vegetables crop group in the USA (six foliar applications at 0.08 kg ai/ha on a 7–10-day interval with a zero-day PHI). Supervised residue trials matching this GAP are available from the USA.

Mean field trial residues of cyazofamid in cucumber from independent field trials matching the critical GAP (n=4) were: 0.01 and 0.02 (3) mg/kg.

Mean field trial residues of cyazofamid in summer squash from independent field trials matching the critical GAP (n=4) were: 0.02 (2) and 0.04 (2) mg/kg.

Mean field trial residues of cyazofamid in muskmelon from independent field trials matching the critical GAP (n=6) were: < 0.01, 0.02 (3), 0.03 (2), and 0.06 mg/kg.

Noting that the residue trials address crops in the Codex commodity designation Fruiting Vegetables, Cucurbits and that the median residues from each crop are within a 5-fold range, the Meeting determined that a group MRL is appropriate. The cyazofamid residue data across the test crops are not significantly different by the Kruskal-Wallis test; therefore, the Meeting grouped the data together and is estimating a group maximum residue level for Fruiting Vegetables, Cucurbits based on the following cyazofamid residue data set (n=14): < 0.01, 0.01, 0.02 (5), 0.03 (2), 0.04 (2), and 0.06 mg/kg..

Based on those data, the Meeting estimated a maximum residue level for Fruiting Vegetables, Cucurbits of 0.09 mg/kg.

From the trials cited above, residues of CCIM were (n=14): < 0.01 (12) and 0.01 (2) mg/kg. For assessing short-term dietary intake from Fruiting Vegetables, Cucurbits, the HR, from a single sample, is 0.01 mg/kg.

From the trials cited above, the combined residues of cyazofamid and CCIM, expressed as cyazofamid, were (n=14): 0.02 (2), 0.03, 0.04 (7), 0.04, 0.06 (2), and 0.08 mg/kg. For assessing long-term dietary intake from Fruiting Vegetables, Cucurbits, the STMR from that data set is 0.04 mg/kg.

Fruiting vegetables, other than Cucurbits (except Sweet Corn and Mushroom)

The critical GAP is from the registration of cyazofamid on the fruiting vegetables crop group in the USA (six foliar applications at 0.08 kg ai/ha on a 7–10-day interval with a zero-day PHI). Supervised residue trials matching this GAP are available from the USA.

Mean field trial residues of cyazofamid in peppers, sweet (including pimento or pimiento) from independent field trials matching the critical GAP (n=6) were: 0.038, 0.055, 0.058, 0.072, 0.098, and 0.22 mg/kg.

Mean field trial residues of cyazofamid in peppers, chili from independent field trials matching the critical GAP (n=3) were: 0.24, 0.25, and 0.31 mg/kg.

Mean field trial residues of cyazofamid in tomatoes from independent field trials matching the critical GAP (n=14) were: < 0.010, 0.025, 0.030 (2), 0.035, 0.040, 0.050 (4), 0.065, 0.075, 0.11, and 0.15 mg/kg.

Noting that the residue trials in the USA address crops in the Codex commodity designation Fruiting Vegetables, Other Than Cucurbits (except Sweet Corn and Mushrooms), the Meeting considered whether a group MRL is appropriate. Based on the five-fold difference in the median residue values, The Meeting concluded that a group recommendation is appropriate. Analysis of the data set by the Kruskal-Wallis test indicated that the residues are not from the same populations and should not be combined when estimating the maximum residue level. Of the crops in this category, field trials with chilli pepper resulted in the greatest median residue level and greatest overall single-sample residue; however, the number of trials on chilli pepper is insufficient for making a group recommendation and the Meeting decided to make recommendations for the individual crops.

The Meeting estimated a maximum residue levels for sweet peppers at 0.4 mg/kg, for chilli peppers at 0.8 mg/kg, and for tomato at 0.2 mg/kg. Furthermore, the Meeting extrapolated the tomato data to eggplant and estimated a maximum residue level for eggplant at 0.2 mg/kg.

From the trials cited above, residues of CCIM and their associated HRs (from single samples) for assessing short-term dietary intake were as follows:

Sweet pepper (n=5): < 0.01 (4) and 0.012 mg/kg [HR = 0.014 mg/kg]

Chili pepper (n=3): 0.012 and 0.014 (2) mg/kg [HR = 0.017 mg/kg]

Tomato (n=15): < 0.01 (13), 0.01, and 0.015 mg/kg [HR = 0.02 mg/kg]; and by extension,

Eggplant: [HR = 0.02 mg/kg].

From the trials cited above, the combined residues of cyazofamid and CCIM, expressed as cyazofamid, and their associated STMRs for assessing long-term dietary intake were as follows:

Sweet pepper (n=5): 0.05, 0.07, 0.07, 0.09, and 0.24 mg/kg [STMR = 0.072 mg/kg]

Chilli pepper (n=3): 0.27 (2) and 0.33 mg/kg [STMR = 0.027 mg/kg];

Tomato (n=15): 0.02 (2), 0.04 (3), 0.05, 0.06 (5), 0.08, 0.09, 0.13, and 0.16 mg/kg [STMR = 0.06 mg/kg]; and by extension, Eggplant: [STMR = 0.06 mg/kg].

Leafy Vegetables (Including Brassica Leafy Vegetables)

The critical GAPs are from the registration of cyazofamid on the leafy greens crop subgroup in the USA (six foliar applications at 0.08 kg ai/ha on a 7–10-day interval with a zero-day PHI) and *Brassica* (Cole) leafy vegetables crop group in the USA (for mustard greens; one soil application at 0.753 kg ai/ha followed by five foliar applications at 0.08 kg ai/ha on a 7–10-day interval with a zero-day PHI). Supervised residue trials matching this GAP are available from Canada (head lettuce only) and the USA.

Mean field trial residues of cyazofamid in head lettuce from independent field trials matching the critical GAP (n=11) were: 0.070, 0.20, 0.26, 0.46, 0.63 (2), 0.73, 1.2, 1.5, 1.7, and 1.8 mg/kg.

Mean field trial residues of cyazofamid in leaf lettuce from independent field trials matching the critical GAP (n=11) were: 0.53, 0.76, 0.87, 0.89, 1.4, 1.8, 2.7, 2.8, 3.0, 4.0, and 4.4 mg/kg.

Mean field trial residues of cyazofamid in mustard greens from independent field trials matching the critical GAP (n=9) were: 1.4, 1.9, 3.3, 3.4, 3.5, 3.7, 5.5, 6.0, and 6.3 mg/kg.

Mean field trial residues of cyazofamid in spinach from independent field trials matching the critical GAP (n=10) were: 1.6, 2.0 (2), 2.2, 2.9, 3.3, 3.4, 3.6, 4.6, and 6.4 mg/kg.

Noting that the residue trials address crops in the Codex commodity designation Leafy Vegetables, the Meeting considered whether a group MRL is appropriate. The differences in median residue values across all four crops is greater than five-fold, indicating that a crop group recommendation is not appropriate. As median residue values for head lettuce, leaf lettuce, and spinach are within a five-fold range, the Meeting decided to make a recommendation for leafy vegetables, except Brassica leafy vegetables and to use data from mustard greens to make a recommendation for Brassica leafy vegetables.

Analysis of the residue data for lettuces and spinach by Kruskal-Wallis indicates that the residues are not from the same population and should not be combined when estimating the maximum residue level. Of these crops, the data from spinach has the highest median and highest residue.

On the basis of the data from spinach, the Meeting estimated a maximum residue level for Leafy Vegetables, except Brassica Leafy Vegetables at 10 mg/kg.

From the trials cited above, residues of CCIM and their associated HRs (from single samples) for assessing short-term dietary intake were as follows:

Head lettuce (n=11): < 0.010 (4), 0.01, 0.011, 0.013, 0.017 (2), 0.022, and 0.026 mg/kg [HR = 0.029 mg/kg];

Leaf lettuce (n=11): 0.011, 0.012, 0.016, 0.021, 0.025, 0.027, 0.037, 0.041 (2), 0.042, and 0.044 mg/kg [HR = 0.05 mg/kg];

Spinach (n=10): 0.029, 0.034, 0.045, 0.049, 0.05, 0.059, 0.088, 0.093, 0.12, and 0.14 mg/kg [HR = 0.15 mg/kg].

From the trials cited above, the combined residues of cyazofamid and CCIM, expressed as cyazofamid, and their associated STMRs for assessing long-term dietary intake were as follows:

Head lettuce (n=11): 0.08, 0.21, 0.27, 0.47, 0.64, 0.65, 0.74, 1.2, 1.5, 1.7, and 1.8 mg/kg [STMR = 0.65 mg/kg]

Leaf lettuce (n=11): 0.55, 0.80, 0.89, 0.93, 1.4, 1.8, 2.8, 2.9, 3.0, 4.1, and 4.5 mg/kg [STMR = 1.8 mg/kg];

Spinach (n=10): 1.6, 2.0, 2.1, 2.2, 3.0, 3.4, 3.5, 3.7, 4.8, and 6.6 mg/kg [STMR = 3.2 mg/kg].

For estimating dietary intake of the combined residues of cyazofamid and CCIM from leafy vegetables, except Brassica leafy vegetables, the data from spinach provide the highest residue estimate, with an STMR of 3.2 mg/kg.

For Brassica leafy vegetables, the Meeting estimated a maximum residue level of 15 mg/kg based on the data from mustard greens.

Residues of CCIM were (n=9): 0.032, 0.035 (2), 0.05, 0.053, 0.092, 0.11, 0.15, and 0.18 mg/kg. For assessing short-term dietary intake from Brassica leafy vegetables, the HR, from a single sample, is 0.19 mg/kg.

Combined residues of cyazofamid and CCIM in Mustard Greens were (n=9): 1.6, 2.0, 3.3, 3.5, 3.7, 4.0, 5.6, 6.1, and 6.4 mg/kg. For assessing long-term dietary intake from Brassica leafy vegetables, the STMR from that data set is 3.7 mg/kg.

Beans and beans, shelled

The critical GAP is from the registration of cyazofamid on beans (succulent podded and succulent shelled) in the USA (six foliar applications at 0.08 kg ai/ha on a 7–14-day interval with a zero-day PHI). Supervised residue trials in lima beans matching this GAP are available from the USA.

Mean field trial residues of cyazofamid in lima beans from independent field trials matching the critical GAP (n=6) were: < 0.010 (5) and 0.040 mg/kg.

Mean field trial residues of cyazofamid in snap beans from independent field trials matching the critical GAP (n=8) were: 0.018, 0.046, 0.059, 0.10, 0.12, 0.19, and 0.20 (2) mg/kg.

Noting that the residue trials in the USA address crops in the Codex commodity designation Legume Vegetables, the Meeting considered whether a group MRL is appropriate. Based on the spread in the median residue values, the Meeting determined that the residues from the trials are too dissimilar and that a group MRL is not appropriate.

The Meeting used the residue data from lima beans to estimate a maximum residue level for beans, shelled of 0.07 mg/kg.

From the trials cited above, residues of CCIM were (n=6): < 0.01 (6) mg/kg. For assessing short-term dietary intake from beans, shelled, the HR, from a single sample, is 0.01 mg/kg.

From the trials cited above, the combined residues of cyazofamid and CCIM, expressed as cyazofamid, were (n=6): 0.025 (5), and 0.06 mg/kg. For assessing long-term dietary intake from beans, shelled, the STMR from that data set is 0.025 mg/kg.

The Meeting used the residue data from snap beans to estimate a maximum residue level for beans, except broad bean and soya bean of 0.4 mg/kg.

From the trials cited above, residues of CCIM were (n=8): < 0.01 (8) mg/kg. For assessing short-term dietary intake from beans, except broad bean and soya bean, the HR, from a single sample, is 0.01 mg/kg.

From the trials cited above, the combined residues of cyazofamid and CCIM, expressed as cyazofamid, were (n=8): 0.04, 0.06, 0.07, 0.12, 0.13, 0.20, and 0.21 (2) mg/kg. For assessing long-term dietary intake from beans, except broad bean and soya bean the STMR from that data set is 0.125 mg/kg.

Carrot and potato

The critical GAP for carrots is from the registration of cyazofamid on carrots in the USA (five foliar applications at 0.175 kg ai/ha on a 14–21-day interval with a 14-day PHI). Supervised residue trials matching this GAP are available from Canada and the USA.

Mean field trial residues of cyazofamid in carrots from independent field trials matching the critical GAP (n=15) were: < 0.010 (9), 0.022 (2), 0.027, 0.029, 0.034, and 0.039 mg/kg. Carrot samples were stored frozen for 91 to 443 days prior to analysis. Stability of cyazofamid in carrots during frozen storage was not demonstrated (58% remaining at 374 days, no other time points sampled). As a result, the Meeting did not estimate a maximum residue level, HR, or STMR for carrot.

The critical GAP for potatoes is from the registration of cyazofamid on potatoes in Brazil (six foliar applications at 0.1 kg ai/ha on a 7–10-day interval with a seven-day PHI). The submitted residue trials conducted in Brazil did not match the critical GAP. However, supervised residue trials matching this GAP are available from the USA.

Mean field trial residues of cyazofamid in potatoes from independent field trials matching the critical GAP (n=23) were: < 0.010 (23). A single sample from an exaggerated rate (10-fold for the final application only) had a quantifiable residue of cyazofamid (0.02 mg/kg)

Based on those data and the results from the metabolism study, the Meeting estimated a maximum residue level for potato of 0.01* mg/kg.

From the trials cited above, residues of CCIM were: < 0.01 mg/kg. For assessing short-term dietary intake from potato, the HR, from a single sample, is 0.01 mg/kg.

From the trials cited above, the combined residues of cyazofamid and CCIM, expressed as cyazofamid, were (n=23): < 0.025 (23) mg/kg. Noting the low residue at the exaggerated rate, the Meeting decided to set the STMR at 0.01 mg/kg for assessing long-term dietary intake from potato.

Basil and hops

In basil, the critical GAP is from the registration in the USA (nine foliar applications at 0.088 kg ai/ha on a 10–14-day interval with a zero-day PHI). Mean field trial residues of cyazofamid in basil (sweet) from independent field trials conducted in the USA and matching the critical GAP (n=4) were: 2.5, 2.9, 7.2, and 9.4 mg/kg.

Stability of CCIM in sweet basil was not demonstrated (47% remaining at 284 days, the only time point analysed). As the data are insufficient for evaluating dietary intake, the Meeting is not making a recommendation for residues of cyazofamid in sweet basil.

In hops, the critical GAP is from the registration in the USA (six foliar applications at 0.06–0.08 kg ai/ha on a 7–10-day interval with a 3-day PHI). Mean field trial residues of cyazofamid in dried cones from independent field trials conducted in Canada and the USA and matching the critical GAP (with DAT ranging from 2 to 4 days; n=5) were: 2.5, 2.9, 3.2, 6.3, and 7.4 mg/kg.

Based on those data, the Meeting estimated a maximum residue level for hops (dried cones) of 15 mg/kg.

From the trials cited above, residues of CCIM were (n=5): 0.13, 0.17, 0.18, 0.24, and 0.44 mg/kg. For assessing short-term dietary intake from hops (dried cones), the HR, from a single sample, is 0.45 mg/kg.

From the trials cited above, the combined residues of cyazofamid and CCIM, expressed as cyazofamid, were (n=5): 3.1, 3.2, 3.6, 6.5, and 7.5 mg/kg. For assessing long-term dietary intake from hops (dried cones), the STMR from that data set is 3.6 mg/kg.

Fate of residues during processing

High-temperature hydrolysis

The Meeting received a study investigating the high-temperature hydrolysis of cyazofamid. Samples of aqueous buffered solutions were spiked with cyazofamid 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. Cyazofamid was readily hydrolysed to CCIM (ca. 80% for pasteurisation and 100% for both baking/brewing/boiling and sterilisation).

Based on the results of the high-temperature hydrolysis study, the Meeting assumed 100% yield in the conversion of cyazofamid to CCIM in all foods other than those specified as "raw" when conducting the short-term intake assessment for CCIM.

Residues after processing

In basil, the critical GAP is from the registration in the USA (nine foliar applications at 0.088 kg ai/ha on a 10–14-day interval with a zero-day PHI). Mean field trial residues of cyazofamid in dried basil from independent field trials conducted in the USA and matching the critical GAP (n=6) were: 9.7, 13, 14 (3), and 40 mg/kg

Stability of CCIM in basil (dry) was not demonstrated (59% remaining at 297 days, the only time point analysed). As the data are insufficient for evaluating dietary intake, the Meeting is not making a recommendation for residues of cyazofamid in basil (dry).

The Meeting received data depicting the concentration/dilution of residues during processing of grapes into raisins, must and wine; tomato into paste and puree; and potatoes into wet peel, chip, and flake commodities. Processed commodities were derived using simulated commercial practices. The residue data are supported by adequate analytical methods. Storage stability data demonstrate that residues of cyazofamid and CCIM are stable in those commodities under the conditions and storage periods used in the processing studies. Residues in raw and processed commodities are supported by adequate concurrent recovery data, with the exception of cyazofamid in raisins (67±10%) and CCIM in potato chips (68±4%).

Cyazofamid did not concentrate in any processed commodity. As no concentration of residues was observed, recommendations for maximum residue levels for grapes, tomatoes, or potatoes processed commodities are not necessary. The Meeting noted that for the potato commodities, residues were < LOQ in all samples and processing factors could not be calculated; however, the tubers used in the processing study were treated at an exaggerated rate such that quantifiable residues are not expected in processed commodities even if concentration is occurring upon processing.

For estimating short-term dietary intake, the Meeting based processing factors on the combined residues of cyazofamid (as CCIM equivalents) and CCIM in raw commodities and residues of CCIM only in processed commodities. When residues were < 0.01 in a sample, they were assumed to be 0.01 for purposes of deriving a processing factor.

For grapes, the combined residues of cyazofamid (as CCIM equivalents) and CCIM from field trials at the critical GAP were: 0.017, 0.033, 0.037, 0.050, 0.070, and 0.45 mg/kg, with an STMR of 0.044 mg/kg and an HR, from a single sample, of 0.47 mg/kg.

For tomatoes, the combined residues of cyazofamid (as CCIM equivalents) and CCIM from field trials at the critical GAP were: 0.017, 0.027, 0.030 (2), 0.033, 0.037, 0.044 (4), 0.054, 0.060, 0.075, and 0.11 mg/kg, with an STMR of 0.044 mg/kg and an HR, from a single sample, of 0.12 mg/kg.

For dried hops, the combined residues of cyazofamid (as CCIM equivalents) and CCIM from field trials at the critical GAP were: 2.1 (2), 2.4, 4.4, 5.0, and 5.1 mg/kg, with an STMR of 3.4 mg/kg and an HR, from a single sample, of 5.4 mg/kg.

For estimating long-term dietary intake, the Meeting based processing factors on the combined residues of cyazofamid and CCIM, expressed as cyazofamid, in raw and processed commodities. For all raw and processed commodities except potato, residues of parent or CCIM were quantifiable and processing factors could be derived. When residues were < 0.01 in a sample, they were assumed to be 0.01 for purposes of deriving a processing factor.

Crop	Processed commodity	Long-term processing factor [a]	Short-term yield factor [b]	Long-term processing factor [a]	Short-term yield factor [b]	STMR-P (Cyazofamid + CCIM), mg/kg	STMR-P (CCIM), mg/kg	HR-P (CCIM), mg/kg
Grape	Fruit (RAC)	–	–	–	–	STMR [c] = 0.06	STMR [d] = 0.044	HR [d] = 0.47
	Dried	0.22	0.064	0.22	0.064	0.013	0.0028	0.030
	Must	0.3, 0.5 (2), 0.59, 1.3, 1.8, 1.9	0.11, 0.25, 0.3 (3), 0.33	0.59	0.3	0.035	0.013	0.14
	Wine	0.18, 0.5 (7), 0.55, 0.66	0.11, 0.3 (7), 0.33, 0.5	0.5	0.3	0.03	0.013	0.14
Tomato	Fruit (RAC)	–	–	–	–	STMR = 0.06	STMR [d] =	HR [d] = 0.12

Crop	Processed commodity	Long-term processing factor [a]	Short-term yield factor [b]	Long-term processing factor [a]	Short-term yield factor [b]	STMR-P (Cyazofamid + CCIM), mg/kg	STMR-P (CCIM), mg/kg	HR-P (CCIM), mg/kg
							0.044	
	Paste	0.72	0.54	0.72	0.54	0.043	0.024	0.069
	Puree	0.45	0.27	0.45	0.27	0.027	0.012	0.034
Potato	Tuber (RAC)	–	–	–	–	STMR [c] = 0.01	STMR [d] = 0.01	HR [d] = 0.01
	Chips	Not calculated		Not calculated		0.01	0.01	0.01
	Flakes	Not calculated		Not calculated		0.01	0.01	0.01
	Wet peel	Not calculated		Not calculated		0.01	0.01	0.01
Hops	Dried cones (RAC)	–	–	–	–	STMR [c] = 3.6	STMR [d] = 3.4	HR [d] = 5.4
	Beer	0.002	0.0014	0.002	0.0014	0.0072	0.0048	0.0076

[a] [Cyazofamid + CCIM (cyazofamid equivalents) in the processed commodity] ÷ [cyazofamid + CCIM (cyazofamid equivalents) in the raw commodity].

[b] CCIM in the processed commodity ÷ [cyazofamid (CCIM equivalents) + CCIM in the raw commodity].

[c] Cyazofamid + CCIM (cyazofamid equivalents)

[d] Cyazofamid (CCIM equivalents) + CCIM

Residues in animal commodities

The Meeting has not made a determination as to the residue definitions for compliance and dietary intake for animal commodities. Furthermore, the Meeting did not receive animal feeding studies or residue data for livestock feedstuffs from some crops considered in this appraisal (grape: grape pomace, beans: vines). The Meeting did not make a recommendation for animal commodities.

RECOMMENDATIONS

Definition of the residue for compliance with the MRLs for plant commodities: *Cyazofamid*.

Definition of the residue for estimating long-term dietary intake from plant commodities: *Cyazofamid plus CCIM, expressed as cyazofamid*.

Definition of the residue for estimating short-term dietary intake from plant commodities (to be compared to the ARfD for CCIM; an ARfD was determined to be unnecessary for cyazofamid): *CCIM*.

Definition of the residue for compliance with the MRLs and for dietary intake for animal commodities: *Not defined.*

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
VP 0061	Beans, except broad bean and soya bean	0.4	--	0.125 Cyaz	--
				0.01 CC-R	0.01 CC-R
				0.017 CC-C	0.042 CC-C
VP 0062	Beans, shelled	0.07	--	0.025 Cyaz	--
				0.01 CC-R	0.01 CC-R
				0.084 CC-C	0.16 CC-C
VB 0040	Brassica (cole or cabbage) vegetables, Head cabbages, Flowerhead brassicas	1.5	--	0.31 Cyaz	--
				0.01 CC-R	0.025 CC-R
				0.22 CC-C	0.64 CC-C

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
VL 0054	Brassica leafy vegetables	15	--	3.7 Cyaz	--
				0.053 CC-R	0.19 CC-R
				2.4 CC-C	4.8 CC-C
VO 0440	Egg plant	0.2		0.06 Cyaz	--
				0.01 CC-R	0.02 CC-R
				0.044 CC-C	0.13 CC-C
VC 0045	Fruiting vegetables, Cucurbits	0.09	--	0.04 Cyaz	--
				0.01 CC-R	0.01 CC-R
				0.027 CC-C	0.057 CC-C
FB 0269	Grapes	1.5	--	0.06 Cyaz	--
				0.01 CC-R	0.01 CC-R
				0.044 CC-C	0.47 CC-C
DH 1100	Hops, dry	15	--	3.6 Cyaz	--
				3.4 CC-C	5.4 CC-C
VL 0053	Leafy vegetables (except Brassica leafy vegetables)	10	--	3.2 Cyaz	--
				0.054 CC-R	0.15 CC-R
				2.2 CC-C	4.5 CC-C
VO 0445	Peppers, sweet (including Pimento or pimiento)	0.4	--	0.072 Cyaz	--
				0.01 CC-R	0.014 CC-R
				0.054 CC-C	0.2 CC-C
VO 0444	Peppers, chili	0.8	--	0.27 Cyaz	--
				0.014 CC-R	0.017 CC-R
				0.18 CC-C	0.23 CC-C
VR 0589	Potato	0.01*	--	0.01	--
				0.017 CC-R, CC-C	0.017 CC-R, CC-C
VO 0448	Tomato	0.2	--	0.06 Cyaz	--
				0.01 CC-R	0.02 CC-R
				0.044 CC-C	0.13 CC-C
	Cyazofamid + CCIM (long-term only)				
DF 0269	Dried grapes (=currants, raisins and sultanas)			0.013	
	Grapes – Must			0.035	
	Grapes – Wine			0.03	
	Cabbage - raw			--	
	Cabbage – not raw			--	
VW 0448	Tomato – Paste			0.043	
MW 0448	Tomato – Purée			0.027	
	Head lettuce – raw			--	
	Head lettuce – not raw			--	
	Leaf lettuce – raw			--	
	Leaf lettuce – not raw			--	
	Potato – all forms			0.025	
DH 1100	Hops, Dry			3.6	
	Hops – Beer			0.0072	
	CCIM (short-term only)				
DF 0269	Dried grapes (=currants, raisins and sultanas)			0.0028	0.03
	Grapes – Must			0.013	0.14
	Grapes – Wine			0.013	0.14
	Cabbage - raw			0.01	0.025
	Cabbage – not raw			0.22	0.64

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
VW 0448	Tomato – Paste			0.024	0.065
MW 0448	Tomato – Puree			0.012	0.032
	Head lettuce – raw			0.011	0.029
	Head lettuce – not raw			0.43	1.4
	Leaf lettuce – raw			0.027	0.05
	Leaf lettuce – not raw			1.2	3.1
	Potato – all forms			0.017	0.017
DH 1100	Hops, Dry			3.4	5.4
	Hops – Beer			0.0048	0.0076

FUTURE WORK

As future work, the Meeting recommends that methods be developed to assay residues of CCBA (free and conjugated) in animal commodities, and that any such methods include suitable digestion steps for liver.

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of cyazofamid were calculated for the 17 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the current Meeting. The ADI for cyazofamid is 0–0.2 mg/kg bw. The calculated IEDIs for cyazofamid were 0–4% of the maximum ADI.

The Meeting concluded that the long-term intakes of residues of cyazofamid, when cyazofamid 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 CCIM 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 CCIM is 0.2 mg/kg bw. The calculated maximum IESTI for CCIM was 90% of the ARfD for all commodities. The Meeting concluded that the short-term intake of residues of CCIM resulting from uses of cyazofamid, when cyazofamid is used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

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CYPRODINIL (207)

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EXPLANATION

Cyprodinil is a fungicide belonging to the anilinopyridine group. It is a systemic foliar and seed dressing fungicide that acts as an inhibitor of methionine biosynthesis. Cyprodinil has been registered in many countries to control a range of fungal diseases in cereals, grapes, pome fruit, stone fruit, strawberries, vegetables, field crops and ornamentals, and as a seed dressing for barley.

Cyprodinil was firstly evaluated by JMPR in 2003, when an ADI of 0–0.03 mg/kg bw/day was established. An ARfD was deemed to be unnecessary. A residue definition of cyprodinil was recommended for plant and animal commodities, for both compliance with MRLs and estimation of dietary intake. The residue is fat soluble.

At the Forty-sixth session of the CCPR (2014), cyprodinil was scheduled for evaluation of additional use patterns by the 2015 JMPR.

The Meeting received residue data for oilseed rape and potato, and the proposal to extrapolate from carrot to ginseng.

METHODS OF RESIDUE ANALYSIS

Plant matrices

Method REM 141.01

Method REM 141.01(Dieterle, 1989) was evaluated by the 2003 JMPR. Homogenized samples were extracted with aqueous methanol. The extract was cleaned-up on a cation exchange cartridge. HPLC (single-column or two column-switching systems) with UV detection (λ max 270) was used for the final measurement. The LOQ for plant material was 0.02-0.05 mg/kg. The validation data included a wide range of high-water content crops as well as cereal grains (starchy). As the method was used for the determination of cyprodinil residues in potatoes, no further validation was conducted.

Method number AG-631B

Method AG-631B (Williams, R.K. 1998), with minor modifications was evaluated by the 2013 JMPR. Additional validation on rape seed matrices (seed and meal) are included in the supervised trials. Rape seed and meal samples were extracted by shaking with a methanol/water mixture at room temperature. After centrifugation, a 20 mL aliquot was taken and 2 mL of 1 M HCl was added. The extract was eluted through a SPE column with a methanol/ammonia mixture. The eluent was evaporated to near dryness and reconstituted with methanol. The extract was brought to 10 mL with methanol and bottled water and then diluted to 100 mL final volume and analysed by LC/MS/MS (quantification transition: 226.1 → 93.1). The method was verified at an LOQ of 0.02 mg/kg and an LOD of 0.006 mg/kg for canola seed and meal.

The following modifications were made to the reference method:

1. The extracts were centrifuged at 5000 rpm instead of being filtered.

2. Diethylene glycol diethyl ether was not added.

3. Extracts were brought to 10 mL final volume instead of 2 mL final volume.

These modifications were made to improve the method's ruggedness and make it suitable for LC/MS/MS analysis.

Table 1 Recovery of cyprodinil from rape seed and rape seed meal using method AG-631B

Commodity	Fortification level (mg/kg)	No. of analyses (n)	Recovery (%)	Mean recovery (%)	% RSD	Reference (Author, Year)
Rape seed	0.02	4	73, 85, 82, 88	83	7.7	Williams, R.K. 1998
	0.1	2	79, 92			
	0.2	2	86, 76			
Rape seed meal	0.02	4	97, 88, 80, 86	97	12	Williams, R.K. 1998
	0.1	2	102, 113			
	0.2	2	104, 109			

Method AG-597B

The principle of method AG-579B (Campbell, D, D, 1996) for the determination of cyprodinil in oil is as follows: 10 g sample of rape seed oil samples were shaken with acetonitrile saturated with hexane. The partition was repeated four more times and the acetonitrile layers combined. The extract was evaporated to less than 5 mL and brought to 10 mL in acetonitrile. The extract was diluted to a suitable final volume and analysed by LC/MS/MS (226.0-108.2). The method was verified at an LOQ of 0.01 mg/kg and an LOD of 0.0033 mg/kg for refined oil.

Table 2 Recovery of cyprodinil from rape seed oil using method A-597B

Commodity	Fortification level (mg/kg)	No. of analyses (n)	Recovery (%)	Mean recovery (%)	% RSD	Reference (Author, Year)
Refined rape seed oil	0.01	4	107, 104, 92, 84	97	8.8	Campbell, D.D. 1996
	0.05	2	107, 89			
	0.1	2	96, 99			

Stability of pesticide residues in stored analytical samples

The stability of cyprodinil residues was investigated concurrently with sample storage as part of the analytical phase of the residue trials at intervals of 0, 3, 6 and 9 months frozen storage in rape seed, meal and oil. Cyprodinil residues are stable in rape seed, meal and oil stored frozen for at least 9 months.

Table 3 Recovery of cyprodinil in stored samples of rape seed and processed rape seed products

Matrix	Fortification lever(ppm)	Storage interval (months)				Reference
		0	3	6	9	
Rape seed	0.2	74	89	80	101	Sagan, K., 2009
Rape seed meal	0.2	99	107	97	78	
Rape seed oil	0.1	102	100	106	105	

Further storage stability data was evaluated by JMPR for the 2003 evaluation of cyprodinil. A study by Kissling (1995) evaluated the stability (at -18 °C) of incurred cyprodinil residues in grapes, apples, wheat ears, and wheat stalks, and of fortified residues in strawberries, potatoes, and wine. Acceptable stability was observed in all of these matrices over 24 months.

Additional storage stability data were also evaluated by the 2013 JMPR. Storage stability data for Avocado, Beans (dry), Blueberry, Broccoli, Cabbage, Mustard greens, Raspberry, Cantaloupe, Cucumber, Squash, Peppers, Tomato (fruit, puree, paste), Basil (fresh), Chives (fresh), Kiwifruit, Lettuce, Spinach, Lemon(dried pulp, juice, oil), Lychee, Parsley (fresh, dried), Carrot, Radish(top, roots), Strawberry, Watercress, Apple and Pear were determined concurrently with sample storage as part of the analytical phase of the residue trials. Cyprodinil was shown to be stable for periods up to 601 days in a wide range of frozen plant matrices.

USE PATTERN

Cyprodinil is registered in the Brazil for use on potatoes, Canada for use on oilseed rape and the USA for use on ginseng, and are summarized in Table 4.

Table 4 Registered uses of cyprodinil in Brazil, Canada and the USA

Crop	Country	Formulation		Application				PHI (days)
		g ai/kg	type	Method	(g ai/ha)	Water L/ha	No	
Potato	Brazil	750	WG	Foliar spray	250	500	4	7
Oilseed rape (canola)	Canada	375	WG	Foliar spray	365.6	>200	1	35
Ginseng	USA	375	WG	Foliar Spray	365.6	>140	4	7

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received information on cyprodinil supervised field residue trials for potatoes and oilseed rape.

Root and tuber vegetables

Potatoes

Three supervised trials with cyprodinil on potatoes were conducted in Brazil (two trials in 1997) and South Africa (one trial in 1992).

In trials conducted in South Africa, 5 foliar applications of cyprodinil (500 WP formulation) were applied at a rate of 175 or 300 g ai/ha. Samples of tubers were collected at PHIs of 0–63 days following the final application.

In Brazil, 5 or 6 applications of cyprodinil (750 WG formulation) were applied at rate of 250–500 g ai/ha). Samples of tubers were collected at PHIs of 0–63 days following the final application in each trial.

Samples were immediately frozen and maintained in frozen storage for periods of 82 to 329 days prior to extraction, and were analysed with the modified method REM 141.01

Table 5 Results of residue trials conducted with cyprodinil in potatoes in Brazil (750 g/kg WG formulation) and South Africa (500WP formulation)

Location, Trial no., Year (Variety)	Application					PHI (days)	Crop Part	Residue (mg/kg)	Reference
	Formulation	Growth Stage	Rate (g ai/ha)	Volume (L/ha)	No.				
Fazenda Vista Alegre, Monte Mor, Brazil , FR 049 and 50/96, 1997, (Achat)	750 WG	BBCH 19	250		6	0	Tuber	< 0.02	FR 049-50/96,
		BBCH 24	250			3	Tuber	< 0.02	
		BBCH 31	250			7	Tuber	< 0.02	
		BBCH 43	250			10	Tuber	< 0.02	
		BBCH 45	250			15	Tuber	< 0.02	
		BBCH 47	250						
	750WG	BBCH 19	500		6	0	Tuber	< 0.02	
		BBCH 24	500			3	Tuber	< 0.02	
		BBCH 31	500			7	Tuber	< 0.02	
		BBCH 43	500			10	Tuber	< 0.02	
		BBCH 45	500			15	Tuber	< 0.02	
		BBCH 47	500						
Sitia Quilombo, Divinolandia, SP, Brazil, FR 051-52/96, 1997, (Monalisa)	750WG	BBCH 19	250		6				FR 051-52/96,
		BBCH 24	250			3	Tuber	< 0.02	
		BBCH 31	250			7	Tuber	< 0.02	
		BBCH 43	250			10	Tuber	< 0.02	
		BBCH 45	250			15	Tuber	< 0.02	
		BBCH 47	250						
	750WG	BBCH 19	500		6	0	Tuber	< 0.02	

Location, Trial no., Year (Variety)	Application					PHI (days)	Crop Part	Residue (mg/kg)	Reference
	Formulation	Growth Stage	Rate (g ai/ha)	Volume (L/ha)	No.				
		BBCH 24	500			3	Tuber	< 0.02	
		BBCH 31	500			7	Tuber	< 0.02	
		BBCH 43	500			10	Tuber	< 0.02	
		BBCH 45	500			15	Tuber	< 0.02	
		BBCH 47	500						
Bultfontein, South Africa, 2168-91, 1992, (BP 1)	500 WP	Begin flower drop to End Flower drop	175	480	5	0	Tuber	< 0.02	2169-91
			175	480		7	Tuber	< 0.02	
			175	480		13	Tuber	< 0.02	
			175	480		28	Tuber	< 0.02	
			175	480		63	Tuber	< 0.02	
Bultfontein, South Africa, 2169-91, 1992, (BP 1)	500 WP	Begin flower drop - End Flower drop	300	480	5	0	Tuber	< 0.02	2169-91
			300	480		7	Tuber	< 0.02	
			300	480		13	Tuber	< 0.02	
			300	480		28	Tuber	< 0.02	
			300	480		63	Tuber	< 0.02	

Oilseeds

Rape seed

Sixteen supervised trials with cyprodinil on canola (oilseed rape) were conducted in Canada in 2009. Fourteen of the trials were conducted in region 14 but at only nine field sites. Thus, there were only nine independent trials. One application of cyprodinil (WG formulation) was made at the rate of 365.6 g ai/ha with a PHI of 35 days, with adjuvant added. Samples of rape seed were collected at normal commercial harvest, 35 to 53 days after application.

Samples were immediately frozen and maintained in frozen storage for periods of up to 200 days prior to extraction. Residues of cyprodinil in seed and meal were determined using method AG-631B and residues of cyprodinil in oil were determined using method AG-597B.

Table 6 Summary of residue data from Canada supporting the Canada GAP for use of cyprodinil on oilseed rape

Location, Trial no., Year (Variety)	Application					PHI (days)	Crop Part	Residue (mg/kg)	Reference
	Formulation	Growth Stage	Rate (g ai/ha)	Volume (L/ha.)	No.				
Elm Creek, MB, Canada, CER04169/07, 2009, (5030)	WG	BBCH 57 - 62	345.9			48	Seed	< 0.02	CER04169/07
Delisle, SK, Canada, CER04169/07, 2009, (5108)	WG	BBCH 62 - 63	362.7			35	Seed	< 0.02	
Minto, MB, Canada, CER04169/07, 2009, (Liberty Link Invigor 5020)	WG	BBCH 55 - 62	367.1			44	Seed	< 0.02	
						48	Seed	< 0.02	
						53	Seed	< 0.02	
						57	Seed	< 0.02	
Minto, MB, Canada, CER04169/07, 2009 , (Invigor 5108)	WG	BBCH 62 - 63	368.4			37	Seed	< 0.02	
Boissevain, MB, Canada, CER04169/07, 2009 , (Liberty 5030)	WG	BBCH 55 - 63	369.3			52	Seed	< 0.02	
		BBCH 55 - 63	1119.7			52	Seed	< 0.02	
							Meal	< 0.02	
							Oil	< 0.01	

Location, Trial no., Year (Variety)	Application					PHI (days)	Crop Part	Residue (mg/kg)	Reference
	Formulation	Growth Stage	Rate (g ai/ha)	Volume (L/ha.)	No.				
Boissevain, MB, Canada, CER04169/07, 2009, (Round-Up Ready 9551)	WG	BBCH 52 - 63 (Majority of plot was BBCH 62-63)	364.1			46	Seed	< 0.02	
Rosthern, SK, Canada, CER04169/07, 2009, (5020)	WG	BBCH 62 - 63	366.3			35	Seed	< 0.02	
						42	Seed	< 0.02	
						49	Seed	< 0.02	
						56	Seed	< 0.02	
Rosthern, SK, Canada, CER04169/07, 2009, (5030)	WG	BBCH 62 - 63	378.2			53	Seed	< 0.02	
Hepburn, SK ,Canada, CER04169/07, 2009, 45H72	WG	BBCH 62 - 63	375.7			38	Seed	< 0.02	
	WG	BBCH 62 - 63	1126.9			38	Seed	< 0.02	
							Meal	< 0.02	
							Oil	< 0.01	
Hepburn, SK, Canada, CER04169/07, 2009, (45H73)	WG	BBCH 62 - 63	366.2			38	Seed	< 0.02	
Innisfail, AB, Canada, CER04169/07, 2009, (5108)	WG	BBCH 62 - 66	390.6			41	Seed	< 0.02	
Innisfail, AB, Canada, CER04169/07, 2009, (9551)	WG	BBCH 62 - 63	382.8			52	Seed	< 0.02	
Penhold, AB, Canada, CER04169/07, 2009, (5020)	WG	BBCH 62 - 63	371.8			41	Seed	Mean = <u>< 0.02</u> (0.021, 0.017)	
Penhold, AB, Canada, CER04169/07, 2009, (9551)	WG	BBCH 62 - 63	374.9			52	Seed	< 0.02	
Sylvan Lake, AB, Canada, CER04169/07, 2009, (5020)	WG	BBCH 62 - 63	367.4			42	Seed	< 0.02	
Sylvan Lake, AB, Canada CER04169/07, 2009, (5020)	WG	BBCH 65 - 67	375.2			42	Seed	Mean = <u>< 0.02</u> (< 0.02, < 0.02)	

LOQ for seed is 0.02mg/kg. LOQ for oil is 0.01mg/kg

FATE OF RESIDUES IN PROCESSING

The determination of cyprodinil residues in processed fractions of oilseed rape was included in the residue study conducted in Canada. The application rate of cyprodinil was 1098 g ai/ha, 3-times the label rate. The process included seed cleaning, seed pro-conditioning and flaking, seed cooking, pressing the flake to mechanically remove a portion of the oil, solvent extraction of the press-cake to remove the remainder of the oil, and desolventizing and toasting of the meal. No residues (<LOQ) were found in seed, meal and oil. Therefore, no processing factors can be established because no measurable residues were found in the seed samples before processing.

APPRAISAL

Cyprodinil was first evaluated for residues and toxicological aspects by the 2003 JMPR. An ADI of 0–0.03 mg/kg bw for cyprodinil was established, and an ARfD was concluded as unnecessary. The residue definition was established as cyprodinil for both compliance with MRLs and dietary risk assessment for both plant and animal commodities. The residue is fat soluble.

Cyprodinil was evaluated by 2013 JMPR for additional crops. A number of Codex Maximum Residue limits for cyprodinil were established. Cyprodinil was scheduled by the Forty-sixth CCPR meeting in 2014 for evaluation of residue data for additional crops by the JMPR.

Methods of analysis

The Meeting received two analytical methods for determination of cyprodinil residues in plant matrices which are relevant to this evaluation. The LOQ for the HPLC-MS/MS (226.01–93.10) methods for rapeseed and meal was 0.02 mg/kg, and for rape seed oil, 0.01 mg/kg.

Stability of residues in stored analytical samples

The Meeting received information on the storage stability of cyprodinil residues in plant matrices from trials conducted in conjunction with the residue studies submitted to the Meeting. These data and stability data from JMPR 2003 and 2013 covers the maximum storage period for samples in the residue studies submitted to this Meeting.

Residues of supervised trials on crops

The Meeting received supervised trial data for application of cyprodinil to oilseed rape, potatoes, and carrots, which was evaluated by 2013 JMPR.

Potato

Cyprodinil is registered in the Brazil for use on potatoes at a GAP of 4× 0.25 kg ai/ha and PHI of 7-days.

The residues of cyprodinil in potatoes from two trials conducted in Brazil and one trial in South Africa matching the Brazilian GAP were all < 0.02 mg/kg (LOQ). The meeting noted that three trials were insufficient to make a recommendation for a maximum residue level for potatoes.

Ginseng

The meeting received the request to extrapolate the maximum residue level from carrots to ginseng. The 2013 Meeting received supervised residue trials of carrots matching the US GAP. The Meeting noted that although the US GAP for ginseng is the same as that for carrots, the growth traits and cultivation practices are significantly different, and agreed not to extrapolate from carrots to ginseng.

Rape seed

Cyprodinil is registered in Canada for use on rape seed at a GAP of 1× 0.365 kg ai/ha and a 35-day PHI.

Nine independent residue trials were conducted in rapeseed at GAP in Canada. Residues in seed of rape seed at the 35 day PHI were all < 0.02 mg/kg (n=9).

Based on the residues from the Canadian trials, the Meeting estimated a maximum residue level of 0.02 mg/kg for seed of rape seed and an STMR of 0.02 mg/kg.

Processing studies

A processing study for oilseed rape was evaluated by the current Meeting in which the application rate of cyprodinil was 1098 g ai/ha, 3-times the label rate. No residues (< LOQ), were found in seed, meal and oil, and therefore no processing factors could be established.

Residues in animal commodities

Farm animal dietary burden

Dietary burden calculations incorporating all commodities considered by the current, 2003 and 2013 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. The animal dietary burden is the same as the results from 2013 meeting, and the Meeting confirmed the previous recommendation of MRLs in animal products.

	US/CAN		EU		AU		Japan	
	Max.	Mean	Max.	Mean	Max.	Mean	Max.	Mean
Beef cattle	0.91	0.37	13.9	1.8	5.8	1.4	0.46	0.46
Dairy cattle	1.7	0.87	13.5	1.4	23.3	1.8	0.26	0.26
Poultry—broiler	0.49	0.49	0.80	0.54	0.12	0.12	0.066	0.066
Poultry—layer	0.49	0.49	4.1	0.76	0.12	0.12	–	–

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed are appropriate for establishing maximum residue limits and for an IEDI assessment.

Definition of the residue for plant and animal commodities for compliance with MRLs and for estimation of dietary intake: *cyprodinil.*

The residue is fat soluble

CCN	**Commodity**	**Recommended Maximum residue level (mg/kg)**		**STMR or STMR-P mg/kg**	**HR or HR-P mg/kg**
		New	**Previous**		
SO 0495	Rape seed	0.02		0.02	

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Dietary Intakes (IEDIs) of cyprodinil were calculated for the 17 GEMS/food cluster diets using STMRs/STMR-Ps estimated by the current Meeting and by the 2003 JMPR. The ADI is 0–0.03 mg/kg bw and the calculated IEDIs were 6–70% of the maximum ADI (0.03 mg/kg bw). The Meeting concluded that the long-term intakes of residues of cyprodinil, resulting from the uses considered by the current Meeting and by the 2003 JMPR are unlikely to present a public health concern.

Short-term intake

The 2003 JMPR decided that an ARfD was unnecessary and concluded that the short-term intake of cyprodinil residues is unlikely to present a public health concern.

REFERENCES

Code	Author(s)	Year	Title
Report No. AG-631B	Williams, Robert	1998	Analytical Method AG-631B for the Determination of Residues of CGA219417 in Crops by High Performance Liquid Chromatography with Column Switching. Not published.
Report No. AG-597B	Campbell D.D.	1996	Analytical method for the determination of CGA 173506 in crops by high performance liquid chromatography including validation data. Not published.
Report No. FR 049-96 , FR 050-96	Machado Thais R.	1997	Determination of the residue level of stated potato peels after application of the fungicide Unix 750 WG, with the objective to provide data for the registration team to present to the authorities. Not published.
Report No. FR 051-52-96	Gebara Amir B.	1997	Determination of the concentration of Cyprodinil residues in potato tubers after application of Unix 750 WG fungicide.Not published.
Report No.2168-91	Dieterle R.	1993	CGA219417, WP 50, Potatoes, South Africa. Not published
Report No.2169-91	Dieterle R.	1993a	CGA219417, WP 50, Potatoes, South Africa. Not published
Report No. CER 04169/07	Sagan K.	2009	Amendment_A9219B - Residue Levels on Canola Seed and Processed Fractions, Meal and Refined Oil, from Trials Conducted with SWITCH 62.5 WG in Canada during 2007 (Fludioxonil/Cyprodinil WG). Not published.
Report No. 07090	Chen H.	2002	Cyprodinil and Fludioxonil: Magnitude of the Residue on Carrot. Not published.

DIFENOCONAZOLE (224)

First draft was prepared by Dr Anita Stromberg, National Food Agency, Uppsala Sweden

EXPLANATION

Difenoconazole is a systemic triazole fungicide and acts by inhibition of demethylation during ergosterol synthesis. It is applied by foliar spray or seed treatment and controls a broad-spectrum of foliar, seed and soil-borne diseases caused by *Ascomycetes*, *Basidiomycetes* and *Deuteromycetes* on a variety of crops. Difenoconazole was evaluated for the first time by JMPR 2007. The 2007 Meeting established an acceptable daily intake (ADI) of 0–0.01 mg/kg bw and an acute reference dose (ARfD) of 0.3 mg/kg bw. In 2007, 2010 and 2013, the JMPR evaluated the compound for residues and recommended a number of maximum residue levels.

Difenoconazole was listed by the 46th session of CCPR (2014) for evaluation for additional MRLs. The current Meeting received from the manufacturer additional analytical methods, processing data from soya beans, oilseed rape and rice, GAP information and residue trial data from uses on strawberry, avocado, soya beans, cotton, peanut, rice and oilseed rape (canola).

IDENTITY

The 2007 Meeting noted that the structural formula for difenoconazole contains two chiral carbons resulting in a *cis-trans* pair diastereoisomers. The current Meeting noted that the presented analytical methods not are stereo-selective for the *cis*- and *trans*- isomers

ANALYTICAL METHODS

The current Meeting received new analytical method descriptions and validation data for parent difenoconazole. Methods were validated for all crop matrices; the LOQ were 0.01 mg/kg for determination of difenoconazole with procedural recoveries by matrix in the range of 70–122% at various fortification levels. A summary of the analytical methods for difenoconazole is provided below.

Method, analyte	Matrix	Extraction	Clean-up	Detection, LOQ
Method REM 147.08 Difenoconazole	Plant material 2007 JMPR) Current Meeting: Validation data on oilseed rape seed, meal and refined oil.	Refluxing with methanol-ammonia for 2 hours. Elution with dichloromethane.	Solid-phase extraction (SPE)	LC-MS/MS Difenoconazole LOQ 0.01 mg/kg
Method POPIT MET.032 Difenoconazole	Plant material Validation data on soya beans and peanuts	high-speed homogenisation with a acetone/water mixture (2:1; v/v)	Filtration/centrifugation	HPLC-MS/MS Difenoconazole The ion transition m/z 406→251 is used for quantification LOQ 0.01 mg/kg
Method POPIT MET.033, rev.31 Difenoconazole	Plant material Validation data on avocado, cotton, peanut, rice, soya beans and	high-speed homogenisation with a acetone/water mixture (2:1; v/v)	Filtration/centrifugation	HPLC-MS/MS Difenoconazole The ion transition m/z 406→251 is used for quantification and

Method, analyte	Matrix	Extraction	Clean-up	Detection, LOQ
	strawberry			the ion transition m/z 406→111 for confirmation, LOQ 0.01 mg/kg

Plant materials

Oilseed rape (canola)

The analytical method REM 147.08 was validated for oilseed rape seed and the processed fraction meal and refined oil by Sagan, K (2012 SYN545192) for residues of difenoconazole with an LOQ of 0.01 mg/kg. The method REM 147.08 was reviewed by JMPR in 2007. The recovery (% recovery) and repeatability (RSD) is summarized in Table 1 below.

Table 1 Recovery and repeatability data for the method REM 147.08 for difenoconazole oilseed rape

Commodity	Fortification level (mg/kg)	No of analysis	Recovery (%)	Mean recovery (%)	% RSD
Oilseed Rape (seed)	0.010	4	81,121, 114, 112	107	17
	0.10	4	122, 118, 97, 102	110	11
	0.20	4	91 ,94 ,85	90	5.1
Oilseed Rape (meal)	0.010	3	98,89,100	96	6.1
	0.10	3	89, 103, 115	102	13
	0.20	3	85, 92, 84	87	5.0
Oilseed Rape (oil)	0.010	3	82, 100, 83	88	11
	0.10	3	98, 99,84	94	9.0

Other plant materials

The analytical method POPIT MET.032 was developed to determine and quantify difenoconazole in plant material by Vopi K *et al.* (2010 Syngenta file no. CGA169374_10882).

Residues of difenoconazole are extracted from plant matrices by high-speed homogenisation with an acetone/water mixture (2:1; v/v). The suspension is either filtrated (soya beans) or centrifuged (peanut) and brought to volume with extraction solvent. An aliquot of the extract is evaporated and reconstituted in acetonitrile/water (1:1; v/v). After filtration of the final sample solution, residues of difenoconazole are determined by liquid chromatography with tandem mass spectrometric detection (HPLC-MS/MS). Quantification is performed with calibration curves using 6 standard solutions (from 1×10^{-4} to 3.2×10^{-3} µg/mL, ½ to 16 times the LOQ). The ion transition m/z 406→251 is used for quantification. The method has a validated LOQ of 0.01 mg/kg for soya beans and peanut.

Table 2 Recovery and repeatability data for the method POPIT MET.032 for difenoconazole in soya beans and peanuts

Commodity	Fortification level (mg/kg)	No of analysis	Recovery (%)	Mean recovery (%)	% RSD
Soya (beans)	0.010	8	88; 91; 92; 93; 94; 96; 97; 99	94	3.8
	0.10	6	78; 83; 90; 92; 93; 95	89	7.5
	1.0	5	74; 77; 79; 79; 83	78	4.2
Peanut (kernels)	0.010	7	70; 72; 72; 72; 73; 74; 80	73	4
	0.11	5	70; 82; 83; 85; 90	82	9

The analytical method POPIT MET.033 was developed to determine and quantify difenoconazole in plant material by Maslowski, R *et al*. (2008 Syngenta file no. CGA169374_10881).

Residues of difenoconazole are extracted from plant matrices by high-speed homogenisation with an acetone/water mixture (2:1; v/v). The suspension is filtrated (cotton) and brought to volume by extraction solvent. After filtration of the final sample solution, residues of difenoconazole are determined by liquid chromatography with tandem mass spectrometric detection (HPLC-MS/MS). Quantification is performed with calibration curves using 6 standard solutions. The ion transition m/z 406→251 is used for quantification and the ion transition m/z 406-111 for confirmation. The method has a validated LOQ of 0.01 mg/kg for difenoconazole in avocado, cotton, peanut, rice, soya beans and strawberry.

The recovery (% recovery) and repeatability (RSD) of residues from parent difenoconazole in crop matrices in current evaluation for MRLs is summarized in Table 3 below.

Table 3 Recovery and repeatability data for the method POPIT MET.033 for difenoconazole in various plant matrices

Commodity	Fortification level (mg/kg)	No of analysis	Recovery (%)	Mean recovery (%)	% RSD
Avocado	0.011	7	81; 83; 83; 83; 84; 84; 87	84	2.2
	0.11	5	89; 90; 90; 90; 93	90	1.7
	2.2	5	89; 91; 91; 91; 92	91	1.2
Cotton	0.010	8	75; 79; 80; 81; 85; 88; 91; 92	84	7.3
	0.10	6	81; 82; 82; 82; 87; 91	84	4.7
Peanut	0.010	7	78; 81; 83; 85; 85; 86; 87	84	3.8
	0.10	3	88; 88; 88; 90; 90	89	1.2
Rice	0.01	7	94; 95; 96; 97; 99; 99; 103	98	3.1
	0.1	5	96; 101; 104; 105; 109	103	4.7
	0.51	5	102; 106; 107; 108; 109	106	2.5
Soya beans	0.011	7	90; 92; 92; 93; 94; 95; 96	93	2.2
	0.11	5	88; 88; 88; 89; 89	88	0.6
Strawberry	0.01	5	78; 79; 80; 80; 81; 83; 85	81	3.0
	0.1	5	76; 77; 77; 80; 80	78	2.4
	0.3	5	88; 88; 91; 91; 94	90	2.8
	2	5	87; 87; 91; 92; 95	90	3.8

USE PATTERN

Difenoconazole is a systemic fungicide which belongs to the triazole chemical group of fungicides. Information on registered uses including labels from countries trials had been carried out was provided to the Meeting by one manufacturer. The representative uses relating to crops under consideration for additional MRLs and revising some of the existing CODEX MRLs are summarized in the following table.

Table 4 Registered uses of difenoconazole from labels provided

Crop	Country	Application details								
		type	Met-hod	kg ai/ha	Water L/ha	Crop growth stage	No	Interval (days)	PHI	Comments
Straw-berry	USA	126 g ai/L SC	Foliar spray	0.129	> 94[a]; 140 [b]	Prior to disease onset when conditions conductive for disease	4	7-14	0	Not more than 2 sequential applications before alternating to another fungicide with different mode of action. max 0.21 kg ai/ha per crop and season
Avo-cado	Brazil	250 g ai/L EC	Foliar spray	0.050	500-1000	start at flowering, end when fruit is around 5 cm	4	14	14	ground/ aerial application
Soya bean	USA	126 g ai./L SC	Foliar spray	0.129	> 19[b]	Prior to disease onset when conditions conductive for disease	2	7-10	14	Do not feed soybean hay, forage or silage max 0.25 k g ai/ha per season
Rice	Italy	125 g ai/L SC	Foliar spray	0.125	200-400	BBCH 21-29[d]	2	-	28	ground application
Cotton	Brazil	250 g ai/L EC	Foliar spray	0.075	200-400	when first symptom occur	3	10-15	21	ground/ aerial application
Peanut	Brazil	250 g ai/L EC	Foliar spray	0.0875	100-200	when first symptom occur	3	-	22	ground/ aerial application
Oilseed Rape (canola)	Canada	250 g ai/L EC	Foliar spray	0.125	110-170[c]	BBCH 12-18[e] or BBCH 62-65[f]	1	-	30	ground/ aerial application max 0.125 kg ai/ha per season

[a] For aerial applications

[b] For ground applications

[c] When applying difenoconazole at typical herbicide timing it is recommended to use 50-110 L/ha water

[d] Between beginning and end of tillering.

[e] Apply during rosette stage between 2nd true leaf and bolting. (*Leptosphaeria maculans)* Virulent Black Leg

[f] Apply at 20-50% bloom *(Sclerotinia sclerotiorum)* Sclerotinia Stem Rot

Conditions of the supervised residue trials were generally well reported in detailed field reports. In most trials plots treated plots were not replicated but where results were reported from replicate plots, these are presented as individual values. Most field reports provided data on the sprayers used and their calibration, and reports provided data on 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.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

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.

The Meeting received information on supervised field trials involving difenoconazole for the following crops and commodities:

Group	Crop commodity	Portion of commodity to which MRL apply	Countries	Table No
FB, Berries and other small fruits	Strawberry, fruit	Whole fruit	USA	5
FI, Assorted tropical and sub-tropical fruits – inedible peel	Avocado, fruit	Whole commodity after removal of obviously decomposed or withered leaves	Brazil	6
Pulses,	Soya, seed	Whole commodity	USA	7
GC, Cereal grain	Rice, grain, straw forage	Whole commodity	Europe	8
SO, Oilseed	Cotton, seed	Whole kernel after removal of the seed	Brazil	9
	Peanut, whole plant	Whole kernel after removal of the seed	Brazil	10
	Oilseed rape, seed	Whole kernel after removal of the seed	Canada	11
Animal feeds	Rice forage		Europe	12
	Rice straw		Europe	13

Strawberry

Nine independent supervised residue field trials on strawberries were conducted in USA during growing season 2008-2009. Four foliar applications of difenoconazole (EC formulation) at a target rate of 0.129 kg ai/ha were made with a seven day interval. Duplicate samples (fruits) were taken seven days after the third application and immediately after the last (fourth) application. Samples were stored frozen for a maximum of eight months.

Analysis of difenoconazole was made using LC-MS/MS and method REM 147.08. The limit of quantification was 0.01 mg/kg and the mean recovery was in the range of 70–109% at fortification levels of (n=1–2) 0.01, 0.5, 1.0 and 2.0 mg/kg.

Samples were analysed for triazole metabolites by Analytical Method 160 using LC/MS/MS and Morse Laboratories, Inc. The limit of quantitation (LOQ) for all analytes (as respective parent equivalents) for strawberries was 0.01 mg/kg. The limit of detection (LOD) based on the smallest standard that can be detected is 0.0015 ng/kg. The mean recovery for the metabolites was triazole (87±8%), triazole alanine (92±7%) and triazole acetic (102±7%) at fortification levels (n=16) 0.01, 0.10, 0.02 and 0.5 mg/kg.

Table 5 Residues in strawberry after foliar applications of difenoconazole in field trials from USA

STRAWBERRY Country year (variety)	Application			Residues (mean values in parenthesis) mg/kg mature fruit							Reference
	kg ai/ha	no	(BBCH)	DAT	Difenoconazole	1,2,4,-Triazole	Triazole alanine		Triazole acetic acid		Study: T002101-7 CGA169374_50035
Critical GAP in USA; apply 0.129 kg ai/ha 4 times at a 7-14 day intervals. PHI 0 days											
					treated	treated	treated	control	treated	control	
USA (NY) 2007 Penn Yan (Honeoye)	0.131 0.131 0.128 0.129	1 2 3 4	65 73 81 89	7[c]	0.31, 0.25 (0.28)	< 0.01, < 0.01 (< 0.01)	0.02, 0.02 (0.02)	0.02	< 0.01, < 0.01 (0.01)	< 0.01	Trial: E03NY078481
				0[d]	0.64, 0.66 (0.65)	nd, < 0.01 (< 0.01)	0.03, 0.03 (0.03)	0.02	< 0.01, < 0.01 (0.01)	< 0.01	
USA (NC) 2008 Seven Springs (Camarosa)	0.129 0.129 0.125 0.130	1 2 3 4	81 81 83 85	7[c]	0.20, 0.19 (0.20)	< 0.01,< 0.01	0.02, 0.02 (0.02)	< 0.01	< 0.01,< 0.01 (< 0.01)	< 0.01	Trial: E10NC078482
				0[d]	0.38, 0.43 (0.41)	< 0.01,< 0.01 (< 0.01)	0.02, 0.02 (0.02)	< 0.01	< 0.01,< 0.01 (< 0.01)	< 0.01	
USA (FL) 2008 (Treasures)	0.132 0.127 0.130 0.126	1 2 3 4	81-85[a] 73-81 81-85[a] 81-85[b]	7[c]	0.13, 0.18 (0.16)	< 0.01,< 0.01 (< 0.01)	0.05, 0.05 (0.05)	< 0.01	< 0.01,< 0.01 (< 0.01)	< 0.01	Trial: E16FL078483
				0[d]	0.19, 0.19 (0.19)	< 0.01,< 0.01 (< 0.01)	0.04, 0.05 (0.05)	< 0.01	< 0.01,< 0.01 (< 0.01)	< 0.01	
USA (MN) 2008 Wimauma (Mesabi)	0.132 0.130 0.129 0.137	1 2 3 4	63 65 73 88	7[c]	0.14, 0.20 (0.17)	< 0.01,< 0.01 (< 0.01)	< 0.01 < 0.01	(< 0.01) < 0.01	< 0.01,< 0.01 (< 0.01)	< 0.01	Trial: C12MN078484
				0[d]	0.49, 0.36 (0.43)	< 0.01,< 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01	< 0.01,< 0.01 (< 0.01)	< 0.01	
USA (CA) Santa Maria 2008 (Albino)	0.130 0.130 0.129 0.129	1 2 3 4	89 89 89 89	7[c]	0.22, 0.24 (0.23)	< 0.01,< 0.01	0.07, 0.07 (0.07)	0.02	< 0.01,< 0.01	< 0.01	Trial: W30CA078485
				0[d]	0.41, 0.55 (0.48)	< 0.01,< 0.01 (< 0.01)	0.08, 0.07 (0.08)	0.02	< 0.01,< 0.01 (< 0.01)	< 0.01	
				1[d]	0.63	< 0.01	0.07	0.02	0.01	< 0.01	
				3[d]	0.47	< 0.01	0.09	0.03	0.02	< 0.01	
				5[d]	0.42	< 0.01	0.09	0.03	0.02	< 0.01	
USA (CA) 2008 Madera (Seascape)	0.130 0.131 0.129 0.131	1 2 3 4	71 75 79 79	7[c]	0.26, 0.31 (0.29)	< 0.01,< 0.01 (< 0.01)	0.04, 0.03 (0.03)	< 0.01	< 0.01,< 0.01 (< 0.01)	< 0.01	Trial: W29CA078486
				0[d]	0.72, 0.58 (0.65)	< 0.01,< 0.01 (< 0.01)	0.02, 0.04 (0.03)	0.01	< 0.01,< 0.01 (< 0.01)	< 0.01	
USA (CA) 2008 Madera (Chandler)	0.130 0.131 0.130 0.131	1 2 3 4	73 77 79 79	7[c]	0.54, 0.59 (0.57)	< 0.01,< 0.01 (< 0.01)	0.04, 0.05 (0.05)	< 0.01	< 0.01 < 0.0.01 (< 0.01)	< 0.01	Trial: W29CA078487
				0[d]	1.20, 1.22 (1,21)	< 0.01,< 0.01 (< 0.01)	0.06, 0.03 (0.05)	< 0.01	< 0.01,< 0.01 (< 0.01)	< 0.01	
USA (WA) 2008 Mount	0.125 0.123 0.131	1 2 3	73-81 81 85	7[c]	0.13, 0.09 (0.11)	< 0.01,< 0.01 (< 0.01)	< 0.01,< 0.0.01 (< 0.01)	< 0.01	< 0.01,< 0.01 (< 0.01)	< 0.01	Trial: W19WA078488

STRAWBERRY Country year (variety)	Application			Residues (mean values in parenthesis) mg/kg mature fruit							Reference
	kg ai/ha	no	(BBCH)	DAT	Difenoconazole	1,2,4,-Triazole	Triazole alanine		Triazole acetic acid		Study: T002101-7 CGA169374_50035
Critical GAP in USA; apply 0.129 kg ai/ha 4 times at a 7-14 day intervals. PHI 0 days											
					treated	treated	treated	control	treated	control	
Vernon (Puget Reliance)	0.128	4	81-85[a]	0[d]	0.07, 0.07 (0.07)	< 0.01,< 0.0.01 (< 0.01)	< 0.01,< 0.0.0.01 (< 0.01)	< 0.01	< 0.01,< 0.0.01 (< 0.01)	< 0.01	
USA (CA) Guadalupe 2009 (Albino)	0.131 0.131 0.130 0.131	1 2 3 4	75 75 75 75	7[c]	0.23, 0.25 (0.24)	< 0.01,< 0.0.01 (< 0.01)	0.09, 0.08 (0.09)	0.03	0.02, 0.03	< 0.01	Trial: W33CA098489
				0[d]	0.35, 0.39 (0.37)	< 0.01,< 0.0.01 (< 0.01)	0.07, 0.08 (0.08)	0.03	0.02, 0.02	< 0.01	

DAT = days after third or fourth (last) application

[a] Ripe berries

[b] Green, ripe fruit and flowers

[c] samples taken after third treatment

[d] samples taken after fourth treatment

nd = not detected

Avocado

Four independent supervised residue decline field trials were conducted on avocado in Brazil during growing season 2007-2008. Four foliar applications of difenoconazole (SC formulation) at a rate of 0.05 kg ai/ha were made with a fourteen day interval. Single samples (avocado fruits) were collected and stored frozen for a maximum of 8.9 months. This storage period is covered by the storage stability studies (24 months)

Analysis of parent difenoconazole was made using HPLC-MS/MS and method POPIT MET.033. The limit of quantification was 0.01 mg/kg and the mean recovery was between 84±2.2% to 91±1.2% at fortification levels of (n=2) 0.01, 0.11 and 2.2 mg/kg.

Table 6 Residues in avocado after foliar application of difenoconazole from field trials in Brazil

Location	Application			Residues Fruit		Reference
AVOCADO Country, year (variety	Kg ai/ha	no	BBCH	DAT	Difenoconazole (mg/kg)	No A13703G_10284
Brazil (Mogi, Mirimi, SP) 2007/2008 (Giada)	0.050	1	71-73	0	0.13	Study/ Trial M08071-LZF1
	0.050	2	73	3	0.12	
	0.050	3	73-75	7	0.05	
	0.050	4	75-77	14	0.05	
				21	0.03	
Brazil (SP) 2008 (Hass)	0.050	1	76	0	0.29	Study/ Trial M08071-LZF2
	0.050	2	76	3	0.33	
	0.050	3	77	7	0.20	
	0.050	4	77	14	0.26	
				21	0.18	
Brazil (Taquaritnga, SP) 2007/2008	0.050	1	69-71	0	0.12	Study/ Trial: M08071-LZF3
	0.050	2	71	3	0.06	
	0.050	3	71	7	0.07	
	0.050	4	85	14	0.05	

Location	Application			Residues Fruit		Reference
AVOCADO Country, year (variety	Kg ai/ha	no	BBCH	DAT	Difenoconazole (mg/kg)	No A13703G_10284
(Giada)				21	0.01	
Brazil	0.050	1	75	0	0.04	Study/
(MG)	0.050	2	76	3	0.05	Trial
2008	0.050	3	78	7	0.02	M08071-JJB
	0.050	4	79	14	0.02	
(Margarida)				21	0.01	

DAT = days after last treatment

Soya beans (dry)

Eighteen independent supervised residue field trials on soya beans were conducted in USA during growing season 2008. Two foliar applications of difenoconazole (EC formulation) at a target rate of 0.129 kg ai/ha were made at an interval of seven to ten days. Duplicate samples of soya beans were collected except in the residue decline trials when single samples were taken. Samples were stored frozen for a maximum of 4.8 months. This storage period is covered by the storage stability studies (24 months).

Analysis of parent difenoconazole was made using LC-MS/MS and method REM 147.08. The limit of quantification was 0.01 mg/kg and the mean recovery was 100±11% (n=22) at fortification levels of 0.01–10.0 mg/kg.

Samples were also analysed for the triazole metabolites using LC/MS/MS and Morse Laboratories, Inc. (Analytical Method 160). The limit of quantitation (LOQ) for all analytes (as respective parent equivalents) for soybeans was 0.01 mg/kg. The limit of detection (LOD) for all metabolites based on the smallest detectable standard was 0.00003 µg/mL for 1, 2, 3-triazole and triazole alanine in all matrices. The LOD for triazole acetic acid was 0.00005 µg/mL in all matrices. The mean recovery for the metabolites was triazole (91±11%), triazole alanine (90±10%) and triazole acetic (99±7.4%) at fortification levels of 0.01 and 0.1 mg/kg (n=18).

Table 7 Residues in soya beans after foliar application of difenoconazole from field trials in USA

SOYA BEAN Country, year (variety)	Application			Residues (mean value in parenthesis) mg/kg beans							Reference
	kg ai/ ha	no	(BBCH)	DAT	Difenoconazole	1,2,4,-Triazole	Triazole alanine		Triazole acetic acid		Study: T002400-07 and ML 08-1488-SYN No A7402T_10144
Critical GAP USA; apply 0.129 kg ai/ha maximum 2 times at 7-10 days intervals. PHI 14 days											
					treated	treated	treated	control	treated	control	Trial
USA (NC)	0.127	12	88	0	0.44	nd	0.20	0.1	< 0.01	< 0.01	Trial:
Seven Spring[a]	0.124		88	7	0.02	nd	0.15	0.1	< 0.01	< 0.01	E10NC081261
				14	< 0.01, < 0.01 (0.01)	nd, nd	0.12, 0.13 (0.13)	0.09	< 0.01, < 0.01	< 0.01	
2008				20	< 0.01	nd	0.162	0.11	< 0.01	< 0.01	
(DKB 64-51 (SE 74480))				28	< 0.01	< 0.01	0.12	0.16	< 0.01	< 0.01	
USA (IA)	0.121	1	93	0	0.08	nd,	0.069	0.098	< 0.01,	< 0.01	Trial:
2008	0.123	2	95						< 0.01		C30IA081274
Bagely				7	< 0.01, < 0.01	nd, 0.01	0.082	0.12	< 0.01	< 0.01	
(93M11)				14	0.019, 0.018	nd, nd	0.088,	0.08	< 0.01	< 0.01	

SOYA BEAN Country, year (variety)	Application			Residues (mean value in parenthesis) mg/kg beans							Reference
	kg ai/ ha	no	(BB CH)	DAT	Difenoconazole	1,2,4,-Triazole	Triazole alanine		Triazole acetic acid		Study: T002400-07 and ML 08-1488-SYN No A7402T_10144
Critical GAP USA; apply 0.129 kg ai/ha maximum 2 times at 7-10 days intervals. PHI 14 days											
					treated	treated	treated	control	treated	control	Trial
					(0.019)		0.092 (0.09)				
				21	< 0.01	nd	0.08	0.12	< 0.01	< 0.01	
				33	0.016	nd	0.042	0.09	< 0.01	< 0.01	
USA (NC) 2008 Seven Spring[a] (95M50	0.123 0.124	1 2	93 88	14	< 0.01, < 0.01 (0.01)	nd, nd	0.043, 0.046 (0.05)	0.085	< 0.01, < 0.01	< 0.01	Trial: E10NC081262
USA (MO) 2008 Fisks (Armor 47G7)	0.124 0.123	1 2	81 87	14	< 0.01, 0.014 (0.012)	nd, nd	0.114, 0.09 (0.10)	0.134	< 0.01, < 0.01	< 0.01	Trial: C23MO081263
USA 2008 (Washington, LA) (AG5605)	0.129 0.124	1 2	82 85	14	0.012, 0.019 (0.016)	nd, nd	0.068, 0.037 (0.05)	0.066	< 0.01, < 0.01	< 0.01	Trial: E18LA081264
USA 2008 (Washington, LA) (AG5605)	0.124 0.123	1 2	82 85	14	0.042, 0.038 (0.04)	< 0.01, < 0.01	0.089, 0.073 (0.08)	0.062	< 0.01, < 0.01	< 0.01	Trial: E18LA081265
USA (MO) 2008 Oregon (Pioneer 93M11)	0.123 0.127	1 2	R6 R6-R7	14	0.012, 0.013 (0.013)	< 0.01, nd	0.084, 0.085 (0.09)	0.053	< 0.01, < 0.01	< 0.01	Trial: C19MO081266
USA (MO) 2008 St Joseph (Pioneer) 93M96)	0.126 0.124	1 2	R5 R6	14	< 0.01, < 0.01, (0.01)	nd, nd	0.154, 0.191 (0.17)	0.09	< 0.01, < 0.01	< 0.01	Trial:, C19MO081267
USA (WI) 2008 Dunn (S17-A1)	0.122 0.119	1 2	75 81	14	< 0.01, < 0.01, (0.01)	nd, nd	0.07, 0.078 (0.074)	0.11	< 0.01, < 0.01	< 0.01	Trial: C08WI1081268
USA (WI) 2008 Fitchburg (S17-V2)	0.125 0.125	1 2	74 80	14	0.026, 0.015 (0.021)	nd, nd	0.086, 0.086 (0.09)	0.07	< 0.01, < 0.01	< 0.01	Trial: C08WI1081269
USA (ND) 2008 Asgrow AG0202	0.123 0.125	1 2	82 88	15	< 0.01, < 0.01 (0.01)	nd, nd	0.118, 0.198 (0.16)	0.110	< 0.01, < 0.01	< 0.01	Trial: C13ND081270
USA (NE) York, (NC+2A46RR)	0.123 0.125	1 2	93 95-97	11	< 0.01, < 0.01 (0.01)	nd, nd	0.129, 0.126 (0.13)	0.146	< 0.01, < 0.01	< 0.01	Trial: E13NE081271
USA (Osceola, NE) 2008 (NC+2A46RR)	0.122 0.123	1 2	93-95 97	13	< 0.01, < 0.01 (0.01)	nd, nd	0.156, 0.178 (0.17)	0.162	< 0.01, < 0.01	< 0.01	Trial: E13NE081272

SOYA BEAN Country, year (variety)	Application			Residues (mean value in parenthesis) mg/kg beans							Reference
	kg ai/ ha	no	(BB CH)	DAT	Difenoconazole	1,2,4,-Triazole	Triazole alanine		Triazole acetic acid		Study: T002400-07 and ML 08-1488-SYN No A7402T_10144
Critical GAP USA; apply 0.129 kg ai/ha maximum 2 times at 7-10 days intervals. PHI 14 days											
					treated	treated	treated	control	treated	control	Trial
USA (IA) Berkely 2008 (93M11)	0.121 0.126	1 2	79 95	14	< 0.01, < 0.01 (0.01)	nd, nd	0.079, 0.077 (0.08)	0.059	< 0.01, < 0.01	< 0.01	Trial: C301A081273
USA 2008 (Lime Springs, IA) (52726085)	0.125 0.125	1 2	86 88	14	0.022, 0.15 (0.087)	nd, nd	0.0590, 0.0635 (0.06)	0.052	< 0.01, < 0.01	< 0.01	Trial E19A081275
USA 2008 (Lime Springs, IA) (52726085)	0.123 0.123	1 2	86 88	14	0.067, 0.092 (0.079)	< 0.01, < 0.01	0.034, 0.0295 (0.03)	0.037	< 0.01, < 0.01	< 0.01	Trial E19A081276
USA (IA) 2008 Richland (Pioneer 93M11)	0.124 0.125	1 2	79 83	15	< 0.01, < 0.01 (0.01)	nd, nd	0.0575, 0.0462, (0.05)	0.036	< 0.01, < 0.01	< 0.01	Trial C18A081277
USA (IA) Hedrick 2008 (Pioneer 93M11)	0.123 0.124	1 2	79 95	14	< 0.01, < 0.01 (0.01)	nd, < 0.01	0.0555 0.0418 (0.05)	0.036	< 0.01, < 0.01	< 0.01	Trial C18A081278
USA (ND) Gardner 2008 5B077RR	0.127 0.124	2	85 87	14	< 0.01, < 0.01 (0.01)	nd, < 0.01	0.310 0.324 (0.32)	0.299	0.023,0.026 (0.03)	0.016	Trial C12MN081279
USA (MN) Perley 2008 (5A009RR)	0.130 0.121	2	85 89	14	< 0.01, < 0.01 (0.01)	nd, nd	0.264, 0.282 (0.28)	0.332	0.013 0.013 (0.01)	0.013	Trial C12MN081280

DAT = days after last treatment

nd = not detected

[a] Different due to different of 2.5 weeks difference in application times and different cultvars

R5 = BBCH 50-59Beginning Seed: Seed in one of the four uppermost nodes with fully developed leaves is 1/8 in. long.

R6 = BBCH60-69 Full Seed: Pod containing a green seed filling the pod cavity is present at one of the top four nodes.

R7 =BBCH 70-79 Beginning Maturity: One normal pod on the main stem has reached its mature pod colour. At this stage, the crop is safe from a killing frost.

Rice

Eight independent supervised residue field trials on rice were conducted in Italy in 2009 and 2010. Two foliar applications of difenoconazole (EC or SC) were made with a fifteen days interval at a target rate of 0.125 kg ai/ha. Duplicate samples of whole plant, grain and straw were collected and

maintained in frozen storage for periods up to 14 months for whole plants and 13 months for grain and straw. This storage period is covered by the storage stability studies (24 months)

Analysis of parent difenoconazole (on one of the duplicate sample) was made using LC-MS/MS and method REM 147.08. The limit of quantification was 0.01 and the mean recovery was 102±14% (whole plant), 108±12% (grain) and 105±10% for straw at fortification levels of (n=1–2) 0.01, 0.1 and 8 mg/kg.

Analysis of the metabolites was made using Syngenta method GRM053.01A for triazole metabolites T, TA, TAA and triazole lactic acid (TLA). The method is validated for cereals (including rice) whole plant, grain and straw with a LOQ of 0.01 mg/kg for each metabolite.

Table 8 Residues in rice grain after foliar application of difenoconazole from field trials in Europe

RICE Country, year (variety) Trial no	Application			Residues* mg/kg								Reference
	g ai/ ha	**no**	(BBCH)	DAT	Matrix	Difenoconazole	Triazole alanine	Triazole acetic acid		Triazole lactic acid		No A7402T_ 10138, 10139, No A13703G _10496
Critical GAP EU; apply 0.125 kg ai/ha maximum 2 times at BBCH 21-29 with a 15 days interval. PHI 28 days.												
						treated	treated	control	treated	control	treated	control	
Europe Italy 2009 (Ercole S-09-01473-01	133[a]	1	71-74 83	21	Grain	0.85	0.07	-	0.05	-	< 0.01	-	Study: S09-01473
	132[a]	2	83	28	Grain	0.76	0.06	0.03	0.06	0.02	< 0.01	< 0.01	
Europe Italy 2009 (ValoneNano) S-09-01473-02	118[a]	1	71-75	21	Grain	0.9	0.03	-	0.04	-	< 0.01	-	Study: S09-01473
	122[a]	2	73-77	28	Grain	0.85	0.03	0.02	0.04	0.01	< 0.01	< 0.01	
Europe Italy 2010 (Ercole) S10-00370-01[g]	133[a]	1	69-73	21	Grain	0.75	0.12	-	0.07	-	< 0.01	-	Study: S10-00370
	133[a]	2	77-83	28	Grain	0.68	0.12	0.06	0.09	0.07	< 0.01	< 0.01	
Europe Italy 2010 (Scudo) S10-00370-02[f]	127[a]	1	69-73	21	Grain	1.2	0.40	-	0.33	-	0.01	-	Study: S10-00370
	113[a]	2	77-83	28	Grain	1.1	0.33	0.24	0.26	0.25	< 0.01	< 0.01	
Europe	144[b]	1	69	21	Grain	0.84	0.09	0.03	0.05	0.03	< 0.01	< 0.01	Study:

RICE Country, year (variety) Trial no	Application			Residues* mg/kg									Reference
	g ai/ ha	**no**	(BBCH)	DAT	Matrix	Difenoconazole	Triazole alanine		Triazole acetic acid		Triazole lactic acid		No A7402T_10138, 10139, No A13703G_10496
Critical GAP EU; apply 0.125 kg ai/ha maximum 2 times at BBCH 21-29 with a 15 days interval. PHI 28 days.													
						treated	treated	control	treated	control	treated	control	
Italy 2010 (Volano) S10-00370-01	144 [b]	2	72-73										S10-00372
				28	Grain	0.86	0.08	-	0.04	-	< 0.01	-	
Europe Italy 2010 (Scudo) S10-00370-02 [e]	147 [b]	1	69-73	21	Grain	1.8, 1.3 (1.6)	0.27 [c]	0.36 [c]	0.14 [c]	0.18 [c]	< 0.01 [c]	< 0.01 [c]	Study: S10-00372
	145 [b]	2	76										
				28	Grain	1.4	0.39	-	0.17	-	< 0.01	-	
Europe Italy 2010 (Ercole) S10-00370-03 [d]	146 [b]	1	69-73	21	Grain	0.95	0.10	0.06	0.05	0.03	< 0.01	< 0.01	Study: S10-00372
	146 [b]	2	77-83										
				28	Grain	0.78	0.13	-	0.04	-	< 0.01	-	
Europe Italy 2010 (SIS R215) S10-00370-03	143 [b]	1	83-85	21	Grain	1.2	0.20	0.08	0.07	0.04	< 0.01	< 0.01	Study: S10-00372
	139 [b]	2	85-87										
				28	Grain	1.1	0.17		0.07		< 0.01		

*1,2,4-triazole was measured but was not detected in any trial, therefore not reported here.

- Data not available

[a] EC formulation

[b] SC formulation in mixture with azoxystrobin

DAT = days after last treatment

nd = not detected

Cotton

Eight independent supervised residue field trials on cotton were conducted in Brazil during growing season 2006 and 2007/08. Four trials were made with four foliar applications of difenoconazole (SC formulation) at a target rate of 0.075 kg ai/ha, an interval of 14 days and sampling after 7, 4 and 21 days. An additional four trials were made with five foliar applications of difenoconazole (SC formulation) at a target rate of 0.075 kg ai/ha, an interval of 21 days (after the last two applications)

and sampling after 30 days. Single samples (cotton bolls) were taken and stored frozen maximum 8.1 months. This storage period is covered by the storage stability studies (24 months).

Analysis of parent difenoconazole in seeds was made using HPLC-MS/MS and method POPIT MET.033. The limit of quantification was 0.01 mg/kg for and the mean recovery was 84±5% at fortification levels of (n=8) 0.01 and 0.1 mg/kg.

Table 9 Residues in cotton after foliar application of difenoconazole from field trials in Brazil

COTTON Country, year (variety	Application				Residues			Reference
	g ai/ha	no	interval days	BBCH	DAT	matrix	Difenoconazole (mg/kg)	No A13703G_10323, No A15265A_10006
Critical GAP Brazil; apply 0.075 kg ai/ha maximum 3 times at 10-15 days intervals. PHI 21 days								
Brazil (Holambra)	75	4	0	71	7	seed	0.02	Study: M05022 Trial: M505022-LZF1
			14	75	14	seed	0.02	
			14	81	21	seed	0.02	
2006			14	87				
(IAC24,)								
Brazil (Bandeiantes)	75	4	0	73	7	seed	0.02	Study: M05022 Trial: M505022-LZF2
			14	79	14	seed	0.02	
			14	79-80	21	seed	0.02	
2006			14	80				
(IPR 96)								
Brazil (Uberlandia)	75	4	0	73	7	seed	0.04	Study: M05022 Trial: M05022-JJB1
			14	79	14	seed	0.01	
			14	81	21	seed	< 0.01	
2006			14	83				
(IPR 96)								
Brazil (Guaira)	75	4	0	75	7	seed	0.01	Study: M05022 Trial: M05022-JJB2
			14	77	14	seed	0.02	
			14	79	21	seed	0.01	
2006 (Delta Penta)			14	83				
Brazil (Coelho)	75	5	0	13-19				Study: M08065 Trial: M08065 -LZF1
			21	29				
			21	40				
2007/08			14	51				
(Delta Oppal,)			77	70	30	seed	< 0.01	
Brazil (Bandeirantes)	75	5	0	12				Study: M08065 Trial: M08065 LZF2
			20	21-22				
			22	39				
2007/08			45	71				
(Copetec 401)			52	71-73	30	seed	< 0.01	
Brazil (Uberlandia)	75	5	0	14				Study: M08065 Trial: M08065 JJB1
			21	18-19				
			21	57				
2007/08			14	60				
(Nu Opal,)			99	81	30	seed	< 0.01	
Brazil (Goiania)	75	5	0	14				Study: M08065 Trial: M08065 -JJB2
			21	22				
			21	60				
2007/08			14	63				
(Nu Opal,)			66	80	30	seed	< 0.01	

DAT = days after last treatment

Peanut

Eight supervised residue field trials on peanuts were conducted in Brazil during growing seasons 2008 and 2009/10. Four trials were made with six foliar applications (SC formulation) at a rate of 0.125 kg ai/ha. Single samples (peanut plants) were collected 14, 22 and 38 days after last application and after the plants were dried, the pods were removed from the plants and threshed using a small machine. The seeds were stored frozen at maximum 7.6 months. This storage period is covered by the storage stability studies (24 months).

Analysis of parent difenoconazole from seeds in these trials was made using method HPLC-MS/MS and method POPIT MET.033. The limit of quantification was 0.01 mg/kg and the mean recovery was between 84±3% to 89±2% at fortification levels of (n=5–7) 0.01 and 0.1 mg/kg.

An additional four field trials were conducted in Brazil during growing season 2007/08 with three foliar applications at rate of 0.0875 kg difenoconazole (EC formulation). Single samples (peanut plants) were sampled and peanut kernel stored frozen for a maximum of 4.5 months. The storage period is covered by the storage stability studies (24 months).

Analysis of parent difenoconazole from seeds in these trials was made using HPLC-MS/MS and method POPIT MET.032. The limit of quantification was 0.01 mg/kg and the mean recovery was between 73±3% to 82±9% at fortification levels of (n=5–7) 0.01 and 0.1 mg/kg.

Table 10 Residues in peanut kernel after foliar application of difenoconazole in field trials from Brazil

PEANUT Country, year (variety	Application				Residues			Reference
	kg ai/ha	no	interval days	BBCH	DAT	matrix*	Difenoconazole (mg/kg)	No A16976A_10030, No A7402N_10001
Critical GAP Brazil; apply 0.0875 kg ai/ha maximum 3 times, interval not defined. PHI 22 days.								
Brazil (Sao Palo,) 2009/10 (Runner)	0.125	6	0	59-60	7	peanuts	< 0.01	Study: M10070 Trial: M10070-LZF
			13	61-63	14	peanuts	< 0.01	
			14	63-65	22	peanuts	< 0.01	
			14	65-67	28	peanuts	< 0.01	
			14	73-75				
			14	78-79				
Brazil (Parana) 2009/10 (Super Tatu)	0.125	6	0	60	7	peanuts	< 0.01	Study: M10070 Trial: M10070-JJB
			14	67	14	peanuts	< 0.01	
			14	71	22	peanuts	< 0.01	
			14	75	28	peanuts	< 0.01	
			14	79			< 0.01	
			14	81			< 0.01	
Brazil (Jacoboticabal, Sao Palo) 2009/10 (Alto oleico)	0.125	6	0	13-14	7	peanuts	< 0.01	Study: M10070 Trial: M10070-AMA1
			14	23-29	14	peanuts	< 0.01	
			14	51-61	22	peanuts	< 0.01	
			14	63-67	28	peanuts	< 0.01	
			14	69-71				
			14	75				

PEANUT Country, year (variety	Application				Residues			Reference
	kg ai/ha	no	interval days	BBCH	DAT	matrix*	Difenoconazole (mg/kg)	No A16976A_10030, No A7402N_10001
Critical GAP Brazil; apply 0.0875 kg ai/ha maximum 3 times, interval not defined. PHI 22 days.								
Brazil (Vista Alegro do Alto, Sao Paulo) 2009/10 (Alto oleico)	0.125	6	0	13-15	7	peanuts	< 0.01	Study: M10070 Trial: M10070-AMA1
			14	55	14	peanuts	< 0.01	
			14	61	22	peanuts	< 0.01	
			14	65	28	peanuts	< 0.01	
			14	69			< 0.01	
			14	69			< 0.01	
Brazil (Sao Paulo) 2008 (Super Tatu Vermelho)	0.088	3	0	73	14	peanuts	< 0.01	Study: M08013 Trial: M08013-LZF1
			7	75	22	peanuts	< 0.01	
			7	76-77	28	peanuts	< 0.01	
Brazil (Parana) 2008 (Tatu Vermelho)	0.088	3	0	77	14	peanuts	< 0.01	Study: M08013 Trial: M08013-LZF2
			7	77-79	22	peanuts	< 0.01	
			7	70-80	28	peanuts	< 0.01	
Brazil (Goias) 2008 (Tatu)	0.088	3	0	77	14	peanuts	< 0.01	Study: M08013 Trial: M08013-JJB1
			7	79	22	peanuts	< 0.01	
			7	82	28	peanuts	< 0.01	
Brazil (Minas Gerais) 2008 (Tatu)	0.088	3	0	79-81	14	peanuts	< 0.01	Study: M08013 Trial: M08013-JJB2
			7	81-83	22	peanuts	< 0.01	
			7	83-85	28	peanuts	< 0.01	

DAT = days after last treatment

*Peanut plants were sampled. After the plants were dried, the pods were removed from the pods. Threshing was done on a small machine

Rape seed (Canola)

Thirteen independent supervised field trials on oilseed rape were conducted in Canada during growing season 2011. One foliar application (EC formulation) was made at the target rate of 0.125 kg ai/ha. Duplicate samples of were collected 30 days after the application. Rape seed samples were stored frozen for periods up to 4.7 months. This storage period is covered by the storage stability studies (24 months).

Analysis of parent difenoconazole from seeds in these trials was made using LC-MS/MS and method REM 147.08. The limit of quantification was 0.01 mg/kg for and the mean recovery was between 88±11% to 107±17% at fortification levels of (n=3–4) 0.01, 0.1 and 0.2 mg/kg.

Table 11 Residues of parent difenoconazole in oilseed rape from field trials in Canada

OILSEED RAPE, (CANOLA) Country, year (variety	Application					Residues (mean value in parenthesis)			Reference
	g ai/hl	water L/ha	kg ai/ha	no	BBCH	DAT	matrix	Difenoconazole (mg/kg)	No A15457B_50038, Study: CER 05903/11
GAP Canada; apply 0.125 kg ai/ha one time at BBCH 12-18[a]or at BBCH 62-65[b]. PHI 30 days.									
Canada (Elm Creek, MB) 2011 (1841 RR)	302	45	0.136		69-73	29	seed	0.017, 0.013 (0.015)	Trial: T938
Canada (Morden, MB) 2011 (1841 RR)	305	45	0.137		67-69	30	seed	0.81, 0.043 (0.062)	Trial: T938C
Canada (Kinley, SK) 2011 (1841 RR)	282	45	0.127	1	67-71	30	seed	0.056, 0.070 (0.063)	Trial: T939
Canada (Kinley, SK) 2011 (72-55) RR)	63	200	0.126	1	69-73	30	seed	0.023, 0.023 (0.023)	Trial: T940
Canada (Elgin, MB) 2011 (72-55) RR)	277	45	0.125	1	68	30	seed	0.042, 0.024 (0.033)	Trial: T941
Canada (Elgin, MB) 2011 (72-55) RR)	63	200	0.125	1	78-79	30	seed	0.036, 0.021 (0.029)	Trial: T942
Canada (Rosthern, SK) 2011 (1841 RR)	65	200	0.130	1	73-76	31	seed	0.031, 0.044 (0.038)	Trial: T943
Canada (Minto, MB) 2011 (72-55) RR)	62	200	0.123	1	67	35	seed	< 0.01, < 0.01 (< 0.01)	Trial: T944
Canada (Alvena, SK) 2011 (72-55) RR)	58	200	0.116	1	65-66	31	seed	0.010, 0.019, (0.015)	Trial: T945
Canada (Fort Sask.AB) 2011 (72-55) RR)	65	200	0.129	1	67-71	32	seed	0.040; 0.026 (0.033)	Trial: T946
Canada (Minto, MB) 2011 (1841 RR)	62	200	0.124	1	67	25	seed	0.025	Trial: T947
						30		< 0.01, 0.012 (< 0.01)	
						35		< 0.01	
						40		< 0.01	

OILSEED RAPE, (CANOLA) Country, year (variety	Application					Residues (mean value in parenthesis)			Reference
	g ai/hl	water L/ha	kg ai/ha	no	BBCH	DAT	matrix	Difenoconazole (mg/kg)	No A15457B_50038, Study: CER 05903/11
GAP Canada; apply 0.125 kg ai/ha one time at BBCH 12-18[a]or at BBCH 62-65[b]. PHI 30 days.									
Canada (Elgin, MB) 2011 (1841 RR)	62	200	0.124	1	68	31	seed	0.011, < 0.01 (0.011)	Trial: T948
Canada (Rosthern, SK) 2011 (72-55 RR)	65	200	0.130	1	73-76	31	seed	0.037, 0.035 (0.036)	Trial: T949

DAT = days after last treatment

[a] Virulent Black Leg

[b] Sclerotinia Stem Rot

Animal feeds

Rice straw and whole crops silage,

For information on the trials see, Table 8.

Table 12 Residues of difenoconazole in rice whole crop silage following foliar application in field trials from Europe

RICE Country, year (variety)	Application			Residues* mg/kg									Reference
	g ai/ha	no	(BBCH)	DAT	Matrix	Difenoconazole	Triazole alanine		Triazole acetic acid		Triazole lactic acid		No A7402T_10138, 10139, No A13703G_10496
Critical GAP EU; apply 0.125 kg ai/ha maximum 2 times at 15 days interval. PHI 28 days.													
						treated	treated	control	treated	control	treated	control	
Europe Italy 2009 (Ercole	133[a]	1	71-74 83	0	Whole plant	3.5	0.01	0.01	0.03	0.02	0.01	0.02	Study: S09-01473 Trial no: S-09-01473-01
	132 [a]	2	83	7	Whole plant	1.8	0.04		0.05		0.02		
				14	Whole plant	1.4	0.04		0.04		0.02		
Europe Italy 2009 (ValoneNano)	118 [a]	1	71-75	0	Whole plant	6.3	< 0.01	< 0.01	0.03	0.03	0.02	0.02	Study: S09-01473 Trial no: S-09-01473-02
				7	Whole plant	2.6	< 0.01	-	0.04		0.02		
				14	Whole plant	1.6	< 0.02	-	0.05		0.02		
Europe Italy 2010 (Ercole)	133 [a]	1	69-73	0	Whole plant	6.1	0.07	0.05	0.07	0.07	0.02	0.03	Study: S10-00370 Trial no: S10-00370-01[g]
	133 [a]	2	77-83	7	Whole plant	2.1	0.04		0.07		0.02		
				14	Whole plant	1.4	0.06		0.10		0.01		
Europe Italy	127 [a]	1	69-73	0	Whole plant	5.2	0.17	0.16	0.19	0.24	0.09	0.09	Study: S10-00370
	113 [a]	2	77-	7	Whole	2.6	0.15		0.20		0.06		Trial no:

RICE Country, year (variety)	Application			Residues* mg/kg									Reference
	g ai/ ha	no	(BBCH)	DAT	Matrix	Difenoconazole	Triazole alanine		Triazole acetic acid		Triazole lactic acid		No A7402T_10138, 10139, No A13703G_10496
Critical GAP EU; apply 0.125 kg ai/ha maximum 2 times at 15 days interval. PHI 28 days.													
						treated	treated	control	treated	control	treated	control	
2010			83		plant								S10-00370-02[f]
(Scudo)				14	Whole plant	1.4	0.20		0.22		0.06		
Europe Italy	144[b]	1	69	0	Whole plant	3.7	0.02	0.02	0.06	0.04	0.04	0.03	Study: S10-00372
2010	144 [b]	2	72-73	7	Whole plant	3.3	0.04		0.07		0.05		Trial no: S10-00370-01
(Volano)				14	Whole plant	2.5	0.02		0.07		0.03		
Europe Italy	147 [b]	1	69-73	0	Whole plant	4.6	0.19	0.13	0.20	0.13	0.06	0.06	Study: S10-00372
2010	145 [b]	2	76	7	Whole plant	2.8	0.20		0.24		0.06		Trial no: S10-00370-02[e]
(Scudo)				14	Whole plant	2.5	0.17		0.23		0.06		
Europe Italy	146 [b]	1	69-73	0	Whole plant	5.6	0.06	0.04	0.08	0.05	0.02	0.02	Study: S10-00372
2010	146 [b]	2	77-83	7	Whole plant	2.4	0.06		0.08		0.01		Trial no: S10-00370-03 [d]
(Ercole)				14	Whole plant	1.8	0.07		0.06		0.02		
Europe Italy	143 [b]	1	83-85	0	Whole plant	4.6	0.13	0.06	0.11	0.06	0.04	0.04	Study: S10-00372
2010	139 [b]	2	85-87	7	Whole plant	2.9	0.12		0.10		0.04		Trial no: S10-00370-03
(SIS R215)				14	Whole plant	2.5	0.09		0.06		0.04		

*1,2,4-triazole was measured but was not detected in any trial.

- Data not available

[a] EC formulation

[b] SC formulation in mixture with azoxystrobin

[c] Treated and untreated grain samples 21 DAT have been mixed up

DAT = days after last treatment

nd = not detected

Table 13 Residues of difenoconazole in rice straw following foliar application in field trials from Europe

RICE Country, year (variety)	Application			Residues* mg/kg									Reference
	g ai/ ha	no	(BBCH)	PHI	Matrix	Difenoconazole	Triazole alanine		Triazole acetic acid		Triazole lactic acid		No A7402T_10138, 10139, No A13703G_10496
Critical GAP EU; apply 0.125 kg ai/ha maximum 2 times at 15 days interval. PHI 28 days.													
						treated	treated	control	treated	control	treated	control	
Europe Italy	133[a]	1	71-74 83	0									Study: S09-01473 Trial no:

RICE Country, year (variety)	Application			Residues* mg/kg									Reference
	g ai/ ha	no	(BBCH)	PHI	Matrix	Difenoconazole	Triazole alanine		Triazole acetic acid		Triazole lactic acid		No A7402T_10138, 10139, No A13703G_10496
Critical GAP EU; apply 0.125 kg ai/ha maximum 2 times at 15 days interval. PHI 28 days.													
						treated	treated	control	treated	control	treated	control	
2009 (Ercole	132 [a]	2	83	21	Straw	1.1	< 0.01		0.06		0.03		S-09-01473-01
				28	Straw	1.0	< 0.01	< 0.01	0.07	0.02	0.03	0.02	
Europe Italy	118 [a]	1	71-75	0									Study: S09-01473
	122 [a]	2	73-77	21	Straw	1.4	< 0.01		0.08		0.03		Trial no:
2009				28	Straw	1.4	< 0.01	< 0.01	0.09	0.03	0.02	0.02	S-09-01473-02
(ValoneNano)													
Europe Italy	133 [a]	1	69-73	0									Study: S10-00370
	133 [a]	2	77-83										Trial no:
2010													S10-00370-01[g]
(Ercole)				28	Straw	2.6	0.01	0.02	0.12	0.10	0.05	0.03	
Europe Italy	127 [a]	1	69-73	0									Study: S10-00370
	113 [a]	2	77-83	21	Straw	1.9	0.05		0.28		0.12		Trial no:
2010													S10-00370-02[f]
(Scudo)				28	Straw	1.6	0.03	0.04	0.30	0.21	0.12	0.15	
Europe Italy	144 [b]	1	69	0									Study: S10-00372
	144 [b]	2	72-73										Trial no:
2010				21	Straw	2.3	< 0.01	< 0.01	0.09	0.08	0.07	0.04	S10-00370-01
(Volano)				28	Straw	1.8	0.01		0.09		0.07		
Europe Italy	147 [b]	1	69-73	0									Study: S10-00372
	145 [b]	2	76										Trial no:
2010				21	Straw	3.0	0.03	0.04	0.38	0.23	0.16	0.11	S10-00370-02[e]
(Scudo)				28	Straw	2.2	0.04		0.29		0.11		
Europe Italy	146 [b]	1	69-73	0									Study: S10-00372
	146 [b]	2	77-83										Trial no:
2010													S10-00370-03 [d]
				21	Straw	4.3	0.02	0.01	0.14	0.08	0.05	0.04	
(Ercole)				28	Straw	2.2	0.02		0.11		0.05		
Europe Italy	143 [b]	1	83-85	0									Study: S10-00372
	139 [b]	2	85-87										Trial no:
2010													S10-00370-03
				21	Straw	2.2	0.03	0.02	0.16	0.10	0.13	0.08	
(SIS R215)				28	Straw	2.0	0.03		0.17		0.11		

*1,2,4-triazole was measured but was not detected in any trial.

- Data not available

[a] EC formulation

[b] SC formulation in mixture with azoxystrobin

[c] Treated and untreated grain samples 21 DAT have been mixed up

DAT = days after last treatment

nd = not detected

FATE OF RESIDUES IN STORAGE AND PROCESSING

Residues after processing

As a measure of the transfer of residues into processed products, a processing factor was used, which is defined as:

Processing factor (Pf) parent difenoconazole =

$$\frac{\text{Residues in processed product (mg/kg)}}{\text{Residues in raw agricultural commodity (mg/kg)}}$$

Processing factor (PF) for each triazole metabolite =

$$\frac{\text{Residues in treated processed product – residues in untreated processed product (mg/kg)}}{\text{Residues in treated raw agricultural commodity (RAC) – residues in untreated RAC (mg/kg)}}$$

If residues in the RAC were below 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 in 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). If residues in the processed commodity were below what was found in untreated processed commodity no processing factor was calculated.

Soya beans

Two studies on the conduct of difenoconazole during processing of soya bean into meal, hulls and refined oil and one study for the processing into aspired grains was conducted by Willard, TR and Mäyer JT (2008, T002400-07). Field trials of soya bean was treated with two applications at a target rate of 0.65 kg ai/ha. Samples of soya beans were collected 14 days after the last application. Duplicate field samples and processed fractions were analysed for parent difenoconazole using method REM 147.08. LOQ for parent difenoconazole was 0.01 mg/kg and the mean recovery was in meal 108% at fortification level of (n=2) 0.01–5.0, in hulls 106% at fortification level of (n=2) 0.01–5.0, in refined oil 88% at fortification level of (n=2) 0.01–0.05 and in aspirated grain fraction (AEG) 112±6.6% at fortification level (n=4) 0.01–250 mg/kg- Each triazole metabolite was analysed using method No 160 rev.2 Morse Laboratories. LOQ for all triazole analytes were 0.01 mg/kg.

Samples of RAC (soya beans) were stored frozen for a maximum of 4.8 months and the duration of the storage for the processed fractions meals, hulls, refined oil and aspired grain fractions were 3.2, 5.5, 3.2 and 10.4 months, respectively.

Processing of meal, hulls and refined oil

Cleaned whole soybeans were fed into a roller mill to crack the hull and liberate the kernel. After hulling, the material was passed through an aspirator to separate hull and kernel material. The moisture content of the kernel material was determined and adjusted to 13.5%. Kernel material was heated to 71–79 °C and flaked in a flaking roll with a gap setting of 0.2–0.33 mm. Flakes were extruded in a continuous processor, where they were turned into collets by direct steam injection and compression. After extrusion, the collets were oven dried, placed in stainless steel batch extractors and submerged in hexane at 49–60 °C. After 30 minutes, the hexane was drained and fresh hexane was added to repeat the cycle twice.

The solvent was evaporated from the extracted flakes and the oil fraction to give meal and crude oil. The crude oil was treated with sodium hydroxide to remove free fatty acids. The neutralized oil was centrifuged and the supernatant, refined oil decanted.

Processing of aspirated grain fraction

To generate aspirated grain fractions (AGF), the samples were placed in a dust generation room containing a holding bin, two bucket conveyors, and a screw conveyor. As the samples were moved for 120 minutes in the system, aspiration was used to remove light impurities (grain dust). Light impurities were classified by sieving using 2.36, 2.0, 1.18, 0.85 and 0.425 mm sieves. After classification of each sample, the material collected through the 2.36 mm sieve was recombined to produce one aspirated grain fraction.

Residues determined in soya bean and processed fractions meal, hulls, refined oil and aspirated grain fraction are shown in table 14 and 15.

Table 14 Residues from parent difenoconazole in soya beans (RAC and processed fractions)

Trial Location, year , (variety), dose rate, interval DALT	Processed fraction	Difenoconazole parent, mg/kg (mean value in parenthesis)	Processing factor parent
C13ND081270 USA, (ND) 2008 (Asgrow) 0.614+0.608 kg ai/ha interval 7days, DALT=14	Soya bean, seeds (RAC)	< 0.01, 0.0128 < 0.0247 (0.016)	-
	Meal	< 0.01, < 0.01 (< 0.01)	0.63
	Hulls	0.0540, 0.0536 (0.054)	3.38
	Refined Oil	0.0158, 0.0180 (0.017)	1.06
C12MN081279 USA, (ND) 2008 (5B077RR) 0.618+0.634 interval 7 days, DALT=14	Soya bean (RAC)	0.049, 0.074, 0.107 (0.077)	
	Meal	< 0.01, < 0.01 (< 0.01)	0.13
	Hulls	0.045, 0.048 (0.047)	0.61
	Refined Oil	0.028, 0.036 (0.032)	0.42
C12MN081281 USA,(ND) 2008 (5A009RR) 0.618+0.621 interval 7 days, DALT=12	Soya bean , seed (RAC)	0.363, 0.31, 0.368 (0.347)	
	AGF	190, 214, 244 (216)	622

Table 15 Levels of triazole metabolites from difenoconazole in soya bean (RAC and processed fractions) *In parenthesis average of the three replicates*

Trial Location, year , (variety), dose rate, interval DALT	Matrix	Treatment 1=control 2=treated	1,2,4 Triazole mg/kg	Pf	Triazole alanine mg/kg	Pf	Triazole acetic acid mg/kg	Pf
C13ND081270 USA, (ND) 2008 (Asgrow) 0.614+0.608 kg ai/ha interval 7 days, DALT=14	Soya bean (RAC)	1	nd		0.068		< 0.01	
	Soya bean (RAC)	2	nd		0.113		< 0.01	
	Soya bean (RAC)	2	nd		0.164		< 0.01	
	Soya bean (RAC)	2	nd (nd)		0.160 (0.146)		< 0.01 (< 0.01)	
	Meal	1	nd		0.113		< 0.01	
	Meal	2	nd	< 0.01	0.143,	0.45	< 0.01	0.01
	Meal	2	nd (nd)		0.152 (0.148)		< 0.01 (nd)	
	Hulls	1	nd		0.026		< 0.01	
	Hulls	2	nd	< 0.01	0.052	0.31	< 0.01	0.01
	Hulls	2	nd		0.049 (0.05)		< 0.01	

Trial Location, year , (variety), dose rate, interval DALT	Matrix	Treatment 1=control 2=treated	1,2,4 Triazole mg/kg	Pf	Triazole alanine mg/kg	Pf	Triazole acetic acid mg/kg	Pf
			(nd)				(< 0.01)	
	Refined Oil	1	nd		nd		< 0.01	
	Refined Oil	2	nd	< 0.01	nd	< 0.01	< 0.01	0.01
	Refined Oil	2	nd (nd)		nd (nd)		< 0.01 (< 0.01)	
C12MN081279 USA, (ND) 2008 (5B077RR) 0.618+0.634 interval 7 days, DALT=14	Soya bean (RAC)	1	nd		0.396		0.017	
	Soya bean (RAC)	2	nd		0.555		0.019	
	Soya bean (RAC)	2	nd		0.585		0.02	
	Soya bean (RAC)	2	nd (nd)		0.605 (0.582)		0.022 (0.02)	
	Meal	1	< 0.01		0.388		0.028	
	Meal	2	< 0.01	0.01	0.545	0.91	0.034	1.5
	Meal	2	< 0.01 (< 0.01)		0.570 (0.558)		0.032 (0.033)	
	Hulls	1	< 0.01		0.182		0.014	
	Hulls	2	< 0.01	0.01	0.221	0.22	0.013	-
	Hulls	2	< 0.01 (< 0.01)		0.226 (0.224)		0.01 (0.012)*	
	Refined Oil	1	nd		nd		nd	
	Refined Oil	2	nd	< 0.01	nd	< 0.01	nd	< 0.01
	Refined Oil	2	nd (nd)		nd (nd)		nd (nd)	
C12MN081281 USA, (ND) 2008 (5A009RR) 0.618+0.621 interval 7 days, DALT=12	Soya bean (RAC)	1	< 0.01		0.600		0.027	
	Soya bean (RAC)	2	nd		0.615		0.032	
	Soya bean (RAC)	2	< 0.01		0.605		0.035	
	Soya bean (RAC)	2	nd (< 0.01)		0.590 (0.60)		0.033 (0.033)	
	AGF	1	< 0.01		0.342		0.030	
	AGF	2	0.026	2.4	0.132	-	0.214	33.84
	AGF	2	0.021		0.106		0.205	
	AGF	2	0.024 (0.024)*		0.113 (0.117)		0.224 (0.214)	

Pf: Processing factor

Treatment 1 Untreated control, one sample per trial

Treatment 2 Treated twice with 0.65 kg ai/ha at ca 7 day interval starting 28 days prior to harvest of mature seed

- not calculated due to a reduced amount in treated processed soya bean than in untreated processed soya bean, or not detected in treated or untreated processed soya beans.

AGF: Aspirated Grain Fraction

Rice

A study on the behaviour of difenoconazole during processing of rice was conducted by Yozgatli HP, and Breyer N (2010, S10-02953, No. A7402T_10217). Two field trials of rice were treated with two applications of difenoconazole with a target rate of 0.25 kg ai/ha. Samples of rice grain were collected at 21 and 28 days after the final application. Rice (grain) was processed into polished rice, parboiled rice, cooked rice and rice flour. Two mass-balance studies to determine the accountability of the residue and two follow-up studies were conducted to determine residue transfer on each process.

Field samples and processed fractions were analysed for parent difenoconazole using method REM 147.08 and LOQ was 0.01 mg/kg. The RAC (rice grain) and processed fractions were stored in the freezer ≤ 18 °C for a maximum of 17 months.

Cleaning and husking

Grain samples from the field were dried if required to achieve a moisture content of 12.1–14.2%. The rice was then cleaned using a sample cleaner. Shriveled (undeveloped and broken) grain was sorted out (< 1.9 mm). Samples of cleaned grain, shriveled grain and impurities were taken.

A portion of the cleaned grain was husked with a rubber husker. Samples of husks, brown rice and abrasion / broken grain were taken.

Polishing

Brown rice was processed into bran and polished rice. If the period between husking and polishing was more than 12 hours, an additional sample of brown rice was taken before polishing. The brown rice was then polished using a vertical shelling machine (abrasive decortication). Samples of bran / rub-off and polished rice were taken.

Parboiling

Samples of cleaned grain were taken before the parboiling process. The cleaned rice was steeped in water and heated to 76–85 °C. The steeped grain was stored in its closed container at room temperature and had a moisture content of 37.1–47.3 % at the end of the procedure (duration 3–4.4 h). Excess steeping water (which was not absorbed) was removed. A sample of the steeping water was taken.

The steeped grain was transferred to an autoclave and steamed at 104–115 °C for about 15 min. Samples of steamed grain and steaming water were taken before the steamed grain was transferred to the drying oven. The grain was dried for 16 h at temperatures between 36 °C and 88 °C until a final moisture content of 7.6–14.9 % was achieved. A sample of parboiled rice was taken.

The parboiled rice was husked using a rubber husker and samples of husks, parboiled brown rice and abrasion / broken grain were taken.

The husked parboiled brown rice was then polished using a vertical shelling machine. Samples of bran / rub-off and polished parboiled rice were taken.

Cooking

Samples of each type of rice were taken just before cooking.

Brown rice was cooked for 50–75 min in boiling water (97–100 °C) and a sample of cooked brown rice was taken. Brown parboiled rice was cooked for 65–85 min in boiling water (99–102 °C) and a sample of cooked parboiled brown rice was taken. Polished rice was cooked for 31–62 min in boiling water (98–100 °C) and a sample of cooked rice was taken. Polished parboiled rice was cooked for 48–68 min in boiling water (98–104 °C) and a sample of cooked parboiled rice was taken.

Milling flour

Samples of polished rice and polished parboiled rice were taken just before milling. Polished rice was milled using a cross beater mill and a sample of flour (polished rice) was taken. Similarly, polished parboiled rice was milled using a cross beater mill and a sample of flour (parboiled rice) was taken.

A summary flow chart of the overall processing scheme is given in Figure 1.

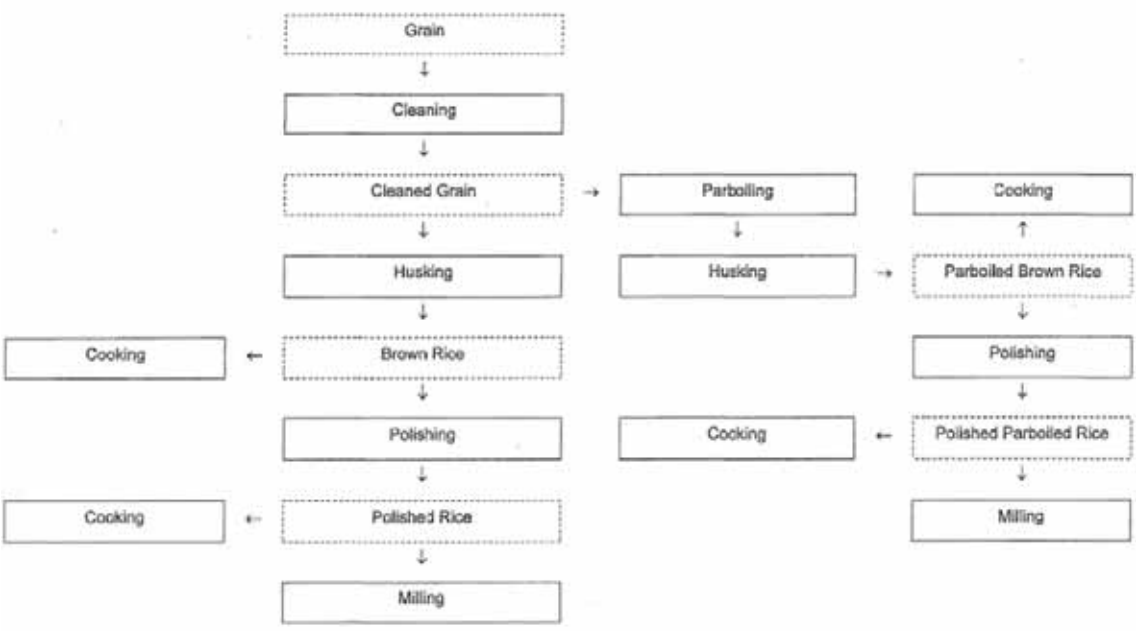


Figure 1 Processing Scheme for Rice grain

Study 1

Table 16a Residues from parent difenoconazole in rice grain (RAC and processed fractions)

Trial Location, year, (variety), dose rate, interval , DALT	Processed fraction	Difenoconazole parent mg/kg (mean in parenthesis)	processing factor
S10-02953-01 Italy, 2010 (Scudo) 258+256 g ai/ha interval 15 days, DALT = 24 Sandy clay loam	Rice grain, field (RAC)	3.0	-
	Mass balance trial (S10-02953-01-006)		
	Cleaning and husking		
	Grain, not cleaned	2.4	
	Cleaned grain	1.9, 2.5 (2.2)	1.09
	Impurities	7.6	3.45
	Shriveled grain	1.4	0.64
	Husks	8.4	3.50
	Abrasion/broken grain	4.0	1.82
	Brown rice	0.15	0.07
	Polishing		
	Brown rice	0.28	0.13
	Bran/rub rice	0.28	0.13
	Polished rice	0.041	0.02
		Parboiling	
	Cleaned grain	2.4	
	Steeping water	0.04	0.02
	Steamed grain	1.3	0.54
	Steaming water	< 0.01	0.004
	Parboiled rice	2.0	0.83
	Husks	5.4	2.25
	Abrasion/broken grain	2.2	0.92
	Parboiled brown rice	0.84	0.35

Trial Location, year, (variety), dose rate, interval , DALT	Processed fraction	Difenoconazole parent mg/kg (mean in parenthesis)	processing factor
	Bran/rub-off	3.3	1.38
	Polished parboiled rice	0.56	0.23
		Cooking	
	Cooked brown rice	0.12	0.05
	Cooked parboiled rice	0.51	0.21
	Cooked rice	0.023	0.01
	Cooked parboiled brown rice	0.22	0.09
		Milling	
	Flour (polished rice)	0.054	0.02
	Flour (parboiled rice)	0.44	0.18
	Follow-up-trial (S10-02953-01-007)		
	Cleaning and husking		
	Grain, not cleaned	2.9	-
	Cleaned grain	2.1, 1.9 (2.0)	0.69
	Husks	8.6	4.30
	Brown rice	0.14	0.07
	Polishing		
	Brown rice	0.10	0.05
	Bran/rub rice	0.25	0.13
	Polished rice	0.027	0.01
	Parboiling		
	Cleaned grain	1.9	0.66
	Parboiled rice	1.7	0.59
	Husks	5.1	1.76
	Parboiled brown rice	0.88	0.30
	Bran/rub-off	3.0	1.03
	Polished parboiled rice	0.49	0.17
	Cooking		
	Cooked brown rice	0.14	0.05
	Cooked parboiled rice	0.35	0.12
	Cooked rice	0.011	0.004
	Cooked parboiled brown rice	0.25	0.12
	Milling		
	Flour (polished rice)	0.039	0.01
	Flour (parboiled rice)	0.42	0.14

Study 2

Table 16b Residues from parent difenoconazole in rice grain (RAC and processed fractions)

Trial Location, year, (variety), dose rate, interval, DALT	Processed fraction	Difenoconazole parent (mg/kg)	processing factor
	Rice grain, field (RA	1.7	-
S10-02953-02 Italy, 2010 (Ercole)	Mass balance trial ((S10-02953-01-006)		
	Cleaning and husking		
	Grain, not cleaned	2.0	
	Cleaned grain	1.8, 1.2 (2.0)	1.0
251+252 g ai/ha interval 15 days, DALT = 21	Impurities	5.6	2.8
	Shriveled grain	1.8	0.9
	Husks	8.4	4.2
	Abrasion/broken grain	1.8	0.9
	Brown rice	0.077	0.04
Sandy clay loam	Polishing		
	Brown rice	0.09	0.05
	Bran/rub rice	0.28	0.14
	Polished rice	0.017	0.009
		Parboiling	
	Cleaned grain	1.4	0.7
	Steeping water	0.019	0.01

Trial Location, year, (variety), dose rate, interval, DALT	Processed fraction	Difenoconazole parent (mg/kg)	processing factor
	Steamed grain	0.88	0.63
	Steaming water	0.001	00007
	Parboiled rice	1.5	1.07
	Husks	5.2	3.71
	Abrasion/broken grain	2.1	1.5
	Parboiled brown rice	0.76	0.54
	Bran/rub-off	2.1	1.5
	Polished parboiled rice	0.35	0.25
	Cooking		
	Cooked brown rice	0.062	0.04
	Cooked parboiled rice	0.37	0.26
	Cooked rice	< 0.01	0.007
	Cooked parboiled brown rice	0.18	0.13
	Milling		
	Flour (polished rice)	0.016	0.01
	Flour (parboiled rice)	0.37	0.26
	Follow-up-trial (S10-02953-01-007)		
	Cleaning and husking		
	Grain, not cleaned	1.9	
	Cleaned grain	1.3, 1.3, (1.3)	0.68
	Husks	8.0	4.21
	Brown rice	0.074	0.04
	Polishing		
	Brown rice	0.099	0.05
	Bran/rub rice	0.38	0.2
	Polished rice	0.013	0.007
	Parboiling		
	Cleaned grain	1.5	0.79
	Parboiled rice	1.7	1.13
	Husks	4.7	3.13
	Parboiled brown rice	0.68	0.45
	Bran/rub-off	1.9	1.27
	Polished parboiled rice	0.37	0.25
	Cooking		
	Cooked brown rice	0.045	0.03
	Cooked parboiled rice	0.37	0.25
	Cooked rice	< 0.01	0.003
	Cooked parboiled brown rice	0.17	0.11
	Milling		
	Flour (polished rice)	0.013	0.009
	Flour (parboiled rice)	0.35	0.23

Table 17 Summary of parent difenoconazole residues in rice grain processed commodities from trials made in Italy

Processed fraction	Processing factors	Processing factors (mean)
Cleaned grain	1.09, 0.69, 1.0, 0.68	0.85
Husks	3.5, 4.3, 4.2, 4.21	4.05
Bran/rub-off	0.13, 0.13, 0.14, 0.2	0.15
Brown rice	0.07, 0.07, 0.04, 0.04	0.06
Parboiled rice	0.83, 0.59, 1.07, 1.13	0.91
Parboiled brown rice	0.35, 0.30, 0.54, 0.45	0.41
Polished rice	0.02, 0.01, 0.009, 0.007	0.01
Polished parboiled rice	0.23, 0.17, 0.25, 0.25	0.23

Processed fraction	Processing factors	Processing factors (mean)
Cooked brown rice	0.05, 0.05, 0.04, 0.03	0.04
Cooked parboiled rice	0.21, 0.12, 0.26, 0.25	0.21
Cooked rice	0.01, 0.004, 0.007, 0.003	0.006
Cooked parboiled brown rice	0.09, 0.12, 0.13, 0.11	0.11
Flour (polished rice)	0.02, 0.01, 0.01, 0.009	0.01
Flour (parboiled rice)	0.18, 0.14, 0.26, 0.23	0.20

Rape seed

Two studies on the behaviour of difenoconazole during processing of rape seed into meal and refined oil was conducted by Sagen K (2011, CER 05903/11). Field trials of oilseed were treated with one application of the target rate 0.375 kg ai/ha. Samples were harvested 30 days after the application. Rape seed was used for the production of meal and refined oil. Field samples and processed fractions (single samples) were analysed for parent difenoconazole using method REM 147.08. The LOQ was 0.01 mg/kg. The duration of storage for the processed fractions press-cake meal and refined oil were 3.2 months and 1.6 months, respectively.

- Whole oilseed rape seeds were flaked
- Flakes were pressed to separate the oil
- The extracted meal was air dried
- A sample of air dried meal was heat treated to duplicate toasting of rape seed meal
- The pressed and extracted oils were combined
- The crude solvent oil and the centrifuged press oil were blended, acid degummed, refined, washed with water and bleached
- The bleached oil was deodorized.

Figure 2 Processing scheme for rape seed

Table 18 Residues from parent difenoconazole in rape seed (RAC and processed fractions)

Trial Location, year, (variety), dose rate, interval , DALT	Processed Fraction	Difenoconazole parent, mg/kg	processing factors
Trial T948 Canada, (Elgin MB) 2011 (1841 RR) 0.367 kg ai/ha, DALT = 31	rape seed	0.033	
	meal	0.014	0.42
	oil	< 0.01	0.3
Trial T949 Canada, (Rosthern SK) 2011 (72-55 RR) 0.390 kg ai/ha, DALT = 31	rape seed	0.18	
	meal	0.12	0.67
	oil	< 0.01	0.06

Table 19 Summary of calculated processing factors in soya bean, rice and oilseed rape from difenoconazole treated raw commodities

RAC	Processed fraction	Calculated processing factors				PF best estimate
		Difenoconazole	1,2,4 Triazole*	Triazole alanine**	Triazole lactic acid***	
soya bean	Meal	0.63, 0.01	< 0.01, 0.01,	0.45, 0.91	0.01, 1.5	
	Hulls	3.38, 0.61	< 0.01, 0.01,	0.31, 0.22	0.01, -	
	Oil (refined)	1.06, 0.42	< 0.01, < 0.01,	< 0.01, < 0.01,	0.01, < 0.01	
	AGF	622	2.4	-	33.8	
rice	Husks	3.5, 4.3, 4.2, 4.21	nm	nm	nm	
	Bran/rub-off	0.13, 0.13, 0.14, 0.2	nm	nm	nm	
	Brown rice	0.07, 0.07, 0.04, 0.04	nm	nm	nm	
	Parboiled rice	0.83, 0.59, 1.07, 1.13	nm	nm	nm	
	Parboiled brown rice	0.35, 0.30, 0.54, 0.45	nm	nm	nm	
	Polished rice	0.02, 0.01, 0.009, 0.007	nm	nm	nm	
	Polished parboiled rice	0.23, 0.17, 0.25, 0.25	nm	nm	nm	
	Cooked brown rice	0.05, 0.05, 0.04, 0.03	nm	nm	nm	
	Cooked parboiled rice	0.21, 0.12, 0.26, 0.25	nm	nm	nm	
	Cooked rice	0.01, 0.004, 0.007, 0.003	nm	nm	nm	
	Cooked parboiled brown rice	0.09, 0.12, 0.13, 0.11	nm	nm	nm	
	Flour (polished rice)	0.02, 0.01, 0.01, 0.009	nm	nm	nm	
	Flour (parboiled rice)	0.18, 0.14, 0.26, 0.23	nm	nm	nm	
Oilseed rape	meal	0.42, 0.67	nm	nm	nm	
	refined oil	0.3, 0.06	nm	nm	nm	

- not calculated due to less occurrence in treated processed soya bean than in untreated processed soya beans.

nm: not measured

APPRAISAL

Difenoconazole is a systemic triazole fungicide and acts by inhibition of demethylation during ergosterol synthesis. It is applied by foliar spray or seed treatment and controls a broad spectrum of foliar, seed and soil-borne diseases caused by Ascomycetes, Basidiomycetes and Deuteromycetes, on a variety of crops. Difenoconazole was evaluated for the first time by JMPR 2007. The 2007 Meeting established an acceptable daily intake (ADI) of 0–0.01 mg/kg bw and an acute reference dose (ARfD) of 0.3 mg/kg bw. Maximum residue levels for a number of commodities were recommended by JMPR in 2007, 2010 and 2013.

Definition of residues for plant products (compliance with MRLs and dietary intake assessment): *difenoconazole*.

Definition of residues for animal products: sum of difenoconazole and CGA 205375 (1-[2-chloro-4-(4-chloro-phenoxy)-phenyl]-2-(1, 2, 4-triazol)-1-yl-ethanol), expressed as difenoconazole.

Difenoconazole was listed by the Forty-sixth Session of CCPR (2014) for the review of additional maximum residue levels. GAP information with supporting residue studies in strawberries, avocadoes, soya beans, cotton, peanuts, rice and oilseed rape (canola) was evaluated by the present Meeting.

Methods of analysis

The analytical method used for determination of difenoconazole residues in samples derived from supervised field trials and processing studies in strawberries, soya beans, rice and oilseed was evaluated by previous Meetings.

Two new pre-registration methods for plant matrices were presented to the 2015 Meeting. In these methods difenoconazole is extracted by high-speed homogenisation with an acetone/water mixture (2:1). After clean-up the residues were determined by (HPLC-MS/MS).The method has a validated LOQ of 0.01 mg/kg for difenoconazole in avocadoes, cotton, oilseed rape including processed commodities, peanuts, rice, soya beans and strawberries. The methods were used for determination of difenoconazole residues in samples from supervised field trials on cotton and peanuts presented to the current Meeting.

Stability of pesticide residues in stored analytical samples

The stability of residues from difenoconazole in stored samples was evaluated by the 2007 Meeting. The periods of demonstrated stability cover the frozen storage intervals used in the residue trials for which maximum residue levels were estimated.

Results of supervised residue trials on crops

The Meeting received new supervised trial data for foliar application of difenoconazole (EC or SC formulations) on strawberries, avocadoes, soya beans, rice, cotton, peanuts and oilseed rape, and noted that residue data from rice, soya beans and oilseed rape also were provided to the 2007 JMPR.

The results from new trials and those previously reported by the 2007 JMPR which either matched the critical GAP, or when results could be proportionally adjusted to reflect GAP application rates, were considered in estimating maximum residue levels, STMRs and HRs for the commodities for which GAP information was available. The proportionality approach was considered to scale the results from trials where the application rates range from 0.3× GAP to 4× GAP and where all other parameters matched the critical GAP.

Strawberry

Data from supervised trials on strawberries from USA conducted in 2008 and 2009 were presented to the Meeting. The critical GAP in USA is maximum foliar applications up to 0.129 kg/ha, an application interval of 7–14 days and a PHI of 0 days. The maximum application rate for difenoconazole is 0.515 kg ai/ha per crop and season.

Strawberries belong to the high acid category and storage data covering this category was not evaluated by 2007 JMPR and not included in the residue trials. As difenoconazole has a pKa of 1.1 an estimation of maximum residue levels was not made.

Avocado

Four independent supervised trials from Brazil conducted in 2007 and 2008 were presented to the Meeting. The critical GAP in Brazil is four foliar applications of 0.05 kg ai/ha at BBCH 62–79 (starting at flowering until fruit is around 5 cm) and with intervals of 14 days. The PHI is 14 days.

The trials from Brazil (4× 0.05 kg ai/ha at BBCH 71–79, interval 14 days, PHI 14 days) matched the critical GAP. Residues of difenoconazole in avocado fruits 14 days after the last application were (n=4) 0.02, 0.05 (2) and 0.26 mg/kg. The highest residue of 0.26 mg/kg was measured in an individual fruit sample.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in avocado of 0.6 mg/kg, 0.05 mg/kg and 0.26 mg/kg, respectively.

Soya bean (dry)

Twenty one supervised trials from USA conducted in 2008 were presented to the Meeting. The critical GAP in USA is two foliar applications of 0.129 kg ai/ha, with an interval of seven days and a PHI of 14 days.

Six trials from Brazil (2× 0.075 kg ai/ha and a PHI of 30 days) presented to the 2007 JMPR did not match the critical GAP.

Eighteen independent trials from USA (2× 0.129 kg ai/ha, interval 7–10 days, PHI 14 days) matched the critical GAP. Residues of difenoconazole in soya beans were (n=18) < 0.01(12), 0.012, 0.013, 0.019, 0.021, 0.04 and 0.087 mg/kg. The highest residue of 0.15 mg/kg was measured in individual seed samples.

The Meeting estimated a maximum residue level and STMR value for difenoconazole in soya bean seeds of 0.1 mg/kg and 0.01 mg/kg, respectively. The Meeting withdraws its previous recommendation of 0.02* mg/kg for maximum residue level for soya beans (dry).

Rice

Eight supervised trials from Europe (Italy) conducted in 2009 and 2010 were presented to the current Meeting. A registered label was not available to the Meeting and an estimation of a maximum residue level was not made.

Cotton

Eight independent supervised trials from Brazil conducted in 2006–2008 were presented to the Meeting. The critical GAP in Brazil is three foliar applications of 0.075 kg ai/ha, an interval of 10–15 days and a PHI of 21 days.

Four trials (5× 0.075 kg ai/ha, BBCH 13–81, interval 21 days, PHI 30 days) were not according to GAP. Samples were only taken 30 days after last application, and the applications were two more than specified in the critical GAP.

Four trials were made with four applications of 0.075 kg ai/ha starting from BBCH 71 up to BBCH 83 and a PHI of 21 days. These trials matched the critical GAP from Brazil. Residues

of difenoconazole in cotton were (n=4) < 0.01, 0.01 and 0.02 (2) mg/kg. An estimation of maximum residue levels was not made as four trials were considered insufficient.

Oilseeds

Peanut

Eight independent supervised trials from Brazil conducted in 2008–2010 were presented to the Meeting. The critical GAP in Brazil is three applications of 0.0875 kg ai/ha and a PHI of 22 day.

Four of the trials (3× 0.088 kg ai/ha, PHI 22 days) were according to the critical GAP and residues of parent difenoconazole were not detected. Another four trials (6× 0.125 kg ai/ha) were conducted as residue decline trials and residues of parent difenoconazole was not found.

As residues of difenoconazole not was detected at an exaggerated number of applications and application rates, the Meeting concluded a zero residue situation occurs after application of difenoconazole to peanuts in accordance with the Brazilian critical GAP.

Residues of difenoconazole in peanuts from eight independent trials matching GAP were (n=8) < 0.01 mg/kg.

The Meeting estimated a maximum residue level and STMR values for difenoconazole in peanut kernels of 0.01* mg/kg and 0 mg/kg, respectively.

Rape seed (canola)

Data from supervised trials on rape seed (canola) from Canada conducted in 2011 were presented to the Meeting. The critical GAP in Canada is one foliar application of 0.125 kg ai/ha and a PHI of 30 days.

Nine independent trials from Canada matching the critical GAP were available to the Meeting. Residues from difenoconazole in rape seed were (n=9) < 0.01, 0.011 (1), 0.015 (2), 0.033 (2), 0.038, 0.062 and 0.063 mg/kg.

The Meeting estimates a maximum residue level, and STMR value for difenoconazole in oilseed rape (rape seed) of 0.15 mg/kg and 0.03 mg/kg, respectively. The Meeting replaces its previous recommendation of 0.05 mg/kg for the maximum residue level for rape seed.

Animal feeds

Rape seed (canola), forage, fodder

Residue data for rape seed forage was not presented to the Meeting.

Soya bean

The Meeting noted that the GAP for difenoconazole in USA does not permit soya bean hay, forage or silage as animal feeds.

Rice whole crop (silage), and straw

Eight supervised trials from Europe (Italy) conducted in 2009 and 2010 were presented to the Meeting. Forage and straw samples were collected. A registered GAP was not available for rice. An estimation of maximum residues levels was not made.

Fate of residues during processing

The 2007 JMPR reported that difenoconazole was essentially stable during the hydrolysis conditions simulating food processing conditions and also estimated processing factors for a range of commodities. Relevant processing factors for difenoconazole and STMR-Ps for the commodities

considered at this Meeting and used for dietary intake and risk assessment or for estimating livestock animal burden are summarized below.

Raw agricultural commodity	Processed commodity	Processing factors [a] (mean)	RAC (mg/kg)	STMR-P
			STMR	mg/kg
Soya bean	RAC		0.01	
	Meal	0.38		0.004
	Hulls	2		0.02
	Oil (refined)	0.8		0.08
	AGF [b]	622		6.22
Rape seed (canola)	RAC		0.03	
	Meal	0.55		0.016
	Refined oil	0.05		0.002

[a] The processing factor is the ratio of the total residue in the processed item divided by the total residue in the RAC

[b] Aspirated grain fraction

The Meeting noted that in the studies available difenoconazole residues did not concentrate in food commodities during processing. In feed commodities however residues increased in soya bean hulls and soya bean aspirated grain fractions (AGF).

Residues in animal commodities

Estimated dietary burdens of farm animals

The dietary burdens for beef cattle and dairy cattle were calculated using the OECD diets listed in Appendix IX of the 2009 edition of the FAO Manual. Potential feed items included: almond hulls, cabbage heads and leaves, bean vines, carrot hulls, canola meal, grape pomace, pea vines, potato culls, potato process waste, soya beans, soya bean aspirated grain fraction, sunflower meal, and wheat grain and hay.

The estimated the dietary burden for cattle and poultry and were not significantly different from the dietary burdens estimated by the 2013 JMPR. The only additional feed item included was soya bean.

The Meeting confirmed the previous recommendations for animal commodities.

RECOMMENDATIONS

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

Definition of residue for plant products (compliance with MRLs and dietary intake assessment): *difenoconazole.*

Definition of residue for animal products (compliance with MRLs and dietary intake assessment): sum of difenoconazole and CGA 205375 (1-[2-chloro-4-(4-chloro-phenoxy)-phenyl]-2-(1, 2,4-triazol)-1-yl-ethanol), expressed as difenoconazole.

The residue is fat soluble (2007 JMPR Meeting).

CCN	**Commodity**	**Recommended Maximum residue level (mg/kg)**		**STMR or STMR-P mg/kg**	**HR or HR-P mg/kg**
		New	**Previous**		
FI 0326	Avocado	0.6		0.05	0.26
SO 0697	Peanut	0.1 *		0	
SO 0495	Rape seed	0.15	0.05	0.03	
VD 0541	Soya bean (dry)	0.1	0.02 *	0.01	

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
OR 0541	Soya bean oil, refined			0.08	
OR 0495	Rape seed oil, edible			0.002	
AB 0541	Soya bean hulls			0.02	
AB 1265	Soya bean meal			0.004	
	Soya bean asp gr fn[a]			6.22	

[a] aspirated grain fraction

DIETARY RISK ASSESSMENT

Long-term intake

The IEDI of difenoconazole based on the STMRs estimated by this and previous Meetings for the 17 GEMS/Food regional diets were 7–70% of the maximum ADI of 0.01 mg/kg bw (see Annex 3 of the 2015 Report). The Meeting concluded that the long-term dietary intake of residues of difenoconazole is unlikely to present a public health concern.

Short-term intake

The ARfD for difenoconazole is 0.3 mg/kg bw. The International Estimated Short-Term (IESTI) of difenoconazole for the commodities for which STMR, HR and maximum residue levels were estimated by the current Meeting are shown in Annex 4 to the 2015 Report. The IESTI represented a maximum of 3% of the ARfD. The Meeting concluded that the short-term intake of difenoconazole residues from uses considered by the current Meeting was unlikely to present a public health concern.

REFERECENCE

File number	Author	Year	Title, Institute, Report reference
No A13703G_10284	Casallanovo F., Maslowski K.	2008	Amistar Top - Residues of Azoxystrobin, R2303'0 and Difenoconazole in Avocados - Brazil 2007/08 Syngenta Crop Protection AG, Basel, CH, M08071 GLP, not published
No A13703G_10323	Casallanovo F., de Gois F.	2006	Priori Top - Residues of Azoxystrobin, R230310 and Difenoconazole in cotton - Brazil, 2005-06 Syngenta Crop Protection AG, Basel, CH, M05022 GLP, not published
No A15265A_10006	Casallanovo F., Volpi R., Suzuki L.	2008	Cypress - Residues of Cyproconazole in cotton - Brazil, 2007-08 Syngenta Crop Protection AG, Basel, CH, M08065 GLP, not published
No A16976A_10030	Draetta M	2014	A16976A - Residue Magnitude of Difenoconazole and Chlorothalonil in Peanuts - Brazil, 2009-10 Syngenta Crop Protection AG, Basel, CH, M10070 GLP, not published
EFSA Journal 2014; 12 (10): 3882	EFSA	2014	Reasoned opinion on the modification of the existing MRLs for difenoconazole in lettuce and other salad plants including Brassicae and in basil (mint)

File number	Author	Year	Title, Institute, Report reference
Efsa Journal 2011; 9 (1): 1967	EFSA	2011	Conclusion on the peer review of the pesticide risk assessment of the active substance difenoconazole
No CGA169374_50035	Hamilton L.	2009	Difenoconazole - Magnitude of the Residues in or on Strawberries Syngenta Crop Protection AG, Basel, CH, Morse Laboratories, Inc., Sacramento, USA, T002401-07 GLP, not published
No A7402N_10001	Marconi F., Volpi R	2008	Score - Residues of Difenoconazole in peanut - Brazil 2007-08 Syngenta Crop Protection AG, Basel, CH, M08013 GLP, not published
No A15457B_50038	Sagan K.	2012	SYN545192 EC (A15457B), Difenoconazole EC (A7402T), Propiconazole EC (A6097AC) and Propiconazole/Azoxystrobin SU (A13705V) Residue Levels on canola Seed and Processed Fractions (Meal and Refined Oil) in Canada during 2011 Syngenta Crop Protection AG, Basel, CH, CER 05903/11, 12SYN312.REP GLP, not published
No A7402T_10144	Willard T., Mayer T.	2009	Difenoconazole - Magnitude of residues in or on soybean Syngenta Crop Protection AG, Basel, CH, Morse Laboratories, LLC, Sacramento, USA, T002400-07 GLP, not published
No A7402T_10138	Yozgatli H., Amann S.	2011	Difenoconazole - Residue Study on Rice in Italy in 2009 Syngenta Crop Protection AG, Basel, CH, Eurofins Agroscience Services DE, S09-01473 GLP, not published
No A7402T_10217	Yozgatli H., Breyer N	2013	Difenoconazole - Residue Study on Rice and Processed Specimens in Italy in 2010 Syngenta Crop Protection AG, Basel, CH, Eurofins Agroscience Services DE, S10-02953 GLP, not published
No A7402T_10139	Yozgatli H.	2011	Azoxystrobin, Difenoconazole - Residue Study on Rice in Italy in 2010 Syngenta Crop Protection AG, Basel, CH, Eurofins Agroscience Services DE, S10-00372 GLP, not published
No A13703G_10496	Yozgatli H., Amann S.	2011	Azoxystrobin, Difenoconazole - Residue Study on Rice in Italy in 2010 Syngenta Crop Protection AG, Basel, CH, Eurofins Agroscience Services DE, S10-00372 GLP, not published

ETHEPHON (106)

First draft prepared by Dr Yukiko Yamada, Ministry of Agriculture, Forestry and Fisheries, Tokyo, Japan

EXPLANATION

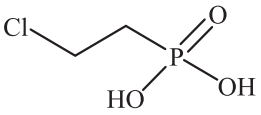
Ethephon, 2-chloroethylphosphonic acid, is a systemic plant growth regulator belonging to the phosphonate family. It is readily absorbed by the plant and releases ethylene, a natural plant hormone. Ethylene not only influences directly several physiological processes, such as ripening and maturation, but also stimulates the endogenous ethylene production. It has been registered in many countries for a variety of crops, including fruits, vegetables, cereals and oilseed crops.

Ethephon was first evaluated by JMPR in 1977 as a new compound, and has been reviewed for residues in 1978, 1983, 1985, 1994 (Periodic Review) and 1994. Currently there are 26 Codex MRLs for ethephon. It was listed in the Priority List by the 46th Session of CCPR in 2014 for toxicological and residue evaluation by the current Meeting in the CCPR Periodic Review Programme.

The Meeting received information on identity, metabolism and environmental fate, residue analysis, use patterns, supervised trials (on apples, cherries, grapes, figs, olives, pineapples, tomatoes, cereals, and cotton), processing, and animal feeding studies.

Matrix	Fortification, mg/kg	n	Range of recoveries, %	Mean recovery, %	CV, %	Ref. method
Blueberry	0.05	3	90–100	96	5.3	
	0.5	4	84–94	89	5.2	

IDENTITY

ISO common name:	Ethephon
Chemical name	
IUPAC:	2-Chloroethylphosphonic acid
CAS:	(2-Chloroethyl)phosphonic acid
CAS Registry No.:	16672-87-0
CIPAC No.:	373
Structural formula:	
Molecular formula:	C2H6ClO3P
Molecular weight:	144.5

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

Property	Results	Reference
Appearance	White crystalline powder (98.5%)	Mühlberger, 2001 (PA01/031) [M-207237-01-1]
Odour	No characteristic odour (98.5%)	Mühlberger, 2001 (PA01/031) [M-207237-01-1]
Melting point	73.3 °C (98.5%)	Smeykal, 2001 (20010301.01) [M-203841-01-1]
Boiling point	Decomposes at 250–400 °C (under	Smeykal, 2001 (20010301.01)

Property	Results	Reference
	nitrogen) (98.5%)	[M-203841-01-1]
Relative density	1.65 kg/m^3 at 20 °C (98.5%)	Schneider, 2001 (B 031/2001) [M-204865-01-1)]
Vapour pressure	< 1.0×10^{-3} Pa (from 18 to 80 °C) (98.5%)	Smeykal, 2001 (20010301.02) [M-203843-01-1]
Volatility (Henry's law constant)	< 1.45×10^{-7} Pa m^3 mol^{-1}	Bascou, 2002 (C019663) [M-208014-01-1]
Solubility in water	At 21–24 °C pH < 0.2: > 1000 g/L pH 4: 800 g/L pH > 5: decomposition and no solubility could be determined (98.5% and 98.0%)	Mühlberger, 2002 (PA01/018) [M-206704-01-1]
Solubility in organic solvents	Solubility at 20 °C n-Heptane: < 0.3 mg/L p-Xylene: 82.5 mg/L 1,2-Dichloroethane: 832 mg/L Methanol: > 600 g/L Acetone: > 600 g/L Ethyl acetate: > 600 g/L Acetonitrile: > 600 g/L Dimethylsulfoxide: > 600 g/L (98.5%)	Mühlberger, 2001 (PA01/019) [M-204740-01-1]
Partition coefficient	Log P_{ow} at room temperature: pH 2: –0.63 pH 7: –1.89 pH 10: –1.81 (98.5%)	Mühlberger, 2002 (PA01/020) [M-206706-01-1]
Hydrolysis	DT_{50} values at 25 °C: pH 5: 73.5 days pH 7: 2.4 days pH 9: 1.0 day (linear-regression)	Das, 1990 (ISSI 89150) [M-187629-01-1]
Photochemical degradation	Rate constant k at 25 °C and pH 5 from linear regression: k_2 under irradiated conditions, 9.39 10^{-04} h^{-1} (DT_{50} 61 days of 12 hours irradiation/day); k_1 under non-irradiated conditions, 5.22 10^{-04} h^{-1} (DT_{50} 111 days of 12 hours darkness/day). Net rate constant k_3 due to irradiation alone, $k_3 = k_2 - k_1 = 4.17E^{-04}$ h^{-1} (Net DT_{50} 139 days of 12 hours irradiation/day). Degradation product: ethylene (max. 15.3% and 23.1% in non-irradiated and irradiated samples, respectively).	Das, 1990 (ISSI 89151) [M-187632-01-1]
Dissociation constant	At 21 °C $pK_1 = 2.82$ $pK_2 = 7.21$ (98.5%)	Mühlberger, 2002 (PA01/017) [M-206703-01-1]

Technical material

Property	Results	Reference
Active ingredient	Not less than 910 g/kg	FAO Specification 373/TC/S/F (1997) Ethephon technical
Impurities	MEPHA (Mono 2-chloroethyl ester, 2-chloroethyl phosphonic acid): maximum 20 g/kg 1,2-Dichloroethane: maximum 0.5 g/kg	FAO Specification 373/TC/S/F (1997) Ethephon technical
Appearance	Greyish-white coloured, waxy solid without extraneous matter	FAO Specification 373/TC/S/F (1997) Ethephon technical
pH	1.5 to 2.0	FAO Specification 373/TC/S/F (1997) Ethephon technical

Technical concentrate

Property	Results	Reference
Impurities	MEPHA: maximum 2% of declared ethephon content 1,2-Dichloroethane: maximum 0.05% of the declared ethephon content Material insoluble in water: The product shall pass through a 250 µm test sieve and not more than 1 g/kg shall remain on a 150 µm test sieve. Water: shall not be less than the following figure: {1000 – (measured ethephon content in g/kg)/0.91} – 15	FAO Specification 373/TK/S/F (2000) Ethephon technical concentrate
pH	1.5 to 2.0	FAO Specification 373/TK/S/F (2000) Ethephon technical concentrate
Appearance	Viscous colourless liquid (71.5%)	Bascou, 2001 (R&D/CRLD/AN/0015211) [M-184641-01-1]
Odour	No characteristic odour (71.5%)	Bascou, 2001 (R&D/CRLD/AN/0015211) [M-184641-01-1]
Flammability	No flash point up to 111 °C (boiling temp.) (71.4/70.2%)	Francois, 1999 (99-308-SEC) [M-179319-01-1]
Auto-flammability	Self-ignition temperature: 490 °C (70.2%)	Francois, 1999 (99-308-SEC) [M-179319-01-1]
Explosive properties	Not explosive (70.2%)	Francois, 1999 (99-308-SEC) [M-179319-01-1]

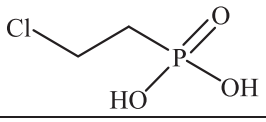
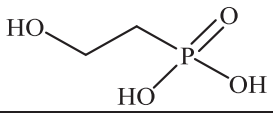
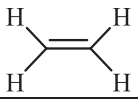
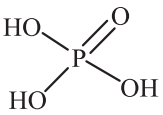
Formulations

Ethephon is mainly formulated as a soluble concentrate (SL). Concentrations are between 120 and 730 g/L. Combinations with chlormequat chloride or cyclanilide are also available for specific uses. Formulations are applied as foliar sprays by either ground or aerial equipment. Available formulations are listed below:

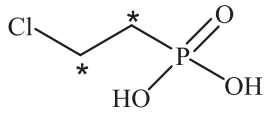
- Soluble liquid (SL) formulations containing either 120 g ai/L, 240 g ai/L, 250 g ai/L, 480 g ai/L, 660 g ai/L or 720 g ai/L
- Soluble liquid (SL) formulations containing a mixture of ethephon + chlormequat-chloride (150 g ai/L + 300 g ai/L or 180 g ai/L + 360 g ai/L ethephon + chlormequat-chloride, respectively)
- Suspension concentrate (SC) formulations containing a mixture of ethephon + cyclanilide (480 g ai/L + 60 g ai/L or 720 g ai/L + 45 g ai/L or 731 g ai/L + 49.5 g ai/L ethephon + cyclanilide, respectively)

METABOLISM AND ENVIRONMENTAL FATE

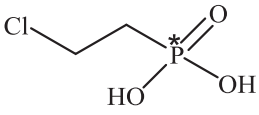
The following links code numbers and structure or description of the compounds appearing in the various metabolism and environmental fate studies.

Name or Code (MW)	IUPAC Name	Structure	Found in:
Ethephon (144.5) Syn: V-1283, S-1283, YI-5301, SCAL-5001	2-Chloroethylphophonic acid		Plants, Animals, Soils
HEPA (126.05)	(2-Hydroxyethyl)-phosphonic acid		Plants, Animals, Soils
Ethylene (28.05)	Ethylene		Plants, Animals
Phosphoric acid (94.97) or Phosphate anion	Phosphoric acid		Plants, Animals

The Meeting received information on plant and animal metabolism for ethephon, its environmental fate in soil and residues in rotational crops. The fate and behaviour of ethephon in plants, animals and soil were investigated using the radio-labelled ethephon with ^{14}C as shown in Figure 1. The radio-labelled ethephon with ^{32}P was also used in the metabolism study in pineapple.



1,2-^{14}C-ethephon ([U-^{14}C]-ethephon, ^{14}C-ethephon)



^{32}P-ethephon

Figure 1 Radio-labelled test materials used in the metabolism and environmental fate studies

In the metabolism and environmental studies, the total radioactive residues were expressed in ethephon equivalents unless otherwise stated.

Plant Metabolism

The Meeting received information on metabolism of ethephon in various plants (mostly fruit and seed crops) in support of supervised trials: pineapple, melon (cantaloupe), tomato, wheat, hazelnut and cotton. Information was also available from the published scientific literature on apple, peach, cherry, grape, squash and cucumber.

Pineapple

The metabolism of ethephon was studied in pineapple using [^{32}P]ethephon and [^{14}C]ethephon (Anonymous, 1968, ETH/M21, [M-188023-01-1]). The technical material used in the study was a mixture of 70% ethephon and 30% monochloroethyl ester. However, the monochloroethyl ester was later removed from all formulations intended for crop use and therefore its metabolism is not relevant for the current uses of ethephon.

In the first experiment, pineapple plants grown in the field were treated with an application to individual leaves of 300 mg of a formulation mixture containing [^{32}P]ethephon and its monochloroethyl ester approximately 5 months before harvesting of fruit. A separate group of pineapple plants was treated with 300 mg of a formulation mixture containing ^{32}P-sodium acid phosphate to investigate the uptake and distribution of phosphate, ethylene and chloride (all expected metabolites of ethephon) under the pH conditions normally found in plant tissues. Plants that were harvested with a longer PHI received a larger amount of ^{32}P-labelled compound due to the short half-life of ^{32}P (14.2 days).

One to 118 days after treatment, the above-ground portions were harvested. Samples were rinsed with water, homogenized and extracted with benzene and then methanol. The post-extraction solids were analysed for radioactivity by combustion. Liquid extracts were analysed by liquid scintillation counting (LSC).

On the day of application and three days after application (DAT), most of the radioactivity was recovered in the water wash. No radioactivity was found in the benzene extract or in the post-extraction solids. More than three days after treatment, little or no radioactivity was recovered in the water wash. On 118 DAT, approximately 40% of the radioactivity remained in the post-extraction solids, and was almost same for plants treated with [^{32}P]ethephon and with [^{32}P]phosphate.

TLC analysis of the water washes and methanol extracts showed complete degradation of ethephon in/on pineapple leaves long before formation of the fruits. No ethephon was found in immature fruits, or in fruits of leaves harvested 1 month before full maturity of the fruits.

In the second experiment, a pineapple leaf was spotted with a solution of [^{14}C]ethephon in methanol, and air-dried. Then the treated area was excised and sliced. The leaf slices were inserted into a sealed two-necked flask. A continuous stream of nitrogen was passed over the slices and led to an absorber tower containing a solution of 0.25 M mercuric perchlorate in perchloric acid to absorb [^{14}C]ethylene. The amount of [^{14}C]ethylene absorbed was determined by LSC for 8 consecutive days, after which time the leaf slices were freeze-dried, and the remaining radioactivity was determined by combustion.

Over the 8-day duration, 40.1% of the applied [^{14}C]ethephon was metabolized to [^{14}C]ethylene, and 36.3% of the applied radioactivity remained in the leaf. The low recovery is attributed to losses during freeze-drying.

In an additional static experiment, a treated pineapple leaf slice was cut into strips and placed in the centre annular ring of a Conway micro diffusion dish. A 0.5 mL aliquot of absorber solution was placed in the inner compartment and the apparatus sealed and left for 72 hours. The absorber solution was analysed by LSC. The leaf strips were extracted with methanol, the extract was diluted with water and then extracted with benzene. The methanol and benzene extracts were analysed by TLC, and the post-extraction solids were analysed by combustion. A portion of the methanol extract was treated with 5 N NaOH to convert the [^{14}C]ethephon to [^{14}C]ethylene. The resulting [^{14}C]ethylene was trapped in the perchlorate absorber and analysed by LSC.

After 72 hours, 25.2% of the applied [^{14}C]ethephon was converted to [^{14}C]ethylene. Of the radioactivity remaining in the leaf, 63.3% of the applied radioactivity (AR) was extracted with methanol, of which 40.1% AR reacted with NaOH to form [^{14}C]ethylene and was therefore characterized as [^{14}C]ethephon. TLC analysis of the methanol extract showed that parent ethephon was the only component of the residue, (Table 1).

Table 1 Recovery of ^{14}C- residues from an excised pineapple leaf slices following application of [^{14}C]ethephon (static experiment)

Fraction	% of Applied Radioactivity
[^{14}C]-ethylene	25.2
Methanol extract	63.3
Radioactivity evolved after treatment of methanol extract with NaOH (presumed to be [^{14}C]ethylene)	40.1
Benzene extract of methanol extract	< 0.1
Post-extraction solids	9.2

In the third experiment, nine pineapple plants were treated shortly (7, 14 or 21 days) before harvest of mature fruit with a spray application of [^{14}C]ethephon at 9 kg ai/ha, and transferred to uncoated cellophane chambers. Cellophane is impervious to ethylene but permeable to air and water vapour. In three of the boxes, glass tubing was inserted and connected to absorber towers filled with mercuric perchlorate-perchloric acid solution to absorb the ethylene evolved. Using a vacuum pump, air was passed through the chamber into the absorber towers at a rate of 1 air change/hour. The absorber solution was changed after 18, 46, 94, 118, 166 and 202 hours, and the radioactivity was determined by LSC. Plants were harvested after 1 hour to 21 days, and sectioned into fruit, top leaves, lower leaves and stump. The fruits were further sub-divided into crown, shell and bottom leaflets ('shell'), shell scrapings, fruit cylinder and core. Samples were frozen in dry ice and ground to a fine powder. The total radioactive residue in each fraction was determined by combustion analysis. Aliquots of each fraction were extracted with benzene and methanol, and the extracts were analysed by LSC. The radioactivity remaining unextracted was determined by combustion.

Very little or no radioactivity was found in the benzene extracts, and therefore these were not analysed further. Selected methanol extracts were analysed for ethephon by TLC. [^{14}C]Ethylene was evolved at an approximately constant rate from the treated plants. Little radioactivity was translocated into the pineapple flesh. TLC analysis showed that the bulk of the radioactivity remained in/on the plants and was found to comprise almost entirely unchanged [^{14}C]ethephon. An additional unidentified minor component of the ^{14}C-residue in pineapple shell and shell scrapings was also found in some stored standard solutions and was therefore postulated to be an impurity in the starting material rather than a metabolite. The distribution of residues in the pineapple fractions is shown in Table 2.

Table 2 Distribution of ^{14}C- residues in pineapple fractions

Application timing (days before normal harvest): Time after treatment	% of Total Radioactivity							
	Top third leaves	Shell	Shell scrapings	Stump	Cylinder	Core	Crown	Lower leaves
21: 45 hours	7.6	16.4	5.9	2.1	0.5	0.1	16.9	50.4
21: 6 days	20.5	23.9	3.7	1.4	0.9	0.1	9.8	39.3
21: 1 hour	50.6	33.6	9.2	2.3	3.4	0.8		
21: 6 hours	66.8	19.6	8.2	4.1	1.0	0.2		
21: 21 hours	42.7	42.1	11.2	2.1	0.2	< 0.1		
21: 45 hours	20.6	50.2	18.1	6.4	1.6	0.2	Not collected	
21: 3 days	41.5	39.8	14.7	1.4	1.7	0.4		
21: 6 days	40.6	47.3	7.3	2.7	1.8	0.2		
21: 9 days	38.7	49.0	9.2	1.4	1.3	0.2		
14: 9 days	26.7	47.0	21.9	2.1	2.1	0.3		
7: 7 days (fully mature)	78.6	8.6	11.8	0.8	0.3	< 0.1		

Melon (Cantaloupe)

Melon plants grown under field conditions were treated with a foliar spray of an SL formulation followed by a localised application of [^{14}C]ethephon to the leaves proximal or distal to the peduncle

(fruit stalk), or directly to the melon rind covering about 40% of the surface area (Palmer, Lewis, Johnson and Smith, 1970, ETH/20, [M-188017-01-1]). The fruits were protected after treatment using a cheesecloth bag and were harvested after 3 days. Surface residues were removed by washing the treated leaves or melon rind with 20% aqueous methanol followed by two water washes. Each melon was separated into rind, flesh and seeds, the samples were cut into thin ribbons and then frozen. The remaining vines were collected and frozen. Samples were freeze-dried and ground into a fine powder, and then extracted with either benzene plus methanol, water and methanol/chloroform (2:1), or water and chloroform.

The methanol extracts from benzene and methanol were combined, acidified and concentrated by rotary evaporation. The concentrated extract was acidified, made up to volume with methanol, and ethyl ether added to precipitate the co-extracted plant material. The combined methanol/water extracts were concentrated by rotary evaporation, acidified, and ethyl ether added to precipitate the ether insoluble residue. This extraction scheme resulted in more complete extraction of radioactivity.

Radioactivity in the methanol, or methanol/water extracts was determined by LSC. Radioactivity in non-aqueous solvents and insoluble plant residues was determined by low beta gas flow counting. Metabolite profiling was performed by radio-TLC using cellulose or silica plates.

Surface washing removed 37.2–47.8% of the AR from the treated melons and 21.4–42.9% of the AR from the treated distal leaves. The treated proximal leaves senesced and desiccated rapidly and therefore two leaves were lost and a low recovery was obtained from the third leaf. Similar but less severe ageing of the proximal leaf was observed on other vines with ripened melons (Table 3).

Table 3 Radioactive residues recovered in surface washes following application of [^{14}C]ethephon to different portions of melon plant

Plant portion to which [^{14}C]ethephon was applied	% of Applied Radioactivity [a]
Melon fruit rind	37.2–47.8
Distal leaf	21.4–42.9
Proximal leaf	12.2 [b]

[a] Range of three replicates

[b] Value for one replicate only. Proximal leaf desiccated and shattered in two replicates.

The total recovered radioactivity from the melon fruits after surface-washing was 6.90% of the AR following application to the melon rind, 1.14% following application to the distal leaf and 1.70% following application to the proximal leaf (Table 4).

Table 4 Radioactive residues in melon sections following application of [^{14}C]ethephon

Plant portion to which [^{14}C]ethephon was applied	% of Applied Radioactivity [a]			
	Rind	Flesh	Seed	Total
Melon fruit rind [b]	6.35	0.06	0.15	6.90
Distal leaf	0.60	0.47	0.07	1.14
Proximal leaf	0.87	0.67	0.14	1.70

[a] Average of three replicates.

[b] After surface washing.

Most (96–98%) of the radioactivity remained in the rind following topical application to the melon rind (Table 5).

Table 5 Distribution of radioactive residues in melon sections following application of [^{14}C]ethephon

Plant portion to which [^{14}C]ethephon was applied	% of Total Radioactivity [a]		
	Rind	Flesh	Seed
Melon fruit rind	96.3–97.8	0.6–1.4	1.5–2.4

Plant portion to which [^{14}C]ethephon was applied	% of Total Radioactivity [a]		
	Rind	Flesh	Seed
Distal leaf	33.1–67.9	30.8–58.8	1.3–15.7
Proximal leaf	29.0–80.7	13.6–61.1	5.7–13.3

[a] Range of three replicates

Ethephon was the only radioactive residue component identified by TLC (Table 6). No other radioactive component was detected.

Table 6 Concentration of [^{14}C]ethephon in melon sections following application of [^{14}C]ethephon

Plant portion to which [^{14}C]ethephon was applied	[^{14}C]Ethephon (µg/kg) [a]		
	Rind	Flesh	Seed
Melon fruit rind	14–34	0.04–0.11	0.60–2.3
Distal leaf	0.82–5.3	0.39–0.76	0.21–0.74
Proximal leaf	1.6–6.2	0.33–1.3	0.83–1.7

[a] Range of three replicates

Tomato

Tomato plants in outdoor plots were treated with a foliar application of [^{14}C]ethephon at 1.46 kg ai/ha and a water volume of 480 L/ha (Smith, 2002, CZ00E500, [M-240722-01-2]). The application timing was at the 'green mature' or 'colour break' stage of development. Tomato fruits were harvested on day 0 and 5 and 12 days after treatment (DAT). The 0 and 5 DAT samples were surface-washed with methanol, and then chopped and extracted with methanol. The 12 DAT samples were ground with dry ice and the total radioactivity was determined by combustion. The 12 DAT samples were subsequently extracted with methanol. Radioactivity in extracts was determined by LSC, and post-extraction solids were analysed by combustion analysis and LSC. Extracts were analysed by HPLC and TLC, and identification of ethephon and HEPA was performed by co-chromatography with reference standards.

The majority of the radioactive residue on 0 DAT was recovered in the surface wash, and most of the remainder was extracted with methanol. At 5 DAT, only 18% of the total radioactive residue (TRR) was recovered in the surface wash and the majority was extracted with methanol. Only 4.6% TRR remained unextracted. At 12 DAT, methanol extraction recovered 98% TRR, leaving only 2.3% TRR unextracted (Table 7).

Table 7 Total radioactive residues in tomato fruit after foliar application of [^{14}C]ethephon at 1.46 kg ai/ha

Fraction	0 DAT		5 DAT		12 DAT	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Methanol surface wash	6.5	83.7	0.31	18.4	Not performed	
Methanol extraction	1.3	16.0	1.3	77.1	1.1	97.8
Total extracted	7.8	99.7	1.6	95.5	1.1	97.8
Unextracted residue	0.025	0.4	0.078	4.6	0.026	2.3
TRR by extraction	7.8	100	1.7	100	1.2	106
TRR by combustion	Not performed		Not performed		1.1	100

The main component of the radioactive residue found in tomato fruit was ethephon (96, 70 and 59% TRR on 0, 5 and 12 DAT, respectively). The concentration of ethephon decreased over the time period in the study from 7.5 mg/kg at 0 DAT to 0.68 mg/kg at 12 DAT. The only significant metabolite was HEPA, amounting to 13–15% TRR in fruits of 5 and 12 DAT (Table 8). There were two other discernible metabolites that chromatographed close to HEPA, but both accounted for < 5% TRR and were not identified. The remainder of the unidentified radioactivity was polar in nature and did not exceed 8.5% TRR.

In tomato plants ethephon was metabolised by replacement of the chlorine in the 2-position with a hydroxy group to form HEPA; like in all other plants whose metabolism of ethephon was studied, the majority of the [^{14}C]ethephon applied was decomposed to volatile ethylene and phosphate.

Table 8 Identification of radioactive residues in tomato fruit after foliar application of [^{14}C]ethephon at 1.46 kg ai/ha

Fraction/compound	0 DAT		5 DAT		12 dayDAT	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
TRR by extraction	7.8	100	1.7	100	1.2	106
Total extracted	7.8	99.7	1.6	95.5	1.1	97.8
Ethephon						
—Methanol surface wash	6.3	81.2	0.3	18.1	–	–
—Methanol extract	1.2	14.9	0.9	51.5	0.71	59.4
—Total	7.5	96.1	1.2	69.6	0.71	59.4
HEPA						
—Methanol surface wash	0.14	1.8	0.01	0.3	–	–
—Methanol extract	0.02	0.2	0.25	14.7	0.16	13.2
—Total	0.16	2.0	0.26	15.0	0.16	13.2
Total identified	7.7	98.1	1.4	84.8	0.87	72.6
Unextracted residue	0.025	0.4	0.078	4.6	0.028	2.3

Wheat

Wheat plants at the forage stage (BBCH 39) in outdoor plots were treated with a foliar application of [^{14}C]ethephon at a normal field rate of 0.36 kg ai/ha and at a 10× rate of 3.6 kg ai/ha in a water volume of approximately 250 L/ha (Smith, 2002, CZ00E501, [M-240723-01-1]). Samples were harvested on 0 (forage), 14 (hay) and 34 (grain and straw) DAT. The 0 and 14 DAT samples were surface-washed with methanol, and then chopped and extracted with methanol. The 34 DAT samples were homogenized and extracted with methanol. The post-extraction solids from the grain (1× and 10× rate) and straw (1× rate) were subjected to acid hydrolysis with 5% HCl, yielding an acid hydrolysate. The residual fibres were extracted with methanol and then acetonitrile, yielding a post-hydrolysis extract and non-extractable residue. Radioactivity in extracts was determined by LSC, and post-extraction solids were analysed by combustion analysis and LSC. The TRR in the 0 and 14 DAT samples were determined by extraction and combustion of the residue. The TRR in the 34 DAT samples was determined by combustion. Extracts were concentrated and analysed by HPLC and TLC, and identification of ethephon and HEPA was performed by co-chromatography with reference standards.

For both application rates at 0 DAT, about half the radioactivity was quickly absorbed into the leaves. On 14 DAT, only a small amount of the applied radioactivity remained on the leaf surface (1.1% TRR) and almost all the radioactivity was recovered in the methanol extract, with about 5% TRR remaining unextracted (Table 9).

On 14 and 34 DAT, the majority of radioactivity was recovered in methanol extracts of plant parts (hay and straw) regardless of the dose used; radioactivity was similarly distributed in methanol surface wash and methanol extract of forage on 0 DAT. Unextracted residues were about 5% in 14 DAT hay but 10% (1×) and 26% (10×) in 34 DAT straw.

Methanol extraction could recover only 28 and 22% TRR from grain (34 DAT) samples after the low and high doses. Acid hydrolysis of remaining solid with 5% HCl released 56 and 71% TRR and extraction of the post-hydrolysis solids with methanol and then acetonitrile further released a total of 9.9% and 4.3% TRR. This indicates the significance of conjugates in grains. Unextracted residues were 1.8–6.0% TRR.

Table 9 Total radioactive residues in wheat fractions after foliar application of [^{14}C]ethephon at 0.36 kg ai/ha (1× rate) or 3.6 kg ai/ha (10× rate)

	Forage, 0 DAT		Hay, 14 DAT		Grain, 34 DAT		Straw, 34 DAT	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Application at 0.36 kg ai/ha (1× rate)								
Methanol wash	16.31	44.8	0.06	1.1	Not performed		Not performed	
Methanol extraction	19.94	54.9	4.79	94.1	0.30	27.5	1.38	57.8
Acid hydrolysate	Not performed		Not performed		0.60	56.1	0.47	19.9
Post-hydrolysis extract	Not performed		Not performed		0.11	9.9	0.29	12.4
Total extracted	36.25	99.7	4.85	95.2	1.00	93.5	2.14	90.1
Unextracted residue	0.12	0.4	0.23	4.9	0.06	6.0	0.23	10.1
TRR by extraction	36.37	100	5.09	100	1.07	99.5	2.37	100.2
TRR by combustion	Not performed		Not performed		1.07	100	2.37	100
Application at 3.6 kg ai/ha (10× rate)								
Methanol wash	110.56	45.6	0.22	1.2	Not performed		Not performed	
Methanol extraction	133.32	54.3	17.42	93.7	0.75	22.0	16.52	73.6
Acid hydrolysate	Not performed		Not performed		2.42	71.4	Not performed	
Post-hydrolysis extract	Not performed		Not performed		0.15	4.3	Not performed	
Total extracted	243.88	99.9	17.64	94.9	3.32	97.7	16.52	73.6
Unextracted residue	0.66	0.3	0.96	5.2	0.06	1.8	5.93	26.4
TRR by extraction	244.54	100.2	18.60	100.1	3.38	99.5	22.45	100
TRR by combustion	Not performed		Not performed		3.39	100	22.45	100

At all harvest times, most of TRR was attributed to the sum of ethephon and HEPA, and were the only residues identified. In 0 DAT forage (1× rate), the recovered radioactivity was primarily unchanged ethephon (Table 10).

In the 14 DAT hay, the major radioactive residue was HEPA with 72% TRR and 3.7 mg/kg followed by ethephon with 20% TRR and 1.0 mg/kg in the methanol extract. In the 34 DAT straw, the major radioactive residue was ethephon at 62% TRR (47% TRR in methanol extract, 9.3% in acid hydrolysate and 5.9% TRR in extracts of post acid hydrolysis solid) and 1.5 mg/kg.

In 34 DAT grain, HEPA was found at a similar level as ethephon after the low dose: HEPA, 48% TRR (14% TRR in methanol extract, 29% TRR in acid hydrolysate and 5.5% TRR in extracts post-hydrolysis solid) and 0.51 mg/kg; and ethephon, 44% TRR (13% TRR in methanol extract, 26% TRR in acid hydrolysate and 4.4% TRR in extracts of post-hydrolysis solid) and 0.47 mg/kg. After the higher dose, approximately two times larger amounts of HEPA was found than ethephon (HEPA, total of 60% TRR and 2.0 mg/kg; and ethephon, total of 32% TRR and 1.1 mg/kg). No other metabolites exceeded 3% of TRR.

In total, in 14 and 34 DAT samples, 88–92% of the radioactive residue was identified as ethephon and HEPA, with no other single metabolite comprising more than 2.6% TRR.

Table 10 Identification of radioactive residues in wheat fractions after foliar application of [^{14}C]ethephon at 0.36 kg ai/ha (1× rate) or 3.6 kg ai/ha (10× rate)

	Forage, 0 DAT		Hay, 14 DAT		Grain, 34 DAT		Straw, 34 DAT	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Application at 0.36 kg ai/ha (1× rate)								
TRR	36.4	100	5.09	100	1.07	100	2.37	100
Total extracted	36.3	99.7	4.85	95.2	1.00	93.5	2.14	90.1
Ethephon								
—Methanol wash	16.0	43.9	–	–	–	–	–	–
—Methanol extract	18.9	52.0	1.00	19.7	0.14	13.0	1.12	47.1
—Acid hydrolysate	–	–		–	0.28	26.1	0.22	9.3
—Post-hydrolysis ext.	–	–		–	0.05	4.4	0.14	5.9
—Total	34.9	95.9	1.00	19.7	0.47	43.5	1.48	62.3
HEPA								
—Methanol wash	0.15	0.4	–	–	–	–	–	–
—Methanol extract	0.58	1.6	3.67	72.2	0.15	13.6	0.22	9.1

	Forage, 0 DAT		Hay, 14 DAT		Grain, 34 DAT		Straw, 34 DAT	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
—Acid hydrolysate	–	–	–	–	0.31	28.6	0.25	10.6
—Post-hydrolysis ext.	–	–	–	–	0.06	5.5	0.15	6.4
—Total	0.73	2.0	3.67	72.2	0.51	47.7	0.62	26.1
Total identified	33.6	97.9	4.68	91.9	0.98	91.2	2.09	88.4
Unextracted residue	0.14	0.4	0.23	4.9	0.06	6.0	0.24	10.1

	Grain (34 day)	
	mg/kg	% TRR
Application at 3.6 kg ai/ha kg ai/ha (10× rate)		
TRR	3.39	100
Total extracted	3.32	97.7
Ethephon		
—Methanol extract	0.28	8.3
—Acid hydrolysate	0.74	21.8
—Post-hydrolysis ext.	0.04	1.2
—Total	1.08	31.8
HEPA		
—Methanol extract	0.41	12.1
—Acid hydrolysate	1.53	45.1
—Post-hydrolysis ext.	0.11	3.2
—Total	2.04	60.3
Total identified	3.12	92.1
Unextractable residue	0.06	1.8

In summary, ethephon is metabolised in wheat to form HEPA. The residue in 0 DAT wheat forage comprised mainly ethephon, with low levels of HEPA. In hay, grain and straw, the residue consisted of ethephon and HEPA; no other metabolites were identified.

Hazelnut (Filberts)

Two filbert trees (in the Codex Classification of Foods and Animal Feeds, the entry “Filberts” refers to “Hazelnuts” with the description, “among other *Corylus maxima*, Mill; and the “Hazelnuts”, include *C maxima* and *C. avellana*.) were treated with a foliar spray of non-radio-labelled ethephon at 1000 mg/kg and, six hours later, 2960 kBq [^{14}C]ethephon was applied to two branches of the trees (Anonymous, 1972, [M-188020-01-1]). One branch had 36 leaves and 9 nuts in husks, of which the upper surfaces of 18 leaves and two husks were treated. The other branch had 35 leaves and 11 nuts in husks, of which 15 leaves and three husks were treated.

The treated branches were separately enclosed in a screen cage wrapped in a plastic bag. Small holes in the bags allowed air to enter and flow through the bags. The bags were fitted with tubing which was connected to a gas trapping system consisting of an absorber containing water-saturated n-butanol and a mercuric perchlorate-perchloric acid solution to absorb ethylene. Air was drawn through the gas trapping system at a rate of 475 cm^3/minute. Ethylene absorption was continued for 7 days. [^{14}C]Ethylene in the absorber solution was measured using a liquid scintillation spectrometer.

Filbert nuts were harvested 7 and 14 DAT. Two different types of nut samples were collected: those treated directly on the husk, and those from limbs with treated leaves. Samples were frozen after collection. Nuts were separated into kernels, shells and husks and the samples were ground. TRR were determined by combustion analysis. The 7 DAT nutmeat was extracted by soxhlet extraction for 4 hours with benzene followed by methanol. The benzene extract did not contain any radioactivity and was discarded. The extracted residue was analysed by combustion.

The methanol extract was acidified, concentrated by rotary evaporation and then under nitrogen. The resulting extract was acidified, treated with diethyl ether and centrifuged. The resulting extract was concentrated, diluted with methanol and extracted with isooctane. The isooctane did not contain the radioactive residue and was discarded. The remaining methanol

extract was cleaned-up using a silica gel column and analysed by paper chromatography. The alkaline decomposition of the radioactive residue was investigated by treating an aliquot of the filbert extract with methanol/20% potassium hydroxide solution (1:1 v/v), by refluxing at 60 °C for 8 hours. A sample of control filbert extract was spiked with [^{14}C]ethephon and treated with alkali in the same way.

A significant amount of applied radioactivity was released over the 7 day period after treatment with [^{14}C]ethephon. The greatest amount of ethylene was release on the first day after treatment, gradually declining over the 7 day period (Table 11).

Only a small amount of the applied radioactivity was translocated onto the kernels (nutmeat) 7 DAT: 0.002 mg/kg and 0.87 mg/kg following application to the leaves and husk, respectively. The amount remaining in the kernels was even lower 14 DAT: 0.002 mg/kg and 0.14 mg/kg following application to the leaves and husk, respectively (Tables 12 and 13).

The 7 DAT nutmeat was extracted with benzene to remove the fats/oils. No radioactive residues were detected in the benzene fraction. Extraction with methanol released 98% of the TRR, with a further 1.6% remaining unextracted. After clean-up of the methanol extract, paper chromatography showed that the residue in nutmeat consisted of ethephon. No other radio-labelled component was detected. The presence of ethephon was confirmed by demonstrating that the radioactive residue in the nutmeat extract completely decomposed when treated with a strong base, as the alkaline treatment of [^{14}C]ethephon-spiked control extract confirmed this behaviour (of ethephon having been treated with a strong base).

Table 11 Release of [^{14}C]ethylene after application of [^{14}C]ethephon to filberts

DAT	[^{14}C]Ethylene released	
	dpm	kBq
1	4778000	79.9
2	2378000	87.0
3	2280700	42.2
4–7	1786700	30.4

Table 12 Distribution of [^{14}C]residues in filberts

Plant portion to which [^{14}C]ethephon was applied	DAT	[^{14}C]Ethephon Residue (dpm) [a]		
		Nutmeat	Husks	Shells
Leaves	7	24	566	34
Husks	7	8150	309900	4482
Leaves	14	19	10380	8
Husks	14	1311	257700	1161

[a] Average of three replicates

Table 13 Concentration of [^{14}C]ethephon in filbert kernels

Plant portion to which [^{14}C]ethephon was applied	7 DAT		14 DAT	
	mg/kg	Dpm	mg/kg [a]	dpm [a]
Leaves	0.002	24	0.002	19
Husk	0.87	8150	0.14	1311

[a] Average of three replicates

Cotton

Cotton plants in outdoor plots were treated with a foliar application of [^{14}C]ethephon at a rate of 1.40 kg ai/ha in a water volume of approximately 500 L/ha (Smith, 2003, 601CZ, [M-240888-01-2]). The application timing corresponded to a 7 day PHI. Samples of treated cotton leaves were collected 0 DAT, immediately after the application had dried. The remaining plants were harvested 7 DAT according to normal agricultural practices, and separated into gin trash, lint and seed. The lint was not analysed further. The 0 DAT samples were surface-washed with acetonitrile, and then extracted with

acetonitrile. The mature (7 DAT) samples were frozen, ground and combusted to determine the TRR. The gin trash samples were extracted with methanol:water (9:1). The seed samples were extracted with methanol. The post-extraction solids from the gin trash and seed were hydrolysed with a mixture of concentrated HCl and water (1:7), yielding an acid hydrolysate. Radioactivity in extracts was determined by LSC, and post-extraction solids were analysed by combustion analysis and LSC. The TRR in the 0 DAT leaf samples was determined by extraction and combustion of the residue. The TRR in the 7 DAT samples was determined by combustion. Extracts were concentrated and analysed by HPLC, and identification of ethephon and HEPA was performed by comparison of retention times with radio-labelled reference standards. Identification was confirmed by TLC.

Radioactive residues recovered in leaves at 0 DAT (237 mg/kg) declined rapidly over 7 days after application. Gin trash and seed samples from 7 DAT (final harvest) contained TRR of 31.4 mg/kg and 0.82 mg/kg, respectively (Table). The percentage of residue extracted from leaves harvested 0 DAT by acetonitrile wash and extraction was relatively low (in total 62.5% TRR), but this extraction was used only for the residue levels at 0 DAT and to develop extraction methods for the 7 DAT samples. Methanol extraction of mature gin trash (with the addition of water at a ratio 1:9 of methanol) and seed proved very effective, recovering 89% TRR in gin trash and 82% TRR in seeds respectively. Acid hydrolysis with HCl:water (1:7) further recovered the majority of the remainder of the residue (11% TRR in gin trash and 17% TRR in seeds), leaving only 0.2% TRR remaining unextracted, potentially fibre-bound, in the gin trash and 1.2% in the cotton seed.

Table 14 Total radioactive residues in cotton after foliar application of [^{14}C]ethephon at 1.40 kg ai/ha

Fraction	Leaves, 0 DAT		Gin Trash, 7 DAT		Seeds, 7 DAT	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Acetonitrile wash	160.15	61.6	Not performed		Not performed	
Solvent extraction	1.35	0.9	27.81	88.6	0.67	82.1
Acid hydrolysate	Not performed		3.52	11.2	0.14	16.8
Total extracted	161.50	62.5	31.33	99.8	0.81	98.9
Unextracted residue	75.77	37.6	0.08	0.2	0.01	1.2
TRR by extraction	237.27	100	31.41	100	0.82	100
TRR by combustion	Not performed		31.41	100	0.82	100

The predominant radioactive residue in gin trash was ethephon at 93% TRR (84% TRR in the methanol:water extract and 9.3% TRR in acid hydrolysate) and 30 mg/kg and 78% TRR (66% TRR in the methanol extract and 12% in acid hydrolysate) and 0.64 mg/kg in seeds. HEPA was low at a total of 1.7% TRR and 0.52 mg/kg in gin trash and 9.6% TRR and 0.08 mg/kg in seeds. A total of 88–95% of the residue in these RACs was identified as ethephon and HEPA, with no other single metabolite comprising more than 1.9% of the residue.

Table 15 Identification of residues in cotton after foliar application of [^{14}C]ethephon at 1.40 kg ai/ha

	Leaves (0 day)		Gin Trash (7 day)		Seeds (7 day)	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
TRR by extraction	237.3	100	31.4	100	0.82	100
Total extracted	160.2 [a]	61.6 [a]	31.3	99.8	0.81	98.9
Ethephon						
—Surface wash	156.3	59.2	–	–	–	–
—Extract (methanol or Methanol + water, 9:1)	–	–	26.3	83.7	0.54	66.1
—Acid hydrolysate	–	–	2.9	9.3	0.10	12.2
—Total	156.3	59.2	29.2	93.0	0.64	78.3
HEPA						
—Surface wash	0.24	0.2	–	–	–	–
—Extract (methanol or Methanol + water, 9:1)	–	–		1.3	0.06	7.7
—Acid hydrolysate	–	–		0.4	0.02	1.9
—Total	0.24	0.2	0.52	1.7	0.08	9.6

	Leaves (0 day)		Gin Trash (7 day)		Seeds (7 day)	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Total identified	156.5	59.4	30.20	94.7	0.72	87.8
Unextractable residue	75.8	37.6	0.08	0.2	0.01	1.2

[a] For leaves, only the surface wash was profiled

The majority of the ethephon applied to cotton is decomposed to volatile ethylene and phosphates. The metabolic pathway for ethephon in cotton was replacement of the chlorine atom in the 2-position with a hydroxyl function to give HEPA. The main residue found in cotton leaves, gin trash and seed was parent ethephon.

Data from Published Literature

Apple and Cherry (Edgerton and Hatch, 1972)

Radioactive ethephon labelled with ^{14}C was applied (500 ppm with 0.1% of Tween 20) to leaf and fruit surfaces of selected branches of apple and cherry trees 6 to 10 days before normal harvest dates. Samples were collected periodically following application and analysed with appropriate extraction and counting procedures. The level of radioactive ethephon increased in the fruit for about 48 to 72 hr, then decreased to a low level after 6 days. No intermediate metabolites were detected in the fruits. It was found that the majority of the ethephon in the fruits moved there from the application on adjacent leaves; relatively small amounts moved directly into the fruit from surface application. Radioactive ethylene was detected within 12 hr after application of the [^{14}C]ethephon on the leaf surfaces.

Cherry (Gilbert et al., 1975)

The metabolism of [^{14}C]ethephon was investigated after application to the leaves of cherry trees. In extracts from cherry leaves harvested 3 and 11 days after treatment, a metabolite was detected by TLC. The ratio of metabolite to ethephon was greater at 11 days than at 3 days after application. Based on the fact that the metabolite could also be chromatographed on an anion exchange resin column, it was suggested that the metabolite contains an intact phosphonic acid or other anionic group. Characterisation by mass spectrometry was not possible due to matrix interferences.

Peach (Giulivo et al., 1981)

The translocation and metabolism of 1,2-[^{14}C]ethephon was investigated in Andross peach trees at the end of Stage 1 of fruit development. [^{14}C]Ethephon was applied to the fruit surface or to the abaxial surface of the basal leaf of a developing shoot. Translocation did not occur following application to the fruit, but did occur following application to the leaf. TLC analysis indicated that the translocated radioactivity was associated with sugars. However the binding to sugars was not a metabolic reaction.

Grape

The translocation of [^{14}C]ethephon was investigated after spray application to grapevines (Weaver *et al*, 1972). At 7 days after treatment, 62% of the recovered radioactivity remained on the surface of the treated grape berries. In concentrated extracts of methanol-washed grape berries, parent ethephon was detected by TLC, but no radioactive metabolite was found. Application of ethephon to the first leaf above the cluster, or to a berry pedicel or peduncle, failed to result in measurable translocation of ethephon into the berries.

The uptake, translocation and fate of [^{14}C]ethephon in detached grapevine leaves and intact shoots was investigated (Nir and Lavee, 1981). Mature Perlette leaves were treated with [^{14}C]ethephon and the leaves put under constant fluorescent light (9 W/m^2) for 48–120 hours. Recovery of radioactivity from detached leaves was 53–61% after 48 hours, and reduced to 25% after 120 hours. Translocation was found to be mainly basipetal, and this was confirmed by autoradiography.

When young leaves near the apex of young detached cardinal shoots were treated with [^{14}C]ethephon, recovery after 48 hours was 85.5%. 7.5% remained on the leaf surface and 78% was extracted from the shoots. There was almost no translocation to other parts of the shoots.

Application of [^{14}C]ethephon to different sites on the upper parts of young growing shoots (cut surface, shoot apex and mature leaves) showed that translocation was very slight and after 4 hours, recovery was 58–72%. In mature leaves, only 2.4% of the radioactivity had penetrated the tissue, whereas 21–26% had penetrated the apical tissues. Translocation of [^{14}C]ethephon was very slight and most of the applied compound remained at the application site for many hours. No measurement of the loss of ^{14}C as volatiles was made.

Squash, Cucumber and Tomato (Yamaguchi et al, 1971)

The fate of [^{14}C]ethephon was investigated after application to squash, cucumber and tomato plants. At 7 days after application of a [^{14}C]ethephon solution to tomato leaves, about 15% of the radioactivity was recovered from the treated leaves and about 50% had been converted to [^{14}C]ethylene. About 12% of the radioactivity applied was translocated to immature fruits on the same branch. Analysis by paper chromatography showed that the radioactivity recovered from the fruit surface and tissue extracts comprised parent ethephon.

After injection of [^{14}C]ethephon into petioles of summer squash, more than 20% of the applied radioactivity was converted to [^{14}C]ethylene during the first day, followed by slightly less than 15% in the second day. There was a rapid decline in radioactivity in the petioles after the first day which was accompanied by translocation of radioactivity to other parts of the seedlings. One day after application, the radioactive residue comprised mainly ethephon. At 2 days after application the presence of an unknown metabolite was noted and at 6 days after treatment the amount of the unknown metabolite at the site of application was greater than that of ethephon. The translocated radioactivity was all in the form of the unidentified metabolite.

Four days after an application of [^{14}C]ethephon solution to cucumber leaves and fruits, about 40% of the total remaining radioactivity was found to be ethephon. No identification of characterization of the remaining 60% of the radioactive residue was performed.

This paper indicates that the main route of metabolism of ethephon in tomato is conversion to ethylene, and translocation of ethephon occurs. In contrast, in summer squash, besides the formation of ethylene, an unidentified metabolite is formed which is translocated to other parts of the plants whereas translocation of ethephon is not observed. In tomato tissue, the radioactive residue comprised [^{14}C]ethephon, but in squash seedlings much of the radioactivity was present in the form of the unidentified metabolite.

Walnut (Martin et al, 1972)

[^{14}C]Ethephon applied to a walnut leaflet was found to penetrate and translocate rapidly in young plants, but more slowly in older plants. The compound translocated to the kernel at higher levels when applied to a leaflet than when applied to the hull, but levels of radioactivity were low in both cases. Between 5–7 days after application, the amount of radioactivity in the kernel decreased markedly. It was concluded from the decrease in radioactivity that [^{14}C]ethephon in the leaves, hull, shell and kernel was metabolised. TLC analysis revealed the presence of [^{14}C]ethephon in leaf, hull and kernel extracts; however, no metabolites remained in the plant tissue that could be detected by TLC. No measurement of [^{14}C]ethylene was made in this study.

Proposed metabolic pathway of ethephon in plants

The metabolism of ethephon in a wide range of crops were studied. Information taken from published literature was also provided. Recent studies on tomatoes, wheat and cotton (2002–2003) and older studies (1968–1981) on apples, cherries, peaches, grapes, pineapples, cantaloupes, summer squash, cucumbers, tomatoes, filberts and walnuts showed similar metabolism of ethephon.

In the tomato study, the plants were foliarly-treated with 1.44 kg ai/ha of [^{14}C]ethephon. Parent ethephon was found to be the major residue component in tomato fruit harvested 0, 5 and 12 days after treatment. HEPA represented up to 15% of the total radioactive residue.

In the wheat study [^{14}C]ethephon was foliar sprayed at the rate of 0.36 kg ai/ha when the plants had reached the ligule stage (BBCH 39). At mature harvest, grain showed similar levels of parent ethephon and HEPA, whereas straw was found to contain higher levels of ethephon than of HEPA.

In the cotton study, the plants were treated with 1.40 kg ai/ha of [^{14}C]ethephon. The majority of the residue in cotton seed and gin trash harvested 7 days after treatment was parent ethephon. HEPA represented 1.7% of the total radioactive residue in gin trash and 9.6% in seed.

Overall, the main degradation route of ethephon was shown to involve decomposition of ethephon to ethylene and phosphates. The ethylene is rapidly released into the atmosphere while the phosphates are taken up in the natural phosphate cycle of the plant. However, part of the applied ethephon is metabolized according to a different metabolic pathway that results in the formation of the metabolite HEPA. HEPA is further metabolized by incorporation of the two carbon atoms in natural bio-molecules. The proposed metabolic pathway of ethephon in plants is presented below.

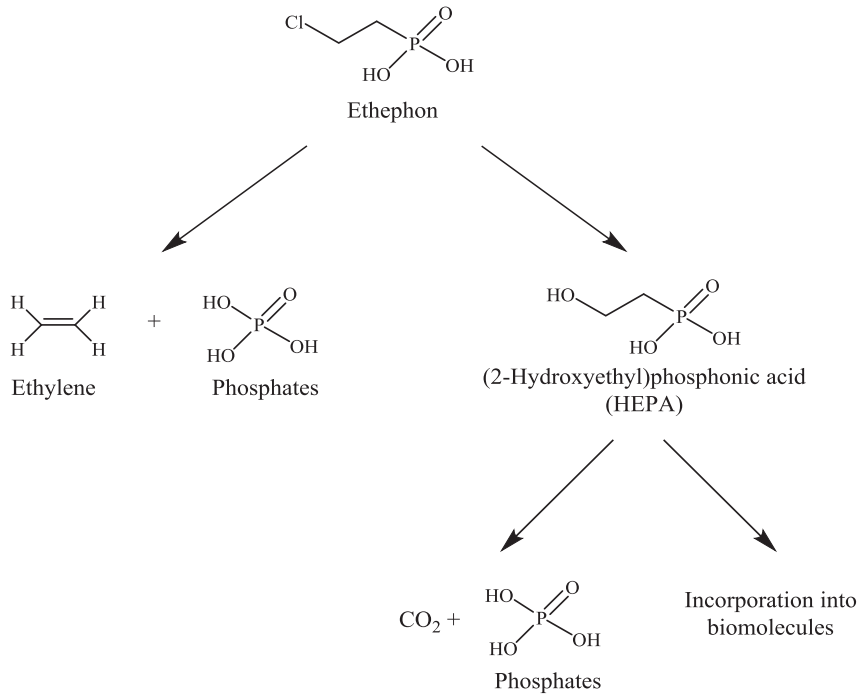


Figure 2 Proposed Metabolic Pathway of Ethephon in Plants

Animal Metabolism

The Meeting received information on the results of studies on lactating goats and laying hens which were fed [^{14}C]ethephon.

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

After oral administration of ethephon to rats, absorption was rapid with a Tmax of 1.0–1.3 hours and 1.9–2.5 hours after a single oral dose of 50 or 1000 mg/kg bw, respectively. Six days after a single dose, tissue and carcass contained only 0.08% or less of administered radioactivity. Highest concentrations were found in liver and kidney. Radioactivity was excreted in urine (47–60%), expired air (18–21%, mainly ethylene) and faeces (4–6.5%), indicating that at least 65% of the administered dose was absorbed. Ethephon was mainly metabolized to ethylene and to a small extent to HEPA.

Lactating goats

The metabolism of ethephon in the lactating goat (Nubian and Alpine/Nubian cross) has been studied using [^{14}C]ethephon (Huhtanen *et al.*, 1984, ETH/M3, [M-187423-01-1]; Fisher, 2005, C046890, [M-223288-02-1]). The [^{14}C]ethephon was administered twice daily orally in capsules to two lactating goats for seven consecutive days. One dose followed the morning milking, and the other followed the afternoon milking. The goats received mean daily doses of 0.37 and 0.46 mg/kg bodyweight/day, respectively, equivalent to a dose level of approximately 10 ppm in the diet. A third goat served as a control animal.

Urine, faeces, milk and blood samples were collected daily. Milk samples were collected twice daily, in the morning and in the afternoon, approximately ten hours later, immediately prior to dosing. Selected milk sub-samples were separated into skimmed milk and milk fat by centrifugation. Whole blood samples were collected from each animal immediately prior to the afternoon dose. On Day 6, blood was collected from each goat at intervals of 0.25, 0.5, 1, 2, 4, 6, 8 and 10 hours after the morning dose. Volatile compounds were collected for 24 hours on the seventh day of the study. Carbon dioxide was trapped using 10% aqueous potassium hydroxide and ethylene was trapped using mercuric perchlorate solution. The animals were sacrificed approximately 16 hours after the final dose, and the following tissues were collected: liver, kidney, heart, composite fat, skeletal muscle, blood, and contents of the stomach and small and large intestine.

Radioactivity was quantified by LSC. Liquid samples (milk and urine) were analysed directly by LSC. Solid samples (tissues and intestinal contents) were analysed by oxidative combustion followed by LSC.

Freeze-dried sub-samples of liver were extracted with ether and then methanol. Extracts were radio-assayed and the remaining solids were analysed by combustion. Proteins and glycogen from the liver were isolated and analysed by combustion. Levels of ethephon in tissues, urine and milk were determined by base hydrolysis to ethylene which was trapped in a mercuric perchlorate solution.

A major proportion of the administered dose was released as volatiles in the form of ethylene (29% of administered dose) and CO_2 (2% of administered dose). Urinary excretion accounted for 19% and faecal excretion about 7% of the administered dose. Only 3.3% was excreted in milk and 3% remained in tissues on Day 7 (Table). The low total recovery (64%) was attributed to the difficulties in trapping large amounts of volatile compounds and the fact that volatile compounds were only collected over a 24 hour period.

Table 16 Distribution of radioactivity in tissues, milk and excreta from goats following oral administration of [^{14}C]ethephon at a nominal dietary concentration of 10 ppm for 7 days

Fraction	% of Administered dose
[^{14}C]ethylene [a]	29 [a]
$^{14}CO_2$ [a]	2.0 [a]
Urine	19
Faeces	6.7
Milk	3.3
Tissues	3.0
Gut contents	0.84
Total Recovery	64

[a] [^{14}C]ethylene and $^{14}CO_2$ were collected only over a 24-hour period on Day 7

Kidney and liver contained the highest total radioactive residue, at 1.2 and 1.0 mg/kg, respectively. TRRs in heart and muscle were low at 0.16 and 0.10 mg/kg, respectively, whilst fat contained a TRR of 0.50 mg/kg (Table).

Table 17 Average concentration of radioactive residues in tissues of goats sacrificed 16 hours after oral administration of [^{14}C]ethephon at a nominal 10 ppm for 7 days

Tissue	TRR, mg/kg
Kidney	1.2
Liver	1.0
Fat	0.50
Heart	0.16
Muscle	0.10

Average radioactive residue levels in whole milk were 0.28 mg/kg on Day 1, 0.36 mg/kg on Day 2 and 0.37 mg/kg on Day 3. Radioactive residue levels in milk increased until the afternoon milking on Day 3, where a plateau level of about 0.42 mg/kg was reached (Table). The milk fat fraction contained 45% of the radioactivity in milk. Radioactive residue concentrations in skimmed milk were 0.15–0.20 mg/kg, whilst those in milk fat were 3.03–4.18 mg/kg. As ethephon is hydrophilic and not expected to partition into fat, the residue in milk fat was attributed to incorporation of ^{14}C via [^{14}C]acetate into milk fats.

Table 18 Average concentration of radioactive residues in milk from goats during oral administration of [^{14}C]ethephon at a nominal 10 ppm for 7 days

Time, days	Average concentration, mg/kg
0.5	0.081
1.0	0.279
1.5	0.318
2.0	0.357
2.5	0.366
3.0	0.371
3.5	0.420
4.0	0.380
4.5	0.394
5.0	0.427
5.5	0.423
6.0	0.405
6.5	0.422
7.0	0.419

For the determination of ethephon, base degradation method was used to convert parent ethephon to ethylene. The analytical results indicate that no ethephon were present in fat, muscle, liver and milk. Kidney was the only tissue which yielded measurable levels of ethylene after base hydrolysis, equivalent to ethephon levels of 0.0085 mg/kg. Extraction of liver with ether released 5.3% TRR, extraction with methanol released a further 63.7% TRR, and 27.2% TRR remained in the post-extraction solids. Precipitation with trichloroacetic acid showed that 12.4% TRR in liver was associated with proteins. Radioactivity was also found to be associated with liver glycogen.

The incorporation of radiocarbon into liver protein, glycogen and fats as well as the elimination of $^{14}CO_2$ demonstrated that ethephon was incorporated into natural products possibly through an acetate-like intermediate. It was observed that radioactive carbon was present in milk fat and fat tissue, which indicates metabolic degradation of ethephon to a less hydrophilic compound.

The results show that significant amounts of the parent ethephon are degraded to ethylene and respired. The absence of parent ethephon in tissues demonstrated the complete metabolic degradation of ethephon, probably through an acetate-like intermediate. The study indicated that

there is low potential for transfer of residues of ethephon and/or its metabolites to milk, meat or meat by-products in ruminants after dietary exposure to ethephon.

Laying Hens

In the first study (Byrd, 1992, 9015C, [M-179283-01-1]), eight hens received daily oral capsule doses of [^{14}C]ethephon for five consecutive days at a rate equivalent to 53 ppm diet. Three hens in Group I were individually housed in metabolism cages designed to collect expired ethylene in a 2 M mercuric perchlorate trap solution and CO_2 in a 2 M sodium hydroxide solution. Five hens in Group II were individually housed in layer cages. Five other hens in Group III served as controls, and were individually housed in layer cages. Eggs were collected twice daily and excreta were collected once daily. Blood was collected prior to termination. Hens were terminated 22–23 hours after the final dose, and the following tissues collected: liver, kidney, muscle, fat, gastrointestinal tract and contents. A cage wash sample was collected after termination.

Liver, kidney, muscle, fat, yolk (Day 4) and excreta (Day 5) were freeze-dried and sequentially extracted with hexane and methanol using soxhlet extraction. The hexane and methanol extracts were pooled and the unextracted residues were subjected to enzyme hydrolysis (glucuronidase and sulphatase), and acid and base hydrolysis. The hydrolysates were extracted with dichloromethane but no radioactivity in any of hydrolysates partitioned into the organic layer. Radioactivity in extracts and hydrolysates was determined by LSC. Solid samples were analysed by combustion and LSC. Radioactivity in extracts was characterised by radio TLC.

The majority of the radioactivity (58% of the administered dose) was recovered as ethylene in the mercuric perchlorate trap solution. The identity of ethylene was confirmed by GC/MS headspace analysis. The amount of radioactivity trapped as $^{14}CO_2$ was negligible. A significant amount (26–30%) of the administered dose was recovered in the excreta. Radioactive residues in the CO_2 trap, eggs and tissues accounted for less than 1% of the total radioactivity administered (Table).

Table 19 Distribution of radioactivity in tissues, eggs and excreta from hens following oral administration of [^{14}C]ethephon at a nominal dietary concentration of 53 ppm for 5 days

Sample	% of Administered dose
[^{14}C]ethylene	58
$^{14}CO_2$	< 1
Excreta	26, 30[a]
Eggs (whole)	< 0.1
White	0.00
Yolk	0.05
Liver	0.05
Kidneys	0.01
Muscle	0.03
Fat	0.01
Plasma	0.01
Erythrocytes	0.01

[a] Group in the metabolism cages (Group I) and layer cages (Group II), respectively

The highest TRR among tissues was found in liver (0.31 mg/kg), followed by kidneys (0.23 mg/kg) and fat (0.15 mg/kg) (Table). The TRR in eggs increased to a plateau level of about 0.18 mg/kg (mean of Groups II and III) after 4 days (Table).

Table 20 Concentration of radioactive residues in tissues of hens following oral administration of [^{14}C]ethephon at a nominal 53 ppm for 5 days

Tissue	TRR, mg/kg
Eggs	0.18 [a]
White	0.042[a]
Yolk	0.45 [b]
Liver	0.31

Tissue	TRR, mg/kg
Kidneys	0.20
Muscle	0.023
Fat	0.15
Plasma	0.078
Erythrocytes	0.063

[a] Highest residue concentration (found on Day 4)

[b] Highest residue concentration (found on Day 5)

Table 21 Mean concentration of radioactive residues in eggs from hens following oral administration of [^{14}C]ethephon at a nominal 53 ppm for 5 days

Study day	Average concentration, mg/kg					
	Group I (expired air cage)			Group II (layer cage)		
	White	Yolk	Whole egg	White	Yolk	Whole egg
1	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
2	0.014	0.028	0.019	0.016	0.043	0.025
3	0.028	0.180	0.078	0.028	0.199	0.086
4	0.041	0.205	0.149	0.043	0.541	0.216
5	0.033	0.408	0.154	0.034	0.509	0.203

Soxhlet extraction released the largest amount of radioactivity from all samples, except excreta. In excreta, the majority of the radioactivity was released by enzyme and acid hydrolysis. For all tissues, more than 75% of the residue was characterized. In liver and kidney, the radioactive residue was less readily extracted by solvent and 27% TRR in liver and 41% TRR in kidney remained unextracted. The extracted residue from liver and egg yolk could not be characterized by TLC due to the low amount of radioactivity in the extracts and interference from co-extractives.

Table 22 Characterisation of residues in tissues, egg yolk (Day 4) and excreta (Day 5)

Fraction	Liver		Kidney		Muscle		Fat		Egg yolk (Day 4)		Excreta (Day 5)	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Soxhlet extraction	0.16	52	0.083	41	0.012	53	0.15	101	0.27	72	1.0	8.1
Enzyme hydrolysis	< 0.01	2.3	< 0.01	2.5	< 0.01	2.5	< 0.01	0.0	0.025	6.7	5.8	45
Acid hydrolysis	< 0.01	0.5	< 0.01	0.2	< 0.01	0.9	< 0.01	0.9	< 0.01	2.4	2.2	17
Base hydrolysis	< 0.01	2.3	< 0.01	1.8	< 0.01	0.0	< 0.01	3.6	< 0.01	2.1	1.1	8.9
Bound residues	0.084	27	0.083	41	< 0.01	0.4	< 0.01	0.5	0.041	11	2.9	22
Total recovery		84		87		83		106		95		101

The results indicate that the metabolism of ethephon proceeds almost exclusively by hydrolysis and dechlorination to ethylene, which is then expired. It appears that incorporation of the two carbon moiety into cellular components may result as no other radioactive metabolite could be isolated in tissue extracts.

In the second study (Schocken, 1995, 94-10-5526, [M-188154-01-1]), two groups (Groups II and III) of five hens received daily gavage doses of [^{14}C]ethephon for five consecutive days at a rate equivalent to 59 ppm diet (Group II) or 67 ppm diet (Group III). Five hens in Group II were individually housed in metabolism cages designed to collect expired ethylene in a 2 M mercuric perchlorate trap solution and CO_2 in a 2 M sodium hydroxide solution. Five hens in Group III were individually housed in layer cages. Three hens in Group I served as controls

and were individually housed in layer cages. Eggs were collected twice daily, and excreta were collected once daily. Blood was collected prior to termination. Hens were terminated 9–10 hours after the final dose, and the following tissues collected: liver, kidney, muscle, fat, gastrointestinal tract and contents.

Radioactivity in liquid samples was determined by LSC. Solid samples were analysed by combustion and LSC. [^{14}C]ethylene was confirmed by GC/MS headspace analysis of the mercuric perchlorate trap. $^{14}CO_2$ in the sodium hydroxide trap was determined by barium carbonate precipitation.

Fat and egg yolk samples were extracted with hexane/tetrahydrofuran. Other tissue samples were extracted with methanol/water. Fat and egg yolk were saponified with methanolic potassium hydroxide and analysed by LC/MS and/or HPLC to identify radio-labelled fatty acids, cholesterol and glycerol. The post-extraction solids from muscle, kidney, liver, egg white and egg yolk samples from Group III were digested with protease. Aliquots of hydrolysates were further hydrolysed with 6 N HCl. The protease and acid hydrolysates were profiled by HPLC to detect the presence of radio-labelled amino acids. The remaining solids were analysed by combustion.

The majority of the radioactivity was recovered in excreta, accounting for about one third of the administered dose. Radioactive residues in tissues accounted for 0.12–0.14% of the dose, with the highest concentrations in kidney (0.71–1.1 mg/kg) and liver (0.63–0.90 mg/kg) and lowest concentrations in fat (0.051–0.091 mg/kg) and muscle (0.051–0.058 mg/kg). Radioactive residues in egg white and egg yolk accounted for 0.03% and 0.07–0.10% of the dose, respectively. Due to leakage in the gas collection system, a total of only 2.7% of the administered dose was recovered in the expired volatiles trap (Table).

Table 23 Distribution of radioactivity in tissues, eggs and excreta from hens following oral administration of [14C]ethephon for 5 days

Fraction	% of Administered dose	
	59 ppm Diet (Group II) (expired air cage)	67 ppm Diet (Group III) (layer cage)
[^{14}C]ethylene	2.66 [a]	Not collected
$^{14}CO_2$	0.03	Not collected
Excreta	26	36
Egg white	0.03	0.03
Egg yolk	0.07	0.10
Tissues	0.12	0.14

[a] Recovery of expired [^{14}C]ethylene is not representative due to leakage in the gas collecting system

The highest TRR among tissues was found in kidneys (1.1 and 0.71 mg/kg), followed by liver (0.90 and 0.63 mg/kg) (Table). The TRR in eggs increased to a level of about 0.40 mg/kg (mean of Groups II and III) after 5 days (Table).

Table 24 Mean concentration of radioactive residues in tissues of hens following oral administration of [^{14}C]ethephon for 5 days

Tissue	TRR, mg/kg	
	59 ppm Diet (Group II) (expired air cage)	67 ppm Diet (Group III) (layer cage)
Egg white	0.10	0.10
Egg yolk	1.0	1.0
Liver	0.90	0.63
Kidneys	1.1	0.71
Muscle	0.058	0.051
Fat	0.091	0.051

Table 25 Mean concentration of radioactive residues in eggs from hens following oral administration of [^{14}C]ethephon for 5 days

Study day	Average concentration, mg/kg						Mean whole egg [a], mg/kg
	59 ppm Diet (Group II) (expired air cage)			67 ppm Diet (Group III) (layer cage)			
	White	Yolk	Whole egg	White	Yolk	Whole egg	
1	0.001	0.001	0.001	0.002	0.000	0.001	0.001
2	0.029	0.003	0.020	0.046	0.006	0.033	0.027
3	0.095	0.248	0.148	0.069	0.265	0.134	0.141
4	0.098	0.579	0.299	0.100	0.657	0.283	0.291
5	0.098	1.035	0.420	0.086	1.014	0.384	0.402

[a] Average concentration in whole eggs from Group II and Group III

Ethephon and HEPA were both identified in muscle, liver and kidney. Radioactivity in egg white and yolk was mainly incorporated into amino acids (57% TRR) and fatty acids/cholesterol (74–77% TRR), respectively. In organs, radioactivity was also incorporated into amino acids (up to 35% TRR in muscle). In fat, the only characterised fraction was fatty acids/cholesterol (39–44% TRR). The unknown fractions in Group III liver included a metabolite at 0.039 mg/kg, a multi-component peak (with no individual component exceeding 0.033 mg/kg) and a region of unidentified radioactivity (0.023 mg/kg) which could represent polypeptides. The unknowns in Group III kidney included two metabolites at levels of 0.015 and 0.045 mg/kg, as well as a multi-component peak (with no individual component exceeding 0.050 mg/kg) and a region of unidentified radioactivity (0.059 mg/kg) which could represent polypeptides. Unidentified residues in other Group III matrices were below 0.05 mg/kg. Bound residues from Group III samples, which had been subjected to protease hydrolysis in addition to solvent extraction, were all below 0.035 mg/kg (Table).

Table 26 Characterisation and identification of residues in tissues and eggs (Day 4) of laying hens following oral administration of [^{14}C]ethephon for 5 days

	Liver		Kidney		Muscle		Fat		Egg white		Egg yolk	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
59 ppm diet (Group II—expired air cage)												
Extracted	0.64	71	0.69	64	0.045	79	0.087	96	0.094	94	0.96	95
Unextracted residue	0.16	18	0.13	12	0.026	45	0.012	13	0.001	0.5	0.17	16
Total recovered	0.80	89	0.82	75	0.071	124	0.099	108	0.095	95	1.12	111
Ethephon	0.15	17	0.42	38	0.017	29	nd	nd	nd	nd	nd	nd
HEPA	0.11	12	0.10	9	0.013	22	nd	nd	nd	nd	nd	nd
Polypeptides	–	–	–	–	–	–	–	–	0.093	93	–	–
Amino acids	0.17	19	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Fatty acids/ cholesterol	Nd	nd	nd	nd	nd	nd	0.040	44	nd	nd	0.78	77
Glycerol	Nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	0.022	2
Total identified	0.43	48	0.52	48	0.030	52	0.040	44	0.093	93	0.80	79
Unidentified	0.20	23	0.17	16	0.015	26	0.038	42	0.001	1	0.16	16
67 ppm diet (Group III—layer cage)												
Extracted	0.40	64	0.55	78	0.024	47	0.048	93	0.065	65	0.83	82
Protease hydrolysis	0.11	17	0.078	11	0.018	35	nd	nd	0.003	3	0.12	12
Unextracted residue	0.034	5	0.015	2	0.006	12	0.005	10	0.007	7	0.013	1
Total recovered	0.54	86	0.65	91	0.049	96	0.054	105	0.075	75	0.97	96

	Liver		Kidney		Muscle		Fat		Egg white		Egg yolk	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Ethephon	0.11	17	0.30	42	0.006	12	nd	nd	nd	nd	nd	nd
HEPA	0.10	16	0.096	14	0.009	18	nd	nd	nd	nd	nd	nd
Amino acids	0.084	13	0.019	3	0.018	35	nd	nd	0.057	57	0.091	9
Fatty acids/ cholesterol	Nd	nd	nd	nd	nd	nd	0.020	39	nd	nd	0.75	74
Glycerol	Nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	0.033	3
Total identified	0.29	46	0.42	59	0.033	65	0.020	39	0.057	57	0.84	83
Unidentified	0.22	40	0.22	30	0.009	18	0.036	71	0.008	8	0.031	3

nd = Not determined

The radioactivity present in excreta was almost completely extracted with methanol. For the Group III samples which were treated with protease, a further 2.3% TRR was released by protease and 0.6% TRR remained bound. The major radioactive residue in excreta was ethephon, accounting for 83% TRR for the Group II hens and 88% TRR for the Group III hens, and represents the unabsorbed dose. The metabolite (2-hydroxyethyl)phosphonic acid (HEPA) accounted for 4.4–6.5% TRR. In the Group III excreta, an unknown metabolite was detected at 4.6% TRR. No radioactive amino acids, fatty acids/cholesterol or glycerol were detected.

Table 27 Characterisation and identification of residues in excreta

	% TRR in Excreta	
	59 ppm Diet (Group II) (expired air cage)	67 ppm Diet (Group III) (layer cage)
Extractable	92.4	100.5
Protease hydrolysis	Not performed	2.3
Unextracted residue	2.5	0.6
Total recovered	94.9	103.4
Ethephon	83.4	87.8
HEPA	6.5	4.4
Total identified	89.9	92.2
Unidentified	–	4.6

nd = Not determined

The results indicate that ethephon metabolism in laying hens is postulated to involve the direct release of ethylene from parent ethephon, as well as the competitive removal of chlorine to form HEPA, which is further metabolised to release CO_2, and intermediates which can enter biochemical pathways, leading to the biosynthesis of proteins and lipids. The highest residue levels were found in liver, kidney and egg. Ethephon and HEPA were the major components of the residue in liver and kidney, whereas in egg yolk, most of the radioactivity was incorporated into fatty acids and cholesterol.

Proposed metabolic pathway of ethephon in animals

The metabolism of [^{14}C]ethephon was studied in lactating goats and laying hens. Orally administered [^{14}C]ethephon is rapidly eliminated either in the excreta or as [^{14}C]ethylene in expired air. The main route of metabolism is degradation/metabolism to [^{14}C]ethylene, and to a much lesser degree to $^{14}CO_2$.

A similar route of metabolism of ethephon to ethylene is seen in rats, goats and hens. In livestock, radioactivity was found in fat (fatty acids/cholesterol and glycerol), proteins (polypeptides and amino acids) and glycogen, demonstrating that metabolic degradation of ethephon through an acetate-like intermediate in the tricarboxylic acid cycle was occurring. Ethephon and the metabolite HEPA were found only at low levels in tissues. The proposed metabolic pathway of ethephon in animals is presented below.

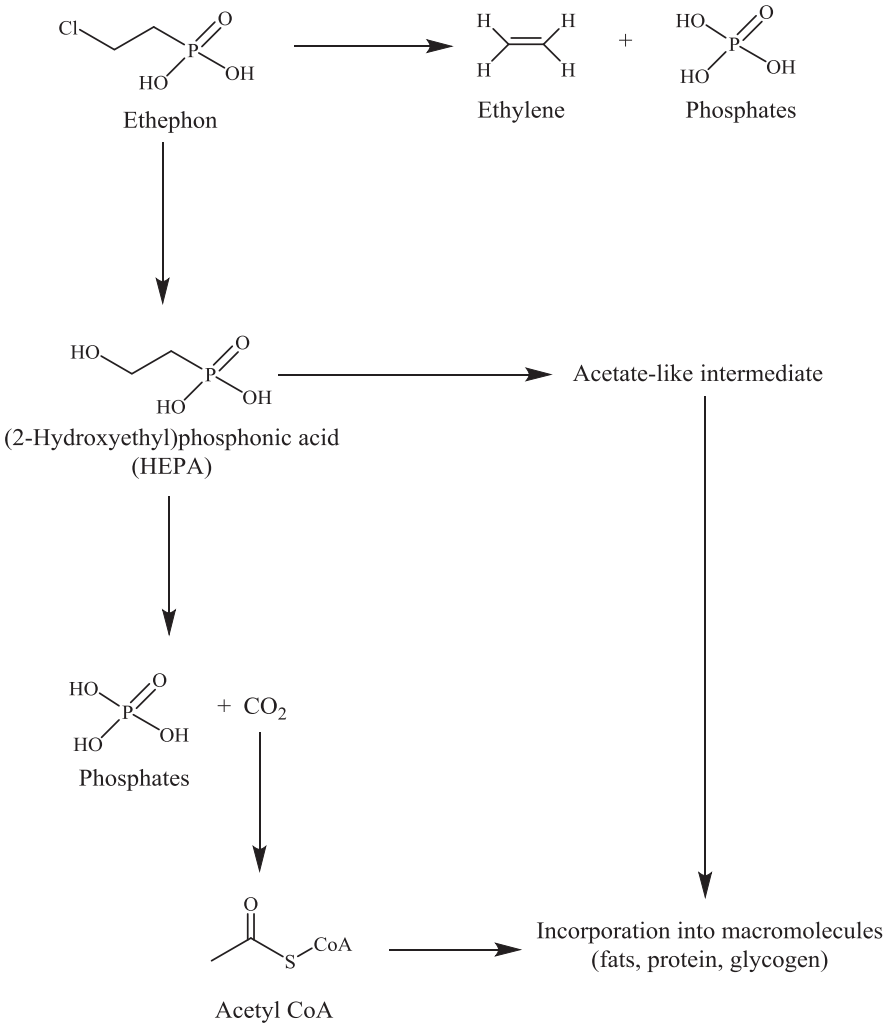


Figure 3 Proposed metabolic pathway for ethephon in animals

Environmental Fate in Soil

The Meeting received information on hydrolysis, photochemical degradation, aerobic and anaerobic degradation of ethephon in soil, photolysis of ethephon on soil, ethephon field dissipation, and residues in rotational crops.

Hydrolysis

The results of the hydrolysis study are summarized in the Physical and Chemical Properties section.

Photochemical degradation

The photolysis of ethephon in water was investigated under artificial sunlight in acetate buffer at pH 5 (Das, 1990, ISSI 89151, Bayer Ref: M-187634-01-1). [^{14}C]Ethephon was mixed with non-radio-labelled ethephon and dissolved in sterile acetate buffer and irradiated continuously using a xenon arc

simulated sunlight source (> 290 nm, 510.5 W/m^2) for up to 360 hours at 25 ± 1 °C; for control, ethephon in the acetate buffer was kept in darkness. Samples were taken at 0, 12, 36, 84, 168, 252 and 360 hours during irradiation. The samples were acidified immediately with HCl solution to prevent breakdown of ethephon during analysis. [^{14}C]ethylene was quantified by flushing the headspace with oxygen, and analysing the gas mixture by sample oxidation/LSC. The identity of ethylene was confirmed by GC-MS analysis of the headspace gases. The test solutions were evaporated to dryness and methylated with diazomethane for GC-MS analysis.

The mean recovery of radioactivity was 96.6% from the irradiated samples and 96.5% from the non-irradiated samples. The pH was confirmed in both the irradiated and non-irradiated samples. Microscopic analysis at 0 and 360 hours confirmed the sterility of the test solutions. GC-MS analysis confirmed the identity of [^{14}C]ethephon and [^{14}C]ethylene as the only major degradate in the test solution. [^{14}C]Ethylene was the only ethephon-related compound in the headspace.

The quantities of radioactive components in irradiated and non-irradiated test solutions of pH 5 buffer treated with [^{14}C]ethephon are presented in Table. In both irradiated and non-irradiated samples, [^{14}C]ethylene was the only major degradate. The calculated DT_{50} was 29.4 days for irradiated samples, and 51.4 days for non-irradiated samples.

Table 28 Recovery of radioactivity as [^{14}C]ethephon and [^{14}C]ethylene in an aqueous photolysis study

Time (hours)	Mean % recovery of total applied radioactivity			
	Irradiated samples (n=2)		Non-irradiated samples (n=2)	
	[^{14}C]ethephon	[^{14}C]ethylene	[^{14}C]ethephon	[^{14}C]ethylene
0	99.9	0.1	99.9	0.1
12	96.2	1.2	96.0	0.5
36	93.4	1.2	94.6	0.5
84	92.7	2.5	91.3	2.4
168	81.4	17.0	85.7	11.0
252	76.0	20.2	84.8	12.0
360	71.2	23.1	81.7	15.2

Aerobic degradation

In the first study, the aerobic degradation of ethephon was investigated in four soils for 180 days (Burr, 2001, C016772, [M-203033-01-1]). [^{14}C]Ethephon was applied to each soil at a rate equivalent to 2.24 kg ai/ha. The soils were incubated aerobically in the dark with 45% maximum water holding capacity at 10 °C or 20 °C under continuous air flow. Three traps containing a saturated solution of pyridinium hydrogen bromide per bromide (PHBPB) were used to collect [^{14}C]ethylene, and a 2 M KOH trap to collect $^{14}CO_2$. Soil samples were taken at 0, 1, 3, 7, 14, 27–28, 56–60, 77–80, 100–102, 120–123, 150–152 and 180 days after treatment. The soil was extracted with phosphoric acid followed by methanol.

The total recovered radioactivity decreased over time during the test (below 70% in the sandy loam, sandy silt loam and clay loam after approximately 102 days). This is probably due to problems with trapping [^{14}C]ethylene, caused by loss of dibromoethane (which is volatile) between trap removal and sampling.

Significant quantities of a volatile metabolite (ethylene) were found in the PHBPB traps, at up to approximately 60% applied radioactivity. Small amounts of (2-hydroxyethyl) phosphonic acid (HEPA) were detected in the soil samples (< 10% applied radioactivity), which is therefore regarded as a minor metabolite (Table 29).

Table 29 Recovery of radioactivity in soil after application of [^{14}C]ethephon

Days after appl.	% of Applied radioactivity						
	Extract 1 Phosphoric acid	% Ethephon in Extract 1	Extract 2 Methanol	Volatiles in PHBPB traps (ethylene)	Volatiles in KOH trap (CO_2)	Unextracted Residue	Total

Days after appl.	% of Applied radioactivity						
	Extract 1 Phosphoric acid	% Ethephon in Extract 1	Extract 2 Methanol	Volatiles in PHBPB traps (ethylene)	Volatiles in KOH trap (CO_2)	Unextracted Residue	Total
	Clay loam soil (00/18), 20 °C. Soil pH 6.9.						
0.02	80.76	80.76	n.a.	n.a.	n.a.	28.93	109.69
1	66.94	66.41	n.a.	0.00	0.00	24.99	92.07
3	66.34	65.15	n.a.	0.00	0.00	33.88	100.54
7	54.51	47.07	2.85	–	–	33.08	90.44
14	49.58	47.17	n.a.	1.61	0.00	46.88	98.06
28	44.34	41.61	n.a.	2.24	0.40	46.18	93.16
56	35.01	30.85	2.09	6.12	0.23	48.74	92.20
80	29.16	24.82	2.50	7.53	0.44	43.77	83.39
100	28.23	23.35	2.88	6.42	0.18	53.40	91.12
123	27.16	21.50	2.30	5.19	0.16	46.77	81.58
152	18.04	14.68	2.05	6.81	0.04	50.64	77.57
180	9.76	4.33	1.87	8.17	0.10	49.71	69.61
	Clay loam soil (00/18), 10 °C. Soil pH 6.9.						
0.02	85.98	83.71	2.34	n.a.	n.a.	11.68	100.0
1	74.77	74.42	0.00	0.02	0.00	19.42	94.21
3	72.38	72.39	0.00	0.00	0.00	27.89	100.72
7	71.25	68.31	2.86	0.00	0.00	26.74	100.85
14	61.78	60.37	0.00	0.13	0.00	26.68	88.59
28	61.31	59.00	0.00	0.18	0.00	34.08	95.57
56	52.99	50.29	2.59	0.61	0.00	40.11	96.30
80	48.96	45.55	3.06	0.86	0.01	51.72	104.60
100	35.08	29.95	4.07	1.44	0.00	60.44	101.03
123	63.33	58.70	4.37	1.32	0.01	23.97	93.00
152	32.01	29.38	3.97	4.35	0.03	43.62	83.98
180	19.12	17.38	4.07	12.59	0.04	34.86	70.68
	Sandy loam soil (00/14), 20 °C. Soil pH 6.8.						
0.02	90.25	90.25	2.15	n.a.	n.a.	8.66	101.06
1	78.06	78.06	3.23	0.27	0.00	18.85	100.41
3	69.26	69.26	3.68	0.71	0.00	21.62	95.27
7	59.86	59.86	3.68	0.60	0.00	29.14	93.28
14	47.50	47.50	3.05	1.51	0.00	32.81	84.87
27	34.50	34.50	2.64	24.80	0.00	28.54	90.48
60	15.00	15.00	1.97	55.98	0.00	13.02	85.97
77	7.14	7.14	1.26	58.09	0.29	11.87	78.65
102	4.87	3.10	0.97	51.27	0.58	11.02	68.70
120	5.31	1.00	1.74	17.33	0.85	13.54	38.76
150	2.48	1.26	0.85	36.44	0.02	12.06	51.85
180	2.21	1.36	0.64	31.90	0.41	10.57	45.73
	Sandy silt loam soil (00/15), 20 °C. Soil pH 5.9.						
0.02	73.06	73.06	1.24	n.a.	n.a.	26.01	100.31
1	65.80	65.80	1.51	0.00	0.00	38.51	105.85
3	63.23	63.23	2.18	0.07	0.00	33.19	98.67
7	54.88	54.88	2.56	0.10	0.00	38.42	95.96
14	50.81	50.45	1.84	2.16	0.00	43.60	98.41
27	44.17	44.17	2.27	8.89	0.00	42.88	98.21
60	42.05	41.27	4.57	15.73	0.68	19.21	82.24
77	38.74	38.15	4.50	11.26	0.12	20.60	75.22
102	33.05	33.05	3.24	22.23	0.41	18.95	77.88
120	20.70	20.70	2.23	13.89	0.30	18.55	55.67
150	19.49	17.22	2.22	8.25	0.36	18.62	48.94
180	14.06	12.42	1.77	22.58	0.30	17.08	55.78
	Clay loam soil (00/16), 20 °C. Soil pH 7.6.						
0.02	82.03	81.32	2.40	n.a.	n.a.	11.10	95.53
1	62.56	61.62	2.30	4.65	0.00	18.08	87.59
3	48.21	47.62	4.63	10.57	0.00	20.02	83.43
7	25.50	24.85	2.52	14.36	0.00	28.79	71.17
14	10.82	10.39	1.16	35.94	0.00	30.09	78.01

Days after appl.	% of Applied radioactivity						
	Extract 1 Phosphoric acid	% Ethephon in Extract 1	Extract 2 Methanol	Volatiles in PHBPB traps (ethylene)	Volatiles in KOH trap (CO_2)	Unextracted Residue	Total
27	1.89	1.18	0.84	51.12	0.00	27.60	81.45
60	0.74	0.50	0.67	53.37	0.00	19.17	73.95
77	0.59	0.36	0.63	43.69	0.00	20.73	65.64
102	0.52		0.54	52.67	0.09	20.14	73.96
120	0.40		0.60	39.74	0.25	23.19	64.18
150	0.33		0.39	62.06	0.32	17.45	80.54
180	0.28		0.46	24.82	0.21	20.35	46.11

n.a. = Not applicable

– = Data not available (traps not aliquotted in error)

The rate of degradation of ethephon under aerobic conditions was also determined. Degradation of ethephon in soil under aerobic conditions depended on the pH of the soil and the temperature, being more rapid at higher pH values and at higher temperatures. The DT_{50} values at 20 °C ranged from 2.7 to 37.6 days. The DT_{50} value at 10 °C (for a clay loam soil) was slower (51.4 days) than the DT_{50} for the same soil at 20 °C (22.2 days). The DT_{50} and DT_{90} values for ethephon in aerobic soils are presented below in Table 30.

Table 30 DT_{50} and DT_{90} values of [^{14}C]ethephon in aerobic soils

Temp.	Clay loam (00/18) Soil pH 7.6.		Sandy loam (00/14) Soil pH 5.9.		Sandy silt loam (00/15) Soil pH 6.8.		Clay loam (00/16) Soil pH 6.9.	
	DT_{50} (days)	DT_{90} (days)	DT_{50} (days)	DT_{90} (days)	DT_{50} (days)	DT_{90} (days)	DT_{50} (days)	DT_{90} (days)
20 °C	22.2	160	14.2	60.7	37.6	173	2.7	12.5
10 °C	51.4	254						

In the second study, the aerobic degradation of ethephon was investigated in a sandy loam soil for 30 days (Das, 1991, ISSI 90031, [M-187639-01-1]). [^{14}C]Ethephon was applied to the soil at a rate of 10.2 µg/g dry weight of soil. The soil was incubated aerobically in the dark with 75% maximum water holding capacity at 25 ± 1 °C under the airtight conditions. Soil samples were taken at 0, 1, 3, 7, 14, 21 and 30 days after treatment.

[^{14}C]Ethylene was quantified by flushing the headspace with oxygen (Headspace 1). The resulting gas mixture was fed through a NaOH solution (to capture $^{14}CO_2$) and then into a biological sample oxidiser. During oxidization, the [^{14}C]ethylene was quantitatively converted to $^{14}CO_2$ which was trapped in a scintillation cocktail. The soil was extracted with methanol and then with 1.0 N NaOH solution to hydrolyse the ethephon to ethylene. Immediately after addition of the NaOH solution, the vessels were sealed and the headspace contents sampled as described previously (Headspace 2). The headspace gases were analysed by GC-MS to confirm the identity of ethylene. Methanol soil extracts were methylated with diazomethane for GC-MS analysis. Alkaline soil extracts were neutralized and cleaned-up and then analysed in the same way as the methanol extracts.

The mean recovery was 97.3% of applied radioactivity. The oxygen content was 8.9 mg/L at the beginning and 8.7 mg/L at the end of the study, confirming aerobic incubation conditions. The results are summarized in Table 31.

Table 31 Recovery of radioactivity in sandy loam soil after application of [^{14}C]ethephon

Days after appl.	% of Applied radioactivity						
	Extract 1 Methanol	Headspace 1	Extract 2 NaOH solution	Headspace 2	Unextracted Residue	Extract 1 + Headspace 2 (sub-total)	Total
	Sandy loam soil, 25 °C. Soil pH 6.1.						
0	97.2	1.2	< 0.1	2.4	0.9	99.6	101.7
1	75.8	1.1	1.4	16.4	2.2	92.2	96.8
3	63.3	3.8	6.5	16.3	8.0	79.6	97.8
7	47.0	11.6	23.2	10.6	6.7	57.6	99.0
14	29.2	11.6	43.6	5.1	8.6	34.3	98.0
21	21.4	15.0	46.0	3.4	10.8	24.8	96.6
30	0.3	8.5	63.5	4.4	14.6	4.7	91.4

GC-MS analysis showed that ethylene was the only compound in all the headspace fractions (Headspace 1 and 2). Ethephon and phosphoric acid were identified in the methanol extract of soil. HEPA was found in large quantities in the alkaline soil extract, but this may be an artefact caused by the alkaline extraction procedure.

The formation and detection of HEPA was investigated in more detail in a separate study (Lowden and Oddy, 2000, 202534, [M-198831-01-1]). Significant loss of radioactivity was found for [^{14}C]ethephon in 0.1 M or 1.0 M NaOH solutions after incubation at room temperature for 2 hours or 2 days. No loss of radioactivity was found in acidic solutions (0.1 M phosphoric acid or 0.1 M acetic acid). In tests to determine the best extraction solvent to use for soil, phosphoric acid gave the highest recoveries. Freeze-drying of the phosphoric acid extract gives a quantitative recovery of applied radioactivity. Methanol gives a poor recovery of radioactivity from soil samples. Extraction of soil with NaOH solution causes ethephon to transform to HEPA.

In the third aerobic degradation study, the degradation of ethephon was investigated in a soil different from those used by Burr for 44 days (Fitzmaurice, 2003, CX/02/32, [M-232779-01-1]). [^{14}C]Ethephon was applied to a clay loam soil at a rate of 2.24 µg/kg. The soil was incubated aerobically in the dark with 45% maximum water holding capacity at 20 °C under continuous air flow. $^{14}CO_2$ was trapped in an individual trap per flask and a merged trap prior to the air passing over a bed of cuprous oxide at 800 °C to convert volatile hydrocarbons to CO_2, which was trapped in two more CO_2 traps. Soil samples were taken at 0, 1, 3, 7, 14, 21, 38 and 44 days after treatment. The soil was extracted with acetonitrile/water (80:20 v/v) followed by 0.1 M phosphoric acid, and then washed with acetone.

Extracts were analysed by LSC, and post-extraction solids by combustion/LSC. The acetonitrile/water and acetone extracts were concentrated by evaporation and analysed by HPLC. Phosphoric acid extracts were concentrated by freeze-drying. Identification was by co-chromatography with ethephon and HEPA reference standards. Phosphoric acid extracts were analysed by LC-MS/MS for confirmation. The unextracted radioactivity was further characterized by fractionation of the soil organic matter. Volatile traps were treated with sodium carbonate chloride and barium chloride to characterize the radioactivity present.

The recovery of radioactivity ranged from 90.1–107.7% of applied radioactivity. Procedural recoveries ranged from 90.2 to 106.8%. The amount of extracted radioactivity decreased over time from an initial 98.8% to 16.3% on Day 44. Unextracted residues gradually increased to a maximum of 34% at Day 21 and were 27% at 44 days. The largest proportion of non-extracted radioactivity (12.6–19%) was associated with the fulvic acid soil fraction. The amount of radioactivity recovered as volatiles increased to 46.9% at Day 44. CO_2 evolution reached a maximum of 22.8% at Day 38. Ethylene accounted for 24.6% at Day 44.

Ethephon degraded from 98.7 to 10.8% of the applied radioactivity after 44 days. Minor amounts of HEPA (up to 1.6%) were detected. The presence of ethephon and HEPA was confirmed in the phosphoric acid extracts of Day 0 and Day 44. Ethylene was found at up to 25.6% of applied radioactivity. CO_2 and unextracted residues accounted for 22 and 27% of the

applied radioactivity after 44 days. Three unidentified polar degradates were found as minor metabolites (< 5%), (Table 32).

Table 32 Recovery of radioactivity in soil after application of [^{14}C]ethephon

Days after appl.	% of Applied radioactivity						
	Extracted residue	Ethylene trap	Unextracted Residue	CO_2	Total (mass balance)	Ethephon	HEPA
	Clay loam soil, 20 °C. Soil pH 7.9.						
0	98.8	n.d.	8.8	0.0	107.7	98.7	n.d.
1	90.3	3.6	13.2	0.01	107.2	90.2	0.10
3	70.6	10.5	19.4	0.7	101.2	69.9	0.11
7	54.6	12.2	25.9	3.2	95.8	52.4	0.98
14	38.8	15.2	32.1	12.4	98.6	36.2	0.05
21	30.4	18.0	34	15.7	98.0	28.2	0.04
38	18.6	22.8	27.1	22.8	91.4	15.0	0.43
44	16.3	25.6	27.0	22.3	90.2	10.8	1.56

The DT_{50} and DT_{90} were 6 days and 63 days, respectively. For the five soils for which the DT_{50} and DT_{90} were calculated above, the mean DT_{50} and DT_{90} values were 16.5 days and 93.8 days, respectively.

Anaerobic Degradation

The anaerobic degradation of ethephon was studied in a flooded clay loamy soil for 30 days (Oddy, 2001, C013378, [M-204496-01-1]). [^{14}C]Ethephon was applied to the soil at a rate equivalent to 2.24 kg ai/ha. The soil was incubated anaerobically in the dark at 20 ± 2 °C. Soil samples were taken at 0, 6 and 12 hours and 1, 2, 4, 7, 14 and 30 days after treatment. Four traps containing saturated solution of pyridinium hydrogen bromide per bromide (PHBPB) were used to collect [^{14}C]ethylene, and the fifth trap contained 2 M KOH to collect $^{14}CO_2$.

The extraction and recovery of radioactivity from anaerobic soil after application of [^{14}C]ethephon is summarized in Table 33. Recoveries were in the range 90–110% of applied radioactivity at all time-points, except at 2 days where the mean recovery was 86%. At the end of the incubation period, 94% of the applied radioactivity was found in the PHBPB traps. This was identified by GC-MS as dibromoethane from the reaction of ethylene with bromine. At 30 days, < 5% of the applied radioactivity was recovered in the water phase, the remaining radioactivity was found in the soil. Small amounts of HEPA (max 3.7% of applied radioactivity after 12 hours) were detected in the water phase. Ethylene was found in the water phase at up to 18.5% of applied radioactivity at 6 days. The amount of ethylene in the water phase declined to 0.4% after 14 days. Two minor metabolites were also detected.

Table 33 Extraction and recovery of radioactivity from an anaerobic soil after application of [^{14}C]ethephon

Time after application	% of Applied radioactivity							
	Water phase	% ethephon in water phase	Soil extract	% ethephon in soil extract	KOH trap (CO_2)	PHBPB trap (ethylene)	Unextracted residue	Total
0 hours	103.88	90.46	0.00	0.00	0.00	0.00	0.00	103.88
6 hours	89.00	66.05	8.14	7.90	0.00	3.09	1.11	101.33
12 hours	85.97	70.13	7.03	6.90	0.00	1.80	1.01	95.81
1 day	66.63	54.01	10.12	9.96	0.01	12.75	1.62	91.13
2 days	48.17	40.02	10.98	10.46	0.03	25.49	1.70	86.36
4 days	21.14	18.56	15.27	13.93	0.04	68.18	3.17	107.76
7 days	13.60	8.94	10.38	8.99	0.05	71.02	2.88	97.88
14 days	5.85	2.24	7.00	6.28	0.00	80.54	2.34	95.72
30 days	2.65	n.a.	2.67	n.a.	0.03	94.06	2.05	101.43

The major compound identified in soil was [^{14}C]ethephon. Five minor metabolites were detected in the soil at levels below 5% applied radioactivity.

The rate of degradation of ethephon under anaerobic conditions was determined. The DT_{50} and DT_{90} values for ethephon in an anaerobic soil system are presented below in Table 34.

Table 34 DT_{50} and DT_{90} values of [^{14}C]ethephon in an anaerobic soil system

Parameter	DT_{50} (days)	DT_{90} (days)
Anaerobic soil system	2.2	8.8

Soil Photolysis

The photolytic degradation of ethephon was studied on a clay loamy soil (Hatcher and Oddy, 2001, 202650, [M-199517-01-1]). [^{14}C]ethephon was applied to 1 cm thick layers of soil at a concentration equivalent to an application rate of 2.24 kg ai/ha. The soil layers was maintained at 45% of maximum water holding capacity under artificial sunlight from a xenon lamp at 20 ± 2 °C. A non-irradiated group was maintained in the dark at 20 ± 2 °C. Moistened CO_2-free air was supplied at a continuous rate, and the effluent air led through a series of four traps. Three traps containing saturated solution of PHBPB were used to collect [^{14}C]ethylene, and the last trap contained 2 M KOH to collect $^{14}CO_2$. Traps were replaced at 6, 12, 16 and 23 days after application. Duplicate soil samples were taken at 0, 1, 2, 5, 10, 21 and 30 days after treatment. Soil was extracted with 0.2 M phosphoric acid solution, the extracts neutralised and analysed directly by HPLC. Quantification of extracts and trapping solutions was done by LSC. Unextracted radioactivity in soil was analysed by combustion LSC.

Total recoveries of radioactivity for irradiated soils were in the range 76–106% of applied radioactivity. After 21 days, the mean recovery decrease to below 90%, probably due to incomplete trapping of ethylene by the PHBPB traps. The extraction and recovery of radioactivity from irradiated soil following application of [^{14}C]ethephon are presented in Table 35. For comparison, the extraction and recovery of radioactivity from non-irradiated soil following application of [^{14}C]ethephon are also presented.

In the irradiated experiment, after 30 days, 45.1% of the applied radioactivity was recovered in the phosphoric acid extract and 20.7% remained unextractable. 12.3% was found in the PHBPB traps and is attributed to ethylene. Small amounts of HEPA were detected in the soil extract, reaching a maximum of 10.6% applied radioactivity after 10 days, and decreasing to 8.7% after 30 days. The main component in soil was [^{14}C]ethephon. Four minor metabolites were detected in soil extracts at levels below 3% applied radioactivity.

At the end of the non-irradiated experiment, 49.4% applied radioactivity was recovered in the phosphoric acid extract and 19.6% remained unextractable. 7.8% was found in the PHBPB traps (ethylene). A further 5.7% was recovered in the KOH trap and characterised by precipitation as $^{14}CO_2$. The main component in soil was [^{14}C]ethephon, and small amounts of HEPA were detected in the extract (5.7% after 30 days). Three minor metabolites were detected in soil extracts at levels below 3% of the applied radioactivity

Table 35 Extraction and recovery of radioactivity from soil following application of [^{14}C]ethephon

Sampling time, days	% of applied radioactivity					
	H_3PO_4 extract	% ethephon in soil extract	KOH trap (CO_2)	PHBPB trap (ethylene)	Unextracted residue	Total
Irradiated						
0	100.52	99.11	0.00	0.00	0.80	101.32
1	96.80	90.62	0.00	0.27	2.85	99.92
2	96.03	87.95	0.00	0.80	3.42	100.25
5	89.91	76.76	0.02	2.85	8.79	101.57
10	72.96	59.08	0.05	4.52	15.12	92.64
21	54.74	43.51	0.99	7.29	18.34	81.35
30	45.14	32.55	0.50	12.25	20.68	78.56
Non-irradiated						

Sampling time, days	% of applied radioactivity					
	H_3PO_4 extract	% ethephon in soil extract	KOH trap (CO_2)	PHBPB trap (ethylene)	Unextracted residue	Total
0	100.52	99.11	0.00	0.00	0.80	101.32
1	95.84	89.90	0.08	1.13	3.22	100.26
2	92.42	86.44	0.00	0.51	4.32	97.26
5	83.29	77.74	1.08	2.91	8.67	95.95
10	71.08	62.99	2.21	4.30	14.87	92.46
21	59.90	51.96	4.97	5.69	16.96	87.51
30	49.40	40.47	5.67	7.78	19.60	82.45

The result indicates that the degradation pathway did not differ between the irradiated and non-irradiated soils. The rate of degradation of ethephon was slightly enhanced by irradiation. The DT_{50} and DT_{90} of [^{14}C]ethephon in non-irradiated and irradiated soil are shown below.

Table 36 DT_{50} and DT_{90} values of [^{14}C]ethephon in irradiated and non-irradiated soil

Parameter	DT_{50} (days)	DT_{90} (days)
Irradiated soil	16.5	57.8
Non-irradiated soil	20.7	74.4

Field Dissipation

The dissipation of ethephon was studied in three soils under field conditions in the USA (Norris, 1991, 41011, [M-187653-01-1]). The study was carried out over a period of four months under the growing conditions of tomatoes, cotton and spring wheat. Sites in the USA (California, North Carolina and Washington) were selected with plot areas of 960–1600 m^2. The field were tilled, the crops were planted and ethephon was applied at each location as follows:

Table 37 Dissipation of ethephon in soils

Trial location	Crop	Formulation	Application rate	Soil characterization (0–15 cm depth)
California	Tomato	SL, 22% ethephon	1.85 kg ai/ha	Loam, pH 7.8, 1.5% OM
North Carolina	Cotton	SL. 55% ethephon	2.25 kg ai/ha	Sand, pH 6.6, 0.7% OM
Washington	Spring wheat	SL, 40% ethephon	1.86 kg ai/ha [a]	Loamy sand, pH 7.1, 1.2% OM

[a] For the Washington site, the actual application rate was 3.3× the nominal rate of 0.56 kg ai/ha due to a calculation error

Crops were grown according to local standard agronomic practices. At the California and Washington trials sites, crops were irrigated in order to maintain a viable crop. Directly after application, 0–15 cm depth soil cores were collected. Soil cores collected at later intervals were segmented into 0–15, 15–30, 30–45, 45–60, 60–75 and 75–90 cm depth increments. After air-drying and sieving (2 mm), endogenous ethylene was removed from the soil samples. The soil was then subjected to alkaline hydrolysis to convert any ethephon present to ethylene, which was measured by GC analysis of the headspace.

The procedural recoveries for ethephon were in the range 63–104% (RSD 13%, n=78). The procedural recoveries were similar for soil from the three sites. In addition to the soil samples, filter paper strips placed in the field during application were analysed for evaluation of the application rate achieved.

Following application, residues of ethephon declined with time. Residues found in soil after application were 0.73–1.2 mg/kg, and assuming a soil density of 1.6 g/cm, fairly well matched the application rate as determined in the field (except for the Washington site). Residues declined to 0.01–0.03 mg/kg within 60–120 days. The majority of the residues were found in the top soil (0–15 cm), except for the Washington trial, which was attributed to excessive irrigation shortly after application, causing ethephon to penetrate into the soil as deep as 45–60 cm.

Dissipation seems to be temperature dependent, i.e. fastest in the south (North Carolina) and slowest in the north (Washington). Dissipation of ethephon in soil follows first order kinetics. The DT_{50} and DT_{90} values for dissipation of ethephon in soil under local field conditions are 6.8–20 days and 22–66 days, respectively (Table 38).

Table 38 DT_{50} and DT_{90} values for three USA soils

Location	Crop	Application rate, kg ai/ha	DT_{50}, days	DT_{90}, days	Function	Regression coefficient
California	Tomato	1.85	12 20	66	1st order linear 1st order non-linear	–0.986
North Carolina	Cotton	2.25	6.8 6.8	22	1st order linear 1st order non-linear	–0.964
Washington	Spring wheat	1.86	25 15	65	1st order linear 1st order non-linear	–0.986

Proposed degradation pathway in soil

Under aerobic, anaerobic and photolytic conditions, the route of degradation was similar with ethylene being formed as the major metabolite. Small amounts of HEPA and CO_2 are formed. The proposed degradation pathway of ethephon in soil is shown below.

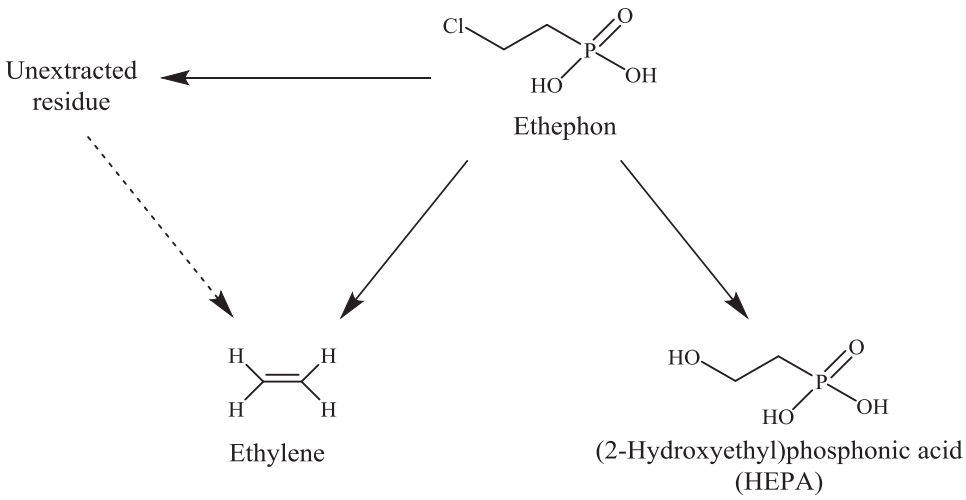


Figure 4 Proposed degradation pathway of ethephon in soil

Residues in Succeeding or Rotational Crops

A confined accumulation study on rotational crops was conducted with [^{14}C]ethephon using wheat, collards and radish (Miller, 1994, EC-91-158, [M-187425-01-1]). The test material was applied to bare plots in plastic containers containing a sandy loam soil, at an application rate of 2.36 kg ai/ha. Crops were planted into the treated soil at plant-back intervals (PBI) of 30, 120 and 379 days after treatment (DAT) after thorough manual mixing of soil (ca. 10 cm). Mature crops were harvested 54–62 days after planting (radishes), 68–91 days after planting (collards) and 110–158 days after planting (wheat). Immature wheat foliage was harvested 47–68 days after planting. Soil samples were collected at each planting and harvest interval. Total radioactive residues (TRR) in crops and soil were determined by combustion LSC.

Crop matrices were homogenized and subsequently extracted with hexane, ethyl acetate and methanol, and then by soxhlet extraction using acidified methanol. The extracts were combined and analysed using HPLC with UV and radiochemical detection. Radioactive components were co-chromatographed with ethephon and HEPA.

The unextracted residue was subjected to a sequential extraction procedure to give water-soluble polysaccharide (potassium phosphate extraction), starch (alpha-amylase digestion), protein (Pronase E digestion), pectin (sodium acetate/ EDTA extraction at pH 4.5), lignin (sodium chlorite digestion at 70 °C), hemi-cellulose (24% potassium hydroxide digestion) and cellulose (72% sulphuric acid digestion at 100 °C) fractions. These fractions were derivatized with phenyl hydrazine and oxidized to $^{14}CO_2$ in order to investigate the incorporation of [^{14}C]ethephon into biomolecules.

TRR in soil at a depth of 0–10 cm following application of [^{14}C]ethephon are summarized in Table 39. At 525 DAT, no detectable radioactive residue was observed in soil at a depth of 20–40 cm, and not more than 0.04 mg/kg was found at 10–20 cm depth.

Table 39 Total radioactive residues in soil at 0–10 cm depth after treatment with [^{14}C]ethephon

DAT	0	30	97	118	120	167	188	230	379	440	470	525
TRR in soil, mg/kg	2.0	1.5	1.2	0.71	0.69	0.73	0.86	0.94	0.33	0.20	0.23	0.22

TRRs in radishes, collards and wheat following application of [^{14}C]ethephon are summarized in Table 40. The highest TRRs were found in the 30 day PBI wheat straw (0.49 mg/kg) and grain (0.35 mg/kg), but in the 379 day PBI samples, 0.03 and 0.02 mg/kg, respectively. Extracted radioactive residues were low (< 50% of TRR) at all time points. Most of the extracted radioactivity from the crop matrices was released by extraction with methanol and the soxhlet extraction with acidified methanol, whereas hardly any radioactivity was extracted with hexane and ethyl acetate. The total extracted residue did not exceed 0.07 mg/kg in any sample analysed. Only the extracts containing residues above 0.01 mg/kg were subjected to HPLC analysis. Low levels of ethephon and HEPA were present in some extracts analysed (30 day PBI radish root and foliage, collard, wheat forage and straw, 120 day PBI radish root and wheat forage, and 379 day PBI wheat grain), and no unidentified metabolites were detected at significant concentrations.

Table 40 Total radioactive residues in rotational crops planted 30, 120 and 379 days after soil application of [^{14}C]ethephon

Crop matrices	Harvest time	TRR	Solvent-extracted radioactive residue [a]	
	DAT	mg/kg	mg/kg	% TRR
30 day PBI				
Radish foliage	98	0.07	0.03	33
Radish roots	98	0.07	0.02	38
Collards	117	0.11	0.03	35
Immature wheat forage	98	0.14	0.05	43
Wheat grain	188	0.35	0.02	8.1
Wheat straw	188	0.49	0.07	15
120 day PBI				
Radish foliage	174	0.07	0.02	29
Radish roots	174	0.06	0.03	49
Collards	188	0.05	0.02	42
Immature wheat forage	167	0.12	0.04	40
Wheat grain	230	0.13	0.02	18
Wheat straw	230	0.19	0.05	24
379 day PBI				
Radish foliage	441	0.01	< 0.01	11
Radish roots	441	0.00	< 0.01	21
Collards	470	0.01	< 0.01	1.4
Immature wheat forage	441	0.01	< 0.01	0.6
Wheat grain	523	0.02	0.01	23
Wheat straw	523	0.03	< 0.01	21

[a] Sum of extractions with hexane, ethyl acetate, methanol and acidified methanol

All crop samples contained radioactive residues in the post-extraction solids. In general, 30 day PBI wheat contained the highest unextracted residues. In these samples, the cellulose fractions from wheat grain and straw were 0.07 mg/kg (20% TRR) and 0.12 mg/kg (25% TRR), respectively. Radioactivity in other biomolecule fractions was found to be lower, (Table 41).

Table 41 Characterization of the unextracted residue by solvents in 30 day and 120 day PBI crop samples

Fraction	30 day PBI Collards		30 day PBI Wheat grain		30 day PBI Wheat straw		30 day PBI Wheat foliage	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Buffer fraction	0.01	6.4	0.02	5.1	0.01	1.5	0.00	3.3
Starch fraction	0.01	11.1	0.03	9.0	0.01	2.5	0.00	3.1
Protein fraction	0.03	27.6	0.05	14.0	0.01	2.0	0.04	30.3
Pectin fraction	0.00	2.8	0.01	2.2	0.01	1.0	0.01	5.9
Lignin fraction	0.00	1.6	0.01	4.2	0.01	1.6	0.01	4.3
Hemi-cellulose fraction	0.01	8.3	0.05	13.9	0.06	12.6	0.02	11.2
Cellulose fraction	0.01	9.5	0.07	20.1	0.12	24.8	0.02	14.7
Filters + ash	0.00	3.9	0.01	4.7	0.15	31.3	0.01	11.1
Solvent extracts	0.03	36.1	0.02	7.3	0.07	13.7	0.05	37.3
Total recovery	0.10	107.2	0.27	80.5	0.45	91.0	0.16	121.1
TRR	0.11		0.35		0.49		0.14	
Fraction	120 day PBI Radish tops		120 day PBI Wheat grain		120 day PBI Wheat straw		120 day PBI Wheat foliage	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Buffer fraction	0.01	18.9	0.00	2.5	0.00	2.2	0.00	3.6
Starch fraction	0.00	6.2	0.01	10.5	0.00	1.1	0.00	3.3
Protein fraction	0.03	34.2	0.02	16.0	0.01	2.8	0.02	15.6
Pectin fraction	0.01	14.0	0.01	4.1	0.00	2.2	0.00	4.0
Lignin fraction	0.01	7.9	0.01	4.2	0.00	1.4	0.00	3.1
Hemi-cellulose fraction	0.01	8.2	0.01	8.9	0.02	12.7	0.01	10.3
Cellulose fraction	0.01	15.7	0.02	17.4	0.04	22.7	0.01	9.7
Filters + ash	0.01	11.6	0.00	3.8	0.04	21.4	0.01	6.0
Solvent extracts	0.02	36.0	0.02	15.3	0.05	23.8	0.04	34.5
Total recovery	0.11	152.8	0.10	82.6	0.16	90.4	0.09	90.2
TRR	0.07		0.13		0.19		0.12	

Overall, [^{14}C]ethephon residues declined steadily in soil. Radioactivity in mature plant samples paralleled or decreased at an even faster rate compared to the soil levels. In plant extracts, no radioactive peaks greater than 0.01 mg/kg were detected. Very low levels of ethephon and HEPA were detected in radishes, collards and wheat. Most of the radioactivity in the crop samples was attributable to incorporation into natural plant constituents.

The metabolism in rotational crops is similar to that seen in primary crops, with degradation to HEPA and natural incorporation into biomolecules.

Residue analytical methods

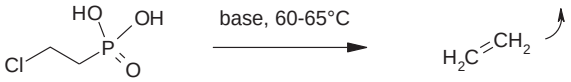
The Meeting received information on analytical methods together with validation data for residues of ethephon in plant, and animal matrices.

The analytical methods presented in this section are based on three different principles:

Ethylene-release method

This method was widely used in studies performed in the USA, and involves base hydrolysis of the residue to ethylene, with measurement of the released ethylene by GC-FID. The samples are first heated in a solution of tartaric acid in order to remove the endogenous ethylene. Thereafter the solution is made basic and heated again in capped bottles which allow headspace samples to be collected. By this procedure the ethephon present in the samples decomposes to ethylene, which is

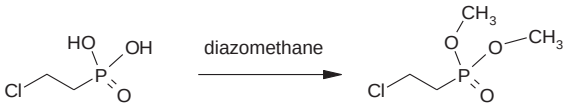
determined by headspace GC/FID. The released ethylene allows the ethephon residues in the sample to be quantified. The LOQ is typically 0.02–0.10 mg/kg.



ethephon **ethylene**

Derivatisation to methyl ester method

This method was used in the earlier studies performed in Europe and involves extraction with methanol and derivatisation of the ethephon residue with diazomethane to give the methyl ester, with measurement by GC-NPD or GC-FPD in phosphorus mode. The LOQ is typically 0.05–0.20 mg/kg.



ethephon **ethephon dimethyl ester**

LC-MS/MS method

This is a highly specific method which has been used in the more recent studies and involves extraction of the ethephon residue, sample clean-up and measurement by LC-MS/MS. The LOQ is typically 0.05 mg/kg.

Detailed descriptions of all these analytical methods are presented below. Validation data for methods on plant and animal matrices are summarized in Table 42.

Analytical Methods for Determination of Ethephon Residues

Analytical methods for plant matrices

Method: 11-94 (Ethylene release method) (Nygren, 1994, 11-94, [M-188198-01-1])

Analyte:	Ethephon	GC-FID	
LOQ:	0.10 mg/kg in fig, 0.02 in pineapple, 0.07 mg/kg in cotton seed,		
Description:	The ground sample is placed in a 250 mL pressure bottle with a crown cap that has a provision for withdrawing a headspace sample with a syringe. An aqueous tartaric acid-surfactant solution is added. The bottle is then capped, heated to about 60 °C and periodically agitated to drive any endogenous ethylene from the sample. After one hour heating, the cap is removed and the bottle is flushed with a gentle stream of nitrogen to remove any released ethylene. The sample is allowed to cool to room temperature. Trisodium phosphate, sufficient to make the sample basic, is added. The bottle is immediately capped and heated for one hour with periodic agitation to convert any ethephon residues into ethylene. The ethylene accumulates in the headspace and is quantified by gas chromatography with flame ionisation detection.		

Method: Union Carbide, 1981 (Diazomethane method) (Conn, 1992, SARS-89-24, [M-187553-01-1])

Method title “Detailed Method of Analysis for Residues of (2-Chloroethyl)Phosphonic Acid (Ethephon) in Wheat and Barley Grain, Straw and Milling Fractions”			
Analyte:	Ethephon	GC-FPD	
LOQ:	0.05 mg/kg in wheat grain and straw		

Description:	This method is the predecessor of SOP 90074 Samples are hard-frozen, ground and freeze-dried. Grain samples are soxhlet extracted with methanol for 4 hours. Straw samples are soxhlet extracted with 1% citric acid in methanol for 4 hours. The pH is adjusted by the addition of 10% methanolic hydrochloric acid. An aliquot of the extract is concentrated, 10% methanolic HCl added and solid materials precipitated by the addition of diethyl ether. After centrifugation and concentration of the liquid extract to ca. 1 mL, the ethephon residues are methylated with diazomethane. Straw samples are subjected to an additional clean-up step using a florisil column. The thus formed ethephon dimethyl ester is measured by means of gas chromatography with flame photometric detection.

Method: SOP–90070 (Diazomethane method) (Nygren, 1990, SOP–90070, [M-163159-01-1])

Analyte:	Ethephon	GC-FPD or GC-NPD	
LOQ:	0.05 mg/kg in wheat grain and straw, 0.02 in tomato		
Description:	This method is essentially the same as Method SOP 90074. Samples are hard-frozen, ground (in case of solid matrices) and freeze-dried. Samples are soxhlet extracted with methanol for 4 hours. The pH is adjusted by the addition of 10% methanolic hydrochloric acid. An aliquot of the extract is concentrated, 10% methanolic HCl added and solid materials precipitated by the addition of diethyl ether. After centrifugation and concentration of the liquid extract to ca. 1 mL, the ethephon residues are methylated with diazomethane. The thus formed ethephon dimethyl ester is measured by means of gas chromatography with either flame photometric detection in the phosphorous mode or with nitrogen phosphorus detection. Minor adjustments of the general procedure may be necessary to adapt for individual crops.		

Method: SOP 90069 (Diazomethane method) (Nygren, 1991, 89-REN-WA-S, [M-187529-01-1])

Analyte:	Ethephon	GC-NPD	
LOQ:	0.01 mg/kg in macadamia nuts		
Description:	Samples are hard-frozen, ground and freeze-dried. Samples are soxhlet extracted with methanol for 4 hours. The pH of the extract is adjusted by the addition of 10% methanolic hydrochloric acid. The acidified extract is frozen overnight to solidify the extracted lipid material. The methanolic solution is separated from the lipid material, concentrated and the solid materials in the extract precipitated by the addition of diethyl ether and separated by centrifugation. The resulting extract is concentrated and residues of ethephon methylated with diazomethane. The ethephon dimethyl ester is analysed by gas chromatography with nitrogen phosphorus detection.		

Method: SOP 90074 (Diazomethane method) (Eckert, 1992, Report: RP-01-89I, [M-187521-01-1])

Analyte:	Ethephon	GC-FPD	
LOQ:	0.05 mg/kg in wheat grain and straw		
Description:	Samples are hard-frozen, ground and freeze-dried. Grain samples are soxhlet extracted with methanol for 4 hours. Straw samples are soxhlet extracted with 1% citric acid in methanol for 4 hours. The pH is adjusted by the addition of 10% methanolic hydrochloric acid. An aliquot of the extract is concentrated, 10% methanolic HCl added and solid materials precipitated by the addition of diethyl ether. After centrifugation and concentration of the liquid extract to ca. 1 mL, the ethephon residues are methylated with diazomethane. Straw samples are subjected to an additional clean-up step using a florisil column. The thus formed ethephon dimethyl ester is measured by means of gas chromatography with flame photometric detection in phosphorus mode.		

Method: SOP 90075 (Diazomethane method) (Eckert, 1992, RP-01-89J, [M-187525-01-1])

Analyte:	Ethephon	GC-FPD	
LOQ:	0.05 mg/kg in cotton seed		

Description:	Cotton seed, hulls and meal: Samples are hard-frozen, ground and freeze-dried. Samples are soxhlet extracted with methanol for 4 hours. The pH of the extract is adjusted by the addition of 10% methanolic hydrochloric acid. An aliquot of the extract is concentrated, 10% methanolic HCl added and solid materials precipitated by the addition of diethyl ether. After centrifugation and concentration of the liquid extract to ca. 1 mL, the ethephon residues are methylated with diazomethane. The resulting ethephon dimethyl ester is analysed by gas chromatography with flame photometric detection in phosphorus mode or alkali flame thermionic detection. Cottonseed oil and soapstock: Samples are extracted by vortex mixing with 1% methanolic citric acid for 1 minute. After centrifugation, the upper methanol phase is removed and reserved, and the extraction procedure repeated a further two times. The combined methanol extracts are concentrated and solid materials precipitated by the addition of diethyl ether. After centrifugation and concentration of the liquid extract to ca. 1 mL, the ethephon residues are methylated with diazomethane. The resulting ethephon dimethyl ester is analysed by gas chromatography with flame photometric detection in phosphorus mode or alkali flame thermionic detection.

Method: Analytical methods for pesticide residues in foodstuffs, Sixth edition, June 1996: Ethephon (Diazomethane method) [M-208923-01-1])

Analyte:	Ethephon	GC-FPD	
LOQ:	0.1 mg/kg		
Description:	This is an official method for determination of ethephon in foodstuffs of plant origin, which has been published by the Dutch General Inspectorate for Health Protection. This method is similar to method SOP 90070 except that ethyl acetate is used as an extraction solvent instead of methanol. The ground samples (50 g) are extracted with ethyl acetate (400 mL) in presence of sulphuric acid, magnesium sulphate and sodium sulphate. An aliquot of the extract (100 mL) is methylated with diazomethane and then concentrated on a rotary evaporator. The ethephon dimethyl ester present in the final extract is measured by means of gas chromatography with flame photometric detection in the phosphorus mode. If necessary the final extract can first be cleaned up by treatment with charcoal.		

Method: HVA 12/89 (Diazomethane method) (Maestracci, 1998, R&D/CRLD/AN/msa/9816152, [M-165702-02-1])

Analyte:	Ethephon	GC-FPD	
LOQ:	0.10 mg/kg in pineapple (skin and flesh), cotton (seed and lint)		
Description:	Samples are extracted by homogenization with methanol, filtered, and the extraction repeated. The combined extract is concentrated and made up to a known volume with methanol. An aliquot of the extract is diluted with diethyl ether and acidified with acetic acid. The ethephon is methylated with diazomethane and residues determined by gas chromatography with flame photometric detection in the phosphorus mode. Quantification is done by external standardisation.		

Method: HVA SOP 10071 (Diazomethane method) (Fuchsbichler, 2002, HVA SOP 10071, [M-210331-01-1])

Analyte:	Ethephon and HEPA	GC-FPD	
LOQ:	0.05 mg/kg		
Description:	Samples are extracted by homogenization with methanol, filtered, concentrated and made up to a known volume with methanol. An aliquot of the extract is liquid/liquid partitioned into diethyl ether, the diethyl ether dried with sodium sulphate and evaporated to 1–2 ml. Ethephon and HEPA are methylated with diazomethane and residues determined by gas chromatography with flame photometric detection in the phosphorus mode. Quantification is done by external standardisation. For sweet pepper, an additional clean up on silica gel is necessary prior to GC-determination.		

Method: V5229/01 (LC-MS/MS method) (Kerkdijk, 1994, V5229/01, [M-226290-01-1])

Analyte:	Ethephon and HEPA	LC-MS/MS	
LOQ:	0.05 mg/kg in apple, cherry and sweet pepper		
Description:	Samples are extracted by high speed blending with demineralized water. The extract is centrifuged and filtered to give a clear supernatant. The pH of the supernatant is adjusted to pH 4–5 using 1 N formic acid solution. A further clean-up is performed by solid phase extraction (SPE) using SDB1 columns. The resulting eluate is analysed by liquid chromatography with tandem mass spectrometric detection (LC/MS/MS) in the negative electrospray mode. Ethephon is monitored by means of the MS/MS transition at m/z 142.9 → 107.0 (^{35}Cl isotope) and HEPA at m/z 124.9 → 94.9.		

Method: 00902 (LC-MS/MS method) (Oel & Bardel, 2005, MR-128/04, [M-247578-01-1])(Independent-laboratory-validated)

Analyte:	Ethephon	LC-MS/MS	
LOQ:	0.05 mg/kg in tomato, wheat grain, orange, olive		
Description:	Residues of ethephon are extracted from plant material by high speed blending with methanol/water/formic acid (90/10/0.1, v/v/v). For dry matrices (e. g. cereal grain) the sample must be soaked prior to blending and some cysteine hydrochloride is added to extraction solvent. For dry matrices it is also possible to use microwave extraction instead of high speed blending. After concentration to dryness the extract is reconstituted in water/methanol/formic acid (80/20/0.5, v/v/v). The reconstituted extract is analysed by liquid chromatography with tandem mass spectrometric detection (LC/MS/MS) using a triple-quadrupole apparatus that is operated in the negative electrospray mode. Ethephon is monitored by means of the MS/MS transitions m/z 143 → 107 (^{35}Cl isotope) and/or m/z 145 → 107 (^{37}Cl isotope). Satisfactory chromatographic separation is achieved on a C_{18} column with polar embedding (Synergi Fusion-RP 80Å, 150×4.6 mm, 4 µm). Elution is performed using water/methanol (80/20, v/v) acidified with 0.5% formic acid as the mobile phase.		

Method: 00918 (LC-MS/MS method) (Oel & Bardel, 2005, MR-173/04, [M-248933-01-1])

Analyte:	Ethephon and HEPA	LC-MS/MS	
LOQ:	0.05 mg/kg in cereal green material, grain and straw		
Description:	The residues of ethephon and HEPA are extracted from cereal green material, straw, and grain by high speed blending with methanol/water/formic acid (50/50/0.1, v/v/v). Samples of straw and grain must be soaked prior to blending. Alternately it is possible to extract residues from cereal grain by microwave extraction using the same solvent mixture. The raw extracts are cleaned-up on an SPE Bond Elut ENV cartridge. For determination of ethephon and HEPA an aliquot of the eluate is concentrated to dryness and reconstituted in 0.01% formic acid. The final extracts are measured by liquid chromatography with tandem mass spectrometric detection (LC/MS/MS) using a triple-quadrupole apparatus that is operated in the negative electrospray mode. Satisfactory chromatographic separation is achieved an ion chromatography column (Metrosep A Supp 4) with an aqueous solution of ammonium carbonate (15 mmol/L) as mobile phase. Ethephon is monitored by means of the MS/MS transition m/z 143 → 107, while HEPA is monitored by means of the MS/MS transition m/z 125 → 95.		

Method: 00903 and 00903/E001 (LC-MS/MS method) (Oel & Bardel, 2005, MR-131/04, [M-254165-01-1])

Analyte:	Ethephon and HEPA	LC-MS/MS	
LOQ:	0.05 mg/kg in grapes, apple, tomato, olives and processed fractions		
Description:	Apple and grape samples are extracted by soaking and then high speed blending with 0.01% formic acid. The extract is filtered under vacuum to give a clear supernatant. Tomato matrices are extracted by soaking and then high speed blending with 0.01% formic acid. Celite is added to the extract, which is then filtered under vacuum to give a clear supernatant. The filtered extract is cleaned-up by solid phase extraction (SPE) using Bond Elut ENV columns. The resulting eluate is filtered and analysed by liquid chromatography with tandem mass spectrometric detection (LC/MS/MS) in the negative ion mode. Ethephon is monitored by means of the MS/MS transition at m/z 143 → 107 (^{35}Cl isotope) and HEPA at m/z 125 → 95. The method can be performed using either internal or external standards.		

Method: 01429 (LC-MS/MS method)(Schulte and Sruskus, 2015, MR-14/100)

Analyte	Ethephon and HEPA	LC-MS/MS	
LOQ:	For both compounds: 0.01 mg/kg in cereal grains and 0.05 mg/kg in cereal green material and straw		
Description:	The residues were extracted from cereal green material by blending two times with methanol. For cereal straw and grain the residues were extracted by blending three times with methanol followed by hydrolysis/extraction with a mixture of hydrochloric acid (32%)/water (1/7, v/v) at 50 °C overnight. After addition of isotopically labeled internal standards the extracts were analysed by HPLC-MS/MS using a cation exchange column (e.g. Luna SCX 5 µm, 150 × 2 mm) in the HILIC (Hydrophilic Interaction Liquid Chromatography) mode. The mass spectrometer was operated in the negative ionization mode using the mass transitions m/z 142.9 → 106.8 for the quantitation of ethephon and m/z 125.0 → 94.8 for the quantitation of the metabolite HEPA.		

Method Validation

Validation data of the methods used for determining ethephon in plant and animal commodities from related studies are summarized below.

Table 42 Summary of Method Validation

Matrix	Analyte	Fortification, mg/kg	n	Recovery (%)		% RSD	Method	Reference
				Range	Mean			
Plant commodities—ethylene release method								
Barley grain	Ethephon	0.16 0.8 2	4 4 4	88–97 92–100 95–102	94 96 98	4.8 3.5 3.4	11-94 (EC-92-228)	Nygren, 1993, EC-92-228, [M-179285-01-1]
Wheat grain	Ethephon	0.16 0.8 2	8 8 8	86–98 97–102 97–104	92 99 101	4.6 1.9 2.2	11-94 (EC-92-228)	Nygren, 1993, EC-92-228, [M-179285-01-1]
Barley straw	Ethephon	0.82 4.1 10	8 8 8	87–106 83–105 93–105	94 98 97	6.5 7.2 3.8	11-94 (EC-92-228)	Nygren, 1993, EC-92-228, [M-179285-01-1]
Wheat straw	Ethephon	0.82 4.1 10	4 4 4	113–130 99–106 94–101	122 102 98	6.0 2.9 3.6	11-94 (EC-92-228)	Nygren, 1993, EC-92-228, [M-179285-01-1]
Apples	Ethephon	0.02-1 0.05 5	4 4 4	94–102 93–102 94–105	97 97 99	3.6 4.4 5.2	11-94 (EC-92-228)	Nygren, 1993, EC-92-228, [M-179285-01-1]
Tomatoes	Ethephon	0.02 0.1 2	4 4 4	101–107 98–102 98–100	104 100 100	2.4 1.7 1.0	11-94 (EC-92-228)	Nygren, 1993, EC-92-228, [M-179285-01-1]
Grapes	Ethephon	0.02 0.1 2	8 8 8	89–95 99–106 92–104	91 102 99	2.0 2.5 3.5	11-94 (EC-92-228)	Nygren, 1993, EC-92-228, [M-179285-01-1]
Cherries	Ethephon	0.02 0.1 10	8 8 8	74–92 80–103 85–101	82 94 96	8.3 7.2 6.5	11-94 (EC-92-228)	Nygren, 1993, EC-92-228, [M-179285-01-1]
Pineapple	Ethephon	0.02 0.1 2	8 8 8	89–108 89–112 100–120	99 105 105	6.4 3.5 6.3	11-94 (EC-92-228)	Nygren, 1993, EC-92-228, [M-179285-01-1]
Cotton seed	Ethephon	0.07 0.35 2	4 4 4	82–92 94–97 87–95	87 95 90	5.1 1.6 3.8	11-94 (EC-92-228)	Nygren, 1993, EC-92-228, [M-179285-01-1]
Plant commodities—diazomethane method								
Wheat grain (6% FFAP column packing)	Ethephon	0.05 0.25 0.5	2 2 2	85–120 86–98 98–110	103 92 104		Union Carbide, 1981	Conn, 1992, SARS-90-24P, [M-187550-01-1]
Wheat straw (6% FFAP column packing)	Ethephon	0.05 0.25 0.5	2 2 2	100 79–94 100–108	100 87 104		Union Carbide, 1981	Conn, 1992, SARS-90-24P, [M-187550-01-1]
Wheat grain (20% OV-11 column packing)	Ethephon	0.05	1	78			Union Carbide, 1981	Conn, 1992, SARS-90-24P, [M-187550-01-1]
Wheat straw (20% OV-11 column packing)	Ethephon	0.05 0.1	1 1	108 79			Union Carbide, 1981	Conn, 1992, SARS-90-24P, [M-187550-01-1]
Wheat grain	Ethephon	0.05 0.2 0.5	6 6 6	72–97 70–92 81–01	86 83 87	9.7 10.5 8.6	SOP 90074	Eckert, 1992, RP-01-89I, [M-187521-01-1]
Wheat straw	Ethephon	0.05 0.2 2	6 6 6	87–111 71–112 85–106	96 96 96	9.2 15.1 9.0	SOP 90074	Eckert, 1992, RP-01-89H, [M-187519-01-1]
Apple	Ethephon	0.05 0.2 1	6 6 6	76–94 69–106 84–108	86 84 91	8.4 15.6 9.9	SOP–90070	Eckert, 1992, RP-01-89C, [M-187515-01-1]

Matrix	Analyte	Fortification, mg/kg	n	Recovery (%)		% RSD	Method	Reference
				Range	Mean			
Tomato	Ethephon	0.05 0.2 0.5	6 6 6	75–89 72–93 83–100	82 83 88	6.6 8.8 6.8	SOP–90070	Eckert, 1992, RP-01-89A, [M-187533-01-1]
Grapes	Ethephon	0.05 0.2 0.5	6 6 6	69–79 81–96 79–101	72 87 87	7.3 6.2 9.1	SOP–90070	Eckert, 1992, RP-01-89D, [M-187544-01-1]
Blackberry	Ethephon	0.05 0.2 1	6 6 6	85–112 78–105 82–92	94 88 88	10.8 11.0 4.4	SOP–90070	Eckert, 1992, RP-01-89B, [M-187511-01-1]
Pineapple fruit	Ethephon	0.05 0.2 0.5	6 6 6	77–118 88–96 77–94	93 92 88	15.8 3.8 7.0	SOP–90070	Eckert, 1992, RP-01-89E, [M-187540-01-1]
Pineapple forage	Ethephon	0.05 0.2 0.5	6 6 6	70–85 79–86 79–92	77 82 85	7.7 3.2 5.2	SOP–90070	Eckert, 1992, RP-01-89F, [M-187538-01-1]
Tomato dry pomace	Ethephon	0.02 0.2 2	3 3 3	86–104 90–104 70–120	96 96 93	9.5 7.7 27.1	SOP–90070	Nygren, 1991, USA89E30, [M-187599-01-1]
Tomato canned fresh juice	Ethephon	0.02 0.2 2	3 2 3	88–122 88–94 70–91	109 91 81	16.7 4.7 13.0	SOP–90070	Nygren, 1991, USA89E30, [M-187599-01-1]
Tomatoes	Ethephon	0.02 0.2 2	3 3 3	72–88 76–90 77–100	77 82 90	11.9 8.8 13.2	SOP–90070	Nygren, 1991, USA89E16, [M-187596-01-1]
Tomatoes	Ethephon	0.01 0.2 2	3 3 3	72–76 80–85 100–105	74 82 102	2 3 3	SOP–90070	Dorschner, 2008, 00250, [M-301374-01-1]
Apples	Ethephon	0.05 0.2 2	3 3 3	82–110 82–105 99–108	95 97 104	14.7 13.6 4.4	SOP–90070	Nygren, 1990, USA89E32, [M-187583-01-1]
Apple dry pomace	Ethephon	0.05 0.2 2	3 3 3	80–97 84–98 92–108	86 91 103	10.8 7.7 9.0	SOP–90070	Nygren, 1990, USA89E32, [M-187583-01-1]
Apple juice	Ethephon	0.05 0.2 2	3 3 3	79–98 102–108 88–104	91 105 95	11.5 2.9 8.6	SOP–90070	Nygren, 1990, USA89E32, [M-187583-01-1]
Grapes	Ethephon	0.1 0.2 0.4 0.5 2	3 1 1 1 1	73–93 75 70 78 85	83 75 70 78 85	12.1 – – – –	Similar to SOP – 90070 Based on ‘Analytical methods for pesticide residues in foodstuffs’, 5th edition, 1988	Grolleau, 1997, EA950185, [M-188232-01-1]
Barley plant	Ethephon	0.05 0.5 10	8 8 7	71–93 74–108 75–104	86 89 88	9.1 12.2 9.8	HVA SOP 10071	Fuchsbichler, 2002, HVA SOP 10071, [M-210331-01-1]
Barley grain	Ethephon	0.05 0.5	7 7	69–97 69–98	85 84	11.1 11.9	HVA SOP 10071	Fuchsbichler, 2002, HVA SOP 10071, [M-210331-01-1]
Barley straw	Ethephon	0.05 0.5	8 9	67–104 81–104	89 89	15.4 10.4	HVA SOP 10071	Fuchsbichler, 2002, HVA SOP 10071, [M-210331-01-1]
Wheat plant	Ethephon	0.05 0.5 10	3 3 3	80–96 86–91 70–104	88 89 88	7.4 2.6 15.9	HVA SOP 10071	Fuchsbichler, 2002, HVA SOP 10071, [M-210331-01-1]
Wheat grain	Ethephon	0.05 0.5	4 4	77–93 78–110	83 90	7.7 13.6	HVA SOP 10071	Fuchsbichler, 2002, HVA SOP 10071, [M-210331-01-1]

Matrix	Analyte	Fortification, mg/kg	n	Recovery (%)		% RSD	Method	Reference
				Range	Mean			
Wheat straw	Ethephon	0.05 0.5	5 4	78–92 82–93	82 87	6.9 4.6	HVA SOP 10071	Fuchsbichler, 2002, HVA SOP 10071, [M-210331-01-1]
Apple	Ethephon	0.05 0.5 1	10 9 2	74–112 81–108 95–99	92 94 97	12.6 8.0 –	HVA SOP 10071	Fuchsbichler, 2002, HVA SOP 10071, [M-210331-01-1]
Cherry	Ethephon	0.05 0.5 1 3	9 9 1 5	69–95 70–104 94 80–94	84 90 94 89	11.8 12.1 – 5.5	HVA SOP 10071	Fuchsbichler, 2002, HVA SOP 10071, [M-210331-01-1]
Tomato	Ethephon	0.05 0.5 1 2	12 11 1 1	73–112 76–104 88 108	85 89 88 108	12.5 8.8 – –	HVA SOP 10071	Fuchsbichler, 2002, HVA SOP 10071, [M-210331-01-1]
Sweet peppers	Ethephon	0.05 0.5 1 3	6 6 1 1	85–98 83–104 82 96	92 94 82 96	5.7 8.7 – –	HVA SOP 10071	Fuchsbichler, 2002, HVA SOP 10071, [M-210331-01-1]
Pineapple skin	Ethephon	0.1 0.2 0.5	1 1 1	82 114 76	82 114 76	– – –	HVA 12/89	Maestracci, 1998, R&D/CRLD/AN/msa/9816152, [M-165702-02-1
Pineapple flesh	Ethephon	0.1 0.2 0.5	1 1 1	89 76 82	89 76 82	– – –	HVA 12/89	Maestracci, 1998, R&D/CRLD/AN/msa/9816152, [M-165702-02-1
Cotton seed	Ethephon	0.1	3	80–82	81	1.2	HVA 12/89	Richard & Muller, 1995, R&D/CRLD/AN/bd/9515891, [M-163122-01-1]
Cotton lint	Ethephon	0.1 2 20	2 1 1	78–93 88 70	86 88 70	– – –	HVA 12/89	Richard & Muller, 1995, R&D/CRLD/AN/bd/9515911, [M-163133-01-1]
Cotton seed	Ethephon	0.1 0.5	2 1	111–115 69	113 69	– –	HVA 12/89	Richard & Muller, 1995, R&D/CRLD/AN/bd/9515911, [M-163133-01-1]
Cotton lint	Ethephon	0.1 0.5 2	1 1 1	86 89 74	86 89 74	– – –	HVA 12/89	Muller, 1996, R&D/CRLD/AN/bd/9516706, [M-163236-01-1]
Cotton seed	Ethephon	0.1 0.5	1 1	115 75	75 115	– –	HVA 12/89	Muller, 1996, R&D/CRLD/AN/bd/9516706, [M-163236-01-1]
Cotton seed	Ethephon	0.1 0.5 3	1 1 1	98 85 73	98 85 73	– – –	HVA 12/89	Muller, 1996, R&D/CRLD/AN/vg/9516705, [M-163240-01-1]
Walnut nutmeat	Ethephon	0.2	11	67–112	80	17.0	SOP 90069	Nygren, 1991, 89-REN-WA-S, [M-187529-01-1]
Cotton seed	Ethephon	0.05 0.2 2	6 6 6	63–138 77–98 74–87	93 90 80	28.5 8.4 6.5	SOP 90075	Eckert, 1992, RP-01-89J, [M-187525-01-1]
Plant commodities—LC/MS/MS method								

Matrix	Analyte	Fortification, mg/kg	n	Recovery (%)		% RSD	Method	Reference
				Range	Mean			
Apple	Ethephon	0.05 0.5	5 5	92–108 86–93	99 90	6.4 3.1	V5229/01	Kerkdijk, 1994, V5229/01, [M-226290-01-1]
Cherry	Ethephon	0.05 0.5	5 5	92–98 92–102	95 97	2.5 4.3	V5229/01	Kerkdijk, 1994, V5229/01, [M-226290-01-1]
Sweet peppers	Ethephon	0.05 0.5	5 5	103–109 75–91	107 88	2.5 8.6	V5229/01	Kerkdijk, 1994, V5229/01, [M-226290-01-1]
Tomato	Ethephon m/z 143 → 107	0.05 0.5	5 5	98–103 99–103	101 102	1.9 1.8	00902	Oel & Bardel, 2005, MR-128/04, [M-247578-01-1]
Tomato	Ethephon m/z 145 → 107	0.05 0.5	5 5	98–103 97–101	100 99	1.9 1.5	00902	Oel & Bardel, 2005, MR-128/04, [M-247578-01-1]
Wheat grain (conventional extraction)	Ethephon m/z 143 → 107	0.05 0.5	5 5	86–94 90–96	89 92	3.8 2.5	00902	Oel & Bardel, 2005, MR-128/04, [M-247578-01-1]
Wheat grain (conventional extraction)	Ethephon m/z 145 → 107	0.05 0.5	5 5	85–90 84–91	87 86	2.4 3.6	00902	Oel & Bardel, 2005, MR-128/04, [M-247578-01-1]
Wheat grain (microwave extraction)	Ethephon m/z 143 → 107	0.05 0.5	5 5	93–99 92–100	96 95	2.5 3.1	00902	Oel & Bardel, 2005, MR-128/04, [M-247578-01-1]
Wheat grain (microwave extraction)	Ethephon m/z 145 → 107	0.05 0.5	5 5	94–99 92–100	97 95	2.1 3.8	00902	Oel & Bardel, 2005, MR-128/04, [M-247578-01-1]
Orange	Ethephon m/z 143 → 107	0.05 0.5	5 5	95–103 96–107	98 101	3.2 4.4	00902	Oel & Bardel, 2005, MR-128/04, [M-247578-01-1]
Orange	Ethephon m/z 145 → 107	0.05 0.5	5 5	96–99 95–104	97 99	1.3 3.8	00902	Oel & Bardel, 2005, MR-128/04, [M-247578-01-1]
Olive	Ethephon m/z 143 → 107	0.05 0.5	5 5	95–104 98–101	101 100	3.7 1.1	00902	Oel & Bardel, 2005, MR-128/04, [M-247578-01-1]
Olive	Ethephon m/z 145 → 107	0.05 0.5	5 5	97–104 101–104	100 103	2.6 1.1	00902	Oel & Bardel, 2005, MR-128/04, [M-247578-01-1]
Tomato	Ethephon m/z 143 → 107	0.05 0.5	5 5	95–100 105–110	97 108	2.0 2.0	00902	Ballesteros, 2005, MR-029/05, [M-247677-01-1]
Wheat grain (conventional extraction)	Ethephon m/z 143 → 107	0.05 0.5	5 5	77–89 91–98	85 93	5.7 3.1	00902	Ballesteros, 2005, MR-029/05, [M-247677-01-1]
Olive	Ethephon m/z 143 → 107	0.05 0.5	5 5	98–99 95–102	98 99	0.5 2.9	00902	Ballesteros, 2005, MR-029/05, [M-247677-01-1]
Wheat green material	Ethephon	0.05 0.5	5 5	77–89 79–82	84 80	5.2 1.4	00918	Oel & Bardel, 2005, MR-173/04, [M-248933-01-1]
Wheat straw	Ethephon	0.05 0.5	5 5	77–88 81–87	81 84	6.1 3.3	00918	Oel & Bardel, 2005, MR-173/04, [M-248933-01-1]
Wheat grain	Ethephon	0.05 0.5	5 5	66, 70–77 65–69	71 67	4.5 3.4	00918	Oel & Bardel, 2005, MR-173/04, [M-248933-01-1]

Matrix	Analyte	Fortification, mg/kg	n	Recovery (%)		% RSD	Method	Reference
				Range	Mean			
Wheat grain (microwave extraction)	Ethephon	0.05 0.5	5 5	77–86 76–83	82 79	5.7 2.4	00918	Oel & Bardel, 2005, MR-173/04, [M-248933-01-1]
Barley green material	Ethephon	0.05 0.5	3 3	100–103 99–102	102 101	1.5 1.7	00918	Oel & Bardel, 2005, MR-173/04, [M-248933-01-1]
Barley straw	Ethephon	0.05 0.5	3 3	70–75 76–77	72 76	3.7 0.8	00918	Oel & Bardel, 2005, MR-173/04, [M-248933-01-1]
Barley grain (microwave extraction)	Ethephon	0.05 0.5	3 3	93–98 93–96	95 95	2.6 1.6	00918	Oel & Bardel, 2005, MR-173/04, [M-248933-01-1]
Olive	Ethephon	0.05 0.5	5 5	77–81 100–106	79 104	1.9 2.3	00918	Schulte, 2014, MR 13/083, [M-463954-01-1]
Olive oil	Ethephon	0.05 0.5	3 3	83–86 101–108	84 104	2.1 3.7	00918	Schulte, 2014, MR 13/083, [M-463954-01-1]
Olive	Ethephon	0.05 0.5	5 5	84–102 93–107	91 99	8.6 5.4	00903/E001	Schulte, 2014, MR 13/083, [M-463954-01-1]
Grape berry	Ethephon	0.05 0.5	3 3	93–98 94–97	96 96	2.6 1.8	00903/E001	Oel & Bardel, 2005, MR-131/04, [M-254165-01-1]
Grape juice	Ethephon	0.05 0.5	3 3	105–118 116–117	112 116	5.9 0.5	00903/E001	Oel & Bardel, 2005, MR-131/04, [M-254165-01-1]
Grape must	Ethephon	0.05 0.5	3 3	99–106 74–107	102 89	3.4 18.8	00903/E001	Oel & Bardel, 2005, MR-131/04, [M-254165-01-1]
Wine	Ethephon	0.05 0.5	3 3	95–104 104–105	100 105	4.5 0.6	00903/E001	Oel & Bardel, 2005, MR-131/04, [M-254165-01-1]
Grape pomace	Ethephon	0.05 0.5	3 3	74–81 84–86	77 85	4.5 1.4	00903/E001	Oel & Bardel, 2005, MR-131/04, [M-254165-01-1]
Apple fruit	Ethephon	0.05 0.5	3 3	105–109 102–110	107 106	2.0 3.8	00903/E001	Oel & Bardel, 2005, MR-131/04, [M-254165-01-1]
Apple juice	Ethephon	0.05 0.5	3 3	101–105 103–107	103 104	1.9 2.2	00903/E001	Oel & Bardel, 2005, MR-131/04, [M-254165-01-1]
Apple washing water	Ethephon	0.05 0.5	3 3	96–104 100–101	100 100	4.1 0.6	00903/E001	Oel & Bardel, 2005, MR-131/04, [M-254165-01-1]
Apple sauce	Ethephon	0.05 0.5	3 3	92–119 87–104	101 96	15.1 8.9	00903/E001	Oel & Bardel, 2005, MR-131/04, [M-254165-01-1]
Apple pomace	Ethephon	0.05 0.5	3 3	89–90 93–97	90 95	0.6 2.1	00903/E001	Oel & Bardel, 2005, MR-131/04, [M-254165-01-1]
Tomato fruit	Ethephon	0.05 0.5	3 3	98–105 101–103	102 12	3.5 1.0	00903/E001	Oel & Bardel, 2005, MR-131/04, [M-254165-01-1]
Tomato juice	Ethephon	0.05 0.5	3 3	98–104 101–108	101 105	3.0 3.4	00903/E001	Oel & Bardel, 2005, MR-131/04, [M-254165-01-1]
Tomato pomace	Ethephon	0.05 0.5	3 3	86–91 88–92	89 90	3.0 2.3	00903/E001	Oel & Bardel, 2005, MR-131/04, [M-254165-01-1]

Matrix	Analyte	Fortification, mg/kg	n	Recovery (%)		% RSD	Method	Reference
				Range	Mean			
Tomato puree	Ethephon	0.05 0.5	3 3	95–101 92–101	98 98	3.1 5.3	00903/E001	Oel & Bardel, 2005, MR-131/04, [M-254165-01-1]
Wheat grain	Ethephon	0.01 0.1	5 5	93–107 64, 93–98	100 89	5.2 15.8	01429	Schulte & Druskus, 2015, MR-14/100
Wheat straw	Ethephon	0.05 0.5	5 5	84–95 87–89	88 88	4.6 1.1	01429	Schulte & Druskus, 2015, MR-14/100
Wheat green material	Ethephon	0.05 0.5	5 5	98–109 87–104	102 95	4.2 7.5	01429	Schulte & Druskus, 2015, MR-14/100
Wheat grain	HEPA	0.01 0.1	5 5	88–98 76–103	94 96	4.4 11.7	01429	Schulte & Druskus, 2015, MR-14/100
Wheat straw	HEPA	0.05 0.5	5 5	85–98 86–95	91 90	5.4 4.1	01429	Schulte & Druskus, 2015, MR-14/100
What green material	HEPA	0.05 0.5	5 5	94–108 97–115	99 108	6.0 6.6	01429	Schulte & Druskus, 2015, MR-14/100

Analytical methods for animal matrices

Method: 18980A 9-REN-74-76 (Ethylene release method) (Leonard, 1993, EC-92-198, [M-187997-01-1])

Analyte:	Ethephon	GC-FID	
LOQ:	0.01 mg/kg in meat, milk and egg		
Description:	This is the same as method 11-94, with some minor modifications for analysing animal tissues. The sample is placed in a pressure bottle with a crown cap that has a provision for withdrawing a headspace sample with a syringe. Water and an aqueous tartaric acid-surfactant solution are added. The bottle is then capped, heated to 60–65 °C and periodically agitated to drive any endogenous ethylene from the sample. After one hour heating, the headspace gases are released, the sample shaken for 5 minutes and then incubated at 60–65 °C with periodic agitation for a further 30 minutes. After shaking for a further 5 minutes, the cap is removed and the bottle is flushed with a gentle stream of nitrogen to remove any released ethylene. The sample is allowed to cool to room temperature. Trisodium phosphate, sufficient to make the sample basic, is added. The bottle is immediately capped and heated for one hour at 60–65 °C with periodic agitation to convert any ethephon residues into ethylene. The ethylene accumulates in the headspace and is quantified by gas chromatography with flame ionisation detection.		

Method: 11-94 (Ethylene release method) (Nygren, 1994, 11-94, [M-188198-01-1])

Analyte:	Ethephon	GC-FID
LOQ:	0.002 mg/kg in milk and eggs, 0.01 mg/kg in tissues	
Description:	The sample is placed in a pressure bottle with a crown cap that has a provision for withdrawing a headspace sample with a syringe. Water and an aqueous tartaric acid-surfactant solution are added. The bottle is then capped, heated to 60–65 °C and periodically agitated to drive any endogenous ethylene from the sample. After one hour heating, the headspace gases are released, the sample shaken for 5 minutes and then incubated at 60–65 °C with periodic agitation for a further 30 minutes. After shaking for a further 5 minutes, the cap is removed and the bottle is flushed with a gentle stream of nitrogen to remove any released ethylene. The sample is allowed to cool to room temperature. Trisodium phosphate, sufficient to make the sample basic, is added. The bottle is immediately capped and heated for one hour at 60–65 °C with periodic agitation to convert any ethephon residues into ethylene. The ethylene accumulates in the headspace and is quantified by gas chromatography with flame ionisation detection. A radiovalidation performed using poultry liver containing incurred residues of [^{14}C]ethephon. The sample was analysed twice using the ethylene-release method. Both analyses indicated a residue level of 0.048 mg/kg. The same sample was analysed using a radiometric technique, which yielded an ethephon concentration of 0.041 mg/kg. The two values therefore are in good agreement, indicating that the ethylene-release method adequately determines the concentration of ethephon residues in animal tissues.	

Method: 00995 (LC-MS/MS method) (Bardel, 2006, MR-054/06 and Amendment 1, [M-274047-02-1])

Analyte:	Ethephon	LC-MS/MS	
LOQ:	0.01 mg/kg in milk, 0.05 mg/kg in meat (muscle), fat, kidney and egg		

Description:	Residues of ethephon are extracted from milk, fat, meat, and kidney by high speed blending with methanol/water/formic acid (90/10/0.1, v/v/v). Residue extraction from egg samples is performed according to a similar procedure, except that some cysteine hydrochloride is added to the extraction solvent. In all cases the extract is cleaned-up on a styrene divinyl benzene SPE column (Varian Bond Elut ENV), concentrated to dryness and reconstituted in water/methanol/formic acid (80/20/0.5, v/v/v). The reconstituted extract is analysed by liquid chromatography with tandem mass spectrometric detection (LC/MS/MS) using a triple-quadrupole apparatus that is operated in the negative electrospray mode. Ethephon is monitored by means of the MS/MS transitions m/z 143 → 107 (^{35}Cl isotope) and/or m/z 145 → 107 (^{37}Cl isotope). Satisfactory chromatographic separation is achieved on a C_{18} column with polar embedding (Synergi Fusion-RP 80Å, 150×4.6 mm, 4 µm). Elution is performed using water/methanol (74/26, v/v) acidified with 0.5% formic acid as the mobile phase.

Table 43 Method Validation

Matrix	Analyte	Fortification, mg/kg	n	Recovery (%)		% RSD	Method	Reference
				Range	Mean			
Animal commodities—ethylene release method								
Milk	Ethephon	0.002 0.004 0.01 0.02 0.04 0.1	1 1 6 1 4 4	88 96 92–104 115 97–102 98–100	88 96 99 115 99 100	– – 4.5 – 2.6 1.0	11-94	Wells-Knecht, 1996, 96E08334, [M-188195-01-1]
Bovine fat	Ethephon	0.01 0.4	1 1	68 72	68 72	– –	11-94	Wells-Knecht, 1996, 96E08334, [M-188195-01-1]
Bovine kidney	Ethephon	0.01 0.1 1 10 12	1 1 1 1 1	71 101 99 102 96	71 101 99 102 96	– – – – –	11-94	Wells-Knecht, 1996, 96E08334, [M-188195-01-1]
Bovine liver	Ethephon	0.01 0.4 2	1 1 1	113 99 102	113 99 102	– – –	11-94	Wells-Knecht, 1996, 96E08334, [M-188195-01-1]
Bovine muscle	Ethephon	0.01 0.4	1 1	94 101	94 101	– –	11-94	Wells-Knecht, 1996, 96E08334, [M-188195-01-1]
Eggs	Ethephon	0.002 0.004 0.005 0.01 0.02 0.1	3 3 1 4 3 3	90–102 93–102 96 100–105 99–102 98–101	95 98 96 102 101 100	6.4 4.7 – 2.3 1.7 1.7	11-94	Wells-Knecht, 1996, 96E08335, [M-188192-01-1]
Poultry liver	Ethephon	0.01 0.1 0.5	1 2 1	115 106–107 90	115 107 90	– – –	11-94	Wells-Knecht, 1996, 96E08335, [M-188192-01-1]
Poultry muscle	Ethephon	0.004 0.01 0.1	2 2 1	84–98 101–107 102	91 104 102	– – –	11-94	Wells-Knecht, 1996, 96E08335, [M-188192-01-1]
Poultry skin + fat	Ethephon	0.004 0.01 0.2	2 2 1	81–89 89–93 93	85 91 93	– – –	11-94	Wells-Knecht, 1996, 96E08335, [M-188192-01-1]
Milk	Ethephon	0.01 0.05 0.1	4 4 4	96–106 95–109 94–103	100 101 99	4.8 5.9 4.9	18980A 9-REN-74-76	Leonard, 1993, EC-92-198, [M-187997-01-1]
Egg	Ethephon	0.01 0.05 0.1	4 4 4	79–93 92–98 93–98	86 96 95	6.4 2.6 2.2	18980A 9-REN-74-76	Leonard, 1993, EC-92-198, [M-187997-01-1]
Meat	Ethephon	0.01 0.05 0.1	4 4 4	73–105 80–107 77–84	93 93 81	15.4 11.8 3.1	18980A 9-REN-74-76	Leonard, 1993, EC-92-198, [M-187997-01-1]
Egg	Ethephon m/z 143 → 107	0.05 0.5	5 5	68–74 95–107	72 101	3.5 4.4	00995	Cavaillé, 2007, MR-06/164, [M-283314-01-1]

Multi-residue Methods

DFG S 19

The applicability of multi-methods has been investigated (Fuchsbichler, 2000, HVA 24/00, [M-184660-01-1]). Multi-residue methods for products of plant origin typically involve extraction with acetone or ethyl acetate. Ethephon is known to be a very hydrophilic compound but it is also readily soluble in acetone and ethyl acetate (solubility > 600 g/L).

Wheat grain was chosen as crop material for the experimental assessment. The samples were fortified with ethephon at 2 or 10 mg/kg. Two variants of the German multi-residue method DFG S19 were investigated. Extraction was performed with acetone/water (2:1, v:v). The extract was cleaned-up by liquid/liquid partition. Depending on the variant of the method, this was done either with dichloromethane or with a mixture of cyclohexane and ethyl acetate. At this stage, the organic phase was dried with sodium sulphate and reacted with (trimethylsilyl) diazomethane in order to methylate any ethephon residues. The methylated extracts were analysed by gas chromatography with flame photometric detection (GC/FPD). There was no ethephon dimethyl ester, indicating that the extraction procedure was not appropriate to ethephon. The same result was found when blank reagents were fortified at the beginning of the procedure. Therefore, the problem was not due to any effect of the crop matrix. In order to demonstrate the accuracy of the derivatisation reaction, control samples and reagent blanks were fortified with ethephon after the extraction step. In this case the concentrations determined by GC/FPD were between 84% and 105% of the theoretical value, therefore validating the derivatisation procedure.

An alternate extraction procedure was investigated similarly. The samples were homogenised with ethyl acetate and sodium sulphate. The extracts were filtered and reacted with (trimethylsilyl) diazomethane. The amounts of ethephon dimethyl ester determined by GC/FPD were less than 30% of the theoretical value. This was a better result than with the DFG S19 extraction procedure, but still insufficient to develop a reliable method.

The study shows that instead of using diazomethane it is possible to perform the methylation with (dimethylsilyl) diazomethane, which is a less hazardous reagent. However, acetone and ethyl acetate are not suitable extraction solvents for ethephon. The extraction procedures used in the classical multi-residue enforcement methods therefore do not work for ethephon.

Storage Stability under Frozen Conditions

Plant commodities

The stability of ethephon residues in commodities has been investigated in high water content commodities (apples, cherries, melons, peppers and tomatoes), high acid content commodities (grapes, blackberries and pineapples), high starch content commodity (wheat) and high oil content commodities (walnuts and cotton) stored under frozen conditions. In all studies on raw agriculture commodities except on wheat and cotton seed, 20 g homogenized control samples were fortified with ethephon. In studies on wheat and cotton seed, 10 g, 5 g and 10 or 5 g of homogenized wheat grain, wheat straw and cotton seed, respectively, were fortified. In studies on apple juice and cottonseed oil, 25 g and 10 g of control samples were fortified.

The stability of ethephon residues has also been investigated in freeze-dried commodities stored at room temperature (apples, cherries, grapes, blackberries, pineapples, melons, peppers, tomatoes and walnuts) because freeze-drying is part of analytical methods. All stored samples were spiked prior to freeze-drying but, for procedural recovery, samples were spiked after freeze-drying.

Conditions and results of storage stability studies are summarized in Table 44 (under frozen conditions) and Table 45 (freeze-dried samples at room temperature). Percent of ethephon remaining was not corrected for procedural recoveries.

Plant matrices

Table 44 Storage stability ethephon in various matrices under frozen conditions

Fortification, mg/kg	Storage temp., °C	Storage time, month	Ethephon, % Remaining	Procedural recovery, %	Analytical method	Reference
Apple						
0.5	–20	0	91, 85	89	SOP 90070	Eckert, 1992, RP-01-89C, [M-187515-01-1]
		1	92, 87	90		
		2	102, 100	99		
		4	101, 92	102		
		6	81, 90	94		
		9	67, 70	79		
		12	70, 69	89		
		18	93, 90	102		
		24	83, 84	88		
Sweet cherry						
1.0	–15	0	91, 95	84	SOP 90070	Nygren, 1992,89-REN-CH-S, [M-187505-01-1]
		1	112, 110	116		
		2	105, 91	112		
		6	91, 93	103		
		9	93, 70	77		
		12	86, 85	99		
		18	97, 80	104		
		24	102, 90	98		
Grape						
0.5	–20	0	78, 91	89	SOP 90070	Eckert, 1992, RP-01-89D, [M-187544-01-1]
		1	70, 78	83		
		2	84, 81	84		
		4	110, 104	93		
		6	99, 76	93		
		9	78, 93	103		
		12	125, 110	112		
		18	73, 75	83		
		24	88, 71	83		
Blackberry						
1.0	–20	0	102, 82	88	SOP 90070	Eckert, 1992, RP-01-89B, [M-187511-01-1]
		1	98, 99	89		
		2	95, 96	91		
		4	100, 108	93		
		6	95, 87	91		
		9	75, 73	86		
		12	114, 92	91		
		18	75, 110	91		
		24	83, 96	95		
Pineapple fruit						
0.5	–20	0	86, 86	83	SOP 90070	Eckert, 1992, RP-01-89E, [M-187540-01-1]
		1	88, 93	79		
		2	95, 95	93		
		4	97, 117	94		
		6	108, 106	98		
		9	90, 90	102		
		12	87, 89	99		
		18	117, 112	110		
		24	77, 98	86		
Pineapple forage						
0.5	–20	0	82, 79	76	SOP 90070	Eckert, 1992, RP-01-89F, [M-187538-01-1]
		1	95, 85	79		

Fortification, mg/kg	Storage temp., °C	Storage time, month	Ethephon, % Remaining	Procedural recovery, %	Analytical method	Reference
		2	91, 86	90		
		4	106, 82	100		
		6	81, 72	92		
		9	85, 88	85		
		12	82, 89	89		
		18	84, 95	93		
		24	85, 98	83		
Cantaloup						
0.5	–20	0	79, 89	104	SOP 90070	Eckert, 1993, RP-01-89G, [M-187507-01-1]
		1	79, 90	76		
		2	96, 86	83		
		4	96, 96	105		
		6	107, 99	99		
		9	102, 92	90		
		12	84, 84	93		
		18	75, 80	80		
		24	82, 82	77		
		30	113, 111	105		
		36	98, 98	104		
Sweet pepper						
1.0	–15	0	120, 110	130	SOP 90070	Nygren, 1992, 89-REN-P-S, [M-187542-01-1]
		2	120, 110	110		
		4	100, 100	98		
		6	100, 87	110		
		9	92, 78	100		
		12	88, 96	85		
		18	110, 120	110		
		24	120, 130	130		
Tomato						
0.5	–20	0	96, 84	91	SOP 90070	Eckert, 1992, RP-01-89A, [M-187533-01-1]
		1	76, 78	93		
		2	118, 84	102		
		4	84, 100	81		
		6	74, 72	75		
		9	61, 82	71		
		12	104, 89	104		
		18	97, 75	78		
		24	99, 107	97		
Wheat grain						
0.5	–20	0	86, 89	75	SOP 90074	Eckert, 1992, RP-01-89I, [M-187521-01-1]
		1	88, 74	90		
		2	118, 98	98		
		4	72, 76	88		
		6	104, 111	111		
		9	100, 78	89		
		12	94, 79	90		
		18	103, 82	89		
		24	90, 79	92		
Wheat straw						
1.0	–20	0	98, 94	85	SOP 90074	Eckert, 1992, RP-01-89H, [M-187519-01-1]
		1	92, 83	80		
		2	88, 75	82		
		4	86, 66	82		
		6	109, 121	93		
		9	87, 87	76		
		12	72, 66	78		
		18	101, 76	91		
		24	90, 108	90		
Walnut nutmeat (English walnut) [e]						
0.2	< –15	0	31, 40	112	SOP 90069	Nygren, 1991, 89-REN-

Fortification, mg/kg	Storage temp., °C	Storage time, month	Ethephon, % Remaining	Procedural recovery, %	Analytical method	Reference
		0 [a]	107, 84 126, 87	72 70		WA-S, [M-187529-01-1]
		1	84, 93	81		
		3	108, 105	64		
		5	69, 74	87		
		5 [b]	66, 83	89		
Cottonseed (10 g homogenized sample) [e]						
1.0	–20	0	76, 86	93	SOP 90075	Eckert, 1992, RP-01-89J, [M-187525-01-1]
		1	89, 98	103		
		2	84, 81	102		
		4	98, 79	108		
		6	89, 72	108		
		9	66, 72	92		
		12	77, 83	79		
		18	57, 65 (46, 65) [c]	94 (77) [c]		
		24 (25) [d]	76, 92 (90, 96) [d]	74 (91) [d]		
Cottonseed (5 g homogenized sample), stored at room temperature in the dark						
0.5	Room temp.	0 day	91, 91, 100, 97, 97 (mean: 95)	96, 91 (mean: 94)	00918	Schmeer and Reineke, 2010, MR-09/053, [M-384885-01-1]
	In the dark	28 days	16, 6, 10 (mean: 11)	100, 94 (mean: 97)		
		35 days	9, 5, 9 (mean: 7.7)	93, 93 (mean: 93)		
Apple juice						
0.20	–20	0	102, 102	103	EC-92-228	Nygren, 1995, EC-94-253, [M-188009-01-1]
		1	99, 100	105		
		2	103, 100	102		
		3	104, 105	104		
		6	104, 104	101		
		9	108, 97	100		
		12	106, 105	100.5		
Cottonseed oil						
0.20	–20	0	94, 95	91	EC-92-228	Nygren, 1995, EC-94-253, [M-188009-01-1]
		1	92, 95	96		
		2	92, 90	93		
		3	80, 82	90		
		6	88, 89	90		
		9	104, 107	102.5		
		12	96.5, 97	94		

[a] Additional set of “Day 0” samples

[b] Additional set of “Day 5” samples

[c] For reanalysis of the samples after 18 months in parentheses

[d] For reanalysis of the samples after 25 months in parentheses

[e] No indication in the study report about whether data were adjusted for procedural recovery

Table 45 Storage stability of ethephon in various freeze-dried matrices at room temperature

Fortification, mg/kg	Storage time, month	Ethephon, % Remaining	Procedural recovery, %	Analytical method	Reference
Apple					
0.5	0	91, 85	89	SOP 90070	Eckert, 1992, RP-01-89C, [M-187515-01-1]
	1	83, 75	81		
	2	97, 101	95		
	4	86, 112	98		
	6	96, 100	93		
	9	77, 83	86		

Fortification, mg/kg	Storage time, month	Ethephon, % Remaining	Procedural recovery, %	Analytical method	Reference
	12	87, 85	87		
	18	95, 89	95		
	24	77, 89	87		
Sweet cherry					
1.0	0	91, 95	84	SOP 90070	Nygren, 1992,89-REN-CH-S, [M-187505-01-1]
	1	111, 97	108		
	2	105, 95	80		
	6	94, 110	105		
	9	104, 89	104		
	12	89, 89	82		
	18	81, 70	96		
	24	83, 85	101		
Grape					
0.5	0	78, 91	89	SOP 90070	Eckert, 1992, RP-01-89D, [M-187544-01-1]
	1	71, 65	77		
	2	81, 87	75		
	4	121, 117	110		
	6	74, 80	82		
	9	64, 70	78		
	12	86, 108	100		
	18	88, 76	98		
	24	79, 92	82		
Blackberry					
1.0	0	102, 82	88	SOP 90070	Eckert, 1992, RP-01-89B, [M-187511-01-1]
	1	82, 86	93		
	2	98, 105	101		
	4	108, 71	104		
	6	101, 90	92		
	9	85, 76	85		
	12	97, 99	87		
	18	99, 64	95		
	24	71, 68	82		
Pineapple fruit					
1.0	0	86, 86	83	SOP 90070	Eckert, 1992, RP-01-89E, [M-187540-01-1]
	1	89, 86	73		
	2	100, 93	89		
	4	90, 90	90		
	6	92, 102	82		
	9	103, 91	89		
	12	98, 94	104		
	18	106, 86	102		
	24	75, 84	87		
Pineapple forage					
0.5	0	82, 79	76	SOP 90070	Eckert, 1992, RP-01-89F, [M-187538-01-1]
	1	73, 86	74		
	2	92, 99	85		
	4	92, 90	90		
	6	87, 88	85		
	9	76, 74	90		
	12	49, 70 (51, 53)	81 (85) [a]		
	18	77, 77	96		
	24	52, 59 (56, 63)	89 (83) [b]		
Cantaloup					
0.5	0	79, 89	104	SOP 90070	Eckert, 1993, RP-01-89G, [M-187507-01-1]
	1	80,76	83		
	2	76, 64	73		
	4	102, 93	88		
	6	59, 37 (47, 38) [c]	89 (106) [c]		
	18	12, 12	81		

Fortification, mg/kg	Storage time, month	Ethephon, % Remaining	Procedural recovery, %	Analytical method	Reference
Sweet pepper					
1.0	0	120, 110	130	SOP 90070	Nygren, 1992, 89-REN-P-S, [M-187542-01-1]
	2	110, 100	130		
	4	92, 93	82		
	6	62, 83 (97, 85) [c]	120 (110) [c]		
	9	47, 57 (70, 60) [c]	96 (98) [c]		
	12	42, 46	87		
	18	37, 36	130		
Tomato					
0.5	0	96, 84	91	SOP 90070	Eckert, 1992, RP-01-89A, [M-187533-01-1]
	1	90, 77	88		
	2	103, 100	83		
	4	82, 87	103		
	6	61, 70	71		
	9	61, 78	80		
	12	97, 97	97		
	18	68, 58	81		
	24	102, 84	98		
Walnut nutmeat (English walnut) [f]					
0.2	0	31, 40	112	SOP 90069	Nygren, 1991, 89-REN-WA-S, [M-187529-01-1]
	0 [d]	107, 84 126, 87	72 70		
	1	91, 74	88		
	5	64, 51	67		
	5 [e]	42, 77	79		
	6	73, 83	73		

[a] Value in parentheses: for reanalysis of the samples after 12 months

[b] Value in parentheses: for reanalysis of the samples after 24 months

[c] Value in parentheses: for reanalysis of the samples

[d] Additional set of “day 0” samples

[e] Additional set of “day 5” samples

[f] No indication in the study report about whether data were adjusted for procedural recovery.

1.

The results showed that ethephon was stable for at least the following periods under frozen conditions:

Table 46 Summary of storage stability of ethephon in various plant matrices under frozen conditions

Matrix	Storage temp., °C	Stable period (at least)	Note
Apple	–20 (–18 to –26)	24 months	Longest period tested
Sweet cherry	–15	24 months	Longest period tested
Grape	–20 (–18 to –26)	24 months	Longest period tested
Blackberry	–20 (–18 to –26)	24 months	Longest period tested
Pineapple fruit	–20 (–18 to –26)	24 months	Longest period tested
Pineapple forage	–20 (–18 to –26)	24 months	Longest period tested
Cantaloupe	–20 (–18 to –26)	36 months	Longest period tested
Sweet pepper	–15	24 months	Longest period tested
Tomato	–20 (–18 to –26)	24 months	Longest period tested
Wheat grain	–20 (–18 to –26)	24 months	Longest period tested
Wheat straw	–20 (–18 to –26)	24 months	Longest period tested
Walnut	–15	3 months	Not conclusive due to analytical uncertainty
Cottonseed	–20 (–18 to –26)	25 months	Longest period tested (some uncertainty)
Apple juice	–20	12 months	Longest period tested
Cottonseed oil	–20	12 months	Longest period tested

Ethephon was shown to be stable during storage at room temperature after freeze-drying for the longest period tested (24 months) in apples, sweet cherries, grapes, blackberries, pineapple fruit, and tomato samples.

However, ethephon was stable up to only 9 months in pineapple forage, 4 months in cantaloupe, and 6 months in sweet pepper samples during storage at room temperature after freeze-drying. Due to significant analytical uncertainty, it was also not possible to determine storage stability of freeze-dried walnut samples at room temperature.

Animal Commodities

A storage stability study was conducted on meat, milk and eggs in 1992–1993 (Leonard, 1993, EC-92-198, [M-187997-01-1]).

Bovine meat was trimmed, and ground to homogeneity. Eggs were removed from their shells and beaten to a homogenous mixture. Milk was used as received. The prepared control samples (40 g) were fortified with ethephon at a concentration of 0.10 mg/kg and then stored frozen at about –20 °C. Samples were analysed using the ethylene release method 18980A 9-REN-74-76

The results showed that ethephon was stable when stored frozen (actual temperature: –10 to –23 °C) for the longest periods tested: in milk for 4 months, in meat 12 months and in eggs 15 months.

Table 47 Storage stability of ethephon in animal matrices at a fortification level of 0.1 mg/kg and at -20 ºC

Time, month	Ethephon, % Remaining	Procedural recovery, %
Bovine milk		
0	95, 99	97
1	99, 98	100
2	98, 89	93
3	93, 94	99
4	96, 97	96
Bovine meat		
0	93, 106	97
1	91, 93	97
2	96, 91	99
3	96, 94	94
4	97, 86	97
6	95, 94	95
9	92, 92	95
12	91, 89	85
Poultry eggs		
0	95, 90	93
1	96, 102	95
2	101, 88	99
3	94, 93	91
4	97, 96	102
6	96, 92	88
9	92, 89	92
12	94, 90	94
15	93, 92	94

USE PATTERN

Ethephon is registered in many countries for use on cereals (wheat, barley, rye and rice) to increase resistance to lodging through straw shortening and strengthening; fruits and vegetables to promote

fruit maturity (early and uniform ripening and colouring of mature fruits); and on cotton to promote uniform boll opening and enhance defoliation.

Ethephon is mainly formulated as a soluble concentrate (SL). Combinations with chlormequat chloride are also used for cereals, and combinations with cyclanilide are used for cotton. Formulations are applied as foliar sprays by either ground or aerial equipment, except for applications to figs in Brazil where ethephon is applied directly to fruits using brushes or other equipment for even distribution.

For the purposes of estimating maximum residue levels, only the registered uses in countries relevant for the submitted supervised trials are recorded in Table 48.

For cereals, where there is a long interval between application and harvest, PHIs are often not given on the label. The PHI is described by the vegetative growth between applications; the labels give the growth stage at application. Therefore for cereals in the table below, both the PHI (where available) and the application timing (growth stage at application) are given.

Table 48 Registered Uses of Ethephon

Crop	Country	Form. Ethephon conc Type	Application				PHI (days)/Application timing Notes
			Max rate, g ai/ha	Max. Spray conc., g ai/hL	Water volume (L/ha)	No. (max g ai/ha/ season)	
Pome fruits							
Apple	Austria	660 g/L SL	198		500 L/ha/m crown height	2 (396 g ai/ha)	91/ BBCH 59–31
Apple (cider varieties)	France	120 g/L SL		48		1–2	10/ Pre-bloom or post-bloom
Apple (other varieties)	France	120 g/L SL		36		1–2	10/ 15–20 days before expected harvest date
Apple	Italy	480 g/L SL		48	1500–2000	– (768 g ai/ha)	14/ 14–20 days before harvest
Stone fruits							
Cherry	Austria	660 g/L SL	357		500 L/ha/m crown height	1	7/ BBCH 79–89
Cherry	France	120 g/L SL		36		1	10
Cherry, sour	Netherlands	480 g/L SL	360			1	7/ 7–10 days before harvest
Berries and other small fruits							
Grape	France	180 g/L SL	450		100–200	1	28/ 15–30% berries ripe
Assorted tropical fruits and sub-tropical fruits—edible peel							
Fig	Brazil	720 g/L SL		936		1	5 fruit in bloom stage with pink ostioles. Apply directly to fruit using brushes with sponge tip or any other equipment that evenly distribute the mixture over the fruit.
Olive for oil and table olive	Italy	480 g/L SL	1st 450 2nd 600	1st 36 2nd 48	1250	2	11 (1st appl. 18 days before harvest)
Assorted tropical fruits and sub-tropical fruits—inedible peel							

Crop	Country	Form. Ethephon conc Type	Application				PHI (days)/Application timing Notes
			Max rate, g ai/ha	Max. Spray conc., g ai/hL	Water volume (L/ha)	No. (max g ai/ha/ season)	
Pineapple	Belize, El Salvador, Honduras, Domenican Rep.	480 g/L SL	1920		2000–3000	1 or 2 (1920 g ai/ha)	7–14/ Apply 1–2 weeks before first round of harvesting (common label)
Pineapple	Brazil	720 g/L SL	936 (Dec, Jan, Feb)		200–500 30 (aerial)	1	14
Pineapple	Costa Rica, Panama, Guatemala	720 g/L SL	936		2000–3000	1	7–14 (common label)
Pineapple	Costa Rica, Panama, Guatemala	720 g/L SL	1152		1000	1 or 2 (1152 g ai/ha)	7–14/ Apply 1–2 weeks before first round of harvesting (common label)
Pineapple	Costa Rica	480 g/L SL	1200		2800–3800	2 (2400 g ai/ha)	1
Pineapple	Costa Rica	480 g/L SL	1200		2800–3800	2 (2400 g ai/ha)	1
Pineapple	Costa Rica	480 g/L SL	1200		2000–3000	2 (1920 g ai/ha)	1
Pineapple	Kenya	480 g/L SL	480		3000	1	–/ Apply when plants are ready to be forced to flower
Pineapple	Kenya	480 g/L SL	1920		500–1000	1	7
Fruiting vegetables, other than cucurbits							
Tomato (except cherry tomato)	Austria	660 g/L SL	594		1200	1	7/ BBCH 81–85
Tomato	Bolivia	240 g/L SL	1920		100–400	1	21
Tomato	Canada	240 g/L SL	1536		30–500	1	14–21/ Apply when 5–30% of fruits partly red or red
Tomato (for fresh consumption)	France	120 g/L SL		192		1	7/ Apply after harvest of first fruits, when max fruits on 1st to 3rd trusses. 10–15 days before last harvest
Tomato (for processing)	France	120 g/L SL	1680		800–1000	1	7/ Apply when 20–25% of fruits are red
Tomato (for fresh consumption)	Italy	480 g/L SL		120		1	7/ Apply when 40–60% of fruits are ripe and remaining fruits are at mature green stage. Can be divided into two applications
Tomato (for processing)	Italy	480 g/L SL	1920		1000 (for determined variety)	2 (1920 g ai/ha)	
Tomato	Netherlands	480 g/L SL		48		1	–/ senescent crops
Cereal grains							
Barley, winter	Austria	660 g/L SL	462		100–300	1	– / BBCH 32–49
Barley, spring	Austria	660 g/L SL	330		100–300	1	– / BBCH 37–51

Crop	Country	Form. Ethephon conc Type	Application				PHI (days)/Application timing Notes
			Max rate, g ai/ha	Max. Spray conc., g ai/hL	Water volume (L/ha)	No. (max g ai/ha/ season)	
Barley, winter	Belgium	480 g/L SL	600		200–400	1	– / BBCH 37–39
Barley, spring	Belgium	480 g/L SL	384		200–400	1	– / BBCH 37–39
Barley, spring	France	480 g/L SL	360		100–200	1	56 / BBCH 32–39
Barley, winter	France	480 g/L SL	480		100–200	1	56 / BBCH 32–39
Barley, spring	France	150 g/L SL	225			1	– / BBCH 31–37 (+ chlormequat-chloride 300 g/L)
Barley, winter	France	150 g/L SL	375			1	– / BBCH 31–39 (+ chlormequat-chloride 300 g/L)
Barley, winter	Germany	660 g/L SL	462		100–300	1	– / BBCH 32–49
Barley, spring	Germany	660 g/L SL	330		100–300	1	– / BBCH 37–49
Barley, winter	Poland	480 g/L SL	720		150–300	1	– / BBCH 32–39
Barley, spring	Poland	480 g/L SL	360		150–300	1	– / BBCH 32–49
Barley, winter	UK	480 g/L SL	480		100–400	– (480 g ai/ha)	– / BBCH 32–49
Barley, spring	UK	480 g/L SL	240		100–400	– (240 g ai/ha)	– / BBCH 32–49
Rye, winter	Austria	660 g/L SL	726		100–300	1	– / BBCH 37–49
Rye	Belgium	480 g/L SL	720		200–400	1	– / BBCH 39–45
Rye, winter	Germany	660 g/L SL	726		100–300	1	– / BBCH 37–49
Rye, winter	UK	480 g/L SL	480		100–400	– (480 g ai/ha)	– BBCH 37–49
Triticale, winter	Austria	660 g/L SL	495		100–300	1	– / BBCH 37–39
Triticale	Belgium	480 g/L SL	600		200–400	1	– / BBCH 37–45
Triticale	France	480 g/L SL	480		100–200	1	70 / BBCH 32–39
Triticale	France	150 g/L SL (+ chlormequat-chloride 300 g/L)	375			1	– / BBCH 31–37
Triticale, winter	Germany	660 g/L SL	495		100–300	1	– / BBCH 37–49
Triticale	Poland	480 g/L SL	480		150–300	1	– / BBCH 32–37
Triticale, winter	UK	480 g/L SL	480		100–400	– (480 g ai/ha)	– / BBCH 37–47
Wheat	Austria	660 g/L SL	462		100–300	1	– / BBCH 37–51
Wheat, winter	Belgium	480 g/L SL	600		200–400	1	– / BBCH 37–45
Wheat, winter	Canada	240 g/L SL	600		30–300	1	35 / BBCH 37–49
Wheat, spring	Canada	240 g/L SL	360		30–300	1	35 / BBCH 37–49

Crop	Country	Form. Ethephon conc Type	Application				PHI (days)/Application timing Notes
			Max rate, g ai/ha	Max. Spray conc., g ai/hL	Water volume (L/ha)	No. (max g ai/ha/ season)	
Wheat, hard, winter	France	480 g/L SL	480		100–200	1	70 / BBCH 39
Wheat, soft, winter	France	480 g/L SL	288		100–200	1	56 / BBCH 39
Wheat, hard, winter	France	150 g/L SL	375			1	– / BBCH 31–37 (+ chlormequat-chloride 300 g/L)
Wheat, soft, winter	France	150 g/L SL	300			1	– / BBCH 31–37 (+ chlormequat-chloride 300 g/L)
Wheat	Germany	660 g/L SL	462		100–300	1	– / BBCH 37–51
Wheat, winter Wheat, spring	Poland	480 g/L SL	360		150–300	1	– / BBCH 31–37
Wheat, winter	UK	480 g/L SL	360		100–400	– (360 g ai/ha)	– / BBCH 37–47
Oilseeds							
Cotton	Greece	480 g/L SL	1440		500–600	1	7/ BBCH 82–84
Cotton	Brazil	480 g/L SC	1200		200–500	1	7/ Apply at 90% boll maturity (+ cyclanilide 60 g/L)
Cotton	USA	720 g/L SC	2240		28–47 aerial 94–234 ground	1	7 (+ cyclanilide 45 g/L)
Cotton	USA	720 g/L SL	2240		19–94	1	7

RESIDUES RESULTING FROM SUPERVISED TRIALS

Supervised trials have been conducted on the following crops: apples, cherries, grapes, figs, olives, pineapples, tomatoes, cereal grains (wheat, barley and rye) and cotton. The results of these supervised trials are summarized in the following tables:

Crop Group	Commodity	Country/Region, year of trials	Table No.
Pome fruit	Apple	Europe, 2000, 2002, 2006, 2007	49
Stone fruit	Cherries	Europe, 2000, 2002, 2009	50
Berries and other small fruits	Grapes	Europe, 1995, 2006, 2009	51
Assorted tropical and sub-tropical fruits—edible peel	Fig	Brazil, 2004, 2005	52
	Olive	Europe, 2007, 2008	53
Assorted tropical and sub-tropical fruits—inedible peel	Pineapple	Brazil, 1994, 1997, 2005 Costa Rica, 1998 Côte d'Ivoire, 1997, 1999 USA, 1989	54
Fruiting vegetables, other than cucurbits	Tomatoes	Europe, 1999, 2000, 2001, 2004 USA, 1989, 1990, 1991, 2005	55

Crop Group	Commodity	Country/Region, year of trials	Table No.
Cereal grains	Barley	Europe, 2000, 2001, 2004, 2006, 2007, 2008	56
			57
	Rye	Europe, 2013, 2014	58
	Wheat	Europe, 2006, 2007	59
		Europe, 2000, 2001, 2004, 2006, 2007	60
		Europe, 2013, 2014	61
		USA, 1981, 1989	
Oilseeds	Cotton	Europe, 1993, 1994, 1995, 2008	62
		USA, 1989, 1993, 1994	
		Brazil, 1996, 2006	
Primary animal feed	Barley	(See above)	63, 64
	Rye		65
	Wheat		66, 67, 68

In addition to the description and details of the field trials and analytical methods, each study report includes procedural recoveries and in some cases a summary of the method validation.

In the trials where multiple analyses are 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.

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. Where a higher residue value was obtained at a later PHI, the higher value has been used.

Apple

A total of eighteen supervised trials were conducted on apples in France, Germany, the UK, Italy, Spain, Portugal and Greece. A 480 g/L SL formulation was applied as a foliar spray at BBCH 78–89 at a rate of 0.35–0.42 kg ai/ha. In studies 00-551 and 00-550, residues of ethephon were determined using method HVA SOP 10071. In study 02R792, residues of ethephon were determined using method V5229/01. In studies RA-2514/06 and RA-2576/07, residues of ethephon were determined using method 00903, supplement E001. The maximum period of storage of frozen samples was 406 days at < –18 ºC.

Table 49 Ethephon residues in apples resulting from supervised trials in Europe

APPLE Trial No Country, year (Variety)	Application					DALT days	Ethephon mg/kg	Reference
	Form. (g ai/L & type)	kg ai/ha	kg ai/hL	Water (L/ha)	No			
GAP, France	120 g/L SL		0.036		1	10		
GAP, Italy	480 g/L SL		0.048	1500–2000	1	14		
00551AM1 Saulty, France, 2000 (Canada Grise)	480 g/L SL	0.35	0.035	1000	1	10	0.40	Ballesteros, 2002, R&D/CRLD/AN/0215010 (M-209123-01-1)

APPLE Trial No Country, year (Variety)	Application					DALT days	Ethephon mg/kg	Reference
	Form. (g ai/L & type)	kg ai/ha	kg ai/hL	Water (L/ha)	No			
00551RS1 Damard, France, 2000 (Idared)	480 g/L SL	0.35	0.035	1000	1	11	0.27	Ballesteros, 2002, R&D/CRLD/AN/0215010 (M-209123-01-1)
00550RN1 Bellevue, France, 2000 (Judeline)	480 g/L SL	0.36	0.035	1029	1	0 3 7 10	0.62 0.54 0.62 0.26	Ballesteros, 2002, R&D/CRLD/AN/0215012 (M-210409-01-1)
00550RS1 Monthurel, France, 2000 (Judeline)	480 g/L SL	0.42	0.035	1201	1	0 3 7 10	0.39 0.15 0.22 0.075	Ballesteros, 2002, R&D/CRLD/AN/0215012 (M-210409-01-1)
02R792-1 Soucelles, France, 2002 (Golden Delicious)	480 g/L SL	0.36	0.072	500	1	0 3 7 10 14 21	0.47 0.84 0.68 0.31 0.40 0.28	Sonder, 2004, 02 R 792 (M-220915-01-1)
02R792-2 Cheille, France, 2002 (Gala)	480 g/L SL	0.36	0.067	550	1	0 3 7 10 14 21	0.29 0.30 0.34 0.13 0.13 0.12	Sonder, 2004, 02 R 792 (M-220915-01-1)
02R792-3 Geisenheim, Germany, 2002 (Jonagold)	480 g/L SL	0.36	0.045	800	1	0 3 7 10 14 21	0.13 0.19 0.20 0.14 0.11 0.14	Sonder, 2004, 02 R 792 (M-220915-01-1)
02R792-4 Wurzen-Roitzsch, Germany, 2002 (Rubin)	480 g/L SL	0.36	0.036	1000	1	0 3 7 10 14 21	0.11 0.11 0.15 0.059 0.051 < 0.05	Sonder, 2004, 02 R 792 (M-220915-01-1)
02R792-5 Royston, UK, 2002 (Bramley)	480 g/L SL	0.36	0.072	500	1	0 3 7 10 14 22	0.18 0.13 < 0.05 0.081 < 0.05 < 0.05	Sonder, 2004, 02 R 792 (M-220915-01-1)
R 2006 0116/6 Pernes les Fontaines, France, 2006 (Galaxy)	480 g/L SL	0.36	0.036	1000	1	0 7 10 14 21	0.25 0.17 < 0.05 < 0.05 < 0.05	Billian, 2007, RA-2514/06 (M-292470-01-1)
R 2006 0245/6 Bologna, Italy, 2006 (Golden)	480 g/L SL	0.36	0.036	1000	1	0 7 10 14 21	0.17 0.21 0.15 0.12 0.08	Billian, 2007, RA-2514/06 (M-292470-01-1)
R 2006 0246/4 Torrelavit, Spain, 2006 (Golden)	480 g/L SL	0.39	0.045	856	1	0 7 10 14 21	0.48 0.64 0.49 0.31 0.09	Billian, 2007, RA-2514/06 (M-292470-01-1)

APPLE Trial No Country, year (Variety)	Application					DALT days	Ethephon mg/kg	Reference
	Form. (g ai/L & type)	kg ai/ha	kg ai/hL	Water (L/ha)	No			
R 2006 0247/2 Peral-Cadaval, Portugal, 2006 (Fuji)	480 g/L SL	0.36	0.045	800	1	0 7 10 14 21	0.41 0.20 0.07 0.09 0.06	Billian, 2007, RA-2514/06 (M-292470-01-1)
R 2006 0248/0 Tripotamos, Greece, 2006 (Jonagold Red)	480 g/L SL	0.36	0.048	750	1	0 7 10 14 21	0.14 0.16 0.13 0.15 0.09	Billian, 2007, RA-2514/06 (M-292470-01-1)
R 2007 0176/4 Eyragues, France, 2007 (Brock field)	480 g/L SL	0.36	0.036	1000	1	0 7 10 14 21	0.18 0.25 0.24 0.18 0.16	Billian, Erler & Wolters, 2008, RA-2576/07 (M-311032-01-1)
R 2007 0188/8 Zevio, Italy, 2007 (Golden Rainders)	480 g/L SL	0.36	0.036	1000	1	0 7 10 14 21	0.19 0.08 0.07 < 0.05 < 0.05	Billian, Erler & Wolters, 2008, RA-2576/07 (M-311032-01-1)
R 2007 0189/6 Caldes de Malavella-Girona, Spain, 2007 (Golden Smoothy)	480 g/L SL	0.36	0.036	1000	1	0 7 9 14 21	0.19 0.25 0.15 0.14 0.07	Billian, Erler & Wolters, 2008, RA-2576/07 (M-311032-01-1)
R 2007 0191/8 Tripotamos, Greece, 2007 (Jonagold Red)	480 g/L SL	0.36	0.036	1000	1	0 7 10 14 21	0.09 0.07 0.08 0.05 < 0.05	Billian, Erler & Wolters, 2008, RA-2576/07 (M-311032-01-1)

Cherries

A total of fifteen supervised trials were conducted on cherries in France, Italy, Spain, Greece, Belgium and the Netherlands. A 480 g/L SL formulation was applied as a foliar spray to cherry trees at BBCH 76–89 at a rate of 0.35–0.36 kg ai/ha. In general, residues were determined in the whole fruit at earlier time points, and in the pitted fruit at the last time point, and the residue in the whole fruit was calculated. Whether whole fruit or pitted fruit was analysed is specified in the following Table. In the trials conducted in 2000, residues of ethephon were determined using method HVA SOP 10071. In the trials conducted in 2002, residues of ethephon were determined using method V5229/01. In the trials conducted in 2009, residues of ethephon were determined using method 00903, supplement E001. The maximum period of storage of frozen samples at < –18 ºC was 483 days.

Table 50 Ethephon residues in cherries resulting from supervised trials in Europe

CHERRY Trial No Country, year (Variety)	Application					DALT days	Portion analysed	Ethephon mg/kg	Reference
	Form. (g ai/L & type)	kg ai/ha	kg ai/hL	Water (L/ha)	No				
GAP, Austria	660 g/L SL	0.36		500 L/h a/m crown height	1	7			

CHERRY Trial No Country, year (Variety)	Application					DALT days	Portion analysed	Ethephon mg/kg	Reference
	Form. (g ai/L & type)	kg ai/ha	kg ai/hL	Water (L/ha)	No				
GAP, Netherlands	480 g/L SL	0.36			1	7			
00552AV1 Malaucene, France, 2000 (Napoleon)	480 g/L SL	0.35	0.036	962	1	0 3 7 11 11	Whole fruit Whole fruit Whole fruit Pitted fruit Whole fruit (calculated)	0.55 0.65 0.65 0.53 0.48	Ballesteros, 2002, R&D/CRLD/AN/mr/ 0115439 (M-208089-01-1)
00552TL1 Belcastel, France, 2000 (Stark)	480 g/L SL	0.35	0.035	1000	1	0 2 7 10 10	Whole fruit Whole fruit Whole fruit Pitted fruit Whole fruit (calculated)	0.54 0.66 1.40 0.64 0.59	Ballesteros, 2002, R&D/CRLD/AN/mr/ 0115439 (M-208089-01-1)
00553AV1 L'Isle s/la Sorge, France, 2000 (Napoleon)	480 g/L SL	0.35	0.035	1000	1	10 10	Pitted fruit Whole fruit (calculated)	0.17 0.15	Ballesteros, 2002, R&D/CRLD/AN/01154 58 (M-208961-01-1)
00553TL1 Adge, France, 2000 (Van)	480 g/L SL	0.35	0.035	1000	1	9 9	Pitted fruit Whole fruit (calculated)	2.9 2.7	Ballesteros, 2002, R&D/CRLD/AN/01154 58 (M-208961-01-1)
00554BKA1 Fougerolles, France, 2000 (Bechat thermo)	480 g/L SL	0.35	0.035	997	1	0 3 7 10 10	Whole fruit Whole fruit Whole fruit Pitted fruit Whole fruit (calculated)	0.65 1.2 0.91 0.50 0.42	Ballesteros, 2002, R&D/CRLD/AN/02150 09 (M-210351-01-1)
00554BKA2 Saxon Sion, France, 2000 (Montmorency)	480 g/L SL	0.35	0.035	1000	1	0 3 7 10 10	Whole fruit Whole fruit Whole fruit Pitted fruit Whole fruit (calculated)	2.1 2.6 0.30 0.15 0.14	Ballesteros, 2002, R&D/CRLD/AN/02150 09 (M-210351-01-1)
00555BKA1 Fourgerolles, France, 2000 (Marie-Jean Diaude)	480 g/L SL	0.35	0.035	993	1	9 9	Pitted fruit Whole fruit (calculated)	0.61 0.52	Ballesteros, 2002, R&D/CRLD/AN/02150 13 (M-210352-01-1)
00555BKA2 Saint Maurice s/les Cotes, France, 2000 (Griotte à jus clair)	480 g/L SL	0.36	0.035	1008	1	9 9	Pitted fruit Whole fruit (calculated)	0.36 0.33	Ballesteros, 2002, R&D/CRLD/AN/02150 13 (M-210352-01-1)
02R795-1 Boe, France, 2002 (Coralise)	480 g/L SL	0.36	0.036	1000	1	0 4 7 11 11	Whole fruit Whole fruit Whole fruit Pitted fruit Whole fruit (calculated)	2.7 2.3 2.3 1.8 1.6	Sonder, 2004, 02 R 795 (M-220921-01-1)
02R795-2 Malaucene, France, 2002 (Bigareau Napolélon)	480 g/L SL	0.36	0.037	972	1	0 9 0 9	Pitted fruit Whole fruit (calculated)	0.77 0.93 0.66 0.67	Sonder, 2004, 02 R 795 (M-220921-01-1)

CHERRY Trial No Country, year (Variety)	Application					DALT days	Portion analysed	Ethephon mg/kg	Reference
	Form. (g ai/L & type)	kg ai/ha	kg ai/hL	Water (L/ha)	No				
02R795-3 Andria, Italy, 2002 (Ferrovia)	480 g/L SL	0.36	0.043	834	1	0 4 7 10 0 4 7 10	Pitted fruit Whole fruit (calculated)	1.5 1.8 1.7 2.3 1.0 1.6 1.5 2.0	Sonder, 2004, 02 R 795 (M-220921-01-1)
02R795-4 Segorbe, Spain, 2002 (Precoz De Bernat)	480 g/L SL	0.36	0.023	1550	1	0 10 10	Whole fruit Pitted fruit Whole fruit (calculated)	0.30 0.76 0.64	Sonder, 2004, 02 R 795 (M-220921-01-1)
02R795-5 Lokindros, Greece, 2002 (Bourla)	480 g/L SL	0.36	0.024	1500	1	0 9 9	Whole fruit Pitted fruit Whole fruit (calculated)	0.57 0.40 0.37	Sonder, 2004, 02 R 795 (M-220921-01-1)
09-2147-01 Rosoux, Belgium, 2009 (Regina)	480 g/L SL	0.36	0.030	1200	1	0 4 7 10 14	Whole fruit	0.25 0.42 0.44 0.31 0.31	Uceda and Meilland-Berthier, 2011, 09-2147 (M-403958-01-1)
09-2147-02 ND Wognum, Netherlands, 2009 (Regina)	480 g/L SL	0.36	0.024	1500	1	0 4 7 10 14	Whole fruit	0.16 0.21 0.28 0.23 0.21	Uceda and Meilland-Berthier, 2011, 09-2147 (M-403958-01-1)

Grapes

Ten supervised trials were conducted on grapes in France. A 180 g/L SL formulation was applied once as a foliar spray to grape vines at BBCH 83–85 at a rate of 0.45–0.47 kg ai/ha. In the trials conducted in 1995, residues of ethephon were determined using the analytical method referenced in "Analytical Method for Residues of Pesticides" Part II-89, 5th Edition, SDU Publishers, The Netherlands (1988). This method is similar to SOP 90070 and was validated on grapes prior to use. The LOQ was 0.10 mg/kg. In the trials conducted in 2006 and 2009, residues of ethephon were determined using method 00903, supplement E001. The maximum period of storage of frozen samples at < –18 ºC was 447 days.

Table 51 Ethephon residues in grapes resulting from supervised trials in Europe

GRAPES Trial No Country, year (Variety)	Application					DALT days	Ethephon mg/kg	Reference
	Form. (g ai/L & type)	kg ai/ha	kg ai/hL	Water (L/ha)	No			
GAP, France	180 g/L SL	0.45		100–200	1	28		
EA950185-FR01 Mercurol, France, 1995 (Syrah)	180 g/L SL	0.45	0.45	99	1	0 25 35	0.80 0.35 0.37	Grolleau, 1997, EA950185 (M-188232-01-1)
EA950185-FR02 Pouzillac, France, 1995 (Grenache)	180 g/L SL	0.47	0.45	105	1	0 25 35	1.02 0.17 0.25	Grolleau, 1997, EA950185 (M-188232-01-1)

GRAPES Trial No Country, year (Variety)	Application					DALT days	Ethephon mg/kg	Reference
	Form. (g ai/L & type)	kg ai/ha	kg ai/hL	Water (L/ha)	No			
R 2006 0333/9 Blere, France, 2006 (Cabernet franc)	180 g/L SL	0.45	0.23	200	1	0 10 21 28 35	0.58 1.5 0.74 0.52 0.39	Billian, Lorenz, Telscher, 2005, RA-2562/06 (M-294217-01-1)
R 2006 0411/4 Saint Nicolas de Bourgueil, France, 2006 (Cabernet franc)	180 g/L SL	0.45	0.23	200	1	0 10 21 28 35	0.53 0.58 0.45 0.21 0.21	Billian, Lorenz, Telscher, 2005, RA-2562/06 (M-294217-01-1)
R 2006 0334/7 Fronton, France, 2006 (Négrette)	180 g/L SL	0.45	0.23	200	1	0 10 21 28 35	0.81 0.68 0.24 0.18 0.13	Billian, Telscher, 2005, RA-2563/06 (M-294366-01-1)
R 2006 0412/2 Laudun, France, 2006 (Merlot)	180 g/L SL	0.45	0.23	200	1	0 10 21 28 35	0.63 0.09 0.07 0.05 < 0.05	Billian, Telscher, 2005, RA-2563/06 (M-294366-01-1)
09-2176-01 La Chapelle de Guinchay, France, 2009 (Gamay)	180 g/L SL	0.45	0.23	200	1	0 10 21 28 35	0.42 0.30 0.09 0.05 0.07	Uceda, Meilland, Berthier, 2011, 09-2176 (M-403873-01-1)
09-2176-02 Athee sur Cher, France, 2009 (Gamay)	180 g/L SL	0.45	0.23	200	1	0 10 21 28 35	0.34 0.25 0.28 0.20 0.16	Uceda, Meilland, Berthier, 2011, 09-2176 (M-403873-01-1)
09-2176-03 Vendeuvre du poitou, France, 2009 (Gamay)	180 g/L SL	0.45	0.23	200	1	0 10 21 28 35	0.38 0.27 0.16 0.10 0.14	Uceda, Meilland, Berthier, 2011, 09-2176 (M-403873-01-1)
09-2176-04 Fonton, France, 2009 (Negrette)	180 g/L SL	0.45	0.23	200	1	0 9 21 28 35	0.31 0.57 0.32 0.18 0.18	Uceda, Meilland, Berthier, 2011, 09-2176 (M-403873-01-1)

Fig

Six supervised trials were conducted in 2004–2005 on figs in Brazil. For the trials conducted in 2004, brush application was carried out with a 240 g/L SL formulation at the harvest growth stage. For the trials conducted in 2005, application used a 720 g/L SL formulation at the harvest growth stage. In the trials conducted in 2004, residues of ethephon were determined using the analytical method referenced in "Analytical Methods for Pesticide Residues in Foodstuffs" 6th Edition, part II, The Netherlands, 1996, with some modifications. In the trials conducted in 2005, residues of ethephon were determined using the analytical method 11-94. The maximum period of storage of frozen samples at < –20 ºC was 8 months.

Table 52 Ethephon residues in figs resulting from supervised trials in Brazil

FIG	Application	DALT	Ethephon	Reference

Trial No Country, year (Variety)	Form. (g ai/L & type)	kg ai/ha	kg ai/hL	Water (L/ha)	No	days	mg/kg	
GAP, Brazil	720 g/L SL		0.94		1	5		
1(R04MA1) Valinhos, Brazil, 2004 (Figo Roxo de Valinhos)	240 g/L SL		24	0.5	1	0 1 3 5 7	2.7 1.3 0.8 0.2 0.2	Trevizan, de Baptista, 2004, 102/5373/04 (M-284626-01-2)
	240 g/L SL		24	1.0	1	5	0.2	
2(R04MA01-P1) Monte Mor, Brazil, 2004 (Figo Roxo de Valinhos)	240 g/L SL		24	0.5	1	5	< 0.2	Trevizan, de Baptista, 2004, 102/5374/04 (M-284634-01-2)
	240 g/L SL		24	1.0	1	5	< 0.2	
3(R04MA01-P2) Caldas-MG, Brazil, 2004 (Figo Roxo de Valinhos)	240 g/L SL		24	0.5	1	5	0.6	Trevizan, de Baptista, 2004, 102/5375/04 (M-284637-01-2)
	240 g/L SL		24	1.0	1	5	0.9	
HR05BRA008-P1 Piracicaba, Brazil, 2005 (Roxo de Valinhos)	720 g/L SL		0.94	25	1	5	0.75	Galhiane, Santos, 2005, RA-925/05 (M-284675-01-2)
	720 g/L SL		1.9	25	1	5	1.32	
HR05BRA008-P2 Valinhos, Brazil, 2005 (Roxo de Valinhos)	720 g/L SL		0.94	25	1	5	0.71	Galhiane, Santos, 2005, RA-926/05 (M-284678-01-2)
	720 g/L SL		1.9	25	1	5	1.25	
HR05BRA008-P3 Itatiba, Brazil, 2005 (Roxo de Valinhos)	720 g/L SL		0.94	25	1	5	0.73	Galhiane, Santos, 2005, RA-927/05 (M-284681-01-2)
	720 g/L SL		1.9	25	1	5	1.34	

Olives

Eight supervised trials were conducted in 2007–2008 on olives in Spain. In the 2007 trials, a 480 g/L SL formulation was applied twice as a foliar spray to olives trees at BBCH 79–81 at a rate of 0.35–0.41 + 0.47–0.50 kg ai/ha and a 7-day interval between applications. In the 2008 trials, a 480 g/L SL formulation was applied twice as a foliar spray to olives trees at BBCH 78–87 at a rate of 0.48 + 0.62 kg ai/ha and a 7–8 day interval between applications. In the trials conducted in 2007, residues of ethephon were determined using method 00903. In the trials conducted in 2008, residues of ethephon were determined using method 00903, supplement E001. The maximum period of storage of frozen samples at < –18 ºC was 12 months for olives, 7 months for table olives and 11.5 months for oil.

Table 53 Ethephon residues in olives resulting from supervised trials in Europe

OLIVES Trial Country, year (Variety)	Application					DALT days	Ethephon mg/kg	Reference
	Formulation (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No			
GAP, Italy	480 g/L SL	1st 0.45 2nd 0.60	1st 36 2nd 48	1250	2	11		
07 D OL BY P01 Arahal, Spain, 2007 (Manzanillo)	480 g/L SL	0.35 0.47	0.036 0.048	968 971	2	11	4.3	Fernandez, 2009, 07 D OL BY P/A (M-352734-01-1)

OLIVES Trial Country, year (Variety)	Application					DALT days	Ethephon mg/kg	Reference
	Formulation (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No			
07 D OL BY P02 Huevar del Aljarafe, Spain, 2007 (Manzanillo)	480 g/L SL	0.41 0.50	0.036 0.048	1132 1045	2	11	2.2	Fernandez, 2009, 07 D OL BY P/A (M-352734-01-1)
07 D OL BY P03 La Puebla de Cazalla, Spain, 2007 (Hojiblanca)	480 g/L SL	0.35 0.47	0.036 0.048	974 983	2	11	2.5	Fernandez, 2009, 07 D OL BY P/A (M-352734-01-1)
07 D OL BY P04 Herrera, Spain, 2007 (Hojiblanca)	480 g/L SL	0.37 0.47	0.036 0.048	1020 981	2	11	1.6	Fernandez, 2009, 07 D OL BY P/A (M-352734-01-1)
08-2053-01 Sevilla, Spain, 2008 (Manzanillo)	480 g/L SL	0.48 0.62	0.044 0.057	1100 1100	2	11	0.90	Billian, 2009, 08-2053 (M-350265-02-1)
08-2053-02 Osuna, Spain, 2008 (Manzanillo)	480 g/L SL	0.48 0.62	0.044 0.057	1100 1100	2	11	2.60	Billian, 2009, 08-2053 (M-350265-02-1)
08-2053-03 Antequera, Spain, 2008 (Hojiblanco)	480 g/L SL	0.48 0.62	0.044 0.057	1100 1100	2	11	0.85	Billian, 2009, 08-2053 (M-350265-02-1)
08-2053-03 La Rambla, Spain, 2008 (Hojiblanco)	480 g/L SL	0.48 0.62	0.044 0.057	1100 1100	2	10	0.98	Billian, 2009, 08-2053 (M-350265-02-1)

Pineapple

Pineapple plants may be treated early to induce flowering or close to harvest to induce ripening/colouration of the pineapple fruit. The pre-flowering application is not expected to result in measurable residues. Treatment for fruit ripening/colouration close to harvest (typical PHI 1–14 days) is the most critical use and will result in the highest residues in the fruit. In the trials conducted in Brazil, Costa Rica and Côte d'Ivoire, pineapples have been treated close to harvest for fruit ripening/colouration.

Five supervised trials were conducted in Brazil. The plots were sprayed with a 240 g/L SL formulation once at 0.96 kg ai/ha or 1.92 kg ai/ha. All samples were analysed using ethylene release method (Method 11-94 for the 2005 trials). The maximum period of frozen storage of frozen samples was 1.5 months.

Two supervised trials have been conducted in 1998 in Costa Rica. The plots were sprayed with a 480 g/L SL formulation at an application rate of 1.59 kg ai/ha. Samples were separated into flesh and peel, after removal of the crown. The maximum period of storage of frozen samples at < –18 ºC was 3 months.

Two supervised trials were conducted in 1997 and 1999 in Côte d'Ivoire. The plots were sprayed with a 480 g/L SL formulation at a rate of 1.43–1.44 kg ai/ha.

Samples of peel and flesh of pineapple fruit from trials in Costa Rica and Côte d'Ivoire were analysed using method HVA 12/89. Residues in whole fruit were determined by calculation

from the residues in peel and flesh. The maximum period of storage of frozen samples was 6 months.

Six supervised trials have been conducted in 1989 in the USA (Hawaii). Four trials were conducted in Oahu, and two in Maui (no specific description about the locations). Each plot was divided into three subplots which were sprayed with a 480 g/L SL formulation at a rate of 2.24 + 1.12 kg ai/ha or 2× 2.24 kg ai/ha. Samples were analysed using method SOP 90070. The maximum period of storage of frozen samples was 11 months.

Table 54 Ethephon residues in pineapples resulting from supervised trials in Brazil, Costa Rica, Côte d'Ivoire and the USA

PINEAPPLE Trial No Country, year (Variety)	Application					DALT days	Portion analysed	Ethephon mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No				
GAP, Belize, El Salvador, Honduras, Domenican Rep	480 g/L SL	1.92 [a]		2000–3000	1–2	7–14			[a] Can be divided into two applications (i.e., seasonal max: 1.92)
GAP, Brazil	720 g/L SL	0.94		30–500	1	14			
GAP, Costa Rica	480 SL	1.2		2000–3000	2	1			
GAP, Costa Rica, Panama, Guatemala	720 g/L SL	0.94		2000–3000	1	7–14			
GAP, Costa Rica, Panama, Guatemala	720 g/L SL	1.2		1000	1	7–14			
GAP, Kenya	480 g/L SL	1.92		3000	1	7			
BRAZIL									
039/94PC-01 Sao Paolo, Brazil, 1994 (variety not reported)	240 g/L SL	0.96	–	–	1	0 4 8 13 18	Fruit	0.47 0.41 0.46 0.20 0.13	Garcia, 1994, CP-1997 PA-081/94 (M-188144-02-1)
	240 g/L SL	1.90	–	–	1	0 4 8 13 18	Fruit	1.12 0.87 1.21 0.90 0.48	
060/96 PC-1 Faz Sao Carlos-Holambra, Brazil, 1996 (Pérola)	240 g/L SL	0.96	0.24	400	1	14	Fruit	< 0.05	Guimaraes, 1997, 4170 (M-421140-01-1)
	240 g/L SL	1.92	0.48	400	1	14	Fruit	< 0.05	
HR05BRA0004-P1 Frutal MG, Brazil, 2005 (Havaiana)	240 g/L SL	0.96	0.24	400	1	14	Fruit	0.15	Galhiane, Santos, 2005, RA-966/05 (M-284613-02-1)
	240 g/L SL	1.92	0.48	400	1	14	Fruit	0.22	
HR05BRA0004-P2 Uberlandia MG, Brazil, 2005 (Havai)	240 g/L SL	0.96	0.24	400	1	14	Fruit	0.11	Galhiane, Santos, 2005, RA-967/05 (M-284618-02-1)
	240 g/L SL	1.92	0.48	400	1	14	Fruit	0.21	

PINEAPPLE Trial No Country, year (Variety)	Application					DALT days	Portion analysed	Ethephon mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No				
HR05BRA0004-P3 Ribeirao SP, Brazil, 2005 (Havai)	240 g/L SL	0.96	0.24	400	1	14	Fruit	0.19	Galhiane, Santos, 2005, RA-968/05 (M-284623-02-1)
	240 g/L SL	1.92	0.48	400	1	14	Fruit	0.24	
Costa Rica									
98622XX1 Buenos Aires, Costa Rica, 1998 (Del Monte Gold)	480 g/L SL	1.59	0.13	1273	1	0 2 3 7 0 2 3 7 0 2 3 7	Pulp Peel Whole fruit, calculated	< 0.10 0.11 < 0.10 < 0.10 0.38 0.41 0.13 < 0.10 0.19 0.20 0.11 < 0.10	Maestracci, 1998, R&D/CRLD/ AN/msa/ 9816197 (M-165714-01-1)
98622XX2 Buenos Aires, Costa Rica, 1998 (Del Monte Gold)	480 g/L SL	1.59	0.13	1215	1	0 2 3 7 0 2 3 7 0 2 3 7	Pulp Peel Whole fruit, calculated	< 0.10 < 0.10 < 0.10 < 0.10 0.14 < 0.10 < 0.10 < 0.10 0.11 < 0.10 < 0.10 < 0.10	Maestracci, 1998, R&D/CRLD/ AN/msa/ 9816197 (M-165714-01-1)
Côte d'Ivoire									
97766CI1 Yamoussoukro, Côte d'Ivoire, 1997 (Cayenne Lisse)	480 g/L SL	1.43	0.048	2978	1	0 2 3 7 0 2 3 7 0 2 3 7	Pulp Peel Whole fruit, calculated	< 0.10 < 0.10 < 0.10 < 0.10 0.51 0.31 0.64 0.13 0.21 0.16 0.28 0.11	Maestracci, 1998, R&D/CRLD/ AN/msa/ 9816152 (M-165702-02-1)

PINEAPPLE Trial No Country, year (Variety)	Application					DALT days	Portion analysed	Ethephon mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No				
98761C1 Yamoussoukro Côte d'Ivoire, 1999 (Cayenne Lisse)	480 g/L SL	1.44	0.048	3000	1	0 2 3 7 0 2 3 7 0 2 3 7	Pulp Peel Whole fruit, calculated	0.21 0.25 0.13 0.13 1.7 1.6 1.6 2.7 0.72 0.67 0.59 0.97	Baudet 1998, R&D/CRLD/ AN/mr/ 9916533 (M-179309-01-1)
USA									
89-130-P2 Honolulu Co, HI, USA, 1989 (Smooth Cayenne)	480 g/L SL	2.24 1.12	0.24 0.12	935 935	2	1 2 4 8	Whole fruit	0.06, 0.08, 0.15 [a] 0.05 0.04 0.03	Nygren, 1992, USA89E27, [M- 187578-01-1
89-130-P3 Honolulu Co, HI, USA,1989 (Smooth Cayenne)	480 g/L SL	2.24 2.24	0.24 0.24	935 935	2	1 2 4 8	Whole fruit	0.22 0.12 0.13 0.08	Nygren, 1992, USA89E27, [M- 187578-01-1
89-131-P2 Honolulu Co, HI, USA, 1989 (Smooth Cayenne)	480 g/L SL	2.24 1.12	0.24 0.12	935 935	2	1 2 4 8	Whole fruit	0.17, 0.11, 0.22 0.11 0.03 < 0.02	Nygren, 1992, USA89E27, [M- 187578-01-1
89-131-P3 Honolulu Co, HI, USA, 1989 (Smooth Cayenne)	480 g/L SL	2.24 2.24	0.24 0.24	935 935	2	1 2 4 8	Whole fruit	0.38 0.07 0.09 0.06	Nygren, 1992, USA89E27, [M- 187578-01-1
89-132-P2 Oahu Co., HI, USA, 1989 (Smooth Cayenne)	480 g/L SL	2.24 1.12	0.24 0.12	935 935	2	1 2 4 8	Whole fruit	n.a. 0.29 0.32 0.32	Nygren, 1992, USA89E27, [M- 187578-01-1
89-132-P3 Oahu Co., HI, USA, 1989 (Smooth Cayenne)	480 g/L SL	2.24 2.24	0.24 0.24	935 935	2	1 2 4 8	Whole fruit	0.67 0.41 0.98 0.72	Nygren, 1992, USA89E27, [M- 187578-01-1
89-133-P2 Oahu Co., HI, USA, 1989 (Smooth Cayenne)	480 g/L SL	2.24 1.12	0.24 0.12	935 935	2	1 2 4 8	Whole fruit	0.52, 0.71, 0.72 0.67 0.42 0.27	Nygren, 1992, USA89E27, [M- 187578-01-1
89-133-P3 Oahu Co., HI, USA,1989 (Smooth Cayenne)	480 g/L SL	2.24 2.24	0.24 0.24	935 935	2	1 2 4 8	Whole fruit	1.27 0.86 0.75 0.69	Nygren, 1992, USA89E27, [M- 187578-01-1

PINEAPPLE Trial No Country, year (Variety)	Application					DALT days	Portion analysed	Ethephon mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No				
89-134-P2 Maui Co., HI, USA, 1989 (Champaka)	480 g/L SL	2.45- 2.56 1.12	0.26- 0.27 0.12	935 935	2	1 2 4 8	Whole fruit	0.30, 0.19, 0.28 0.17 0.32 0.23	Nygren, 1992, USA89E27, [M- 187578-01-1
89-134-P3 Maui Co., HI, USA, 1989 (Champaka)	480 g/L SL	2.24 2.24	0.24 0.24	935 935	2	1 2 4 8	Whole fruit	0.62 0.40 0.36 0.76	Nygren, 1992, USA89E27, [M- 187578-01-1
89-135-P2 Maui Co., HI, USA, 1989 (Champaka)	480 g/L SL	2.62- 2.99 1.12	0.28- 0.32 0.12	935 935	2	1 2 4 8	Whole fruit	0.33, 0.42, 0.35 0.11 0.16 0.17	Nygren, 1992, USA89E27, [M- 187578-01-1
89-135-P3 Maui Co., HI, USA, 1989 (Champaka)	480 g/L SL	2.24 2.24	0.24 0.24	935 935	2	1 2 4 8	Whole fruit	0.74 0.26 0.59 0.48	Nygren, 1992, USA89E27, [M- 187578-01-1

[a] Results of three subplots. The highest residue concentration is selected.

Tomato

A total of twelve supervised trials were conducted on outdoor (field) grown tomatoes in Greece, Italy, Portugal and Spain. A total of nine supervised trials were conducted on indoor tomatoes in France, the Netherlands and Spain in 1999, 2000 and 2001. A 480 g/L SL formulation was applied as a foliar spray to outdoor (field) tomatoes at BBCH 84–89 at a rate of 1.68 kg ai/ha, or to indoor tomatoes at BBCH 60–89 at 1.42–1.47 kg ai/ha. In the 1999–2001 studies, residues of ethephon were determined using method HVA SOP 10071. In the 2004 study, residues of ethephon were determined using method 00903, supplement E001. The maximum period of storage of frozen samples was 642 days (21 months).

Twelve supervised trials were conducted in 1989–1991 on outdoor (field) grown tomato and three trials in 2005 on indoor tomato in the USA. A 240 g/L SL formulation was applied as a single foliar spray to outdoor (field) tomatoes at a rate of 1.73–2.14 kg ai/ha, or to indoor tomatoes at 1.38–1.42 kg ai/ha. In one field tomato trial (89-138), ethephon had been applied prior to the trial commencing, and the total ethephon application rate was 2.43 kg ai/ha. Residues of ethephon from the trials reported in 1991, 1992 and 2008 were determined using method SOP 90070. Residues of ethephon from the trials reported in 1995 were determined using method EC-92-228 (ethylene release method). The maximum period of storage frozen samples at –15 °C was 26 months.

Table 55 Ethephon residues in tomatoes resulting from supervised trials in Europe and the USA.

TOMATO Trial No Country, year (Variety)	Application					DALT days	Ethephon mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No			
GAP, Italy	480 g/L SL	1.92 [b]		1000	1–2	7		)
EUROPE/OUTDOOR (FIELD)								
DR00EUS522 ESP0201 Brenes, Spain, 2000 (Inca)	480 g/L SL	1.68	0.17	1000	1	0 3 7	1.5 1.1 0.78	Hees, 2001, DR00EUS522 (M- 203527-01-1)
DR00EUS522 ITA0101 Bologna, Italy, 2000 (Nun 7491)	480 g/L SL	1.68	0.17	1000	1	0 3 7	1.2 0.23 0.24	Hees, 2001, DR00EUS522 (M- 203527-01-1)

TOMATO Trial No Country, year (Variety)	Application					DALT days	Ethephon mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No			
DR00EUS522 ITA0201 Andria, Italy, 2000 (Faino)	480 g/L SL	1.68	0.17	1000	1	0 3 7	1.6 0.65 0.78	Hees, 2001, DR00EUS522 (M-203527-01-1)
DR00EUS522 GRC0101, Korifi-Imathia, Greece, 2000 (Titano M)	480 g/L SL	1.68	0.17	1000	1	0 3 7	0.56 0.52 0.62	Hees, 2001, DR00EUS522 (M-203527-01-1)
01R773-1 Utrea Sevilla, Spain, 2001 (Odin)	480 g/L SL	1.68	0.34	500	1	0 3 7	1.6 0.95 0.45	Davies, 2002, 01R773 (M-215341-01-1)
01R773-2 Brenes Sevilla, Spain, 2001 (Inca)	480 g/L SL	1.68	0.34	500	1	0 3 7	0.93 0.85 0.68	Davies, 2002, 01R773 (M-215341-01-1)
01R773-3 Molfetta, Italy, 2001 (Denaro)	480 g/L SL	1.68	0.24	700	1	0 3 7	1.9 1.1 0.5	Davies, 2002, 01R773 (M-215341-01-1)
01R773-4 Vrachia-Tessaloniki, Greece, 2001 (Titano)	480 g/L SL	1.68	0.34	500	1	0 3 7	0.35 0.45 0.46	Davies, 2002, 01R773 (M-215341-01-1)
01R773-5 Korifi-Imathia, Greece, 2001 (Rio Grande)	480 g/L SL	1.68	0.34	500	1	0 3 7	0.58 0.65 0.40	Davies, 2002, 01R773 (M-215341-01-1)
R 2004 0468/9 Gava, Spain, 2004 (Malpica)	480 g/L SL	1.68	0.21	800	1	0 4 7	0.95 0.46 0.30	Bardel, 2005, RA-2065/04 (M-261821-01-1)
R 2004 0469/7 Aldeia, Portugal, 2004 (H-9661)	480 g/L SL	1.68	0.21	800	1	0 3 7 10	1.1 0.80 0.57 0.17	Bardel, 2005, RA-2065/04 (M-261821-01-1)
R 2004 0470/0 Bolognia, Italy, 2004 (Missouri)	480 g/L SL	1.68	0.21	800	1	0 3 7 10	1.2 1.7 0.55 0.49	Bardel, 2005, RA-2065/04 (M-261821-01-1)
EUROPE/INDOOR								
DR00EUI520 FRA0301 Marcellus, France, 2000 (Vekio)	480 g/L SL	1.44	0.096	1500	1	0 3 7	0.25 0.44 0.79	Hees, 2001, DR00EUI520 (M-202477-01-1)
DR00EUI520 FRA0302 Villefranche du Queyran, France, 2000 (Félicia)	480 g/L SL	1.44	0.096	1500	1	0 3 7	0.45 0.48 0.45	Hees, 2001, DR00EUI520 (M-202477-01-1)
00582NL1 Huissen, Netherlands, 1999 (Elegance)	480 g/L SL	1.42	0.095	1488	1	7	0.51	Ballesteros, 2002, R&D/CRLD/AN/0215069 (M-210410-01-1)
00582NL2 Ooserhout, Netherlands, 1999 (Tomcat)	480 g/L SL	1.47	0.095	1538	1	7	0.69	Ballesteros, 2002, R&D/CRLD/AN/0215069 (M-210410-01-1)

TOMATO Trial No Country, year (Variety)	Application					DALT days	Ethephon mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No			
01R791-1 Puebla de Vicar, Spain, 2001 (Eldiez)	480 g/L SL	1.44	0.12	1250	1	0 3 7	0.88 1.1 0.68	Davies, 2002, 01R791 (M-210553-01-1)
01R791-2 ND Zwaagdik, Netherlands, 2001 (Fergie (F6197))	480 g/L SL	1.44	0.096	1500	1	0 3 7	0.86 1.4 0.66	Davies, 2002, 01R791 (M-210553-01-1)
01R791-3 ND Zwaagdik,Netherlands, 2001 (Rapsodie)	480 g/L SL	1.44	0.096	1500	1	0 3 7	0.57 0.31 0.52	Davies, 2002, 01R791 (M-210553-01-1)
01R791-4 ND Zwaagdik,Netherlands, 2001 (Fergie (F6197))	480 g/L SL	1.44	0.096	1500	1	0 3 7	0.61 0.34 0.31	Davies, 2002, 01R791 (M-210553-01-1)
01R791-5 ND Zwaagdik,Netherlands, 2001 (Rapsodie)	480 g/L SL	1.44	0.096	1500	1	0 3 7	0.41 0.16 0.36	Davies, 2002, 01R791 (M-210553-01-1)
GAP, Canada	240 g/L SL	1.54		30–500	1	No specific PHI set, harvest at maturity, generally 14–21 days after treatment		
USA/OOUTDOOR (FIELD)								
89-119 Imperial Co., CA, USA, 1989 (U.C. 82)	240 g/L SL	1.75	2.3	76	1	0 3 7	0.18 (0.14, 0.21,0.18) [a] 0.10 (0.14, 0.06, 0.10) 0.09 (0.07, 0.12, 0.08)	Nygren, 1991, USA89E30 (M-187599-01-1)
89-120 Imperial Co., CA, USA, 1989 (U.C. 82)	240 g/L SL	2.14	1.05	204	1	0 3 7	0.48 (0.71, 0.47, 0.26) 0.44 (0.65, 0.34, 0.32) 0.27 (0.23, 0.42, 0.17)	Nygren, 1991, USA89E30 (M-187599-01-1)
89-136 Solano Co., CA, USA, 1989 (Sun Seed 5715)	240 g/L SL	1.80	1.9	93	1	3 7 14	0.66 (0.34, 1.1, 0.54) 0.92 (1.0, 0.81, 0.95) 0.69 (0.63, 0.72, 0.73)	Nygren, 1991, USA89E30 (M-187599-01-1)
89-137 Solano Co., CA, USA, 1989 (Sun Seed 5715)	240 g/L SL	2.00	0.97	206	1	3 7 14	0.02 < 0.02 (< 0.02, < 0.02, < 0.02) 0.15 (0.05, 0.17, 0.22)	Nygren, 1991, USA89E30 (M-187599-01-1)
89-138 Sacrament Co., CA, USA, 1989 (1643)	240 g/L SL	1.27 1.16	1.3 0.93	93 125	2	3	0.73 (0.68, 0.86, 0.64)	Nygren, 1991, USA89E30 (M-187599-01-1)
90-492 Collier Co., FL, USA, 1990 (Sunny)	240 g/L SL	1.80	0.97	187	1	3 7 14	< 0.02 (< 0.02, < 0.02, < 0.02) < 0.02 (< 0.02, < 0.02, < 0.02) < 0.02 (< 0.02, < 0.02, < 0.02)	Nygren, 1992, USA90E16 (M-187596-01-1)
90-493 Collier Co., FL, USA, 1990 (Sunny)	240 g/L SL	1.80	1.9	93	1	3 7 14	0.32 (0.16, 0.47, 0.32) 0.06 (0.05, < 0.02, 0.11) 0.06 (0.05, 0.05, 0.07)	Nygren, 1992, USA90E16 (M-187596-01-1)

TOMATO Trial No Country, year (Variety)	Application					DALT days	Ethephon mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No			
91-307 Stanislaus Co., CA, USA, 1991 (Ace)	240 g/L SL	1.75	0.53	329	1	3 7 14	1.66 (1.64, 2.24, 1.09) 0.97 (0.51, 1.24, 1.16) 0.63 (0.78, 0.66, 0.44)	Nygren, 1995, USA91E16 (M-187891-01-1)
91-308 Stanislaus Co., CA, USA, 1991 (Ace)	240 g/L SL	1.76	1.36	129	1	3 7 11	1.24 (1.06, 1.29, 1.37) 0.81 (0.93, 0.66, 0.83) 0.37 (0.29, 0.44, 0.39)	Nygren, 1995, USA91E16 (M-187891-01-1)
91-309 Stanislaus Co., CA, USA, 1991 (Ace)	240 g/L SL	1.80	0.55	329	1	3 7 14	0.55 (0.48, 0.61) 0.35 (0.43, 0.25, 0.36) 0.15 (0.22, 0.12, 0.12)	Nygren, 1995, USA91E16 (M-187891-01-1)
91-310 Stanislause Co., CA, USA, 1991 (Ace)	240 g/L SL	1.73	1.34	129	1	3 7 14	0.62 (0.69, 0.69, 0.49) 0.68 (0.75, 0.40, 0.89) 0.67 (0.40, 0.34, 1.27)	Nygren, 1995, USA91E16 (M-187891-01-1)
91-311 Collier Co., FL, USA, 1991 (BHN)	240 g/L SL	1.80	0.38	469	1	3 7 10	0.30 (0.17, 0.36, 0.37) 0.08 (0.12, 0.07, 0.04) 0.05 (0.06, 0.05, 0.04)	Nygren, 1995, USA91E16 (M-187891-01-1)
USA/INDOOR								
00250.05-CO13 Fort Collins, CO, USA, 2005 (Trust F1)	240 g/L SL	1.42	0.75	189	1	1 2	0.58 (0.56, 0.60) 0.70 (0.83, 0.56)	Dorschner, 2008, IR4 PR No 00250 (M-301374-01-1)
00250.05-FL37 Citra, FL, USA, 2005 (FL47)	240 g/L SL	1.41	0.49	289	1	1 2	0.60 (0.32, 0.88) 0.98 (0.85, 1.1)	Dorschner, 2008, IR4 PR No 00250 (M-301374-01-1)
00250.05-TX25 Weslaco, TX, USA, 2005 (Super sweet 100)	240 g/L SL	1.38	0.41	340	1	1 2	1.70 (2.0, 1.4) 1.80 (2.0, 1.6)	Dorschner, 2008, IR4 PR No 00250 (M-301374-01-1)

[a] Mean residue. Analytical results of replicate samples were in parentheses

[b] Can be divided into two applications (i.e., seasonal max, 1.92)

Cereal grains

Barley

A total of fifty-three supervised trials were conducted in Europe with a foliar spray:

- Fourteen at a rate of 1× 480 g ai/ha, application at BBCH 45–51 (one trial at BBCH 55), (determination using method HVA SOP 10071)
- Eight trials at a rate of 1× 225 g ai/ha, application at BBCH 39–41, (determination using method 00918)
- Ten trials at a rate of 1× 380 g ai/ha, application at BBCH 37–39, (determination using method 00918)
- Five trials at a rate of 1× 670–720 g ai/ha (nominal rate 720 g ai/ha), application at BBCH 37–39 (determination using method 00918).

In all studies, the maximum period of storage of frozen samples at around –18 °C was 14 months.

A total of 16 new trials were conducted to determine the magnitude of the residues of ethephon in/on barley (grain, green materials and straw) after one spraying application with ethephon SL 480 during the 2013 and 2014 seasons with one foliar application in Europe:

- Eight trials at a rate of 480 g ai/ha at BBCH 39
- Eight trials at 480 g ai/ha at BBCH 51.

The samples were stored frozen (–18 °C) for a maximum of 647 days. In these sixteen trials residues of ethephon and HEPA were determined by Method 01429, HPLC-MS/MS method in which grains and straw were extracted first with methanol and then by a mixture of concentrated hydrochloric acid and water (1/7, v/v) at 50 °C to convert conjugated ethephon and HEPA to free ethephon and HEPA. The extracts and acid hydrolysates were combined for analysis.

Table 56 Ethephon residues in barley grains resulting from supervised trials in Europe

BARLEY Trial No Country, year (Variety)	Application					DALT days	Ethephon mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No			
GAP, Germany	660 g/L SL	0.462		100–300	1	–	Application timing BBCH 32–49	
GAP, UK	480 g/L SL	0.48		100–400	–	–	Application timing BBCH 32–49 Maximum total rate 0.48 kg ai/ha	
DR00EUS525 ITA0101 Bologna, Italy, 2000 (Express)	480 g/L SL	0.48 (BBCH 47)	0.16	300	1	48	< 0.05	Hees, 2001, DR00EUS525 (M-199982-01-1)
DR00EUS525 ITA0102 S. Mauro Pascoli, Italy, 2000 (Extra)	480 g/L SL	0.48 (BBCH 45)	0.16	300	1	47	< 0.05	Hees, 2001, DR00EUS525 (M-199982-01-1)
00547BX1 Marignac, France, 2000 (Sunrise)	480 g/L SL	0.48 (BBCH 45)	0.14	333	1	52	0.06	Ballasteros, 2001, R&D/CRLD/AN/mr/0115430 (M-208093-01-1)
00547TL1 Gardouch, France, 2000 (Esterel)	480 g/L SL	0.48 (BBCH 47)	0.19	250	1	62	0.06	Ballasteros, 2001, R&D/CRLD/AN/mr/0115430 (M-208093-01-1)
01R761-1 Ronchères, France, 2001 (Platine)	480 g/L SL	0.48 (BBCH 47)	0.19	250	1	69	< 0.05	Davies, 2002, 01R761 (M-209901-01-1)
01R761-2 Hargicourt, France, 2001 (Muscat)	480 g/L SL	0.48 (BBCH 49)	0.19	250	1	54	0.05	Davies, 2002, 01R761 (M-209901-01-1)
01R761-3 Braintree, UK, 2001 (Regina)	480 g/L SL	0.48 (BBCH 55)	0.19	252	1	58	0.23	Davies, 2002, 01R761 (M-209901-01-1)
01R761-4 Weilerswist, Germany, 2001 (Theresa)	480 g/L SL	0.48 (BBCH 51)	0.16	300	1	60	< 0.05	Davies, 2002, 01R761 (M-209901-01-1)
01R761-5 Zschortau, Germany, 2001 (Landi)	480 g/L SL	0.48 (BBCH 49)	0.16	300	1	66	< 0.05	Davies, 2002, 01R761 (M-209901-01-1)

BARLEY Trial No Country, year (Variety)	Application					DALT days	Ethephon mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No			
01R771-1 Senestis, France, 2001 (Platine)	480 g/L SL	0.48 (BBCH 45)	0.19	250	1	64	< 0.05	Davies, 2002, 01R771 (M-210307-01-1)
01R771-2 Toussieux, France, 2001 (Ladoga)	480 g/L SL	0.48 (BBCH 47)	0.19	250	1	63	< 0.05	Davies, 2002, 01R771 (M-210307-01-1)
01R771-3 Genas, France, 2001 (Ladoga)	480 g/L SL	0.48 (BBCH 47)	0.19	250	1	57	< 0.05	Davies, 2002, 01R771 (M-210307-01-1)
01R771-4 Alberone Di Cento, Italy, 2001 (Sonora)	480 g/L SL	0.48 (BBCH 47)	0.14	350	1	35	0.29	Davies, 2002, 01R771 (M-210307-01-1)
01R771-5 Xirochori-Kilkis, Greece, 2001 (Athinaida)	480 g/L SL	0.48 (BBCH 47)	0.16	300	1	50	< 0.05	Davies, 2002, 01R771 (M-210307-01-1)
GAP, France	480 g/L SL	0.48		100–200	1	56	Application timing BBCH 32–39	
R 2004 0577/4 Monospita, Greece, 2004 (Kannon (distiho))	450 g/L SL [a]	0.38 (BBCH 39)	0.125	300	1	54	< 0.05	Bardel & Wolters, 2005, RA-2093/04 (M-251235-01-1)
R 2004 0578/2 Bologna, Italy, 2004 (Marjorie)	450 g/L SL [a]	0.38 (BBCH 39)	0.125	300	1	54	< 0.05	Bardel & Wolters, 2005, RA-2093/04 (M-251235-01-1)
R 2004 0579/0 Vouillé, France, 2004 (Scarlette)	450 g/L SL [a]	0.38 (BBCH 39)	0.125	300	1	56	< 0.05	Bardel & Wolters, 2005, RA-2093/04 (M-251235-01-1)
R 2004 0580/4 Balaguer, Spain, 2004 (Prestige)	450 g/L SL [a]	0.38 (BBCH 39)	0.125	300	1	53	< 0.05	Bardel & Wolters, 2005, RA-2093/04 (M-251235-01-1)
R 2004 0572/3 Lund, Sweden, 2004 (Bombay)	450 g/L SL [a]	0.38 (BBCH 39)	0.125	300	1	80	< 0.05	Bardel & Wolters, 2005, RA-2092/04 (M-251366-01-1)
R 2004 0573/1 Leverkusen, Germany, 2004 (Condesse)	450 g/L SL [a]	0.38 (BBCH 37)	0.125	300	1	85	< 0.05	Bardel & Wolters, 2005, RA-2092/04 (M-251366-01-1)
R 2004 0575/8 Weri-Obernergstraße, Germany, 2004 (Intro)	450 g/L SL [a]	0.38 (BBCH 39)	0.125	300	1	77	< 0.05	Bardel & Wolters, 2005, RA-2092/04 (M-251366-01-1)
R 2004 0576/6 Fresnoy les Roye, France, 2004 (Esterel)	450 g/L SL [a]	0.38 (BBCH 39)	0.125	300	1	67	< 0.057	Bardel & Wolters, 2005, RA-2092/04 (M-251366-01-1)
R 2006 0126/3 Neuville de Poitou, France, 2006 (Abondance)	450 g/L SL [a]	0.38 (BBCH 39)	0.125	300	1	56 59	< 0.05 (ear) < 0.05	Billian & Erler, 2007, RA-2519/06 (M-290151-01-1)
R 2006 0299/5 Tarascon, France, 2006 (Baraka)	450 g/L SL [a]	0.38 (BBCH 39)	0.125	300	1	55 60	0.22 (ear) 0.09	Billian & Erler, 2007, RA-2519/06 (M-290151-01-1)

BARLEY Trial No Country, year (Variety)	Application					DALT days	Ethephon mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No			
GAP, Poland	480 g/L SL	0.72		150– 300	1	–	Application timing BBCH 32–39	
R 2006 0117/4 Beuvraignes, France, 2006 (Colibri)	480 g/L SL	0.67 (BBCH 37)	0.22	300	1	56 76	< 0.05 < 0.05	Billian & Telscher, 2007, RA-2515/06 (M-294373-01-1)
R 2006 0286/3 Welver-Flerke, Germany, 2006 (Duet)	480 g/L SL	0.67 (BBCH 37)	0.22	300	1	55 68	< 0.05 < 0.05	Billian & Telscher, 2007, RA-2515/06 (M-294373-01-1)
R 2006 0285/5 Hoxne/Nreye, UK, 2006 (Sequel)	480 g/L SL	0.67 (BBCH 39)	0.22	300	1	56 74	< 0.05 < 0.05	Billian & Telscher, 2007, RA-2515/06 (M-294373-01-1)
R 2007 0172/1 Chaussy, France, 2007 (Sibéria)	480 g/L SL	0.72 (BBCH 37)	0.24	300	1	56 75	< 0.05 < 0.05	Billian, 2008, RA-2573/07 (M-311809-01-1)
R 2007 0181/0 Lund, Sweden, 2007 (Bombay)	480 g/L SL	0.72 (BBCH 37)	0.24	300	1	56 70	< 0.05 < 0.05	Billian, 2008, RA-2573/07 (M-311809-01-1)
GAP, France	450 g/L SL [a]	0.23		100– 200	1	–	Application timing BBCH 31–37	
R 2004 0581/2 Le Thil en Vexin, France, 2004 (Scarlet)	450 g/L SL [a]	0.23 (BBCH 39)	0.075	300	1	57	< 0.05	Bardel & Wolters, 2005, RA-2094/04 (M-249305-02-1)
R 2004 0582/0 Staffanstorp, Sweden, 2004 (Pasadena)	450 g/L SL [a]	0.23 (BBCH 39)	0.075	300	1	79	< 0.05	Bardel & Wolters, 2005, RA-2094/04 (M-249305-02-1)
R 2004 0583/9 Burscheid, Germany, 2004 (Scarlett)	450 g/L SL [a]	0.23 (BBCH 39)	0.075	300	1	61	< 0.05	Bardel & Wolters, 2005, RA-2094/04 (M-249305-02-1)
R 2004 0584/7 Gersthofen, Germany, 2004 (Ursa)	450 g/L SL [a]	0.23 (BBCH 39)	0.075	300	1	65	< 0.05	Bardel & Wolters, 2005, RA-2094/04 (M-249305-02-1)
R 2004 0585/5 Saint Germain sur Renon, France, 2004 (Nevada)	450 g/L SL [a]	0.23 (BBCH 41)	0.075	300	1	52	< 0.05	Bardel & Wolters, 2005, RA-2095/04 (M-251234-01-1)
R 2004 0586/3 Bologna, Italy, 2004 (Federal)	450 g/L SL [a]	0.23 (BBCH 39)	0.075	300	1	52	< 0.05	Bardel & Wolters, 2005, RA-2095/04 (M-251234-01-1)
R 2004 0587/1 Tarascon, France, 2004 (Baraka)	450 g/L SL [a]	0.23 (BBCH 39)	0.075	300	1	44	< 0.05	Bardel & Wolters, 2005, RA-2095/04 (M-251234-01-1)
R 2004 0589/8 Golegã, Portugal, 2004 (Scarlett)	450 g/L SL [a]	0.23 (BBCH 39)	0.075	300	1	61	< 0.05	Bardel & Wolters, 2005, RA-2095/04 (M-251234-01-1)

[a] 450 g/L SL formulation (150 g/L ethephon + 300 g/L chlormequat-chloride)

Table 57 Ethephon and HEPA residues in barley grains resulting from supervised trials in Europe obtained using an analytical method involving acid hydrolysis/extraction

BARLEY Trial No Country, year (Variety)	Application					DALT days	Ethephon mg/kg	HEPA mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No				
GAP, Germay	660 g/L SL	0.462		100–300	1	–	Application timing BBCH 32–49		
GAP, UK	480 g/L SL	0.48		100–400	–	–	Application timing BBCH 32–49 Maximum total rate 0.48 kg ai/ha		
13-2027-01 Burscheid, Germany, 2013 (Duett)	480 SL	0.48 (BBCH 51)	0.16	300	1	59	0.13	0.019 (c, 0.013)	Schulte & Berkum, 2015, 13-2027 M-526906-01-1
13-2027-02 Diegem, Belgium, 2013 (Meridian)	480 SL	0.51 (BBCH 51)	0.19	267	1	55	0.067	< 0.01	Schulte & Berkum, 2015, 13-2027 M-526906-01-1
13-2027-03 Mijdrecht, Netherlands, 2013 (Malabar)	480 SL	0.48 (BBCH 51)	0.16	300	1	56	0.73	0.086	Schulte & Berkum, 2015, 13-2027 M-526906-01-1
13-2027-04 Cambridge, United Kingdom, 2013 (Cassata)	480 SL	0.48 (BBCH 51)	0.24	200	1	68	0.23	0.055	Schulte & Berkum, 2015, 13-2027 M-526906-01-1
14-2022-01 Langenfeld, Germany, 2014 (Naomie)	480 SL	0.54 (BBCH 51)	0.16	336	1	78	0.031	0.016	Schulte & Berkum, 2015, 14-2022
14-2022-02 Burscheid, Germany, 2014 (Leibnitz)	480 SL	0.48 (BBCH 51)	0.16	300	1	64	0.41	0.055 (c, 0.054)	Schulte & Berkum, 2015, 14-2022
14-2022-03 Lyon Cedex 09, France, 2014 (Obite Winter)	480 SL	0.48 (BBCH 51)	0.16	300	1	56	0.090	0.021	Schulte & Berkum, 2015, 14-2022
14-2022-04 Cambridge CB4 0WB, United Kingdom, 2014 (Cassatta Typical UK variety)	480 SL	0.48 (BBCH 55)	0.24	200	1	73	0.16	0.047 (c, 0.011)	Schulte & Berkum, 2015, 14-2022
GAP, France	480 g/L SL	0.48		100–200	1	56	Application timing BBCH 32–39		
13-2028-01 Ceaux en Loudun, France, 2013 (Cervoise)	480 SL	0.48 (BBCH 39)	0.16	300	1	71	0.035	< 0.01	Schulte & Berkum, 2015, 13-2028 M-529491-01-1
13-2028-02 Les Franqueses del Valles, Spain, 2013 (Graphic)	480 SL	0.48 (BBCH 39)	0.16	400	1	72	0.21	0.069	Schulte & Berkum, 2015, 13-2028 M-529491-01-1
13-2028-03 Citavecchia, Italy, 2013 (Quench, Distichous barley)	480 SL	0.48 (BBCH 39)	0.16	300	1	62	0.041	0.012	Schulte & Berkum, 2015, 13-2028 M-529491-01-1
13-2028-04 Bologna, Italy, 2013 (Federal)	480 SL	0.48 (BBCH 39)	0.24	350	1	64	0.021	0.070 (c, 0.060)	Schulte & Berkum, 2015, 13-2028 M-529491-01-1
14-2020-01 Ceaux en Loudun, France, 2014 (Limpid Winter Barley)	480 SL	0.48 (BBCH 39)	0.16	300	1	72	0.14	0.026	Schulte & Berkum, 2015, 14-2020

BARLEY Trial No Country, year (Variety)	Application					DALT days	Ethephon mg/kg	HEPA mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No				
14-2020-02 Les Franqueses del Valles, Spain, 2014 (Graphic winterbarley)	480 SL	0.41 (BBCH 43)	0.12	342	1	64	0.039	0.013	Schulte & Berkum, 2015, 14-2020
14-2020-03 Bologna, Italy, 2014 (Lutece Winter variety)	480 SL	0.48 (BBCH 39)	0.12	400	1	64	0.047	< 0.01	Schulte & Berkum, 2015, 14-2020
14-2020-04 Kristoni Village, Greece, 2014 (Mucho Early, six row, USA)	480 SL	0.48 (BBCH 39)	0.16	300	1	63	0.034	0.014	Schulte & Berkum, 2015, 14-2020

Rye

Nine supervised trials were conducted in 2006–2007 in France, UK, Sweden and Germany. A 480 g/L SL formulation was applied as a foliar spray to rye at BBCH 49 at a rate of 0.67–0.72 kg ai/ha. Samples of green material were collected after 0, 7 and 20-21 days, ears and rest of plant after 42–49 days, and mature grain and straw after 70–103 days. Residues of ethephon were determined using method 00918.

The maximum period of storage of frozen samples at –18 °C was 11.4 months.

Table 58 Ethephon residues in rye grains resulting from supervised trials in Europe

RYE Trial No. Country, year (Variety)	Application					DALT days	Portion analysed	Ethephon mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No				
GAP, Germany	660 g/L SL	0.73		100–300	1	–	Application timing BBCH 37–49		
R 2006 0119/0 Le Plessier, France, 2006 (Picasso)	480 g/L SL	0.67 (BBCH 49)	0.22	300	1	49 75	Ear Grain	0.08 < 0.05	Billian & Telscher, 2007, RA-2516/06 (M-294780-02-1)
R 2006 0287/1 Thetford, UK, 2006 (Ursus)	480 g/L SL	0.67 (BBCH 49)	0.22	300	1	49 88	Ear Grain	0.11 0.07	Billian & Telscher, 2007, RA-2516/06 (M-294780-02-1)
R 2006 0289/8 Svedala, Sweden, 2006 (Matador)	480 g/L SL	0.67 (BBCH 49)	0.22	300	1	49 71	Ear Grain	0.07 < 0.05	Billian & Telscher, 2007, RA-2516/06 (M-294780-02-1)
R 2006 0290/1 Anneville Ambourville, France, 2006 (Canovus)	480 g/L SL	0.67 (BBCH 49)	0.22	300	1	49 70	Ear Grain	0.10 0.06	Billian & Telscher, 2007, RA-2516/06 (M-294780-02-1)
R 2006 0292/8 Beiersdorf, Germany, 2006 (Rekrut)	480 g/L SL	0.67 (BBCH 49)	0.22	300	1	49 77	Ear Grain	0.14 0.06	Billian & Telscher, 2007, RA-2516/06 (M-294780-02-1)
R 2007 0174/8 Le Plessier Rosainvillers, France, 2007 (Picasso)	480 g/L SL	0.72 (BBCH 49)	0.24	300	1	49 85	Ear Grain Straw	0.12 < 0.05	Billian, Erler & Wolters, 2008, RA-2574/07 (M-318501-01-1)

RYE Trial No. Country, year (Variety)	Application					DALT days	Portion analysed	Ethephon mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No				
R 2007 0182/9 Burscheid, Germany, 2007 (Fernando)	480 g/L SL	0.72 (BBCH 49)	0.24	300	1	49 86	Ear Grain	< 0.05 < 0.05	Billian, Erler & Wolters, 2008, RA-2574/07 (M-318501-01-1)
R 2007 0184/5 Anneville Ambourville, France, 2007 (Caroass)	480 g/L SL	0.72 (BBCH 49)	0.24	300	1	48 83	Ear Grain	0.09 < 0.05	Billian, Erler & Wolters, 2008, RA-2574/07 (M-318501-01-1)
R 2007 0183/7 Thetford, UK, 2007 (Visello)	480 g/L SL	0.72 (BBCH 49)	0.24	300	1	42 103	Ear Grain	0.06 < 0.05	Billian, Erler & Wolters, 2008, RA-2574/07 (M-318501-01-1)

Wheat

A total of forty-three supervised trials were conducted in Europe with one foliar application:

- Nine trials at a rate of 480 g ai/ha, application at BBCH 37–39 (method HVA SOP 10071)
- Five trials at a rate of 480 g ai/ha, application at BBCH 49–51 (method HVA SOP 10071)
- Eight trials at a rate of 375 g ai/ha, application at BBCH 37 (one trial at BBCH 41–45), (method 00918)
- Five trials at a rate of 670–720 g ai/ha (nominal rate 720 g ai/ha), application at BBCH 39 (one trial at BBCH 49) (method 00918).

In all above studies, the maximum period of storage of frozen samples at around –18 °C was 12.2 months.

During the 2013 and 2014 seasons, a total of 16 trials were conducted in Europe to determine the magnitude of the residues of ethephon in/on wheat, soft (grain, green materials and straw) after one spraying application with Ethephon SL 480:

- Eight at a rate of 480 g ai/ha at BBCH 51
- Eight at a rate of 480 g ai/ha at BBCH 39.

Residues of ethephon in trials in 2013 and 2014 were determined by Method 01429, HPLC-MS/MS method in which grains and straw were extracted first with methanol and then by a mixture of concentrated hydrochloric acid and water (1/7, v/v) at 50 °C to convert conjugated ethephon and HEPA to free ethephon and HEPA. The extracts and acid hydrolysates are combined for analysis.

The samples were stored frozen (–18 °C) for a maximum of 713 days.

Table 59 Ethephon residues in wheat grains resulting from supervised trials in Europe

WHEAT Trial No. Country, year (Variety)	Application					DALT days	Portion analysed	Ethephon mg/kg	Reference
	Form (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No				
GAP, Germany	660 g/L SL	0.46		100–300	1	–	Application timing BBCH 37–51		
01R762-1 Braslou, France, 2001 (Isengrain)	480 g/L SL	0.48 (BBCH 51)	0.19	250	1	70	Grain	< 0.05	Davies, 2002, 01R762 (M-210306-01-1)

WHEAT Trial No. Country, year (Variety)	Application					DALT days	Portion analysed	Ethephon mg/kg	Reference
	Form (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No				
01R762-2 Courdoux, France, 2001 (Ritmo)	480 g/L SL	0.48 (BBCH 49)	0.24	200	1	66	Grain	< 0.05	Davies, 2002, 01R762 (M-210306-01-1)
01R762-3 Cambridge, UK, 2001 (Claire)	480 g/L SL	0.48 (BBCH 49)	0.16	302	1	72	Grain	< 0.05	Davies, 2002, 01R762 (M-210306-01-1)
01R762-4 Weilerswist, Germany, 2001 (Drifter)	480 g/L SL	0.48 (BBCH 49)	0.16	300	1	66	Grain	0.06	Davies, 2002, 01R762 (M-210306-01-1)
01R762-5 Zschortau, Germany, 2001 (Petrus)	480 g/L SL	0.48 (BBCH 49)	0.16	300	1	71	Grain	< 0.05	Davies, 2002, 01R762 (M-210306-01-1)
GAP, France	480 g/L SL	0.48		100–200	1	70	Application timing BBCH 32–39		
00548BX1 Chaunac, France, 2000 (Aztec)	480 g/L SL	0.48 (BBCH 38)	0.14	333	1	90	Grain	< 0.05	Ballasteros, 2002, R&D/CRLD/AN/mr/0115433 (M-208087-01-1)
00548LY1 La Boisse, France, 2000 (Cyrano)	480 g/L SL	0.48 (BBCH 39)	0.15	320	1	78	Grain	< 0.05	Ballasteros, 2002, R&D/CRLD/AN/mr/0115433 (M-208087-01-1)
00549BX1 Tugeras, France, 2000 (Hyno-valea)	480 g/L SL	0.47 (BBCH 39)	0.14	333	1	90	Grain	< 0.05	Ballasteros, 2002, R&D/CRLD/AN/mr/0115434 (M-208091-01-1)
00549TL1 Baziege, France, 2000 (Tremie)	480 g/L SL	0.48 (BBCH 37-39)	0.17	278	1	91	Grain	< 0.05	Ballasteros, 2002, R&D/CRLD/AN/mr/0115434 (M-208091-01-1)
01R772-1 Boe, France, 2001 (Soissons)	480 g/L SL	0.48 (BBCH 39)	0.19	250	1	74	Grain	< 0.05	Davies, 2002, 01R772 (M-210308-01-1)
01R772-2 Saint Romain De Jeolienas, France, 2001 (Aztec)	480 g/L SL	0.48 (BBCH 39)	0.19	250	1	74	Grain	< 0.05	Davies, 2002, 01R772 (M-210308-01-1)
01R772-3 Dodici Morelli, Italy, 2001 (Centauro)	480 g/L SL	0.48 (BBCH 39)	0.14	350	1	57	Grain	< 0.05	Davies, 2002, 01R772 (M-210308-01-1)
01R772-4 Paradas Sevilla, Spain, 2001 (Simeto)	480 g/L SL	0.48 (BBCH 39)	0.16	300	1	78	Grain	< 0.05	Davies, 2002, 01R772 (M-210308-01-1)
01R772-5 Alcala de Guadaira Sevilla, Spain, 2001 (Sula)	480 g/L SL	0.48 (BBCH 39)	0.16	300	1	76	Grain	< 0.05	Davies, 2002, 01R772 (M-210308-01-1)
GAP, France	450 g/L SL [a]	0.38		100–200	1	–	Application timing BBCH 31–37		

WHEAT Trial No. Country, year (Variety)	Application					DALT days	Portion analysed	Ethephon mg/kg	Reference
	Form (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No				
R 2004 0564/2 Staffanstorp, Sweden, 2004 (Marshall)	450 g/L SL [a]	0.38 (BBCH 37)	0.13	300	1	85	Grain	< 0.05	Bardel, 2005, RA-2090/04 (M-251226-01-1)
R 2004 0565/0 Leverkusen, Germany, 2004 (Batis)	450 g/L SL [a]	0.38 (BBCH 37)	0.13	300	1	92	Grain	< 0.05	Bardel, 2005, RA-2090/04 (M-251226-01-1)
R 2004 0566/9 Werl-Oberbergstraße, Germany, 2004 (Winnetou)	450 g/L SL [a]	0.38 (BBCH 37)	0.13	300	1	81	Grain	< 0.05	Bardel, 2005, RA-2090/04 (M-251226-01-1)
R 2004 0567/7 Villettes, France, 2004 (Orvantis)	450 g/L SL [a]	0.38 (BBCH 37)	0.13	300	1	84	Grain	< 0.05	Bardel, 2005, RA-2090/04 (M-251226-01-1)
R 2004 0568/5 Kilkis, Greece, 2004 (Mexicalli)	450 g/L SL [a]	0.38 (BBCH 37)	0.12	300	1	57	Grain	< 0.05	Bardel, 2005, RA-2091/04 (M-251236-02-1)
R 2004 0569/3 Gargas, France, 2004 (Garric)	450 g/L SL [a]	0.38 (BBCH 37)	0.12	300	1	77	Grain	< 0.05	Bardel, 2005, RA-2091/04 (M-251236-02-1)
R 2004 0570/7 Brenes, Spain, 2004 (Don Pedro)	450 g/L SL [a]	0.38 (BBCH 41–45)	0.12	300	1	78	Grain	< 0.05	Bardel, 2005, RA-2091/04 (M-251236-02-1)
R 2004 0571/5 Pereiro/Alenquer, Portugal, 2004 (Sula)	450 g/L SL [a]	0.38 (BBCH 37)	0.12	300	1	82	Grain	< 0.05	Bardel, 2005, RA-2091/04 (M-251236-02-1)
GAP, Belgium	480 g/L SL	0.60		200–400	1	–	Application timing BBCH 37–45		
R 2006 0123/9 Chaussy, France, 2006 (Isengrain)	480 g/L SL	0.67 (BBCH 39)	0.22	300	1	56 64	Ear Grain	0.09 0.06	Billian & Telscher, 2007, RA-2517/06 (M-294528-01-1)
R 2006 0293/6 Bury St Edmunds, UK, 2006 (Einstein)	480 g/L SL	0.67 (BBCH 39)	0.22	300	1	56 68	Ear Grain Straw	< 0.05 < 0.05	Billian & Telscher, 2007, RA-2517/06 (M-294528-01-1)
R 2006 0294/4 Leverkusen, Germany, 2006 (Batis)	480 g/L SL	0.67 (BBCH 39)	0.22	300	1	56 73	Ear Grain	< 0.05 < 0.05	Billian & Telscher, 2007, RA-2517/06 (M-294528-01-1)
R 2007 0175/6 Chambourg sur Indre, France, 2007 (Apache)	480 g/L SL	0.72 (BBCH 39)	0.24	300	1	56 85	Ear Grain	0.07 < 0.05	Billian, 2008, RA-2575/07 (M-312007-01-1)
R 2007 0186/1 Werl-Westönnen, Germany, 2007 (Ritmo)	480 g/L SL	0.77 (BBCH 49)	0.24	321	1	56 65	Ear Grain	0.09 < 0.05	Billian, 2008, RA-2575/07 (M-312007-01-1)

[a] 450 g/L SL formulation (150 g/L ethephon + 300 g/L chlormequat-chloride)

Table 60 Ethephon and HEPA residues in wheat grains resulting from supervised trials in Europe obtained using an analytical method involving acid hydrolysis/extraction

WHEAT Trial No Country, year (Variety)	Application					DALT days	Ethephon mg/kg	HEPA mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No				
GAP, Germany	660 g/L SL	0.46		100–300	1	–	Application timing BBCH 37–51		
13-2029-01 Bursheid, Germany 2013 (Winnetou Soft)	480 SL	0.48 (BBCH 51)	0.16	300	1	75	0.059	0.027	Schulte & Berkum, 2015, 13-2029 M-529493-01-1
13-2029-02 Villars-Perwin, Belgium, 2013 (Matrix Soft)	480 SL	0.48 (BBCH 51)	0.16	300	1	61	0.059	0.029	Schulte & Berkum, 2015, 13-2029 M-529493-01-1
13-2029-03 Little Shelford CB22 5EU, United Kingdom 2013 (Claire Soft)	480 SL	0.48 (BBCH 51)	0.24	200	1	74	0.11	0.080	Schulte & Berkum, 2015, 13-2029 M-529493-01-1
14-2018-01 Vechta – Langförden, Germany, 2014 (Winnetou mass-wheat)	480 SL	0.48 (BBCH 51)	0.16	300	1	71	0.083	0.031 (c, 0.013)	Schulte & Berkum, 2015, 14-2018 M-532267-01-1
14-2018-02 Burscheid, Germany 2014 (Tobak)	480 SL	0.48 (BBCH 51)	0.16	300	1	68	0.14	0.040	Schulte & Berkum, 2015, 14-2018 M-532267-01-1
14-2018-03 SG8 8S Great Chishill, United Kingdom, 2014 (Solstice Milling)	480 SL	0.48 (BBCH 51)	0.24	200	1	64	0.23	0.089 (c, 0.043)	Schulte & Berkum, 2015, 14-2018 M-532267-01-1
14-2018-04 France Chambourg sur Indre, 2014 (Touareg Winter)	480 SL	0.48 (BBCH 51)	0.16	300	1	77	0.052	0.019 (c, 0.015)	Schulte & Berkum, 2015, 14-2018 M-532267-01-1
14-2018-05 Slootdorp, Netherlands 2014	480 SL	0.48 (BBCH 51)	0.12	400	1	54	0.31	0.046	Schulte & Berkum, 2015, 14-2018 M-532267-01-1
GAP, France	480 g/L SL	0.48		100–200	1	70	Application timing BBCH 32–39		
14-2019-01 Gargas, France 2014 (Solehio Soft)	480 SL	0.48 (BBCH 39)	0.16	300	1	77	0.025	0.019 (c, 0.023)	Schulte & Berkum, 2015, 14-2019 M-532272-01-1
14-2019-02 Brenes, Spain 2014 (Don Pedro)	480 SL	0.48 (BBCH 39)	0.16	400	1	72	0.011	0.019	Schulte & Berkum, 2015, 14-2019 M-532272-01-1

WHEAT Trial No Country, year (Variety)	Application					DALT days	Ethephon mg/kg	HEPA mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No				
14-2019-03 Bologna, Italy 2014 (Mieti Winter)	480 SL	0.48 (BBCH 39)	0.12	300	1	58	0.10	0.042	Schulte & Berkum, 2015, 14-2019 M-532272-01-1
14-2019-04 Aramanha-Santarem, Portugal, 2014 (Artur Nick 2)	480 SL	0.48 (BBCH 39)	0.16	300	1	110	0.043	0.031 (c, 0.029)	Schulte & Berkum, 2015, 14-2019 M-532272-01-1
13-2030-01 Castelnau d'estretefonds, France, 2013 (Hystar Soft)	480 SL	0.48 (BBCH 39)	0.16	300	1	80	0.049	0.037 (c, 0.017)	Schulte & Berkum, 2015, 13-2030 M-529488-01-1
13-2030-02 El Campillo, Spain, 2013 (Artur Nick Soft)	480 SL	0.52 (BBCH 39)	0.16	322	1	64	0.057	0.029	Schulte & Berkum, 2015, 13-2030 M-529488-01-1
13-2030-03 Tarquinia, Italy 2013 (Quality Soft)	480 SL	0.48 (BBCH 39)	0.16	300	1	63	0.13	0.044	Schulte & Berkum, 2015, 13-2030 M-529488-01-1
13-2030-04 Bologna, Italy 2013 (Serio Soft)	480 SL	0.48 (BBCH 39)	0.14	350	1	62	0.010	0.014	Schulte & Berkum, 2015, 13-2030 M-529488-01-1

Supervised trials in USA

Sixteen supervised trials were conducted in wheat.

In the 1981 trials, a 480 g/L SL formulation was applied as a single foliar broadcast spray to wheat at a rate of 0.56–0.59 or 0.84 kg ai/ha. Application was made at the early-late boot growth stage. Residues of ethephon were determined using a method similar to SOP 90074, entitled “Detailed Method of Analysis for residues of (2-Chloroethyl)Phosphonic Acid (Ethephon) in Wheat and Barley Grain, Straw and Milling Fractions”, dated December 1981.

In the 1989 trials, a 480 g/L SL formulation was applied as a single foliar spray to wheat at a rate of 0.56 kg ai/ha. Application was made at the late boot to inflorescence emergence growth stage. Residues of ethephon were determined using the same method as above.

The samples were stored frozen at approximately –20 °C for the maximum period of storage was 5 months for the 1981 study and 29 months for the 1989 study.

Table 61 Ethephon residues in wheat grains resulting from supervised trials in the USA

WHEAT Trial Country, year (Variety)	Application					DALT	Ethephon mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No	days		
GAP, Canada	240 g/L SL	0.60		30–300	1	35	Application from BBCH 37–49	
10223-W1 Arkansas City, Kansas, USA, 1981 (Newton)	480 g/L SL	0.84 (late boot)	–	–	1	55	0.16 (0.17, 0.17, 0.15, 0.13) [a]	Harrison, 1981, 10223 (M-187972-01-1)

WHEAT Trial Country, year (Variety)	Application					DALT	Ethephon mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No	days		
10223-W2 Landisville, Pennsylvania, USA, 1981 (Redcoat)	480 g/L SL	0.84 (boot)	–	–	1	49	0.07 (0.07, 0.08, 0.05, 0.09)	Harrison, 1981, 10223 (M-187972-01-1)
10223-W3 Skaneateles, New York, USA, 1981 (Hauser)	480 g/L SL	0.84 (boot)	–	–	1	41	0.15 (0.15, 0.06, 0.12, 0.27)	Harrison, 1981, 10223 (M-187972-01-1)
10223-W4 Newton, Iowa, USA, 1981 (Sage Hard Red)	480 g/L SL	0.56 (early boot)	–	–	1	54	0.04 (0.02, 0.06, 0.04)	Harrison, 1981, 10223 (M-187972-01-1)
10223-W5 Sandusky, Michigan, USA, 1981 (Arthur)	480 g/L SL	0.56 (early boot)	–	–	1	62	0.15 (0.08, 0.18, 0.19)	Harrison, 1981, 10223 (M-187972-01-1)
10223-W6 Newcastle, Ohio, USA, 1981 (Titan)	480 g/L SL	0.56 (early boot)	–	–	1	63	0.03 (0.04, 0.04, 0.03, < 0.02)	Harrison, 1981, 10223 (M-187972-01-1)
10223-W7 Glyndon, Minnesota, USA, 1981 (Era)	480 g/L SL	0.56 (early boot)	–	–	1	57	0.02 (< 0.02, < 0.02, 0.02, 0.02)	Harrison, 1981, 10223 (M-187972-01-1)
10223-W8 Powell, Wyoming, USA, 1981 (Prodax)	480 g/L SL	0.84 (boot)	–	–	1	57	0.34 (0.26, 0.36, 0.39)	Harrison, 1981, 10223 (M-187972-01-1)
10223-W9 Warsaw, Illinois, USA, 1981 (Pioneer)	480 g/L SL	0.56 (mid boot)	–	–	1	64	< 0.02 (< 0.02,< 0.02,< 0.02)	Harrison, 1981, 10223 (M-187972-01-1)
10223-W10 Rock Springs, Pennsylvania, USA, 1981 (Titan)	480 g/L SL	0.56	–	–	1	48	0.04 (0.05, 0.03, 0.04, 0.05)	Harrison, 1981, 10223 (M-187972-01-1)
10223-W11 Elora, Ontario, Canada, 1981 (Frederick)	480 g/L SL	0.59	–	–	1	53	0.35	Harrison, 1981, 10223 (M-187972-01-1)
SARS-89-CO-24 Brighton, Colorado, USA, 1989 (Hawk)	480 g/L SL	0.56 (aerial) (late boot to 1/4 inflorescence emerged)	2.0	28	1	35 40 60	0.65 (0.65, 0.70, 0.60) 0.58 (0.58, 0.50, 0.67) 0.23 (0.29, 0.17, 0.23)	Conn, 1992, SARS-89-24 (M-187553-01-1)
		0.56 (ground) (late boot to 1/4 inflorescence emerged)	0.83	67	1	35 40 60	0.61 (0.61, 0.61, 0.60) 0.40 (0.48, 0.42, 0.30) 0.16 (0.15, 0.18, 0.14)	

WHEAT Trial Country, year (Variety)	Application					DALT	Ethephon mg/kg	Reference
	Form. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No	days		
SARS-89-KS-24 Sedan, Kansas, USA, 1989 (Thinderbird)	480 g/L SL	0.56 (aerial) (3/4 inflorescence emerged)	2.1	27	1	35 40 60	0.68 (0.94, 0.28, 0.82) 0.33 (0.35, 0.27, 0.38) 0.10 (0.08, 0.14, 0.09)	Conn, 1992, SARS-89-24 (M-187553-01-1)
		0.56 (ground) (3/4 inflorescence emerged)	0.86	65	1	35 40 60	0.53 (0.56, 0.52, 0.52) 0.33 (0.29, 0.34, 0.35) 0.10 (0.08, 0.09, 0.12)	
SARS-89-MN-24 East Grand Forks, Minnesota, USA, 1989 (Marshall)	480 g/L SL	0.56 (aerial) (late boot)	2.0	28	1	35 41 59	0.08 (0.07, 0.08, 0.08) 0.08 (0.07, 0.08, 0.10) < 0.05 (< 0.05, < 0.05, < 0.05)	Conn, 1992, SARS-89-24 (M-187553-01-1)
		0.56 (ground) (late boot)	0.86	65	1	35 41 59	0.13 (0.12, 0.13, 0.14) 0.12 (0.11, 0.12, 0.14) 0.05 (< 0.05, 0.05, < 0.05)	
SARS-89-ND-24 Northwood, North Dakota, USA, 1989 (Butte 86)	480 g/L SL	0.56 (aerial) (late boot)	2.0	28	1	35 40 60	0.33 (0.30, 0.36, 0.32) 0.15 (0.18, 0.13, 0.15) 0.08 (0.09, 0.07, 0.07)	Conn, 1992, SARS-89-24 (M-187553-01-1)
		0.56 (ground) (late boot)	0.86	65	1	35 40 60	0.25 (0.30, 0.24, 0.21) 0.14 (0.15, 0.14, 0.14) 0.08 (0.09, 0.06, 0.10)	
SARS-89-WA-24 Ephrata, Washington, USA, 1989 (Madson)	480 g/L SL	0.56 (aerial) (late boot)	2.0	28	1	40 60 70	0.15 (0.14, 0.12, 0.18) 0.14 (0.11, 0.14, 0.16) 0.07 (0.08, 0.07, 0.07)	Conn, 1992, SARS-89-24 (M-187553-01-1)
		0.56 (ground) (late boot)	0.73	77	1	40 60 70	0.30 (0.31, 0.40, 0.20) 0.24 (0.24, 0.25, 0.23) 0.15 (0.14, 0.13, 0.19)	

[a] Mean residue. Analytical results of replicate samples were in parentheses.

Cotton seed

A total of ten supervised trials were conducted in Greece and Spain. A 540 g/L SC formulation was applied as a foliar spray to cotton at a nominal rate of 1.44 kg ai/ha (actual rate range 1.41–1.53 kg ai/ha). In the trials conducted in Greece in 1993 and 1995, an additional plot was treated at a nominal rate of 2.88 kg ai/ha (actual rate range 2.79–2.93 kg ai/ha). In the 1993–1995 studies, residues of ethephon were determined using method HVA 12/89. In the 2008 study, residues of ethephon were determined using method 00918. The maximum period of storage of frozen cotton seed samples, except described below, at < –18 °C was 14 months.

In trial 93739GR1, samples were stored at room temperature for 3 months and then frozen (–20 °C) for 13 months prior to analysis. As storage stability data indicate that residues of ethephon are not stable in cotton seed when stored at room temperature, these data will not be considered in the estimation of maximum residue level. In trials 94681SE1, 94681SE2 and 94681SE3, samples were stored in a cold room for 1 month and then frozen (–20 °C) for 4 months prior to analysis.

A total of forty-one supervised trials were conducted in the USA. In the 1989 trials, a 720 g/L SL formulation was applied as a single foliar spray to cotton at a rate of 2.24 kg ai/ha by ground or aerial application. Residues of ethephon were determined using method SOP 90075. In the 1993 trials, a 540 g/L SC formulation was applied as a single foliar spray to cotton at a nominal rate of 2.24 kg ai/ha by ground application. Residues of ethephon were determined using method EC-92-228. In the 1994 trials, a 540 g/L SC formulation was applied as a single foliar

spray to cotton at a nominal rate of 2.24 kg ai/ha by ground application. Residues of ethephon in seed and gin trash were determined using method EC-92-228. The maximum period of storage of frozen samples at < –10 °C was 12 months for seed and 6.3 months for gin trash.

A total of seven supervised trials were conducted in Brazil. In the 1996 trials, a 480 g/L SL formulation was applied as a foliar spray to cotton at a nominal rate of 1.44 kg ai/ha in one plot and at 2.88 kg ai/ha in the other. In the 2006 trials (HR06BR008-P1 to -P4), a 540 g/L SC formulation was applied as a foliar spray to cotton at a nominal rate of 1.20 kg ai/ha. Residues of ethephon were determined using method 11-94 (ethylene release). The maximum period of storage of frozen samples at < –10 °C was 12 months.

Table 62 Ethephon residues in cotton seed resulting from supervised trials in Europe, the USA and Brazil

COTTON Trial No. Country, year (Variety)	Application					DALT days	Ethephon mg/kg	Reference
	Form. (g ai/L & type)	kg ai/ha	kg ai/hL	Water (L/ha)	No			
GAP, Greece	480 g/L SL	1.44		500–600	1	7		
EUROPE								
93739GR1 Arma Thiva-Viotia, Greece, 1993 (Zeta II)	540 g/L SC [a]	1.44	0.36	400	1	7	< 0.10	Richard & Muller, 1995, R&D/CRLD/AN/bd/ 9515891 (M-163122-01-1)
	540 g/L SC [a]	2.88	0.72	400	1	7	0.12	
94681SE1 Carlota-AL, Spain, 1994 (Cnema 111)	540 g/L SC [a]	1.44	0.36	400	1	0 3 7	< 0.10 0.35 0.59	Richard & Muller, 1995, R&D/CRLD/AN/bd/ 9515911 (M-163133-01-1)
94681SE2 Carlota-ZA, Spain, 1994 (Cnema 111)	540 g/L SC [a]	1.44	0.36	400	1	0 3 7	< 0.10 0.15 0.30	Richard & Muller, 1995, R&D/CRLD/AN/bd/ 9515911 (M-163133-01-1)
94681SE3 Ecija, Spain, 1994 (Cnema 111)	540 g/L SC [a]	1.44	0.36	400	1	0 3 7	2.09 0.29 1.13	Richard & Muller, 1995, R&D/CRLD/AN/bd/ 9515911 (M-163133-01-1)
95723SE1 Ciatr Sevilla, Spain, 1995 (Corona)	540 g/L SC [a]	1.44	0.33	440	1	7	0.19	Muller, 1996, R&D/CRLD/AN/bd/ 9516706 (M-163236-01-1)
95705GR1 Nicaea-Larissa, Greece, 1995 (Zeta 2)	540 g/L SC [a]	1.48	0.16	911	1	8	< 0.10	Muller, 1996, R&D/CRLD/AN/vg/ 9516705 (M-163240-01-1)
	540 g/L SC [a]	2.79	0.31	911	1	8	0.20	
95705GR2 Larissa, Greece, 1995 (Zeta 2)	540 g/L SC [a]	1.46	0.16	912	1	8	< 0.10	Muller, 1996, R&D/CRLD/AN/vg/ 9516705 (M-163240-01-1)
	540 g/L SC [a]	2.93	0.32	912	1	8	< 0.10	
95705GR3 Stavros-Lamia, Greece, 1995 (Zeta 2)	540 g/L SC [a]	1.41	0.16	892	1	8	0.35	Muller, 1996, R&D/CRLD/AN/vg/ 9516705 (M-163240-01-1)
	540 g/L SC [a]	2.93	0.33	892	1	8	0.23	

COTTON Trial No. Country, year (Variety)	Application					DALT days	Ethephon mg/kg	Reference
	Form. (g ai/L & type)	kg ai/ha	kg ai/hL	Water (L/ha)	No			
08-2023-01 Aiginion-Pieria, Greece, 2008 (Carmen)	540 g/L SC [a]	1.44	0.29	500	1	0 7	2.3 (boll) 0.10	Billian, Reineke, Krusell, 2009, 08-2023 (M-360139-01-1)
08-2023-02 Lebrija, Spain, 2008 (Celia)	540 g/L SC [a]	1.53	0.29	533	1	0 7	1.7 (boll) 0.07	Billian, Reineke, Krusell, 2009, 08-2023 (M-360139-01-1)
GAP, USA	765 g/L SC [b]	2.24		28–234	1	7		
GAP, USA	720 g/L SC	2.24		19–94	1	7		
USA								
89-156 Harmon Co., OK, USA, 1989 (Stoneville 483)	720 g/L SL	2.24 (aerial)	11.4	19.7	1	7 10 14	0.23 (0.24, 0.26, 0.20) 0.47 (0.45, 0.51, 0.45) 0.56 (0.77, 0.76, 0.14)	Nygren, 1991, USA89I03 (M-187602-01-1)
89-157 Harmon Co., OK, USA, 1989 (Stoneville 483)	720 g/L SL	2.24 (ground)	1.34	167	1	7 10 14	0.58 (0.64, 0.52, 0.59) 0.75 (0.78, 0.75, 0.72) 0.70 (0.65, 0.83, 0.62)	Nygren, 1991, USA89I03 (M-187602-01-1)
89-159 Maricopa Co., AZ, USA, 1989 (DP-L90)	720 g/L SL	2.24 (aerial)	4.79	46.7	1	7 10 14	0.55 (0.49, 0.57, 0.58) 0.95 (1.2, 0.64, 1.0) 0.45 (0.55, 0.10, 0.71)	Nygren, 1991, USA89I03 (M-187602-01-1)
89-160 Maricopa Co., AZ, USA, 1989 (DP-L70)	720 g/L SL	2.24 (ground)	2.08	107	1	7 10 14	2.4 (2.1, 2.9, 2.1) 2.2 (2.7, 1.8, 2.0) 1.9 (2.4, 2.0, 1.4)	Nygren, 1991, USA89I03 (M-187602-01-1)
89-161 Lonoke Co., AR, USA, 1989 (Stoneville 506)	720 g/L SL	2.24 (ground)	1.60	140	1	7 10 14	0.10 (0.10, 0.09, 0.10) 0.18 (0.20, 0.17, 0.16) 0.24 (0.22, 0.33, 0.18)	Nygren, 1991, USA89I03 (M-187602-01-1)
89-162 Tulare Co., CA, USA, 1989 (Acala GC-510)	720 g/L SL	2.24 (aerial)	4.79	46.7	1	7 10 14	0.10 (0.08, 0.09, 0.12) 0.09 (0.16, 0.05, 0.07) 0.16 (0.22, 0.12, 0.15)	Nygren, 1991, USA89I03 (M-187602-01-1)
89-163 Wharton Co., TX, USA, 1989 (DES 119)	720 g/L SL	2.24 (ground)	1.58	142	1	7 10 14	0.06 (0.07, 0.05, 0.06) 0.05 (0.05, 0.04, 0.06) 0.02 (< 0.02, < 0.02, 0.03)	Nygren, 1991, USA89I03 (M-187602-01-1)
89-164 Wharton Co., TX, USA, 1989 (DES 119)	720 g/L SL	2.24 (aerial)	12.0	18.7	1	7 10 14	0.03 (< 0.02, 0.06, < 0.02) < 0.02 (< 0.02, < 0.02, < 0.02) < 0.02 (< 0.02,< 0.02, < 0.02)	Nygren, 1991, USA89I03 (M-187602-01-1)
89-165 Burke Co., GA, USA, 1989 (DPL 90)	720 g/L SL	2.24 (ground)	1.44	155	1	7 10 14	0.31 (0.35, 0.36, 0.21) 0.34 (0.38, 0.37, 0.27) < 0.02 (< 0.02, < 0.02, < 0.02)	Nygren, 1991, USA89I03 (M-187602-01-1)
89-166 Burke Co., GA, USA, 1989 (DPL 90)	720 g/L SL	2.24 (aerial)	10.4	21.5	1	7 10 14	0.65 (0.82, 0.51, 0.61) 0.35 (0.54, 0.07, 0.45) 0.36 (0.43, 0.26, 0.39)	Nygren, 1991, USA89I03 (M-187602-01-1)

COTTON Trial No. Country, year (Variety)	Application					DALT days	Ethephon mg/kg	Reference
	Form. (g ai/L & type)	kg ai/ha	kg ai/hL	Water (L/ha)	No			
89-167 Hale Co., TX, USA, 1989 (Paymaster 145)	720 g/L SL	2.24 (aerial)	12.0	18.6	1	7 10 14	0.54 (0.42, 1.0, 0.21) 0.91 (0.42, 1.5, 0.82) 1.42 (0.25, 2.0, 2.0)	Nygren, 1991, USA89I03 (M-187602-01-1)
89-168 Lynn Co., TX, USA, 1989 (Paymaster HS26)	720 g/L SL	2.24 (ground)	1.70	132	1	7 10 14	0.46 (0.42, 0.50, 0.47) 0.86 (0.78, 0.69, 1.1) 0.70 (0.58, 0.71, 0.80)	Nygren, 1991, USA89I03 (M-187602-01-1)
89-169 Curry Co., NM, USA, 1989 (Paymaster 792)	720 g/L SL	2.24 (ground)	1.70	132	1	7 10 14	1.5 (1.6, 1.6, 1.4) 1.1 (1.3, 0.92, 1.1) 1.5 (1.6, 1.2, 1.8)	Nygren, 1991, USA89I03 (M-187602-01-1)
89-170 Sharkey Co., MS, USA, 1989 (DPL 50)	720 g/L SL	2.24 (ground)	1.63	137	1	7 10 14	0.50 (0.69, 0.35, 0.45) 0.09 (0.10, 0.05, 0.12) 0.10 (0.12, 0.08, 0.11)	Nygren, 1991, USA89I03 (M-187602-01-1)
89-171 Sharkey Co., MS, USA, 1989 (DPL 50)	720 g/L SL	2.24 (ground)	1.63	137	1	7 10 14	0.61 (0.54, 0.80, 0.49) 0.42 (0.21, 0.90, 0.16) 0.07 (0.09, 0.11, < 0.02)	Nygren, 1991, USA89I03 (M-187602-01-1)
89-172 Sharkey Co., MS, USA, 1989 (DPL 50)	720 g/L SL	2.24 (aerial)	12.1	18.5	1	7 11 14	0.44 (0.42, 0.34, 0.56) 0.16 (0.24, 0.12, 0.11) 0.22 (0.03, 0.58, 0.04)	Nygren, 1991, USA89I03 (M-187602-01-1)
89-173 Tulare Co., CA, USA, 1989 (Acala GC510)	720 g/L SL	2.24 (aerial)	4.89	45.8	1	7 10 14	0.35 (0.25, 0.28, 0.53) 0.21 (0.28, 0.12, 0.22) 0.05 (0.04, 0.05, 0.05)	Nygren, 1991, USA89I03 (M-187602-01-1)
89-174 Fresno Co., CA, USA, 1989 (GC-510)	720 g/L SL	2.24 (ground)	1.20	187	1	7 10 14	0.36 (0.54, 0.30, 0.25) 0.16 (0.19, 0.12, 0.18) 0.19 (0.14, 0.22, 0.21)	Nygren, 1991, USA89I03 (M-187602-01-1)
89-175 Lonoke Co., AR, USA, 1989 (DPL 50)	720 g/L SL	2.24 (aerial)	11.5	19.5	1	7 11 14	0.03 (< 0.02, 0.03, 0.03) < 0.02 (< 0.02, < 0.02, < 0.02) 0.11 (0.06, 0.18, 0.10)	Nygren, 1991, USA89I03 (M-187602-01-1)
93-0257 Hale Co., TX, USA, 1993 (Paymaster HS-200)	540 g/L SC	2.20 (ground)	1.30	168	1	7	0.59 (0.50, 0.52, 0.76)	See, 1994, USA93I03R (M-252199-01-1)
93-0258 Lenoir, NC, USA, 1993 (Chembred 1135)	540 g/L SC	2.26 (ground)	1.61	140	1	7	0.23 (0.20, 0.23, 0.26)	See, 1994, USA93I03R (M-252199-01-1)
93-0259 Yuma Co., AZ, USA, 1993 (Deltapine 50)	540 g/L SC	2.22 (ground)	1.58	140	1	7	2.42 (2.20, 2.59, 2.48)	See, 1994, USA93I03R (M-252199-01-1)
93-0260 Fresno, CA, USA, 1993 (Acala SJ-2)	540 g/L SC	3.18 (ground)	2.27	140	1	7	0.59 (1.25, 0.23, 0.29)	See, 1994, USA93I03R (M-252199-01-1)
93-0261 Backgate, AR, USA, 1993 (D&PL50)	540 g/L SC	2.35 (ground)	1.68	140	1	7	0.11 (0.12, 0.10, 0.12)	See, 1994, USA93I03R (M-252199-01-1)

COTTON Trial No. Country, year (Variety)	Application					DALT days	Ethephon mg/kg	Reference
	Form. (g ai/L & type)	kg ai/ha	kg ai/hL	Water (L/ha)	No			
93-0262 Barnwell Co., SC, USA, 1993 (Delta Pine 90-Acala)	540 g/L SC	2.33 (ground)	1.66	140	1	7	0.55 (0.69, 0.56, 0.40)	See, 1994, USA93I03R (M-252199-01-1)
93-0263 Mitchel Co., GA, USA, 1993 (Deltapine 90-Acala)	540 g/L SC	2.24 (ground)	1.20	187	1	7	0.10 (0.12, 0.11, 0.06)	See, 1994, USA93I03R (M-252199-01-1)
93-0264 Fresno Co., CA, USA, 1993 (GS 10)	540 g/L SC	3.77 (ground)	2.02	187	1	7	0.99 (1.06, 1.12, 0.80)	See, 1994, USA93I03R (M-252199-01-1)
93-0265 Crittenden Co., AR, USA, 1993 (Stoneville 453)	540 g/L SC	2.13 (ground)	1.52	140	1	7	0.41 (0.42, 0.41, 0.41)	See, 1994, USA93I03R (M-252199-01-1)
93-0266 St. Landry, LA, USA, 1993 (Deltapine 50)	540 g/L SC	2.26 (ground)	1.61	140	1	7	0.26 (0.21, 0.34, 0.22)	See, 1994, USA93I03R (M-252199-01-1)
94-0284 Wharton Co., TX, USA, 1994 (Deltapine 20)	540 g/L SC	2.31 (ground)	1.54	150	1	7	0.16 (0.17, 0.14)	See, 1995, USA94I01R (M-253436-01-1)
94-0285 Castro Co., TX, USA, 1994 (Paymaster 145)	540 g/L SC	2.25 (ground)	1.53	147	1	7	2.88 (2.48, 3.28)	See, 1995, USA94I01R (M-253436-01-1)
94-0286 Floyd Co., TX, USA, 1994 (Paymaster HS-200)	540 g/L SC	2.43 (ground)	1.54	158	1	7	0.69 (0.70, 0.67)	See, 1995, USA94I01R (M-253436-01-1)
94-0287 Fresno, CA, USA, 1994 (Maxxa)	540 g/L SC	2.22 (ground)	1.59	139	1	7	0.18 (0.15, 0.21)	See, 1995, USA94I01R (M-253436-01-1)
94-0288 Washington Co., MS, USA, 1994 (DPL 50)	540 g/L SC	2.26 (ground)	1.59	142	1	7	0.54 (0.56, 0.52)	See, 1995, USA94I01R (M-253436-01-1)
94-0289 Houseton Co., AL, USA, 1994 (DPL 5415)	540 g/L SC	2.28 (ground)	1.64	139	1	8	0.26 (0.29, 0.22)	See, 1995, USA94I01R (M-253436-01-1)
94-0290 Madera Co., CA, USA, 1994 (Maxa)	540 g/L SC	2.17 (ground)	1.18	184	1	6	2.73 (2.34, 3.12)	See, 1995, USA94I01R (M-253436-01-1)
94-0291 Fayette Co., TN, USA, 1994 (Stoneville 453)	540 g/L SC	2.25 (ground)	1.57	143	1	7	1.18 (1.38, 0.97)	See, 1995, USA94I01R (M-253436-01-1)
94-0292 Crittenden Co., AR, USA, 1994 (Stoneville 453)	540 g/L SC	2.27 (ground)	1.62	140	1	7	0.09 (0.096, 0.080)	See, 1995, USA94I01R (M-253436-01-1)

COTTON Trial No. Country, year (Variety)	Application					DALT days	Ethephon mg/kg	Reference
	Form. (g ai/L & type)	kg ai/ha	kg ai/hL	Water (L/ha)	No			
94-0293 Burleson Co., TX, USA, 1994 (DP&L 5415)	540 g/L SC	2.24 (ground)	1.56	144	1	9	0.12 (0.12, 0.12)	See, 1995, USA94I01R (M-253436-01-1)
94-0393 Hale Co., TX, USA, 1994 (Paymaster HS-200)	540 g/L SC	2.32 (ground)	1.53	151	1	7	4.93 (4.21, 5.65)	See, 1995, USA94I01R (M-253436-01-1)
94-0394 Hale Co., TX, USA, 1994 (Paymaster 145)	540 g/L SC	2.27 (ground)	1.45	157	1	7	2.29 (2.32, 2.26)	See, 1995, USA94I01R (M-253436-01-1)
GAP, Brazil	540 g/L SC [a]	1.20		200–500	1	7		
GAP, Peru	720 g/L SL	1.44			1	7–14		
BRAZIL								
003/97-PC-01 Holambra SP, Brazil, 1996 (IAC-20)	480 g/L SL	1.44	–	–	1	7	< 0.20	Garcia, 1997, CP-2466/97 (M-188222-01-1)
	480 g/L SL	2.88	–	–	1	7	< 0.20	
055/96-PC EAE Paulinia SP, Brazil, 1996 (IAC-22)	480 g/L SL	1.44	0.36	400	1	7	< 0.20	Garcia & Oliverira, 1997, CP-2435/97 (M-253467-02-1)
	480 g/L SL	2.88	0.72	400	1	7	< 0.20	
056/96-PC Holambra SP, Brazil, 1996 (IAC-20)	480 g/L SL	1.44	0.36	400	1	7	< 0.20	Garcia & Oliverira, 1997, CP-2436/97 (M-253470-02-1)
	480 g/L SL	2.88	0.72	400	1	7	< 0.20	
HR06BRA008-P1 Paulinia SP, Brazil, 2006 (Delta Opal)	540 g/L SC [a]	1.20	0.24	500	1	7	< 0.10	Galhiane & Santos, 2006, RA-218/06 (M-285068-01-2)
HR06BRA008-P2 Rondonopolis MT Brazil, 2006 (Delta Opal)	540 g/L SC [a]	1.20	0.24	500	1	7	< 0.10	Galhiane & Santos, 2006, RA-219/06 (M-285070-01-2)
HR06BRA008-P3 Costa Rica MS, Brazil, 2006 (Delta Opal)	540 g/L SC [a]	1.20	0.24	500	1	7	< 0.10	Galhiane & Santos, 2006, RA-220/06 (M-285073-01-2)
HR06BRA008-P4 Rio Verde GO, Brazil, 2006 (FMX 966)	540 g/L SC [a]	1.20	0.24	500	1	7	< 0.10	Galhiane & Santos, 2006, RA-221/06 (M-285075-01-2)

[a] 540 g/L SC formulation (480 g/L ethephon + 60 g/L cyclanilide)

[b] 765 g/L SC formulation (720 g/L ethephon + 45 g/L cyclanilide)

Primary feed commodities

Barley forage and straw

Thirty-seven supervised trials have been conducted in barley in Europe.

Table 63 Ethephon residues in barley forage and straw resulting from supervised trials in Europe

BARLEY	Application					DALT	Portion analysed	Ethephon	Reference
Trial No. Country, year (Variety)	Form (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No	days		mg/kg	
GAP, Germay	660 g/L SL	0.462		100– 300	1	–	Application timing BBCH 32–49		
GAP, UK	480 g/L SL	0.48		100– 400	–	–	Application timing BBCH 32–49 Maximum total rate 0.48 kg ai/ha		
DR00EUS525 ITA0101 Bologna, Italy, 2000 (Express)	480 g/L SL	0.48 (BBCH 47)	0.16	300	ß1	0 11 48	Green plant Green plant Straw	10 0.23 0.08	Hees, 2001, DR00EUS525 (M-199982-01-1)
DR00EUS525 ITA0102 S. Mauro Pascoli, Italy, 2000 (Extra)	480 g/L SL	0.48 (BBCH 45)	0.16	300	1	0 18 47	Green plant Green plant Straw	11 0.35 0.63	Hees, 2001, DR00EUS525 (M-199982-01-1)
00547BX1 Marignac, France, 2000 (Sunrise)	480 g/L SL	0.48 (BBCH 45)	0.14	333	1	0 14 27 52	Green plant Green plant Green plant Straw	8.1 1.3 0.47 0.25	Ballasteros, 2001, R&D/CRLD/AN/mr/0115430 (M-208093-01-1)
00547TL1 Gardouch, France, 2000 (Esterel)	480 g/L SL	0.48 (BBCH 47)	0.19	250	1	0 14 25 62	Green plant Green plant Green plant Straw	5.1 1.9 0.90 0.43	Ballasteros, 2001, R&D/CRLD/AN/mr/0115430 (M-208093-01-1)
01R761-1 Ronchères, France, 2001 (Platine)	480 g/L SL	0.48 (BBCH 47)	0.19	250	1	0 69	Green plant Straw	5.7 0.06	Davies, 2002, 01R761 (M-209901-01-1)
01R761-2 Hargicourt, France, 2001 (Muscat)	480 g/L SL	0.48 (BBCH 49)	0.19	250	1	0 12 28 54	Green plant Green plant Green plant Straw	2.6 0.86 0.48 0.21	Davies, 2002, 01R761 (M-209901-01-1)
01R761-3 Braintree, UK, 2001 (Regina)	480 g/L SL	0.48 (BBCH 55)	0.19	252	1	0 6 28 58	Green plant Green plant Green plant Straw	9.3 5.8 1.6 0.95	Davies, 2002, 01R761 (M-209901-01-1)
01R761-4 Weilerswist, Germany, 2001 (Theresa)	480 g/L SL	0.48 (BBCH 51)	0.16	300	1	0 9 35 60	Green plant Green plant Green plant Straw	6.2 0.80 0.66 1.1	Davies, 2002, 01R761 (M-209901-01-1)
01R761-5 Zschortau, Germany, 2001 (Landi)	480 g/L SL	0.48 (BBCH 49)	0.16	300	1	0 66	Green plant Straw	4.2 0.33	Davies, 2002, 01R761 (M-209901-01-1)
01R771-1 Senestis, France, 2001 (Platine)	480 g/L SL	0.48 (BBCH 45)	0.19	250	1	0 64	Green plant Straw	9.4 < 0.05	Davies, 2002, 01R771 (M-210307-01-1)
01R771-2 Toussieux, France, 2001 (Ladoga)	480 g/L SL	0.48 (BBCH 47)	0.19	250	1	0 20 27 63	Green plant Green plant Green plant Straw	8.4 1.1 0.81 0.09	Davies, 2002, 01R771 (M-210307-01-1)
01R771-3 Genas, France, 2001 (Ladoga)	480 g/L SL	0.48 (BBCH 47)	0.19	250	1	0 14 33 57	Green plant Green plant Green plant Straw	4.8 2.1 1.4 0.36	Davies, 2002, 01R771 (M-210307-01-1)

BARLEY	Application					DALT	Portion analysed	Ethephon	Reference
Trial No. Country, year (Variety)	Form (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No	days		mg/kg	
01R771-4 Alberone Di Cento, Italy, 2001 (Sonora)	480 g/L SL	0.48 (BBCH 47)	0.14	350	1	0 9 20 35	Green plant Green plant Green plant Straw	4.4 5.2 1.7 0.24	Davies, 2002, 01R771 (M-210307-01-1)
01R771-5 Xirochori-Kilkis, Greece, 2001 (Athinaida)	480 g/L SL	0.48 (BBCH 47)	0.16	300	1	0 50	Green plant Straw	3.0 < 0.05	Davies, 2002, 01R771 (M-210307-01-1)
GAP, France	480 g/L SL	0.48		100–200	1	56	Application timing BBCH 32–39		
R 2004 0577/4 Monospita, Greece, 2004 (Kannon (distiho))	450 g/L SL [a]	0.38 (BBCH 39)	0.125	300	1	0 11 54	Green plant Green plant Straw	8.1 0.20 < 0.05	Bardel & Wolters, 2005, RA-2093/04 (M-251235-01-1)
R 2004 0578/2 Bologna, Italy, 2004 (Marjorie)	450 g/L SL [a]	0.38 (BBCH 39)	0.125	300	1	0 12 54	Green plant Green plant Straw	6.0 0.09 < 0.05	Bardel & Wolters, 2005, RA-2093/04 (M-251235-01-1)
R 2004 0579/0 Vouillé, France, 2004 (Scarlette)	450 g/L SL [a]	0.38 (BBCH 39)	0.125	300	1	0 20 56	Green plant Green plant Straw	6.7 0.21 0.12	Bardel & Wolters, 2005, RA-2093/04 (M-251235-01-1)
R 2004 0580/4 Balaguer, Spain, 2004 (Prestige)	450 g/L SL [a]	0.38 (BBCH 39)	0.125	300	1	0 15 53	Green plant Green plant Straw	5.2 0.30 0.27	Bardel & Wolters, 2005, RA-2093/04 (M-251235-01-1)
R 2004 0572/3 Lund, Sweden, 2004 (Bombay)	450 g/L SL [a]	0.38 (BBCH 39)	0.125	300	1	0 21 80	Green plant Green plant Straw	9.5 0.45 0.07	Bardel & Wolters, 2005, RA-2092/04 (M-251366-01-1)
R 2004 0573/1 Leverkusen, Germany, 2004 (Condesse)	450 g/L SL [a]	0.38 (BBCH 37)	0.125	300	1	0 19 85	Green plant Green plant Straw	3.9 < 0.05 < 0.05	Bardel & Wolters, 2005, RA-2092/04 (M-251366-01-1)
R 2004 0575/8 Weri-Obernergstraße, Germany, 2004 (Intro)	450 g/L SL [a]	0.38 (BBCH 39)	0.125	300	1	0 17 77	Green plant Green plant Straw	5.3 0.11 < 0.05	Bardel & Wolters, 2005, RA-2092/04 (M-251366-01-1)
R 2004 0576/6 Fresnoy les Roye, France, 2004 (Esterel)	450 g/L SL [a]	0.38 (BBCH 39)	0.125	300	1	0 17 67	Green plant Green plant Straw	6.2 0.38 0.07	Bardel & Wolters, 2005, RA-2092/04 (M-251366-01-1)
R 2006 0126/3 Neuville de Poitou, France, 2006 (Abondance)	450 g/L SL [a]	0.38 (BBCH 39)	0.125	300	1	0 7 21 59	Green plant Green plant Green plant Straw	5.6 1.1 0.22 0.13	Billian & Erler, 2007, RA-2519/06 (M-290151-01-1)
R 2006 0299/5 Tarascon, France, 2006 (Baraka)	450 g/L SL [a]	0.38 (BBCH 39)	0.125	300	1	0 7 21 60	Green plant Green plant Green plant Straw	8.3 3.4 2.1 1.6	Billian & Erler, 2007, RA-2519/06 (M-290151-01-1)

BARLEY	Application					DALT	Portion analysed	Ethephon	Reference
Trial No. Country, year (Variety)	Form (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No	days		mg/kg	
GAP, Poland	480 g/L SL	0.72		150– 300	1	–	Application timing BBCH 32–39		
R 2006 0117/4 Beuvraignes, France, 2006 (Colibri)	480 g/L SL	0.67 (BBCH 37)	0.22	300	1	0 7 21 76	Green plant Green plant Green plant Straw	13 1.1 0.26 < 0.05	Billian & Telscher, 2007, RA-2515/06 (M-294373-01-1)
R 2006 0286/3 Welver-Flerke, Germany, 2006 (Duet)	480 g/L SL	0.67 (BBCH 37)	0.22	300	1	0 7 21 68	Green plant Green plant Green plant Straw	7.1 0.28 0.07 < 0.05	Billian & Telscher, 2007, RA-2515/06 (M-294373-01-1)
R 2006 0285/5 Hoxne/Nreye, UK, 2006 (Sequel)	480 g/L SL	0.67 (BBCH 39)	0.22	300	1	0 6 20 74	Green plant Green plant Green plant Straw	9.6 0.60 0.22 0.13	Billian & Telscher, 2007, RA-2515/06 (M-294373-01-1)
R 2007 0172/1 Chaussy, France, 2007 (Sibéria)	480 g/L SL	0.72 (BBCH 37)	0.24	300	1	0 7 21 56 75	Green plant Green plant Green plant Rest of plant Straw	8.9 4.3 0.24 < 0.05 < 0.05	Billian, 2008, RA-2573/07 (M-311809-01-1)
R 2007 0181/0 Lund, Sweden, 2007 (Bombay)	480 g/L SL	0.72 (BBCH 37)	0.24	300	1	0 7 21 56 70	Green plant Green plant Green plant Rest of plant Straw	6.0 0.39 0.08 < 0.05 < 0.05	Billian, 2008, RA-2573/07 (M-311809-01-1)
GAP, France	450 g/L SL [a]	0.23		100– 200	1	–	Application timing BBCH 31–37		
R 2004 0581/2 Le Thil en Vexin, France, 2004 (Scarlet)	450 g/L SL [a]	0.23 (BBCH 39)	0.075	300	1	0 21 57	Green plant Green plant Straw	3.7 0.28 < 0.05	Bardel & Wolters, 2005, RA-2094/04 (M-249305-02-1)
R 2004 0582/0 Staffanstorp, Sweden, 2004 (Pasadena)	450 g/L SL [a]	0.23 (BBCH 39)	0.075	300	1	0 15 79	Green plant Green plant Straw	5.9 0.16 0.05	Bardel & Wolters, 2005, RA-2094/04 (M-249305-02-1)
R 2004 0583/9 Burscheid, Germany, 2004 (Scarlett)	450 g/L SL [a]	0.23 (BBCH 39)	0.075	300	1	0 11 61	Green plant Green plant Straw	4.5 0.21 < 0.05	Bardel & Wolters, 2005, RA-2094/04 (M-249305-02-1)
R 2004 0584/7 Gersthofen, Germany, 2004 (Ursa)	450 g/L SL [a]	0.23 (BBCH 39)	0.075	300	1	0 10 65	Green plant Green plant Straw	3.0 0.11 < 0.05	Bardel & Wolters, 2005, RA-2094/04 (M-249305-02-1)
R 2004 0585/5 Saint Germain sur Renon, France, 2004 (Nevada)	450 g/L SL [a]	0.23 (BBCH 41)	0.075	300	1	0 17 52	Green plant Green plant Straw	5.2 0.27 < 0.05	Bardel & Wolters, 2005, RA-2095/04 (M-251234-01-1)
R 2004 0586/3 Bologna, Italy, 2004 (Federal)	450 g/L SL [a]	0.23 (BBCH 39)	0.075	300	1	0 14 52	Green plant Green plant Straw	5.4 0.28 0.10	Bardel & Wolters, 2005, RA-2095/04 (M-251234-01-1)
R 2004 0587/1 Tarascon, France, 2004 (Baraka)	450 g/L SL [a]	0.23 (BBCH 39)	0.075	300	1	0 6 44	Green plant Green plant Straw	7.5 3.9 3.7	Bardel & Wolters, 2005, RA-2095/04 (M-251234-01-1)

BARLEY	Application					DALT	Portion analysed	Ethephon	Reference
Trial No. Country, year (Variety)	Form (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No	days		mg/kg	
R 2004 0589/8 Golegã, Portugal, 2004 (Scarlett)	450 g/L SL [a]	0.23 (BBCH 39)	0.075	300	1	0 21 61	Green plant Green plant Straw	4.1 0.16 0.19	Bardel & Wolters, 2005, RA-2095/04 (M-251234-01-1)

[a] 450 g/L SL formulation (150 g/L ethephon + 300 g/L chlormequat-chloride)

Table 64 Ethephon residues in barley forage and straw resulting from supervised trials in Europe obtained using an analytical method involving acid hydrolysis/extraction

BARLEY Trial No Country, year (Variety)	Application					DALA days	Portion analysed	Ethephon mg/kg	HEPA mg/kg	Reference
	Form.(g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No					
13-2027-01 Burscheid, Germany, 2013 (Duett)	480 SL	0.48 (BBCH 51)	0.16	300	1	0 7 14 21 24 59	Green plant Green plant Green plant Green plant Green plant Straw	6.2 0.61 0.55 0.26 0.43 0.51	0.091 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	Schulte & Berkum, 2015, 13-2027 M-526906-01-1
13-2027-02 Diegem, Belgium, 2013 (Meridian)	480 SL	0.51 (BBCH 51)	0.19	267	1	0 33 55	Green plant Green plant Straw	3.2 < 0.05 0.35	< 0.05 < 0.05 < 0.05	Schulte & Berkum, 2015, 13-2027 M-526906-01-1
13-2027-03 Mijdrecht, Netherlands, 2013 (Malabar)	480 SL	0.48 (BBCH 51)	0.16	300	1	0 7 14 21 43 56	Green plant Green plant Green plant Green plant Green plant Straw	7.9 3.8 0.85 0.57 0.27 1.5	0.094 0.088 0.085 0.076 0.059 < 0.05	Schulte & Berkum, 2015, 13-2027 M-526906-01-1
13-2027-04 Cambridge, United Kingdom, 2013 (Cassata)	480 SL	0.48 (BBCH 51)	0.24	200	1	0 34 68	Green plant Green plant Straw	6.6 0.36 3.6	0.093 < 0.05 0.066	Schulte & Berkum, 2015, 13-2027 M-526906-01-1
14-2022-01 Langenfeld, Germany, 2014 (Naomie)	480 SL	0.54 (BBCH 51)	0.16	336	1	0 7 14 21 36 78	Green plant Green plant Green plant Green plant Green plant Straw	6.2 0.50 0.29 0.17 0.086 0.64	0.12 < 0.05 < 0.05 < 0.05 < 0.05 0.055	Schulte & Berkum, 2015, 14-2022
14-2022-02 Burscheid, Germany, 2014 (Leibnitz)	480 SL	0.48 (BBCH 51)	0.16	300	1	0 21 64	Green plant Green plant Straw	7.7 0.37 1.2	0.12 < 0.05 0.063 (c, 0.061)	Schulte & Berkum, 2015, 14-2022
14-2022-03 Lyon Cedex 09, France, 2014 (Obite Winter)	480 SL	0.48 (BBCH 51)	0.16	300	1	0 7 14 21 28 56	Green plant Green plant Green plant Green plant Green plant Straw	6.6 0.34 0.15 0.10 < 0.05 0.43	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	Schulte & Berkum, 2015, 14-2022
14-2022-04 Cambridge CB4 0WB, United Kingdom, 2014 (Cassatta Typical UK variety)	480 SL	0.48 (BBCH 55)	0.24	200	1	0 34 73	Green plant Green plant Straw	7.3 0.13 0.78 (c, 0.088)	0.072 0.050 < 0.05	Schulte & Berkum, 2015, 14-2022

BARLEY Trial No Country, year (Variety)	Application					DALA days	Portion analysed	Ethephon mg/kg	HEPA mg/kg	Reference
	Form.(g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No					
GAP, France	480 g/ L SL	0.48		100–200	1	56	Application timing BBCH 32-39			
13-2028-01 Ceaux en Loudun, France, 2013 (Cervoise)	480 SL	0.48 (BBCH 39)	0.16	300	1	0 7 12 21 39 71	Green plant Green plant Green plant Green plant Green plant Straw	4.5 0.24 0.15 0.092 < 0.05 0.23	0.053 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	Schulte & Berkum, 2015, 13- 2028 M-529491- 01-1
13-2028-02 Les Franqueses del Valles, Spain, 2013 (Graphic)	480 SL	0.48 (BBCH 39)	0.16	400	1	0 27 72	Green plant Green plant Straw	4.2 0.26 1.7	0.058 (c, 0.081) < 0.05 0.17 (c, 0.17)	Schulte & Berkum, 2015, 13- 2028 M-529491- 01-1
13-2028-03 Citavecchia, Italy, 2013 (Quench, Distichous barley)	480 SL	0.48 (BBCH 39)	0.16	300	1	0 7 14 21 24 62	Green plant Green plant Green plant Green plant Green plant Straw	5.9 0.44 0.087 0.078 0.051 0.39	0.051 < 0.05 < 0.05 < 0.05 < 0.05 0.054	Schulte & Berkum, 2015, 13- 2028 M-529491- 01-1
13-2028-04 Bologna, Italy, 2013 (Federal)	480 SL	0.48 (BBCH 39)	0.24	350	1	0 29 64	Green plant Green plant Straw	3.5 < 0.05 0.24	< 0.05 < 0.05 < 0.05	Schulte & Berkum, 2015, 13- 2028 M-529491- 01-1
14-2020-01 Ceaux en Loudun, France, 2014 (Limpid Winter Barley)	480 SL	0.48 (BBCH 39)	0.16	300	1	0 7 14 21 42 72	Green plant Green plant Green plant Green plant Green plant Straw	5.6 3.0 3.0 0.38 0.095 1.1	0.069 0.055 0.055 < 0.05 < 0.05 < 0.05	Schulte & Berkum, 2015, 14- 2020
14-2020-02 Les Franqueses del Valles, Spain, 2014 (Graphic winterbarley)	480 SL	0.41 (BBCH 43)	0.12	342	1	0 29 64	Green plant Green plant Straw	6.6 0.36 0.97	0.14 < 0.05 0.080	Schulte & Berkum, 2015, 14- 2020
14-2020-03 Bologna, Italy, 2014 (Lutece Winter variety)	480 SL	0.48 (BBCH 39)	0.12	400	1	0 6 14 20 29 64	Green plant Green plant Green plant Green plant Green plant Straw	3.3 1.2 0.34 0.10 < 0.05 0.39	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	Schulte & Berkum, 2015, 14- 2020
14-2020-04 Kristoni Village, Greece, 2014 (Mucho Early, six row, USA)	480 SL	0.48 (BBCH 39)	0.16	300	1	0 48 63	Green plant Green plant Straw	8.2 < 0.05 0.35	0.14 < 0.05 < 0.05	Schulte & Berkum, 2015, 14- 2020

Rye forage and straw

Nine supervised trials have been conducted in rye in Europe.

Table 65 Ethephon residues in rye forage and straw resulting from supervised trials in Europe

RYE	Application					DALT	Portion analysed	Ethephon	Reference
Trial Country, year (Variety)	Formul. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No	days		mg/kg	

RYE	Application					DALT	Portion analysed	Ethephon	Reference
Trial Country, year (Variety)	Formul. (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No	days		mg/kg	
GAP, Austria	660 g/L SL	0.73		100–300	1	–	Application timing BBCH 37–49		
GAP, Germany	660 g/L SL	0.73		100–300	1	–	Application timing BBCH 37–49		
R 2006 0119/0 Le Plessier, France, 2006 (Picasso)	480 g/L SL	0.67 (BBCH 49)	0.22	300	1	0 7 21 75	Green plant Green plant Green plant Straw	6.4 0.31 0.18 0.26	Billian & Telscher, 2007, RA-2516/06 (M-294780-02-1)
R 2006 0287/1 Thetford, UK, 2006 (Ursus)	480 g/L SL	0.67 (BBCH 49)	0.22	300	1	0 7 21 88	Green plant Green plant Green plant Straw	9.6 0.76 0.51 0.34	Billian & Telscher, 2007, RA-2516/06 (M-294780-02-1)
R 2006 0289/8 Svedala, Sweden, 2006 (Matador)	480 g/L SL	0.67 (BBCH 49)	0.22	300	1	0 7 21 71	Green plant Green plant Green plant Straw	13 0.66 0.31 0.33	Billian & Telscher, 2007, RA-2516/06 (M-294780-02-1)
R 2006 0290/1 Anneville Ambourville, France, 2006 (Canovus)	480 g/L SL	0.67 (BBCH 49)	0.22	300	1	0 7 21 70	Green plant Green plant Green plant Straw	7.7 0.53 0.25 0.21	Billian & Telscher, 2007, RA-2516/06 (M-294780-02-1)
R 2006 0292/8 Beiersdorf, Germany, 2006 (Rekrut)	480 g/L SL	0.67 (BBCH 49)	0.22	300	1	0 7 21 77	Green plant Green plant Green plant Straw	9.2 1.1 0.47 0.12	Billian & Telscher, 2007, RA-2516/06 (M-294780-02-1)
R 2007 0174/8 Le Plessier Rosainvillers, France, 2007 (Picasso)	480 g/L SL	0.72 (BBCH 49)	0.24	300	1	0 7 21 49 85	Green plant Green plant Green plant Rest of plant Straw	7.2 1.8 0.24 0.69 0.11	Billian, Erler & Wolters, 2008, RA-2574/07 (M-318501-01-1)
R 2007 0182/9 Burscheid, Germany, 2007 (Fernando)	480 g/L SL	0.72 (BBCH 49)	0.24	300	1	0 7 21 49 86	Green plant Green plant Green plant Rest of plant Straw	4.4 2.5 0.12 0.07 < 0.05	Billian, Erler & Wolters, 2008, RA-2574/07 (M-318501-01-1)
R 2007 0184/5 Anneville Ambourville, France, 2007 (Caroass)	480 g/L SL	0.72 (BBCH 49)	0.24	300	1	0 7 20 48 83	Green plant Green plant Green plant Rest of plant Straw	9.4 1.2 0.28 0.21 0.14	Billian, Erler & Wolters, 2008, RA-2574/07 (M-318501-01-1)
R 2007 0183/7 Thetford, UK, 2007 (Visello)	480 g/L SL	0.72 (BBCH 49)	0.24	300	1	0 7 21 42 103	Green plant Green plant Green plant Rest of plant Straw	9.1 0.52 0.34 0.16 0.07	Billian, Erler & Wolters, 2008, RA-2574/07 (M-318501-01-1)

Wheat forage and straw

Twenty-six supervised trials have been conducted in wheat in Europe and sixteen supervised trials have been conducted in wheat in the USA, which support the use on wheat in Canada.

Table 66 Ethephon residues in wheat forage and straw resulting from supervised trials in Europe

WHEAT	Application					DALT	Portion analysed	Ethephon	Reference
Trial Country, year (Variety)	Form (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No	days		mg/kg	
GAP, Austria	660 g/L SL	0.46		100–300	1	–	Application timing BBCH 37–51		
GAP, Germany	660 g/L SL	0.46		100–300	1	–	Application timing BBCH 37–51		
01R762-1 Braslou, France, 2001 (Isengrain)	480 g/L SL	0.48 (BBCH 51)	0.19	250	1	0 70	Green plant Straw	5.2 0.22	Davies, 2002, 01R762 (M-210306-01-1)
01R762-2 Courdoux, France, 2001 (Ritmo)	480 g/L SL	0.48 (BBCH 49)	0.24	200	1	0 8 23 66	Green plant Green plant Green plant Straw	3.5 3.5 1.5 0.14	Davies, 2002, 01R762 (M-210306-01-1)
01R762-3 Cambridge, UK, 2001 (Claire)	480 g/L SL	0.48 (BBCH 49)	0.16	302	1	0 17 29 72	Green plant Green plant Green plant Straw	6.5 0.77 0.56 0.13	Davies, 2002, 01R762 (M-210306-01-1)
01R762-4 Weilerswist, Germany, 2001 (Drifter)	480 g/L SL	0.48 (BBCH 49)	0.16	300	1	0 66	Green plant Straw	6.2 0.51	Davies, 2002, 01R762 (M-210306-01-1)
01R762-5 Zschortau, Germany, 2001 (Petrus)	480 g/L SL	0.48 (BBCH 49)	0.16	300	1	0 15 40 71	Green plant Green plant Green plant Straw	4.0 1.5 0.58 0.38	Davies, 2002, 01R762 (M-210306-01-1)
GAP, France	480 g/L SL	0.48		100–200	1	70	Application timing BBCH 32-39		
00548BX1 Chaunac, France, 2000 (Aztec)	480 g/L SL	0.48 (BBCH 38)	0.14	333	1	0 14 34 90	Green plant Green plant Green plant Straw	7.7 2.2 1.2 0.15	Ballasteros, 2002, R&D/CRLD/AN/mr/ 0115433 (M-208087-01-1)
00548LY1 La Boisse, France, 2000 (Cyrano)	480 g/L SL	0.48 (BBCH 39)	0.15	320	1	0 16 34 78	Green plant Green plant Green plant Straw	7.0 1.2 0.71 0.15	Ballasteros, 2002, R&D/CRLD/AN/mr/ 0115433 (M-208087-01-1)
00549BX1 Tugeras, France, 2000 (Hyno-valea)	480 g/L SL	0.47 (BBCH 39)	0.14	333	1	90	Straw	0.22	Ballasteros, 2002, R&D/CRLD/AN/mr/ 0115434 (M-208091-01-1)
00549TL1 Baziege, France, 2000 (Tremie)	480 g/L SL	0.48 (BBCH 37–39)	0.17	278	1	91	Straw	0.075	Ballasteros, 2002, R&D/CRLD/AN/mr/ 0115434 (M-208091-01-1)
01R772-1 Boe, France, 2001 (Soissons)	480 g/L SL	0.48 (BBCH 39)	0.19	250	1	0 74	Green plant Straw	7.4 0.56	Davies, 2002, 01R772 (M-210308-01-1)

WHEAT	Application					DALT	Portion analysed	Ethephon	Reference
Trial Country, year (Variety)	Form (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No	days		mg/kg	
01R772-2 Saint Romain De Jeolienas, France, 2001 (Aztec)	480 g/L SL	0.48 (BBCH 39)	0.19	250	1	0 25 35 74	Green plant Green plant Green plant Straw	14 1.5 1.1 < 0.05 0.45	Davies, 2002, 01R772 (M-210308-01-1)
01R772-3 Dodici Morelli, Italy, 2001 (Centauro)	480 g/L SL	0.48 (BBCH 39)	0.14	350	1	0 10 31 57	Green plant Green plant Green plant Straw	12 3.1 1.2 1.3	Davies, 2002, 01R772 (M-210308-01-1)
01R772-4 Paradas Sevilla, Spain, 2001 (Simeto)	480 g/L SL	0.48 (BBCH 39)	0.16	300	1	0 16 29 78	Green plant Green plant Green plant Straw	18 5.7 2.9 0.46	Davies, 2002, 01R772 (M-210308-01-1)
01R772-5 Alcala de Guadaira Sevilla, Spain, 2001 (Sula)	480 g/L SL	0.48 (BBCH 39)	0.16	300	1	0 15 29 76	Green plant Green plant Green plant Straw	14 4.3 3.0 0.12	Davies, 2002, 01R772 (M-210308-01-1)
GAP, France	450 g/L SL [a]	0.38		100–200	1	–	Application timing BBCH 31–37		
R 2004 0564/2 Staffanstorp, Sweden, 2004 (Marshall)	450 g/L SL [a]	0.38 (BBCH 37)	0.13	300	1	0 28 85	Green plant Green plant Straw	7.2 0.27 0.18	Bardel, 2005, RA-2090/04 (M-251226-01-1)
R 2004 0565/0 Leverkusen, Germany, 2004 (Batis)	450 g/L SL [a]	0.38 (BBCH 37)	0.13	300	1	0 42 92	Green plant Green plant Straw	4.9 < 0.05 < 0.05	Bardel, 2005, RA-2090/04 (M-251226-01-1)
R 2004 0566/9 Werl-Oberbergstraße, Germany, 2004 (Winnetou)	450 g/L SL [a]	0.38 (BBCH 37)	0.13	300	1	0 23 81	Green plant Green plant Straw	3.1 0.09 0.06	Bardel, 2005, RA-2090/04 (M-251226-01-1)
R 2004 0567/7 Villettes, France, 2004 (Orvantis)	450 g/L SL [a]	0.38 (BBCH 37)	0.13	300	1	0 36 84	Green plant Green plant Straw	4.5 0.41 0.45	Bardel, 2005, RA-2090/04 (M-251226-01-1)
R 2004 0568/5 Kilkis, Greece, 2004 (Mexicalli)	450 g/L SL [a]	0.38 (BBCH 37)	0.12	300	1	0 9 57	Green plant Green plant Straw	8.3 8.5 0.10	Bardel, 2005, RA-2091/04 (M-251236-02-1)
R 2004 0569/3 Gargas, France, 2004 (Garric)	450 g/L SL [a]	0.38 (BBCH 37)	0.12	300	1	0 25 77	Green plant Green plant Straw	6.1 0.09 0.15	Bardel, 2005, RA-2091/04 (M-251236-02-1)
R 2004 0570/7 Brenes, Spain, 2004 (Don Pedro)	450 g/L SL [a]	0.38 (BBCH 41-45)	0.12	300	1	0 14 78	Green plant Green plant Straw	10 0.26 0.10	Bardel, 2005, RA-2091/04 (M-251236-02-1)
R 2004 0571/5 Pereiro/Alenquer, Portugal, 2004 (Sula)	450 g/L SL [a]	0.38 (BBCH 37)	0.12	300	1	0 35 82 82	Green plant Green plant Straw	6.0 0.07 0.11	Bardel, 2005, RA-2091/04 (M-251236-02-1)
GAP, Belgium	480 g/L SL	0.60		200–400	1	–	Application timing BBCH 37–45		

WHEAT	Application					DALT	Portion analysed	Ethephon	Reference
Trial Country, year (Variety)	Form (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No	days		mg/kg	
R 2006 0123/9 Chaussy, France, 2006 (Isengrain)	480 g/L SL	0.67 (BBCH 39)	0.22	300	1	0 7 21 64	Green plant Green plant Green plant Straw	8.9 0.82 0.54 0.37	Billian & Telscher, 2007, RA-2517/06 (M-294528-01-1)
R 2006 0293/6 Bury St Edmunds, UK, 2006 (Einstein)	480 g/L SL	0.67 (BBCH 39)	0.22	300	1	0 7 21 68	Green plant Green plant Green plant Straw	9.0 0.22 0.11 0.18	Billian & Telscher, 2007, RA-2517/06 (M-294528-01-1)
R 2006 0294/4 Leverkusen, Germany, 2006 (Batis)	480 g/L SL	0.67 (BBCH 39)	0.22	300	1	0 7 21 73	Green plant Green plant Green plant Straw	8.1 0.28 0.14 0.08	Billian & Telscher, 2007, RA-2517/06 (M-294528-01-1)
R 2007 0175/6 Chambourg sur Indre, France, 2007 (Apache)	480 g/L SL	0.72 (BBCH 39)	0.24	300	1	0 7 21 56 85	Green plant Green plant Green plant Rest of plant Straw	11 6.0 0.40 0.23 0.29	Billian, 2008, RA-2575/07 (M-312007-01-1)
R 2007 0186/1 Werl-Westönnen, Germany, 2007 (Ritmo)	480 g/L SL	0.77 (BBCH 49)	0.24	321	1	0 7 21 56 65	Green plant Green plant Green plant Rest of plant Straw	7.6 0.45 0.21 0.18 0.18	Billian, 2008, RA-2575/07 (M-312007-01-1)

[a] 450 g/L SL formulation (150 g/L ethephon + 300 g/L chlormequat-chloride)

Table 67 Ethephon and HEPA residues in wheat forage and straw resulting from supervised trials in Europe obtained using an analytical method involving acid hydrolysis/extraction

WHEAT Trial No Country, year (Variety)	Application					DALA days	Portion analysed	Ethephon mg/kg	HEPA mg/kg	Reference
	Form.(g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No					
GAP, Germany	660 g/L SL	0.46		100–300	1	–	Application timing BBCH 37–51			
13-2029-01 Bursheid, Germany 2013 (Winnetou Soft)	480 SL	0.48 (BBCH 51)	0.16	300	1	0 7 14 21 23 75	Green plant Green plant Green plant Green plant Green plant Straw	3.3 0.46 0.21 0.17 0.17 0.36	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 0.050	Schulte & Berkum, 2015, 13-2029 M-529493-01-1

WHEAT Trial No Country, year (Variety)	Application					DALA days	Portion analysed	Ethephon mg/kg	HEPA mg/kg	Reference
	Form.(g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No					
13-2029-02 Villars-Perwin, Belgium, 2013 (Matrix Soft)	480 SL	0.48 (BBCH 51)	0.16	300	1	0 8 14 21 29 61	Green plant Green plant Green plant Green plant Green plant Straw	3.1 0.16 0.11 0.11 0.11 0.66	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	Schulte & Berkum, 2015, 13-2029 M-529493-01-1
13-2029-03 Little Shelford CB22 5EU, United Kingdom 2013 (Claire Soft)	480 SL	0.48 (BBCH 51)	0.24	200	1	0 38 74	Green plant Green plant Straw	7.5 0.32 1.3	0.076 0.050 0.083	Schulte & Berkum, 2015, 13-2029 M-529493-01-1
14-2018-01 Vechta – Langförden, Germany, 2014 (Winnetou mass-wheat)	480 SL	0.48 (BBCH 51)	0.16	300	1	0 8 14 21 29 71	Green plant Green plant Green plant Green plant Green plant Straw	4.9 0.28 0.29 0.23 0.22 0.44	0.085 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	Schulte & Berkum, 2015, 14-2018 M-532267-01-1
14-2018-02 Burscheid, Germany 2014 (Tobak)	480 SL	0.48 (BBCH 51)	0.16	300	1	0 26 68	Green plant Green plant Straw	7.0 0.23 1.2	0.078 < 0.05 0.15 (c, 0.23)	Schulte & Berkum, 2015, 14-2018 M-532267-01-1
14-2018-03 SG8 8S Great Chishill, United Kingdom, 2014 (Solstice Milling)	480 SL	0.48 (BBCH 51)	0.24	200	1	0 7 15 22 36 64	Green plant Green plant Green plant Green plant Green plant Straw	7.0 0.39 0.27 0.17 0.12 1.2	0.073 < 0.05 < 0.05 < 0.05 < 0.05 0.055	Schulte & Berkum, 2015, 14-2018 M-532267-01-1
14-2018-04 France Chambourg sur Indre, 2014 (Touareg Winter)	480 SL	0.48 (BBCH 51)	0.16	300	1	0 35 77	Green plant Green plant Straw	7.2 0.071 0.57	0.087 < 0.05 < 0.05	Schulte & Berkum, 2015, 14-2018 M-532267-01-1
14-2018-05 Slootdorp, Netherlands 2014	480 SL	0.48 (BBCH 51)	0.12	400	1	0 32 54	Green plant Green plant Straw	5.9 0.23 1.5	0.062 < 0.05 < 0.05	Schulte & Berkum, 2015, 14-2018 M-532267-01-1
GAP, France	480 g/L SL	0.48		100–200	1	70	Application timing BBCH 32–39			

WHEAT Trial No Country, year (Variety)	Application					DALA days	Portion analysed	Ethephon mg/kg	HEPA mg/kg	Reference
	Form.(g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No					
14-2019-01 Gargas, France 2014 (Solehio Soft)	480 SL	0.48 (BBCH 39)	0.16	300	1	0 7 14 21 41 77	Green plant Green plant Green plant Green plant Green plant Straw	7.1 0.27 0.16 0.12 < 0.05 0.29	0.13 < 0.05 < 0.05 < 0.05 < 0.05 0.079	Schulte & Berkum, 2015, 14-2019 M-532272-01-1
14-2019-02 Brenes, Spain 2014 (Don Pedro)	480 SL	0.48 (BBCH 39)	0.16	400	1	0 39 72	Green plant Green plant Straw	6.4 < 0.05 0.21	0.087 < 0.05 0.092 (c, 0.12)	Schulte & Berkum, 2015, 14-2019 M-532272-01-1
14-2019-03 Bologna, Italy 2014 (Mieti Winter)	480 SL	0.48 (BBCH 39)	0.12	300	1	0 7 14 21 30 58	Green plant Green plant Green plant Green plant Green plant Straw	10 0.82 0.30 0.30 0.26 1.2	0.12 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	Schulte & Berkum, 2015, 14-2019 M-532272-01-1
14-2019-04 Aramanha-Santarem, Portugal, 2014 (Artur Nick 2)	480 SL	0.48 (BBCH 39)	0.16	300	1	0 60 110	Green plant Green plant Straw	16 0.075 0.44	0.21 < 0.05 0.084 (c, 0.061)	Schulte & Berkum, 2015, 14-2019 M-532272-01-1
13-2030-01 Castelnau d'estretefonds, France, 2013 (Hystar Soft)	480 SL	0.48 (BBCH 39)	0.16	300	1	0 7 14 21 45 80	Green plant Green plant Green plant Green plant Green plant Straw	5.7 0.50 0.31 0.24 0.16 0.86	0.27 < 0.05 < 0.05 < 0.05 < 0.05 0.051	Schulte & Berkum, 2015, 13-2030 M-529488-01-1
13-2030-02 El Campillo, Spain, 2013 (Artur Nick Soft)	480 SL	0.52 (BBCH 39)	0.16	322	1	0 43 64	Green plant Green plant Straw	17 0.21 0.84	0.24 < 0.05 < 0.05	Schulte & Berkum, 2015, 13-2030 M-529488-01-1

WHEAT Trial No Country, year (Variety)	Application					DALA days	Portion analysed	Ethephon mg/kg	HEPA mg/kg	Reference
	Form.(g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No					
13-2030-03 Tarquinia, Italy 2013 (Quality Soft)	480 SL	0.48 (BBCH 39)	0.16	300	1	0 7 14 21 24 63	Green plant Green plant Green plant Green plant Green plant Straw	6.9 0.48 0.17 0.19 0.16 1.7	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 0.12	Schulte & Berkum, 2015, 13-2030 M-529488-01-1
13-2030-04 Bologna, Italy 2013 (Serio Soft)	480 SL	0.48 (BBCH 39)	0.14	350	1	0 25 62	Green plant Green plant Straw	5.6 0.050 0.30	0.11 < 0.05 0.058	Schulte & Berkum, 2015, 13-2030 M-529488-01-1

Table 68 Residues of ethephon in wheat straw resulting from supervised trials in the USA

WHEAT	Application					DALT	Ethephon	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No	days	mg/kg	
GAP, Canada	240 g/L SL	0.60		30–300	1	35	Application from BBCH 37–49	
10223-W1 Arkansas City, Kansas, USA, 1981 (Newton)	480 g/L SL	0.84 (late boot)	–	–	1	55	0.28 (0.08, 0.63, 0.27, 0.14) [a]	Harrison, 1981, 10223 (M-187972-01-1)
10223-W2 Landisville, Pennsylvania, USA, 1981 (Redcoat)	480 g/L SL	0.84 (boot)	–	–	1	49	0.59 (< 0.02, 0.30, 0.81, 1.21)	Harrison, 1981, 10223 (M-187972-01-1)
10223-W3 Skaneateles, New York, USA, 1981 (Hauser)	480 g/L SL	0.84 (boot)	–	–	1	41	5.84 (7.60, 3.43, 4.61, 7.71)	Harrison, 1981, 10223 (M-187972-01-1)
10223-W4 Newton, Iowa, USA, 1981 (Sage Hard Red)	480 g/L SL	0.56 (early boot)	–	–	1	54	0.39 (0.29, 0.40, 0.49)	Harrison, 1981, 10223 (M-187972-01-1)
10223-W5 Sandusky, Michigan, USA, 1981 (Arthur)	480 g/L SL	0.56 (early boot)	–	–	1	62	3.37 (4.44, 2.19, 3.48)	Harrison, 1981, 10223 (M-187972-01-1)
10223-W6 Newcastle, Ohio, USA, 1981 (Titan)	480 g/L SL	0.56 (early boot)	–	–	1	57	0.05 (< 0.02, 0.11, 0.06)	Harrison, 1981, 10223 (M-187972-01-1)

WHEAT	Application					DALT	Ethephon	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No	days	mg/kg	
10223-W7 Glyndon, Minnesota, USA, 1981 (Era)	480 g/L SL	0.84 (boot)	–	–	1	57	4.24 (4.51, 3.66, 3.66, 5.11)	Harrison, 1981, 10223 (M-187972-01-1)
10223-W8 Powell, Wyoming, USA, 1981 (Prodax)	480 g/L SL	0.56 (mid boot)	–	–	1	64	0.16	Harrison, 1981, 10223 (M-187972-01-1)
10223-W9 Warsaw, Illinois, USA, 1981 (Pioneer)	480 g/L SL	0.56	–	–	1	48	1.33 (1.36, 1.28, 1.40, 1.30)	Harrison, 1981, 10223 (M-187972-01-1)
SARS-89-CO-24 Brighton, Colorado, USA, 1989 (Hawk)	480 g/L SL	0.56 (aerial) (late boot to 1/4 inflorescence emerged)	2.0	28	1	35 40 60	1.3 (1.1, 1.4, 1.5) 1.7 (1.5, 1.7, 1.9) 3.4 (4.5, 3.3, 2.3)	Conn, 1992, SARS-89-24 (M-187553-01-1)
		0.56 (ground) (late boot to 1/4 inflorescence emerged)	0.83	67	1	35 40 60	1.5 (1.5, 1.5, 1.6) 1.5 (1.6, 1.4, 1.4) 1.3 (1.2, 1.3, 1.4)	
SARS-89-KS-24 Sedan, Kansas, USA, 1989 (Thinderbird)	480 g/L SL	0.56 (aerial) (3/4 inflorescence emerged)	2.1	27	1	35 40 60	3.2 (4.3, 2.4, 3.0) 1.1 (0.99, 0.83, 1.4) 0.31 (0.39, 0.34, 0.21)	Conn, 1992, SARS-89-24 (M-187553-01-1)
		0.56 (ground) (3/4 inflorescence emerged)	0.86	65	1	35 40 60	2.7 (2.5, 3.1, 2.6) 1.3 (1.1, 1.5, 1.3) 0.78 (0.56, 0.86, 0.91)	
SARS-89-MN-24 East Grand Forks, Minnesota, USA, 1989 (Marshall)	480 g/L SL	0.56 (aerial) (late boot)	2.0	28	1	35 41 59	1.0 (1.1, 0.98, 0.96) 1.3 (1.0, 1.2, 1.6) 0.29 (0.25, 0.39, 0.24)	Conn, 1992, SARS-89-24 (M-187553-01-1)
		0.56 (ground) (late boot)	0.86	65	1	35 41 59	1.4 (0.84, 1.9, 1.5) 1.7 (1.6, 1.6, 1.8) 0.66 (0.56, 0.77, 0.64)	
SARS-89-ND-24 Northwood, North Dakota, USA, 1989 (Butte 86)	480 g/L SL	0.56 (aerial) (late boot)	2.0	28	1	35 40 60	2.7 (3.4, 2.3, 2.5) 1.6 (2.4, 0.72, 1.7)) 0.20 (0.39, 0.09, 0.11)	Conn, 1992, SARS-89-24 (M-187553-01-1)

WHEAT	Application					DALT	Ethephon	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	kg ai/hL	Water (L/ha)	No	days	mg/kg	
		0.56 (ground) (late boot)	0.86	65	1	35 40 60	2.0 (2.5, 1.6, 1.9) 1.4 (1.3, 1.5, 1.5) 0.33 (0.43, 0.34, 0.23)	
SARS-89-WA-24 Ephrata, Washington, USA, 1989 (Madson)	480 g/L SL	0.56 (aerial) (late boot)	2.0	28	1	40 60 70	0.95 (1.5, 0.63, 0.73) 0.95 (0.85, 0.90, 1.1) 1.5 (1.7, 1.2, 1.5)	Conn, 1992, SARS-89-24 (M-187553-01-1)
		0.56 (ground) (late boot)	0.73	77	1	40 60 70	1.2 (0.41, 1.4, 1.8) 1.8 (1.9, 1.7, 1.9) 1.3 (1.3, 1.1, 1.6)	

[a] Mean residue. Analytical results of replicate samples are in parentheses

Table 69 Ethephon residues in cotton lint and gin trash resulting from supervised trials in Europe, the USA and Brazil

COTTON Trial No. Country, year (Variety)	Application					DALT days	Portion analysed	Ethephon mg/kg	Reference
	Form. (g ai/L & type)	kg ai/ha	kg ai/hL	Water (L/ha)	No				
GAP, Greece	480 g/L SL	1.44		500–600	1	7			
EUROPE									
94681SE1 Carlota-AL, Spain, 1994 (Cnema 111)	540 g/L SC [b]	1.44	0.36	400	1	0 3 7	Lint	0.12 11.5 10.1	Richard & Muller, 1995, R&D/CRLD/AN/bd/ 9515911 (M-163133-01-1)
94681SE2 Carlota-ZA, Spain, 1994 (Cnema 111)	540 g/L SC [b]	1.44	0.36	400	1	0 3 7	Lint	< 0.10 8.08 1.92	Richard & Muller, 1995, R&D/CRLD/AN/bd/ 9515911 (M-163133-01-1)
94681SE3 Ecija, Spain, 1994 (Cnema 111)	540 g/L SC [b]	1.44	0.36	400	1	0 3 7	Lint	< 0.10 33.3 14.4	Richard & Muller, 1995, R&D/CRLD/AN/bd/ 9515911 (M-163133-01-1)
95723SE1 Ciatr Sevilla, Spain, 1995 (Corona)	540 g/L SC [b]	1.44	0.33	440	1	7	Lint	2.06	Muller, 1996, R&D/CRLD/AN/bd/ 9516706 (M-163236-01-1)
GAP, USA	765 g/L SC	2.24		28-234	1	7			
GAP, USA	720 g/L SC	2.24		19-94	1	7			
USA									

COTTON Trial No. Country, year (Variety)	Application					DALT days	Portion analysed	Ethephon mg/kg	Reference
	Form. (g ai/L & type)	kg ai/ha	kg ai/hL	Water (L/ha)	No				
94-0284 Wharton Co., TX, USA, 1994 (Deltapine 20)	540 g/L SC	2.31 (ground)	1.54	150	1	7	Gin trash	8.41 (8.63, 8.18) [a]	See, 1995, USA94I01R (M-253436-01-1)
94-0285 Castro Co., TX, USA, 1994 (Paymaster 145)	540 g/L SC	2.25 (ground)	1.53	147	1	7	Gin trash	40.5 (43.4, 37.5)	See, 1995, USA94I01R (M-253436-01-1)
94-0286 Floyd Co., TX, USA, 1994 (Paymaster HS-200)	540 g/L SC	2.43 (ground)	1.54	158	1	7	Gin trash	11.1 (10.5, 11.7)	See, 1995, USA94I01R (M-253436-01-1)
94-0287 Fresno, CA, USA, 1994 (Maxxa)	540 g/L SC	2.22 (ground)	1.59	139	1	7	Gin trash	17.1 (15.3, 18.8)	See, 1995, USA94I01R (M-253436-01-1)
94-0288 Washington Co., MS, USA, 1994 (DPL 50)	540 g/L SC	2.26 (ground)	1.59	142	1	7	Gin trash	54.2 (56.3, 52.0)	See, 1995, USA94I01R (M-253436-01-1)
94-0289 Houseton Co., AL, USA, 1994 (DPL 5415)	540 g/L SC	2.28 (ground)	1.64	139	1	8	Gin trash	45.5 (41.1, 49.8)	See, 1995, USA94I01R (M-253436-01-1)
94-0290 Madera Co., CA, USA, 1994 (Maxa)	540 g/L SC	2.17 (ground)	1.18	184	1	6	Gin trash	150 (141, 158)	See, 1995, USA94I01R (M-253436-01-1)
94-0291 Fayette Co., TN, USA, 1994 (Stoneville 453)	540 g/L SC	2.25 (ground)	1.57	143	1	7	Gin trash	25.1 (25.8, 24.4)	See, 1995, USA94I01R (M-253436-01-1)
94-0292 Crittenden Co., AR, USA, 1994 (Stoneville 453)	540 g/L SC	2.27 (ground)	1.62	140	1	7	Gin trash	13.5 (12.0, 15.0)	See, 1995, USA94I01R (M-253436-01-1)
94-0293 Burleson Co., TX, USA, 1994 (DP&L 5415)	540 g/L SC	2.24 (ground)	1.56	144	1	9	Gin trash	6.66 (6.46, 6.86)	See, 1995, USA94I01R (M-253436-01-1)
94-0393 Hale Co., TX, USA, 1994 (Paymaster HS-200)	540 g/L SC	2.32 (ground)	1.53	151	1	7	Gin trash	55.7 (44.5, 66.8)	See, 1995, USA94I01R (M-253436-01-1)
94-0394 Hale Co., TX, USA, 1994 (Paymaster 145)	540 g/L SC	2.27 (ground)	1.45	157	1	7	Gin trash	28.9 (26.9, 30.8)	See, 1995, USA94I01R (M-253436-01-1)

[a] Mean residue. Analytical results in parentheses

[b] 540 g/L SL formulation (180 g/L ethephon + 360 g/L chlormequat-chloride)

FATE OF RESIDUES IN STORAGE AND IN PROCESSING

Information and Data from Residues in Processed Commodities

The Meeting received information on hydrolysis relevant to food processing; and processing of apples, grapes, olives, tomatoes, barley, wheat, and cotton seed to their respective processed commodities.

Hydrolysis

The hydrolytic behaviour of ethephon was investigated under conditions relevant to major food processing operations such as pasteurization (20 minutes at 90 °C, pH 4), brewing, baking and boiling (60 minutes at 100 °C, pH 5) and sterilisation (20 minutes at 120 °C, pH 6) using [^{14}C]ethephon (Selzer, 2002, CP02/001, [M-211072-01-1).

[^{14}C]Ethephon was spiked into citrate buffer solutions which were adjusted to the required pH-value with sodium hydroxide. For each set of conditions there were two trials at a spiking level of 0.1 mg/L and two trials at a level of 1.0 mg/L. The spiked buffer solutions were heated in closed stainless steel reaction vessels using either a water bath or an autoclave. The heating time was measured from the moment when the temperature inside the vessels reached the required value. At the end of the fixed time the vessels were immersed immediately in an ice bath. After cooling, the outlets of the vessels were connected to a series of adsorption bottles containing a saturated solution of pyridinium hydrobromide perbromide (PHB) and the headspace gas was passed through the bottles in order to trap the ethylene formed during the test.

The total radioactivity remaining in the buffer solutions and the radioactivity trapped in the bottles were measured by LSC. The individual compounds present in the buffer solutions were identified and quantified by HPLC against reference standards. In order to characterize the radioactive compounds released in the gaseous phase, a series of trials was performed under the same conditions with unlabelled ethephon and the gaseous phase was analysed by GC/FID.

The overall radioactivity recovery was in the range of 82 to 95%, except in three trials where the recovery was only about 50% because of losses during the gas trapping procedure (Table 70).

Under the conditions representative of pasteurization, more than 80% of the ethephon remained unchanged and about 10% was decomposed to ethylene. Besides the parent compound, very small amounts of HEPA and an unknown compound were formed in the buffer solution. Under the conditions representative of brewing, baking, boiling and sterilization, degradation of ethephon was complete. Based on the trials which gave acceptable overall recoveries, at least 75% of the substance was decomposed to ethylene. The buffer solutions contained small quantities of HEPA and an unknown compound, but these amounted to less than 10% of the initial radioactivity.

Table 70 Quantification and characterization of radioactivity recovered under hydrolysis conditions simulating processing

Simulated process	Initial level of ethephon	Total radioactivity recovered (%[a])	Radioactivity in solution (%[a])				Ethylene (%[a])
			Total	Ethephon	HEPA	Unknown	
Pasteurisation (90 °C, pH 4, 20 min)	0.1 mg/L	93.0	83.31	80.29	1.46	1.56	9.67
	1.0 mg/L	93.4	82.55	80.74	0.93	0.66	10.82
Baking, brewing, boiling (100 °C, pH 5, 60 min)	0.1 mg/L	82.6	6.94	n.d.	4.70	2.24	75.64
	1.0 mg/L	85.7	8.76	n.d.	7.86	0.90	76.93

Simulated process	Initial level of ethephon	Total radioactivity recovered (%[a])	Radioactivity in solution (%[a])				Ethylene (%[a])
			Total	Ethephon	HEPA	Unknown	
Sterilization (120 °C, pH 6, 20 min)	0.1 mg/L	51.2 [b]	11.72	n.d.	2.66	5.44	39.45 [b]
	1.0 mg/L	82.6	4.14	n.d.	2.91	1.23	78.45

[a] All results are expressed as percentage of initial radioactivity and represent the mean of two replicates, except for brewing, baking and boiling at 1.0 mg/L, for which the results of only one trial are shown, due to low overall recovery in the other trial (49.0% of initial radioactivity).

[b] For sterilisation at 0.1 mg/L both trials resulted in low overall recoveries, probably due to a leak in the gas trapping system and underestimation of the ethylene released.

Apples

The first study was conducted in the USA during 1989–1990 on processing of apples harvested at a DALT of 7 days in one trial in Washington into juice and wet and dry pomace (Nygren, 1990, USA89E32, [M-187583-01-1]). Apples were stored frozen prior to processing. The processing procedure consisted of first washing the thawed apples. The use of frozen apples resulted in a high yield of juice end compared to normal commercial processing attributed to the partial destruction of cell walls during freezing. A flow chart of the processing operations is shown below with analysed fractions underlined.

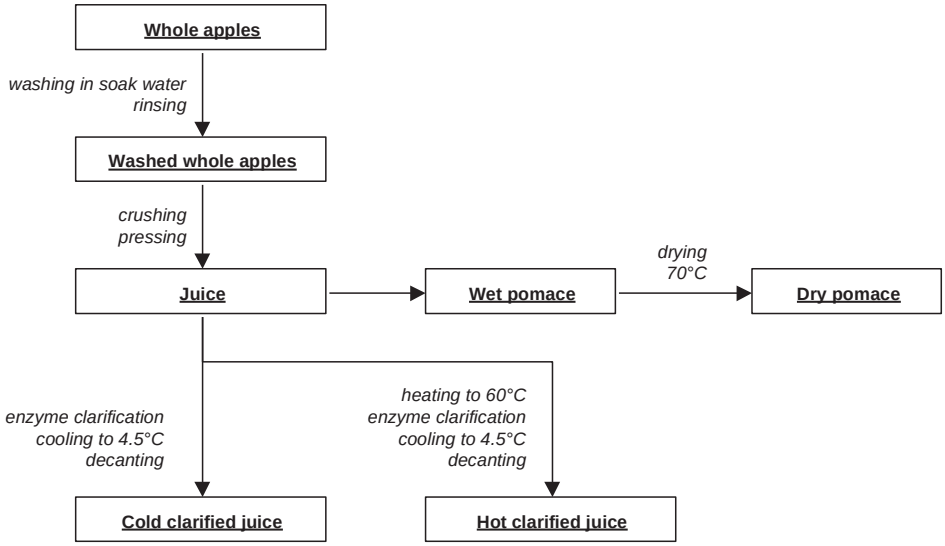


Figure 5 Apple processing

Residues of ethephon were determined using method SOP 90070. The LOQ was 0.05 mg/kg in apples and processed fractions. In the method validation, recoveries at fortification levels of 0.20–2.0 mg/kg were 104% in fruit, 76–85% in wet pomace, 105% in dry pomace, 98% in fresh juice, 64–107% in cold clarified juice and 95% in hot clarified juice.

The samples were frozen after collection (< –16 °C) and stored frozen until extraction and analysis. The maximum period of storage was 13 months for apple fruit and 9 months for processed commodities.

Residues determined in apple fruit and processed fractions are shown in Table 71. Results indicate that ethephon residues concentrate from whole fresh fruit to juice with processing factors

between 1.24 (fresh juice) and 1.57 (cold clarified juice). This result may be accounted for by the high water solubility of ethephon. A processing factor of almost 2 was observed in dried pomace as compared to whole fresh fruit. This indicates that part of the ethephon residues were eliminated during the drying process, probably due to co-sublimation with the water.

Table 71 Residues of ethephon in apple fruit and processed commodities

Commodities	Ethephon, mg/kg	Processing factor
Apple fruit (RAC)	0.37	1.0
Washed apple fruit	0.28	0.8
Wet pomace	0.24	0.6
Dry pomace	0.73	2.0
Fresh juice	0.46	1.2
Cold clarified juice	0.58	1.6
Hot clarified juice	0.56	1.5

The second study was conducted in Europe during 2003 on processing of apples harvested at a DALT of 14 days in a total of four trials in Europe (two in Italy, one in Portugal and one in Spain) into apple sauce, juice and wet and dry pomace (Bardel, Hoffmann & Eberhardt, 2005, RA-3610/03, [M-254102-01-1]). Apples were stored frozen prior to processing. Apples were partially defrosted and washed with tap water before processing.

The apples were then crushed and pressed into raw juice and wet pomace. The juice was filtered and subjected to ultrafiltration for 2–4 hours at room temperature. The resulting cleared juice was filtered to obtain clear apple juice, and pasteurised in glass bottles to give pasteurised juice.

The washed and thawed apples were cut into small pieces manually with a knife and then placed in a stainless steel pot. Water was added and the apples heated until all fruit parts were soft (cooking time 20 minutes at 96–99 °C). The apples were then crushed using a stainless steel food mill to remove cores and peel (pomace) and yield raw sauce. The pomace was discarded. The raw apple sauce was filled into a preserving bottle and pasteurised to give pasteurised sauce.

Residues of ethephon were determined by method 00903/E001 using LC-MS/MS. The LOQ was 0.05 mg/kg in apples and processed fractions. In the method validation, mean recoveries at fortification levels of 0.05–0.5 mg/kg were 104% in fruit, 104% in juice, 92% in pomace, 99% in washings and 96% in sauce/raw stewed fruit.

The samples were frozen after collection (< –14 °C) and stored frozen until extraction and analysis. The maximum period of storage was 462 days (< 16 months) for apple fruit and 71 days (2.3 months) for processed commodities.

Residues determined in apple fruit and processed fractions are shown in Table 72. At harvest, residues in apples were 0.06–0.63 mg/kg. Residues in the processed commodities were < 0.05–0.41 mg/kg in apple sauce, < 0.05–0.30 mg/kg in juice and < 0.05–0.71 mg/kg in wet pomace. Processing factors were calculated to be, 0.4–< 0.8, < 0.4–< 0.8 and 0.3–1.1, for apple sauce, juice and wet pomace, respectively.

Table 72 Residues of ethephon in apple fruit and processed commodities

Trial	Commodities	Ethephon, mg/kg	Processing factor
R2003 0153/7 Italy	Apple fruit (RAC)	0.14	
	Washed apple fruit	0.15	
	Washing water	0.07	
	Preparation of apple sauce		
	Raw sauce	0.08	
	Pasteurised sauce	0.07	0.5
	Preparation of apple juice		
	Wet pomace	0.06	0.4

Trial	Commodities	Ethephon, mg/kg	Processing factor
	Raw juice	< 0.05	
	Pasteurised juice	< 0.05	< 0.4
R2003 0423/4 Italy	Apple fruit (RAC)	0.06	
	Washed apple fruit	< 0.05	
	Washing water	< 0.05	
	Preparation of apple sauce		
	Raw sauce	< 0.05	
	Pasteurised sauce	< 0.05	< 0.8
	Preparation of apple juice		
	Wet pomace	< 0.05	< 0.8
	Raw juice	< 0.05	
	Pasteurised juice	< 0.05	< 0.8
R2003 0424/2 Portugal	Apple fruit (RAC)	0.63	
	Washed apple fruit	0.40	
	Washing water	0.18	
	Preparation of apple sauce		
	Raw sauce	0.26	
	Pasteurised sauce	0.24	0.4
	Preparation of apple juice		
	Wet pomace	0.71	1.1
	Raw juice	0.31	
	Pasteurised juice	0.30	0.5
R2003 0425/0 Spain	Apple fruit (RAC)	0.39	
	Washed apple fruit	0.32	
	Washing water	0.08	
	Preparation of apple sauce		
	Raw sauce	0.41	
	Pasteurised sauce	0.42	1.1
	Preparation of apple juice		
	Wet pomace	0.10	0.3
	Raw juice	0.16	
	Pasteurised juice	0.15	0.4

Grapes

The first study was conducted in the USA on processing of grapes harvested at a DALT of 42–47 days in six field trials in California during 1978 into dried grape and raisin waste (Harrison, 1979, 279C2, [M-188057-01-1]). Ethephon was applied as a foliar spray to Thompson seedless grapes at a rate of 0.56 kg ai/ha. The processing procedure is not reported.

The samples of grape berries, dried grapes and raisin waste were analysed according to the analytical method (AmChem Products Inc., 1975). The LOQ was 0.01 mg/kg. The mean recovery at a fortification level of 0.20 mg/kg in grapes was 107% (RSD 15.5%), at 0.40 mg/kg in dried grapes was 105% (RSD 4.6%), and at 10.0 mg/kg in raisin waste was 102% (RSD 8.9%).

The samples were stored frozen (–34 °C) until they were freeze dried, and the freeze-dried samples then stored frozen (–12 °C) or at ambient temperature. The maximum period of storage was 5 months.

Residues determined in grapes, dried grapes and raisin waste are shown in Table 73. Results give variable processing factors ranging between 0.79 and 8.5 for dried grapes and 19 and 82 for raisin waste. Considering the relationship of concentrations apple wet pomace and apple dry pomace, it is likely that the processing factor for dried raisin would be higher than 1.

Table 73 Residues of ethephon in grapes, dried grapes and raisin waste (Harrison, 1979)

Trial	Commodities	Ethephon, mg/kg	Processing factor

Trial	Commodities	Ethephon, mg/kg	Processing factor
Dinuba, CA, USA	Grapes (RAC)	0.46	
	Dried grape	0.46	1.0
	Raisin waste	9.28	20
Fresno, CA, USA	Grapes (RAC)	0.47	
	Dried grape	1.49	3.2
	Raisin waste	38.0	82
Parlier, CA, USA	Grapes (RAC)	0.15	
	Dried grape	0.21	1.4
	Raisin waste	3.27	22
Madeira [a], CA, USA	Grapes (RAC)	1.72	
	Dried grape	1.37	0.79
	Raisin waste	31.8	19
Kingsburg, CA, USA	Grapes (RAC)	0.24	
	Dried grape	0.22	0.89
	Raisin waste	4.72	19
Fresno, CA, USA	Grapes (RAC)	0.42	
	Dried grape	3.60	8.5
	Raisin waste	29.7	70

[a] Mite infested trial

Another study was conducted in 1995 on processing of grapes harvested at DALT of 35–38 days in two field trials in France to must and red wine (Grolleau, 1997, EA950185, [M-188232-01-1]).

The samples were shipped refrigerated (approximately 5 °C) to the processing facility on the day of collection in order to start immediately with the wine preparation procedure. The vinification procedure is shown below. Alcoholic fermentation takes about 4 weeks, malolactic fermentation about 3 weeks and clarification about 8 weeks. Several oenological additives were used in the process: potassium metabisulphite, yeast (*Saccharomyces cerevisiae*), sugar, lactic bacteria (*Leuconostoc oenos*), gelatine and metatartaric acid.

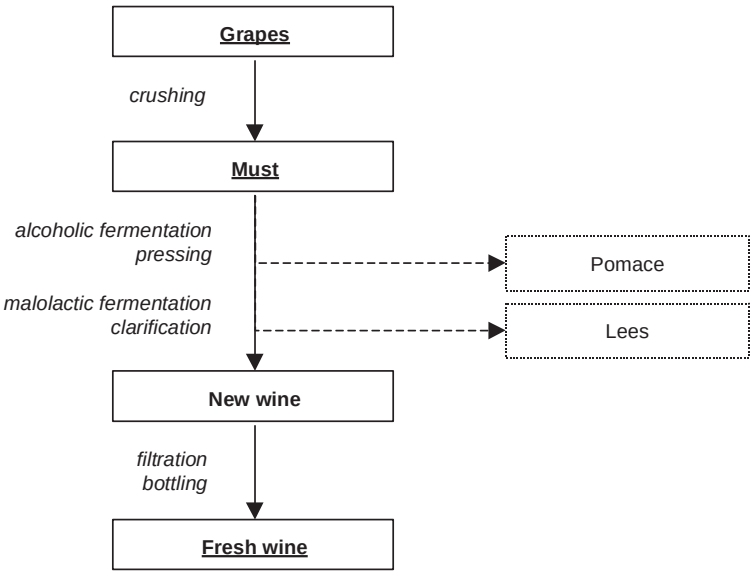


Figure 6 Processing of grapes to red wine

The samples of grape, must and red wine were analysed using the analytical method referenced in “Analytical Method for Residues of Pesticides” Part II-89, 5th Edition, SDU

Publishers, The Netherlands (1988). This method involves extraction with methanol, methylation with diazomethane and determination by GC/FPD and is similar to SOP 90070, and was validated on grapes prior to use. The LOQ was 0.10 mg/kg. Procedural recoveries at fortification levels of 0.10–2.0 mg/kg in grapes were 70–93% (mean 79%, RSD 9.9), at 0.10–0.25 mg/kg in must were 70–90%, and at 0.10–0.50 mg/kg in wine were 90–123%.

The samples were frozen after collection and stored frozen (< –18 ºC) until extraction and analysis. The maximum period of storage was 4 months for grapes and must, and 9 days for wine (after bottling).

Residues determined in grapes, must and red wine are shown in Table 74. The concentrations of ethephon in must were found to be comparable to or slightly lower than the concentrations in whole fruit. A concentration from fruit to wine was found, with processing factors of 1.4 and 2.1.

Table 74 Residues of ethephon in grapes, must and red wine (Grolleau, 1997)

Trial	Processed commodities	Ethephon, mg/kg	Processing factor
EA950185-FR01 France, 1995 (variety Syrah)	Grapes (RAC)	0.37	
	Must	0.34	0.9
	Wine	0.77	2.1
EA950185-FR02 France, 1995 (variety Grenache)	Grapes (RAC)	0.25	
	Must	0.17	0.7
	Wine	0.36	1.4

Two additional studies were conducted in 2003 in which processing of grapes from four trials in Europe (one in Germany, two in France and one in Greece) to raw juice and wine (Bardel & Hoffmann, 2005, RA-3680/03 and Amendment 1, [M-249278-02-1; Bardel & Hoffmann, 2005, RA-3681/03 and Amendment 1, [M-249332-02-1).

Juice was prepared through the following procedure: grapes were destemmed, washed and crushed, and the mash pressed to give raw juice and wet pomace. The raw juice was depectinised by heating for approximately 30 seconds at 80–85 °C, cooled and treated with pectolytic enzyme for 1 hour at room temperature. The cooled juice was filtered and pasteurised, and a juice sample collected.

Wine was prepared through the following procedure: grapes were crushed and destemmed, and the mash heated to 80 °C and then cooled down to fermentation temperature. The mash was pressed in a cloth press, and a sample of the resulting pomace collected. The must was filled into vessels and potassium hyposulphite and bentonite added. After clarifying, the must was decanted from the lees and a sample of must collected. Alcoholic fermentation was started by the addition of pure-culture yeast. After fermentation (approximately 6 weeks), the yeast was removed by decanting and filtration. The young wine was sulphited and finished for 2 months (trial R 2003 0468/4) and for 3 days (trials R 2003 0971/6, R 2003 0469/2 and R 2003 0973/2). The wine was filtered and bottled, and samples of bottled wine collected.

The samples of grapes and processed commodities were analysed using method 00903, which was validated prior to use. The LOQ was 0.05 mg/kg. Mean procedural recoveries at fortification levels of 0.05–0.5 mg/kg were 95% (RSD 5.9) in grapes, 99% (RSD 15.2%) in must, 103% (RDS 4.1%) in wine, 81% (RSD 5.7%) in pomace and 114% (RSD 4.2%) in juice.

The samples were frozen after collection and stored frozen (< –18 ºC) until extraction and analysis. The maximum period of storage was 14 months.

Residues determined in grape, juice, pomace, must and wine are shown in Table 75. The ethephon concentrations in juice, pomace and must were found to be comparable to or lower than those in whole fruit. The processing factors were in the range 0.5–1.1 for juice, 0.4–1.1 for wet pomace, 0.8–1.0 in must and 0.7–1.5 in wine.

Table 75 Residues of ethephon in grape, juice, pomace and wine

Trial	Commodities	Ethephon, mg/kg	Processing factor	Reference
R 2003 0468/4 Germany, 2003 (variety Spätburgunder)	Grapes bunch (RAC)	0.67		Bardel & Hoffmann, 2005, RA-3680/03 and Amendment 1 (M-249278-02-1)
	Grape berries	0.55		
	Juice	0.53	0.8	
	Pomace (wet)	0.76	1.1	
	Must	0.52	0.8	
	Wine (bottled)	0.98	1.5	
R 2003 0971/6 N France, 2003 (variety Gamay)	Grapes bunch (RAC)	0.19		
	Grape berries	0.22		
	Juice	0.21	1.1	
	Pomace (wet)	0.08	0.4	
	Must	0.19	1.0	
	Wine (bottled)	0.14	0.7	
R 2003 0469/2 Greece, 2003 (variety Roditis)	Grapes bunch (RAC)	0.22		Bardel & Hoffmann, 2005, RA-3681/03 and Amendment 1 (M-249332-02-1)
	Grape berries	0.20		
	Juice	0.12	0.5	
	Pomace (wet)	0.14	0.6	
	Must	0.21	1.0	
	Wine (bottled)	0.26	1.2	
R 2003 0973/2 S France, 2003 (variety Syrah)	Grapes bunch (RAC)	0.20		
	Grape berries	0.17		
	Juice	0.14	0.7	
	Pomace (wet)	0.18	0.9	
	Must	0.15	0.8	
	Wine (bottled)	0.19	1.0	

Olive

A study was conducted in 2007 on processing of olives harvested at a DALT of 11 days in four trials in Spain to table olives and olive oil (Fernandez, 2009, 07 D OL BY P/A, [M-352734-01-1]).

Table olives: Olives were placed into a 2–4% NaOH solution and oscillated for 5–8 hours. Afterwards, the olives were immersed in water for 12–20 hours to eliminate the NaOH from the fruit. After watering, the olives were put into a 10% NaCl solution to give table olives.

Olive oil: Olives were washed in tap water, and the washed olives then milled to a pulp. The pulp was mixed in a thermo-malaxer for approximately 30 minutes. Boiling water was added after the first 20 minutes of mixing, to give a water:pulp ratio of 1:1. The mixture was pressed into a liquid phase (oil and water) and solid phase (press cake). The liquid phase was decanted and centrifuged and the raw oil separated. Filtration of the raw oil yielded virgin oil. Soda was added to raw oil and the mixture heated to 60–70 °C for 30 minutes. The oil was separated from the sediment (soap) by filtration to give refined oil.

Residues of ethephon were determined using method 00918. The LOQ was 0.05 mg/kg. Procedural recoveries at fortification levels of 0.05–5.0 mg/kg in olives were within the acceptable range of 70–120%, RSD < 20%. Procedural recoveries at fortification levels of 0.05–0.50 mg/kg in oil were within the acceptable range of 70–120%, RSD < 20%.

The samples were frozen after collection and stored frozen (–18 ºC) until extraction and analysis. The maximum period of storage was 7 months for olives RAC and 7 months for table olives.

Residues determined in olives, table olives and oil are shown in Table 76. Concentrations of ethephon were 1.6–4.3 mg/kg in olive RAC. There is no significant transfer of residues of ethephon into the processed commodities, and residues in table olives and virgin and refined oil were < 0.05 mg/kg in all trials.

Table 76 Residues of ethephon in olives, table olives and olive oil

Trial	Commodities	Ethephon, mg/kg	Processing factor
07 D OL BY P01 Spain, 2007 (variety Manzanillo)	Olives (RAC)	4.3	
	Table olives	< 0.05	< 0.01
07 D OL BY P02 Spain, 2007 (variety Manzanillo)	Olives (RAC)	2.2	
	Table olives	< 0.05	< 0.02
07 D OL BY P03 Spain, 2007 (variety Hojiblanca)	Olives (RAC)	2.5	
	Table olives	< 0.05	< 0.02
	Virgin oil	< 0.05	< 0.02
	Refined oil	< 0.05	< 0.02
07 D OL BY P04 Spain, 2007 (variety Hojiblanca)	Olives (RAC)	1.6	
	Table olives	< 0.05	< 0.03
	Virgin oil	< 0.05	< 0.03
	Refined oil	< 0.05	< 0.03

Tomato

A study was conducted in 1989 on processing of tomatoes harvested at a DALT in 3 days of trials in the USA (California) to juice, paste and puree (Nygren, 1991, USA89E30, [M-187599-01-1]).

The processing was performed using commercial equipment and each of the processed fractions generated was to industry specifications. A simplified flow chart of the processing is shown in the following Figure. The tomato processed fractions collected were fresh whole tomatoes, washed whole tomatoes, wet pomace, dry pomace, canned fresh juice, canned puree, canned paste and canned juice reconstituted from tomato concentrate.

Residues of ethephon were determined using method SOP 90070. The LOQ was 0.02 mg/kg and the method was validated prior to use. The mean procedural recovery at a fortification level of 0.2 mg/kg in tomatoes was 109% (n=9), and recoveries at 0.5 mg/kg in processed commodities were 70–105%.

The samples were frozen after collection (–15 °C) and stored frozen until extraction and analysis. The maximum period of storage was 17 months.

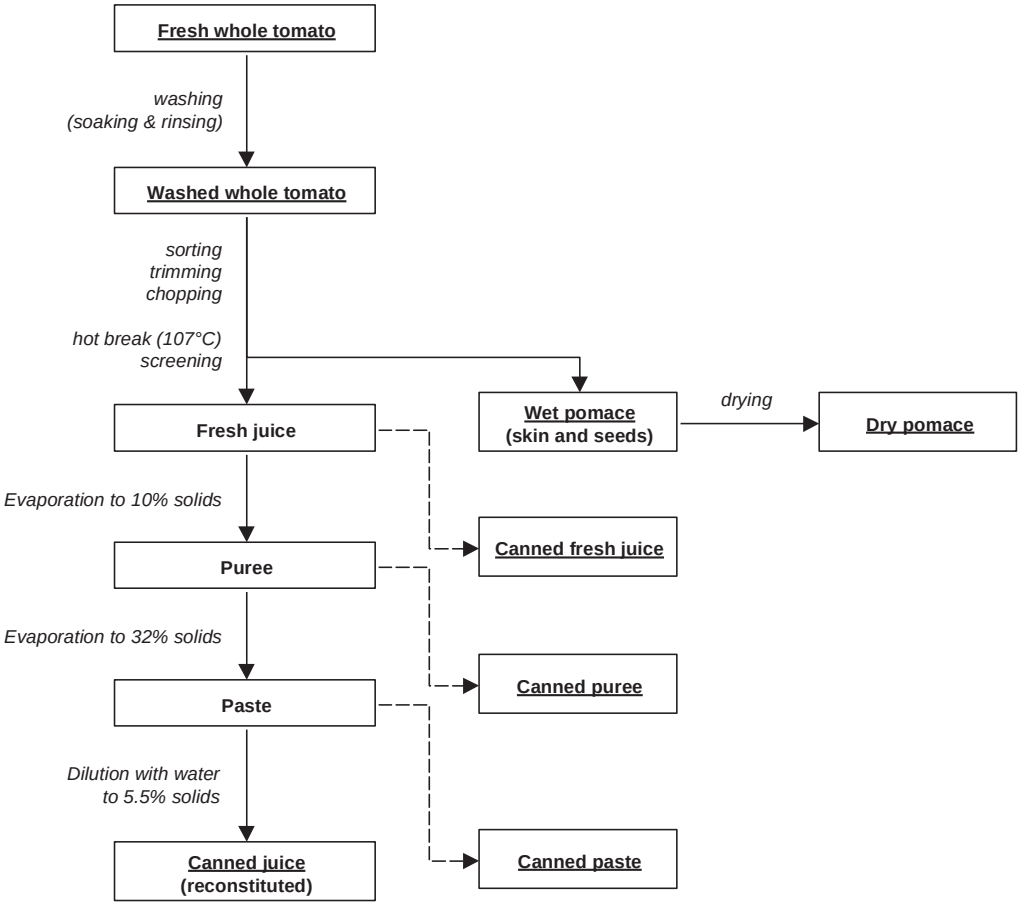


Figure 7 Tomato processing

Residues determined in fresh whole tomato and the tomato processed commodities are shown in Table 77. Ethephon did not concentrate in tomato processed commodities except in dry pomace which has a processing factor of 1.9. The data indicate that ethephon was lost during the preliminary processing, probably by heating.

Table 77 Residues of ethephon in processed tomato commodities (Nygren, 1991)

Trial	Commodities	Ethephon, mg/kg	Processing factor
89-138 CA, USA, 1989 (variety 1643)	Tomatoes (RAC)	0.73	1.0
	Washed fruit	0.68	0.93
	Wet pomace	0.38	0.52
	Dry pomace	1.39	1.9
	Canned fresh juice	0.25	0.34
	Canned puree	0.44	0.60
	Canned paste	0.55	0.75
	Canned juice from concentrate	0.29	0.40

In a published paper, processing of tomato into tomato paste was studied in Italy (Bolzuni & Leoni, Industria Conserve, *60*, 1985, pp 183, [M-188387-01-1). In this study, two lots of tomatoes (approximately 100 kg) containing incurred residues were processed using a procedure commonly used in industrial facilities. Following washing and chopping, the tomatoes were heated to 90 °C and passed through a sieve (opening Ø 0.6 mm) in order to remove seeds and skin. The juice obtained was concentrated into paste by heating at 55 °C under reduced pressure.

Finally, the paste was heated at 90 °C, canned and pasteurised. The samples of canned paste were stored for 9 months prior to analysis.

Analysis was by a method involving freeze drying, extraction with methanol, methylation using diazomethane and determination by means of GC/NPD. Mean procedural recoveries at fortification levels of 0.4 and 2.0 mg/kg in tomatoes were 86–95%, and at 0.2 and 1.0 mg/kg in tomato paste were 75–86%.

Residues determined in fresh whole tomatoes and tomato paste are shown in Table 78. The initial concentrations in the two lots of tomato were 0.27 and 0.36 mg/kg decreasing to 0.13 and 0.21 mg/kg, respectively, in paste with processing factor of 0.5 and 0.6 respectively.

Table 78 Residues of ethephon in tomato paste

Trial	Commodities	Ethephon, mg/kg	Processing factor
Trial 1 (variety UC 82)	Tomatoes (RAC)	0.27	
	Tomato paste	0.13	0.5
Trial 2 (variety UC 82)	Tomatoes (RAC)	0.36	
	Tomato paste	0.21	0.6

A study was conducted in 2004 on processing of tomato from three trials (Spain, Portugal and Italy) to juice, puree and preserve (Bardel, 2005, RA-3065/04, [M-262300-01-1]).

Preparation of juice: Tomatoes were washed in water, and samples of the washing water and washed tomato fruits collected. The washed tomatoes were cut into small pieces and heated with water (100 mL water/kg tomatoes) to 98–100 °C for 15–30 minutes. After this blanching process, the tomato pulp was passed through a strainer to separate raw juice and wet pomace. Sodium chloride was added to the raw juice, and sample of raw juice collected. One part of the raw juice was used for the processing into preserves. The rest of the raw juice was filled into preserving cans and pasteurised. After pasteurisation, a sample of juice was collected.

Preserves: Frozen tomatoes were washed in water and the peel removed. Samples of peel, peeling water and peeled fruits were collected. The peeled tomatoes were filled into preserving cans and raw juice added. The tomato preserves were pasteurised and a sample of tomato preserves collected.

Purée: Tomatoes were washed and then cut into small pieces. The cut tomatoes were heated with water (100 mL water/kg tomatoes) to 98–100 °C for 25–35 minutes. After this blanching process, the tomato pulp was passed through a strainer to separate raw juice and wet pomace. After the addition of sodium chloride, the raw juice was separated into raw purée and tomato liquid by centrifugation. The raw puree was filled into preserving cans and pasteurised. After pasteurisation, a sample of purée was collected.

Residues of ethephon were determined using method 00903, supplement E001. The LOQ was 0.05 mg/kg. The mean procedural recovery at fortification levels of 0.05–5.0 mg/kg in tomatoes was 103% (RSD 3.9%, n=10), at 0.05–0.5 mg/kg in juice was 103% (RSD 2.9%, n=8), at 0.05–0.5 mg/kg in puree was 98% (RSD 3.3%, n=8) and at 0.05–5.0 mg/kg in wet pomace was 90% (RSD 3.5%, n=7).

The samples were frozen after collection (–15 °C) and stored frozen until extraction and analysis. The maximum period of storage was 225 days (7.4 months).

Residues determined in fresh whole tomato and the tomato processed commodities are shown in Table 79. Ethephon did not concentrate in juice, preserves or puree. Residues were 0.30–0.57 mg/kg in fresh tomatoes, and decreased after processing to < 0.05–0.06 mg/kg in juice, < 0.05 mg/kg in puree and < 0.05–0.12 mg/kg in preserve.

Table 79 Residues of ethephon in processed tomato commodities

Trial	Commodities	Ethephon, mg/kg	Processing factor
R 2004 0468/9 Spain, 2004 (variety Malpica)	Tomatoes (RAC)	0.30	
	Fruit, peeled	0.09	
	Fruit, washed	0.13	0.4
	Washings	0.06	
	Juice	< 0.05	< 0.2
	Puree	< 0.05	< 0.2
	Raw juice	< 0.05	
	Preserve	< 0.05	< 0.2
	Wet pomace	< 0.05	< 0.2
	Raw puree	< 0.05	
	Peel, washed	0.11	
	Peeling water	0.08	
R 2004 0469/7 Portugal, 2004 (variety H-9661)	Tomatoes (RAC)	0.57	
	Fruit, peeled	0.09	
	Fruit, washed	0.11	0.2
	Washings	0.28	
	Juice	< 0.05	< 0.1
	Puree	< 0.05	< 0.1
	Raw juice	< 0.05	
	Preserve	< 0.05	< 0.1
	Wet pomace	< 0.05	< 0.1
	Raw puree	< 0.05	
	Peel, washed	0.23	
	Peeling water	0.24	
R 2004 0470/0 Italy, 2004 (variety Missouri)	Tomatoes (RAC)	0.55	
	Fruit, peeled	0.50	
	Fruit, washed	0.51	0.9
	Washings	< 0.05	
	Juice	0.06	0.1
	Puree	< 0.05	< 0.1
	Raw juice	0.48	
	Preserve	0.12	0.2
	Wet pomace	< 0.05	< 0.1
	Raw puree	< 0.05	
	Peel, washed	0.10	
	Peeling water	0.07	

Barley

A study was conducted on processing of barley grains obtained at a DALT of 49 days from a trial in Canada during 1981 to hulls and pearl barley (Harrison, 1981, 10223, [M-187972-01-1]).

Barley grain was milled into pearls as a batch operation. After pearling, the hulls and pearls were separated by sifting. Pearls were ground on a small plate grinder prior to analysis.

Residues of ethephon were determined using a method similar to SOP 90074. The LOQ was 0.05 mg/kg. The average recovery rate in wheat and barley grain at 0.2 mg/kg was 102% (RSD 17%, n=14). The recovery at 0.2 mg/kg in barley hulls was 114%, and in pearls was 99%.

The samples were frozen after collection at approximately –20 °C and stored frozen for 4 months.

Residues determined in barley processed commodities are shown in Table 80. In barley pearls, the ethephon concentration was slightly lower than in the corresponding raw agricultural commodity, while ethephon concentration in hulls was higher with a processing factor of 1.6.

Table 80 Residues of ethephon in barley grain and processed commodities

Trial	Commodities	Ethephon, mg/kg	Processing factor
Canada (variety Bruce)	Barley grain (RAC)	0.62	
	Barley pearls	0.54	0.9
	Barley hulls	1.01	1.6

Wheat

A study was conducted on processing of wheat grains harvested at a DALT of 65 days from one field trial in the USA (Texas) during 1989–1990 to dust, middlings, bran, flour, red dog and germ and shorts (Conn, 1992, SARS-90-24P, [M-187550-01-1]).

Wheat grain samples were processed to simulate industrial practice as closely as possible. The total quantity processed was approximately 81 kg. A simplified flow chart of the processing is shown in the following Figure.

Residues of ethephon were determined using a method similar to SOP 90074. The method involved Soxhlet extraction with methanol, precipitation of interfering materials, methylation using diazomethane and determination by means of GC/FPD. The LOQ was 0.1 mg/kg in red dog and 0.05 mg/kg for wheat grain and all other processed fractions. The average recovery rate at fortification levels of 0.05–1.0 mg/kg was 80% in wheat grain, 97% in dust, 73% in bran, 104% in low grade flour, 66% in patent flour, 60% in shorts and germ, and 74% in red dog. The overall average recovery was 79% (n=15).

Samples were in frozen storage for less than 1 month between harvest and processing and for no longer than 5 months between processing and analysis.

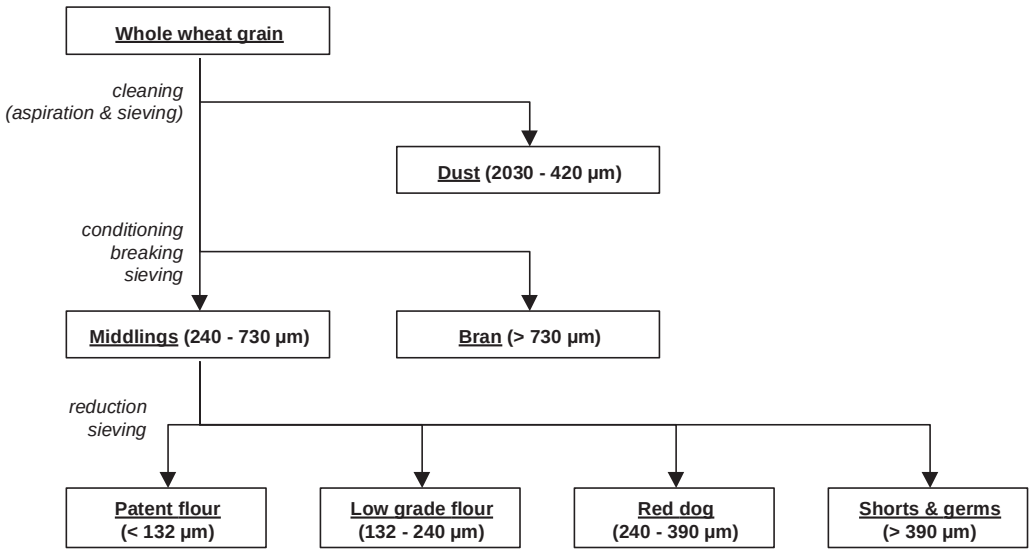


Figure 8 Wheat grain processing

Residues determined in wheat grain and the processed commodities are shown in Table 81. In middlings, low grade flour and patent flour, the residue levels were less than the LOQ (0.05 mg/kg). Measurable residues were found in unprocessed grain, grain dust, bran, shorts and germ and in red dog. Limited concentrations of ethephon residues occurred in bran, shorts and germs, and in red dog, with processing factors of less than 1.5.

Table 81 Residues of ethephon in wheat grain and processed products (Conn, 1992)

Trial	Commodities	Ethephon, mg/kg	Processing factor
USA (Texas) (variety Mitt)	Wheat grain (RAC)	0.17	
	Grain dust	0.10	0.6
	Bran	0.23	1.4
	Middlings	< 0.05	< 0.3
	Low grade flour	< 0.05	< 0.3
	Patent flour	< 0.05	< 0.3
	Shorts and germ	0.25	1.5
	Red dog	0.20	1.2

A second study was conducted on processing of wheat from a trial in Canada during 1981 to bran, flour, germ and shorts (Harrison, 1981, 10223, [M-187972-01-1]). Wheat grain was harvested at a PHI of 53 days.

The wheat grain sample was first brought to 15% moisture content and then milled using an automatic laboratory mill, which separated the ground grain into bran, flour, and a mixture of shorts and germ. The shorts and germ fraction was then manually separated into shorts and germ.

Residues of ethephon were determined using the same method as in the study above. The LOQ was 0.05 mg/kg. The average recovery rate in wheat and barley grain at 0.2 mg/kg was 102% (RSD 17%, n=14). The recovery at 0.2 mg/kg was 98% in shorts, 112% in germ, 84% in flour and 96% in bran.

The samples were frozen after collection at approximately –20 °C and stored frozen for less than 2 months.

Residues determined in wheat processed commodities are shown in Table 82. In wheat flour the residue level of ethephon was lower than in the corresponding raw agricultural commodity. Concentration of ethephon residues occurred in bran, shorts and germ with processing factors in the range of 2.0 to 3.5.

Table 82 Residues of ethephon in wheat grain and processed products (Harrison, 1981)

Trial	Commodities	Ethephon, mg/kg	Processing factor
Canada (variety Frederick)	Wheat grain (RAC)	0.35	
	Wheat bran	1.21	3.5
	Wheat flour	0.02	0.1
	Wheat shorts	0.78	2.2
	Wheat germ	0.71	2.0

A third study was conducted on processing of wheat from a trial in Canada during 1984 into bran and flour (Nygren, 1985, 866R11, [M-187977-01-1]). Three separate grain samples were processed into bran and flour. The shorts and germ fractions were combined during processing to a whole wheat flour fraction.

Residues of ethephon were determined using the same method as above. The method was validated by determination of the recovery rates for control samples fortified at 0.20 mg/kg. The recovery rates were good: 74% in grain, 79% in bran, 85% in flour and 84% in shorts and germ.

The samples were stored frozen (< –34 °C) for less than 12 months.

Residues determined in wheat processed commodities are shown in Table 83. Ethephon concentrations were lower in wheat flour in unprocessed grain. Ethephon residues occurred in bran as well as in shorts and germs (combined to whole wheat flour) with average processing factors of 3.1 and 2.7, respectively. These results compare well with those obtained in the previous study. A material balance is provided in the report which shows that the residues

measured in the milled fractions accounted for 78 to 86% of the residues determined in the corresponding unprocessed wheat grain samples.

Table 83 Residues of ethephon in wheat grain and processed products (Nygren, 1985)

Trial	Commodities	Ethephon, mg/kg	Mean processing factor
Canada (variety Augusta)	Wheat grain (RAC)	0.07, 0.10, 0.07 (mean 0.08)	
	Wheat bran	0.20, 0.22, 0.30 (mean 0.24)	3.1
	Wheat flour	0.02, 0.03, 0.02 (mean 0.02)	0.2
	Whole wheat flour (germ + shorts)	0.25, 0.18, 0.19 (mean 0.21)	2.7

Cotton seed

A study was conducted on processing of cotton seed from a trial in the USA (Louisiana) in 1993–1994 to oil and pomace (Lee, 1994, USA93I04R, [M-203874-01-2]). Three replicate samples (approximately 72 kg each) were harvested by hand 7 days after treatment.

The cotton was processed in such a way as to simulate industrial practice as closely as possible. However, due to the sample size, a batch process was adopted (as opposed to a continuous operation). A simplified flow chart of the processing is shown in the following Figure with analysed fractions underlined. The kernel (with some hull material) was heated (66–76 °C), flaked (82–114 °C) and then exposed to hexane (49–60 °C) to remove the crude oil from the flakes. After the crude oil and the hexane mixture was adjusted to the proper ratio, the crude oil was refined by heating up to 49 °C with sodium hydroxide. Thereafter the soapstock was separated by centrifugation and heated to about 70 °C in order to remove solvent. The refined oil was obtained from the liquid phase by evaporating the hexane. As the refined oil had a dark colour, it was refined again.

Residues of ethephon were analysed by ethylene release with method EC-92-228. The LOQ was 0.07 mg/kg for seed and processed commodities. Procedural recoveries at fortification levels of 0.07–6.0 mg/kg were 95% (RSD 3.2%, n=7) for seed, 108% (RSD 14.6%, n=5) for hulls, 101% (RSD 10.3%, n=5) for meal, 99% (RD 6.7%, n=7) for crude oil, 93% (RSD 14.1%, n=8) for soapstock and refined soapstock) and 100% (RSD 6.0%, n=8) for refined and re-refined oil.

The samples were frozen after collection at < -10 °C and stored frozen until extraction and analysis. The maximum period of storage was 12 months.

Residues determined in the ginned cottonseed and the cottonseed processed fractions are shown in Table 84. The mean ethephon residue level in ginned cottonseed amounted to 4.96 mg/kg. There was no concentration in any of the analysed processed fractions. Mean ethephon concentrations amounted to 0.35 mg/kg in hulls, 0.12 mg/kg in meal and 0.13 mg/kg in soapstock. Ethephon concentrations in crude and refined oil were less than the limit of quantification of 0.07 mg/kg. The data suggest that a significant part of the ethephon residue decomposes during processing.

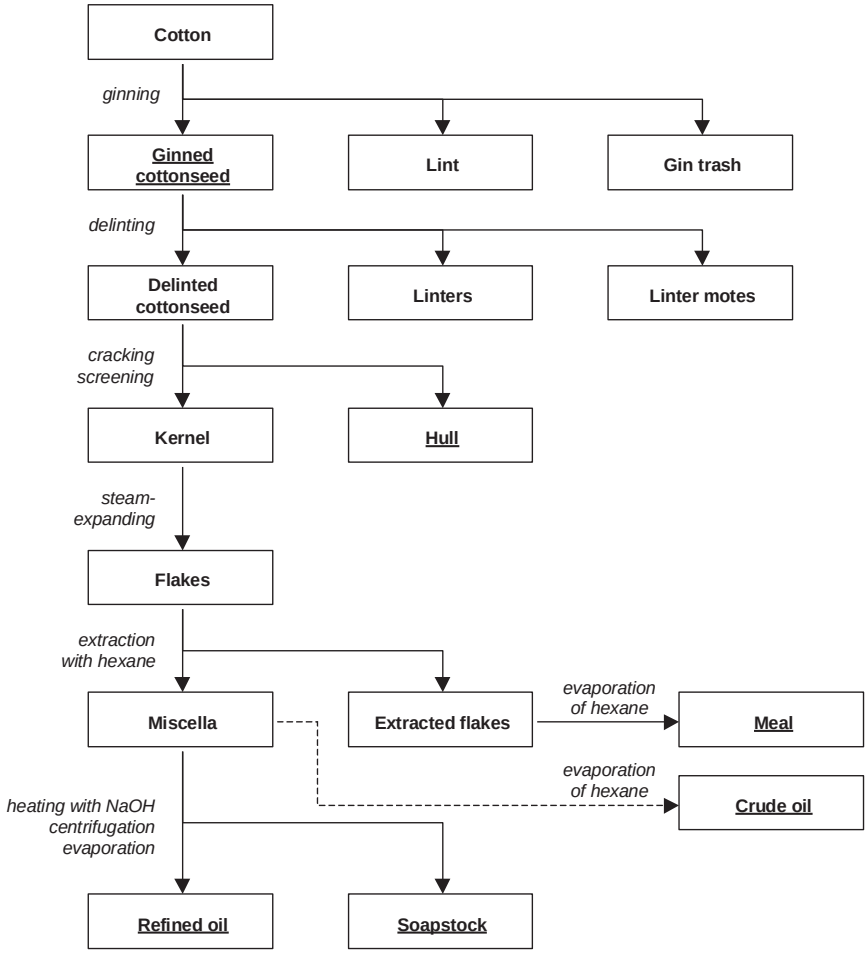


Figure 9 Cotton seed processing

Table 84 Residues of ethephon in cotton seed and processed products (Lee, 1994)

Trial	Commodities	Ethephon, mg/kg	Processing factor
USA (Louisiana) (variety DPL 41)	Cottonseed	5.84, 3.75, 5.30 (mean 4.96)	
	Hulls	0.22, 0.29, 0.55 (mean 0.35)	0.07
	Meal	0.15, 0.07, 0.14 (mean 0.12)	0.02
	Crude oil	< 0.07, < 0.07, < 0.07 (mean < 0.07)	< 0.02
	Soapstock	0.13, 0.15, 0.11 (mean 0.13)	0.03
	Refined oil	< 0.07, < 0.07, < 0.07 (mean < 0.07)	< 0.02
	Refined soapstock	< 0.07, < 0.07, < 0.07 (mean < 0.07)	< 0.02
	Re-refined oil	< 0.07, < 0.07, < 0.07 (mean < 0.07)	< 0.02

Another study was conducted on processing of cotton seed from two field trials in Greece and Spain in 2008 to oil (Billian & Krusell, 2010, 08-3401, [M-367885-01-1]).

The processing included the following steps: conditioning, extraction and refining. Initially pressing was planned, but it was not carried out because the oil content of the cotton seeds was < 25%. Instead, the oil was separated by solvent extraction.

The seeds were defrosted and crushed using a roller mill. After conditioning (adjusting the content of moisture for < 5%), the crushed cotton seeds extracted with n-hexane (2 hours at 60 °C) in a small technical extraction plant. The solvent-oil-mixture (miscella) was pumped into a distillation vessel and the hexane removed by distillation. The hexane was recycled back to the seed for a second hexane extraction step. After distillation, the rest of the solvent was removed from the extracted by rotary evaporation at 50 °C to give solvent extracted oil. The solvent-extracted crushed seed was sampled as meal after storing at room temperature for approximately one day.

The solvent extracted oil was filtered to give pre-clarified crude oil. Refining consisted of hydration, desliming (degumming), neutralization, washing, drying, bleaching, filtration and deodorization

Residues of ethephon were determined using method 00918. The LOQ was 0.05 mg/kg. Mean procedural recoveries at fortification levels of 0.05–15.0 mg/kg were 98% (RSD 6.5%, n=4) in bolls, 88% (RSD 6.4%, n=3) in meal, 91% (RSD 5.5%, n=4) in seed and 94% (RSD 3.2%, n=6) in oil.

The samples were frozen after collection at < –10 °C and stored frozen until extraction and analysis. The maximum period of storage for bolls was 452 days (14.9 months), for seed was 439 days (14.4 months) and for oil and meal was 437 days (14.4 months).

Residues determined in the cottonseed and processed fractions are shown in Table 85. Ethephon concentrations in bolls were 12.7–13.4 mg/kg. Residues in seed were 1.48–2.0 mg/kg. There was no concentration in any of the analysed processed fractions and little transfer of residues into the oil. Residues in all oil fractions were < 0.05 mg/kg and in meal were 0.05–0.14 mg/kg. The mean processing factors are < 0.03 for oil and 0.05 for meal.

Table 85 Residues of ethephon in cotton seed, oil and meal (Billian & Krusell, 2010)

Trial	Commodities	Ethephon, mg/kg	Processing factor
Trial 08-3401-01 Greece (variety Carmen)	Bolls (0 day PHI)	12.7	
	Cotton seed (7 day PHI)	2.0	
	Meal	0.14	0.07
	Solvent extracted oil	< 0.05	< 0.03
	Preclarified crude oil	< 0.05	< 0.03
	Neutralised crude oil	< 0.05	< 0.03
	Refined oil	< 0.05	< 0.03
Trial 08-3401-02 Spain (variety Alexandro)	Bolls (0 day PHI)	13.4	
	Cotton seed (7 day PHI)	1.48	
	Meal	0.05	0.03
	Solvent extracted oil	< 0.05	< 0.03
	Preclarified crude oil	< 0.05	< 0.03
	Neutralised crude oil	< 0.05	< 0.03
	Refined oil	< 0.05	< 0.03

Summary of processing factors

Based on the available processing studies, the processing factors that have been calculated are summarized in Table 86.

Table 86 Summary of processing factors

Commodity	Processed commodities	Processing factor	
		Individual value	Best estimate
Apple	Wet pomace	0.3, 0.4, 0.6, < 0.8, 1.1	0.60

Commodity	Processed commodities	Processing factor	
		Individual value	Best estimate
	Dry pomace	2.0	2.0
	Apple juice	< 0.4, 0.4, 0.5, < 0.8, 1.5	0. 5
	Apple sauce	0.4, 0.5, < 0.8, 1.1	0.5
Grape	Dried grapes	0.79, 0.89, 1.0, 1.4, 3.2, 8.5	1.2
	Grape juice	0.5, 0.7, 0.8, 1.1	0.75
	Wet pomace	0.4, 0.6, 0.9, 1.1	0.75
	Must	0.7, 0.8, 0.8, 0.9, 1.0, 1.0	0.85
	Wine	0.7, 1.0, 1.2, 1.4, 1.5, 2.1,	1.3
Olives	Olive oil (virgin and refined)	< 0.02, < 0.03	< 0.02
	Table olives	< 0.01, < 0.02, < 0.02, < 0.03	< 0.01
Tomato	Wet pomace	< 0.1, < 0.1, < 0.2, 0.52	0.52
	Dry pomace	1.9	1.9
	Tomato juice	< 0.1, 0.1, < 0.2, 0.34	0.22
	Tomato puree	< 0.1, < 0.1, < 0.2, 0.60	0.60
	Tomato paste	0.5, 0.6, 0.75	0.6
	Tomato preserves	< 0.1, < 0.2, 0.2	0.2
Barley	Pearl barley	0.9	0.9
	Barley hulls	1.6	1.6
Wheat	Flour	0.1, 0.2, < 0.3,	0.15
	Wheat germ	2.0	2.0
	Wholemeal flour (germ + shorts)	2.7	2.7
	Wheat bran	1.4, , 3.1, 3.5	3.1
Cotton	Cottonseed refined oil	< 0.02,< 0.03, < 0.03	< 0.02
	Meal	0.02, 0.03, 0.07	0.03

RESIDUES ON ANIMAL PRODUCTS

Livestock feeding studies

Dairy cattle feeding study

As the goat metabolism studies conducted at exaggerated dose rate suggested that residues of ethephon may transfer to edible tissues and mil, a cattle feeding study was conducted (Wells-Knecht, 1996, 96E08334, [M-188195-01-1]). Three groups of three Holstein dairy cows were orally dosed once daily with ethephon in gelatine capsules for 28 consecutive days. One additional cow was maintained as control and received no test compound. One group received an amount of ethephon equivalent to nominally 43 ppm diet (1×, actual mean level = 44 ppm), another was fed 129 ppm diet (3×, actual mean level = 128 ppm), and the last group received 430 ppm diet (10×, actual mean level = 415 ppm).

Milk samples were collected twice daily and, the p.m. milk and the a.m. milk of the following day were combined. Milk samples for each animal were retained for analysis on study days 0, 1, 4, 8, 11, 15, 18, 22, 25 and 27. All cows were sacrificed after 28 days of dosing, within 6 hours after receiving the final dose. Tissues collected were: kidney, liver, fat (composite of omental and peri-renal fat), and muscle (composite of thigh and loin muscle). All samples were frozen at –20 ºC until analysis.

Ethephon was measured in the homogenised tissue samples using analytical method 11-94 (Nygren, 1994, 11-94). The LOQ was 0.01 mg/kg for tissues and 0.002 mg/kg for milk. The concurrent mean recovery in milk was 99±6% (n=17) at fortification levels of 0.002–0.10 mg/kg. The mean recovery in liver was 105±7% (n=3) at fortification levels of 0.01–2.0 mg/kg, in kidney was 94±14% (n=5) at fortification levels of 0.01–12 mg/kg, in fat was 70% (n=2) at fortification levels of 0.01 and 4 mg/kg and in muscle was 98% (n=2) at fortification levels of 0.01 and 0.4 mg/kg.

All milk and tissue samples were analysed within 30 days of sampling, except for the reanalysis of the Day 8 milk samples, which were analysed after 34 days of storage. The results of the reanalysis corresponded with the results of the initial analysis conducted within 30 days of collection. The storage stability study showed that ethephon residues are stable in milk for at least 4 months, and in meat for at least 12 months when stored frozen.

A summary of the residues found in milk is given in in the table below. All milk samples from the control cow did not contain ethephon (ND). Following oral administration to lactating cows for 28 consecutive days, the residues of ethephon in whole milk appeared to plateau after Day 4. At the dose level of 43 ppm diet, residues of ethephon in milk were less than 0.01 mg/kg. Maximum residue concentrations in milk were 0.007 mg/kg at the low dose level, 0.019 mg/kg at the mid dose level and 0.033 mg/kg at the high dose level.

Table 87 Mean residues of ethephon in whole milk during 28 days oral administration to dairy cows

Day sampled [a]	Ethephon in individual cow, mg/kg (Mean ethepon, mg/kg)		
	43 ppm diet	129 ppm diet	430 ppm diet
0	ND, ND, ND	ND, ND, ND	ND, ND, ND
1	0.0068, 0.0074, 0.0074 (mean 0.072)	0.0178, 0.0116, 0.0142 (0.0145)	0.0331, -, 0.0275 (0.0303)
4	0.065, 0.0054, 0.0066 (0.0062)	0.0147, 0.0122, 0.0186 (0.0152)	0.0263, 0.0307, 0.0274 (0.0281)
11	0.0034, 0.0020, 0.0041 (0.0032)	0.0149, 0.0116, 0.0122 (0.0129)	0.0308, 0.0244, 0.0243(0.0265)
15	0.025, 0.025, 0.041 (0.0030)	0.0119, 0.0094, 0.0113 (0.0109)	0.0269, 0.0283, 0.0261 (0.0271)
18	< 0.002, < 0.002, 0.0023 (< 0.002)	0.0110, 0.0077, 0.0112 (0.0100)	0.0322, 0.0179, 0.0249 (0.0250)
22	0.0020, < 0.002, 0.0025 (< 0.002)	0.0108, 0.0067, 0.102 (0.0092)	0.0276, 0.0180, 0.0271 (0.0242)
25	0.0022, < 0.002, 0.0023 (0.002)	0.0149, 0.0050, 0.0084 (0.0094)	0.0267, 0.0197, 0.0251 (0.0238)
27	< 0.002, < 0.002, 0.0023(< 0.002)	0.0069, 0.0095, 0.0138 (0.0101)	0.0251, 0.0257, 0.0323 (0.0277)

[a] Day 8 milk not included in table because of suspect untreated control

A summary of the residues of ethephon in tissue samples from cows fed 43 ppm, 129 ppm, and 430 ppm in the diet of ethephon for 28 days are summarized in the table below. The results show that residues are very low in tissues except in kidney. The residue levels in kidney are up to 7 times higher than the residue level in liver. Residue levels of ethephon in tissue and milk samples are proportional to dose level.

Table 88 Residues of ethephon in tissues from dairy cattle following dosing with ethephon for 28 days

Tissue	Ethephon in individual cow, mg/kg (Mean ethephon, mg/kg)			
	0 ppm diet	43 ppm diet	129 ppm diet	430 ppm diet
Fat	< 0.01	< 0.01, < 0.01, < 0.01 (< 0.01)	0.016, 0.069, 0.037 (0.04)	0.038, 0.029, 0.13 (0.06)
Kidney	0.03	0.64, 0.24, 0.58 (0.49)	2.8, 3.2, 3.5 (3.2)	8.0, 4.6, 10.9 (7.8)
Liver	0.05	0.095, 0.066, 0.085 (0.08)	0.39, 0.65, 0.50 (0.51)	0.85, 0.63, 1.5 (0.99)
Muscle	< 0.01	0.014, < 0.01, 0.016 (0.01)	0.043, 0.061, 0.049 (0.05)	0.11, 0.074, 0.17 (0.12)

Poultry feeding study

A poultry feeding study was conducted (Wells-Knecht, 1996, 96E08335, [M-188192-01-1]). Three groups of ten Leg Horn laying hens were orally dosed once daily with ethephon in gelatine capsules for 28 consecutive days. Each group was sub-divided into three subgroups of three or four hens. One additional group of ten hens was maintained as a control and received no test compound. One group received ethephon at a dose level equivalent to nominally 2.3 ppm diet (1×), another was fed 6.9 ppm diet (3×), and the last group received 23 ppm diet (10×).

Eggs were collected twice daily. Egg samples from study days 0, 1, 4, 8, 11, 15, 18, 22, 25 and 27 were pooled by sub-group. All hens were sacrificed after 28 days of dosing, within 4 hours after receiving the final dose. Tissue samples collected were liver, skin with adhering fat, and muscle (breast and leg). All samples were frozen at –20 ºC until analysis.

Ethephon was measured in the homogenised egg and tissue samples using analytical method 11-94. The LOQ was 0.01 mg/kg for tissues and 0.002 mg/kg for eggs. The concurrent mean recovery in egg was 99 ± 4% (n=17) at fortification levels of 0.002–0.10 mg/kg. The mean recovery in liver was 104 ± 10% (n=4) at fortification levels of 0.01–2.0 mg/kg, in skin with fat was 89 ± 3% (n=5) at fortification levels of 0.004–0.20 mg/kg and in muscle was 98 ± 9% (n=5) at fortification levels of 0.04–0.10 mg/kg.

All egg and tissue samples were analysed within 30 days of sampling.

A summary of the residues found in eggs is given in in the table below. Following oral administration to laying hens for 28 consecutive days, the residues of ethephon in whole eggs from the highest dose group were slightly above or below the LOQ of 0.002 mg/kg with the highest concentration of 0.0036 mg/kg in eggs from sub-group C of the high dose group on Day 8 (mean residue on Day 8 was 0.0029 mg/kg). Eggs from the low and mid dose groups were not analysed.

Table 89 Residues of ethephon in whole egg during 28 days oral administration to laying hens

Day sampled	Ethephon in subgroup, mg/kg (Mean ethephon, mg/kg)			
	0 ppm diet	2.3 ppm diet	6.9 ppm diet	23 ppm diet
0	< 0.002	< 0.002, < 0.002, < 0.002 (< 0.002)	0.002, < 0.002, < 0.002 (< 0.002)	0.002, < 0.002, < 0.002 (< 0.002)
1	< 0.002	Not analysed	Not analysed	0.002, < 0.002, < 0.002 (< 0.002)
4	0.002	Not analysed	Not analysed	0.0023, 0.0027, 0.0028 (0.0026)
8	< 0.002	Not analysed	Not analysed	0.0025, 0.0027, 0.0036 (0.0029)
11	< 0.002	Not analysed	Not analysed	0.002, < 0.002, < 0.002 (< 0.002)
15	< 0.002	Not analysed	Not analysed	0.002, < 0.002, < 0.002 (< 0.002)
18	< 0.002	Not analysed	Not analysed	0.002, < 0.002, < 0.002 (< 0.002)
22	< 0.002	Not analysed	Not analysed	0.0023, 0.0028, < 0.002 (0.0024)
25	< 0.002	Not analysed	Not analysed	0.0023, 0.0023, 0.0024 (0.0023)
27	< 0.002	Not analysed	Not analysed	< 0.002, 0.0024, 0.0024 (0.0023)

A summary of the residues of ethephon in tissue samples from hens fed 2.3 ppm, 6.9 ppm, and 23 ppm in the diet of ethephon for 28 days are summarized in the table below. The results show that residues are very low in tissues from the low dose group. The highest residue level was found in liver at 0.033 mg/kg in the low dose level. Residue levels of ethephon in egg and tissue samples increased proportionally with dose level. At the highest dose level, the maximum residue in liver was 0.29 mg/kg.

Table 90 Residues of ethephon in tissues from laying hens following dosing with ethephon for 28 days

Tissue	Ethephon in subgroup, mg/kg (Mean ethephone, mg/kg)			
	0 ppm diet	2.3 ppm diet	6.9 ppm diet	23 ppm diet
Liver	0.01	0.0028, 0.0033 (0.031)	0.059, 0.058, 0.068 (0.062)	0.29, 0.19, 0.20 (0.23)
Skin + fat	< 0.01	0.011, 0.014 (0.013)	0.024, 0.017, 0.032 (0.024)	0.117, 0.075, 0.087 (0.093)
Muscle	< 0.01	< 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01, 0.015 (0.012)	0.060, 0.023, 0.027 (0.037)

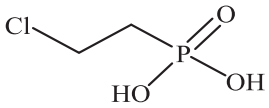
APPRAISAL

Ethephon, 2-chloroethylphosphonic acid, is a systemic plant growth regulator belonging to the phosphonate family. It is readily absorbed by the plant and releases ethylene, a natural plant hormone. Ethylene not only influences directly several physiological processes such as ripening and maturation, but also stimulates the endogenous ethylene production. It has been registered in many countries for a variety of crops, including fruits, vegetables, cereals and oilseed crops.

Ethephon was first evaluated by JMPR in 1977 as a new compound, and then reviewed several times for residues. It was evaluated under the periodic review programme in 1994. The compound was listed in the Priority List by the Forty-sixth Session of CCPR in 2014 for toxicological and residue evaluation by the current Meeting in the CCPR periodic review programme.

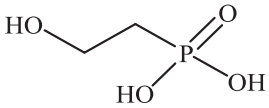
The Meeting received information on identity, metabolism and environmental fate, residue analysis, use pattern, supervised trials (on apples, cherries, grapes, figs, olives, pineapples, tomatoes, cereals, and cotton), processing, and animal feeding studies.

In this Appraisal, the following names were used for referred compounds.



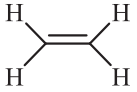
Ethephon

2-Chloroethylphosphonic acid



HEPA

(2-hydroxyethyl)-phosphonic acid



Ethylene

Plant metabolism

The Meeting received information on plant metabolism studies conducted on a variety of plants including information from the published scientific literature. The information dated from 1962 to 2003 and covered peaches, grapes, pineapples, cucumbers, squash, melons, tomatoes, wheat, hazelnuts, walnuts and cotton.

Many studies conducted on various plants indicate the release of ethylene after treatment with ethephon. In several of such studies, methanol, acidified methanol or water was used to extract ethephon from fruits and/or leaves and, where data are available, significant amount of the applied radioactivity (> 60%) or TRR (> 80%) was recovered in the surface wash and solvent extract combined.

The studies involving characterization and identification of other metabolites are described below.

Tomato plants grown outdoor were treated with a foliar spray of uniformly labelled [^{14}C]ethephon at a rate approximating 1.46 kg ai/ha at the "green mature" or "colour break" growth stage and fruits were harvested 0, 5 and 12 days after the treatment (DAT). The majority of the radioactivity was recovered from the methanol surface wash on 0 DAT but 96% (including surface wash) and 98% of the TRR was recovered in methanol extracts of 5 DAT and 12 DAT samples respectively.

The predominant radioactive residue in methanol extract of tomato fruit was ethephon, 70% and 59% of the TRR corresponding to 1.2 mg/kg and 0.68 mg/kg in 5 DAT and 12 DAT was found in fruits, respectively. The concentration of ethephon decreased over the time period in the study from 7.5 mg/kg at 0 DAT to 0.68 mg/kg at 12 DAT. The only significant metabolite found was HEPA accounting for 15% TRR (0.26 mg/kg) on 5 DAT and 13% TRR (0.15 mg/kg) on 12 DAT. No other metabolites exceeded 5% TRR in the methanol extract.

Wheat plants grown outdoor were treated with a foliar spray of [^{14}C]ethephon at a rate of 0.36 kg ai/ha and 3.6 kg ai/ha at the forage stage (BBCH 39) and forage samples were collected on 0 DAT, hay on 14 DAT and grain and straw on 34 DAT. The majority of radioactivity was recovered in methanol extracts of plant parts (hay and straw) on 14 and 34 DAT regardless of the dose used (94% TRR including 1% in surface wash in hay of both doses and 58% and 74% TRR in straw respectively) while radioactivity was similarly distributed in the methanol surface wash and methanol extract (45–46% and 54–55% TRR) of forage on 0 DAT. Unextracted residues were about 5% in 14 DAT for hay and 10% (1×) and 26% (10×) in 34 DAT for straw.

Methanol extraction recovered only 28 and 22% TRR from grain samples after the low and high doses. Acid hydrolysis of the remaining solid released a further 56 and 71% TRR; extraction of the post-hydrolysis solids released a total of 9.9% and 4.3% TRR, respectively. This indicates the presence of significant conjugates in grains. Unextracted residues were 1.8–6.0% TRR.

Most of the TRR was attributed to the sum of ethephon and HEPA. The major radioactive residue in 14 DAT hay was HEPA (72% TRR and 3.7 mg/kg) followed by ethephon (20% TRR and 1.0 mg/kg). In the 34 DAT straw, the major radioactive residue was ethephon (62% TRR and 1.5 mg/kg).

In 34 DAT grain, HEPA was found at a similar level as ethephon after the low dose (HEPA 48% TRR and 0.51 mg/kg and ethephon, 44% and 0.47 mg/kg). After the higher dose, approximately two times larger amount of HEPA was found than ethephon (HEPA, total of 60% TRR and 2.0 mg/kg; and ethephon, total of 32% TRR and 1.1 mg/kg). No other metabolites exceeded 3% of TRR.

Cotton plants grown outdoor were treated with a foliar spray at a rate of 1.4 kg ai/ha seven days before harvest. Plants were harvested at 7 DAT. The majority of radioactivity was recovered in methanol/water (9:1) for gin trash (89% TRR) and in methanol extract for seeds (82% TRR).

The predominant radioactive residue in gin trash was ethephon at 93% TRR and 30 mg/kg; and 78% TRR and 0.64 mg/kg in seeds. HEPA was low, 1.7% TRR and 0.52 mg/kg in gin trash and 9.6% TRR and 0.08 mg/kg in seeds. No other metabolites exceeded 2% of TRR.

In summary, plant metabolism studies conducted on tomatoes, wheat and cotton indicate that the metabolism of ethephon in these plants was qualitatively similar and indicate that radioactivity penetrated into plants after a foliar application and translocated to edible matrices of plants.

After foliar application to plants, ethephon was metabolized to ethylene and phosphates and HEPA which would be either metabolized to carbon dioxide and phosphate or incorporated into biomolecules such as proteins, carbohydrates and lipids after further metabolism.

In tomatoes, cotton, and wheat hay, most radioactivity was recovered from methanol extracts whilst in wheat grains and straw a significant amount of radioactivity was recovered in the acid hydrolysate, suggesting ethephon is present in conjugated forms.

In tomato and cotton, ethephon was the predominant residue with little HEPA present. However, in wheat grains, HEPA and its conjugates were present at a similar concentration as that of ethephon and its conjugates after the 1× dose and approximately two times higher concentration than ethephon after the 10× exaggerated rate in grain. In wheat hay, HEPA was present at 3.5 times higher than ethephon.

Ethephon would be an appropriate marker for plants except cereal grains and straw in which ethephon was significantly metabolised to HEPA and to conjugates of ethephon and HEPA.

Animal metabolism

The Meeting received information on the fate of orally-dosed [^{14}C]ethephon in lactating goats and laying hens.

Metabolism studies on laboratory animals including rats were reviewed in the framework of toxicological evaluation by the current JMPR.

After oral administration of ethephon to rats, absorption was rapid with a Tmax of 1.0–1.3 hours and 1.9–2.5 hours after a single oral dose of 50 or 1000 mg/kg bw, respectively. Six days after a single dose tissue, and carcass contained only 0.08% or less of administered radioactivity. Highest concentrations were found in liver and kidney. Radioactivity was excreted in urine (47–60%), expired air (18–21%, mainly ethylene) and faeces (4–6.5%), indicating that at least 65% of the administered dose was absorbed. Ethephon was mainly metabolized to ethylene and to a small extent to HEPA.

Two lactating goats were orally administered [^{14}C]ethephon twice daily after am and pm milking in capsules for seven consecutive days at 0.37 and 0.46 mg/kg bw/day (approximately 10 ppm in the diet). The goats were sacrificed approximately 16 hours after the last dose.

A significant portion of the administered dose was released as ethylene (29%) and carbon dioxide (2.0%). Radioactivity was also excreted in urine (19%) and faeces (6.7%). In total, milk contained 3.3% of the administered dose, tissues 3.0%, and content of gastro ontestinatl (GI) tract, 0.84%. Amongst tissues, kidney contained the highest radioactivity at 1.2 mg eq/kg followed by liver at 1.0 mg eq/kg. Fat contained 0.50 mg eq/kg, heart 0.16 mg eq/kg and muscle 0.10 mg eq/kg. Over the study period, average TRR in milk increased from 0.081 mg/kg on day 0.5 to a plateau level of 0.42 mg/kg at day 3.5. The fat fraction of milk contained 45% of the TRR in milk; skimmed milk contained 0.15–0.20 mg eq/kg; and milk fat, 3.0–4.2 mg eq/kg.

In order to estimate ethephon, portions of tissues were hydrolyzed by shaking at 40 °C at pH 11 for one hour to transform ethephon to ethylene. Ethylene released by this hydrolysis was 0.4% TRR in kidney corresponding to 0.008 mg/kg ethephon, 0.05% TRR in fat, and 0% TRR in muscle, liver and milk. Radioactivity in the remaining solids were 0.3%, 2.1%, 71% and 35% of the respective TRR in kidney, liver, muscle and fat.

Extraction of a portion of liver with ether released 5.3% TRR, methanol, a further 64% TRR leaving 27% TRR unextracted. Precipitation with trichloroacetic acid resulted in 12% TRR in liver which is associated with proteins. Glycogen was isolated at a concentration of 0.9 mg/kg.

Two studies were provided on metabolism of ethephon in laying hens. In both studies, hens were orally administered either by capsule or gavage [^{14}C]ethephon at a rate equivalent to 53–67 ppm in the diet for five consecutive days. Hens in the first study were sacrificed 22–23 hours after the last dose and those in the second 9–10 hours after the last treatment.

In the first study, the majority of the administered dose (58%) was recovered as expired ethylene while expired carbon dioxide was negligible. In the excreta, 26–30% of the

administered dose was recovered. Liver contained 0.31 mg eq/kg (average), followed by kidney with 0.20 mg eq/kg and fat with 0.15 mg eq/kg. Radioactive residues in the eggs and tissues accounted for less than 1% of the administered dose. Muscle contained 0.023 mg eq/kg showing lower levels than other tissues. Radioactive residues in eggs reached a plateau on Day 4. No identification of metabolites was carried out in this study.

In the second study, approximately one third of the administered dose was recovered in excreta. About 3% of the administered dose was recovered as ethylene but this percentage is not reliable due to the leakage in the experiment. Radioactive residues in the eggs and tissues accounted for less than 1% of the administered dose. Kidneys contained 0.71–1.1 mg eq/kg, liver 0.63–0.90 mg eq/kg, and fat 0.051–0.091 mg eq/kg and muscle, 0.051–0.058 mg eq/kg. Radioactive residues in eggs did not reach a plateau within the study period of 5 days. Higher radioactivity was found in eggs in this study than the first study reaching the level of approximately 0.40 mg eq/kg on Day 5. In eggs, egg yolk contained much higher radioactivity than egg white (1.02 mg eq/kg egg yolk and 0.092 mg eq/kg in egg white).

Ethephon and HEPA were identified in methanol/water extracts of muscle, liver and kidney but not in the hexane/tetrahydrofuran extracts of fat or eggs (both yolk and white). Ethephon was the major residue in kidney accounting for 42% of TRR (0.30 mg/kg) but at a similar level as HEPA in liver (ethephon, 0.11 mg/kg; HEPA, 0.10 mg/kg) and muscle (ethephon, 0.006 mg/kg; HEPA, 0.009 mg/kg). Significant radioactivity was incorporated into amino acids (3–35% of TRR) in these tissues and in fatty acids (around 40% TRR) in fat. Significant amounts of radioactive residues (23 or 40% TRR for liver and 42 or 71% TRR for fat) remain unidentified. In eggs, radioactivity was incorporated into peptides (93% TRR in egg white) and fatty acids/cholesterol/glycerol (77–79% in egg yolk).

In summary, ethephon, when administered orally, was rapidly eliminated either in the excreta or expired as ethylene. Ethephon and HEPA were identified in kidney, liver and muscle in hens. Ethephon was found in kidneys of goats at very low concentrations. Ethephon was metabolized through two routes: metabolized to ethylene and/or to carbon dioxide through HEPA. A similar metabolic pattern was observed in rats, goats and hens. In livestock, radioactivity was found in fatty acids, proteins and glycogen.

Environmental fate

Hydrolysis

Ethephon degrades rapidly at pH 7 and 9 with the half-life of 2.4 and 1.0 day, respectively. At pH 5, it degrades more slowly with a half-life of 73.5 days. Ethylene gas and methylated phosphoric acid were the only degradation products found.

Photochemical degradation

Ethephon showed degradation under continuous irradiation for 360 hours at pH 5 at 25 °C. The half-life was 29 days under irradiation and 51 days without irradiation. Ethephon and ethylene were the only major compounds found. Ethylene was the only degradate of ethephon in the headspace.

Aerobic soil metabolism

The studies on aerobic soil degradation of ethephon in five different soils at 20–25 °C indicate that ethephon applied on soil degraded over time with different rates with the formation of ethylene. DT_{50} values ranged from 2.7–38 days for the five soils tested.

Photolysis on soil surface

Photolysis of ethephon on soil was found to be insignificant. Only ethylene and carbon dioxide were formed.

Field dissipation

Field dissipation studies were conducted at three sites in the USA. In all cases ethephon declined with time. DT_{50} values were 6.8–2 5 days.

Residues in succeeding crops

A confined rotational crop study was conducted to examine the nature and level of residues of ethephon in three succeeding crops (radish, collard and wheat) under outdoor conditions. A single application of radio-labelled ethephon was made on bare plots in plastic containers at a rate of 2.36 kg ai/ha (approximating the highest single application rate for cotton in the USA among approved label rates available to the Meeting). After plant back intervals (PBI) of 30, 120 and 379 days, collard, radish and wheat were planted into the treated soil. Mature radish, collard and wheat were harvested 54–62 days, 68–91 days and 110–158 days after planting. Immature wheat foliage was harvested 47–68 days after planting.

Ethephon declined steadily in soil. Radioactivity in mature plant samples declined in parallel with or faster than the decline in soil. The total extracted radioactive residues were at or lower than 0.07 mg eq/kg in any sample analysed. The solvent extraction recovered 34–37% TRR in 30 day PBI collards, 120 day PBI radish top and 30 day PBI and 120 day PBI wheat forage. As observed in the metabolism study on wheat, only 7.3–24% TRR were extracted by solvents from 30 day PBI and 120 PBI wheat grains and straw.

In the HPLC analysis of plant extracts, where radioactivity was sufficient for characterization, ethephon and HEPA were detected at or below 0.01 mg/kg in the extracts of radish, collard and wheat. No unknown peaks were observed. Sequential treatments of the unextracted radioactive residues for natural components indicated that most of the radioactivity in the plant samples were incorporated into biomolecules, such as starch, proteins, and cellulose fractions.

Overall, ethephon was shown to degrade relatively fast in soil with half-lives around or shorter than the plant back interval of 30 days. The confined succeeding crop study indicated the presence of very low levels of ethephon and HEPA in rotational crops. Therefore, no significant residues of ethephon or HEPA would be expected in rotational crops.

Methods of analysis

Analytical methods for determination of residues of ethephon and its metabolite HEPA were developed for a wide range of matrices of plant and animal origin.

There are three different principles for these analytical methods:

- Ethylene-release by heating in alkaline solution (headspace GC-FID)
- Derivatization to methyl ester using diazomethane (GC-FPD or GC-NPD)
- Extraction: mostly by methanol, acidified methanol or 0.01% formic acid
- LC-MS/MS (m/z 143→ 107 or 145-> 107 and HEPA 125→ 95)
- Extraction: mostly by a mixture of methanol, water and formic acid. Clean-up: mostly with SPE column.

The LC-MS/MS methods were used in the more recent studies.

The methods for plant matrices were validated for ethephon resulting in acceptable mean recoveries and relative standard deviations (RSDs) with the LOQ of 0.01–0.05 mg/kg. They are suitable for determining ethephon in a free form (some methods also for free HEPA).

An LC-MS/MS method was recently developed to determine ethephon and HEPA in both free and conjugated forms in cereal grains, straw and green materials. For the extraction of these compounds, grains and straw were extracted first with methanol and then by a mixture of

concentrated hydrochloric acid and water at 50 °C overnight and the extract and hydrolysate were combined for analysis. For green materials, this acid hydrolysis step was not included. This method was validated for ethephon and HEPA in these matrices resulting in acceptable mean recoveries and RSDs with the LOQ of 0.01 mg/kg for grains and 0.05 mg/kg for straw and green materials.

Methods for animal matrices were validated for ethephon resulting in acceptable mean recoveries and RSDs. The LOQ was 0.002–0.01 mg/kg. They are suitable for determining ethephon in a free form.

A multi-residue method DFG S19 (two variants) was examined for analysis of ethephon in plants for enforcement. However, due to low extraction (30%), this method does not seem appropriate for analysis of ethephon.

Stability of pesticide residues in stored analytical samples

The stability of ethephon was investigated in homogenates of various FROZEN plant and animal matrices at –20–15 °C, at fortification levels 0.2–1.0 mg/kg (plant matrices) or 0.1 mg/kg (animal matrices).

Ethephon was stable when stored frozen for at least 24 months in apples, cherries, grapes, blackberries, pineapples (fruit and forage), melons (36 months), peppers, tomatoes, wheat (grain and straw) and cotton seed (25 months). It was also stable for at least 12 months in apple juice and cotton seed oil.

Ethephon was stable when stored frozen for at least 4 months, the longest period tested, in bovine milk, bovine meat and egg.

Definition of the residue

Plant metabolism studies indicate that ethephon is metabolised in a qualitatively similar pattern in plants. Ethephon penetrates into plants after foliar application and residues of ethephon were found in edible commodities. Ethephon was metabolized to ethylene, which is naturally occurring in plants (but at levels not relevant to MRL setting). Ethephon was metabolized to form HEPA and further metabolized to be incorporated in many biomolecules, such as proteins, carbohydrates and lipids.

In the plants studied, ethephon was the major residue. Except for cereal grains, hay and straw, HEPA was found at much lower concentrations than the parent. In wheat plant fractions, HEPA was present at similar concentrations or higher concentrations than those of ethephon in grain and in hay.

In wheat grains and straw, radioactive residues were recovered at a significant proportion from acid hydrolysate and most of this radioactivity was attributed to ethephon and HEPA. This indicates that ethephon and HEPA were also present in these commodities in the form of conjugates.

The current Meeting considered that HEPA is not a toxicologically relevant metabolite as it does not inhibit cholinesterase activity and the NOAEL for HEPA in a 28-day gavage study in animals is at least two orders of magnitude higher than the NOAEL in humans that formed the basis of the ADI and ARfD.

Residues of ethephon were not expected to occur in significant concentrations in rotational crops.

In summary, the Meeting noted that in cereal grains and straw, presence of ethephon in the form of conjugates is significant. In other plant commodities, the Meeting considered that ethephon would be a good marker for enforcement and for estimation of dietary intake.

One recently developed and validated method, involving methanol extraction and acid hydrolysis/extraction of post methanol-extraction solids is capable of determining total ethephon

in free and conjugated forms in cereal matrices. There are other validated methods suitable for determining ethephon in its free form in plant matrices.

In animal metabolism studies, ethephon was rapidly eliminated either in the excreta or exhaled as ethylene. Ethephon was found at low levels in tissues. No metabolites were significant. The Meeting considered that ethephon is a suitable marker for enforcement and for estimation of dietary intake.

There are validated methods available for the determination of ethephon in its free form in animal matrices.

The log K_{ow} (–1.8 to –0.6 at 20 °C) indicates that ethephon is highly water-soluble. Although radioactive residues were found at higher levels in milk fat and egg yolk than skimmed milk or egg white, they were attributed to radioactivity incorporated into fatty acids. The Meeting concluded that the residue is not fat-soluble.

Based on the above, the Meeting recommended the following residue definitions for plant and animal commodities.

Definition of the residue for plant commodities except cereal grains and straw (for compliance with the MRL and for estimation of dietary intake): *Ethephon.*

Definition of the residue for cereal grains and straw (for compliance with the MRL and for estimation of dietary intake*): Ethephon and its conjugates, expressed as ethephon*.

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

The residue is not fat-soluble.

Results of supervised residue trials on crops

The Meeting received supervised trial data for ethephon on apples, cherries, grapes, figs, olives, pineapples, tomatoes (outdoor and indoor), barley, rye, wheat and cotton using foliar sprays of mostly SL formulations containing various concentrations of ethephon.

As ethephon is reviewed under the periodic review programme, the Meeting decided to withdraw its previous recommendations for blueberries, cantaloupes, peppers, dried chilli peppers, hazelnuts and walnuts due to the lack of data.

Apple

A total of 18 supervised trials were conducted on apples in Europe in 2000, 2002, 2006 and 2007, eight in France, two in Germany, one in the UK, two in Italy, two in Spain, one in Portugal and two in Greece.

Residues of ethephon from 13 trials matching critical GAP for apple in France (0.036 kg ai/hL, one to two applications, and PHI 10 days) were: < 0.05, 0.06, 0.07, 0.08, 0.08, 0.14, 0.15, 0.15, 0.24, 0.26, 0.27, 0.40 and 0.49 mg/kg.

The trials matching GAP in France were appropriate for estimating a maximum residue level. The Meeting estimated a maximum residue level of 0.8 mg/kg for apples to replace the previous recommendation. The Meeting also estimated an STMR of 0.15 mg/kg and an HR of 0.49 mg/kg.

Cherries

A total of 15 supervised trials were conducted on cherries in Europe in 2000, 2002 and 2009, ten in France, one in Italy, one in Spain, one in Greece, one in Belgium and one in the Netherlands.

Residues of ethephon from 13 trials matching GAP in Austria for cherries and in the Netherlands for sour cherries (0.36 kg ai/ha, one application, PHI 7 days) were: 0.28, 0.30, 0.33, 0.37, 0.44, 0.52, 0.65, 0.67, 0.91, 1.4, 2.0, 2.3 and 2.7 mg/kg.

The Meeting estimated a maximum residue level of 5 mg/kg for cherries to replace the previous recommendation and an STMR of 0.65 mg/kg and an HR of 2.7 mg/kg.

Grapes

A total of ten supervised trials were conducted on grapes in France in 1995, 2006 and 2009. The GAP in France for grapes allows one application at a maximum rate of 0.45 kg ai/ha with a PHI of 28 days.

Residues from ten trials matching GAP in France were: 0.05, 0.07, 0.14, 0.18, 0.18, 0.20, 0.21, 0.25, 0.37 and 0.52 mg/kg.

The Meeting estimated a maximum residue level of 0.8 mg/kg for grapes to replace the previous recommendation, an STMR of 0.19 mg/kg and an HR of 0.52 mg/kg.

Fig

Six supervised trials were conducted on figs in Brazil in 2004–2005. GAP in Brazil for figs allows one application of 0.94 kg ai/hL with a PHI of 5 days. Ethephon should be applied directly to fruits using brushes with sponge tips or other equipment for even distribution.

Residues from three trials matching GAP in Brazil were, 0.71, 0.73 and 0.75 mg/kg. The Meeting estimated a maximum residue level of 3 mg/kg, an STMR of 0.73 mg/kg and an HR of 0.75 mg/kg for fig.

Olives

Eight supervised trials were conducted on olives in Spain in 2007–2008. GAP in Italy allows two applications (1st application 18 days before harvest at a rate of 0.45 kg ai/ha and 2nd application 11 days before harvest at 0.60 kg ai/ha) with a PHI of 11 days.

Residues from eight trials matching GAP in Italy were, 0.85, 0.90, 0.98, 1.6, 2.2, 2.5, 2.6 and 4.3 mg/kg.

The Meeting estimated a maximum residue level of 7 mg/kg, an STMR of 1.9 mg/kg and an HR of 4.3 mg/kg for olives.

Pineapple

A total of 15 supervised trials were conducted. Five in Brazil in 1994, 1995 and 2005, two in Costa Rica in 1998, two in Côte d'Ivoire in 1997 and 1998, and six in the USA in 1989.

GAP in Kenya for pineapple allows one application at the maximum rate of 1.92 kg ai/ha with a PHI of 7 days. Residues from trial conducted in Côte d'Ivoire matching this GAP were (n=2): 0.11 and 0.97 mg/kg.

Residues from five trials in Brazil matching GAP in Brazil for pineapple (one application at a maximum rate of 0.94 kg ai/ha with a PHI of 14 days) were: < 0.05, 0.11, 0.15, 0.19 and 0.20 mg/kg.

The trials conducted in the USA involved two applications of ethephon and the rate of the first application was two times higher than GAP in Costa Rica (up to two applications at the maximum rate of 1.2 kg ai/ha with a PHI of 1 day; first one 5–7 months before harvest and second 1–2 weeks before harvest) but it was made six months earlier than the expected harvest time with little impact on the residues at harvest.

In the trial in Costa Rica, pineapple was harvested on 0 DALA but as the decline trials indicated that there was no significant decline from 0 to 1 DALA, the Meeting agreed to use the data from 0 DALA.

Residues from trials conducted in the USA and Costa Rica matching GAP in Costa Rica were (n=4), 0.19, 0.22, 0.42 and 0.72 mg/kg. One trial conducted in Brazil matched GAP in

Costa Rica and residues were (n=1), 0.47 mg/kg. Combined residue dataset was (n=5), 0.19, 0.22, 0.42, 0.47 and 0.72 mg/kg.

As the dataset from five trials matching GAP in Costa Rica would lead to a higher maximum residue level than the dataset from five trials matching GAP in Brazil, the Meeting decided to use the dataset associated with GAP in Costa Rica. The Meeting estimated a maximum residue level of 1.5 mg/kg to replace its previous recommendation.

The Meeting calculated a mean pulp/whole fruit ratio to be 0.29 using residue levels higher than LOQ. Using the mean and highest residue in whole fruit and this ratio, the Meeting estimated an STMR of 0.12 mg/kg and an HR of 0.21 mg/kg for pineapple.

Tomato

A total of 33 supervised trials on tomatoes were conducted. Twenty-one trials were in Europe in 1999, 2000, 2001 and 2004 and 15 in the USA in 1989–1991 and 2005. As the labels provided to the Meeting do not specify outdoor or indoor uses, the Meeting considered both trials conducted outdoor and indoor.

The critical GAP for the European trials was GAP in Italy which allows the maximum rate of 1.92 kg ai/ha which can be divided into two applications with a PHI of 7 days. Residues from 12 outdoor trials in Europe matching GAP in Italy were 0.24, 0.30, 0.40, 0.45, 0.46, 0.5, 0.55, 0.57, 0.62, 0.68, 0.78, and 0.78 mg/kg.

Residues from nine indoor trials matching GAP in Italy were 0.31, 0.36, 0.45, 0.51, 0.52, 0.66, 0.68, 0.69 and 0.79 mg/kg.

Residues from five independent outdoor trials in the USA matching GAP in Canada (one application of 1.54 kg ai/ha, PHI 14–21 days) were 0.05, 0.06, 0.09, 0.67 and 0.69 mg/kg.

As the outdoor and indoor trials conducted in Europe were in compliance with the same GAP of Italy and they were not significantly different according to Mann-Whitney U test, they could be combined to estimate a maximum residue level. Residues in the combined data set were 0.24, 0.30, 0.31, 0.36, 0.40, 0.45, 0.45, 0.46, 0.5, 0.51, 0.52, 0.55, 0.57, 0.62, 0.66, 0.68, 0.68, 0.69, 0.78, 0.78 and 0.79 mg/kg.

The Meeting confirmed the pervious recommendation of 2 mg/kg for tomato and estimated an STMR of 0.52 mg/kg and an HR of 0.79 mg/kg.

Cereal grains

As the residue definition for cereal grains was recommended to be “ethephon and its conjugates, expressed as ethephon”, the Meeting used only those trial data obtained with the recently developed analytical method involving acid hydrolysis to convert ethephon conjugates to free ethephon.

Barley

A total of 53 trials were conducted in Europe in 2000, 2001, 2004, 2007, 2008, 2013 and 2014 on barley.

There are several different groups of GAP in Europe. Critical GAP is either GAP in the UK allowing a maximum single rate of 0.48 kg ai/ha, maximum total rate of 0.48 kg ai/ha, and application timing up to BBCH 49, or GAP in Germany allowing one application at a maximum rate of 0.46 kg ai/ha up to BBCH 49.

Residues from seven trials matching GAP in the UK or Germany were 0.03, 0.07, 0.09, 0.13, 0.23, 0.41, 0.73 mg/kg.

The Meeting estimated, using the dataset matching GAP in the UK or Germany, a maximum residue level of 1.5 mg/kg for barley grains to replace the previous recommendation, and an STMR of 0.13 mg/kg.

Rye

Nine supervised trials were conducted in 2006–2007 in Europe. No data were available on the sum of free and conjugated ethephon in rye grains. (See "Wheat" section below.)

Wheat

A total of 43 supervised trials were conducted on wheat in Europe in 2000, 2001, 2004, 2006, 2007, 2013 and 2014.

There are several different groups of GAP in Europe. Critical GAP is that in Austria and Germany allowing one application at a maximum rate of 0.46 kg ai/ha with application timing up to BBCH 51.

Residues from eight supervised trials matching these GAP were 0.05, 0.06, 0.06, 0.08, 0.11, 0.14, 0.23 and 0.31 mg/kg.

The Meeting estimated, using the dataset from trials matching GAP in Austria and Germany, a maximum residue level of 0.5 mg/kg for wheat grains to replace the previous recommendation, and an STMR of 0.095 mg/kg.

As there are similar GAPs existing for wheat, rye and triticale in countries in Europe, the Meeting decided to extrapolate the maximum residue level and STMR for wheat to rye and triticale.

Cotton seed

A total of ten trials were conducted in Europe in 1993, 1994, 1995 and 2008 on cotton, 41 trials in the USA in 1989, 1993 and 1994, and seven trials in Brazil in 1996 and 2006.

Residues from ten trials conducted in Europe matching GAP in Greece for cotton (one application at a maximum rate of 1.44 kg ai/ha with a PHI of 7 days) were 0.07, < 0.10, < 0.10, < 0.10, 0.10, 0.19, 0.30, 0.35, 0.59 and 1.13 mg/kg.

Residues from six independent trials conducted in Brazil matching GAP in Brazil for cotton (one application at a maximum rate of 1.2 kg ai/ha with a PHI of 7 days) were all below the LOQ: < 0.10 (4) and < 0.20 (2) mg/kg.

Residues from 30 trials matching GAP in the USA for cotton (one application at a maximum rate of 2.24 kg ai/ha with a PHI of 7 days) were 0.06, 0.09, 0.10, 0.11, 0.16, 0.18, 0.23, 0.24, 0.26, 0.26, 0.34, 0.35, 0.36, 0.41, 0.54, 0.55, 0.59, 0.61, 0.65, 0.69, 0.75, 0.86, 1.18, 1.42, 1.50, 2.40, 2.42, 2.73, 2.88 and 4.93 mg/kg.

As the residues from US trials would lead to a higher maximum residue level, the Meeting used the results of the US trials to estimate a maximum residue level. The Meeting estimated a maximum residue level of 6 mg/kg for cotton seed to replace the previous recommendation, and an STMR of 0.545 mg/kg.

Animal feed

Cereal forage

As there is no restriction on feed uses of treated cereal plants, the Meeting used residues in forage samples collected on 0 DALA for cereal forage. Since the determination of ethephon in green materials do not require acid hydrolysis, the Meeting used all available data on barley green material.

Barley forage

Residues in forage collected on 0 DAT from 19 trials matching GAP in the UK or GAP in Germany (a maximum single rate of 0.48 kg ai/ha, maximum total rate of 0.48 kg ai/ha, and application timing up to BBCH 49, or one application at a maximum rate of 0.46 kg ai/ha up to BBCH 49) were 2.6, 3.0, 3.2, 4.2, 4.8, 5.1, 5.7, 6.2, 6.2, 6.2, 6.6, 6.6, 7.7, 7.9, 8.1, 8.4, 9.4, 10 and 11 mg/kg.

Residues from 15 trials matching GAP in France (one application at a maximum application rate of 0.48 kg ai/ha and application timing up to BBCH 39 with a PHI of 56 days) in forage were 3.3, 3.5, 4.2, 4.6, 5.2, 5,6, 5.6, 5.9, 6.0, 6.2, 6.7, 8.1, 8.2, 8.3 and 9.5 mg/kg.

Residues from five trials matching GAP in Poland (one application at a maximum application rate of 0.72 kg ai/ha and application timing up to BBCH 39 were 6.0, 7.1, 8.9, 9.6 and 13 mg/kg.

Residues from seven trials matching another GAP in France (one application at a maximum rate of 0.23 kg ai/ha and application timing up to BBCH 39) were 3.0, 3.7, 4.1, 4.5, 5.2, 5.4, 5.9 and 7.5 mg/kg.

Residues arising from five trials using the application rate of 0.72 kg ai/ha showed higher median and highest residues. Based on this dataset, the Meeting estimated a median residue of 8.9 mg/kg and a highest residue of 13 mg/kg ("as received" basis) for barley forage for animal dietary burden calculation.

Rye forage

Residues in forage collected on 0 DAT from nine trials matching GAP in Germany and Austria (one application at a max rate of 0.73 kg ai/ha, application timing up to BBCH 49) were 4.4, 6.4, 7.2, 7.7, 9.1, 9.2, 9.4, 9.6 and 13 mg/kg.

The Meeting estimated a median and highest residue of 9.1 mg/kg and 13 mg/kg for rye forage on an "as received" basis.

Wheat forage

Residues in forage collected 0 DAT from 17 trials matching GAP in Austria and Germany (one application at a maximum rate of 0.46 kg ai/ha, application timing up to BBCH 51) were 3.1, 3.3, 3.5, 4.0, 4.9, 5.2, 5.9, 6.2, 6.4, 6.5, 7.0, 7.0, 7.1, 7.2, 7.5, 10 and 16 mg/kg.

Residues from 18 trials matching GAP in France (one application at a maximum rate of 0.48 kg ai/ha and application timing up to BBCH 39) were 3.1, 4.5, 4.9, 5.6, 5.7, 6.0, 6.1, 6.9, 7.0, 7.2, 7.4, 7.7, 8.3, 12, 14, 14, 17 and 18 mg/kg

Using the dataset from trials matching GAP in France, the Meeting estimated a median residue of 7.1 mg/kg and a highest residue of 18 mg/kg for wheat forage ("as received" basis).

Cereal straw and fodder, dry

As the residue definition for cereal straw was recommended to be "ethephon and its conjugates, expressed as ethephon", the Meeting used only those trial data obtained using the recently developed analytical method involving acid hydrolysis to convert ethephon conjugates to free ethephon.

Barley straw and fodder, dry

Residues from seven trials matching GAP in the UK or Germany (a maximum single rate of 0.48 kg ai/ha, maximum total rate of 0.48 kg ai/ha, and application timing up to BBCH 49, or one application at a maximum rate of 0.46 kg ai/ha up to BBCH 49) in straw were 0.35, 0.43, 0.51, 0.64, 1.2, 1.5 and 3.6 mg/kg.

Using the data set from the trials matching GAP in the UK or Germany, the Meeting estimated a maximum residue level of 7 mg/kg on a dry weight basis (moisture content of 89%) to replace the previous recommendation. For the purpose of calculation of animal dietary burden, the Meeting estimated a median residue and highest residue of 0.64 mg/kg and 3.6 mg/kg ("as received" basis).

Rye straw and fodder, dry

No data were available on the sum of free and conjugated ethephon in rye straw. (See "Summary of cereal straw and fodder, dry" section below.)

Wheat straw and fodder

Residues from eight trials matching GAP in Austria and Germany (one application at a maximum rate of 0.46 kg ai/ha and application timing up to BBCH 51) in straw were 0.36, 0.44, 0.57, 0.66, 1.2, 1.2, 1.3 and 1.5 mg/kg.

Residues from eight trials matching GAP in France (one application at a maximum rate of 0.48 kg ai/ha and application timing up to BBCH 39 with a PHI of 70 days) in straw were 0.21, 0.29, 0.30, 0.44, 0.84, 0.86, 1.2 and 1.7 mg/kg.

Using the data set from the trials matching GAP in France, the Meeting estimated a median residue of 0.64 mg/kg and a highest residue of 1.7 mg/kg ("as received" basis).

Summary

The Meeting noted that it is not always possible to distinguish straw and fodder of barley, rye, triticale and wheat moving in trade, due to their similarity in appearance. It also noted that there are common or similar GAPs existing for wheat, rye and triticale in countries in Europe. The Meeting decided to extend the maximum residue level recommended for barley straw and fodder at 7 mg/kg on a dry weight basis to straw and fodder of wheat, rye and triticale. The new maximum residue levels for rye and wheat straw and fodder, dry replaces the respective previous recommendations.

The median residue and highest residue estimated for wheat straw and fodder should also apply to rye and triticale straw and fodder, dry.

Cotton gin trash

In 12 US trials, residues in cotton gin trash were analysed and reported. Residues in cotton gin trash from ten trials matching GAP in the USA were: 8.41, 11.1, 13.5, 17.1, 25.1, 28.9, 40.5, 45.5, 54.2 and 55.7 mg/kg. The Meeting estimated a median residue of 27 mg/kg. From the highest residue concentration of individual samples, the Meeting estimated a highest residue of 67 mg/kg.

Fate of residues during processing

High temperature hydrolysis

To simulate the degradation of ethephon during pasteurization, baking, brewing, boiling and sterilisation, the hydrolysis of radio-labelled ethephon was investigated in sterile buffered aqueous solutions.

After incubation at 90 °C (pH 4) for 20 minutes, about 80% of ethephon remained and about 10% was recovered as ethylene. The majority of ethephon was converted to ethylene (76–78%) after incubation at 100 °C (pH 5) for 60 minutes or 120 °C (pH 6) for 20 minutes. Only a minor amount of HEPA was formed.

Processing

The Meeting received information on processing of apple, grapes, olives, tomato, barley, wheat, and cotton seed.

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 apples, grapes, tomatoes, barley, wheat and cotton seed for which maximum residue levels were estimated. Where residues concentrate in processed commodities the Meeting estimated maximum residues levels for these processed commodities using the maximum residue levels for the respective raw agricultural commodities and processing factors.

As no data were available on the processing of fig to dried or dried and candied figs, the Meeting withdrew its previous recommendation on figs, dried and dried and candied.

The processing factor of grape to dried grapes was estimated at 1.2 and therefore a maximum residue level for dried grapes was unnecessary. The Meeting decided to withdraw its previous recommendation on dried grapes.

RAC or Processed commodities	Processing factor		STMR-P	Maximum residue level
	Individual value	Best estimate		
Apple			0.15 (STMR)	0.8
Apple juice	< 0.4, 0.4, 0.5, < 0.8, 1.5	0.5	0.075	–
Apple sauce	0.4, 0.5, < 0.8, 1.1	0.5	0.075	–
Grape			0.19(STMR)	0.8
Dried grapes	0.79, 0.89, 1.0, 1.4, 3.2, 8.5	1.2	0.23	–
Grape juice	0.5, 0.7, 0.8, 1.1	0.75	0.14	–
Must	0.7, 0.8, 0.8, 0.9, 1.0, 1.0	0.85	0.16	–
Wine	0.7, 1.0, 1.2, 1.4, 1.5, 2.1,	1.3	0.25	–
Olives			1.9	–
Olive oil (virgin and refined)	< 0.02, < 0.03	< 0.02	0.038	–
Table olives	< 0.01, < 0.02, < 0.02, < 0.03	< 0.01	0.019	–
Tomato			0.52(STMR)	2
Tomato juice	< 0.1, 0.1, < 0.2, 0.34	0.22	0.18	–
Tomato puree	< 0.1, < 0.1, < 0.2, 0.60	0.60	0.31	–
Tomato paste	0.5, 0.6, 0.75	0.6	0.31	–
Tomato preserves	< 0.1, < 0.2, 0.2	0.2	0.10	–
Barley			0.13(STMR)	1.5
Pearl barley	0.9	0.9	0.12	–
Wheat			0.095 (STMR)	0.5
Flour	0.1, 0.2, < 0.3,	0.15	0.014	–
Wheat germ	2.0	2.0	0.19	1
Wheat bran	1.4, 3.1, 3.5	3.1	0.29	1.5
Cotton seed			0.545 (STMR)	6
Cottonseed refined oil	< 0.02,< 0.03, < 0.03	< 0.02	0.011	–

For the purpose of calculating animal dietary burden, the Meeting estimated the following median residues for feed items.

RAC or Processed commodities	Processing factor		median residue
	Individual value	Best estimate	
Apple			0.15 (STMR)
Wet pomace	0.3, 0.4, 0.6, < 0.8, 1.1	0.5	0.075
Dry pomace	2.0	2.0	0.30
Grape			0.19(STMR)
Wet pomace	0.4, 0.6, 0.9, 1.1	0.75	0.14
Tomato			0.52(STMR)
Wet pomace	< 0.1, < 0.1, < 0.2, 0.52	0.52	0.27
Dry pomace	1.9	1.9	0.99
Barley			0.13(STMR)
Barley hulls	1.6	1.6	0.21
Cotton seed			0.55 (STMR)
Meal	0.02, 0.03, 0.07	0.03	0.016

Residues in animal products

Farm animal feeding studies

Lactating cows received oral administration of ethephon at dose rates equivalent to 44, 128 and 415 ppm in the diet once daily for 28 consecutive days. The residues of ethephon in whole milk appeared to reach plateau after Day 4. Ethephon in milk was 0.007 mg/kg at 44 ppm dose, 0.02 mg/kg at 128 ppm dose, and 0.03 mg/kg at the 415 ppm dose. After a 28 day-administration, the highest concentration of ethephon in kidney was 0.58, 3.2 and 7.8 mg/kg respectively after 44, 128 and 415 ppm dose. In liver, it was 0.08, 0.51 and 0.99 mg/kg. In muscle, the ethephon concentration was much lower at 0.01, 0.05 and 0.12 mg/kg for these dose groups. In fat, at the highest dose, ethephon was present at only 0.06 mg/kg.

Laying hens were orally administered with ethephon at rates equivalent to 2.3, 6.9 and 23 ppm in the diet once daily for 28 consecutive days. The residues of ethephon in whole eggs were very low and those from the highest dose group contained at a maximum 0.0036 mg/kg. Therefore, eggs from the 2.3 ppm and 6.9 diets were not analysed. After 28-day administration, liver contained the highest concentration of ethephon, 0.033 mg/kg at the 2.3 ppm dose, 0.068 at the 6.9 ppm dose and 0.29 ppm at the 23 ppm dose. In skin + fat, it was 0.014, 0.032 and 0.117 mg/kg. In muscle, it was 0.060 at 23 ppm diet.

Estimation of dietary burdens

The maximum and mean dietary burdens were calculated using the highest and median residues of ethephon estimated at the current Meeting on a basis of the OECD Animal Feeding Table. In Australia, use of ethephon-treated cereal green materials as feed is not allowed and cereal forage is not in trade. Residues arising from use of ethephon in barley, rye and wheat forages were not used for calculating animal dietary burden for the Australian diets.

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	4.19	1.65	18.8	9.14	4.04	0.81	0.13	0.13
Dairy cattle	14.5	6.22	18.9 [a]	9.17 [b]	1.46	0.79	0.059	0.059
Broilers	0.11	0.11	0.10	0.10	0.024	0.024	0.015	0.015
Layers	0.11	0.11	7.33 [c]	3.17 [d]	0.024	0.024	0.012	0.012

[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

[c] Suitable for estimating maximum residue levels for eggs, meat, fat and edible offal of poultry

[d] Suitable for estimating STMRs for eggs, meat, fat and edible offal of poultry

Residues in milk and cattle tissues

The maximum and mean dietary burdens in cattle were 18.9 and 9.17 ppm of dry matter diet respectively for estimating a maximum residue level and STMR for milk and edible tissues. The maximum residue levels, STMRs and HRs for relevant commodities of mammal origin were estimated using the residue levels in tissues and milk at 0 and 44 ppm feeding groups.

	Feed level (ppm) for milk residues	Ethephon (mg/kg) in milk	Feed level (ppm) for tissue residues	Ethephon (mg/kg) in			
				Muscle	Liver	Kidney	Fat
Maximum residue level beef or dairy cattle							
Feeding study [a]	0 44	– 0.002	0 44	< 0.01 0.016	0.05 0.095	0.03 0.64	< 0.01 < 0.01
Dietary burden and highest residue	18.9	0.0009	18.8	0.007	0.069	0.29	0.004
STMR beef or dairy cattle							

Feeding study [b]	0 44	– 0.002	0 44	< 0.01 0.01	0.05 0.08	0.03 0.49	< 0.01 < 0.01
Dietary burden and mean residue	9.17	0.0004	8.25	0.002	0.056	0.13	0.002

[a] Highest residues for tissues and mean residue for milk

[b] Mean residues for tissues and mean residue for milk

The level < LOQ at 0 ppm dose is assumed to be 0 mg/kg residue.

The Meeting estimated STMRs of 0.0004, 0.002, 0.056, 0.13 and 0.002 mg/kg, and HRs of 0.0009, 0.007, 0.069, 0.29 and 0.004 mg/kg for milk, meat, liver and kidney respectively.

On a basis of highest residues above, the Meeting estimated maximum residue levels of 0.01*, 0.01*, 0.4 and 0.01* mg/kg for milks mammalian meat, edible offal and fat, respectively.

The previous recommendations for milk of cattle, goats and sheep, meat of cattle, goats, houses, pigs and sheep, and edible offal of cattle, goats, horses, pigs and sheep were withdrawn.

Residues in eggs and chicken tissues

The maximum and mean dietary burdens in poultry were 7.33 and 3.17 ppm of dry matter diet respectively for estimating a maximum residue level and STMR for eggs and edible tissues. The maximum residue levels, STMRs and HRs for relevant commodities of poultry origin were estimated using the residue levels in tissues and eggs at 2.3, 6.9 and 23 ppm feeding groups.

	Feed level (ppm) for egg residues	Ethephon (mg/kg) in			
		Eggs	Muscle	Liver	Fat [a]
Maximum residue level broiler or layer hens					
Feeding study	6.9 23	na 0.0023	0.015 0.060	0.068 0.23	0.032 0.117
Dietary burden and highest residue	7.33	0.00005	0.016	0.072	0.034
STMR broiler or layer hens					
Feeding study	2.3 6.9	na na	< 0.01 0.012	0.031 0.062	0.013 0.024
Dietary burden and mean residue	3.17	0 [b]	0.01	0.037	0.015

[a] From data in fat + skin

[b] At a dose of 23 ppm in the dry matter diet, residues were 0.0036 mg/kg

The Meeting estimated STMR of 0, 0.01, 0.037 and 0.015 mg/kg, and HR of 0.00005, 0.016, 0.072 and 0.034 mg/kg, respectively for poultry eggs, meat, edible offal and fat.

On a basis of HR, the Meeting estimated maximum residue levels of 0.01 *, 0.02, 0.08 and 0.04 mg/kg for eggs, poultry meat, edible offal and fat, respectively. The recommendations for poultry meat and edible offal replace the previous recommendations.

The Meeting withdrew its previous recommendation on chicken eggs.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed in Annex 1 are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for plant commodities except cereal grains and straw (for compliance with the MRL and for estimation of dietary intake): *Ethephon.*

Definition of the residue for cereal grains and straw (for compliance with the MRL and for estimation of dietary intake): *Ethephon and its conjugates, expressed as ethephon*.

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

The residue is not fat-soluble.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FP 0226	Apple	0.8	5	0.15	0.49
GC 0640	Barley	1.5	1	0.13	
AS 0640	Barley straw and fodder, Dry	7 (dw)[b]	5	0.64[a]	3.6 [a]
FB 0020	Blueberries	W	20		
FC 4199	Cantaloupe	W	1		
FS 0013	Cherries	5	10	0.65	2.7
PE 0840	Chicken eggs	W	0.2*		
SO 0691	Cotton seed	6	2	0.55	
DF 0269	Dried grapes	W	5	0.23	
MO 0105	Edible offal (mammalian)	0.4		Kidney 0.056 Liver 0.12	Kidney 0.069 Liver 0.29
MO 0096	Edible offal of cattle, goats, horses, pigs and sheep	W	0.2*		
PE 0112	Eggs	0.01*		0	0.00005
FT 0297	Fig	3		0.73	0.75
DF 0297	Figs, Dried or dried and candied	W	10		
FB 0269	Grapes	0.8	1	0.19	0.52
TN 0666	Hazelnuts	W	0.2		
MF 0100	Mammalian fats (except milk fats)	0.01*		0.002	0.004
MM 0095	Meat (from mammals other than marine mammals)	0.01 *		0.002	0.007
MM 0096	Meat of cattle, goats, horses, pigs and sheep	W	0.1*		
ML 0106	Milks	0.01 *		0.0004	
ML 0107	Milk of cattle, goats and sheep	W	0.05*		
FT 0305	Olives	7		1.9	4.3
VO 0051	Peppers	W	5		
HS 0444	Peppers Chili, dried	W	50		
FI 0353	Pineapple	1.5	2	0.12	0.21
PM 0110	Poultry meat	0.02	0.1*	0.01	0.016
PO 0111	Poultry, Edible offal of	0.08	0.2*	0.037	0.072
PF 0111	Poultry fats	0.04		0.015	0.034
GC 0650	Rye	0.5	1	0.095	
AS 0650	Rye straw and fodder, Dry	7 (dw)	5	0.64 [a]	1.7 [a]
VO 0448	Tomato	2	2	0.52	0.79
GC 0651	Triticale	0.5		0.095	
	Triticale straw and fodder, Dry	7 (dw)		0.64 [a]	1.7 [a]
TN 0678	Walnut	W	0.5		
GC 0654	Wheat	0.5	1	0.095	
CM 0654	Wheat bran	1.5		0.29	
CF 1201	Wheat germ	1		0.19	
AS 0654	Wheat straw and fodder, Dry	7 (dw)	5	0.64 [a]	1.7 [a]
JF 0226	Apple juice			0.075	
	Apple sauce			0.075	
OC 0691	Cotton seed oil, edible			0.011	
DF 0269	Dried grapes (=currants, Raisins and Sultanas)			0.23	
JF 0269	Grape juice			0.14	
	Grape must			0.16	
	Olive oil, virgin and refined			0.038	
DM 0305	Olives, processed			0.019	
	Pearl barley			0.12	

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
JF 0048	Tomato juice			0.18	
VW 0448	Tomato paste			0.31	
	Tomato preserves			0.1	
MW 0448	Tomato puree			0.31	
CF 1211	Wheat four			0.014	
	Wine			0.25	
AB 1230	Apple pomace, wet			0.075	
	Barley forage			8.9	13
	Barley hulls			0.21	
OR 0691	Cotton seed meal			0.016	
AB 1204	Cotton gin trash			27	67
	Grape pomace wet			0.14	
AF 0650	Rye forage (green)			9.1	13
	Tomato pomace wet			0.27	
	Wheat forage			7.1	18

[a] as received basis

[b] dw – dry weight

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Dietary Intakes (IEDIs) of ethephon were calculated for the 17 GEMS/Food cluster diets using STMRs and STMRPs estimated by the current Meetings (see Annex 3 to the 2015 Report). The ADI is 0–0.05 mg/kg bw and the calculated IEDIs were 0–6% of the maximum ADI. The Meeting concluded that the long-term intake of residues of ethephon 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 Intakes (IESTI) of ethephon were calculated for commodities using HRs/HR-Ps and STMRs/STMR-Ps estimated by the current Meeting (see Annex 4 to the 2015 Report). The ARfD is 0.05 mg/kg and the calculated IESTIs were 0–100% of the ARfD for the general population and 0–70% of the ARfD for children. The Meeting concluded that the short-term intake of residues of ethephon, when used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

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RP-01-89A [M-187533-01-1]	Eckert, JA	1992	Determination of the Storage Stability of Ethephon in Tomato Fruit. Enviro-Bio-Tech Ltd., PA 19506, USA. GLP, Unpublished
RP-01-89I [M-187521-01-1]	Eckert, JA	1992	Determination of the Storage Stability of Ethephon in Wheat Grain. Enviro-Bio-Tech Ltd., PA 19506, USA. GLP, Unpublished
RP-01-89H [M-187519-01-1]	Eckert, JA	1992	Determination of the Storage Stability of Ethephon in Wheat Straw. Enviro-Bio-Tech Ltd., PA 19506, USA. GLP, Unpublished
89-REN-WA-S [M-187529-01-1]	Nygren, RE	1992	Storage Stability of Ethephon in/on Walnut Nutmeats. Rhône-Poulenc Ag Company, NC 27709, USA. Non-GLP, Unpublished
RP-01-89J [M-187525-01-1]	Eckert, JA	1992	Determination of the Storage Stability of Ethephon in Cottonseed. Enviro-Bio-Tech Ltd., PA 19506, USA. GLP, Unpublished
MR-09/053 [M-384885-01-1]	Schmeer, K & Reineke, A	2010	Determination of the storage stability of cyclanilide and ethephon and its metabolite HEPA in fortified control samples of cotton seeds during storage at room temperature for 3 months. Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. GLP, Unpublished
RP-01-89D [M-187544-01-1]	Eckert, JA	1992	Determination of the Storage Stability of Ethephon in Grape Berries. Enviro-Bio-Tech Ltd., PA 19506, USA. GLP, Unpublished
RP-01-89B [M-187511-01-1]	Eckert, JA	1992	Determination of the Storage Stability of Ethephon in Blackberry Fruit. Enviro-Bio-Tech Ltd., PA 19506, USA. GLP, Unpublished
RP-01-89E [M-187540-01-1]	Eckert, JA	1992	Determination of the Storage Stability of Ethephon in Pineapple Fruit. Enviro-Bio-Tech Ltd., PA 19506, USA. GLP, Unpublished
RP-01-89F [M-187538-01-1]	Eckert, JA	1992	Determination of the Storage Stability of Ethephon in Pineapple Forage. Enviro-Bio-Tech Ltd., PA 19506, USA. GLP, Unpublished
EC-94-253 [M-188009-01-1]	Nygren, RE	1995	Storage Stability of Ethephon in Apple juice and Cottonseed Oil Spiked with Ethephon. Rhône-Poulenc Ag Company, NC 27709, USA. GLP, Unpublished
EC-92-198	Leonard, MS	1993	Storage Stability of Ethephon in/on Frozen Bovine Meat, Bovine Milk and Chicken Eggs Spiked with Ethephon. Rhône-Poulenc Ag Company, NC 27709, USA. Report No. EC-92-198, File no. 44198 [M-187997-01-1] GLP, Unpublished
R&D/CRLD/ AN/0215010 [M-209123-01-1]	Ballesteros, C	2002	Ethephon and its metabolite (RPA732569), Formulation EXP03149B (SL), North/France/2000—2 trials—Harvest study, Residues in apple (fruit). Aventis Cropscience, F-69009 Lyon, France. GLP, Unpublished

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R&D/CRLD/ AN/0215012 [M-210409-01-1]	Ballesteros, C	2002	Ethephon and its metabolite (RPA732569), Formulation EXP03149B (SL), North/France/2000—2 trials—Decline study, Residues in apple (fruit). Aventis Cropscience, F-69009 Lyon, France. GLP, Unpublished
02 R 792 [M-220915-01-1]	Sonder, KH	2004	Residue Behaviour in Apples European Union (Northern Zone) 2002. Bayer CropScience GmbH, D-65926 Frankfurt, Germany. GLP, Unpublished
RA-2514/06 [M-292470-01-1]	Billian, P	2007	Determination of the residues of ethephon in/on apple after spraying of AE F016382 00 SL40 A1 (480 SL) in the field in Southern France, Italy, Spain, Portugal and Greece. Bayer CropScience AG, D-40789 Monheim, Germany. GLP, Unpublished
RA-2576/07 [M-311032-01-1]	Billian, P, Erler, S & Wolters, A	2008	Determination of the residues of ethephon in/on apple after spraying of AE F016382 00 SL40 A1 (480 SL) in the field in southern France, Italy, Spain and Greece. Bayer CropScience AG, D-40789 Monheim, Germany. GLP, Unpublished
R&D/CRLD/ AN/mr/0115439 [M-208089-01-1]	Ballesteros, C	2002	Ethephon and its metabolite (RPA732569), Formulation EXP03149B (SL), South/France/2000—2 trials—Decline study, Residues in cherry (fruit). Aventis Cropscience, F-69009 Lyon, France. GLP, Unpublished
R&D/CRLD/ AN/0115458 [M-208961-01-1]	Ballesteros, C	2002	Ethephon and its metabolite (RPA732569), Formulation EXP03149B (SL), South/France/2000—2 trials—Harvest study, Residues in cherry (fruit). Aventis Cropscience, F-69009 Lyon, France. GLP, Unpublished
R&D/CRLD/ AN/0215009 [M-210351-01-1]	Ballesteros, C	2002	Ethephon and its metabolite (RPA732569), Formulation EXP03149B (SL), North/France/2000—2 trials—Decline study, Residues in cherry (fruit). Aventis Cropscience, F-69009 Lyon, France. GLP, Unpublished
R&D/CRLD/ AN/0215013 [M-210352-01-1]	Ballesteros, C	2002	Ethephon and its metabolite (RPA732569), Formulation EXP03149B (SL), North/France/2000—2 trials—Harvest study, Residues in cherry (fruit). Aventis Cropscience, F-69009 Lyon, France. GLP, Unpublished
02 R 795 [M-220921-01-1]	Sonder, KH	2004	Residue Behaviour in Cherries, European Union (Southern Zone) 2002. Bayer CropScience GmbH, D-65926 Frankfurt, Germany. GLP, Unpublished
09-2147 [M-403958-01-1]	Uceda, L & Meilland-Berthier, I	2011	Determination of the residues of ethephon in/on cherry, sweet after spraying of Ethephon SL 480 in the field in Belgium and Netherlands. Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. GLP, Unpublished
EA950185 [M-188232-01-1]	Grolleau, G	1997	Magnitude of the Residue of Ethephon on RAC Grapes and processed Fractions after Application of CA1418 at colour-change stage. European Agricultural Services (EAS), F-69007 Lyon, France. GLP, Unpublished
RA-2562/06 [M-294217-01-1]	Billian, P, Lorenz, S & Telscher, M	2005	Determination of the residues of ethephon in/on grape after low-volume spraying of AE F016382 00 SL18 A1 (180 SL) in the field in Northern France. Bayer CropScience AG, D-40789 Monheim, Germany GLP, Unpublished
RA-2563/06 [M-294366-01-1]	Billian, P & Telscher, M	2005	Determination of the residues of ethephon in/on grape after low-volume spraying of AE F016382 00 SL18 A1 (180 SL) in the field in Southern France. Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. GLP, Unpublished
09-2176 [M-403873-01-1]	Uceda, L & Meilland-Berthier, I	2011	Determination of the residues of ethephon in/on grape after spraying, low-volume of Ethephon SL 180 in the field in France (North) and France (South). Bayer CropScience AG, D-40789 Monheim am Rhein, Germany GLP, Unpublished
102/5373/04 [M-284626-01-2	Trevizan, LRP & de Baptista, GC	2004	Determinacao de Residuos de Ethrel em Figo 1(R04MA1)/Valinhos-SP. Laboratorio de Residues de Pesticidas e Analises Cromatograficas, 13418-900 Piracicaba-SP, Brazil. Non-GLP, Unpublished
102/5374/04 [M-284634-01-2	Trevizan, LRP & de Baptista, GC	2004	Determinacao de Residuos de Ethrel em Figo 2(R04MA01-P1)/Monte-Mor-SP. Laboratorio de Residues de Pesticidas e Analises Cromatograficas, 13418-900 Piracicaba-SP, Brazil. Non-GLP, Unpublished
102/5375/04 [M-284637-01-2	Trevizan, LRP & de Baptista, GC	2004	Determinacao de Residuos de Ethrel em Figo 3(R04MA01-P2)/Caldas-SP. Laboratorio de Residues de Pesticidas e Analises Cromatograficas, 13418-900 Piracicaba-SP, Brazil. Non-GLP, Unpublished

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07 D OL BY P/A [M-352734-01-1]	Fernandez, E	2009	Residues of Ethephon in Olives and its Processed Products: Table Olives and Olive Oil (Virgin & Refined), Following Two Applications of Fruitel (480 g/L ethephon) in Tank Mix with Monopotassium Phosphate Under Field Conditions—Spain—Season 2007. Promo-Vert, E-41805 Sevilla, Spain. GLP, Unpublished
08-2053 [M-350265-02-1] (Amendment 1).	Billian, P	2009	Determination of the residues of ethephon in/on olive after spraying of Ethephon SL 480 G in the field in Spain. Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. GLP, Unpublished
CP-1997 PA-081/94 [M-188144-02-1]	Garcia, M	1994	Residue Analysis of Ethephon on Pineapple. Rhodia S.A. Research Center of Paulinia, 13140 Paulinia-SP, Brazil. Non-GLP, Unpublished
4170 [M-421140-01-1]	Guimarães, GAR	1997	Determinação Analítica de Resíduo de Ethephon em Abacaxi. Universidade Federal do Paranã, Centro de Pesquisa e Processamento de Alimentos Convenio Funpar/CEPPA, 81531-970 Paranã, Brazil. Non-GLP, Unpublished
RA-966/05 [M-284613-02-1	Galhiane, MS & Santos, L de S	2005	Relatorio de Estudo de Residuo de Ethrel (Ethephon) em Abacaxi (Analises Realizadas em Fruto). Laboratorio de Quimica Analitica e Cromatografia, 17.033-360 Bauru/SP, Brazil. Non-GLP, Unpublished
RA-967/05 [M-284618-02-1	Galhiane, MS & Santos, L de S	2005	Relatorio de Estudo de Residuo de Ethrel (Ethephon) em Abacaxi (Analises Realizadas em Fruto). Laboratorio de Quimica Analitica e Cromatografia, 17.033-360 Bauru/SP, Brazil. Non-GLP, Unpublished
RA-968/05 [M-284623-02-1	Galhiane, MS & Santos, L de S	2005	Relatorio de Estudo de Residuo de Ethrel (Ethephon) em Abacaxi (Analises Realizadas em Fruto). Laboratorio de Quimica Analitica e Cromatografia, 17.033-360 Bauru/SP, Brazil. Non-GLP, Unpublished
R&D/CRLD/ AN/msa/ 9816197 [M-165714-01-1]	Maestracci, M	1998	Ethephon, Formulation EXP03149B (SL), Trials Costa Rica 1998, Residues in pineapple, Decline study. Rhône-Poulenc Agro, F-69009 Lyon, France. GLP (analytical), non-GLP (field), Unpublished
R&D/CRLD/ AN/msa/ 9816152 [M-165702-02-1]	Maestracci, M	1998	Ethephon, Formulation EXP03149B (SL), Trials Ivory Coast 1997–1998, Residues in pineapple, Decline study. Rhône-Poulenc Agro, F-69009 Lyon, France. GLP (analytical), non-GLP (field), Unpublished
R&D/CRLD/ AN/mr/9916533 [M-179309-01-1]	Baudet, L	1999	Ethephon, Formulation EXP03149B (SL), South/Ivory Coast /1998–1999–1 Decline study trial, Residues in pineapple (flesh and skin). Rhône-Poulenc Agro, F-69009 Lyon, France. GLP (analytical), non-GLP (field), Unpublished 10 November 1999
USA89E27 [M-187578-01-1]	Nygren, RE	1992	Ethrel/Pineapple/Residue. Non-GLP, Unpublished
DR 00 EUS 522 [M-203527-01-1]	Hees, M	2001	Residue Study in industrial field tomatoes European Union [southern zone] 2000, ethephon, AE F016382, water soluble concentrate (SL) 480 g/L. Aventis Cropscience, D-65926 Frankfurt, Germany GLP, Unpublished
01R773 [M-215341-01-1]	Davies, P	2002	Decline of residues in tomatoes, European Union Southern zone 2001, ethephon, AE F016382 watersoluble concentrate (SL) 39.67 % w/w (480 g/L). Bayer CropScience GmbH, D-65926 Frankfurt, Germany GLP, Unpublished
RA-2065/04 [M-261821-01-1]	Bardel, P	2005	Determination of the Residues of Ethephon and HEPA in/on Tomato after Spraying of AE F016382 00 SL40 A1 (480 SL) in the Field in Spain, Portugal and Italy. Bayer CropScience AG, D-40789 Monheim, Germany GLP, Unpublished

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DR 00 EUI 520 [M-202477-01-1]	Hees, M	2001	Residues at harvest in protected tomatoes European Union [indoor] 2000, ethephon, AE F016382, water soluble concentrate (SL) 480 g/L. Aventis Cropscience, D-65926 Frankfurt, Germany GLP, Unpublished
R&D/CRLD/ AN/0215069 [M-210410-01-1]	Ballesteros, C	2002	Ethephon and its metabolite (RPA732569), Formulation EXP03149B (SL), Greenhouse/The Netherlands/1999—2 trials—Harvest study, Residues in tomato (fruit). Aventis Cropscience, F-69009 Lyon, France. GLP, Unpublished
01R791 [M-210553-01-1]	Davies, P	2002	Decline of residues in protected tomatoes, European Union Indoors 2001, ethephon, AE F016382 watersoluble concentrate (SL) 39.67% w/w (480 g/L). Bayer CropScience GmbH, D-65926 Frankfurt, Germany. GLP, Unpublished
USA89E30 [M-187599-01-1]	Nygren, RE	1991	ETHREL/Tomato/Residues. Rhône-Poulenc Ag Company, NC 27709, USA. GLP, Unpublished
USA90E16 [M-187596-01-1]	Nygren, RE	1992	ETHREL/Tomato/Magnitude of residue study. Rhône-Poulenc Ag Company, NC 27709, USA. GLP, Unpublished
USA91E16 [M-187891-01-1]	Nygren, RE	1995	ETHREL® brand plant regulator/Tomato/magnitude of Residue. Rhône-Poulenc Ag Company, NC 27709, USA. GLP, Unpublished
IR-4 PR No 00250 [M-301374-01-1]	Dorschner, K	2008	Ethephon: Magnitude of the Residue on Tomato (Greenhouse) IR-4 Project, Rutgers, The State University of New Jersey, NJ 08540, USA. GLP, Unpublished
DR 00 EUS 525 [M-199982-01-1]	Hees, M	2001	Residue Study in Barley, European Union [southern zone] 2000, Ethephon, Water soluble concentrate, 480 g/L. Aventis CropScience, D-65926 Frankfurt, Germany. GLP, Unpublished
R&D/CRLD/ AN/mr/0115430 [M-208093-01-1]	Ballesteros, C	2001	Ethephon and its metabolite (RPA732569), Formulation EXP03725B (SL), South/France/ 2000—2 Decline study trials, Residues in winter barley (plant, straw and grain). Aventis CropScience, F-69009 Lyon, France. GLP, Unpublished
01R761 [M-209901-01-1]	Davies, P	2002	Residue behaviour in barley, European Union Northern zone 2001, ethephon, AE F016382, water soluble concentrate (SL), 39.83 % w/w (480 g/L). Aventis CropScience, D-65926 Frankfurt, Germany. GLP, Unpublished
01R771 [M-210307-01-1]	Davies, P	2002	Residue behaviour in barley, European Union Southern zone 2001, ethephon, AE F016382, water soluble concentrate (SL), 39.83 % w/w (480 g/L). Aventis CropScience, D-65926 Frankfurt, Germany. GLP, Unpublished
RA-2094/04 [M-249305-02-1]	Report: Bardel, P & Wolters, A Amendment 1: Bardel, P	2005	Determination of the Residues of Ethephon and Chlormequat chloride in/on Spring Barley after Spraying of AE F080286 02 SL40 A1 in the Field in Northern France, Sweden and Germany. Bayer CropScience AG, D-40789 Monheim, Germany. GLP, Unpublished
RA-2095/04 [M-251234-01-1]	Bardel, P & Wolters, A	2005	Determination of the Residues of Ethephon and Chlormequat chloride in/on Spring Barley after Spraying of AE F080286 02 SL40 A1 in the Field in Southern France, Italy and Portugal. Bayer CropScience AG, D-40789 Monheim, Germany. GLP, Unpublished
RA-2093/04 [M-251235-01-1]	Bardel, P & Wolters, A	2005	Determination of the Residues of Ethephon and Chlormequat chloride in/on Winter Barley and Spring Barley after Spraying of AE F080286 02 SL40 A1 in the Field in Greece, Italy, Southern France and Spain. Bayer CropScience AG, D-40789 Monheim, Germany. GLP, Unpublished
RA-2092/04 [M-251366-01-1]	Bardel, P & Wolters, A	2005	Determination of the Residues of Ethephon and Chlormequat Chloride in/on Winter Barley after Spraying of AE F080286 02 SL40 A1 in the Field in Sweden, Germany and Northern France. Bayer CropScience AG, D-40789 Monheim, Germany. GLP, Unpublished
RA-2519/06 [M-290151-01-1]	Billian, P & Erler, S	2007	Determination of the residues of ethephon and chlormequat chloride in/on winter barley after spraying of Ethephon & AEF080286 (450 SL) in the field in Southern France. Bayer CropScience AG, D-40789 Monheim, Germany. GLP, Unpublished
RA-2515/06 [M-294373-01-1]	Billian, P & Telscher, M	2007	Determination of the residues of ethephon in/on winter barley after spraying of AE F016382 00 SL40 A2 (480 SL) in the field in Northern France, the United Kingdom and Germany. Bayer CropScience AG, D-40789 Monheim, Germany. GLP, Unpublished

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RA-2516/06 [M-294780-02-1	Report: Billian, P & Telscher, M Amendment 1: Billian, P	Report: 2007 Amend-ment 1: 2010	Determination of the residues of ethephon in/on winter rye after spraying of AE F016382 00 SL40 A2 (480 SL) in the field in Northern France, the United Kingdom, Sweden and Germany. Bayer CropScience AG, D-40789 Monheim, Germany. GLP, Unpublished
RA-2574/07 [M-318501-01-1]	Billian, P, Erler, S & Wolters, A	2008	Determination of the residues of Ethephon in/on winter rye after spraying of AE F016382 00 SL40 A2 (480 SL) in the field in northern France, Germany and the United Kingdom. Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. GLP, Unpublished
R&D/CRLD/ AN/mr/0115433 [M-208087-01-1]	Ballesteros, C	2002	Ethephon and its metabolite (RPA732569), Formulation EXP03725B (SL), South/France/2000—2 trials—Decline study, Residues in soft winter wheat (plant, straw and grain). Aventis CropScience, F-69009 Lyon, France. GLP, Unpublished
R&D/CRLD/ AN/mr/0115434 [M-208091-01-1]	Ballesteros, C	2002	Ethephon and its metabolite (RPA732569), Formulation EXP03725B (SL), South/France/2000—2 Harvest trials, Residues in soft winter wheat (straw and grain). Aventis CropScience, F-69009 Lyon, France. GLP, Unpublished
01R762 [M-210306-01-1]	Davies, P	2002	Residue behaviour in common wheat, European Union Northern zone 2001, ethephon, (AE F016382), water soluble concentrate (SL) 480 g/L. Aventis CropScience, D-65926 Frankfurt, Germany. GLP, Unpublished
01R772 [M-210308-01-1]	Davies, P	2002	Residue behaviour in wheat, European Union Southern zone 2001, ethephon, (AE F016382), water soluble concentrate (SL), 39.83 % w/w (480 g/L). Aventis CropScience, D-65926 Frankfurt, Germany. GLP, Unpublished
RA-2090/04 [M-251226-01-1]	Bardel, P	2005	Determination of the Residues of Ethephon and Chlormequat Chloride in/on Wheat after Spraying of AE F080286 02 SL40 A1 in the Field in Sweden, Germany and Northern France. Bayer CropScience AG, D-40789 Monheim, Germany. GLP, Unpublished
RA-2091/04 [M-251236-02-1 (Amendment 2)	Bardel, P	2005	Determination of the Residues of Ethephon and Chlormequat Chloride in/on Wheat and Wheat, hard after Spraying of AE F080286 02 SL40 A1 in the Field in Greece, Southern France, Spain and Portugal. Bayer CropScience AG, D-40789 Monheim, Germany. GLP, Unpublished
RA-2517/06 [M-294528-01-1]	Billian, P & Telscher, M	2007	Determination of the residues of ethephon in/on winter wheat after spraying of AE F016382 00 SL40 A2 (480 SL) in the field in Northern France, the United Kingdom and Germany. Bayer CropScience AG, D-40789 Monheim, Germany. GLP, Unpublished
RA-2575/07 [M-312007-01-1]	Billian, P	2008	Determination of the residues of ethephon in/on winter wheat after spraying of AE F016382 00 SL40 A2 (480 SL) in the field in northern France and Germany. Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. GLP, Unpublished
10223 [M-187972-01-1]	Harrison, SL	1981	Residues of Ethephon in wheat and barley resulting from applications of Ethrel® as an anti-lodging agent. Union Carbide Agricultural Products Company Inc., North Carolina, USA. Non-GLP, Unpublished
SARS-89-24 [M-187553-01-1]	Conn, RL	1992	Magnitude of the Residues of Ethephon and Monochloroacetic Acid (MCAA) in or on Wheat. Stewart Pesticide Registration Associates, Inc., Virginia 22202, USA. GLP, Unpublished
R&D/CRLD/ AN/bd/9515891 [M-163122-01-1]	Richard, M & Muller, MA	1995	RPA090946 or Cyclanilide Ethephon Formulation EXP31039A (SC) Greece 1993 Residues in Cotton (seed) Rhône-Poulenc Agrochimie, F-69263 Lyon, France. Non-GLP (field phase), GLP (analytical phase), Unpublished
R&D/CRLD/ AN/bd/9515911 [M-163133-01-1]	Richard, M & Muller, MA	1995	RPA090946 or Cyclanilide Ethephon Formulation EXP31039A (SC) Spain 1994 Residues in Cotton (fibre, seed) Decline study Rhône-Poulenc Agrochimie, F-69263 Lyon, France. GLP, Unpublished
R&D/CRLD/ AN/bd/9516706 [M-163236-01-1]	Muller, MA	1996	RPA090946 or Cyclanilide–Ethephon Formulation EXP31039A (SC) Trial Spain 1995 Residues in Cotton (seed and fibre) Rhône-Poulenc Secteur Agro, F-69009 Lyon, France. GLP, Unpublished

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R&D/CRLD/ AN/vg/9516705 [M-163240-01-1]	Muller, MA	1996	RPA090946 or Cyclanilide–Ethephon Formulation EXP31039A (SC) Trials Greece 1995 Residues in Cotton (seed) Rhône-Poulenc Secteur Agro, F-69009 Lyon, France. GLP, Unpublished
08-2023 [M-360139-01-1]	Billian, P, Reineke, A & Krusell, L	2009	Determination of the residues of cyclanilide and ethephon in/on cotton after spraying of FINISH SC 540 in the field in Greece and Spain. Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. GLP, Unpublished
USA89I03 [M-187602-01-1]	Nygren, RE	1991	PREP/Cotton/Residues. Rhône-Poulenc Ag Company, NC 27709, USA. Non-GLP, Unpublished
USA93I03R [M-252199-01-1]	See, RM	1994	Magnitude of RPA-90946 and Ethephon Residues in/on Seed Cotton Resulting from Foliar Applications of 31039B, 1993. Rhône-Poulenc Ag Company, NC 27709, USA. GLP, Unpublished
USA94I01R [M-253436-01-1]	See, RM	1995	Magnitude of RPA-90946 and Ethephon Residues in/on Seed Cotton Resulting from Foliar Application of 31039B, 1994. Rhône-Poulenc Ag Company, NC 27709, USA. GLP, Unpublished
CP-2466/97 [M-188222-01-1]	Garcia, M	1997	Residue Analysis of Ethephon on Cotton. Rhodia S.A. Research Center of Paulinia, 13.140.000 Paulinia-SP, Brazil. Non-GLP, Unpublished
CP-2435/97 [M-253467-02-1] and M-253467-02-1]	Garcia, M, & de Oliverira, NT	1997	Residue Analysis of Ethephon and Cyclanilide on Cotton. Rhodia S.A. Research Center of Paulinia, 13.140.000 Paulinia-SP, Brazil. Non-GLP, Unpublished
CP-2436/97 [M-253470-02-1] and M-253470-02-1]	Garcia, M & de Oliverira, NT	1997	Residue Analysis of Ethephon and Cyclanilide on Cotton. Rhodia S.A. Research Center of Paulinia, 13.140.000 Paulinia-SP, Brazil. Non-GLP, Unpublished
RA-218/06 [M-285068-01-2	Galhiane, MS & Santos, L de S	2006	Relatorio de Estudo de Resíduo de Finish (Etefon + Cyclanilide) em Algodao (Analises Realizadas em Sementes sem Fibras). Laboratorio de Quimica Analitica e Cromatografia, 17.033-360 Bauru/SP, Brazil. Non-GLP, Unpublished
RA-219/06 [M-285070-01-2]	Galhiane, MS & Santos, L de S	2006	Relatorio de Estudo de Resíduo de Finish (Etefon + Cyclanilide) em Algodao (Analises Realizadas em Sementes sem Fibras). Laboratorio de Quimica Analitica e Cromatografia, 17.033-360 Bauru/SP, Brazil. Non-GLP, Unpublished
RA-220/06 [M-285073-01-2]	Galhiane, MS & Santos, L de S	2006	Relatorio de Estudo de Resíduo de Finish (Etefon + Cyclanilide) em Algodao (Analises Realizadas em Sementes sem Fibras). Laboratorio de Quimica Analitica e Cromatografia, 17.033-360 Bauru/SP, Brazil. Non-GLP, Unpublished
RA-221/06 [M-285075-01-2]	Galhiane, MS & Santos, L de S	2006	Relatorio de Estudo de Resíduo de Finish (Etefon + Cyclanilide) em Algodao (Analises Realizadas em Sementes sem Fibras). Laboratorio de Quimica Analitica e Cromatografia, 17.033-360 Bauru/SP, Brazil. Non-GLP, Unpublished
CP02/001 [M-211072-01-1]	Selzer, J	2002	Ethephon: Investigation of the Nature of the Potential Residue in the Products of Industrial Processing or Household Preparation. Aventis Cropscience, D65629 Frankfurt am Main, Germany. GLP; Unpublished
USA89E32 [M-187583-01-1]	Nygren, RE	1990	Ethrel Apple 1989 Residue Program. Rhône-Poulenc Ag Company, NC 27709, USA. Non-GLP, Unpublished
RA-3610/03 [M-254102-01-1]	Bardel, P, Hoffmann, M & Eberhardt, R	2005	Determination of the Residues of Ethephon in/on Apple (Fruit, Juice, Sauce, Pomace) after Spraying of AE F016382 00 SL40 A1 (480 SL) in Italy, Portugal and Spain. Bayer CropScience AG, D-40789 Monheim, Germany. GLP, Unpublished
M-188057-01-1	Harrison, SL	1979	Residues of Ethephon in grapes and related Foods and Feeds. Amchem Products, Inc., USA. Non-GLP, Unpublished
EA950185 [M-188232-01-1]	Grolleau, G	1997	Magnitude of the Residue of Ethephon in RAC Grapes and Processed Fractions after Application of CA1418 at colour-change stage. European Agricultural Services (EAS), F-69007 Lyon, France. GLP, Unpublished
RA-3680/03 [M-249278-02-1]	Report: Bardel, P, & Hoffmann, M Amendment 1: Schulte, G	Report: 2005a Amend-ment 1: 2013	Determination of the Residues of Ethephon in/on Grape (Juice, Pomace, Must and Wine) after Spraying of AE F016382 00 SL18 A1 (180 SL) in the Field in Germany and Northern France. Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. GLP, Unpublished

Code	Author(s)	Year	Title, Institute, Report reference
RA-3681/03 [M-249332-02-1	Report: Bardel, P & Hoffmann, M Amendment 1: Schulte, G	Report: 2005b Amend-ment 1: 2013	Determination of the Residues of Ethephon in/on Grape (Juice, Pomace, Must and Wine) after Spraying of AE F016382 00 SL18 A1 (180 SL) in the Field in Greece and Southern France. Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. GLP, Unpublished
07 D OL BY P/A [M-352734-01-1]	Fernandez, E	2009	Residues of Ethephon in Olives and its Processed Products: Table Olives and Olive Oil (Virgin and Refined), Following Two Applications of Fruitel (480 g/L ethephon) in Tank Mix with Monopotassium Phosphate under Field Conditions—Spain—Season 2007. Promo-Vert, E-41805, Spain. GLP, Unpublished
R&D/CRLD/ AN/msa/ 9816197 [M-165714-01-1]	Maestracci, M	1998	Ethephon, Formulation EXP03149B (SL), Trials Costa Rica 1998, Residues in pineapple, Decline study. Rhône-Poulenc Agro, F-69009 Lyon, France. GLP (analytical), non-GLP (field), Unpublished
R&D/CRLD/ AN/msa/ 9816152 [M-165702-02-1	Maestracci, M	1998	Ethephon, Formulation EXP03149B (SL), Trials Ivory Coast 1997–1998, Residues in pineapple, Decline study. Rhône-Poulenc Agro, F-69009 Lyon, France. GLP (analytical), non-GLP (field), Unpublished
R&D/CRLD/ AN/mr/9916533 [M-179309-01-1]	Baudet, L	1999	Ethephon, Formulation EXP03149B (SL), South/Ivory Coast /1998–1999—1 Decline study trial, Residues in pineapple (flesh and skin). Rhône-Poulenc Agro, F-69009 Lyon, France. GLP (analytical), non-GLP (field), Unpublished
USA89E30 [M-187599-01-1]	Nygren, RE	1991	Ethrel/Tomato/Residues. Rhône-Poulenc Ag Company, NC 27709, USA. Non-GLP, Unpublished
Industria Conserve, *60*, 1985, pp 183 [M-188387-01-1]	Bolzuni, L & Leoni, C	1985	Residui di Ethephon nel Pomodor Fresco e nel Concentrato di Pomodoro (Ethephon Residues in Fresh Tomatoes and Tomato paste). Industria Conserve, *60*, 1985, pp 183 Non-GLP, Published
RA-3065/04 [M-262300-01-1]	Bardel, P	2005	Determination of the Residues of Ethephon and HEPA in/on Tomato Processed Commodities after Spraying of AE F016382 00 SL40 A1 (480 SL) in the Field in Spain, Portugal and Italy. Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. GLP, Unpublished
SARS-90-24P [M-187550-01-1]	Conn, RL	1992	Magnitude of the Residue of Ethephon on the Processed Fractions of Wheat. Stewart Agricultural Research Services, Inc., MO 63552, USA. GLP, Unpublished
866R11 [M-187977-01-1]	Nygren, RE	1985	Ethephon Residues in Mill Fractions of Treated Wheat Grain. Union Carbide Agricultural Products Company, Inc., North Carolina, USA. Non-GLP, Unpublished
10223 [M-187972-01-1]	Harrison, SL	1981	Residues of Ethephon in Wheat and Barley Resulting from Applications of Ethrel® as an Anti-Lodging Agent. Union Carbide Agricultural Products Company, Inc., North Carolina, USA. Non-GLP; Unpublished
USA93I04R [M-203874-01-2]	Lee, RE	1994	Magnitude of RPA-90946 In/On Cotton Seed and Seed Processing Fractions Resulting From Foliar Applications of 31039B, 1993. Rhône-Poulenc Ag Company, NC 27709, USA. GLP, Unpublished
08-3401 [M-367885-01-1]	Billian, P & Krusell, L	2010	Determination of the residues of cyclanilide and ethephon in/on cotton and processed fractions (extracted meal; crude oil; crude oil, pre-clarified; crude oil, neutralized and oil, refined) after spraying of FINISH SC 540 in the Field in Greece and Spain. Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. GLP, Unpublished
96E08334 [M-188195-01-1]	Wells-Knecht, MC	1996	Ethephon: Magnitude of Residues in Milk and Tissues of Lactating Dairy Cows Rhône-Poulenc Ag Company, NC 27709, USA. GLP, Unpublished
VC070001-06 [M-295429-01-1]	Mackenzie, E	2007	Ethephon—The potential for HEPA residues in ruminants. Battelle UK Ltd., Essex, CM5 0GZ, UK. Non-GLP, Unpublished
96E08335 [M-188192-01-1]	Wells-Knecht, MC	1996	Ethephon: Magnitude of Residues in Tissues and Eggs of Laying Hens Rhône-Poulenc Ag Company, NC 27709, USA. GLP, Unpublished
MR-14/100	Schulte, D & Druskus, M	2015	Validation of the analytical method 01429 for the determination of ethephon and HEPA (2-hydroxyethylphosphonic acid) in/on cereals (green material, straw and grain) by HPLC-MS/MS, Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. GLP, Unpublished

Code	Author(s)	Year	Title, Institute, Report reference
Report: 13-2027 [M-526906-01-1]	Schulte, D & Berkum, S	2015	Determination of the residues of ethephon in/on winter barley after spray application of Ethephon SL 480 in Germany, Belgium, the Netherlands and the United Kingdom Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. GLP, Unpublished
Report:13-2028	Schulte, D & Berkum, S	2015	Determination of the residues of ethephon in/on winter barley after spray application of Ethephon SL 480 in southern France, Spain and Italy Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. Non-GLP, Unpublished
Report:13-2029 [M-529493-01-1]	Schulte, D & Berkum, S	2015	Determination of the residues of ethephon in/on soft wheat after spray application of Ethephon SL 480 in Germany, Belgium and the United Kingdom Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. Non-GLP, Unpublished
Report:13-2030 [M-529488-01-1]	Schulte, D & Berkum, S	2015	Determination of the residues of ethephon in/on soft wheat after spray application of Ethephon SL 480 in southern France, Spain and Italy Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. Non-GLP, Unpublished
Report:14-2018 [M-532267-01-1]	Schulte, D & Berkum, S	2015	Determination of the residues of ethephon in/on winter wheat after spray application of Ethephon SL 480 in Germany, the United Kingdom, northern France and the Netherlands Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. Non-GLP, Unpublished
Report:14-2019 [M-532272-01-1]	Schulte, D & Berkum, S	2015	Determination of the residues of ethephon in/on winter wheat after spray application of Ethephon SL 480 in southern France, Spain, Italy and Portugal Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. Non-GLP, Unpublished
Report:14-2020 [no M number was provided]	Schulte, D & Berkum, S	2015	Determination of the residues of ethephon in/on winter barley after spray application of Ethephon SL 480 in southern France, Spain, Italy and Greece Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. Non-GLP, Unpublished
Report: 14-2022 [M533473-01-1]	Schulte, D & Berkum, S	2015	Determination of the residues of ethephon in/on winter barley after spray application of Ethephon SL 480 in Germany, northern France and the United Kingdom Bayer CropScience AG, D-40789 Monheim am Rhein, Germany. Non-GLP, Unpublished

FLONICAMID (282)

The first draft was prepared by Ms Monique Thomas, Pest Management Regulatory Agency, Canada

EXPLANATION

Flonicamid is a new insecticide for control of aphids and other sucking insects. It belongs to a new class of chemistry known as pyridinecarboxamide. Flonicamid has been registered in Canada since 2009. At the 46th Session of the CCPR (2014), flonicamid was scheduled for evaluation as a new compound by 2015 JMPR.

The Meeting received information on the metabolism of flonicamid in peaches, Bell peppers, potatoes, wheat, lactating goats, laying hens and rotational crops, environmental fate, methods of residue analysis, freezer storage stability, GAP, supervised residue trials on various fruits, vegetables, tree nuts, oil seeds, dried hops, mint and tea, processing studies, as well as livestock feeding studies.

IDENTITY

ISO common name: Flonicamid

Chemical name:

IUPAC: *N*-cyanomethyl-4-(trifluoromethyl)nicotinamide

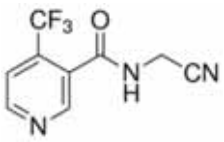
CAS: *N*-(cyanomethyl)-4-(trifluoromethyl)-3-pyridinecarboxamide

CAS Registry. No.: 158062-67-0

CIPAC No.: 763

Trade Name: IKI-220

Structural formula:



Molecular formula: $C_9H_6F_3N_3O$

Molecular weight: 229.16 g/mol

Physical and chemical properties

Property	Findings		Report, Reference
Pure Active Ingredient			
Melting Point	157.5 ºC		010153-1
Mean Relative Density (20 ºC)	1.54 g/mL		
Physical State, colour	solid powder, off white		
Odour	odourless		
Vapour Pressure	Temperature, ºC	Pa	010341-1
	20	9.43×10^{-7} (extrapolated)	

Property	Findings		Report, Reference
	25	2.55×10^{-6} (extrapolated)	
	30	6.48×10^{-6} (experimental)	
	40.1	4.40×10^{-5} (experimental)	
	50.1	2.31×10^{-4} (experimental)	
Solubility in water	5.2 g/L at 20 ºC		010251-1
Solubility in organic solvents (20 ºC)	Solvent	g/L	
	Acetone	163.5	
	Ethyl Acetate	34.2	
	Methanol	104.3	
	Dichloromethane	4.5	0.10250-1
	Toluene	0.55	
	Hexane	0.0002	
	n-Octanol	3	
	Acetonitrile	132.8	
	Isopropyl Alcohol	18.7	
Partition coefficient	1.9 (Log P_{ow} = 0.3) at 29.8 ºC		010252-1
Hydrolysis rate at 25 ºC		DT_{50} (days)	
	pH 5	no hydrolysis	
	pH 7	no hydrolysis	
	pH 9	204 (max. 31% TFNG-AM)	008076-2
at 50 ºC	pH 4	no hydrolysis	
	pH 5	no hydrolysis	
	pH 7	578 (no major degradation products)	
	pH 9	9 (max. 65% TFNG-AM, max. 86% TFNG)	
at 40 ºC	pH 9	17 (max. 63% TFNG-AM, max. 26% TFNG)	
Flonicamid is stable at pH 4 and pH 5. The amide TFNG-AM is formed from this reaction (under alkaline conditions) and can then be hydrolyzed to TFNG.			
Quantum yield	DT_{50} at pH 7 and 23 ºC was 267 days. flonicamid did not degrade in dark controls. The major degradate TFNA-AM, only degraded slightly (from 2.4% at time 0 and increased slightly to 2.9% by Day 15)		011050-1
Dissociation constant	11.60 ± 0.03 in 5% ethanol/water at 20 ± 1 ºC		010141-1
Flammability	Not flammable		20334
Auto-flammability	No relative self-ignition temperature		
Explosive properties	Flonicamid is not a potential explosive and does not have a potential for rapid energy release (decomposition energy = 374 J/g)		
Technical material			
Physical State, colour	Solid powder, light beige		
pH	4.5 at 25 ºC		
Mean Relative Density (20 ºC)	1.531 g/mL		012575-1
Odour	Odourless		
Solubility in organic solvents (20 ºC)	Solvent	g/L	
	Acetone	157.1	
	Ethyl Acetate	34.9	
	Methanol	89	
	Dichloromethane	4	011201-1
	Toluene	0.3	
	Hexane	0.0003	
	n-Octanol	2.6	
	Acetonitrile	111.4	
	Isopropyl Alcohol	14.7	

Formulation

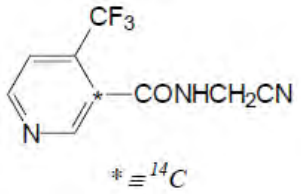
Flonicamid is commercially marketed as a soluble or wettable granule containing 50% flonicamid.

Specification

Flonicamid has not been evaluated by the FAO/WHO Joint Meeting of Pesticide Specifications (JMPS).

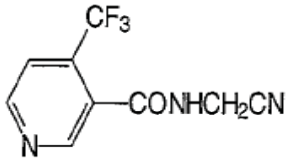
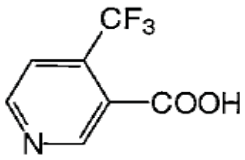
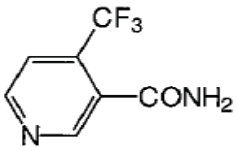
METABOLISM AND ENVIRONMENTAL FATE

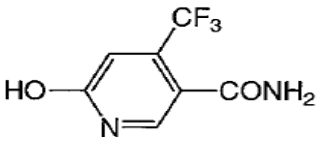
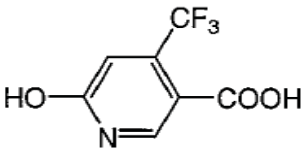
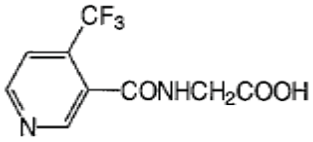
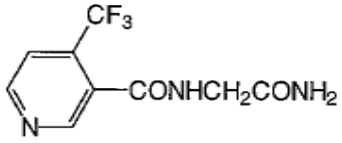
The metabolism and distribution of flonicamid in plants and animals was investigated using ^{14}C-labelled test material as shown below:



Chemical names, structures and code names of metabolites and degradation products of flonicamid are summarized in the following table. Compounds are referred to primarily by the code name.

Code names, chemical names and structures of flonicamid related substances

Code Name	Structure	Chemical Name	Occurrence
Flonicamid IKI-220		*N*-cyanomethyl-4-(trifluoromethyl)nicotinamide	Goat Hen Peach Peppers Potato Wheat Rotational crop (wheat) Soil
TFNA		4-trifluoromethylnicotinic acid	Goat Hen Peach Peppers Potato Wheat Rotational crop (wheat) Soil
TFNA-AM		4-trifluoromethylnicotinamide	Goat Hen Peach Peppers Potato Wheat Rotational crop (wheat)

Code Name	Structure	Chemical Name	Occurrence
OH-TFNA-AM		6-hydroxy-4-trifluoromethylnicotinamide	Goat Hen
TFNA-OH		6-hydroxy-4-trifluoromethylnicotinic acid	Rotational crop (wheat)
TFNG		*N*-(4-trifluoromethylnicotinoyl)glycine	Peach Peppers Potato Wheat Rotational crop (wheat) Soil
TFNG-AM		*N*-(4-trifluoromethylnicotinoyl)glycinamide	Hen Peach Peppers Wheat Rotational crop (wheat)

Plant metabolism

The Meeting received information on the fate of flonicamid radio-labelled at the 3 position of the pyridine ring following foliar application to peaches, Bell peppers, potatoes and wheat (immature and mature).

Peach

Flonicamid, radio-labelled at the 3 position of the pyridine ring (specific activity: 1.67 MBq/mg), formulated as a wettable granule formulation, was applied to single peach trees (variety: Elberta), grown outdoors in individual 1.4 m^2 plots of clay loam. Each tree received two foliar applications, at a 14-day re-treatment interval, at rates of 100 g ai/ha (low rate) or 500 g ai/ha (high rate) per application, resulting in total seasonal rates of 200 g ai/ha or 1000 g ai/ha. Mature fruits and leaves were harvested 21 days following the second application.

To remove surface residues the fruit was washed with deionised water. Following the removal of pits, the surface-washed fruits were then cut into small pieces and homogenised. The homogenates were then centrifuged to give an aqueous fraction (juice) and a solid fraction (pomace). The juice was decanted, total volume measured and the radioactivity measured. The remaining pomace was weighed and then ground with dry ice in a blender.

Radioactivity was measured by liquid scintillation counting (LSC). Dry-ice-ground peach leaves, pomace and PES were combusted in an oxidizer (Table 1).

Overall total radioactive residues (TRRs) in fruits at the low rate and the high rate were 0.10 mg eq/kg and 0.32 mg eq/kg, respectively, while in the leaves, TRRs were higher than those of fruits, 6.24 mg eq/kg at the low rate and 24.21 mg eq/kg at the high rate.

Table 1 TRRs in peach fruits and leaves

Crop part	TRRs (mg eq/kg)	
	Low rate (200 g ai/ha)	High rate (1000 g ai/ha)
Fruits	0.10	0.32
Leaves	6.24	24.21

Subsamples of both pomace and leaves were extracted twice with acetonitrile:water:phosphoric acid (40:60:0.1, v/v/v). The extracts of each subsample were pooled and the PES were air dried. Quantification and characterization/identification of residues were done by HPLC. To determine the ^{14}C residue profiles, fractions were collected from the HPLC effluent and analysed by LSC. Flonicamid and metabolites were isolated and purified from peach juice and leaf extracts of both treatment groups. The isolated radioactive components were purified by reverse phase HPLC. The identification was supported by other methods such as TLC and LC-MS.

Table 2 Distribution of TRRs in mature fruit harvested 21 days following application at low rate and high rate

Fraction	Low Rate (200 g ai/ha)		High Rate (1000 g ai/ha)	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Surface wash	0.006	5.6	0.05	15.3
Flonicamid	0.006	2.7	0.03	8.6
TFNG	0.000	0.2	0.001	0.3
TFNA	0.000	0.3	0.001	0.3
TFNG-AM	0.000	0.2	0.003	0.9
TFNA-AM	0.000	0.2	0.003	0.8
Unknowns	0.000	0.4	0.006	1.9
Polar	0.000	0.2	0.001	0.3
Nonpolar	0.001	1.0	0.007	2.0
Diffuse Radioactivity	0.001	0.6	0.001	0.2
Juice (aqueous fraction)	0.073	73.2	0.205	63.7
Extracted	0.073	73.2	0.205	63.7
Flonicamid	0.020	20.3	0.13	40.2
TFNG	0.005	5.0	0.01	3.0
TFNA	0.040	39.9	0.04	12.9
TFNG-AM	0.001	1.2	0.006	1.8
TFNA-AM	0.001	1.1	0.005	1.6
Unknowns	0.003	3.2	0.007	2.1
Polar	0.002	2.0	0.005	1.5
Nonpolar	–	–	0.001	0.3
Diffuse Radioactivity	0.001	0.6	0.000	0.1
Pomace (solid fraction)	0.021	21.1	0.067	21.0
Extracted	0.019	19.5	0.06	19.1
Flonicamid	0.007	7.1	0.04	11.8
TFNG	0.001	0.8	0.003	1.0
TFNA	0.009	9.0	0.14	4.2
TFNG-AM	0.000	0.3	0.001	0.4
TFNA-AM	0.000	0.3	0.001	0.4
Unknowns	0.001	1.4	0.003	0.9
Polar	0.000	0.4	0.000	0.1
Nonpolar	0.000	0.1	0.000	0.1
Diffuse Radioactivity	0.000	0.1	0.000	0.0
Nonextracted	0.002	1.6	0.006	1.9
Total	0.10	100.0	0.322	100.0

Table 3 Identification/Characterization of TRRs in whole fruit harvested 21 days following application at low rate and high rate

Analyte	Low Rate (200 g ai/ha)		High Rate (1000 g ai/ha)	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Flonicamid	0.033	30.1	0.20	60.6
TFNG	0.006	6.0	0.14	4.3
TFNA	0.049	49.2	0.55	17.4
TFNG-AM	0.001	1.7	0.01	3.1
TFNA-AM	0.001	1.6	0.009	2.8

Analyte	Low Rate (200 g ai/ha)		High Rate (1000 g ai/ha)	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Total Identified	0.09	88.6	0.91	88.2
Total Unidentfied [a]	0.006	6.3	0.017	5.2
Total Characterized [b]	0.003	3.7	0.014	4.3
Total Extracted [c]	0.098	98.3	0.315	98.1
Total Nonextracted	0.002	1.6	0.006	1.9
Total	0.10	100.0	0.322	100.0

[a] Total unidentified = Unknowns + Diffuse Radioactivity

[b] Total Characterized = Polar + Nonpolar

[c] Total extracted = Surface wash + Juice extracted + Pomace extracted

Table 4 Distribution of TRRs in mature leaves harvested 21 days following application at low rate and high rate

	Low Rate (200 g ai/ha)		High Rate (1000 g ai/ha)	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Extracted	5.58	89.2	22.27	92.0
Flonicamid	2.05	32.9	15.72	64.9
TFNG	1.21	19.3	2.06	8.5
TFNA	0.99	15.8	1.28	5.3
TFNG-AM	0.21	3.4	0.40	1.6
TFNA-AM	0.25	4.1	0.48	2.0
Unknowns	0.18	2.8	0.18	0.7
Polar	0.39	6.3	0.92	3.8
Nonpolar	0.29	4.6	1.24	5.1
Diffuse Radioactivity	0.001	0.0	0.002	0.0
Unextracted	0.67	10.8	1.94	8.0
Total	6.25	100.0	24.21	100.0

Representative samples of peach fruit fractions (surface wash, pomace and juice) were extracted and analysed immediately after collection and re-analysed after 5 months of storage. Considering the metabolite profiles from the initial and final analyses were very similar, flonicamid and the metabolites were stable during this storage interval.

According to Table 2, the surface wash removed very little radioactivity, 6–15%, demonstrating limited penetration. The majority of the radioactivity in peach fruits was partitioned in the juice fraction, accounting for 64–73% of the TRR while the radioactivity in pomace represented 21% of the TRR.

While juice was not further extracted with organic solvents, extraction of the pulp with acetonitrile:water:phosphoric acid recovered 92% TRR. When treated at the low rate, flonicamid (30.1% of the TRR). and TFNA (49.2% of TRR) were the predominant residues. In peaches treated at the high rate, flonicamid accounted for 60.6% of the TRR while TFNA accounted for 17.4% of the TRR. All other metabolites, TFNG, TFNG-AM and TFNA- AM, were $\leqslant$ 6% of the TRR.

In leaves, (Table 4), flonicamid accounted for 33–65% of the TRR followed by the major metabolites TFNG (8–19% of the TRR) and TFNA (5–16% of the TRR). All other metabolites, TFNG-AM and TFNA-AM, were $\leqslant$ 6% of the TRR.

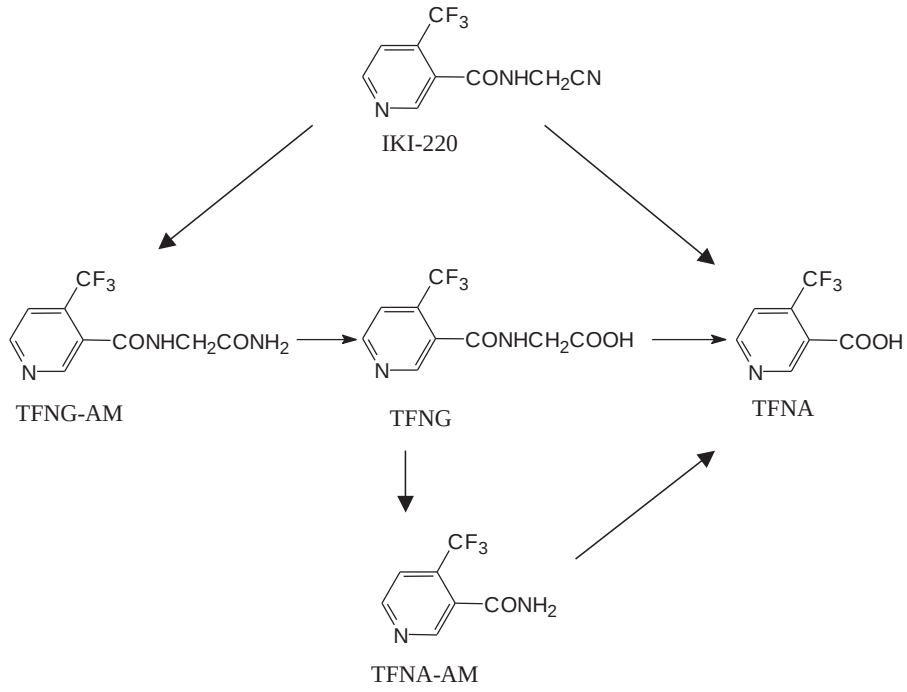


Figure 1 Proposed metabolic pathway in peaches

Bell pepper

Bell pepper plants (variety Wanderbell), grown in individual pots maintained in greenhouses, received a single application of flonicamid, radio-labelled at the 3 position of the pyridine ring (specific activity: 0.182 MBq/mg) and formulated as a 50% wettable granule formulation, at 100 g ai/ha. Bell pepper plants (fruits and leaves) were harvested 7 days and 14 days after application.

Bell pepper fruits and leaves were surface washed with methanol:water (10:90, v/v) prior to being homogenised in a food processor with dry ice. TRRs were determined by combusting triplicate aliquots of the homogenates.

TRRs in leaves decreased from 2.23 mg eq/kg, when harvested 7 days after treatment (DAT) to 1.35 mg eq/kg at 14 DAT. Similarly in fruits, the TRRs decreased insignificantly from 0.17 mg eq/kg (7 DAT) to 0.11 mg eq/kg (14 DAT).

TRRs in each tissue was determined by combusting the samples using an oxidizer. Unextracted residues in post-extraction solids were also determined by combustion.

Table 5 TRRs in bell pepper leaves and fruits

Crop part	TRRs (mg eq/kg)	
	7-DAT	14-DAT
Leaves	2.22	1.35
Fruits	0.17	0.11

Aliquots of fruit and leaf homogenates were extracted twice with methanol:water (50:50) followed by partitioning with hexane and ethyl acetate. The remaining aqueous phase was further separated by open column chromatography. All extracts were analysed by HPLC and TLC.

Table 6 Distribution of radioactivity in bell pepper leaves and fruits

Fraction	7-DAT		14-DAT	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Leaves				
Surface wash	0.81	36.1	0.23	17.3
Extracted	1.35	60.5	1.05	78.2

Fraction	7-DAT		14-DAT	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Hexane	0.004	0.2	0.00	0.0
Ethyl acetate	0.88	39.7	0.46	34.1
Aqueous soluble	0.46	20.6	0.59	44.1
PES	0.08	3.4	0.06	4.3
Total	2.23	100.0	1.35	100.0
Fruits				
Surface wash	0.06	33.6	0.02	18.2
Extracted	0.11	65.6	0.09	80.5
Hexane	0.00	0.0	0.00	0.0
Ethyl acetate	0.10	56.9	0.06	60.8
Aqueous soluble	0.02	8.6	0.02	19.6
PES	0.001	0.8	0.001	1.3
Total	0.17	100.0	0.11	100.0

Table 7 Identification/Characterization of TRRs in mature bell pepper leaves harvested 7 DAT and 14 DAT

	Surface wash		Extracted		Total	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
7 DAT						
Flonicamid	0.81	36.1	0.85	38.2	1.66	74.3
TFNA	n.d.	n.d.	0.04	2.0	0.04	2.0
TFNG	n.d.	n.d.	0.27	12.2	0.27	12.2
TFNA-AM	n.d.	n.d.	0.02	0.7	0.02	0.7
TFNG-AM	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Others	n.d.	n.d.	0.10	4.6	0.10	4.6
Total	0.81	36.1	1.28	57.7	2.09	93.8
14 DAT						
Flonicamid	0.22	16.1	0.42	31.3	0.64	47.4
TFNA	n.d.	n.d.	0.03	2.4	0.03	2.4
TFNG	n.d.	n.d.	0.38	28.2	0.38	28.2
TFNA-AM	n.d.	n.d.	0.02	1.1	0.02	1.1
TFNG-AM	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Others	n.d.	n.d.	0.15	10.8	0.16	12.0
Total	0.24	17.3	0.99	73.7	1.22	91.0

n.d. = Not detected

Others: consists of multiple peaks, each of which accounted for ≤2.1% of the TRR

Table 8 Identification/Characterization of TRRs in mature bell pepper fruits harvested 7 DAT and 14 DAT

	Surface wash		Extracted		Total	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
7 DAT						
Flonicamid	0.06	33.6	0.10	57.8	0.16	91.4
TFNA	n.d.	n.d.	0.001	0.9	0.001	0.9
TFNG	n.d.	n.d.	0.005	2.8	0.005	2.8
TFNA-AM	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
TFNG-AM	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Others	n.d.	n.d.	0.005	2.9	0.005	2.9
Total	0.06	33.6	0.11	64.3	0.17	97.9
14 DAT						
Flonicamid	0.02	17.8	0.06	58.8	0.08	76.6
TFNA	n.d.	n.d.	0.004	3.7	0.004	3.7
TFNG	n.d.	n.d.	0.008	7.8	0.008	7.8
TFNA-AM	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
TFNG-AM	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Others	n.d.	n.d.	0.008	7.1	0.008	7.5

	Surface wash		Extracted		Total	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Total	0.02	18.2	0.08	77.4	0.10	95.6

n.d. = Not detected

Others: consists of multiple peaks, each of which accounted for ≤ 2.0% of the TRR

No information was provided on the duration of storage of the fruit and leaf samples.

While the %TRR in the surface wash decreased with increasing DAT in the leaves and fruit (36% to 17% of the TRR in leaves and 34% to 18% of the TRR in fruits). The extracted TRRs and those in the PES increased with increasing DAT: 61–78% of the TRR and 3–4% of the TRR in leaves, respectively, and 66–81% of the TRR and 0.8–1% of the TRR in fruits, respectively. This trend demonstrates the translocation of the radioactivity from the surface into the leaves and fruits (Table 6).

Analysis of each of the fractions indicated that flonicamid and TFNG were the predominant residues in leaves and fruits at both harvest intervals. In leaves, the parent accounted for 47–74% of the TRR (0.6–1.7 mg/kg) while TFNG accounted for 12–28% of the TRR (0.3–0.4 mg/kg). Similarly in fruits, flonicamid accounted 77–91% of the TRR (0.08–0.16 mg/kg) while TFNG accounted for 3–8% of the TRR (0.005–0.008 mg/kg). All identified metabolites (TFNA, TFNA-AM and TFNG-AM) were either not detected or were ≤ 12% of the TRR.

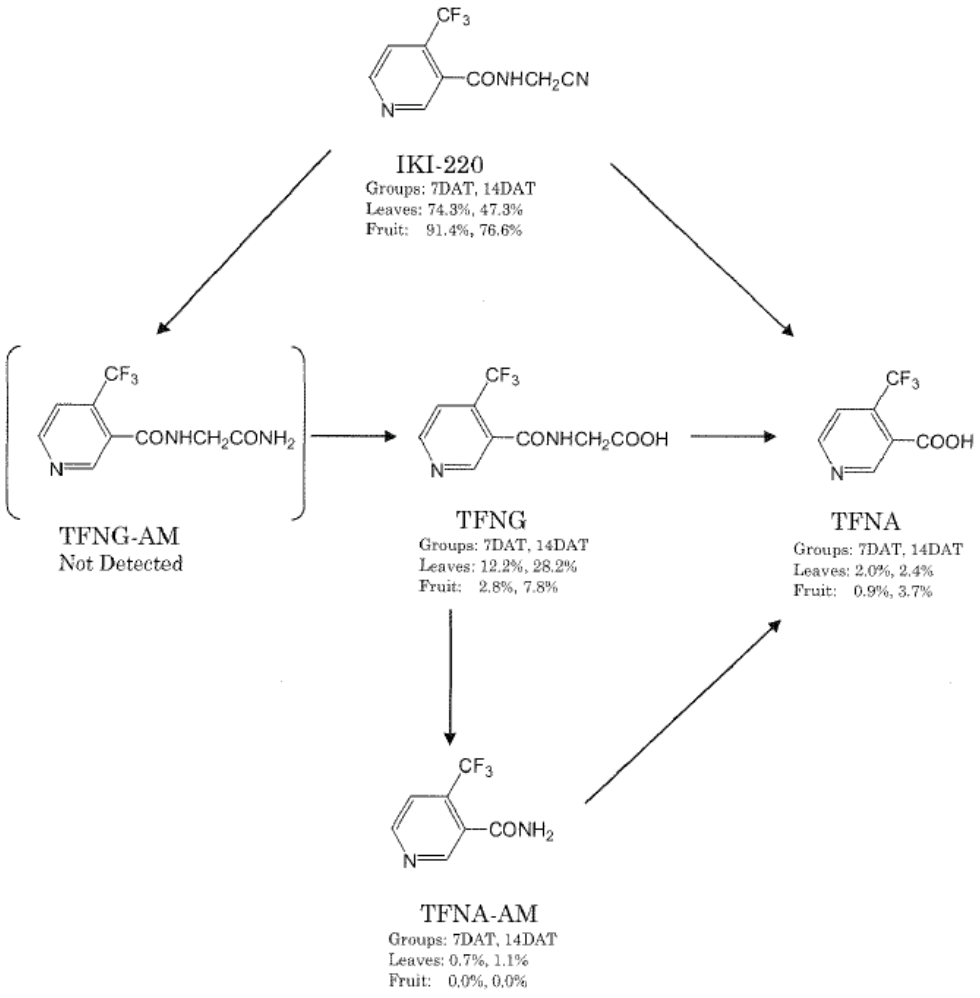


Figure 2 Proposed metabolic pathway in peppers

Potato

Flonicamid, radio-labelled at the 3 position of the pyridine ring (specific activity: 9.08 MBq/mg) and formulated as a 50% wettable powder formulation was applied to potted potato plants (variety Kennebec) maintained outdoors. The plants were treated at either the lower rate of 100 g ai/ha or the higher rate of 500 g ai/ha. Both treatments were repeated after a two-week interval and potato tubers and foliage were harvested 14 days following the second application.

One subsample of potato tubers from each group was washed with ACN:water (80:20, v/v) prior to homogenisation to determine the radioactivity in the surface wash whilst another subsample of potato tubers from each group was homogenised without rinsing the tubers. Potato foliage was processed to isolate the metabolites and to further elucidate the metabolic pathway of flonicamid. Total radioactive residue in each tissue was determined by combusting the samples using an oxidizer. Unextracted residues in PES were also determined by combustion. The radioactivity in the samples was measured by Liquid Scintillation Counting (LSC).

Overall total radioactive residues (TRRs) in unwashed tubers at the low rate and the high rate were 0.11 mg eq/kg and 0.20 mg eq/kg, respectively, whilst those in washed tubers were slightly higher; 0.14 mg eq/kg and 0.53 mg eq/kg. TRRs in mature foliage were higher than those in tubers; 1.53 mg eq/kg at the low rate and 7.67 mg eq/kg at the high rate (Table 9).

Table 9 TRRs in potato tubers and foliage

Crop part	TRRs (mg eq/kg)	
	Low rate (200 g ai/ha)	High rate (1000 g ai/ha)
Unwashed potato tubers	0.11	0.20
Washed potato tubers	0.14	0.53
Foliage	1.53	7.67

Tuber samples were homogenised and consecutively extracted with ACN, ACN:water (80:20, v/v) and twice with ACN:water (50:50, v/v), vortexed, sonicated and centrifuged. The extracts were combined. Foliage samples were homogenised with dry ice and extracted three times with ACN:water:acetic acid (60:40:0.1, v/v/v) and then filtered. The extracts were combined and concentrated.

Metabolites were first identified with HPLC by comparison with reference compounds isolated from repeated HPLC separations of foliage extract of the high treatment group. Most of the isolated metabolites were further purified by normal-phase chromatography. Their identification was supported by other methods such as LC-MS, HPLC on a C8 column and acid hydrolysis (with 3 N HCl).

Table 10 Identification/Characterization of TRRs in unwashed and washed potato tubers following treatment at the low rate and high rate

Fraction	Unwashed Potato Tubers				Washed Potato Tubers			
	Low rate (200 g ai/ha)		High rate (1000 g ai/ha)		Low rate (200 g ai/ha)		High rate (1000 g ai/ha)	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Surface wash	–	–	–	–	0.0004	0.3	0.002	0.4
Extracted	0.098	92.6	0.18	90.9	0.14	94.6	0.49	92.6
Flonicamid	0.006	5.6	0.04	19.3	0.02	11.5	0.04	7.5
TFNG	0.042	39.3	0.05	25.1	0.05	35.9	0.18	33.6
TFNA	0.036	34.4	0.07	33.7	0.05	31.8	0.21	40.0
TFNA conjugate	0.006	6.0	0.01	4.8	0.01	5.2	0.02	4.8
TFNG-AM	0.001	1.0	0.002	1.2	0.001	1.0	0.01	1.1
TFNA-AM	0.001	1.0	0.003	1.4	0.002	1.2	0.01	1.1
PM-3a	0.0	0.0	0.004	1.8	0.006	3.9	n.d.	n.d.
Others	0.006	5.3	0.007	3.7	0.006	4.2	0.02	4.5
Unextracted	0.008	7.4	0.018	9.1	0.007	5.1	0.04	7.0

Fraction	Unwashed Potato Tubers				Washed Potato Tubers			
	Low rate (200 g ai/ha)		High rate (1000 g ai/ha)		Low rate (200 g ai/ha)		High rate (1000 g ai/ha)	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Total	0.11	100	0.20	100	0.14	100	0.53	100.0

PM-3a: Conjugate of TFNA-AM

Table 11 Identification/Characterization of TRRs in foliage following treatment at the low rate and high rate

Fraction	Potato Foliage			
	Low rate (200 g ai/ha)		High rate (1000 g ai/ha)	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Extracted	1.36	88.7	6.91	90.1
Flonicamid	0.15	9.8	1.87	24.5
TFNG	0.56	36.4	2.13	27.8
TFNA	0.26	17.3	0.91	11.9
TFNA conjugate	0.08	5.2	0.30	3.9
TFNG-AM	0.06	4.0	0.22	2.8
TFNA-AM	0.07	4.8	0.60	7.9
PM-1a	0.05	3.2	0.19	2.4
PM-1b	0.06	3.6	0.21	2.7
Others	0.07	4.4	0.47	6.2
Unextracted	0.17	11.3	0.76	9.9
Total	1.53	100	7.67	100

PM-1a/1b: Acid hydrolyzable conjugates of TFNA

Tuber samples (from the low treatment rate) were analysed 10 days and 397 days after being placed in frozen storage. Extracted and bound residues at the 397-day interval were found to be comparable to those at the 10-day interval. The profiles were also similar between the initial and final analysis.

Considering the applications were made to the foliage of the potato plants, the presence of measurable TRRs in the tubers is evidence of translocation of the radioactivity from the foliage to the tubers. Furthermore, while the TRRs in tubers and foliage increased with increased application rate, the distribution of TRRs was relatively the same irrespective of the treatment rate.

The identity of the radioactive residues in the surface wash of tubers was not further investigated as the TRRs were too low. Analysis of each of the extracted fractions of unwashed and washed potato tubers and foliage from the low and high rate demonstrated that the predominant metabolites, TFNA and TFNG, accounted for a significant portion of the TRRs. Moreover, TFNA accounted for 32–40% of the TRR (0.04–0.21 mg eq/kg) in the unwashed and washed tubers and 12–17% TRR (0.26–0.91 mg eq/kg) in the foliage while TFNG accounted for 25–39% of the TRR in tubers (0.04–0.18 mg eq/kg) and 28–36% of the TRR in foliage (0.6–2.1 mg eq/kg). The parent, flonicamid, was also a major residue in tubers (6–12% of the TRR; 0.01–0.04 mg eq/kg) and foliage (10–25% of the TRR; 0.2–1.9 mg eq/kg), but accounted for less than the major metabolites. All other identified metabolites (TFNA conjugate, TFNG-AM< TFNA-AM, PM-1a, PM-1b and PM-3a) accounted for ≤ 6% of the TRR (0.02 mg eq/kg) in tubers and ≤ 8% of the TRR (0.6 mg eq/kg) in foliage. Overall, the general metabolic profile in foliage was similar to that in tubers.

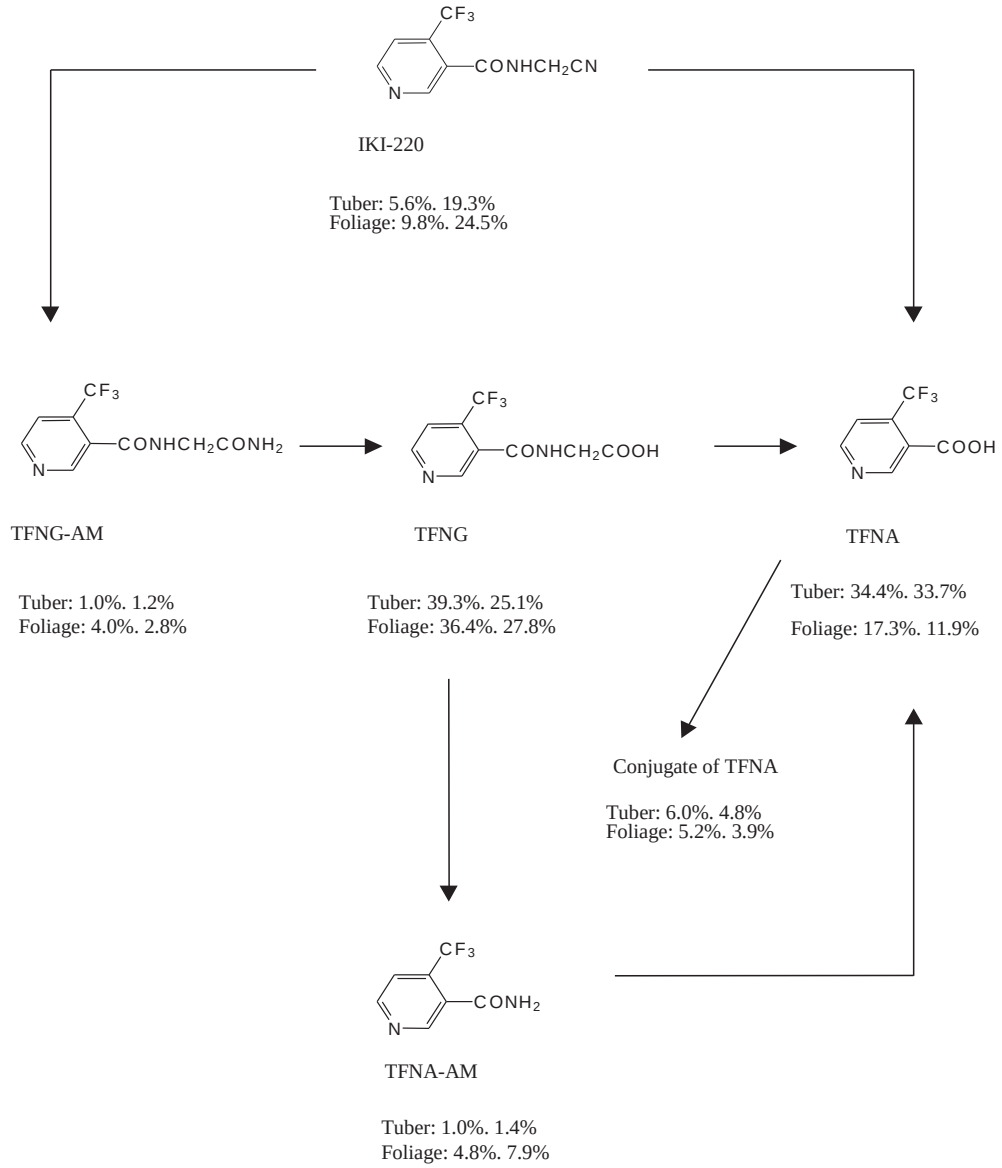


Figure 3 Proposed metabolic pathway in potato

Wheat

Study 1

Spring wheat plants (variety: Kulm) were grown in four separate plots maintained outdoor. Wheat plants grown in Plot I were designated as the control plants. Plants from Plots II and III were treated with flonicamid, radio-labelled at the 3 position of the pyridine ring (specific activity: 9.08 MBq/mg), formulated as a wettable powder, at a single application rate of 100 g ai/ha. Plants in Plot IV were treated twice at 100 g ai/ha/application with a re-treatment interval of 7 days. Forage was harvested 14 days after treatment, from Plot II. Hay was harvested from Plot III, 42 days after treatment. At final harvest, approximately 95 DAT, mature plants from Plot IV were separated into straw, chaff and grain.

The forage, hay, grain, straw and chaff were analysed to determine the total radioactive residue (TRR) levels. The radioactivity was measured with by LSC. Homogenised samples of forage, hay, straw, chaff and grain as well as the PES were combusted in an oxidizer.

Overall residues were lower in the wheat grain sample than the straw or chaff. The TRR levels in the chaff were higher compared to straw potentially because of tissue size differences (higher surface area to weight ratio) assuming a uniform application.

Table 12 Distribution of TRRs in wheat forage, hay, straw, chaff and grain

Plant part	Application rate (g ai/ha)	DAT (days)	TRRs (mg eq/kg)
Forage (Plot II)	100	14	0.648
Hay (Plot III)	100	42	0.951
Straw (Plot IV)	200	95	5.571
Chaff (Plot IV)			6.553
Grain (Plot IV)			2.559

Only samples of forage and hay were analysed to elucidate the nature of the flonicamid residues. These were homogenised in a blender with dry ice, extracted with ACN: water: acetic acid (60:40:0.1, v/v/v), blended with a tissue homogeniser, and then vacuum filtered. The process was repeated twice. The extracts were combined and concentrated by rotary evaporation under vacuum to a small volume. The concentrate was transferred to a vial with appropriate solvent and analysed by HPLC. The radioactivity in the eluate was detected using a radioactivity flow detector.

Table 13 Identification/Characterization of TRRs in wheat forage and hay

Fraction	Forage		Hay	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Extracted	0.58	96.0	0.89	96.0
Flonicamid	0.26	42.8	0.20	21.7
TFNA	0.04	6.5	0.04	3.8
TFNG	0.20	32.7	0.49	52.6
TFNA-AM	0.002	0.3	0.01	1.1
TFNG-AM	0.07	11.0	0.12	13.1
TFNA conjugate	0.004	0.7	n.d.	n.d.
Unknowns	< 0.012	<1.9	0.02	1.6
Polar	n.d.	n.d.	0.02	2.0
Unextracted	0.02	4.0	0.04	4.0
Total	0.60	100.0	0.93	100.0

The extracts were re-analysed by HPLC after storage in the freezer for approximately 2–3 months. Re-analysis confirmed the stability of metabolites in the matrices during storage.

The analysis of forage and hay samples showed that the metabolite profiles were qualitatively similar. Identified residues included flonicamid, TFNA, TFNG, TFNA-AM and TFNG-AM. Differences were observed in the distribution of minor metabolites only. Minor unknown components were observed at less than 0.02 mg eq/kg, $\leqslant$ 2% of the TRR. A trace amount (0.1% of the TRR) of the N-oxide of flonicamid was tentatively identified in forage.

The nature and distribution of metabolites were similar in both wheat forage and hay. The parent compound, flonicamid, accounted for 42.8 and 21.7% of the TRR in forage and hay, respectively. TFNG was the predominant metabolite, accounting for 32.7 and 52.6% of the TRR in forage and hay, respectively. TFNG-AM was present at 11.0–13.1% of the TRR. Metabolites TFNA and TFNA-AM were present at < 7% of the TRR. The unextracted ^{14}C residues in forage and hay represented 0.02–0.04 mg eq/kg (4% of the TRR). Since the samples contained less than 10% of the TRR, the PES were not characterised.

Study 2

Spring wheat plants (variety: Kulm), grown outdoors in metal containers were treated with flonicamid radio-labelled at the 3 position of the pyridine ring (specific activity: 9.08 MBq/mg), formulated as a

wettable granule (WG) and applied on wheat plants as an over-the-top foliar spray at Zadok's stage 86 (soft dough stage), 76 days after sowing. A single application was made at a rate equivalent to 100 g ai/ha. An additional set of wheat plants was treated at a higher rate of 500 g ai/ha. Plants were protected from rain for one week after the spray application. The wheat plants were harvested at maturity, i.e. 21 days after application and separated to straw (leaves and stem), chaff and grain (with hulls attached).

The radioactivity was measured by LSC. Homogenised samples of straw, chaff and grain as well as the PES were combusted in an oxidizer.

The TRRs in wheat straw, chaff and grain samples were 2.03 mg eq/kg, 3.60 mg eq/kg and 0.28 mg eq/kg in the 100 g ai/ha treatment plot (low rate), and 9.28 mg eq/kg, 18.88 mg eq/kg and 1.47 mg eq/kg in the 500 g ai/ha treatment plot (high rate), respectively (Table 14).

Table 14 Distribution of TRRs in wheat straw, chaff and grain

Crop part	TRRs (mg eq/kg)	
	Low rate (100 g ai/ha)	High rate (500 g ai/ha)
Straw	2.03	9.28
Chaff	3.60	18.88
Grain	0.28	1.47

Samples of homogenised straw, grain and chaff were mixed with ACN: water: acetic acid (60:40:0.1, v/v/v), blended with a tissue homogeniser, and then vacuum filtered. This process was repeated twice, following which, the extracts were concentrated and analysed using various HPLC and TLC techniques. The identification of metabolites was supported by other methods such as LC-MS, HPLC on different columns and TLC. The PES were allowed to dry, then combusted to quantitate the unextracted residues. The unextracted residues in the PES obtained by extraction of a second set of subsamples of the normal treatment rate Plot were characterised by acid (1 N HCl) and base hydrolysis (1 N NaOH). For straw and chaff the HCl digestion was followed by treatment with 72% H_2SO_4 to digest the carbohydrate (cellulose) fraction from the matrix leaving behind lignin fraction.

Table 15 Identification/Characterization of TRRs in wheat straw, chaff and grain

Fraction	Straw				Chaff				Grain			
	Low rate (100 g ai/ha)		High rate (500 g ai/ha)		Low rate (100 g ai/ha)		High rate (500 g ai/ha)		Low rate (100 g ai/ha)		High rate (500 g ai/ha)	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Extracted	1.63	80.1	7.42	79.9	2.73	75.7	15.40	81.6	0.25	89.4	1.38	94.3
Flonicamid	1.02	50.2	4.10	44.2	1.47	40.7	8.85	46.9	0.08	29.9	0.35	23.9
TFNG	0.40	19.6	1.98	21.3	0.60	16.6	3.57	18.9	0.11	39.4	0.65	44.1
TFNA	0.04	2.0	0.36	3.8	0.20	5.7	0.57	3.0	0.02	8.1	0.05	3.8
TFNG-AM	0.09	4.5	0.52	5.6	0.19	5.4	0.77	4.1	0.01	3.1	0.08	5.7
TFNA-AM	0.04	1.8	0.22	2.4	0.09	2.5	0.71	3.8	0.02	6.2	0.14	9.5
N-oxide of TFNA-AM	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.008	2.7	0.09	6.1
Unknown (M10)	0.04	2.0	0.09	1.0	0.18	4.9	0.31	1.6	0.0	0.0	0.0	0.0
Others			0.14	1.5			0.61	3.2			0.02	1.5
Unextracted	0.40	19.9	1.86	20.1	0.88	24.3	3.48	18.4	0.03	10.6	0.08	5.7
Total	2.03	100.0	9.28	100.0	3.60	100.0	18.88	100.0	0.28	100.0	1.47	100.0

Homogenized straw, chaff and grain, from the low treatment rate experiment, were extracted and analysed immediately after collection and subsequently stored for 480–505 days in a freezer prior to re-analysis. For all three commodities, extracted and bound residues were found to be comparable to those of the initial extraction. The results indicate a similar metabolite profile for straw and chaff between the first and final analysis; however, for grain a decrease in

the concentration of parent and TFNA with a simultaneous increase in TFNG is observed. This does not have any significant impact on the metabolite profile.

Although the TRRs in wheat straw, chaff and grain increased with increased application rates, the distribution of TRRs was relatively the same irrespective of the treatment rate. Considering the timing of application of the test material and the measurable TRRs in grain, chaff and straw at maturity, there appears to have been translocation of the radioactivity from the site of application to the mature plant parts.

The majority of the radioactivity (76–94% of the TRR) in straw, chaff and grain was extracted with organic solvents. Analysis of the organic fractions indicated that flonicamid and TFNG were the predominant residues at both treatment rates. In straw, chaff and grain, the parent accounted for 44–50% of the TRR (1.0–4.1 mg/kg), 41–47% of the TRR (1.5–8.8 mg/kg) and 24–30% of the TRR (0.08–0.4 mg/kg), respectively. The major metabolite TFNG accounted for approximately 20% of the TRR (0.4–2.0 mg eq/kg), 17–19% of the TRR (0.6–3.6 mg eq/kg) and 39–44% of the TRR (0.11–0.65 mg eq/kg) in straw, chaff and grain, respectively. All identified metabolites (TFNA, TFNG-AM, TFNA-AM and N-oxide of TFNA AM) were either not detected or were $\leqslant$ 8% of the TRR.

The unextracted residues amounted to approximately 20, 24 and 11% of the TRR in straw, chaff and grain, respectively, at the low rate and to about 20, 18 and 6% TRR at the high rate. Samples from the low rate treatment were further characterized by:

- Hydrolysis with 1 N HCl at 40 ºC to release covalently bound residues
- Hydrolysis with 1 N HCl followed by digestion with 72% H2SO4 to determine the reincorporated activity in the carbohydrate (cellulose) and lignin fractions of straw and chaff
- Hydrolysis with 1 N HCl followed by 1 N NaOH.

The radioactivity released following hydrolysis with 1 N HCl accounted for 7% of the TRR (straw), 6% of the TRR (chaff) and 3% TRR (grain) and was identified as either parent and metabolites (straw and chaff) or as metabolites of flonicamid only (grain; TFNG, TFNG-AM and TFNA-AM). Each of the identified metabolites was present at $\leqslant$ 2% of the TRR. As a result of sequential digestion with HCl and H_2SO4, ^{14}C incorporation into carbohydrates amounted to 3% of the TRR in straw and 5% of the TRR in chaff. Base digestion with 1 N NaOH released 56, 61 and 59% of the bound radioactivity in straw, chaff and grain, respectively, corresponding to 12, 14 and 5% of the TRR, respectively. Part of this radioactivity may have been due to polar sugars as released by sulphuric acid digestion, with the remainder of ^{14}C attributed to lignin (straw: 10.9% of the TRR, chaff: 13.6% of the TRR).

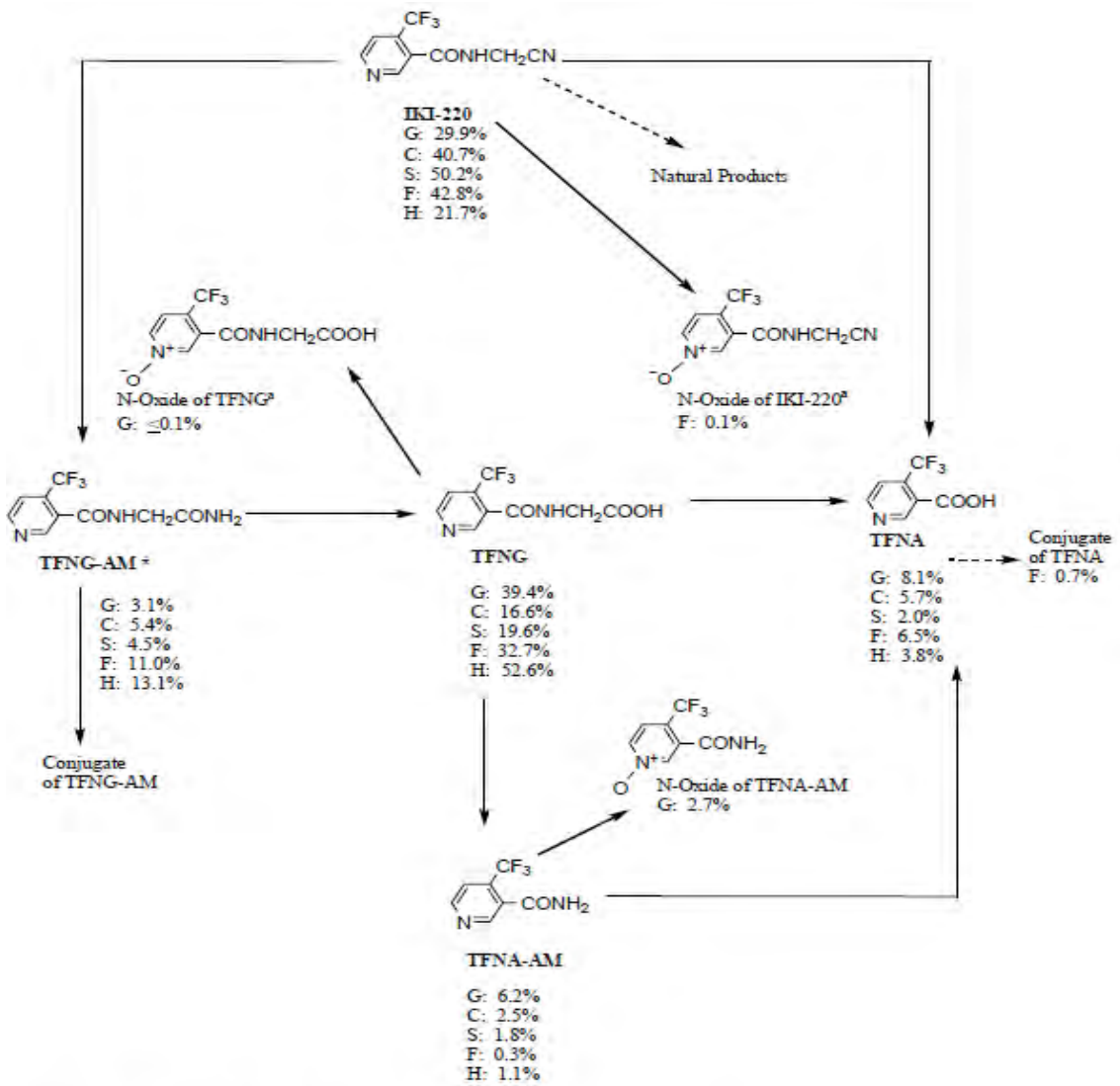


Figure 4 Proposed metabolic pathway in mature wheat

Animal metabolism

The Meeting received information on the fate of 3-pyridine -^{14}C- labelled flonicamid in lactating goats and laying hens. Metabolism in laboratory animals (rat) was summarized and evaluated by the WHO panel of the 2015 JMPR.

Lactating goat

The metabolism of [^{14}C]flonicamid was investigated in two lactating goats (*Capra hircus*), weighing 45–47 kg, dosed orally once daily, using a balling gun, immediately after the morning milking, for 5 consecutive days. The animals were dosed with 3-pyridine-^{14}C-labelled flonicamid (specific activity: 245 µC/mg) at a dose level of 15 mg/day equivalent to 10 ppm in the diet. Milk production ranged from 1.5–1.9 L/day. During the treatment period, milk was collected twice daily, after the morning and evening milking, while urine and faeces were collected once daily. At sacrifice (within 5–8 hours

after the last dose) samples of liver, kidney, muscle (loin and hind leg), fat (omental and peri-renal), heart and GI tract were collected.

Radioactivity in liquid samples (milk, urine, stanchion wash and extracts) was measured LSC. Samples were combusted to verify the total radioactive residues (TRR) prior to extraction. Liver, kidney, muscle, fat, faeces, heart samples and post-extraction solids (PES) were combusted in an oxidizer. The extracted samples were dried before combustion.

The major route of elimination of the radioactivity was via the urine which accounted for 49% of the total administered radioactivity (AD), while faeces accounted for 17–21% of the AD and milk accounted for 1% of the AD. Overall, the tissue burden was low, accounting for < 10% of the AD. The overall recovery of administered radioactivity averaged 95%.

The total radioactive residues (TRRs) were highest in liver (1.2 mg eq/kg), followed by kidney (0.70 mg eq/kg), muscle (0.34–0.39 mg eq/kg) and fat (0.05–0.14 mg eq/kg).

Table 16 Balance of radioactivity in goats following oral administration of [^{14}C]flonicamid for 5 days

Sample	Goat 1		Goat 2	
	%AD	mg eq/kg	%AD	mg eq/kg
Milk	1.18	0.078–0.204	0.97	0.081–0.216
Liver	1.67	1.21	1.71	1.22
Kidney	0.17	0.67	0.15	0.66
Loin muscle	3.80	0.38	3.97	0.39
Hind muscle	3.35	0.34	3.48	0.34
Perirenal fat	0.09	0.14	0.05	0.07
Omental fat	0.07	0.11	0.03	0.05
Heart	0.08	0.22	0.08	0.22
Blood	0.77	0.18	0.88	0.21
Feces	17.06	–	20.59	–
Urine	48.79	–	48.65	–
Cage wash	1.23	–	0.80	–
Subtotal	78.26	–	81.36	–
GI tract	16.62	–	14.09	–
Total Recovery	94.88	–	95.45	–

For collection Days 1–4, evening and morning milk were combined while Day 5 samples consisted of evening milk only. As TRRs were consistently higher in evening milk compared with the morning milk, in the absence of the morning milk on Day 5, ^{14}C-residues were higher than on other collection days.

Table 17 TRRs in goat milk following oral administration of [^{14}C]flonicamid for 5 days

Collection Day	Goat 1		Goat 2	
	mg eq/kg	% AD	mg eq/kg	% AD
Day 1	0.086	0.26	0.081	0.17
Day 2	0.078	0.26	0.090	0.21
Day 3	0.087	0.25	0.090	0.21
Day 4	0.095	0.27	0.096	0.22
Day 5	0.204	0.19	0.216	0.16

Extraction of milk samples with ethanol and ethanol:water (80:20, v/v) and partitioning with hexane released 97–98% of the TRRs. The PES was combusted. The procedure used for the extraction of organs and tissues was relatively similar to that of milk. However, different solvents were used. Kidney and liver samples were extracted with acetonitrile and acetonitrile:water (50:50, v/v) containing 1% acetic acid while muscle samples (loin and rear leg) were extracted with acetonitrile only. The fat samples (omental and peri-renal) were first extracted with hexane and then with acetonitrile. Use of these solvents resulted in extraction efficiencies ranging from 42–57% of the TRRs for organs, 43–52% of the TRRs for muscle and 81–86% of the TRRs for fat.

The unextracted ^{14}C residues in liver, kidney, fat and muscle tissues were sequentially hydrolysed with 1 N HCl and 6 N HCl. The PES from these organs also underwent protease digestion.

Quantification and identification of parent and metabolites were carried out by HPLC (using different solid phases). Purified metabolite isolates were analysed by mass spectrometry.

Table 18 Characterization and identification of radioactivity in goat milk, kidney and liver

Fraction	Milk				Liver				Kidney			
	Goat 1		Goat 2		Goat 1		Goat 2		Goat 1		Goat 2	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Extracted	0.084	97.4	0.09	97.7	0.57	47.3	0.513	42.1	0.362	53.9	0.377	57.3
Flonicamid	n.d.	n.d.	0.001	1.2	0.008	0.6	0.006	0.5	0.009	1.3	0.010	1.6
TFNA	n.d.	n.d.	n.d.	n.d.	0.009	0.4	0.008	0.7	0.009	1.4	0.037	5.6
TFNA unstable conjugate	n.d.	n.d.	0.004	4.6	0.082	6.8	0.051	4.2	0.080	11.9	0.010	1.6
TFNA-AM	0.084	97.4	0.082	91.9	0.355	29.4	0.352	28.9	0.207	30.8	0.270	41.1
6-OH TFNA-AM	n.d.	n.d.	n.d.	n.d.	0.069	5.7	0.078	6.4	0.041	3.2	0.041	6.3
Others	n.d.	n.d.	n.d.	n.d.	0.049	4.1	0.018	1.5	0.016	2.3	0.007	1.1
Unextracted residues	0.002	2.6	0.002	2.3	0.635	52.7	0.705	57.9	0.309	46.1	0.281	42.7
Total	0.087	100.0	0.090	100.0	1.206	100.0	1.218	100.0	0.671	100.0	0.658	100.0

n.d.= Not detected

Table 19 Characterization and identification of radioactivity in goat fat

Fraction	Omental fat				Perirenal fat			
	Goat 1		Goat 2		Goat 1		Goat 2	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Extracted	0.094	85.2	0.038	80.7	0.123	85.9	0.058	81.1
Flonicamid	0.006	5.5	0.001	2.1	0.004	3.2	0.002	2.6
TFNA	0.005	4.6	0.001	2.6	0.009	6.5	0.002	2.2
TFNA unstable conjugate	n.d.	n.d.	0.000	0.9	0.002	1.5	0.000	1.0
TFNA-AM	0.080	72.9	0.035	73.7	0.105	73.5	0.0532	74.1
6-OH TFNA-AM	0.002	1.4	n.d.	n.d.	n.d.	n.d.	0.000	0.4
Others	0.001	0.9	0.001	1.4	0.002	1.2	0.001	0.8
Unextracted residues	0.016	14.8	0.009	19.3	0.020	14.1	0.014	18.9
Total	0.110	100.0	0.047	100.0	0.143	100.0	0.072	100.0

n.d.= Not detected

Table 20 Characterization and identification of radioactivity in goat muscle

Fraction	Loin muscle				Hind leg muscle			
	Goat 1		Goat 2		Goat 1		Goat 2	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Extracted	0.170	44.3	0.167	43.1	0.177	52.1	0.172	50.8
Flonicamid	0.006	1.5	0.004	1.0	0.007	2.0	0.005	1.4
TFNA	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
TFNA unstable conjugate	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
TFNA-AM	0.165	42.8	0.163	42.1	0.170	50.2	0.166	48.8
6-OH TFNA-AM	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.002	n.d.
Others	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Unextracted residues	0.214	55.7	0.220	56.9	0.162	47.9	0.167	49.2
Total	0.385	100.0	0.387	100.0	0.340	100.0	0.339	100.0

n.d.= Not detected

Table 21 Distribution of flonicamid and metabolites released from unextracted residues of selected goat samples

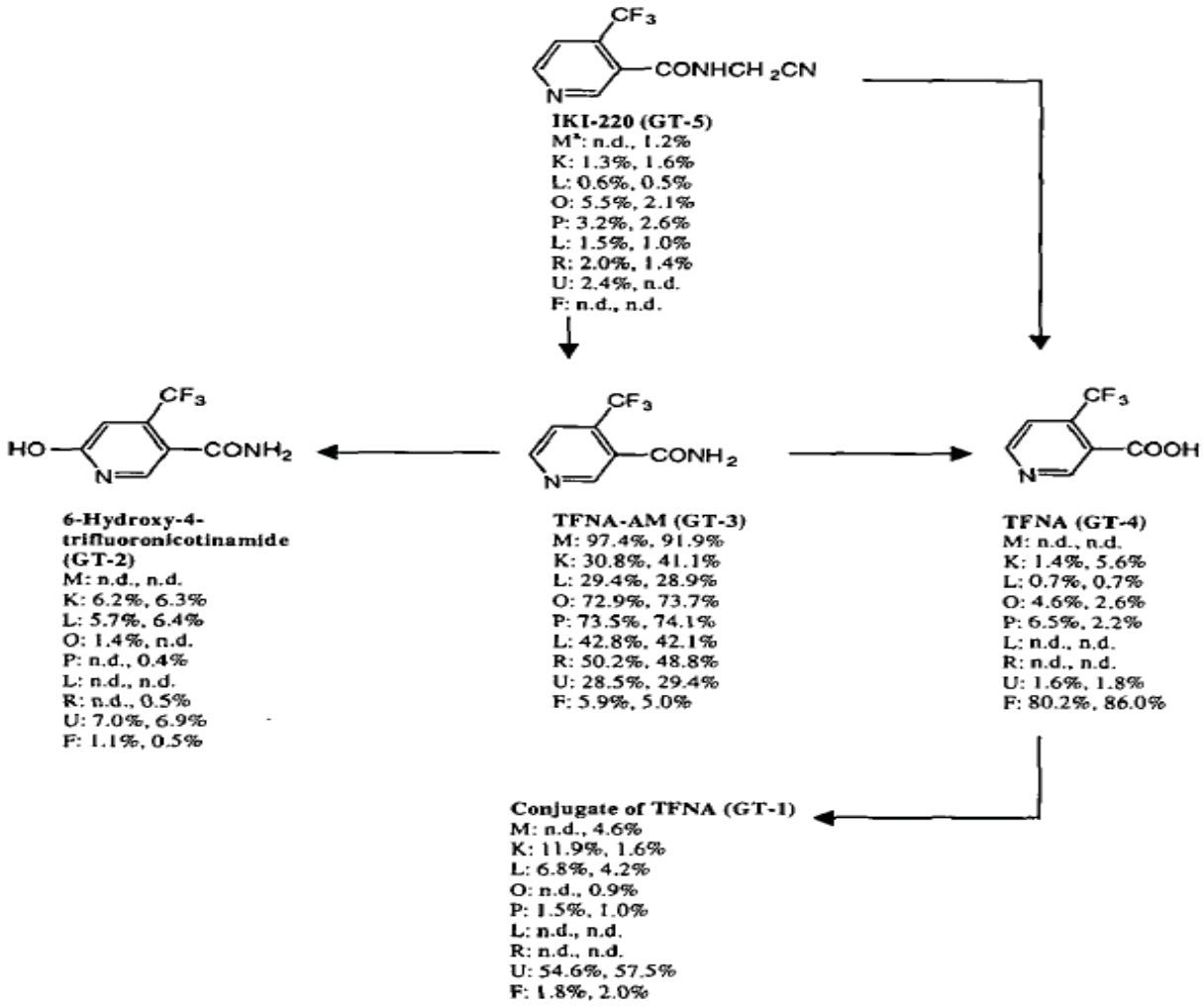
	1 N HCl digestion			6 N HCl digestion			Protease digestion		
	mg/kg	% NER	% TRR	mg/kg	% NER	% TRR	mg/kg	% NER	% TRR
Liver									
Flonicamid	–	–	–	0.006	1.0	0.5	–	–	–
TFNA	–	–	–	0.033	5.2	2.8	–	–	–
TFNA-AM	0.025	4.0	2.1	0.250	39.5	20.8	0.340	53.5	28.2
6-OH TFNA-AM	0.082	13.0	6.5	0.051	8.1	4.2	–	–	–
Kidney									
Flonicamid	n.a.	n.a.	n.a.	–	–	–	n.a.	n.a.	n.a.
TFNA	n.a.	n.a.	n.a.	0.024	7.6	3.5	n.a.	n.a.	n.a.
TFNA-AM	n.a.	n.a.	n.a.	0.169	54.6	25.2	n.a.	n.a.	n.a.
6-OH TFNA-AM	n.a.	n.a.	n.a.	0.13	4.2	1.9	n.a.	n.a.	n.a.
Loin muscle									
Flonicamid	n.a.	n.a.	n.a.	–	–	–	n.a.	n.a.	n.a.
TFNA	n.a.	n.a.	n.a.	–	–	–	n.a.	n.a.	n.a.
TFNA-AM	n.a.	n.a.	n.a.	0.120	56.1	31.2	n.a.	n.a.	n.a.
6-OH TFNA-AM	n.a.	n.a.	n.a.	–	–	–	n.a.	n.a.	n.a.

NER = Unextracted residues

n.a.= Not analysed

All samples of liver, kidney, muscle, fat and Day 3 milk were extracted and analysed within one month of collection, and re-extracted and analysed after 9 months of storage. A comparison of distribution of the TRRs in the initial and final profiles demonstrated minimal changes, indicating stability of the radioactive components under the storage conditions.

Flonicamid was rapidly metabolised in lactating goats, accounting for 0.5–5.5% of the TRRs in tissues and organs. TFNA-AM was the major metabolite in organs (29% of the TRRs in liver, 31–41% of the TRRs in kidney), tissues (74% of the TRRs in fat, 42–50% of the TRRs in muscle) and milk (97% of the TRRs). The metabolite 6-hydroxy TFNA-AM accounted for approximately 3–6% of the TRRs in liver and kidney and less than 1.4% of TRRs in tissue samples and milk.



*M = milk, day 3, K = kidney, L = liver, O = omental fat, P = perirenal fat, L = loin muscle, R = rear leg muscle, U = urine, day 2 and F = feces, day 3.
The values given are % TRR distribution for the samples from goat replicates 1 and 2, respectively.

Figure 5 Proposed metabolic pathway in lactating goats

Laying Hen

Leghorn laying hens (*Gallus domesticus*), weighing 1.37–1.87 kg, were dosed orally once daily for 5 consecutive days with 3-pyridine-^{14}C-labelled flonicamid (specific activity: 1.67 MBq/mg), at 1.3 mg/day, equivalent to 10 mg/kg feed. Eggs were collected twice daily, in the morning before and in the afternoon after administration, while excreta were collected once daily. The average egg production was 95%. The animals were sacrificed approximately 6 h after the last dose and the liver, kidney, thigh muscle, breast muscle, skin and fat were collected and pooled per dose group. Radioactivity in sample solutions was determined by LSC. Solid samples of liver, kidney, muscle, fat, skin, egg and excreta were first combusted in an oxidizer to verify the total radioactive residues (TRR) prior to extraction. Both the dried extracts of tissues and the PES were combusted.

Approximately 91.1% of the administered dose (AD) including 5.7% from the gastrointestinal tract and its contents was recovered. Most of the AD (72.3%) was excreta-related. Total radioactive residues (TRR) in egg white and egg yolk accounted for about 2.4% of AD (1.8% AD in egg white plus 0.6% AD in yolk). The TRR levels in both egg white and egg yolk reached a plateau by Day 3 of dosing. The tissue burden was very low (< 6% of the AD) with highest concentrations found in skin (2.3% of the AD), followed by muscle (evenly

distributed between breast and thigh muscle; each approximately 1.1% of the AD), liver (0.8% of the AD), fat (0.3% of the AD) and kidney (0.2% of the AD). Blood contained 4.7% of the AD.

Table 22 Balance of radioactivity in hens following oral administration of [^{14}C]flonicamid for 5 days

Sample	%AD	mg eq/kg
Egg white	1.84	0.04–0.89
Egg yolk	0.60	0.01–0.68
Liver	0.79	1.18
Kidney	0.22	1.42
Breast muscle	1.13	0.99
Thigh muscle	1.08	0.95
Skin	2.31	0.70
Fat	0.33	0.15
Blood	4.72	1.26
Excreta	67.18	5.20–9.51 [a]
Cage wash	5.16	1.55
Gastrointestinal tract	5.72	–
Total Recovery	91.08	

[a] Excreta collected just before sacrifice

Table 23 TRRs in eggs following oral administration of [^{14}C]flonicamid for 5 days

Day	Egg White		Egg Yolk	
	%TAR	mg/kg eq	%TAR	mg/kg eq
1	0.02	0.04	0.00	0.01
2	0.34	0.56	0.09	0.31
3	0.46	0.74	0.15	0.50
4	0.53	0.87	0.18	0.63
Sacrifice	0.49	0.89	0.18	0.68
Total	1.84		0.60	

Extraction of egg yolk and white (Day 3), liver, kidney, breast and thigh muscle with acetonitrile, and acetonitrile:water (80:20, v/v) containing 1% acetic acid released 81–99% of the TRRs. The radioactivity in each extract and in the PES, after combustion, was quantitated. Skin and fat were extracted in the same manner except that an extraction with hexane was done initially, which resulted in an extraction efficiency of 99% of the TRRs.

Each of the PES of liver and kidney was sequentially hydrolysed with 1 N HCl and 6 N HCl. Aliquots of the PES from liver and kidney were additionally hydrolysed using protease. In a separate experiment, digestion of liver PES was carried out with enzyme.

Quantification and identification of parent and metabolites were carried out by HPLC using different columns. For samples containing low levels of radioactivity, fractions of the effluent were collected and analysed by LSC. Analytical methods (HPLC) were validated with authentic standards and shown to achieve the necessary resolution and sensitivity. HPLC column performance and chromatographic resolution were validated with authentic labelled and non-labelled standards.

Table 24 Characterization and identification of radioactivity in eggs, liver and kidney

Fraction	Day 3 Egg yolk		Day 3 Egg white		Liver		Kidney	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Extracted	0.493	99.3	0.739	99.9	1.117	94.6	1.149	81.2
Flonicamid	0.019	3.8	0.018	2.5	0.004	0.3	0.005	0.4
TFNA	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.020	1.4
TFNA-AM	0.047	94.7	0.710	96.0	1.100	92.9	1.081	76.4
OH TFNA-AM	n.d.	n.d.	n.d.	n.d.	0.001	0.1	0.034	2.4
TFNG-AM	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.002	0.1

Fraction	Day 3 Egg yolk		Day 3 Egg white		Liver		Kidney	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Unknown	n.d.	n.d.	0.002	0.3	0.005	0.4	0.001	0.1
Others	0.004	0.8	0.009	1.2	0.011	0.9	0.006	0.4
Unextracted residues	0.004	0.7	0.0005	0.1	0.063	5.4	0.266	18.8
Total	0.497	100.0	0.740	100.0	1.182	100.0	1.42	100.0

Table 25 Characterization and identification of radioactivity in muscle, skin and fat

Fraction	Breast muscle		Thigh muscle		Skin		Fat	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Extracted	0.988	99.4	0.939	99.1	0.694	98.9	0.147	98.9
Flonicamid	0.006	0.6	0.004	0.4	0.003	0.4	0.001	0.7
TFNA	n.d.	n.d.	0.001	0.1	n.d.	n.d.	n.d.	n.d.
TFNA-AM	0.961	96.8	0.918	96.8	0.677	96.4	0.141	94.7
OH TFNA-AM	0.003	0.3	0.012	1.3	0.002	0.3	0.0007	0.5
TFNG-AM	n.d.	n.d.	0.001	0.1	0.0004	0.1	0.0001	0.1
Unknown	0.011	1.1	0.0003	0.0	0.002	0.3	n.d.	n.d.
Others	0.006	0.6	0.003	0.3	0.009	1.3	0.004	3.0
Unextracted residues	0.006	0.6	0.009	0.9	0.008	1.1	0.002	1.1
Total	0.99	100.0	0.95	100.0	0.70	100.0	0.15	100.0

Table 26 Distribution of metabolites released from unextracted residues of liver and kidney

	1 N HCl digestion			6 N HCl digestion			Protease digestion		
	mg/kg	% NER	% TRR	mg/kg	% NER	% TRR	mg/kg	% NER	% TRR
Liver (NER = 0.063 mg/kg)									
Flonicamid	0.036	56.5	3.0	0.028	43.5	2.3	0.063	100	5.4
TFNA	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	–	–	
TFNA-AM	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.020	–	1.7
OH TFNA-AM	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	–	–	–
Unknown	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.043	–	3.6
Kidney (NER = 0.267 mg/kg)									
Flonicamid	0.090	33.9	6.4	0.176	66.1	12.4	0.257	96.6	–
TFNA	–	–	–	0.021	7.9	1.5	–	–	–
TFNA-AM	0.022	8.3	1.6	0.143	53.6	10.1	0.216	–	15.3
OH TFNA-AM	0.068	25.6	4.8	0.012	4.6	0.9	–	–	–
Unknown	–	–	–	–	–	–	0.041	–	2.9

NER = Unextracted residues

n.a.= Not analysed

All samples of liver, kidney, muscle, fat and Day 3 egg yolks and egg whites were extracted and analysed approximately 9 months after collection. A comparison of distribution of the TRRs in the initial and final profiles demonstrated minimal changes, indicating stability of the radioactive components under the storage conditions.

Flonicamid was rapidly metabolised and excreted with only a very small percentage of the administered dose found in eggs, tissues and organs. TFNA-AM was the predominant metabolite in egg whites and egg yolks (⩽ 96.0% of the TRR), liver (92.9% of the TRR), kidney (76.4% of the TRR) and tissues (96.8% of the TRR in both breast muscle and thigh muscle, 96.4% of the TRR in skin and 94.7% of the TRR in fat).

Other metabolites identified in organs and tissues were OH-TFNA-AM and TFNG-AM; however, neither of these accounted for greater than 4.8% of TRR. One metabolite found in breast muscle and accounting for 1.1% of the TRR remained unidentified (named HN-1).

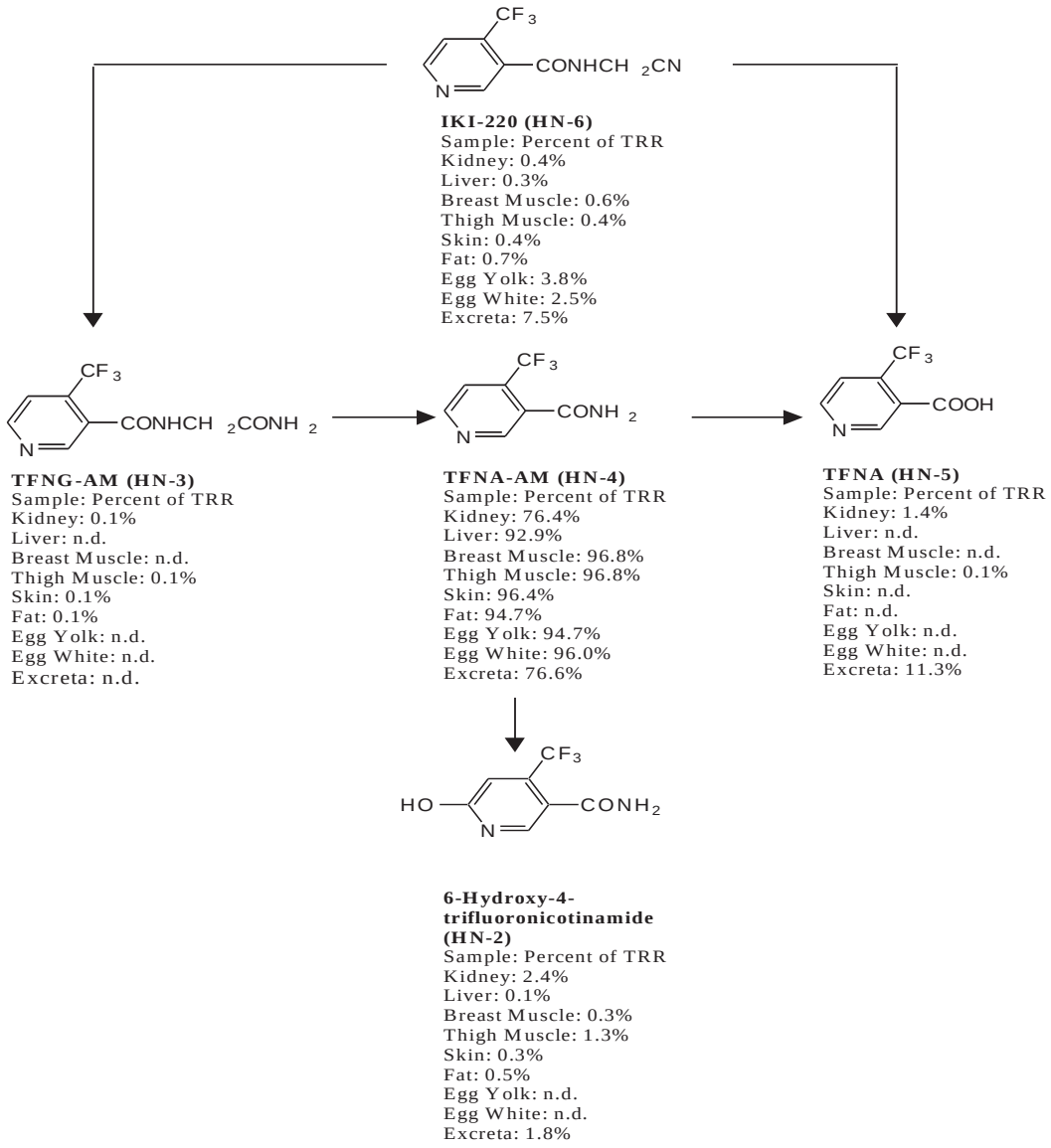


Figure 6 Proposed metabolic pathway in laying hens

Environmental fate in soil

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 flonicamid, supervised residue trials data were received for foliar spray on permanent crops and on annual crops. Therefore, according to the FAO manual, only studies on aerobic degradation, photolysis and rotational crops (confined, field) were evaluated. For information on hydrolysis and photolysis see also Physical and Chemical Properties.

Aerobic degradation

The route of degradation of [^{14}C]flonicamid (specific activity 8.91 MBq/mg) in soil under aerobic conditions was investigated in a biologically active loamy sand soil collected from Madison, Ohio, USA. A subsample was transferred to a growing pot and stored in a greenhouse. Subsamples of the sieved moist soil were weighed into separate plastic bottles and treated after 43 days of equilibration.

The dosing solution was prepared in water and each subsample was treated to produce a soil concentration of 0.1 µg/g (0.1 ppm). The soil sample was connected to a series of traps to retain any volatiles. Duplicate samples of the treated soil were extracted after treatment on Day 0 and after 0.5, 1, 2, 3, 7, 14 and 30 days of incubation.

The average recovery of applied radioactivity (AR) over the 30-day course of the study was 86.3%. The recovery of radiocarbon was low in the definitive experiment for sampling times days 3, 7, 14 and 30 days due to very rapid extensive metabolism and mineralization which formed $^{14}CO_2$.

A second set of soil samples (mass balance experiment) was dosed and sampled at Days 3, 7, 14 and 30 to correct for mass balance and volatiles. The average recovery of applied radioactivity for the mass balance experiment was 93.3%. Extracted residues decreased from 101.4% AR on Day 0 to 13.7% AR after 30 days incubation. Unextracted soil residues increased steadily from 0.7% on Day 0 to 35.2% on Day 30. Evolution of $^{14}CO_2$ increased throughout the study, reaching a maximum of 47.0% AR after thirty days in this experiment.

Table 27 Distribution of radioactivity in loamy sand soil treated with [^{14}C]flonicamid and incubated at 20 °C and 45% WHC_{max} (values are the average of duplicate analyses)

Sampling	Extracted	Unextracted	CO2	Total recovery
	[% AR]			
Day 0	101.4	0.7	NA	102.1
Day 0.5	94.8	1.5	NA	96.3
Day 1	99.7	4.0	0.2	103.9
Day 2	82.7	8.0	0.3	91.0
Day 3	75.8	12.0	8.1 [a]	95.9
Day 7	51.9	30.8	26.1 [a]	108.8
Day 14	20.0	34.9	40.0 [a]	94.9
Day 30	13.7	35.2	47.0 [a]	95.9

NA = Not analysed

[a] Values corrected with the data of the mass balance experiment. In the definitive study, the $^{14}CO_2$ was not trapped efficiently.

Flonicamid rapidly declined from 99.3% AR at Day 0 to 2.3% by Day 30. Five metabolites were identified; TFNA, TFNA-OH, TFNG, TFNG-AM and TFNA-AM. TFNA and TFNA-OH were the major metabolites exceeding 10% AR. TFNA peaked at 36.4% AR on Day 3, before declining to 0% by the end of the 30-day interval. Levels of the metabolite TFNA-OH increased steadily, to 20.2% AR through 7 days, then declined to 0.5% AR at Day 30. The metabolite TFNG-AM reached a maximum of 9.6% AR by Day 0.5, but decreased to 0% on Day 7 and 1.8% AR on Day 30. TFNA-AM remained below 7% AR and TFNG below 3% AR over the course of the experiment. TFNA-AM was present in the dose solution at a level of 2.3% AR. Other more polar, minor components were detected on several days, for a combined total of less than 7% AR. The distribution of metabolites in soil treated with [^{14}C]flonicamid is shown in Table 28.

Table 28 Distribution of extracted components from soil treated with [^{14}C]flonicamid and incubated at 20 °C and 45% WHC_{max} (values are the average of duplicate analyses)

Sampling	Flonicamid	TFNA	TFNA-OH	TFNG	TFNG-AM	TFNA-AM	Others [a]	Total
	[% AR]							
Day 0	99.3	0.0	0.0	0.0	0.0	2.1	0.0	101.4
Day 0.5	66.8	14.9	0.0	0.0	9.6	3.5	0.0	94.8
Day 1	52.1	28.0	5.6	0.8	8.2	5.1	0.0	99.7
Day 2	25.2	33.5	9.7	1.7	5.0	6.2	1.4	82.7
Day 3	13.8	36.4	14.0	0.7	2.3	6.9	1.7	75.8
Day 7	4.6	20.4	20.2	0.0	0.0	5.4	1.3	51.9
Day 14	4.3	1.2	1.9	2.5	2.4	2.1	5.6	20.0
Day 30	2.3	0.0	0.5	1.6	1.8	0.9	6.6	13.7

[a] Region of diffuse radioactivity containing multiple minor components.

Flonicamid degraded rapidly at 20 °C and 45% WHC_{max} in the loamy sand soil with a DT_{50} of 1 day and a DT_{90} of 3.4 days (r^2 = 0.9960), exhibiting first-order decay kinetics.

Table 29 Degradation of [^{14}C]Flonicamid in soil under aerobic conditions

Soil	DT_{50} [days]	DT_{90} [days]	r^2
loamy sand (Madison, Ohio, USA)	1.0	3.4	0.9960

An effort was made to extract larger quantities of bound residues from the PES and to determine if flonicamid or metabolites were less extracted with time. After acid hydrolysis with 6 N HCl (at ca. 40 °C overnight), the additional radioactivity extracted from the PES represented 37% of the bound residues. Approximately 46% of the released radioactivity was organosoluble. HPLC analysis of the organic phase showed negligible amounts of parent material. The majority of residues released from soil PES were polar in nature. Based on HPLC/LSC of the acid extraction, it was concluded that the unextracted residues remaining after the initial extractions did not contain significant amounts of flonicamid (or its known metabolites).

The major degradates observed exceeding 10% of AR were TFNA and TFNA-OH. TFNG-AM, TFNG and TFNA-AM were detected as minor degradates and were formed as intermediate products. Rapid hydrolysis of the cyano group and the resulting amide group led to the formation of TFNG. Further cleavage of the glycine moiety led to the formation of TFNA. TFNA was apparently rapidly hydroxylated to TFNA-OH by micro-organisms. Mineralisation of the radioactive residues to CO_2 and binding to the soil matrix were the terminal steps in soil metabolism of flonicamid. At the end of the 30-day period, approximately half of the applied dose was mineralized to $^{14}CO_2$ and incorporated into the soil organic matter, primarily into the fulvic acid fraction.

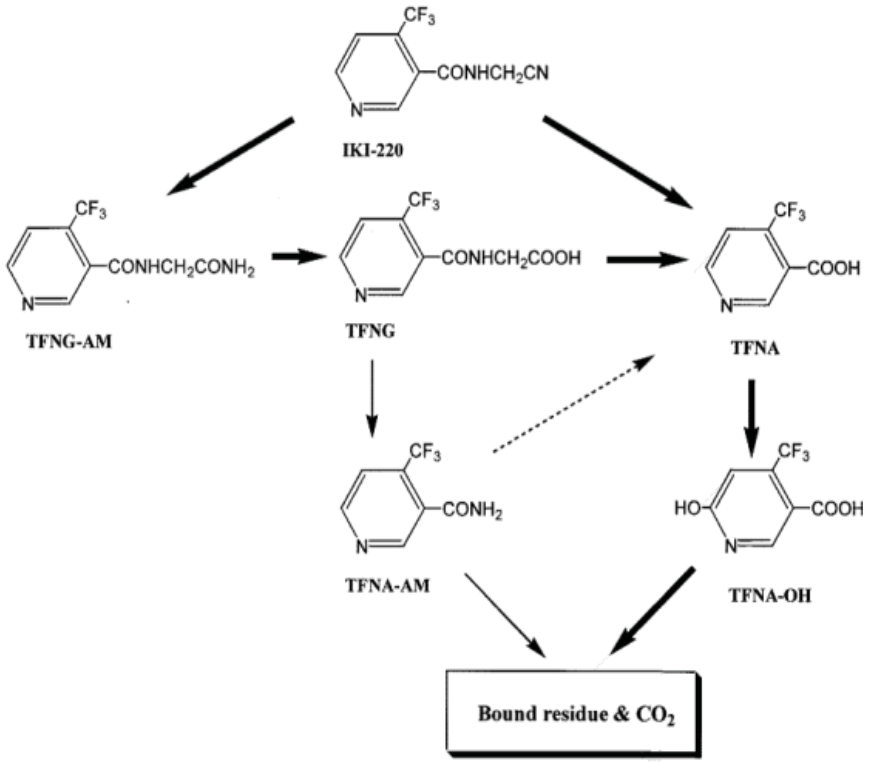


Figure 7 Aerobic degradation pathway in soil

Rate of aerobic degradation in soil

The rate of degradation of [^{14}C]flonicamid, radio-labelled at the 3 position of the pyridine ring (specific activity 9.08 MBq/mg), was investigated in three biologically active soils from the U.K. (Bedfordshire; loamy sand and Birmingham; sandy loam) and one from Germany (LUFA Speyer 2.1; sand) under aerobic conditions at 20 ± 2 °C. In addition, the degradation under aerobic conditions at 10 ± 2 °C was studied in the Bedfordshire soil.

The soil concentration of [^{14}C]flonicamid was 0.1 mg/kg (2 µg/20 g dry soil weight). The moisture content of each subsample was adjusted to approximately 50% of its maximum water holding capacity (MWHC). All three soils were connected to a series of traps to retain any volatiles and maintained in dark environmental chambers at 10 ± 2 °C or 20 ± 2 °C. Soil samples were taken at 0.33, 0.67, 1, 2, 3, 7, 14 and 30 days after treatment.

In UK soils incubated at 20 °C, the total ^{14}C recovery (based on the Day 0 dose) averaged 96% (Table 30). Extracted radioactivity decreased from 98% on Day 0 to 3% after 30 days. For the Bedfordshire soil, unextracted radioactivity reached a maximum of 37.7% by Day 30 and evolution of $^{14}CO_2$ reached 56.6% by Day 30. In Birmingham soil, the PES reached 46.2% by Day 3 and decreased slightly to 43.3% at Day 30 and evolution of $^{14}CO_2$ reached 49.3% by Day 30.

In the German soil, the total ^{14}C recovery (based on the Day 0 dose) averaged 97.2%. Extracted radioactivity decreased from 100.0% on Day 0 to 8.5% at 30 days. The PES reached a maximum of 34.6% by Day 14 and decreased slightly to 29.6% AR by Day 30. Evolution of $^{14}CO_2$ reached 56.2% by Day 30.

In all soils negligible quantities of ^{14}C (< 1%) were detected in the volatile organic compound traps.

Table 30 Mass balance of radioactivity of [^{14}C]flonicamid incubated at 20 °C in three soils (average of duplicates)

Sampling	Bedfordshire					Birmingham					Speyer 2.1				
	Extrac-ted	Unex-tracted	CO_2	VOC	Total	Extrac-ted	Unex-tracted	CO_2	VOC	Total	Extrac-ted	Unex-tracted	CO_2	VOC	Total
	[% of applied radioactivity]														
Day 0	98.1	1.7	NA	NA	99.8	98.4	2.2	NA	NA	100.6	100.0	0.7	NA	NA	100.7
Day 0.33	96.8	3.4	NA	NA	100.2	88.6	8.4	NA	NA	97.0	96.9	2.5	NA	NA	99.4
Day 0.67	90.8	6.3	NA	NA	97.1	81.0	15.8	NA	NA	96.8	96.1	3.9	NA	NA	99.9
Day 1	84.8	12.3	4.1	n.d.	101.2	69.3	23.3	5.3	0.01	97.8	94.5	3.1	1.7	0.01	99.2
Day 2	61.8	19.8	12.4	0.04	94.0	42.6	37.8	14.8	0.1	95.3	84.9	7.7	4.7	0.03	97.3
Day 3	44.1	29.2	21.7	0.08	95.1	21.7	46.2	26.1	0.2	94.1	76.8	9.4	8.5	0.06	94.8
Day 7	8.2	34.1	46.9	0.20	89.4	7.0	46.0	40.8	0.4	94.2	50.2	16.5	25.6	0.24	92.5
Day 14	4.9	36.4	52.3	0.27	93.9	4.6	42.3	45.4	0.5	92.8	13.4	34.6	47.8	0.43	96.2
Day 30	3.2	37.7	56.6	0.31	97.8	2.8	43.3	49.3	0.5	95.9	8.5	29.6	56.2	0.5	94.8

VOC = Volatile organic compounds trapped in ethylene glycol

NA = not analysed

n.d. = Not detected

In Bedfordshire soil incubated at 10 °C, the total ^{14}C recovery (based on the Day 0 dose) from this soil averaged 97.9%. Extracted radio-label decreased from 99.2% on Day 0 to 6.4% after 30 days. The PES reached a maximum of 39.6% and evolution of $^{14}CO_2$ reached 52.4% by Day 30. Negligible quantities of ^{14}C (< 1%) were detected in the volatile organic compounds traps.

Table 31 Mass Balance of radioactivity of [^{14}C]flonicamid incubated at 10 °C in Bedfordshire soil (average of duplicates)

Sampling	Bedfordshire				
	Extracted	Unextracted	CO_2	VOC	Total
	[% of applied dose]				
Day 0	99.2	1.4	NA	NA	100.5
Day 0.33	99.9	2.9	NA	NA	102.8
Day 0.67	95.1	3.9	NA	NA	99.0
Day 1	92.3	4.0	0.7	nd	97.0
Day 2	93.3	5.5	1.9	0.03	100.6
Day 3	82.7	9.4	3.7	0.13	95.8
Day 7	61.9	19.5	12.4	0.15	93.9
Day 14	25.5	32.0	34.8	0.18	92.5
Day 30	6.4	39.6	52.4	0.4	98.8

VOC = Volatile organic compounds trapped in ethylene glycol

NA = Not analysed

Nd = Not detected

[^{14}C]Flonicamid incubated in UK soils at 20 °C declined rapidly from 95% on Day 0 to 0.5% of the applied radioactivity (AR) by Day 14 (Bedfordshire soil) or to 1.5% by Day 7 and non-detectable at Day 14 (Birmingham soil). TFNA rose to a maximum of 19.2–30.6% by Day 1, and then dropped to non-detectable levels by Day 7. TFNA-OH rose to a maximum of 12.1–21.3% by Day 2/3 and declined to non-detectable levels by Day 7. TFNG-AM rose to a maximum of 7.8–9.7% by Day 0.33 and declined to less than 1.0% by Day 3 (Bedfordshire soil) or non-detectable levels by Day 7 (Birmingham soil). Minor metabolites TFNA-AM and TFNG were detected at levels $\leqslant$ 3.7% between Day 0.33 and Day 14. Minor polar components were observed in the HPLC chromatograms at Days 1–14. All polar components were $\leqslant$ 2.2% AR.

In Speyer 2.1 soil, a similar trend was observed where flonicamid decreased from 96.8% of the AD at Day 0 to less than 1% by Day 14. TFNA rose to a maximum of 12.2% by Day 3 then dropped to less than 0.5% of the AD at Day 14. TFNA-OH rose to a maximum of 17.6% by Day 7 and declined to 1.0% of the AD at Day 14. TFNG-AM rose to a maximum of 10.2% by Day 2 and dropped to less than 1% of the applied dose at Day 14. TFNA-AM rose to a maximum of 7.6% at Day 7 and then declined to less than 0.5% by Day 14. Minor metabolite TFNG was detected at levels below 4% between Day 0.33 and Day 14. Several minor polar components were observed in the HPLC chromatograms at Days 2–14. All polar components were less than 7.1% of the applied dose.

Table 32 Distribution of radioactivity in three soils treated with [^{14}C]flonicamid and incubated at 20 °C (average of duplicates)

Sampling [day]	Loamy sand (Bedfordshire)						Sandy loam (Birmingham)						Sand (Speyer 2.1)					
	TFNG-AM	TFNA-AM	TFNG	TFNA-OH	TFNA	Flonicamid	TFNG-AM	TFNA-AM	TFNG	TFNA-OH	TFNA	Flonicamid	TFNG-AM	TFNA-AM	TFNG	TFNA-OH	TFNA	Flonicamid
	[% of applied radioactivity]																	
0	n.d.	2.5 [a]	n.d.	n.d.	n.d.	95.1	n.d.	2.7 [a]	n.d.	n.d.	ND	95.3	n.d.	2.5 [a]	n.d.	n.d.	n.d.	96.8
0.33	9.7	2.9	2.8	2.5	12.1	66.6	7.8	2.5	2.0	3.0	11.6	61.6	4.8	2.4	1.7	n.d.	1.3	86.2
0.67	8.4	3.6	2.6	6.5	22.8	46.6	6.6	2.4	2.0	6.4	17.9	44.9	8.0	3.3	2.3	1.0	4.4	76.7
1	5.6	3.5	2.2	12.4	30.6	29.5	4.9	1.8	1.8	9.7	19.2	29.9	9.8	4.1	3.0	2.0	6.6	68.6
2	1.9	1.6	1.8	20.6	21.8	12.4	2.0	0.3	1.5	12.1	12.7	12.1	10.2	5.5	3.5	5.8	11.3	47.2
3	0.8	0.6	1.8	21.3	13.0	4.9	0.9	0.3	1.6	6.2	4.4	5.1	8.1	7.5	3.4	10.6	12.2	32.9
7	n.d.	0.6	2.9	n.d.	n.d.	1.5	n.d.	n.d.	2.1	n.d.	n.d.	1.5	2.2	7.6	3.9	17.6	6.4	6.6
14	n.d.	n.d.	2.9	n.d.	n.d.	0.5	n.d.	n.d.	2.7	n.d.	n.d.	n.d.	0.7	0.3	3.3	1.0	0.2	0.7
30	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA

[a] TFNA-AM existed as an impurity in the dose test solution at approximately 2.5%

n.d.= Not detected

NA = Not analysed

In Bedfordshire soil incubated at 10 °C parent flonicamid decreased from 96.3% AR at Day 0 to 1.7% by Day 14 (Table 33). TFNA rose to a maximum of 24.3% by Day 3, and then dropped to less than 2% by Day 14. TFNA-OH rose to a maximum of 32.7% by Day 7 and declined to 16.1% at Day 14. TFNG-AM rose to a maximum of 8.1% by Day 0.67 and dropped to less than 1% by Day 14. Minor metabolites TFNA-AM and TFNG were detected at levels below 6% between Day 0.33 and Day 14. Minor polar components were observed in the HPLC chromatograms at Days 2–14. All polar components were less than 2.3% of the administered dose.

Table 33 Distribution of radioactivity in Bedfordshire soil treated with [^{14}C]IKI-220 and incubated at 10 °C (average of duplicates)

Sampling	Loamy sand (Bedfordshire)					
	TFNG-AM	TFNA-AM	TFNG	TFNA-OH	TFNA	Flonicamid
	[% of applied radioactivity]					
Day 0	n.d.	2.6 [a]	n.d.	n.d.	n.d.	96.3
Day 0.33	7.4	3.0	2.4	0.8	5.1	80.6
Day 0.67	8.1	2.9	2.8	2.2	9.3	69.0
Day 1	8.0	3.9	3.9	4.7	15.6	55.6
Day 2	6.6	5.3	3.0	12.3	24.0	41.4
Day 3	4.1	5.4	2.7	20.4	24.3	25.0
Day 7	1.0	4.1	2.5	32.7	11.8	6.7
Day 14	0.5	0.4	2.5	16.1	0.5	1.7
Day 30	NA	NA	NA	NA	NA	NA

[a] TFNA-AM existed as an impurity in the dose test solution at approximately 2.5%

n.d. = Not detected
NA = not analysed

The DT_{50} and DT_{90} values were calculated for each soil set. First-order kinetics were observed for all soils. For the soils incubated at 20 °C the DT_{50} and DT_{90} values ranged from 0.7 to 1.8 days and 2.3 to 6.0 days, respectively. The DT_{50} value was 2.4 days and the DT_{90} value was 7.9 days for the soil incubated at 10 °C. The most rapid degradation kinetics were observed for the Bedfordshire soil and the Birmingham soil at 20 °C. The DT_{50} and DT_{90} values for each soil set are shown in Table 34.

Table 34 Aerobic degradation of flonicamid in soil incubated at 20 °C and 10 °C

Soil		Incubation	DT_{50}	DT_{90}	
Type	Origin	temperature	[days]	[days]	r^2
Loamy sand	Bedfordshire	20 °C	0.7	2.3	0.9898
Sandy loam	Birmingham		0.7	2.4	0.9890
Sand	Speyer 2.1		1.8	6.0	0.9989
Loamy sand	Bedfordshire	10 °C	2.4	7.9	0.9721

TFNA, TFNA-OH and TFNG-AM were the major degradates in all soils over the course of the study which peaked at levels of 12.2 to 30.6%, 12.1 to 32.7% and 7.8 to 10.2% respectively, of the applied radioactivity. Minor degradates TFNG and TFNA-AM were detected at less than 7.7% AR at all sampling points over the course of the study. All of the degradates were metabolised and mineralised to carbon dioxide and immobilised as soil-bound residue.

Soil photolysis

The photochemical degradation of [pyridyl-^{14}C]flonicamid (specific activity 9.08 MBq/mg) was investigated in a loamy sand soil (pH 7.2, 0.98% organic matter, origin Madison, Ohio, USA) under laboratory conditions.

Ten grams (10 g) of dried soil was weighed into each photolysis vessel, made of clear glass, to a depth of approximately 3 mm. The fortification solution of [^{14}C]flonicamid (ca. 50 µL) was added onto the soil surface of each sample jar at a rate of approximately 0.1 mg eq/kg (0.1 µg/g) by means of a syringe. A total of 16 dark control and 16 light exposed samples were prepared. Two additional sample vessels were prepared for each treatment condition at an exaggerated rate (4×) for use in metabolite isolation as required. The temperature of the light-exposed and dark control samples was maintained at 20 ± 1 °C throughout the study. The light exposed and dark control samples were analysed at 0, 1, 3, 7, 9, 11 and 15 days after fortification.

Whilst volatile radioactivity was not trapped, based on the overall recoveries, good material balance was achieved, precluding the requirement for volatile traps.

[^{14}C]Flonicamid decreased from 99.0% of the applied radioactivity (AR) on Day 0 to 59.5% AR after 15 days continuous illumination. Concurrently, the metabolite TFNG-AM was detected in Day 1 sample extracts at 2.9% AR and increased to 29.5% AR by Day 15. TFNA-AM and TFNG were also detected as minor metabolites in the illuminated soils, reaching maximum concentrations of 5.0% (Day 11 and Day 15) and 2.0% AR (Day 15), respectively.

In the dark controls flonicamid decreased from 99.0% of the AR on Day 0 to 80.4% AR after 15 days dark storage. The metabolite TFNG-AM was detected in Day 1 sample extracts at 1.2% AR and increased to 13.8% AR by Day 15 samples. TFNA-AM and TFNG were detected as minor metabolites reaching maximum concentrations of 2.8% (Day 9 and Day 15) and 2.0% AR (Day 15), respectively. The mass balance and ^{14}C distribution of radioactivity from the photochemical degradation of [^{14}C]flonicamid on soil is shown in Table 35.

Table 35 ^{14}C distribution of radioactivity from the photochemical degradation of [^{14}C]flonicamid on soil—light-exposed and dark-control samples

Sampling	Extracted	Flonicamid [a] [% AR]	TFNG-AM [a] [% AR]	TFNA-AM [a] [% AR]	TFNG [a] [% AR]	Bound	Recovery
Exposed							
Day 0	101.3	99.0	–	2.3	–	0.3	101.6
Day 1	97.8	92.0	2.9	2.9	–	0.8	98.5
Day 3	96.5	88.0	5.9	2.6	–	1.2	97.7
Day 7	99.8	79.8	15.0	4.6	0.4	2.0	101.8
Day 9	99.1	77.2	17.0	3.9	1.0	1.7	100.7
Day 11	98.5	69.9	22.0	5.0	1.6	1.7	100.2
Day 15	96.0	59.5	29.5	5.0	2.0	1.7	97.7
Dark							
Day 0	101.3	99.0	–	2.3	–	0.3	101.6
Day 1	99.6	95.8	1.2	2.5	–	0.7	100.3
Day 3	99.4	93.2	3.8	2.3	–	1.1	100.4
Day 7	99.5	88.3	7.8	2.5	1.0	1.2	100.7
Day 9	100.3	86.1	10.1	2.8	1.4	1.2	101.6
Day 11	99.4	85.7	11.1	2.6	–	0.9	100.3
Day 15	98.9	80.4	13.8	2.8	2.0	1.8	100.7

[a] (% ^{14}C in designated fractions) × (% extracted)

Recovery = Extracted plus bound residues

Values are average of duplicate samples

The degradation of flonicamid appears to have followed first order kinetics. A linear regression analysis was performed on the data generated by the HPLC analyses of the samples. The resultant DT_{50} values were 22.4 days for the exposed samples (correlation coefficient (R^2) = 0.9729) and 53.3 days (R^2 = 0.9589) for the dark control samples (Table 36).

Table 36 Calculated values of the DT_{50} from the soil photolysis of flonicamid

	DT_{50} [days]	R^2
Loamy sand (Madison, Ohio, USA)		
exposed	22.4	0.9729
dark controls	53.3	0.9589

Residues in succeeding crops

Confined rotational crop

Flonicamid, radio-labelled at the 3 position of the pyridine ring (specific activity: 9.08 MBq/mg), formulated as a wettable granule (WG) was applied twice to loamy sand soil at a rate equivalent to 100 g ai/ha at an interval of two weeks. Soils were allowed to age under greenhouse conditions after treatment and prior to planting. After the appropriate plant-back intervals (PBIs) of 30, 120 or 360 days, the rotational crops, representative of the root vegetable (carrot), small grain (wheat), and leafy vegetable (lettuce) crop groups, were planted.

The crop samples of lettuce, carrot and wheat (forage, straw, chaff and grain) were homogenised with dry ice and analysed for total ^{14}C residues by combustion analysis. The PES were also analysed by combustion analysis. The TRR combustion data for crop samples are summarized in Table 37.

Table 37 Distribution of TRR levels of harvested crops

Plant-back Interval (days)	Plant part	TRR (mg/kg)	%TRR			
			Extracted	Unextracted	Total Identified	Total Characterized
30	Immature lettuce	0.006	76.4	23.6	45.4	16.5
	Mature lettuce	0.004	59.9	40.1	36.1	20.1
	Immature carrot	0.011	78.4	21.7	Not reported	
	Mature carrot root	0.004	71.6	28.4	48.7	19.3
	Mature carrot foliage	0.019	73.5	26.5	50.8	18.9
	Wheat forage	0.077	92.6	7.4	85.6	3.1
	Wheat straw	0.140	77.7	22.3	62.1	9.8
	Wheat chaff	0.078	82.2	17.8	63.3	12.8
	Wheat grain	0.029	81.5	18.6	73.3	3.9
120	Immature lettuce	0.004	Not extracted			
	Mature lettuce	0.004	Not extracted			
	Immature carrot	0.006	65.9	34.1	48.2	11.0
	Mature carrot root	0.003	Not extracted			
	Mature carrot foliage	0.005	55.0	45.0	39.1	12.1
	Wheat forage	0.009	80.8	19.2	70.1	7.8
	Wheat straw	0.031	73.0	27.0	54.7	9.5
	Wheat chaff	0.023	67.2	32.8	55.9	6.7
	Wheat grain	0.010	67.2	32.9	59.8	2.5
360	Immature lettuce	0.002	n.e.	n.e.	Not analysed	
	Mature lettuce	0.001	n.e.	n.e.		
	Immature carrot	0.003	n.e.	n.e.		
	Mature carrot root	< 0.001	n.e.	n.e.		
	Mature carrot foliage	0.002	n.e.	n.e.		
	Wheat forage	0.007	40.8	59.1		
	Wheat straw	0.017	43.6	56.4		
	Wheat chaff	0.013	47.5	52.5		
	Wheat grain	0.005	27.7	72.3		

Table 38 Identification/Characterization of TRRs

Plant-back Interval (days)	Plant part	%TRR (mg/kg in parentheses)						
		Flonicamid	TFNA	TFNA-OH	TFNG	TFNA-AM	TFNG-AM	Unknowns
30	Immature lettuce	8.9 (0.0)	8.2 (0.0)	4.9 (0.0)	10.0 (0.0)	4.4 (0.0)	9.0 (0.0)	16.5 (0.001)
	Mature lettuce	5.7 (0.0)	1.8 (0.0)	2.0 (0.0)	2.7 (0.0)	3.7 (0.0)	20.2 (0.001)	20.1 (0.001)
	Immature carrot	Not reported						
	Mature carrot root	2.3 (0.0)	1.0 (0.0)	1.9 (0.0)	4.9 (0.0)	15.4 (0.001)	23.2 (0.001)	19.3 (0.001)
	Mature carrot foliage	5.2 (0.001)	2.0 (0.0)	2.2 (0.0)	7.7 (0.001)	7.9 (0.002)	25.8 (0.005)	18.9 (0.004)
	Wheat forage	3.3 (0.003)	11.4 (0.010)	37.8 (0.033)	15.0 (0.013)	6.9 (0.006)	11.2 (0.010)	3.1 (0.003)
	Wheat straw	4.5 (0.007)	2.5 (0.004)	9.6 (0.023)	15.4 (0.023)	8.6 (0.013)	21.5 (0.032)	9.8 (0.014)
	Wheat chaff	4.1 (0.003)	9.4 (0.008)	9.1 (0.008)	18.3 (0.015)	7.5 (0.006)	14.9 (0.013)	12.8 (0.011)
	Wheat grain	5.1 (0.001)	19.6 (0.005)	4.7 (0.001)	36.3 (0.010)	2.2 (0.001)	5.4 (0.002)	3.9 (0.001)
120	Immature	Not further analysed						

Plant-back Interval (days)	Plant part	%TRR (mg/kg in parentheses)						
		Flonicamid	TFNA	TFNA-OH	TFNG	TFNA-AM	TFNG-AM	Unknowns
	lettuce							
	Mature lettuce	Not further analysed						
	Immature carrot	4.7 (0.0)	4.6 (0.0)	4.0 (0.0)	11.0 (0.001)	7.1 (0.0)	16.8 (0.001)	11.0 (0.001)
	Mature carrot root	Not further analysed						
	Mature carrot foliage	1.6 (0.0)	4.0 (0.0)	2.1 (0.0)	7.9 (0.0)	10.8 (0.001)	12.7 (0.001)	12.1 (0.001)
	Wheat forage	10.5 (0.001)	4.5 (0.0)	6.2 (0.001	20.9 (0.002)	8.0 (0.001)	19.9 (0.002)	7.8 (0.001)
	Wheat straw	1.4 (0.0)	2.3 (0.001)	4.4 (0.001)	10.9 (0.004)	10.7 (0.003)	25.0 (0.006)	9.5 (0.003)
	Wheat chaff	5.9 (0.001)	1.8 (0.0)	6.4 (0.002)	15.9 (0.004)	8.2 (0.002)	17.7 (0.004)	6.7 (0.002)
	Wheat grain	12.9 (0.001)	8.6 (0.001)	1.0 (0.0)	32.1 (0.003)	3.0 (0.0)	3.2 (0.0)	2.5 (0.0)

The homogenized tissue samples were extracted three times with acetonitrile/water 40:60 v/v (0.1% phosphoric acid). Each extract was centrifuged or vacuum filtered. The PES was allowed to air dry and then was subjected to combustion analysis. The solvent extracts were pooled then reduced to a small volume by rotary evaporation under reduced pressure. If sufficient residue was detected in the extract or the PES, additional analysis was conducted by HPLC-LSC to characterize the nature of the ^{14}C residue present.

TRRs in all raw agricultural commodities (RACs) declined with prolonged PBIs such that, at the 120-day PBI, no further characterization/identification of the TRRs was performed for immature and mature lettuce and mature carrot roots due to the low total radioactivity. Further to this, at the 360-day PBI, none of the TRRs from any of the crop parts were further subjected to characterization/identification as these were too low.

Overall, extraction of the TRRs with organic solvents released greater than 55% of the TRRs. In most commodities, only small amounts of flonicamid and TFNA-OH were detected with TFNG and TFNG-AM identified as major metabolites. In wheat grain, TFNA was also observed as a major metabolite while in wheat forage, TFNA and TFNA-OH accounted for greater than 10% of the TRRs. TFNA-AM was the only predominant metabolite in mature carrot root.

Field rotational crop

At each of the six field trials conducted in the US, three applications of flonicamid 50WG were made to the primary crop (cotton) at the maximum rate of 0.1 kg ai/ha at 7 ± 1 day intervals, resulting in a total seasonal application rate of approximately 0.31 kg ai/ha. Following harvest of the treated cotton, the rotational crops, wheat (four sites) and turnips (two sites), were planted at 30 and 60 days following the last application. The wheat and turnip samples were taken for analysis at normal maturity for each crop matrix.

Aliquots of homogenised samples were extracted twice with acetonitrile:water (50/50, v/v). Concentrated HCl was added to the combined extracts prior to being filtered and partitioned with ethyl acetate (twice). The combined ethyl acetate extract was evaporated just to dryness and residues taken up in acetonitrile:water (30/70, v/v) and analysed by LC-MS/MS. For wheat straw, an additional SPE clean-up step using a C_{18} cartridge before the partitioning with ethyl acetate was inserted.

Table 39 Maximum residues in rotated crop samples 30 and 60 days following the last application of Flonicamid 50WG to cotton

	Plant back interval [days]	Flonicamid [mg/kg]	TFNG [mg/kg]	TFNA [mg/kg]	TFNA-AM [mg/kg]
Wheat forage	30–32	n.d.	n.d.	< LOQ (< 0.01)	n.d.
Wheat straw	30–32	n.d.	< LOQ (< 0.02)	n.d.	n.d.
Wheat grain	30–32	n.d.	n.d.	< LOQ (< 0.01)	n.d.
Turnip tops	30	n.d.	n.d.	n.d.	n.d.
Turnip roots	30	n.d.	n.d.	n.d.	n.d.
Wheat forage	58–63	n.d.	n.d.	n.d.	n.d.
Wheat straw	58–63	n.d.	n.d.	n.d.	n.d.
Wheat grain	58–63	n.d.	n.d.	n.d.	n.d.
Turnip tops	59–60	n.d.	n.d.	n.d.	n.d.
Turnip roots	59–60	n.d.	n.d.	n.d.	n.d.

n.d. = Not detected

LOD = 0.005 mg eq/kg

No quantifiable residues of flonicamid or its metabolites TFNG, TFNA, and TFNA-AM were detected in any crop matrix in any rotational crop planted at either 30 or 60 days after the last application of flonicamid to the primary crop of cotton.

RESIDUE ANALYSIS

Analytical Methods

The Meeting received descriptions and validation data for analytical methods for residues of flonicamid and its metabolites TFNA-AM, TFNA and TFNG in plant commodities and flonicamid, TFNA-AM, TFNA, TFNG and OH-TFNA-AM in animal commodities. All residue analytical methods rely on LC-MS/ MS. Typical LOQs achieved for plant and animal commodities fall in the range of 0.01–0.02 mg/kg. The LOQs for milk and animal products (liver, kidney, muscle, eggs) were 0.01 mg/kg for each analyte. Methods have been subjected to independent laboratory validation. The methods described briefly below have been used for the analysis of the samples generated during the supervised field trials, processing studies and storage stability investigations.

Table 40 Characterization of Enforcement Analytical Methods for Plant and Animal Commodities

Method ID	Method Type	Detector	Analytes	LOQ/analyte	Matrices	Report
Plant Commodities						
P-3561M	Enforcement	HPLC-MS/MS	Flonicamid TFNA-AM TFNA TFNG	0.01 mg/kg for peach and potato tuber 0.02 mg/kg for wheat straw	Peach Potato tuber Wheat straw	IB-2002-JLW-011-00
	ILV	HPLC-MS/MS	Flonicamid TFNA-AM TFNA TFNG	0.01 mg/kg	Cottonseed	02-0031
P-3822	Enforcement	HPLC-MS/MS	Flonicamid TFNA-AM TFNA TFNG	0.01 mg/kg all matrices 0.02 mg/kg wheat straw and cotton matrices	Various RACs and processed commodities	178MVL05 R1
	ILV	HPLC-MS/MS	Flonicamid TFNA-AM TFNA TFNG	0.01 mg/kg	Various RACs and processed commodities	
AGR/MOA/IKI220-1 v.1	Enforcement	HPLC-MS/MS	Flonicamid TFNA-AM	0.01 mg/kg	Lemon Potato	ISK/IKI/06001

Method ID	Method Type	Detector	Analytes	LOQ/analyte	Matrices	Report
			TFNA TFNG		Oilseed rape Wheat grain Plum Prune	
	ILV	HPLC-MS/MS	Flonicamid TFNA-AM TFNA TFNG	0.01 mg/kg	Lemon Potato Oilseed rape Wheat grain Plum Prune	S09-01231
Animal Commodities						
842993	Enforcement	HPLC-MS/MS	Flonicamid TFNA TFNA-AM OH-TFNA-AM TFNG	0.01 mg/kg	Milk	
844743	Enforcement	HPLC-MS/MS	Flonicamid TFNA TFNA-AM OH-TFNA-AM TFNG	0.01 mg/kg	Bovine muscle, liver, kidney, fat Poultry muscle, liver, fat and eggs	
P-3581	ILV	HPLC-MS/MS	Flonicamid TFNA TFNA-AM OH-TFNA-AM TFNG	0.01 mg/kg	Eggs	178ILV02R1
ADPEN-2K2-1126	ILV	HPLC-MS/MS	Flonicamid TFNA TFNA-AM OH-TFNA-AM TFNG	0.01 mg/kg	Beef muscle	
AGR/MOA/IKI-5	Enforcement	HPLC-MS/MS	Flonicamid TFNA-AM	0.01 mg/kg	Bovine muscle, fat and liver, milk and eggs	S12-04426
P-2960	ILV	HPLC-MS/MS	Flonicamid TFNA-AM	0.01 mg/kg	Bovine muscle, fat and liver, milk and eggs	

Plant Commodities

Method P-3561M

Residues of flonicamid and its metabolites TFNG, TFNA and TFNA-AM were extracted twice with acetonitrile/deionised water (1/1, v/v). After centrifugation, the extracts were combined and evaporated until dryness. The sample extract was then acidified and filtered. In the case of wheat straw, the sample extract underwent clean-up using a C_{18} SPE column eluted with acetonitrile/deionised water (1/4, v/v). The eluate (in the case of wheat straw sample) or the filtrate (in the case of potato tuber or peach sample) was liquid-liquid partitioned twice in ethyl acetate. The ethyl acetate layer was evaporated to near dryness and diluted in acetonitrile/deionised water (3/7, v/v) before quantification by HPLC-MS/MS.

The method underwent successful inter-laboratory validation by EN-CAS laboratories using cottonseed. Average recoveries of flonicamid, TFNG, TFNA and TFNA-AM ranged from 70–110% with RSD of $\leqslant$ 16%, demonstrating good reproducibility.

Method P-3822

The HPLC-MS/MS method P3822 quantifies residues of flonicamid and its metabolites TFNG, TFNA and TFNA-AM in raw agricultural commodities and processed commodities. The extraction and clean-up steps of method P-3822 are very similar to those of P-3561M, however; for oily crop samples (e.g. cotton matrices and potato chips), an additional hexane partition step and acidification is included before filtration.

The method underwent successful inter-laboratory validation by EN-CAS laboratories using the same commodities. Average recoveries of flonicamid, TFNG, TFNA and TFNA-AM ranged from 70–110% with RSD of $\leqslant$ 16% (with the exception of pepper/TFNG at 0.1 mg/kg where the RSD was 33%), demonstrating overall good reproducibility.

AGR/MOA/IKI220-1 v.1

Flonicamid and its major metabolites were extracted with a mixture of acetonitrile/water/acetic acid (60/40/0.1, v/v/v), followed by a washing with hexane (except for potato) and a clean-up using a C_{18} phase SPE cartridge (except for potato and lemon), and followed by a liquid/liquid partition with ethyl acetate. After evaporation to dryness, the residues of flonicamid and its major metabolites were dissolved in a mixture of acetonitrile/water (30/70, v/v) prior to analysis by HPLC-MS/MS using two mass transitions. For oil-seed rape, the TFNA results were confirmed by the use of another liquid chromatographic column.

The method underwent successful inter-laboratory validation by Eurofins laboratories using the same commodities. Average recoveries of flonicamid, TFNG, TFNA and TFNA-AM ranged from 70–110% with RSD of $\leqslant$ 20%, demonstrating good reproducibility.

AATM-R-165

Residues of flonicamid and its metabolites were extracted by shaking with acetonitrile:water (1:1). The extract was then decanted and the extraction was repeated with acetonitrile. An aliquot of the combined acetonitrile extracts was diluted with water, filtered and analysed by ultra performance liquid chromatography (UPLC) with positive-ion electrospray ionization (ESI) tandem mass spectrometry (MS/MS). Quantitation of the analytes was achieved by comparison with mixed external standards of flonicamid and its metabolites.

Due to the nature of the cottonseed oil samples, the method was modified such that cottonseed oil was dissolved in hexane and then partitioned with acetonitrile. An aliquot of the acetonitrile layer was taken and diluted with water and analysed.

Determination of Residues of IKI-220 and its Metabolites TFNG, TFNA and TFNA-AM in Various Crops—Validation of the Method"

This method was used for the freezer storage stability study A-22-00-03 whereby residues of flonicamid and its metabolites TFNG, TFNA and TFNA-AM were extracted with methanol. After filtration, the sample solution was washed with n-hexane, concentrated and then cleaned-up on a C_{18} SPE cartridge. The eluate was evaporated to dryness and methylated with diazomethane/diethylether. After concentration, the residues were liquid-liquid partitioned twice in ethyl acetate. The ethyl acetate layer was filtered through anhydrous sodium sulphate and evaporated to dryness. The residues were purified on a Florisil SPE cartridge and reconstituted in acetone prior to analysis by GC/MSD.

Animal Commodities

Method 842993

Residues of flonicamid and its metabolites TFNA, TFNA-AM, OH-TFNA-AM and TFNG in milk samples were extracted twice with ethanol/water (4/4, v/v). The sample extracts were combined and evaporated to dryness prior to liquid-liquid partitioning twice with hexane. The aqueous phase was evaporated to dryness and the residue was dissolved in water/acetonitrile/trifluoroacetic acid (90/10/0.1, v/v/v) prior to HPLC-MS/MS analysis.

Method 844743

Bovine and Poultry Tissues

Residues of flonicamid and its metabolites TFNA, TFNA-AM, OH-TFNA-AM and TFNG were extracted twice with acetonitrile/water (8/2, v/v). After addition of silicon anti foaming agent to the combined extracts of each tissue/egg sample, the solution was evaporated to dryness. The residue was then dissolved in methanol/water/acetic acid (2000/500/15, v/v/v) prior to liquid-liquid partitioning twice with hexane. The aqueous phase was evaporated to dryness and the residue was dissolved in water/acetonitrile/trifluoroacetic acid (90/10/0.1, v/v/v) prior to clean-up using gel permeation chromatography (GPC). The eluate was evaporated to dryness and redissolved in methanol/water (1/9, v/v) and subject to HPLC-MS/MS analysis.

Eggs

Residues of flonicamid and its metabolites TFNA, TFNA-AM, OH-TFNA-AM and TFNG in egg samples were extracted with acetonitrile/water (8:2; v/v) and the suspension was treated at 60 °C for 1 h. After denaturisation, the process was repeated. The extracts were subsequently combined and partitioned by a liquid-liquid extraction with cyclohexane. The acetonitrile/water phase was evaporated to dryness and the residue was dissolved in methanol/water/acetic acid (2000:500:15; v/v/v) followed by clean-up using GPC. The eluate was evaporated to dryness and the residue was re-dissolved with 30% acetonitrile in water prior to analysis using HPLC-MS/MS.

The method underwent successful inter-laboratory validation by FMC Princeton Environmental Sciences Laboratory using poultry eggs. Average recoveries of flonicamid, TFNG, TFNA and TFNA-AM ranged from 77–108% with RSD of $\leqslant$ 21%, demonstrating good reproducibility.

AGR/MOA/IKI-5

Residues of flonicamid and its metabolite TFNA-AM in samples of bovine, muscle, fat, liver, milk and eggs were extracted with acidified acetonitrile/water (80/20, v/v). All the contents of the dispersive SPE citrate extraction tube were added to the extracts and shaken vigorously by hand, vortexed and centrifuged. The supernatant was evaporated to dryness and residues were dissolved in acetonitrile/ water (10/90, v/v) and filtered (liver only) prior to analysis by HPLC-MS/MS.

The method underwent successful inter-laboratory validation by PTRL Europe using the same commodities. Average recoveries of flonicamid and TFNA-AM were within the range of 70–110% with RSD of $\leqslant$ 20%, demonstrating good reproducibility.

P-3580

While the Meeting did not receive a description of the method P-3580 titled `Radio-validation of Goat Muscle Treated with $^{14}$C-Radio-labelled IKI-220 (F1785) Insecticide and method Validation of Residue Methodology for IKI-220 (F1785) and its Major Metabolites in/on Cow Muscle, Kidney and Liver`, the results of the ILV performed by ADPEN Laboratories was provided, and the method was subsequently renumbered to P-3581. Average recoveries of flonicamid and its metabolites TFNA, TFNA-AM, OH-TFNA-AM and TFNG in bovine muscle were within the range of 82–110% with RSD of $\leq$ 17%, demonstrating good reproducibility.

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 separately from routine sample analysis. These method recovery data, for plant and animal commodities are summarized in Table 41.

Table 41 Method recovery data of flonicamid and metabolites in plants and animal products

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
Plant Commodities								
Wheat Straw	Flonicamid	0.02	3	83–127	109.7	21.4	P-3561M	IB-2002-JLW-011-00
		0.04	3	107–128	114.7	10.1		
		0.1	3	105–122	114	7.5		
	TFNG	0.02	3	66–96	78.3	20		
		0.04	3	72–88	81.3	10.2		
		0.1	3	80–97	86.7	10.5		
	TFNA	0.02	3	84–109	92.3	15.6		
		0.04	3	63–95	83.7	21.4		
		0.1	3	86–110	97.3	12.4		
	TFNA-AM	0.02	3	61–80	70.3	13.5		
		0.04	3	63–71	66	6.6		
		0.1	3	77–86	80.3	6.1		
Peach	Flonicamid	0.01	3	95–113	105.3	8.8		
		0.02	3	105–108	106.3	1.4		
		0.05	3	102–106	104	1.9		
	TFNG	0.01	3	74–95	84.7	12.4		
		0.02	3	96–99	97	1.8		
		0.05	3	102–110	106	3.8		
	TFNA	0.01	3	92–97	94.7	2.7		
		0.02	3	94–108	102.3	7.2		
		0.05	3	105–107	106	0.9		
	TFNA-AM	0.01	3	88–96	93.3	4.9		
		0.02	3	97–103	100	3.0		
		0.05	3	97–98	97.7	0.6		
Potato Tuber	Flonicamid	0.01	3	92–108	98	8.9		
		0.02	3	98–109	103.7	5.3		
		0.05	3	82–106	95.7	12.9		
	TFNG	0.01	3	82–107	91.7	14.6		
		0.02	3	80–95	86.7	8.8		
		0.05	3	74–87	78.7	9.2		
	TFNA	0.01	3	102–115	108	6.1		
		0.02	3	107–122	114.7	6.5		
		0.05	3	93–106	98.3	6.9		
	TFNA-AM	0.01	3	80–98	92	11.3		
		0.02	3	83–89	86.3	3.5		
		0.05	3	73–85	79.3	7.6		
Apple	Flonicamid	0.05	1	84	NA	NA	P-3822	IB-2001-MDG-003
		0.1	4	90–100	94	4.5		
		0.2	1	102	NA	NA		
	TFNA-AM	0.05	1	76	NA	NA		
		0.1	4	73–80	75.7	3		
		0.2	1	90	NA	NA		
	TFNA	0.05	1	79	NA	NA		
		0.1	4	79–86	82.5	3.1		
		0.2	1	96	NA	NA		
	TFNG	0.05	1	69	NA	NA		
		0.1	4	67–77	72.5	5.3		
		0.2	1	90	NA	NA		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
Apple Juice	Flonicamid	0.25	5	90–113	96.2	9.6		01LJL045C
		0.5	1	125	NA	NA		
	TFNA-AM	0.25	5	72–86	78.6	5.4		
		0.5	1	97	NA	NA		
	TFNA	0.25	5	89–105	96.4	6.9		
		0.5	1	136	NA	NA		
	TFNG	0.25	5	65–115	86.4	18.1		
		0.5	1	133	NA	NA		
Apple Pomace	Flonicamid	0.01	2	91	NA	NA		IB-2001-MDG-003
		0.2	2	79–99	89	NA		
	TFNA-AM	0.01	2	75–84	79.5	NA		
		0.2	2	76–89	82.5	NA		
	TFNA	0.01	2	81–83	82	NA		
		0.2	2	100–112	106	NA		
	TFNG	0.01	2	73–107	90	NA		
		0.2	2	104–115	109.5	NA		
Pear	Flonicamid	0.01	1	92	NA	NA		IB-2001-MDG-003
		0.2	2	94–95	94.5	NA		
	TFNA-AM	0.01	1	75	NA	NA		
		0.2	2	74–86	80	NA		
	TFNA	0.01	1	97	NA	NA		
		0.2	2	93–94	93.5	NA		
	TFNG	0.01	1	65	NA	NA		
		0.2	2	73–88	80.5	NA		
Peach	Flonicamid	0.01	3	95–113	105.3	9.3		01LJL071C
		0.02	3	105–108	106.3	1.5		
		0.05	3	102–106	104	2		
	TFNA-AM	0.01	3	88–96	93.3	4.6		
		0.02	3	97–103	100	3		
		0.05	3	97–98	97.7	0.6		
	TFNA	0.01	3	92–97	94.7	2.5		
		0.02	3	97–103	102.3	7.4		
		0.05	3	105–107	106	1		
	TFNG	0.01	3	74–95	84.7	10.5		
		0.02	3	96–99	97	1.7		
		0.05	3	102–110	106	4		
Peach	Flonicamid	0.01	1	97	NA	NA		IB-2001-MDG-005
		0.05	2	107–111	109	NA		
		0.2	1	89	NA	NA		
		0.4	1	108	NA	NA		
	TFNA-AM	0.01	1	100	NA	NA		
		0.05	2	106–112	109	NA		
		0.2	1	82	NA	NA		
		0.4	1	102	NA	NA		
	TFNA	0.01	1	99	NA	NA		
		0.05	2	93–97	95	NA		
		0.2	1	97	NA	NA		
		0.4	1	93	NA	NA		
	TFNG	0.01	1	92	NA	NA		
		0.05	2	98–127	112.5	NA		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.2	1	85	NA	NA		
		0.4	1	120	NA	NA		
Cherry	Flonicamid	0.01	2	95–115	105	NA	P-3822	IB-2001-MDG-005
		0.5	2	85–92	88.5	NA		
	TFNA-AM	0.01	2	92–114	103	NA		
		0.5	2	76–77	76.5	NA		
	TFNA	0.01	2	87–109	98	NA		
		0.5	2	93–106	99.5	NA		
	TFNG	0.01	2	99–119	109	NA		
		0.5	2	77–89	83	NA		
Plum	Flonicamid	0.05	1	85	NA	NA		IB-2001-MDG-005
		0.1	1	83	NA	NA		
		0.2	1	86	NA	NA		
		0.01	2	103–109	106	NA		
		1	1	102	NA	NA		
	TFNA-AM	0.05	1	85	NA	NA		
		0.1	1	74	NA	NA		
		0.2	1	82	NA	NA		
		0.01	2	103–107	105	NA		
		1	1	92	NA	NA		
	TFNA	0.05	1	78	NA	NA		
		0.1	1	84	NA	NA		
		0.2	1	88	NA	NA		
		0.01	2	110	110	NA		
		1	1	106	NA	NA		
	TFNG	0.05	1	77	NA	NA		
		0.1	1	74	NA	NA		
		0.2	1	74	NA	NA		
		0.01	2	96–111	103.5	NA		
		1	1	101	NA	NA		
Prune	Flonicamid	0.01	2	100–109	104.5	NA		IB-2001-MDG-005
		0.5	2	80–90	85	NA		
	TFNA-AM	0.01	2	92–98	95	NA		
		0.5	2	79–82	80.5	NA		
	TFNA	0.01	2	94–105	99.5	NA		
		0.5	2	73–82	77.5	NA		
	TFNG	0.01	2	98–100	99	NA		
		0.5	2	76–79	77.5	NA		
Pepper	Flonicamid	0.01	3	78–99	90	11		IB-2001-MDG-006
		0.05	1	65	NA	NA		
		0.1	1	77	NA	NA		
		0.2	1	87	NA	NA		
		0.5	1	81	NA	NA		
		1	1	104	NA	NA		
	TFNA-AM	0.01	3	68–79	74	6		
		0.05	1	79	NA	NA		
		0.1	1	82	NA	NA		
		0.2	1	64	NA	NA		
		0.5	1	67	NA	NA		
		1	1	67	NA	NA		
	TFNA	0.01	3	81–101	90	8		
		0.05	1	83	NA	NA		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.1	1	94	NA	NA		
		0.2	1	89	NA	NA		
		1	1	96	NA	NA		
	TFNG	0.01	3	63–124	101	33		
		0.05	1	87	NA	NA		
		0.1	1	84	NA	NA		
		0.2	1	66	NA	NA		
		0.5	1	66	NA	NA		
		1	1	77	NA	NA		
Tomato	Flonicamid	0.01	1	131	NA	NA	P-3822	IB-2001-MDG-006/
		0.1	4	88–93	90	2.2		
		1.5	1	96	NA	NA		
	TFNA-AM	0.01	1	80	NA	NA		
		0.1	4	61–76	69.3	6.2		
		1.5	1	88	NA	NA		
	TFNA	0.01	1	136	NA	NA		
		0.1	4	80–98	89.3	7.4		
		1.5	1	90	NA	NA		
	TFNG	0.01	1	74	NA	NA		
		0.1	4	67–80	72.5	5.4		
		1.5	1	89	NA	NA		
Tomato	Flonicamid	0.05	1	87	NA	NA		CA147-A
		0.25	1	102	NA	NA		
		0.5	5	82–111	93.2	11.3		
	TFNA-AM	0.05	1	74	NA	NA		
		0.25	1	79	NA	NA		
		0.5	5	73–89	80.8	7.4		
	TFNA	0.05	1	84	NA	NA		
		0.25	1	102	NA	NA		
		0.5	5	89–122	101.8	13.8		
	TFNG	0.05	1	74	NA	NA		
		0.25	1	112	NA	NA		
		0.5	5	82–120	91.8	16		
Tomato Paste	Flonicamid	0.01	2	75–85	80	NA		CA137-S
		1	2	92–93	92.5	NA		
	TFNA-AM	0.01	2	77–84	80.5	NA		
		1	2	84–85	84.5	NA		
	TFNA	0.01	2	103–111	107	NA		
		1	2	100–101	100.5	NA		
	TFNG	0.01	2	90–92	91	NA		
		1	2	113–115	114	NA		
Tomato Puree	Flonicamid	0.01	2	81–85	83	NA		CA137-V
		0.5	2	97–100	98.5	NA		
	TFNA-AM	0.01	2	70–76	73	NA		
		0.5	2	82–85	83.5	NA		
	TFNA	0.01	2	66–71	68.5	NA		
		0.5	2	93–94	93.5	NA		
	TFNG	0.01	2	66–68	67	NA		
		0.5	2	101–109	105	NA		
Potato Tuber	Flonicamid	0.01	4	92–108	98.8	7.3	P-3822	01JRA/IB-2001-MDG-002
		0.02	3	98–109	103.7	5.5		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.05	5	77–106	89.8	12.2		
		0.1	5	78–102	89	10		
		0.4	1	100	NA	NA		
	TFNA-AM	0.01	4	80–98	89.3	10.1		
		0.02	3	80–89	85.3	4.7		
		0.05	5	63–85	75.2	8.3		
		0.1	5	64–97	79.2	13.8		
		0.4	1	86	NA	NA		
	TFNA	0.01	4	99–115	105.8	7		
		0.02	3	107–122	114.7	7.5		
		0.05	5	73–106	88.8	14		
		0.1	5	82–96	88.6	5.5		
		0.4	1	86	NA	NA		
	TFNG	0.01	4	68–107	85.8	16.1		
		0.02	3	80–95	86.7	7.6		
		0.05	5	69–87	77	6.8		
		0.1	5	64–80	72.2	6.1		
		0.4	1	91	NA	NA		
Potato Tuber	Flonicamid	0.25	5	92–112	103.8	8.4		01JFC667C
		0.5	5	92–124	106.6	15.6		
	TFNA-AM	0.25	5	75–105	86	11.9		
		0.5	5	71–102	88	14.4		
	TFNA	0.25	5	85–119	103.4	12		
		0.5	5	86–104	95	7.7		
	TFNG	0.25	5	78–102	91	9.2		
		0.5	5	86–104	95	7.7		
Potato Flakes	Flonicamid	0.01	2	78–86	82	NA		IB-2001-MDG-002
		0.2	1	96	NA	NA		
		0.5	2	63–89	76	NA		
	TFNA-AM	0.01	2	83–89	86	NA		
		0.2	1	87	NA	NA		
		0.5	2	76–80	78	NA		
	TFNA	0.01	2	91–102	96.5	NA		
		0.2	1	92	NA	NA		
		0.5	2	76–91	83.5	NA		
	TNFG	0.01	2	100–109	104.5	NA		
		0.2	1	87	NA	NA		
		0.5	2	90–95	92.5	NA		
Potato Wet Peel	Flonicamid	0.01	2	101–117	109	NA		IB-2001-MDG-002
		0.1	2	103–117	110	NA		
	TFNA-AM	0.01	2	86–98	92	NA		
		0.1	2	84–98	91	NA		
	TFNA	0.01	2	69–77	73	NA		
		0.1	2	97–113	105	NA		
	TFNG	0.01	2	64–85	74.5	NA		
		0.1	2	90–102	96	NA		
Potato Chips	Flonicamid	0.01	2	71–100	85.5	NA		IB-2001-MDG-002
		0.2	2	100–101	100.5	NA		
	TFNA-AM	0.01	2	83–86	84.5	NA		
		0.2	2	90–92	91	NA		
	TFNA	0.01	2	87–90	88.5	NA		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.2	2	100–103	101.5	NA		
	TFNG	0.01	2	72–73	72.5	NA		
		0.2	2	110–117	103.5	NA		
Cottonseed	Flonicamid	0.02	2	113–118	115.5	NA		IB-2001-MDG-004/99AWC
		0.05	1	76	NA	NA		
		0.1	3	79–105	88.7	14.2		
		0.2	4	78–112	90.5	15		
		0.25	5	78–97	85	7.4		
		0.5	8	84–128	98.5	14.3		
		1	1	93	NA	NA		
	TFNA-AM	0.02	2	78–81	79.5	NA		
		0.05	1	69	NA	NA		
		0.1	3	72–102	84	15.9		
		0.2	4	74–93	80.5	9		
		0.25	5	65–86	74.6	8.7		
		0.5	8	66–104	83.5	14.6		
		1	1	82	NA	NA		
	TFNA	0.02	2	115–128	121.5	11.6		
		0.1	3	81–103	90.7	11.2		
		0.05	1	68	NA	NA		
		0.2	4	77–101	89.3	11.6		
		0.25	5	81–95	89.2	5.6		
		0.5	8	66–120	101	17.6		
		1	1	86	NA	NA		
	TFNG	0.02	2	97–100	98.5	NA		
		0.1	3	69–100	85	15.5		
		0.05	1	71	NA	NA		
		0.2	4	69–97	81.5	12.6		
		0.25	5	71–91	82	7.2		
		0.5	8	77–111	92.9	12.6		
		1	1	83	NA	NA		
Cotton Meal	Flonicamid	0.02	2	81–93	87	NA		IB-2001-MDG-004/99AWC
		0.25	5	73–122	97.6	18		
		0.5	1	99	NA	NA		
		2	2	81–88	84.5	NA		
	TFNA-AM	0.02	2	94–117	105.5	NA		
		0.25	5	67–78	73.2	5.4		
		0.5	1	72	NA	NA		
		2	2	78–79	78.5	NA		
	TFNA	0.02	2	79–119	99	NA		
		0.25	5	76–99	83.2	9.4		
		0.5	1	77	NA	NA		
		2	2	118–123	120.5	NA		
	TFNG	0.02	2	73–108	90.5	NA		
		0.25	5	69–85	79	6.8		
		0.5	1	81	NA	NA		
		2	2	117	117	NA		
Cotton Hulls	Flonicamid	0.02	2	70–90	80	NA		IB-2001-MDG-004/99AWC
		0.5	2	84–125	104.5	NA		
		1	2	100–101	100.5	NA		
		0.25	5	72–100	88	11		
	TFNA-AM	0.02	2	75–83	79	NA		
		0.5	2	80–100	90	NA		
		1	2	76–91	83.5	NA		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.25	5	65–89	77	9.2		
	TFNA	0.02	2	71–92	81.5	NA		
		0.5	2	91–133	112	NA		
		1	2	119–126	122.5	NA		
		0.25	5	86–121	96.6	14.2		
	TFNG	0.02	2	87–95	91	NA		
		0.5	2	80–106	93	NA		
		1	2	99–118	108.5	NA		
		0.25	5	73–92	82.2	7		
Refined Oil	Flonicamid	0.02	2	81–101	91	NA		P06-2/IB-2001-MDG-004/99AWC
		0.25	5	86–125	101.6	15.2		
		0.5	5	82–110	93	14		
		1	1	92	NA	NA		
	TFNA-AM	0.02	2	82–87	84.5	NA		
		0.25	5	65–102	78.8	14.2		
		0.5	5	68–100	84.6	12.3		
		1	1	74	NA	NA		
	TFNA	0.02	2	73–84	78.5	NA		
		0.25	5	69–126	91.2	21.3		
		0.5	5	68–100	84.6	12.3		
		1	1	84	NA	NA		
	TFNG	0.02	2	90–94	92	NA		
		0.25	5	64–114	84.2	18.3		
		0.5	5	63–100	78.4	13.6		
		1	1	80	NA	NA		
Gin Trash	Flonicamid	0.02	2	69	69	NA		IB-2001-MDG-004
		0.5	1	80	NA	NA		
		5	1	91	NA	NA		
	TFNA-AM	0.02	2	114–119	116.5	NA		
		0.5	1	84	NA	NA		
		5	1	81	NA	NA		
	TFNA	0.02	2	85–95	90	NA		
		0.5	1	82	NA	NA		
		5	1	83	NA	NA		
	TNFG	0.02	2	110–111	110.5	NA		
		0.5	1	72	NA	NA		
		5	1	81	NA	NA		
Cucumber	Flonicamid	0.01	1	117	NA	NA		IB-2001-MDG-007
		0.1	2	84–94	89	NA		
	TFNA-AM	0.01	1	85	NA	NA		
		0.1	2	66–79	72.5	NA		
	TFNA	0.01	1	95	NA	NA		
		0.1	2	77–97	87	NA		
	TFNG	0.01	1	75	NA	NA		
		0.1	2	77–80	78.5	NA		
Summer Squash	Flonicamid	0.01	2	70–84	77	NA		IB-2001-MDG-007
		0.05	1	84	NA	NA		
		0.1	2	86–90	88	NA		
	TFNA-AM	0.01	2	74–87	80.5	NA		
		0.05	1	74	NA	NA		
		0.1	2	72–75	73.5	NA		
	TFNA	0.01	2	73–82	77.5	NA		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.05	1	85	NA	NA		
		0.1	2	88–98	93	NA		
	TNFG	0.01	2	72–79	75.5	NA		
		0.05	1	77	NA	NA		
		0.1	2	69–81	75	NA		
Muskmelon	Flonicamid	0.01	2	87–92	89.5	NA		IB-2001-MDG-007
		0.1	1	93	NA	NA		
		0.2	2	95–107	101	NA		
		0.5	1	94	NA	NA		
	TFNA-AM	0.01	2	74–87	80.5	NA		
		0.1	1	71	NA	NA		
		0.2	2	83–84	83.5	NA		
		0.5	1	80	NA	NA		
	TFNA	0.01	2	90–95	92.5	NA		
		0.1	1	89	NA	NA		
		0.2	2	97–102	99.5	NA		
		0.5	1	99	NA	NA		
	TFNG	0.01	2	76–89	82.5	NA		
		0.1	1	70	NA	NA		
		0.2	2	87–88	87.5	NA		
		0.5	1	83	NA	NA		
Wheat Forage	Flonicamid	0.01	1	115	NA	NA		IB-2001-JLW-001/99WDN
		0.1	2	108–122	110	NA		
		0.2	1	93	NA	NA		
		0.25	1	79	NA	NA		
		0.5	5	72–100	92.2	11.5		
	TFNA-AM	0.01	1	85	NA	NA		
		0.1	2	76–92	84	NA		
		0.2	1	78	NA	NA		
		0.25	1	73	NA	NA		
		0.5	5	74–91	82	7.6		
	TFNA	0.01	1	94	NA	NA		
		0.1	2	85–102	93.5	NA		
		0.2	1	83	NA	NA		
		0.25	1	109	NA	NA		
		0.5	5	81–121	97.8	14.8		
	TFNG	0.01	1	75	NA	NA		
		0.1	2	73–102	87.5	NA		
		0.2	1	83	NA	NA		
		0.25	1	80	NA	NA		
		0.5	5	84–94	89.6	3.8		
Wheat Straw	Flonicamid	0.02	4	65–127	98.5	29.4		WCS/IB-2001-JLW-001/99WDN
		0.04	3	107–128	114.7	11.6		
		0.05	4	61–85	74.8	10		
		0.1	5	69–122	97.8	23.2		
		0.25	1	93	NA	NA		
		0.5	5	71–76	78.4	10		
	TFNA-AM	0.02	4	61–80	71.8	8.3		
		0.04	3	63–71	66	4.4		
		0.05	4	72–92	80.3	8.4		
		0.1	5	73–86	79	4.8		
		0.25	1	69	NA	NA		
		0.5	5	62–77	69	6.4		
	TFNA	0.02	4	77–109	88.5	14		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.04	3	63–95	83.7	18		
		0.05	4	73–99	86.8	11		
		0.1	5	81–110	92.8	11.1		
		0.25	1	83	NA	NA		
		0.5	5	71–87	82	6.3		
	TFNG	0.02	4	66–96	82.5	7.6		
		0.04	3	72–88	81.3	8.3		
		0.05	4	85–101	93.8	8		
		0.1	5	80–97	88.4	8		
		0.25	1	76	NA	NA		
		0.5	5	75–89	80	5.6		
Wheat Grain	Flonicamid	0.01	1	121	NA	NA		IB-2001-JLW-001/99WDN
		0.05	1	91	NA	NA		
		0.1	2	79–121	100	NA		
		0.25	5	76–103	85.2	10.6		
		0.5	1	89	NA	NA		
	TFNA-AM	0.01	1	67	NA	NA		
		0.05	1	83	NA	NA		
		0.1	2	79–80	79.5	NA		
		0.25	5	60–80	72	9.7		
		0.5	1	71	NA	NA		
	TFNA	0.01	1	75	NA	NA		
		0.05	1	115	NA	NA		
		0.1	2	96–97	96.5	NA		
		0.25	5	73–105	84.4	12		
		0.5	1	93	NA	NA		
	TFNG	0.01	1	74	NA	NA		
		0.05	1	89	NA	NA		
		0.1	2	83–89	86	NA		
		0.25	5	64–93	78.2	10.5		
		0.5	1	78	NA	NA		
Wheat Bran	Flonicamid	0.25	1	76	NA	NA		99WDN
		0.5	5	83–100	92.2	6.9		
	TFNA-AM	0.25	1	67	NA	NA		
		0.5	5	71–89	78	8.8		
	TFNA	0.25	1	63	NA	NA		
		0.5	5	76–99	88	9.9		
	TFNG	0.25	1	71	NA	NA		
		0.5	5	74–96	83.6	8		
Wheat Germ	Flonicamid	0.25	1	99	NA	NA		02JRA
		0.5	5	80–110	94	11.2		
	TFNA-AM	0.25	1	68	NA	NA		
		0.5	5	67–80	72.4	5.2		
	TFNA	0.25	1	66	NA	NA		
		0.5	5	70–92	78.8	9.7		
	TFNG	0.25	1	80	NA	NA		
		0.5	5	70–92	79.6	9.7		
Wheat Middlings	Flonicamid	0.25	5	76–114	85.6	16		99WDN
		0.5	5	77–89	82.6	6		
	TFNA-AM	0.25	5	80–94	87.8	6.8		
		0.5	5	68–97	84.6	10.7		
	TFNA	0.25	5	84–94	89.6	4.4		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.5	5	75–104	87.6	10.7		
	TFNG	0.25	5	91–105	98.8	5		
		0.5	5	87–118	97	12		
Turnip Tops	Flonicamid	0.01	1	83	NA	NA		IB-2001-JLW-001
		0.05	1	89	NA	NA		
	TFNA-AM	0.01	1	82	NA	NA		
		0.05	1	75	NA	NA		
	TFNA	0.01	1	77	NA	NA		
		0.05	1	73	NA	NA		
	TFNG	0.01	1	76	NA	NA		
		0.05	1	70	NA	NA		
Turnip Roots	Flonicamid	0.01	1	96	NA	NA		IB-2001-JLW-001
		0.1	1	95	NA	NA		
	TFNA-AM	0.01	1	88	NA	NA		
		0.1	1	74	NA	NA		
	TFNA	0.01	1	91	NA	NA		
		0.1	1	70	NA	NA		
	TFNG	0.01	1	94	NA	NA		
		0.1	1	72	NA	NA		
Leaf Lettuce	Flonicamid	0.01	1	79	NA	NA		01JWB
		0.1	1	73	NA	NA		
		0.5	1	84	NA	NA		
		10	1	81	NA	NA		
	TFNA-AM	0.01	1	68	NA	NA		
		0.1	1	74	NA	NA		
		0.5	1	70	NA	NA		
		10	1	73	NA	NA		
	TFNA	0.01	1	72	NA	NA		
		0.1	1	80	NA	NA		
		0.5	1	73	NA	NA		
		10	1	80	NA	NA		
	TFNG	0.01	1	72	NA	NA		
		0.1	1	76	NA	NA		
		0.5	1	75	NA	NA		
		10	1	79	NA	NA		
Head Lettuce	Flonicamid	0.01	2	88–115	101.5	NA		01JWB
		0.1	2	72–85	78.5	NA		
		0.5	2	88–91	89.5	NA		
		1	2	93–115	104	NA		
	TFNA-AM	0.01	2	90–101	95.5	NA		
		0.1	2	72–74	73	NA		
		0.5	2	79	79	NA		
		1	2	80–88	84	NA		
	TFNA	0.01	2	77–113	95	NA		
		0.1	2	73–83	78	NA		
		0.5	2	89–97	93	NA		
		1	2	94–110	102	NA		
	TFNG	0.01	2	122–125	123.5	NA		
		0.1	2	77–78	77.5	NA		
		0.5	2	82–88	85	NA		
		1	2	86–107	96.5	NA		
Celery	Flonicami	0.01	2	66–73	69.5	NA		01JWB

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
	d							
		0.05	1	72	NA	NA		
		0.1	2	75–108	91.5	NA		
		0.25	1	78	NA	NA		
		5	1	83	NA	NA		
	TFNA-AM	0.01	2	76–85	80.5	NA		
		0.05	1	71	NA	NA		
		0.1	2	66–88	77	NA		
		0.25	1	67	NA	NA		
		5	1	72	NA	NA		
	TFNA	0.01	2	76–78	77	NA		
		0.05	1	73	NA	NA		
		0.1	2	91–97	94	NA		
		0.25	1	0.25	NA	NA		
		5	1	77	NA	NA		
	TFNG	0.01	2	112	112	NA		
		0.05	1	81	NA	NA		
		0.1	2	75–98	86.5	NA		
		0.25	1	66	NA	NA		
		5	1	81	NA	NA		
Spinach	Flonicamid	0.01	2	97–100	98.5	NA		01JWB
		0.1	1	88	NA	NA		
		0.2	1	104	NA	NA		
		0.25	5	88–107	96.4	8		
		0.5	1	117	NA	NA		
		2	1	116	NA	NA		
	TFNA-AM	0.01	2	90–94	92	NA		
		0.1	1	66	NA	NA		
		0.2	1	99	NA	NA		
		0.25	5	73–93	83.2	8.2		
		0.5	1	98	NA	NA		
		2	1	88	NA	NA		
	TFNA	0.01	2	83–105	94	NA		
		0.1	1	83	NA	NA		
		0.2	1	108	NA	NA		
		0.25	5	77–117	90.6	16.1		
		0.5	1	118	NA	NA		
		2	1	99	NA	NA		
	TFNG	0.01	2	82–84	83	NA		
		0.1	1	73	NA	NA		
		0.2	1	111	NA	NA		
		0.25	5	68–99	88	13.2		
		0.5	1	101	NA	NA		
		2	1	92	NA	NA		
Broccoli	Flonicamid	0.01	2	74–81	77.5	NA		03WDN
		0.025	1	113	NA	NA		
		1	4	88–95	92	3.2		
	TFNA-AM	0.01	2	82–87	84.5	NA		
		0.025	1	112	NA	NA		
		1	4	77–92	85	6.3		
	TFNA	0.01	2	71–85	78	NA		
		0.025	1	119	NA	NA		
		1	4	78–106	96.3	13.2		
	TFNG	0.01	2	69–72	70.5	NA		
		0.025	1	97	NA	NA		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		1	4	79–98	89.3	8		
Cabbage	Flonicamid	0.01	2	94	94	NA		03WDN
		0.025	1	123	NA	NA		
		0.1	1	123	NA	NA		
		1	2	109–128	118.5	NA		
		2	1	118	NA	NA		
	TFNA-AM	0.01	2	93–97	95	NA		
		0.025	1	108	NA	NA		
		0.1	1	95	NA	NA		
		1	2	86–105	95.5	NA		
		2	1	83	NA	NA		
	TFNA	0.01	2	88	88	NA		
		0.025	1	119	NA	NA		
		0.1	1	78	NA	NA		
		1	2	87–111	99	NA		
		2	1	97	NA	NA		
	TFNG	0.01	2	97–100	NA	NA		
		0.025	1	128	NA	NA		
		0.1	1	87	NA	NA		
		1	2	94–105	99.5	NA		
		2	1	90	NA	NA		
Mustard Greens	Flonicamid	0.5	1	117	NA	NA		03WDN
		2	1	100	NA	NA		
		16	1	99	NA	NA		
	TFNA-AM	0.5	1	90	NA	NA		
		2	1	89	NA	NA		
		16	1	92	NA	NA		
	TFNA	0.5	1	105	NA	NA		
		2	1	107	NA	NA		
		16	1	99	NA	NA		
	TFNG	0.5	1	102	NA	NA		
		2	1	99	NA	NA		
		16	1	106	NA	NA		
Lemon	Flonicamid	0.01	5	86–100	93	6	AGR/MOA/IKI220-1 v.1	ISK/IKI/06001
		0.1	5	76–87	84	5		
	TFNG	0.01	10	68–84	76	7		
		0.1	10	63–85	70	10		
	TFNA	0.01	5	78–89	85	5		
		0.1	5	70–80	77	6		
	TFNA-AM	0.01	10	68–86	78	7		
		0.1	10	56–94	72	15		
Oilseed rape	Flonicamid	0.01	5	77–106	90	13		
		0.1	5	75–96	83	5		
	TFNG	0.01	5	58–84	75	13		
		0.1	5	74–100	86	12		
	TFNA	0.01	5	74–90	81	8		
		0.1	5	67–91	77	13		
	TFNA-AM	0.01	5	67–98	88	14		
		0.1	5	74–101	87	13		
Wheat grain	Flonicamid	0.01	5	102–117	109	5		
		0.1	5	85–110	93	11		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.5	5	84–101	91	8		
	TFNG	0.01	5	85–113	101	12		
		0.1	5	81–113	94	13		
		1.0	5	74–89	83	7		
	TFNA	0.01	5	88–95	92	3		
		0.1	5	87–92	89	3		
	TFNA-AM	0.01	5	94–114	108	8		
		0.1	5	83–116	96	13		
Plum	Flonicamid	0.01	5	89–121	105	12		
		0.1	5	80–102	94	9		
		0.5	5	82–93	89	5		
	TFNG	0.01	5	83–119	103	16		
		0.1	5	77–98	85	10		
	TFNA	0.01	5	95–113	102	7		
		0.1	5	76–105	88	13		
	TFNA-AM	0.01	5	101–113	107	5		
		0.1	5	77–91	82	7		
Prune	Flonicamid	0.01	5	87–103	92	7		
		0.1	5	81–93	89	6		
	TFNG	0.01	5	78–85	81	4		
		0.1	5	73–85	82	6		
	TFNA	0.01	5	75–87	80	7		
		0.1	5	80–97	90	7		
	TFNA-AM	0.01	5	69–75	72	4		
		0.1	5	76–86	83	5		
Animal Commodities								
Milk	Flonicamid	0.01	5	72–81	76	6	842993	
		0.10	5	74–82	79	4		
	TFNA	0.01	5	79–105	94	11		
		0.10	5	80–93	88	6		
	TFNA-AM	0.01	5	78–91	83	6		
		0.10	5	86–97	92	5		
	OH–TFNA-AM	0.01	5	74–83	80	5		
		0.10	5	74–86	82	7		
	TFNG	0.01	5	79–107	95	12		
		0.10	5	71–79	76	5		
Bovine Muscle	Flonicamid	0.01	5	102–108	107	2	844743	
		0.10	5	84–108	92	11		
	TFNA	0.01	5	85–108	98	8		
		0.10	5	95–108	101	6		
	TFNA-AM	0.01	5	95–101	98	3		
		0.10	5	86–106	92	10		
	OH-TFNA-AM	0.01	5	83–100	94	7		
		0.10	5	87–99	94	6		
	TFNG	0.01	5	86–100	95	6		
		0.10	5	95–106	100	5		
Bovine Liver	Flonicamid	0.01	5	72–80	78	4		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.10	5	73–78	75	2		
	TFNA	0.01	5	82–107	91	12		
		0.10	5	82–88	86	3		
	TFNA-AM	0.01	5	79–87	82	4		
		0.10	5	76–82	79	3		
	OH-TFNA-AM	0.01	5	80–94	86	6		
		0.10	5	86–106	95	10		
	TFNG	0.01	5	85–92	87	8		
		0.10	5	94–107	100	6		
Bovine Kidney	Flonicamid	0.01	5	72–101	82	14		
		0.10	5	78–97	87	10		
	TFNA	0.01	5	76–90	83	7		
		0.10	5	89–96	92	4		
	TFNA-AM	0.01	5	90–105	101	6		
		0.10	5	99–106	103	3		
	OH-TFNA-AM	0.01	5	88–105	99	7		
		0.10	5	100–105	102	2		
	TFNG	0.01	5	78–107	91	13		
		0.10	5	88–96	92	4		
Bovine Fat	Flonicamid	0.01	5	108–110	109	1		
		0.10	5	104–108	106	2		
	TFNA	0.01	5	72–108	82	18		
		0.10	5	73–79	75	2		
	TFNA-AM	0.01	5	88–108	96	8		
		0.10	5	89–96	92	3		
	OH-TFNA-AM	0.01	5	71–73	72	1		
		0.10	5	71–83	76	6		
	TFNG	0.01	5	88–99	93	4		
		0.10	5	99–108	104	4		
Poultry Egg	Flonicamid	0.01	5	81–88	85	3		
		0.10	5	92–101	96	4		
	TFNA	0.01	5	70–76	72	3		
		0.10	5	81–98	90	8		
	TFNA-AM	0.01	5	78–86	82	4		
		0.10	5	87–99	93	6		
	OH-TFNA-AM	0.01	5	81–91	87	5		
		0.10	5	94–106	99	5		
	TFNG	0.01	5	74–85	78	5		
		0.10	5	94–110	100	6		
Poultry Muscle	Flonicamid	0.01	2	109–110	110	NA		
		0.10	2	105	105	NA		
		1.0	2	98–99	98	NA		
	TFNA	0.01	2	88	88	NA		
		0.10	2	88–93	90	NA		
		1.0	2	97–101	99	NA		
	TFNA-AM	0.01	2	108–109	108	NA		
		0.10	2	103–106	104	NA		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		1.0	2	98–99	98	NA		
	OH-TFNA-AM	0.01	2	101–105	103	NA		
		0.10	2	104–110	107	NA		
		1.0	2	100–102	101	NA		
	TFNG	0.01	2	89–107	98	NA		
		0.10	2	98–105	101	NA		
		1.0	2	97–98	98	NA		
Poultry Liver	Flonicamid	0.01	2	106–107	106	NA		
		0.10	2	95–97	96	NA		
		1.0	2	96–100	98	NA		
	TFNA	0.01	2	80–94	88	NA		
		0.10	2	90–94	92	NA		
		1.0	2	95–97	96	NA		
	TFNA-AM	0.01	2	107–109	108	NA		
		0.10	2	97–99	98	NA		
		1.0	2	97–98	98	NA		
	OH-TFNA-AM	0.01	2	80–89	84	NA		
		0.10	2	95–97	96	NA		
		1.0	2	92–94	93	NA		
	TFNG	0.01	2	83–90	86	NA		
		0.10	2	93–109	101	NA		
		1.0	2	99–101	100	NA		
Poultry Fat	Flonicamid	0.01	2	107–108	108	NA		
		0.10	2	107–108	107	NA		
		1.0	2	104–110	107	NA		
	TFNA	0.01	2	72–89	81	NA		
		0.10	2	70–76	73	NA		
		1.0	2	72–74	73	NA		
	TFNA-AM	0.01	2	92–104	98	NA		
		0.10	2	96–99	97	NA		
		1.0	2	95–101	98	NA		
	OH-TFNA-AM	0.01	2	75–78	76	NA		
		0.10	2	74–81	78	NA		
		1.0	2	83	83	NA		
	TFNG	0.01	2	100–105	102	NA		
		0.10	2	106–108	107	NA		
		1.0	2	97–105	101	NA		
Milk	Flonicamid	0.01	5	88–92	90	2	AGR/MOA/IKI-5	
		0.10	5	89–92	91	2		
	TFNA-AM	0.01	5	86–90	88	2		
		0.10	5	88–92	90	2		
Eggs	Flonicamid	0.01	5	87–93	90	3		
		0.10	5	92–98	95	3		
	TFNA-AM	0.01	5	91–93	92	2		
		0.10	5	93–96	94	1		
Bovine Muscle	Flonicami	0.01	5	79–87	84	4		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
	d							
		0.10	5	81–88	84	4		
	TFNA-AM	0.01	5	83–88	85	2		
		0.10	5	85–91	87	3		
Bovine Fat	Flonicamid	0.01	5	91–93	92	1		
		0.10	5	88–95	91	3		
	TFNA-AM	0.01	5	91–94	92	1		
		0.10	5	91–96	93	2		
Bovine Liver	Flonicamid	0.01	5	79–84	82	3		
		0.10	5	77–84	81	3		
	TFNA-AM	0.01	5	81–88	83	4		
		0.10	5	80–86	83	3		

Stability of residues in stored analytical samples

Information was received on the freezer storage stability of flonicamid and its metabolites in plant commodities. The storage stability of flonicamid and its metabolites TFNA, TFNA-AM and TFNG are described as follows. The results are shown in Table 42.

Wheat (grain, forage, straw, bran, middling, germ), cottonseed (seed, hulls, meal, refined oil), spinach, potato tuber, apple juice and tomato

Report: P-3570

Study No. 178CSS02R1

Method: P-3561

Description: Untreated control samples were fortified with flonicamid and its metabolites TFNA, TFNA-AM and TFNG at a concentration of 0.5 mg/kg per analyte and then frozen below –17 °C. Samples were analysed immediately after fortification (0 day) and after storage intervals up to 2 years (23 months). At each interval, three stored samples were analysed, with one or more procedural recovery samples (control samples spiked just before analysis at 0.5 mg/kg).

Table 42 Storage Stability of Flonicamid, TFNA, TFNA-AM and TFNG in wheat, cotton, potato, apple and tomato

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
Wheat Grain					
0	0.50, 0.47, 0.53	0.5	100%	76	
3	0.42, 0.37, 0.40	0.4	80%	89	
6	0.39, 0.38, 0.46	0.41	82%	103	
9	0.40, 0.40, 0.45	0.42	84%	86	
15	0.50, 0.49, 0.44	0.48	96%	79	
23	0.48, 0.52, 0.54	0.51	102%	82	
Wheat Forage					
0	0.57, 0.52, 0.53	0.54	100%	79	

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
3	0.52, 0.48, 0.53	0.51	94%	96	
6	0.22, 0.29, 0.68	0.40	74%	72	
9	0.49, 0.48, 0.52	0.50	93%	98	
15	0.43, 0.41, 0.46	0.43	80%	95	
23	0.48, 0.57, 0.55	0.53	98%	100	
Wheat Straw					
0	0.55, 0.58, 0.53	0.55	100%	93	
3	0.52, 0.51, 0.48	0.5	91%	96	
6	0.44, 0.46, 0.40	0.44	80%	75	
9	0.48, 0.50, 0.46	0.48	87%	75	
15	0.44, 0.47, 0.46	0.46	84%	71	
23	0.52, 0.53, 0.53	0.52	95%	75	
Wheat Bran					
0	0.53, 0.38, 0.48	0.46	100%	76	
3	0.49, 0.44, 0.47	0.47	102%	83	
6	0.37, 0.40, 0.35	0.37	80%	91	
9	0.43, 0.45, 0.40	0.43	93%	100	
15	0.47, 0.45, 0.49	0.47	102%	98	
23	0.51, 0.57, 0.53	0.54	117%	89	
Wheat Germ					
0	0.55, 0.50, 0.54	0.53	100%	99	
3	0.59, 0.51, 0.42	0.51	96%	110	
6	0.52, 0.46, 0.53	0.50	94%	80	
9	0.47, 0.45, 0.45	0.46	87%	99	
15	0.39, 0.47, 0.37	0.41	77%	91	
23	0.42, 0.41, 0.44	0.42	79%	90	
Wheat Middling					
0	0.30, 0.34, 0.51	0.38	100%	79	
3	0.24, 0.39, 0.43	0.35	92%	81	
6	0.58, 0.36, 0.58	0.51	134%	76, 89	83
9	0.54, 0.50, 0.53	0.53	139%	77, 89	83
15	0.51, 0.53, 0.60	0.55	145%	82, 77	80
23	0.45, 0.48, 0.50	0.48	126%	114, 77	96
Spinach					
0	0.48, 0.48, 0.50	0.49	100%	89	
3	0.43, 0.44, 0.46	0.44	90%	117	
6	0.62, 0.65, 0.59	0.62	127%	101	
9	0.46, 0.52, 0.47	0.48	98%	88	
15	0.49, 0.41, 0.41	0.44	90%	94	
23	0.45, 0.46, 0.44	0.45	92%	107	
Cottonseed					
0	0.56, 0.55, 0.59	0.57	100%	78	
3	0.46, 0.48, 0.54	0.43	75%	96	
6	0.42, 0.39, 0.38	0.40	70%	86, 106	96
9	0.35, 0.38, 0.38	0.37	65%	97, 105	101
15	0.48, 0.45, 0.47	0.47	82%	80, 84	82
23	0.43, 0.48, 0.53	0.48	84%	84, 90	87
Cotton Hulls					
0	0.30, 0.53, 0.56	0.46	100%	100	
3	0.51, 0.53, 0.56	0.53	92%	125	

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
6	0.56, 0.55, 0.57	0.56	122%	72	
9	0.55, 0.45, 0.54	0.51	111%	93	
15	0.55, 0.58, 0.62	0.58	126%	82	
23	0.61, 0.36, 0.56	0.51	111%	93	
Cotton Meal					
0	0.40, 0.42, 0.41	0.41	100%	100	
3	0.59, 0.50, 0.48	0.52	127%	99	
6	0.58, 0.58, 0.46	0.54	132%	73	
9	0.45, 0.34, 0.44	0.41	100%	103	
15	0.49, 0.48, 0.50	0.49	120%	90	
23	0.44, 0.44, 0.40	0.43	86%	122	
Cotton Refined oil					
0	0.48, 0.45, 0.47	0.47	100%	125	
3	0.49, 0.43, 0.47	0.46	98%	83	
6	0.59, 0.53, 0.47	0.53	113%	103, 110	107
9	0.54, 0.53, 0.55	0.54	115%	86, 83	85
15	0.52, 0.49, 0.54	0.52	111%	90, 92	86
23	0.41, 0.47, 0.42	0.43	91%	104, 107	106
Apple juice					
0	0.52, 0.54, 0.47	0.51	100%	113	
3	0.48, 0.47, 0.43	0.48	75%	125	
6	0.41, 0.47, 0.50	0.46	90%	91	
9	0.46, 0.45, 0.47	0.46	90%	95	
15	0.47, 0.48, 0.56	0.50	98%	92	
23	0.58, 0.55, 0.48	0.54	106%	90	
Tomato					
0	0.52, 0.55, 0.57	0.55	100%	102	
3	0.56, 0.54, 0.52	0.54	98%	111	
6	0.48, 0.43, 0.54	0.48	87%	91	
9	0.45, 0.46, 0.51	0.47	85%	96	
15	0.55, 0.52, 0.51	0.53	96%	82	
23	0.59, 0.55, 0.61	0.58	105%	86	
Potato Tuber					
0	0.46, 0.41, 0.45	0.44	100%	111	
3	0.41, 0.44, 0.44	0.43	79%	124	
6	0.38, 0.40, 0.40	0.39	89%	105, 123	114
9	0.51, 0.50, 0.62	0.54	123%	92 ,97	95
15	0.45, 0.52, 0.42	0.46	105%	99, 97	98
23	0.56, 0.53, 0.49	0.53	120%	112, 92	102
TFNG					
Wheat grain					
0	0.51, 0.49, 0.58	0.53	100%	64	
3	0.46, 0.48, 0.49	0.48	91%	78	
6	0.39, 0.43, 0.46	0.43	81%	93	
9	0.45, 0.51, 0.53	0.49	92%	76	
15	0.58, 0.53, 0.50	0.54	102%	76	
23	0.50, 0.52, 0.50	0.51	96%	82	
Wheat Forage					
0	0.54, 0.52, 0.52	0.53	100%	80	
3	0.53, 0.48, 0.51	0.51	96%	91	
6	0.42, 0.41, 0.41	0.41	77%	91	

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
9	0.56, 0.59, 0.56	0.57	108%	94	
15	0.48, 0.47, 0.47	0.47	89%	88	
23	0.49, 0.56, 0.56	0.54	102%	84	
Wheat Straw					
0	0.63, 0.65, 0.63	0.63	100%	76	
3	0.56, 0.53, 0.53	0.54	114%	75	
6	0.46, 0.49, 0.42	0.46	82%	89	
9	0.47, 0.41, 0.48	0.45	90%	79	
15	0.49, 0.53, 0.50	0.51	100%	81	
23	0.49, 0.55, 0.56	0.53	111%	76	
Wheat Bran					
0	0.53, 0.38, 0.47	0.46	100%	71	
3	0.52, 0.45, 0.46	0.47	102%	74	
6	0.36, 0.41, 0.37	0.38	83%	96	
9	0.45, 0.53, 0.48	0.48	104%	80	
15	0.48, 0.47, 0.50	0.48	104%	85	
23	0.50, 0.46, 0.48	0.51	111%	81	
Wheat Germ					
0	0.52, 0.54, 0.53	0.53	100%	80	
3	0.63, 0.58, 0.59	0.6	113%	92	
6	0.50, 0.47, 0.47	0.48	91%	70	
9	0.56, 0.54, 0.57	0.56	106%	78	
15	0.44, 0.49, 0.51	0.48	91%	87	
23	0.53, 0.60, 0.62	0.58	109%	71	
Wheat Middling					
0	0.48, 0.52, 0.49	0.5	100%	91	
3	0.55, 0.57, 0.55	0.56	112%	90	
6	0.58, 0.40, 0.59	0.52	104%	100, 96	98
9	0.49, 0.56, 0.47	0.51	102%	99, 87	93
15	0.53, 0.55, 0.60	0.56	112%	105, 118	112
23	0.56, 0.53, 0.56	0.55	110%	99, 94	97
Spinach					
0	0.50, 0.50, 0.54	0.52	100%	81	
3	0.43, 0.45, 0.44	0.44	85%	101	
6	0.40, 0.43, 0.39	0.41	79%	96	
9	0.44, 0.48, 0.47	0.46	88%	68	
15	0.55, 0.47, 0.47	0.5	96%	96	
23	0.46, 0.47, 0.46	0.46	88%	99	
Cottonseed					
0	0.56, 0.57, 0.56	0.56	100%	71	
3	0.48, 0.53, 0.53	0.51	91%	87	
6	0.43, 0.38, 0.37	0.39	70%	84, 92	88
9	0.46, 0.42, 0.43	0.44	79%	83, 111	97
15	0.50, 0.47, 0.47	0.48	86%	81, 77	79
23	0.48, 0.50, 0.54	0.51	91%	91, 95	93
Cotton Hulls					
0	0.57, 0.61, 0.61	0.6	100%	92	
3	0.65, 0.63, 0.60	0.63	105%	106	
6	0.46, 0.48, 0.48	0.47	78%	84	
9	0.39, 0.54, 0.41	0.45	75%	80	
15	0.54, 0.55, 0.58	0.56	93%	73	
23	0.59, 0.60, 0.55	0.58	97%	82	
Cotton Meal					
0	0.43, 0.45, 0.43	0.43	100%	85	
3	0.56, 0.54, 0.54	0.55	128%	81	
6	0.46, 0.50, 0.44	0.47	109%	75	
9	0.49, 0.42, 0.50	0.47	158%	69	

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
15	0.48, 0.50, 0.52	0.5	116%	84	
23	0.55, 0.52, 0.52	0.53	123%	82	
Cotton Refined Oil					
0	0.42, 0.37, 0.43	0.41	100%	83	
3	0.39, 0.37, 0.42	0.39	151%	63	
6	0.47, 0.60, 0.49	0.52	127%	79, 73	76
9	0.53, 0.56, 0.58	0.56	137%	64, 76	70
15	0.53, 0.48, 0.54	0.52	127%	81, 80	81
23	0.44, 0.51, 0.45	0.47	115%	114, 100	107
Apple Juice					
0	0.55, 0.51, 0.48	0.51	100%	115	
3	0.47, 0.48, 0.49	0.48	71%	133	
6	0.42, 0.46, 0.46	0.45	88%	82	
9	0.41, 0.47, 0.50	0.46	90%	82	
15	0.50, 0.49, 0.57	0.52	102%	88	
23	0.59, 0.62, 0.50	0.57	112%	65	
Tomato					
0	0.59, 0.49, 0.52	0.53	100%	112	
3	0.51, 0.51, 0.51	0.51	96%	120	
6	0.46, 0.45, 0.44	0.45	85%	87	
9	0.48, 0.50, 0.51	0.5	94%	82	
15	0.51, 0.51, 0.49	0.5	94%	88	
23	0.54, 0.60, 0.64	0.59	111%	82	
Potato Tuber					
0	0.48, 0.43, 0.49	0.47	100%	90	
3	0.45, 0.48, 0.46	0.46	98%	104	
6	0.42, 0.40, 0.41	0.41	87%	97, 102	100
9	0.58, 0.48, 0.60	0.53	113%	78, 90	84
15	0.48, 0.54, 0.48	0.5	106%	88, 86	87
23	0.56, 0.54, 0.55	0.55	117%	102, 93	98
TFNA					
Wheat Grain					
0	0.43, 0.47, 0.48	0.46	100%	73	
3	0.47, 0.42, 0.44	0.44	96%	93	
6	0.37, 0.37, 0.42	0.39	85%	105	
9	0.46, 0.51, 0.54	0.5	109%	80	
15	0.53, 0.52, 0.47	0.51	111%	83	
23	0.52, 0.48, 0.53	0.51	111%	81	
Wheat Forage					
0	0.53, 0.48, 0.48	0.49	100%	79	
3	0.52, 0.45, 0.46	0.48	98%	96	
6	0.49, 0.44, 0.39	0.44	90%	72	
9	0.50, 0.53, 0.56	0.53	108%	98	
15	0.40, 0.39, 0.44	0.47	96%	95	
23	0.47, 0.67, 0.62	0.59	120%	100	
Wheat Straw					
0	0.66, 0.67, 0.66	0.66	100%	83	
3	0.57, 0.56, 0.54	0.56	102%	83	
6	0.38, 0.40, 0.39	0.39	70%	84	
9	0.44, 0.42, 0.45	0.44	77%	87	
15	0.45, 0.49, 0.45	0.46	82%	85	
23	0.49, 0.49, 0.50	0.49	105%	71	
Wheat Bran					
0	0.48, 0.40, 0.47	0.45	100%	63	
3	0.47, 0.48, 0.29	0.41	91%	80	
6	0.37, 0.43, 0.36	0.39	87%	99	
9	0.46, 0.53, 0.46	0.48	107%	89	

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
15	0.46, 0.44, 0.46	0.45	100%	96	
23	0.44, 0.53, 0.46	0.48	107%	76	
Wheat Germ					
0	0.59, 0.68, 0.65	0.64	100%	66	
3	0.76, 0.62, 0.72	0.7	109%	71	
6	0.62, 0.54, 0.57	0.58	91%	70	
9	0.50, 0.49, 0.52	0.5	78%	86	
15	0.41, 0.44, 0.46	0.44	69%	92	
23	0.53, 0.53, 0.52	0.53	83%	75	
Wheat Middling					
0	0.49, 0.52, 0.49	0.5	100%	91	
3	0.52, 0.57, 0.54	0.55	110%	89	
6	0.60, 0.38, 0.62	0.53	106%	84, 88	86
9	0.47, 0.58, 0.52	0.52	104%	86, 75	81
15	0.50, 0.53, 0.54	0.52	104%	93, 104	99
23	0.48, 0.51, 0.57	0.52	104%	94, 82	88
Spinach					
0	0.51, 0.48, 0.51	0.5	100%	94	
3	0.42, 0.45, 0.44	0.43	86%	118	
6	0.40, 0.49, 0.45	0.44	88%	85	
9	0.47, 0.54, 0.50	0.5	100%	80	
15	0.51, 0.42, 0.42	0.45	90%	117	
23	0.45, 0.54, 0.51	0.5	100%	77	
Cottonseed					
0	0.56, 0.57, 0.57	0.56	100%	92	
3	0.45, 0.47, 0.51	0.48	86%	108	
6	0.42, 0.36, 0.35	0.38	68%	95, 108	102
9	0.41, 0.39, 0.40	0.4	71%	92, 120	106
15	0.48, 0.47, 0.44	0.46	82%	86, 92	89
23	0.41, 0.46, 0.51	0.46	82%	81, 95	88
Cotton Hulls					
0	0.55, 0.58, 0.57	0.57	100%	121	
3	0.62, 0.59, 0.59	0.6	79%	133	
6	0.53, 0.51, 0.52	0.52	91%	97	
9	0.42, 0.56, 0.40	0.46	81%	90	
15	0.55, 0.55, 0.60	0.57	100%	89	
23	0.60, 0.61, 0.61	0.61	107%	86	
Cotton Meal					
0	0.43, 0.40, 0.48	0.43	100%	76	
3	0.60, 0.57, 0.55	0.58	135%	77	
6	0.56, 0.59, 0.52	0.56	130%	84	
9	0.44, 0.43, 0.44	0.44	102%	77	
15	0.49, 0.51, 0.50	0.5	116%	99	
23	0.50, 0.54, 0.50	0.51	119%	80	
Cottonseed Refined Oil					
0	0.46, 0.45, 0.45	0.45	100%	93	
3	0.41, 0.46, 0.39	0.42	137%	68	
6	0.51, 0.64, 0.55	0.57	127%	82, 77	80
9	0.51, 0.54, 0.58	0.54	120%	69, 89	79
15	0.49, 0.48, 0.53	0.5	111%	86, 89	88
23	0.29, 0.35, 0.42	0.35	78%	126, 100	113
Apple Juice					
0	0.61, 0.56, 0.51	0.56	100%	105	
3	0.49, 0.55, 0.52	0.52	68%	136	
6	0.40, 0.43, 0.46	0.43	77%	101	
9	0.44, 0.44, 0.50	0.46	82%	90	
15	0.45, 0.46, 0.54	0.48	86%	97	

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
23	0.55, 0.53, 0.47	0.52	93%	89	
Tomato					
0	0.53, 0.49, 0.57	0.53	100%	102	
3	0.51, 0.50, 0.47	0.49	76%	122	
6	0.43, 0.43, 0.41	0.43	81%	110	
9	0.48, 0.50, 0.55	0.51	96%	89	
15	0.48, 0.47, 0.46	0.47	89%	95	
23	0.54, 0.54, 0.62	0.57	108%	93	
Potato Tuber					
0	0.46, 0.39, 0.47	0.44	100%	105	
3	0.41, 0.43, 0.44	0.43	79%	124	
6	0.43, 0.39, 0.47	0.43	98%	104, 98	101
9	0.50, 0.47, 0.58	0.52	118%	85, 109	97
15	0.46, 0.49, 0.44	0.46	105%	104, 96	100
23	0.48, 0.50, 0.49	0.49	111%	119, 96	108
TFNA-AM					
Wheat Grain					
0	0.44, 0.48, 0.54	0.48	100%	60	
3	0.40, 0.40, 0.45	0.42	88%	71	
6	0.38, 0.37, 0.44	0.40	83%	80	
9	0.43, 0.43, 0.50	0.45	94%	79	
15	0.55, 0.50, 0.47	0.51	169%	63	
23	0.49, 0.48, 0.50	0.49	102%	78	
Wheat Forage					
0	0.55, 0.51, 0.55	0.54	100%	73	
3	0.51, 0.47, 0.50	0.49	91%	82	
6	0.43, 0.42, 0.39	0.41	76%	91	
9	0.51, 0.53, 0.54	0.52	96%	88	
15	0.45, 0.45, 0.46	0.45	83%	75	
23	0.49, 0.53, 0.55	0.52	96%	74	
Wheat Straw					
0	0.68, 0.70, 0.67	0.68	100%	69	
3	0.60, 0.57, 0.56	0.58	85%	72	
6	0.42, 0.45, 0.39	0.42	62%	77	
9	0.44, 0.44, 0.45	0.44	65%	71	
15	0.46, 0.50, 0.47	0.48	112%	63	
23	0.51, 0.52, 0.51	0.51	121%	62	
Wheat Bran					
0	0.53, 0.42, 0.48	0.48	100%	67	
3	0.50, 0.46, 0.43	0.46	96%	71	
6	0.37, 0.42, 0.38	0.39	81%	89	
9	0.44, 0.47, 0.43	0.45	94%	86	
15	0.45, 0.48, 0.48	0.47	98%	73	
23	0.50, 0.50, 0.48	0.5	104%	71	
Wheat Germ					
0	0.54, 0.55, 0.52	0.54	100%	68	
3	0.62, 0.57, 0.59	0.59	109%	80	
6	0.46, 0.44, 0.45	0.45	123%	68	
9	0.54, 0.52, 0.52	0.53	98%	74	
15	0.40, 0.46, 0.49	0.45	83%	73	
23	0.53, 0.57, 0.56	0.55	152%	67	
Wheat Middling					
0	0.47, 0.53, 0.51	0.5	100%	94	
3	0.53, 0.58, 0.50	0.54	108%	89	
6	0.60, 0.40, 0.58	0.53	106%	93, 87	90
9	0.54, 0.55, 0.57	0.55	110%	81, 82	82
15	0.52, 0.54, 0.57	0.54	108%	91, 97	94

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
23	0.51, 0.54, 0.56	0.54	108%	80, 68	74
Spinach					
0	0.53, 0.53, 0.54	0.53	100%	79	
3	0.43, 0.46, 0.43	0.44	83%	98	
6	0.40, 0.44, 0.37	0.4	75%	93	
9	0.44, 0.49, 0.50	0.48	91%	73	
15	0.55, 0.46, 0.46	0.49	92%	81	
23	0.42, 0.44, 0.47	0.44	83%	90	
Cottonseed					
0	0.56, 0.57, 0.56	0.56	100%	78	
3	0.48, 0.48, 0.52	0.49	88%	96	
6	0.42, 0.38, 0.36	0.38	68%	86, 106	96
9	0.42, 0.39, 0.41	0.41	73%	97, 105	101
15	0.46, 0.43, 0.45	0.45	80%	80, 84	82
23	0.41, 0.47, 0.52	0.47	84%	84, 90	87
Cotton Hulls					
0	0.55, 0.57, 0.59	0.57	100%	89	
3	0.63, 0.58, 0.59	0.6	105%	100	
6	0.46, 0.49, 0.46	0.47	82%	82	
9	0.45, 0.56, 0.43	0.48	84%	76	
15	0.53, 0.57, 0.60	0.57	154%	65	
23	0.65, 0.63, 0.59	0.62	109%	72	
Cotton Meal					
0	0.41, 0.44, 0.42	0.42	100%	78	
3	0.56, 0.54, 0.55	0.55	131%	72	
6	0.49, 0.50, 0.46	0.48	114%	78	
9	0.47, 0.44, 0.45	0.45	160%	67	
15	0.47, 0.48, 0.51	0.49	117%	75	
23	0.55, 0.52, 0.51	0.53	186%	68	
Cottonseed Refined Oil					
0	0.40, 0.39, 0.42	0.4	100%	102	
3	0.41, 0.37, 0.41	0.4	100%	76	
6	0.52, 0.66, 0.57	0.58	145%	77, 71	74
9	0.55, 0.57, 0.59	0.57	207%	65 73	69
15	0.51, 0.46, 0.51	0.49	178%	70, 68	69
23	0.35, 0.48, 0.45	0.43	108%	80, 77	79
Apple Juice					
0	0.55, 0.53, 0.50	0.53	100%	80	
3	0.47, 0.51, 0.49	0.49	92%	97	
6	0.40, 0.44, 0.44	0.42	79%	86	
9	0.44, 0.49, 0.52	0.49	92%	80	
15	0.49, 0.52, 0.57	0.53	100%	75	
23	0.57, 0.61, 0.47	0.55	104%	72	
Tomato					
0	0.60, 0.52, 0.54	0.55	100%	79	
3	0.53, 0.50, 0.53	0.52	95%	89	
6	0.46, 0.42, 0.41	0.43	78%	88	
9	0.51, 0.52, 0.56	0.53	96%	79	
15	0.53, 0.51, 0.51	0.52	95%	73	
23	0.54, 0.57, 0.67	0.59	107%	75	
Potato Tuber					
0	0.48, 0.43, 0.48	0.46	100%	89	
3	0.43, 0.45, 0.45	0.44	96%	101	
6	0.40, 0.38, 0.38	0.39	85%	105, 102	104
9	0.48, 0.48, 0.57	0.51	111%	78, 91	85
15	0.47, 0.53, 0.44	0.48	104%	75, 75	75
23	0.54, 0.54, 0.52	0.53	115%	83, 71	77

Apples, potatoes, wheat grain and wheat straw

Report: Not assigned

Study No. A-22-00-03

Method: "Determination of Residues of IKI-220 and its Metabolites TFNG, TFNA and TFNA-AM in Various Crops—Validation of the Method"

Description: Untreated control samples were fortified with flonicamid and its metabolites TFNA, TFNA-AM and TFNG at a concentration of 0.1 mg/kg per analyte for apple, potato and wheat grain and 0.2 mg/kg for wheat straw and then frozen below–17 °C. Samples were analysed immediately after fortification (0 day) and after storage intervals up to 18 months. At each interval, two stored samples were analysed, with one or more procedural recovery samples (control samples spiked just before analysis).

Table 43 Storage Stability of Flonicamid, TFNA, TFNA-AM and TFNG in apple, potato and wheat

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
Apples					
0		0.093	100%	95, 89, 86	90
3	0.09, 0.09	0.09	97%	93	93
6	0.11, 0.11	0.11	118%	106	106
12	0.11, 0.10	0.11	118%	99	99
18	0.10, 0.09	0.10	108%	89, 87	88
Potatoes					
0		0.09	100%	89, 92, 93	91
3	0.09, 0.10	0.09	100%	107	107
6	0.11, 0.10	0.10	111%	109	109
12	0.10, 0.14	0.12	133%	82	82
18	0.12, 0.10	0.10	111%	113, 96	104
Wheat grain					
0		0.10	100%	93, 92, 98	94
3	0.10, 0.10	0.10	100%	96	96
6	0.08, 0.08	0.08	80%	87	87
12	0.08, 0.10	009	90%	96	96
18	0.09, 0.10	0.10	100%	94, 94	94
Wheat straw					
0		0.19	100%	104, 110, 82	99
3	0.20, 0.18	0.19	100%	90	90
6	0.22, 0.22	0.22	116%	113	113
12	0.21, 0.22	0.21	111%	102	102
18	0.20, 0.23, 0.21	0.22	116%	87, 111	99
TFNG					
Apples					
0	0.09, 0.08	0.09	100%	76, 93, 78	82
3	0.10, 0.09	0.10	111%	91.00	91
6	0.08, 0.09	0.09	100%	74.00	74
12	0.12, 0.11	0.12	133%	56.00	89
18	0.10, 0.10	0.10	111%	94, 102	97
Potatoes					
0		0.08	100%	90, 84, 84	86
3	0.08, 0.09	0.08	100%	102	102
6	0.10, 0.09	0.09	113%	93	93
12	0.09, 0.11	0.10	125%	80	80
18	0.11, 0.10	0.10	125%	106, 91	98
Wheat grain					

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
0		0.08	100%	87, 67, 86	80
3	0.08, 0.08	0.08	100%	78	78
6	0.08, 0.08	0.08	100%	92	92
12	0.17, 0.09	0.13	163%	85	85
18	0.08, 0.08	0.08	100%	83	83
Wheat straw					
0		0.15	100%	79, 84, 87	77
3	0.16, 0.15	0.16	107%	78	78
6	0.18, 0.18	0.18	120%	93	93
12	0.20, 0.20	0.2	133%	100	100
18	0.16, 0.20, 0.23	0.2	133%	68, 124	96
TFNA					
Apples					
0		0.10	100%	90, 81, 83	85
3	0.08, 0.07	0.07	70%	66	66
6	0.08, 0.07	0.07	70%	77	77
12	0.12, 0.11	0.12	120%	83	83
18	0.08, 0.08	0.08	80%	88, 86	87
Potatoes					
0		0.11	100%	115, 106, 113	112
3	0.10, 0.12	0.11	100%	78	78
6	0.08, 0.08	0.08	73%	77	77
12	0.09, 0.11	0.10	91%	80	80
18	0.07, 0.09	0.08	73%	71	71
Wheat grain					
0		0.1	100%	117, 101, 99	106
3	0.07, 0.07	0.07	70%	71	71
6	0.09, 0.08	0.08	80%	89	89
12	0.12, 0.08	0.1	100%	79	79
18	0.07, 0.07	0.07	70%	72	72
Wheat straw					
0		0.18	100%	109, 109, 76	98
3	0.14, 0.13	0.13	72%	72	72
6	0.15, 0.15	0.15	83%	77	77
12	0.19, 0.19	0.19	106%	97	97
18	0.13, 0.15, 0.14	0.14	120%	45, 85	65
TFNA-AM					
Apples					
0		0.08	100%	93, 81, 83	86
3	0.08, 0.09	0.09	113%	97	97
6	0.10, 0.09	0.10	125%	94	94
12	0.11, 0.08	0.10	125%	91	91
18	0.07, 0.10	0.09	113%	83, 100	92
Potatoes					
0		0.08	100%	80, 73, 79	78
3	0.08, 0.09	0.08	100%	89	89
6	0.09, 0.09	0.09	113%	87	87
12	0.08, 0.10	0.09	113%	77	77
18	0.09, 0.10	0.09	113%	78	78
Wheat grain					
0		0.09	100%	93, 84, 99	92
3	0.08, 0.08	0.08	89%	82	82
6	0.08, 0.07	0.08	89%	81	81
12	0.07, 0.08	0.08	89%	76	76
18	0.08, 0.08	0.08	89%	87	87

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
Wheat straw					
0		0.17	100%	85, 86, 80	84
3	0.17, 0.15	0.16	94%	80	80
6	0.19, 0.19	0.19	112%	99	99
12	0.17, 0.17	0.17	100%	87	86
18	0.16, 0.19, 0.20	0.18	106%	70, 107	89

USE PATTERN

The insecticide flonicamid is registered in Canada, the United States, Slovenia, Cyprus and Australia for control of various insects on a variety of crops. The information available to the Meeting on registered uses on various fruits, vegetables, tree nuts, oilseeds, dried hops, mint and tea is summarized in Table 44. The manufacturer submitted labels for all flonicamid uses.

Table 44 Registered uses of flonicamid

Crop	Country	Form.	Application				PHI, Days
			Method	Rate, kg ai/ha	Spray conc., kg ai/hL	No.	
Pome fruits							
Pome fruits	USA	50WG/50SG	Foliar	0.07–0.1	0.01–0.02	3	21
Apples	AUS	500WG	Foliar	NS	0.005–0.01	3	21
Apples, pears	Cyprus	50WG	Foliar	0.06–0.14	0.006–0.01	3	21
Apples	Slovenia	50WG	Foliar	0.07	0.014	3	21
Stone fruits							
Stone fruits	USA	50WG/50SG	Foliar	0.07–0.1	0.01–0.02	3	14
Peaches	Cyprus	50WG	Foliar	0.06–0.07	0.006–0.007	2	14
Plums							35
Peaches	Slovenia	50WG	Foliar	0.07	0.014	3	14
Berries and other small fruit							
Low growing berries	USA	50SG	Foliar	0.1	0.02–0.10	3	0
Brassica (cole or cabbage) vegetables							
Brassica (cole) leafy vegetables	USA	50WG/50SG	Foliar	0.07–0.1	0.07–0.1	3	0
Fruiting vegetables, cucurbits							
Cucurbits	USA	50WG/50SG	Foliar	0.07–0.1	0.07–0.1	3	0
Greenhouse cucumber	USA	50WG/50SG	Foliar	0.15	0.1–0.15	2	0
			Soil	0.15	NS	2	0
Cucurbits	AUS	50WG	Foliar	0.05–0.1	NS	3	1
Cucurbits (Field and Greenhouse)	Cyprus	50WG	Soil	0.05	0.005	3 (total)	3
			Foliar	0.10	NS		
Cucurbits	Slovenia	50WG	Foliar	0.05	0.005	3	1
Fruiting vegetables, other than cucurbits							
Fruiting vegetables	USA	50WG/50SG	Foliar	0.1	0.1	3	0
				0.15	0.15	2	0
Greenhouse tomatoes	USA	50WG/50SG	Foliar	0.15	0.15	2	0
Tomatoes (field and greenhouse)	Cyprus	50WG	Soil	0.05–0.06	0.005–0.006	3 (total)	3
			Foliar	0.10	NS		
Leafy vegetables (including Brassica leafy vegetables)							
Leafy vegetables	USA	50WG/50SG	Foliar	0.07–0.1	0.07–0.3	3	0

Crop	Country	Form.	Application				PHI, Days
			Method	Rate, kg ai/ha	Spray conc., kg ai/hL	No.	
(except Brassica vegetables)							
Brassica (cole) leafy vegetables	USA	50WG/50SG	Foliar	0.07–0.1	0.07–0.3	3	0
Root and tuber vegetables							
Tuberous and corm vegetables	USA	50WG/50SG	Foliar	0.07–0.1	0.07–0.3	3	7
Root vegetables (except sugar beets)	USA	50WG/50SG	Foliar	0.07–0.1	0.07–0.3	3	3
Potatoes	AUS	500WG	Foliar	0.07–0.1	NS	2	14
Potatoes	Slovenia	50WG	Foliar	0.08	0.016	2	14
Stems and petioles							
Leafy vegetables (except Brassica)	USA	50WG/50SG	Foliar	0.07–0.1	0.07–0.1	3	0
Cereal grains							
Wheat, rye, triticale	Slovenia	50WG	Foliar	0.07	0.014	2	28
Tree Nuts							
Tree nuts	USA	50WG/50SG	Foliar	0.07–0.1	0.01–0.02	3	40
Oilseed							
Cotton	USA	50WG/50SG	Foliar	0.05–0.1	0.02–0.05	3	30
Cotton	AUS	500 WG	Foliar	0.05–0.07	NS	2	7
Rape seed	USA	50WG/50SG	Foliar	0.1	0.1	3	7
Herbs							
Mint	USA	50WG/50SG	Foliar	0.07–0.1	0.04–0.05	3	7
Dried herbs							
Hops	USA	50WG/50SG	Foliar	0.06–0.1	0.01–0.02	3	10
Hops	Slovenia	50WG	Foliar	0.09	0.0225	2	21
Straw, fodder and forage of cereal grains and grasses							
Alfalfa seed	USA	50WG/50SG	Foliar	0.1	0.05	2	14 forage and seed 62 hay
Teas							
Tea	Japan	DF	Foliar	0.1	0.01	1	7

NS Not specified

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on supervised field trials for flonicamid uses that produced residues on the following commodities:

Commodity	Group	Table No.
Apples	FP Pome fruits	45
Pears		46
Peaches	FS Stone fruits	47
Cherries		48
Plums		49
Strawberries	FB Berries and small fruits	50
Broccoli	VB Brassica vegetables	51

Commodity	Group	Table No.
Cabbage		52
Cucumber	VC Fruiting vegetables, Cucurbits	53
Melon		54
Summer squash		55
Tomatoes	VO Fruiting vegetables, other than Cucurbits	56
Bell peppers		57
Non-bell peppers		58
Head lettuce	VL Leafy vegetables (including Brassica leafy vegetables)	59
Leaf lettuce		60
Spinach		61
Radish leaves		62
Mustard greens		63
Potato	VR Root and tuber vegetables	64
Carrot		65
Radish		66
Celery	VS Stem and petioles	67
Wheat	Cereal grains	68
Barley		69
Almonds	TN Tree nuts	70
Pecans		71
Pistachios		72
Rape seed	SO Oilseed	73
Cotton seed		74
Mint	HH Herbs	75
Hops	DH Dried herbs	76
Tea	DT Teas	77
Wheat forage and straw	AS Straw, fodder and forage of cereal grains and grasses	78
Barley forage and straw		79
Alfalfa		80
Almond hulls		81
Cottonseed gin trash		82

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.

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.

Pome fruits

Apple

Twelve independent trials were conducted on apples in the US between 1968 and 1995. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days. Apples were harvested 14–21 days after last treatment (DALT).

In Australia. fourteen independent trials were conducted on between 1983 and 2009. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days and apples were harvested 21 DALT.

The analytical method P-3568 (based on method P-3561M) was used to analyse samples collected from the US trials while method AATM-R-165 was used to analyse the samples from the Australian trials. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 35 days for the Australian trials and 297 days (ca. 10 months) for the US trials Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 45.

Table 45 Residues of Flonicamid in Apples Following Foliar Spray with Flonicamid WG Formulation in Regions of North America

Location, year (variety)	Application						DALT, days	Residues, mg/kg				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.07–0.10	0.01–0.02	100–500	3	7	21					
Lyons, NY, 1998 (Jonigold)	WG	0.10	0.01	756	3	7	0	0.065 (0.063, 0.067)	< 0.01 (< 0.01, < 0.01)	0.034 (0.030, 0.038)	< 0.01 (< 0.01, < 0.01)	IB-2001-MDG-003-00-01
							7	0.055 (0.052, 0.058)	< 0.01 (< 0.01, < 0.01)	0.037 (0.035, 0.039)	< 0.01 (< 0.01, < 0.01)	
							14	0.064 (0.062, 0.065)	< 0.01 (< 0.01, < 0.01)	0.037 (0.040, 0.049)	< 0.01 (< 0.01, < 0.01)	
							21	0.033 (0.032, 0.034)	< 0.01 (< 0.01, < 0.01)	0.039 (0.039, 0.039)	< 0.01 (< 0.01, < 0.01)	
							28	0.047 (0.060, 0.034)	< 0.01 (< 0.01, < 0.01)	0.018 (0.018, 0.017)	< 0.01 (< 0.01, < 0.01)	
Dundee, NY, 1973 (Macoun)	WG	0.10	0.01	941	3	7	21	0.037 (0.032, 0.042)	< 0.01 (< 0.01, < 0.01)	0.021 (0.017, 0.024)	< 0.01 (< 0.01, < 0.01)	
Herford, PA, 1968 (Starkrimson Red Delicious)	WG	0.10	0.02	511	3	7	20	0.037 (0.043, 0.031)	< 0.01 (< 0.01, < 0.01)	0.024 (0.011, 0.013)	< 0.01 (< 0.01, < 0.01)	
Cana, VA, 1994 (Red Delicious)	WG	0.10	0.01	940	3	7	20	0.047 (0.045, 0.048)	< 0.01 (< 0.01, < 0.01)	0.018 (0.016, 0.019)	< 0.01 (< 0.01, < 0.01)	
Conklin, MI, 1993 (Golden Delicious)	WG	0.10	0.01	794	3	7	21	0.097 (0.099, 0.095)	< 0.01 (< 0.01, < 0.01)	0.038 (0.038, 0.038)	< 0.01 (< 0.01, < 0.01)	
Menomonie, WI,	WG	0.10	0.02	468	3	7	21	0.066 (0.067,	< 0.01 (< 0.01,	0.018 (0.017,	< 0.01 (< 0.01,	

Location, year (variety)	Application						DALT, days	Residues, mg/kg				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
1985 (Prairie Sky)								0.064)	< 0.01)	0.018)	< 0.01)	
Eckert, CO, 1984 (Red Delicious)	WG	0.10	0.01	749	3	7	21	0.049 (0.044, 0.054)	< 0.01 (< 0.01, < 0.01)	0.018 (0.016, 0.020)	< 0.01 (< 0.01, < 0.01)	
Fairfield, CA, 1992 (Golden Delicious)	WG	0.10	0.02	637	3	7	14	0.111 (0.104, 0.117)	< 0.01 (< 0.01, < 0.01)	0.041 (0.037, 0.044)	< 0.01 (< 0.01, < 0.01)	
Hood River, OR, 1991 (Red Delicious)	WG	0.10	0.02	665	3	7	20	0.057 (0.055, 0.058)	< 0.01 (< 0.01, < 0.01)	0.016 (0.017, 0.015)	< 0.01 (< 0.01, < 0.01)	
Hood River, OR, 1993 (Jonigold)	WG	0.10	0.01	742	3	7	20	0.023 (0.024, 0.022)	< 0.01 (< 0.01, < 0.01)	0.015 (0.014, 0.016)	< 0.01 (< 0.01, < 0.01)	
Hood River, OR, 1995 (Gala)	WG	0.10	0.01	1032	3	7	21	0.039 (0.038, 0.039)	< 0.01 (< 0.01, < 0.01)	0.014 (0.015, 0.012)	< 0.01 (< 0.01, < 0.01)	
Outlook, WA, 1995 (Red Delicious)	WG	0.10	0.01	960	3	7	21	0.052 (0.053, 0.051)	< 0.01 (< 0.01, < 0.01)	0.019 (0.019, 0.019)	< 0.01 (< 0.01, < 0.01)	
AUS GAP	WG	NS	0.005–0.01	100–1000	3	14	21					
Batlow, New South Wales, 2006 (Sundowner)	WG	NS	0.01	2933–3352	3	7	0 14 21 27	0.34 0.16 0.12 0.093	< 0.01 < 0.01 < 0.01 < 0.01	0.017 0.040 0.049 0.054	< 0.01 < 0.01 < 0.01 < 0.01	
Batlow, New South Wales, 2006 (Sundowner)	WG	NS	0.02	2438–2952	3	7	0 14 21 27	0.86 0.24 0.23 0.17	< 0.01 < 0.01 < 0.01 < 0.01	0.032 0.074 0.11 0.11	< 0.01 < 0.01 < 0.01 < 0.01	
Batlow, New South Wales, 2000 (Pink Lady)	WG	NS	0.01	1856–2022	3	7	21	0.24	< 0.01	0.033	< 0.01	UPL-1002
Batlow, New South Wales, 2000 (Pink Lady)	WG	NS	0.02	1800–2078	3	7	21	0.47	< 0.01	0.067	0.011	
Spreyton, Tasmania, 2009 (Pink Lady)	WG	NS	0.01	201–240	3	7	0 14 21 28	0.20 0.097 0.086 0.010	< 0.01 < 0.01 < 0.01 < 0.01	0.010 0.023 0.034 0.045	< 0.01 < 0.01 < 0.01 0.012	
Grove, Tasmania, 1996 (Fuji)	WG	NS	0.02	1600–1659	3	7	21	0.024	< 0.01	0.1	0.018	
Yering,	WG	NS	0.01	4115	3	7	0	0.43	< 0.01	< 0.01	< 0.01	UP

Location, year (variety)	Application						DALT, days	Residues, mg/kg				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Victoria, 1984 (Fuji)				– 4398			14 21 28	0.16 0.15 0.16	< 0.01 < 0.01 < 0.01	0.023 0.029 0.032	0.011 0.018 0.023	L-1108
Yering, Victoria, 1984 (Fuji)	WG	NS	0.02	3978 – 4374	3	7	0 14 21 28	0.59 0.38 0.29 0.25	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.026 0.039 0.040	< 0.01 0.018 0.028 0.033	
Arding, New South Wales, 1983 (Red Delicious)	WG	NS	0.01	2153 – 2500	3	7	21	0.13	< 0.01	0.036	0.012	
Arding, New South Wales, 1983 (Red Delicious)	WG	NS	0.02	2153 – 2361	3	7	21	0.27	< 0.01	0.063	0.023	
Spreyton, Tasmania, 2010 (Golden Delicious)	WG	NS	0.01	1144 – 1337	3	7	21	0.12	< 0.01	0.069	0.01	
Spreyton, Tasmania, 2010 (Golden Delicious)	WG	NS	0.02	1248 – 1381	3	7	21	0.23	< 0.01	0.099	0.019	
Stanthorpe, Queensland, 1985 (Granny Smith)	WG	NS	0.01	2645 – 3043	3	7	0 14 21 28	0.28 0.19 0.22 0.22	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.030 0.035 0.057	< 0.01 0.026 0.033 0.050	
Stanthorpe, Queensland, 1985 (Granny Smith)	WG	NS	0.02	2101 – 3043	3	7	0 14 21 28	0.48 0.38 0.43 0.45	< 0.01 < 0.01 < 0.01 < 0.01	0.011 0.050 0.079 0.13	0.018 0.046 0.071 0.11	

Pear

Six independent trials were conducted on pears in the US between 1962 and 1996. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days. Pears were harvested 14–21 DALT.

The analytical method P-3568 (based on method P-3561M) was used to analyse the samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 329 days (ca. 11 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 46.

Table 46 Residues of Flonicamid in Pears Following Foliar Spray with Flonicamid 50 WG Formulation in Regions of North America

Location,	Application	DAL	Residues (mg/kg)	Ref

year (variety)	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	T, days	Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/WS	0.07–0.10	0.01–0.02	100–500	3	7	21					
Lyons, NY, 1968 (Clapps Favorite)	WG	0.10	0.01	936	3	7	21	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.021 (0.021, 0.020)	< 0.01 (< 0.01, < 0.01)	
Fairfield, CA, 1986 (Bartlett)	WG	0.10	0.02	654	3	7	14	0.018 (0.017, 0.019)	< 0.01 (< 0.01, < 0.01)	0.045 (0.048, 0.041)	< 0.01 (< 0.01, < 0.01)	
Isleton, CA, 1972-1996 (Bartlett)	WG	0.10	0.02	650	3	7	14	0.016 (0.013, 0.018)	< 0.01 (< 0.01, < 0.01)	0.038 (0.031, 0.044)	< 0.01 (< 0.01, < 0.01)	
Soap Lake, WA, 1962 (Bartlett)	WG	0.10	0.005	1880	3	7	21	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.023 (0.023, 0.022)	< 0.01 (< 0.01, < 0.01)	IB-2001_MDG-003-00-01
Hood River, OR, 1994 (Starkrimson)	WG	0.10	0.01	861	3	7	0	0.020 (0.019, 0.020)	< 0.01 (< 0.01, < 0.01)	0.037 (0.040, 0.034)	< 0.01 (< 0.01, < 0.01)	
							7	0.014 ((0.015, 0.013)	< 0.01 (< 0.01, < 0.01)	0.033 (0.036, 0.030)	< 0.01 (< 0.01, < 0.01)	
							14	0.010 (0.010, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.022 (0.020, 0.024)	< 0.01 (< 0.01, < 0.01)	
							21	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.026 (0.030, 0.022)	< 0.01 (< 0.01, < 0.01)	
Zillah, WA, 1985 (Bartlett)	WG	0.099	0.011	905	3	7	21	0.020 (0.019, 0.021)	< 0.01 (< 0.01, < 0.01)	0.031 (0.029, 0.033)	< 0.01 (< 0.01, < 0.01)	

Stone Fruit

Peach

Nine independent trials were conducted on peaches in the US between 1976 and 1998. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days. Peaches were harvested 10–14 DALT.

The analytical method P-3561M was used to analyse the samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was 329 days (ca. 11 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 47.

Table 47 Residues of Flonicamid in Peaches Following Foliar Spray with Flonicamid 50 WG Formulation in Regions of North America

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/WS	0.07–0.10	0.01–0.02	100–500	3	7	14					

Lyons, NY, 1998 (Harcrest)	WG	0.10	0.01	754	3	7	0	0.298 (0.284, 0.311)	< 0.01 (< 0.01, < 0.01)	0.026 (0.026, 0.026)	0.015 (0.015, 0.014)	IB-2001-MDG-005-00-01
							3	0.308 (0.289, 0.326)	< 0.01 (< 0.01, < 0.01)	0.024 (0.024, 0.025)	0.014 (0.013, 0.014)	
							7	0.190 (0.179, 0.201)	< 0.01 (< 0.01, < 0.01)	0.032 (0.037, 0.027)	0.014 (0.015, 0.012)	
							14	0.216 (0.225, 0.207)	< 0.01 (< 0.01, < 0.01)	0.038 (0.050, 0.026)	0.024 (0.026, 0.022)	
Covesville, VA, 1985 (Blake)	WG	0.10	0.02	680	3	7	14	0.087 (0.091, 0.082)	< 0.01 (< 0.01, < 0.01)	0.036 (0.043, 0.028)	0.025 (0.032, 0.018)	
Monetta, SC, 1990 (Crest Haven)	WG	0.10	0.01	829	3	7	14	0.086 (0.096, 0.075)	< 0.01 (< 0.01, < 0.01)	0.020 (0.023, 0.017)	0.012 (0.011, 0.012)	
Kinston, NC, 1995 (Legend)	WG	0.10	0.01	938	3	7	14	0.423 (0.400, 0.446)	< 0.01 (< 0.01, < 0.01)	0.020 (0.019, 0.021)	0.015 (0.014, 0.015)	
Conklin, MI, 1995 (Red Haven)	WG	0.10	0.01	979	3	7	14	0.095 (0.100, 0.090)	< 0.01 (< 0.01, < 0.01)	0.011 (< 0.01, 0.012)	0.012 (0.013, < 0.01)	
Waller, TX, 1989 (Idylwild)	WG	0.11	0.005	2120	3	7	10	0.065 (0.055, 0.074)	< 0.01 (< 0.01, < 0.01)	0.017 (0.014, 0.020)	0.011 (< 0.01, 0.012)	
Winters, CA, 1976 (Fay Elberta)	WG	0.11	0.02	521	3	7	14	0.151 (0.184, 0.117)	< 0.01 (< 0.01, < 0.01)	0.054 (0.065, 0.042)	0.023 (0.027, 0.018)	
Berenda, CA, 1988 (Last Chance)	WG	0.10	0.01	935	3	7	14	0.219 (0.218, 0.220)	< 0.01 (< 0.01, < 0.01)	0.051 (0.054, 0.047)	0.057 (0.060, 0.053)	
Selma, CA, 1996 (September Sun)	WG	0.10	0.01	938	3	7	14	0.134 (0.149, 0.119)	< 0.01 (< 0.01, < 0.01)	0.067 (0.070, 0.063)	0.023 (0.026, 0.020)	

Cherry

Six independent trials were conducted on cherries in the US between 1989 and 1995. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days. Cherries were harvested 14 DALT.

The analytical method P-3561M was used to analyse the samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was 329 days (ca. 11 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 48.

Table 48 Residues of Flonicamid in Cherries Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/WS	0.07–0.10	0.01–0.02	100–500	3	7	14					
Conklin MI, 1995 (Napoleon)	WG	0.10	0.01	945–963	3	7–8	0	0.759 (0.736, 0.782)	< 0.01 (< 0.01, < 0.01)	0.036 (0.031, 0.021)	0.022 (0.022, 0.022)	IB-2001-MDG-005-00-01
							3	0.360 (0.326, 0.394)	< 0.01 (< 0.01, < 0.01)	0.032 (0.027, 0.036)	0.021 (0.019, 0.023)	
							7	0.290 (0.282, 0.297)	< 0.01 (< 0.01, < 0.01)	0.038 (0.042, 0.034)	0.027 (0.026, 0.027)	
							14	0.273 (0.292, 0.253)	< 0.01 (< 0.01, < 0.01)	0.042 (0.042, 0.041)	0.042 (0.045, 0.039)	
Conklin MI, 1993 (Montmorency)	WG	0.10	0.01	935–954	3	7–8	14	0.276 (0.289, 0.262)	< 0.01 (< 0.01, < 0.01)	0.028 (0.028, 0.027)	0.026 (0.028, 0.024)	
Fairfield, CA, 1990 (Ranier)	WG	0.10	0.02	655	3	6	14	0.281 (0.271, 0.290)	< 0.01 (< 0.01, < 0.01)	0.167 (0.161, 0.172)	0.048 (0.044, 0.052)	
Courtland CA, 1992 (Bing)	WG	0.10	0.02	655–673	3	6	14	0.256 (0.238, 0.273)	< 0.01 (< 0.01, < 0.01)	0.044 (0.041, 0.047)	0.030 (0.029, 0.031)	
Parkdale OR, 1994 (Bing)	WG	0.10	0.01	973–1094	3	7	14	0.266 (0.302, 0.230)	< 0.01 (< 0.01, < 0.01)	0.037 (0.040, 0.034)	0.035 (0.037, 0.032)	
Granger WA, 1989, (Bing)	WG	0.10	0.01	926–963	3	7	14	0.365 (0.387, 0.343)	< 0.01 (< 0.01, < 0.01)	0.065 (0.065, 0.064)	0.062 (0.061, 0.063)	

Plum

Five independent trials were conducted on plums in the US between 1980 and 1995. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days. Plums were harvested 14 DALT.

The analytical method P-3561M was used to analyse the samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was 329 days (ca. 11 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 49.

Table 49 Residues of Flonicamid in Plums Following Foliar Spray with Flonicamid 50 WG Formulation in Regions of North America

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.07–0.10	0.01–0.02	100–500	3	7	14					
Conklin MI, 1995 (Stanley)	WG	0.10	0.01	954	3	7	14	0.041 (0.040, 0.042)	0.012 (0.014, < 0.01)	0.016 (0.017, 0.015)	< 0.01 (< 0.01, < 0.01)	IB-2001-MDG-

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Fairfield [a] CA, 1992 (French)	WG	0.10	0.02	505–514	3	5–8	14	0.044 (0.044, 0.044)	0.025 (0.026, 0.023)	0.011 (0.011, < 0.01)	0.012 (0.011, 0.012)	005-00-01
Fairfield [a] CA, 1992 (French)	WG	0.10	0.02	514	3	6–7	14	0.045 (0.051, 0.038)	0.016 (0.019, 0.013)	0.012 (< 0.01, 0.014)	0.01 (0.012, < 0.011)	
Madera CA, 1990 (Fortune)	WG	0.10	0.01	935–963	3	7	0	0.011 (0.011, 0.011)	< 0.01 (< 0.01, < 0.01)	0.040 (0.044, 0.035)	< 0.01 (< 0.01, < 0.01)	
							3	0.012 (0.012, 0.012)	< 0.01 (< 0.01, < 0.01)	0.040 (0.041, 0.038)	< 0.01 (< 0.01, < 0.01)	
							7	0.012 (0.012, 0.012)	< 0.01 (< 0.01, < 0.01)	0.043 (0.041, 0.044)	< 0.01 (< 0.01, < 0.01)	
							14	0.013 (0.012, 0.014)	< 0.01 (< 0.01, < 0.01)	0.045 (0.043, 0.046)	< 0.01 (< 0.01, < 0.01)	
Selma CA, 1997 (Howard Sun)	WG	0.10	0.01	917–926	3	7	14	0.032 (0.041, 0.023)	0.010 (0.010, < 0.01)	0.027 (0.017, 0.037)	0.010 (0.010, < 0.01)	
Hillsboro OR, 1980 (Italian)	WG	0.10	0.01	823–851	3	7	14	0.023 (0.023, 0.023)	< 0.01 (< 0.01, < 0.01)	0.011 (0.011, 0.010)	< 0.01 (< 0.01, < 0.01)	

[a] The last applications at each trial site were made on the same day and varieties were the same rendering the trials dependent

Berries and other small fruits

Strawberry

Eight independent trials were conducted on strawberries in the US in 2008. In all trials, three foliar spray applications of a SG formulation were made with a re-treatment interval of 6–8 days. Strawberries were harvested 14 DALT.

The analytical method P-3561M was used to analyse the samples. The LOQ was determined to be 0.02 mg/kg/analyte.

The maximum period of sample storage at –20 °C was 498 days (ca. 17 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 50.

Table 50 Residues of Flonicamid in Strawberries Following Foliar Spray with Beleaf 50 SG Formulation in Regions of North America

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	SG	0.10	0.02–0.10	100–500	3	7	0					
Salinas, CA, 2008 (Albion) [a]	SG	0.10	0.02–0.04	253–440	3	7	0	0.47 (0.42, 0.51)	< 0.020 (0.020, < 0.020)	0.041 (0.037, 0.044)	0.038 (0.034, 0.042)	9604
Salinas, CA, 2008	SG	0.10	0.02–0.04	299–496	3	6	0	0.59 (0.52,	< 0.020 (0.020,	0.047 (0.044,	0.033 (0.030,	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
(Albion) [a]								0.66)	< 0.020)	0.049)	0.036)	
Parlier, CA, 2008 (Seascape)	SG	0.10	0.025	402–412	3	7	0	0.54 (0.48, 0.60)	< 0.020 (0.020, < 0.020)	0.10 (0.10, 0.10)	0.056 (0.053, 0.58)	
Balm, FL, 2008 (Festival)	SG	0.10	0.027	374	3	8–11	0	0.27 (0.29, 0.24)	< 0.020 (< 0.020, < 0.020)	0.051 (0.058, 0.044)	0.028 (0.032, 0.024)	
Clinton, NC, 2008 (Chandler)	SG	0.10	0.03	318–327	3	7	0	0.33 (0.34, 0.32)	< 0.020 (< 0.020, < 0.020)	0.021 (0.020, 0.022)	< 0.020 (< 0.020, < 0.020)	
							3	0.23 (0.19, 0.27)	< 0.02 (< 0.02, < 0.02)	0.024 (0.024, 0.025)	< 0.02 (< 0.02,, < 0.02)	
							5	0.16 (0.16, 0.15)	< 0.020 (< 0.020, < 0.020)	0.031 (0.030, 0.032)	< 0.020 (< 0.020, < 0.020)	
							7	0.14 (0.14, 0.14)	< 0.020 (< 0.020, < 0.020)	0.037 (0.036, 0.037)	< 0.020 (< 0.020, < 0.020)	
Penn Yan, NY, 2008 (Honeoye)	SG	0.099–0.1	0.034–0.035	281–290	3	7	0	0.41 (0.35, 0.46)	< 0.020 (< 0.020, < 0.020)	0.046 (0.040, 0.051)	0.087 (0.084, 0.090)	
Salem, OR, 2008 (Totem)	SG	0.104–0.105	0.024	430–440	3	6–10	0	0.13 (0.11, 0.15)	< 0.020 (< 0.020, < 0.020)	0.022 (< 0.02, 0.023)	0.021 (< 0.020 , 0.022)	
Arlington, WI, 2008 (Kent)	SG	0.096–0.102	0.029	327–346	3	7–8	0	0.19 (0.20, 0.18)	< 0.020 (< 0.020, < 0.020)	< 0.020 (< 0.020, < 0.020)	< 0.020 (< 0.020, < 0.020)	

[a] The last applications at each site were made 2 months apart, rendering the trials independent

Brassica (Cole or cabbage) vegetables

Broccoli

Six independent trials were conducted on broccoli in the US during the 2003 growing season. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days. Plants were harvested 0 DALT.

The analytical method P-3561M was used to analyse the samples. The LOQ was determined to be 0.025 mg/kg/analyte.

The maximum period of sample storage at –20 °C was 4 days. Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 51.

Table 51 Residues of Flonicamid in Broccoli Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location,	Application	DALT,	Residues (mg/kg)	Ref

year (variety)	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	days	Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.07–0.10	0.07–0.10	100	3	7	0					
East Bernard, TX, 2003 (Early Dividend)	WG	0.10	0.10	95–96	3	6–7	0	0.428 (0.484. 0.372)	< 0.025 (< 0.025, < 0.025)	< 0.025 (0.025, < 0.025)	0.077 (0.086, 0.068)	P-3679
Camarillo CA, 2003 (Marathon)	WG	0.11	0.11	93–95	3	5–8	0	0.373 (0.331, 0.435)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.034 (0.031, 0.036)	
							1	0.432 (0.338, 0.525)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.045 (0.041, 0.048)	
							3	0.308 (0.327, 0.288)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.048 (0.046, 0.049)	
							7	0.178 (0.186, 0.170)	< 0.025 (< 0.025, < 0.025)	0.032 (0.033, 0.030)	0.060 (0.062, 0.057)	
Visalia CA, 2003 (Waltham 29)	WG	0.10	0.10	94	3	7	0	0.462 (0.430, 0.493)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.144 (0.127, 0.161)	
Casa Grande AZ, 2003 (Marathon)	WG	0.10	0.11	95	3	6–7	0	0.499 (0.416, 0.581)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	
Yuma AZ, 2003 (Everest)	WG	0.10	0.11	93–96	3	7–9	0	0.250 (0.268, 0.232)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.027 (0.028, < 0.025)	
Hillsboro OR, 2003 (Packman)	WG	0.10	0.06	153–159	3	7	0	0.553 (0.515, 0.590)	< 0.025 (< 0.025, < 0.025)	0.056 (0.059, 0.053)	0.144 (0.150, 0.137)	

Cabbage

Six independent trials were conducted on cabbage in the US during the 2003 growing season. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days. Cabbage heads were harvested 0 DALT.

The analytical method P-3561M was used to analyse samples. The LOQ was determined to be 0.025 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 180 days (6 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 52.

Table 52 Residues of Flonicamid in Cabbage Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Matrix	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.07–0.10	0.07–0.10	100	3	7	0						
North Rose NY, 2003 (Early Thunder)	WG	0.10	0.10	94	3	7	0	Cabbage w/wrapper leaves	0.062 (0.066, 0.057)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.032 (0.034, 0.029)	P-3679
								Cabbage w/out wrapper leaves	< 0.025 (0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.091 (0.099, 0.082)	0.165 (0.184, 0.145)	
Delmar DE, 2003 (Blue Thunder)	WG	0.10	0.11	93–94	3	6–7	0	Cabbage w/wrapper leaves	0.205 (0.217, 0.193)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.053 (0.055, 0.051)	
								Cabbage w/out wrapper leaves	< 0.025 (0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.085 (0.088, 0.082)	0.141 (0.152, 0.129)	
Jennings FL, 2003 (Bravo)	WG	0.10	0.10	97	3	7	0	Cabbage w/wrapper leaves	1.262 (1.281, 1.243)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.089 (0.087, 0.090)	
								Cabbage w/out wrapper leaves	1.138 (1.067, 1.208)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.072 (0.069, 0.075)	
East Bernard, TX, 2003 (Early Jersey Wakefield)	WG	0.10	0.10	91–97	3	7–8	0	Cabbage w/wrapper leaves	0.288 (0.311, 0.265)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.037 (0.036, 0.037)	
								Cabbage w/out wrapper leaves	0.055 (0.059, 0.050)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	
Ellendale MN, 2003 (Dannish Ball)	WG	0.10	0.12	88–90	3	7	0	Cabbage w/wrapper leaves	0.025 (0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.074 (0.072, 0.075)	0.127 (0.123, 0.130)	
								Cabbage w/out wrapper leaves	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.070 (0.061, 0.079)	0.110 (0.087, 0.132)	
Visalia CA, 2003 (Copenhagan)	WG	0.10	0.11	94	3	7	0	Cabbage w/wrapper leaves	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.031 (0.031, 0.031)	
								Cabbage w/out wrapper leaves	< 0.025, (< 0.025 < 0.025)	< 0.025 (< 0.025, < 0.025)	0.035 (0.033, 0.036)	0.067 (0.064, 0.069)	

Fruiting vegetables, cucurbits

Cucumber

Six independent trials were conducted on field cucumbers in the US during the 2001 growing season. In all trials, three foliar spray applications of a WG formulation were made at 0.10 kg ai/ha with a re-treatment interval of 6–7 days. Cucumbers were harvested 0 DALT. Four independent trials were conducted on greenhouse cucumbers in Canada and the US during the 2008 and 2009 growing seasons. In each of the greenhouse trials, one of the plots was treated with two foliar spray applications of a SG formulation at 0.15 kg ai/ha with a re-treatment interval of 6–7 days. The other plot was treated twice via syringe to the rock wool cubes in which the plants were grown. Application rates were determined using an average plant density of 2.4 plants per square meter, regardless of the actual density in the respective trials. The nominal rate was 0.15 kg ai/ha per application for a total range of 0.30 kg ai/ha per season. In all trials, cucumbers were harvested 0 DALT.

Two independent trials were also conducted on field cucumbers in Australia during the 2011 and 2012 growing seasons. In both trials, three foliar spray applications of a WG formulation were made at 0.10 kg ai/ha or 0.20 kg ai/ha with a re-treatment interval of 7 days. Cucumbers were harvested 0, 1, 3 and 7 DALT.

For the North American trials, the analytical method P-3561M was used to analyse all samples. The LOQ for the field cucumbers was determined to be 0.025 mg/kg/analyte while the LOQ for greenhouse cucumbers was 0.02 mg/kg/analyte. In Australia, the analytical method AATM-R-165 was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 340 days (ca. 11.5 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 53.

Table 53 Residues of Flonicamid in Field and Greenhouse Cucumbers Following Foliar Spray with Flonicamid 50 WG (Field) in North American Regions and Australia and Beleaf 50SG (Greenhouse) in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Field Cucumbers												
US GAP	WG	0.07–0.10	0.07–0.10	100	3	7	0					
Cotton GA, 2001 (Cross Country)	WG	0.10	0.05	187	3	7	0	0.065 (0.063, 0.067)	< 0.01 (< 0.01, < 0.01)	0.042 (0.040, 0.044)	< 0.01 (< 0.01, < 0.01)	IB-2001-MD G-007-00-01
Rose Hill NC, 2001 (Poinsett)	WG	0.10	0.05	187	3	7	0	0.081 (0.086, 0.076)	< 0.01 (< 0.01, < 0.01)	0.052 (0.054, 0.050)	0.027 (0.028, 0.01)	
							1	0.116 (0.118, 0.113)	< 0.01 (< 0.01, < 0.01)	0.085 (0.085, 0.084)	0.082 (0.080, 0.084)	
							3	0.102 (0.094, 0.110)	< 0.01 (< 0.01, < 0.01)	0.060 (0.055, 0.064)	0.067 (0.063, 0.071)	
							7	0.049 (0.042, 0.056)	< 0.01 (< 0.01, < 0.01)	0.067 (0.063, 0.070)	0.075 (0.070, 0.079)	
Hobe Sound FL, 2001	WG	0.10	0.05	187	3	6–7	0	0.073 (0.076, 0.069)	< 0.01 (< 0.01, < 0.01)	0.045 (0.046, 0.044)	0.026 (0.027, 0.024)	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
(Speedway)												
Northwood ND, 2001 (Market-more 76)	WG	0.10	0.05	187	3	7	0	0.055 (0.047, 0.063)	< 0.01 (< 0.01, < 0.01)	0.102 (0.099, 0.104)	0.076 (0.069, 0.082)	
Arkansas WI, 2001 (Eureka)	WG	0.10	0.05	187	3	7	0	0.055 (0.052, 0.058)	< 0.01 (< 0.01, < 0.01)	0.155 (0.145, 0.164)	0.105 (0.098, 0.111)	
Eakly OK, 2001 (Boston pickling)	WG	0.10	0.05	178–187	3	6–7	0	0.039 [a] (0.041, 0.040; 0.038, 0.038)	< 0.01[1] (< 0.01, < 0.01; < 0.01, < 0.01)	0.090[1] (0.059, 0.057; 0.123, 0.121)	0.070[1] (0.043, 0.041; 0.108, 0.086)	
AUS GAP	WG	0.05–0.10			3	14	1					
Bowen, Queensland, 2011 (Black Prince)	WG	0.10	0.02	502	3	7	0	0.031	< 0.01	0.045	0.043	UPL-1003
							1	0.03	< 0.01	0.063	0.051	
							3	0.028	< 0.01	0.06	0.054	
							7	0.029	< 0.01	0.071	0.061	
		0.20	0.04	502	3	7	0	0.059	< 0.01	0.073	0.056	
							1	0.042	< 0.01	0.066	0.052	
							3	0.048	< 0.01	0.054	0.043	
							7	0.048	< 0.01	0.12	0.1	
Bowen, Queensland, 2012 (Gremlin)	WG	0.10	0.03	395	3	7	0	0.034	< 0.01	0.065	0.096	UPL-1007
							1	0.027	< 0.01	0.055	0.074	
							3	0.031	< 0.01	0.063	0.12	
							7	0.019	< 0.01	0.077	0.12	
		0.20	0.05	395	3	7	0	0.052	< 0.01	0.086	0.13	
							1	0.044	< 0.01	0.062	0.1	
							3	0.055	< 0.01	0.073	0.12	
							7	0.014	< 0.01	0.032	0.071	
Greenhouse Cucumbers												
US GAP	SG	0.15	0.15	minimum 100	2	7	0					71512-9
Foliar application												
Fort Collins CO, USA, 2009 (DRL 1061 F1)	SG	0.16	0.03	505–561	2	6	0	0.054 (0.046, 0.061)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	0.02 (0.02 0.02)	
Salisbury MD, USA, 2008 (Samir)	SG	0.15	0.03	468	2	7	0	0.14 (0.14, 0.14)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	0.03 (0.03 0.02)	
Crossville TN, USA, 2008 (DRL 1061 F1)	SG	0.16	0.05	290	2	7	0	0.54 (0.69, 0.39)	<0.02 (<0.02 <0.02)	0.03 (0.03, 0.03)	0.02 (<0.02, 0.03)	
Harrow ON, CAN, 2009 (Pyralis)	SG	0.14–0.15	0.012	1162–1197	2	7	0	0.061 (0.059, 0.062)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	
							3	0.053 (0.052,	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
								0.054)				
							5	0.048 (0.046, 0.050)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	0.02 (<0.02 (0.02)	
							7	0.042 (0.038, 0.048)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	0.03 (0.02, 0.04)	
Soil application												
Fort Collins CO, USA, 2009 (DRL 1061 F1)	SG	0.15	N/A	2	6		0	0.13 (0.13, 0.12)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	
Salisbury MD, USA, 2008 (Samir)	SG	0.15	N/A	2	7		0	0.20 (0.20, 0.20)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	0.11 (0.11, 0.11)	
Crossville TN, USA, 2008 (DRL 1061 F1)	SG	0.15	N/A	2	7		0	0.094 (0.094, 0.094)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	
Harrow ON, CAN, 2009 (Pyralis)	SG	0.15	N/A	2	7		0	0.14 (0.13, 0.14)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	
							3	0.15 (0.14, 0.16)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	
							5	0.16 (0.17, 0.15)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	0.02 (0.03, 0.02)	
							7	0.16 (0.18, 0.13)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	0.04 (0.05, 0.03)	

[a] Mean of four duplicate samples

N/A = Not applicable as treatment was made via syringe to the growth media

Melons

Six independent trials were conducted on melons in the USA during the 2003 growing season. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days. Melons were harvested 0 DALT.

The analytical method P-3561M was used to analyse samples. The LOQ was determined to be 0.01 mg/kg/analyte. On average, for all the trials, 12 fruits were sampled from each control and treated plot. In trials 5, 11, 13 and 17, fruits were quartered and each quarter was placed in a plastic bag and stored in a freezer. In Trial 14, melons were cut and 1/8th of each melon was placed into plastic bags. In Trial 15, the study only reported that fruits were placed into plastic bags. Trials 1 through 4, 6 through 10, 12 and 16 were conducted on cucumbers or summer squash.

Five independent trials were conducted on rockmelons (cantaloupe) in Australia during the 2010, 2011 and 2012 growing seasons. In all trials, three foliar spray applications of a WG

formulation were made at 0.10 kg ai/ha or 0.20 kg ai/ha with a re-treatment interval of 7 days and DALTs of 0, 1, 3 and 7 days.

The analytical method AATM-R-165 was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte. Five commercially mature fruit weighing > 7 kg were collected by hand from five plants per plot and placed in labelled specimen bags. No specimens were collected from the buffer areas of each plot. Sampling was conducted after the spray solution had dried at the 0-day DALT. Any soil adhering to the fruit was removed by brushing not washing. Gloves were worn and changed between treatments. All specimens were double bagged and labelled in accordance with the specimen list defined in the study plan. No information was provided as to whether the melons were cut prior to bagging.

A total of thirteen independent trials were conducted in Southern Europe (France, Italy and Spain) on field and greenhouse-grown melons during the 2003, 2004 and 2011 growing seasons. In the trials conducted during 2003 and 2004, three foliar spray applications of a WG formulation were made at 0.08 kg ai/ha with re-treatment intervals of 4–10 days. In the trials conducted in 2011, three foliar spray applications of a WG formulation were made at 0.05 kg ai/ha with a re-treatment interval of 7 days. In all trials, melons were harvested 0, 1, 2, 3 and/or 7 DALT.

For the 2003 and 2004 trials, the analytical method was based on the method A22-00-02 and adapted to melon peel by changing the C_{18} clean-up. For the 2011 trials, the LC-MS/MS method AGR/MOA/IKI220-1 was used to analyse all samples. For both methods, the LOQ was determined to be 0.01 mg/kg/analyte for each peel and pulp. In general, each harvested fruit was cut in minimum of two slices. From each retained slice, the peel was separated from the pulp.

In total, the maximum period of sample storage at –20 °C was up to 340 days (11.5 months). Storage stability data on water content commodities show that residues are stable for at least 23 months. The results are summarized in Table 54.

Table 54 Residues of Flonicamid in Whole Melons Following Foliar Spray with a 50 WG Formulation of Flonicamid in North American Regions, Australia and Southern EU

Location, year (variety)	Trial No.	Application						DALT, days	Residues (mg/kg)				Ref
		Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP		WG	0.07–0.10	0.07–0.10	100	3	7	0					
Rose Hill NC, 2001 (Hales Best Jumbo)	5	WG	0.10	0.05	187	3	7	0	0.086 (0.089, 0.082)	< 0.01 (< 0.01, < 0.01)	0.107 (0.107, 0.107)	0.044 (0.046, 0.042)	IB-2001-MDG-007-00-01
Arkansaw WI, 2001 (Hybrid Primo)	11	WG	0.10	0.05	187	3	7	0	0.037 (0.036, 0.037)	< 0.01 (< 0.01, < 0.01)	0.054 (0.057, 0.050)	0.028 (0.029, 0.026)	
East Bernard TX, 2001 (Hales Best 36)	13	WG	0.10	0.08	131	3	7	0	0.056 (0.054, 0.058)	< 0.01 (< 0.01, < 0.01)	0.074 (0.076, 0.072)	0.023 (0.023, 0.022)	
Arbuckle CA, 2001 (Tendral Amaraillo Tandio)	14	WG	0.10	0.04	234	3	7	0	0.050 (0.045, 0.055)	< 0.01 (< 0.01, < 0.01)	0.044 (0.041, 0.046)	0.026 (0.023, 0.028)	
Maricopa AZ, 2001 (Olympic Gold)	15	WG	0.10	0.05	178–187	3	7	0	0.019 (0.021, 0.017)	< 0.01 (< 0.01, < 0.01)	0.077 (0.075, 0.079)	0.047 (0.047, 0.046)	
Fresno CA, 2001 (Top Mark)	17	WG	0.10	0.05	187–196	3	7	0	0.031 (0.029, 0.033)	< 0.01 (< 0.01, < 0.01)	0.082 (0.081, 0.083)	0.053 (0.053, 0.052)	
								1	0.019 (0.021, 0.017)	< 0.01 (< 0.01, < 0.01)	0.092 (0.113, 0.071)	0.059 (0.071, 0.046)	

Location, year (variety)	Trial No.	Application						DALT, days	Residues (mg/kg)				Ref
		Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
								3	0.024 (0.024, 0.023)	< 0.01 (< 0.01, < 0.01)	0.113 (0.102, 0.124)	0.082 (0.075, 0.088)	
								7	0.020 (0.014, 0.026)	< 0.01 (< 0.01, < 0.01)	0.153 (0.153, 0.153)	0.125 (0.116, 0.134)	
AUS GAP		WG	0.05–0.10	NS	NS	3		1					
Bowen, Queensland, 2011 (Hotshot)	NA	WG	0.10	0.02	499	3	7	0	0.091	< 0.01	0.065	0.023	UPL-1003
								1	0.17	< 0.01	0.063	0.026	
								3	0.076	< 0.01	0.047	0.017	
								7	0.031	< 0.01	0.049	0.023	
			0.20	0.04	499	3	7	0	0.25	< 0.01	0.14	0.055	
								1	0.25	< 0.01	0.13	0.046	
								3	0.18	< 0.01	0.13	0.06	
								7	0.098	< 0.01	0.17	0.071	
Caversham, Western Australia, 2010 (Sienna)	NA	WG	0.10	0.017	582	3	7	0	0.078	< 0.01	0.034	< 0.01	
								1	0.092	< 0.01	0.049	< 0.01	
								3	0.043	< 0.01	0.04	< 0.01	
								7	0.034	< 0.01	0.12	0.026	
			0.20	0.034	582	3	7	0	0.18	< 0.01	0.053	0.013	
								1	0.14	< 0.01	0.053	0.013	
								3	0.13	< 0.01	0.091	0.021	
								7	0.092	< 0.01	0.13	0.039	
Wallaville, Queensland, 2012 (Caribbean Queen)	NA	WG	0.10	0.02	636	3	7	0	0.038	< 0.01	0.039	0.016	UPL-1007
								1	0.05	< 0.01	0.033	0.012	
								3	0.031	< 0.01	0.048	0.019	
								7	0.05	< 0.01	0.11	0.03	
			0.20	0.03	636	3	7	0	0.058	< 0.01	0.04	0.018	
								1	0.12	< 0.01	0.051	0.026	
								3	0.084	< 0.01	0.052	0.025	
								7	0.12	< 0.01	0.19	0.089	
Caversham, Western Australia, 2011 (Sienna)	NA	WG	0.10	0.017	576	3	7	0	0.13	< 0.01	0.091	0.054	
								1	0.047	< 0.01	0.055	0.038	
								3	0.032	< 0.01	0.074	0.04	
								7	0.036	< 0.01	0.084	0.053	
			0.20	0.03	576	3	7	0	0.083	< 0.01	0.08	0.048	
								1	0.11	< 0.01	0.13	0.083	
								3	0.039	< 0.01	0.13	0.083	
								7	0.066	< 0.01	0.16	0.099	
Whitton, New South Wales, 2011 (Dubloon)	NA	WG	0.10	0.03	317	3	7	0	0.05	< 0.01	0.049	0.015	
								1	0.028	< 0.01	0.04	0.015	
								3	< 0.01	< 0.01	0.042	0.018	
								7	0.021	< 0.01	0.04	0.017	
			0.20	0.06	317	3	7	0	0.11	< 0.01	0.063	0.024	
								1	0.025	< 0.01	0.059	0.02	
								3	0.026	< 0.01	0.046	0.023	
								7	0.044	< 0.01	0.067	0.033	

Location, year (variety)	Application							Commodity	Residues (mg/kg)				Ref.
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Slovenia GAP	WG	0.05	0.005	1000	3	7	1						
Greenhouse melons													
Languedoc Le Cailar, South France, 2003	WG	0.080	0.010	788–802	3	8–9	0	Peel	0.13	< 0.01	0.07	0.05	FA-22-03-
								Pulp	0.01	< 0.01	0.04	0.02	
								Whole	0.06	< 0.01	0.05	0.03	

Location, year (variety)	Application							Commodity	Residues (mg/kg)				Ref.
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Slovenia GAP	WG	0.05	0.005	1000	3	7	1						
(Arpege) [a]								fruit					01/01
							1	Peel	0.08	< 0.01	0.06	0.07	
								Pulp	0.02	< 0.01	0.06	0.02	
								Whole fruit	0.04	< 0.01	0.06	0.04	
							2	Peel	0.09	< 0.01	0.07	0.04	
								Pulp	0.01	< 0.01	0.07	0.04	
								Whole fruit	0.04	< 0.01	0.07	0.04	
							3	Peel	0.08	< 0.01	0.06	0.05	
								Pulp	0.01	< 0.01	0.07	0.02	
								Whole fruit	0.04	< 0.01	0.07	0.03	
							7	Peel	0.06	< 0.01	0.04	0.06	
								Pulp	0.01	< 0.01	0.08	0.06	
								Whole fruit	0.03	< 0.01	0.06	0.06	
Almeria, Spain, 2003 (Cantarino) [b]	WG	0.080	0.085	900–930	3	6–8	0	Peel	0.05	< 0.01	0.02	0.08	FA-22-03-02/01
								Pulp	< 0.01	< 0.01	< 0.01	< 0.01	
								Whole fruit	0.03	< 0.01	0.01	0.04	
							1	Peel	0.07	< 0.01	0.03	0.09	
								Pulp	< 0.01	< 0.01	< 0.01	< 0.01	
								Whole fruit	0.04	< 0.01	0.02	0.05	
							2	Peel	0.03	< 0.01	0.03	0.10	
								Pulp	< 0.01	< 0.01	< 0.01	0.04	
								Whole fruit	0.02	< 0.01	0.02	0.07	
							3	Peel	0.04	< 0.01	< 0.01	0.05	
								Pulp	< 0.01	< 0.01	< 0.01	0.02	
								Whole fruit	0.02	< 0.01	< 0.01	0.04	
							7	Peel	0.01	< 0.01	0.06	0.16	
								Pulp	< 0.01	< 0.01	< 0.01	< 0.01	
								Whole fruit	0.01	< 0.01	0.03	0.08	
Veneto, Italy, 2003 (Tazio) [c]	WG	0.080–0.082	0.010	801–819	3	6–7	0	Peel	0.07	< 0.01	0.05	0.05	FA-22-03-03/01
								Pulp	< 0.01	< 0.01	0.03	0.02	
								Whole fruit	0.02	< 0.01	0.04	0.03	
							1	Peel	0.10	< 0.01	0.06	0.05	
								Pulp	< 0.01	< 0.01	0.03	0.03	
								Whole fruit	0.03	< 0.01	0.04	0.05	
							2	Peel	0.09	< 0.01	0.06	0.08	
								Pulp	< 0.01	< 0.01	0.04	0.04	
								Whole fruit	0.03	< 0.01	0.05	0.05	
							3	Peel	0.05	< 0.01	0.08	0.10	
								Pulp	0.01	< 0.01	0.04	0.06	
								Whole fruit	0.02	< 0.01	0.05	0.07	
							7	Peel	0.08	< 0.01	0.13	0.17	
								Pulp	< 0.01	< 0.01	0.04	0.05	
								Whole fruit	0.02	< 0.01	0.07	0.09	
Murcia, Spain,	WG	0.079–	0.083–	944–	3	8	1	Peel	0.04	< 0.01	0.09	0.19	FA-

Location, year (variety)	Application							Commodity	Residues (mg/kg)				Ref.
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Slovenia GAP	WG	0.05	0.005	1000	3	7	1						
2004 (Cantagrillo) [e]		0.081	0.084	964				Pulp	< 0.01	< 0.01	0.02	0.02	22-04-02
								Whole fruit	0.02	< 0.01	0.05	0.09	
Aquitaine, South France, 2004 (Amigo)	WG	0.079–0.081	0.010	788–813	3	6–9	1	Peel	0.05	< 0.01	0.14	0.09	FA-22-04-04/02
								Pulp	< 0.01	< 0.01	0.02	0.01	
								Whole fruit	0.02	< 0.01	0.06	0.03	
Field Melons													
Valencia, Spain, 2003 (Piel Sapo) [b]	WG	0.080	0.0084	932–960	3	6–8	1	Peel	0.07	< 0.01	0.03	0.10	FA-22-03-02/02
								Pulp	< 0.01	< 0.01	< 0.01	< 0.01	
								Whole fruit	0.04	< 0.01	0.02	0.05	
Emilia Romagna, Italy, 2003 (Bingo) [c]	WG	0.078–0.083	0.013	584–620	3	4–10	1	Peel	0.10	< 0.01	0.06	0.09	FA-22-03-03/02
								Pulp	< 0.01	< 0.01	0.03	0.04	
								Whole fruit	0.03	< 0.01	0.02	0.03	
Emilia Romagna, Italy, 2004 (Colorado) [d]	WG	0.080–0.082	0.013	599–618	3	7	0	Peel	0.10	< 0.01	0.06	0.05	FA-22-04-02
								Pulp	< 0.01	< 0.01	0.03	0.02	
								Whole fruit	0.04	< 0.01	0.04	0.03	
							1	Peel	0.03	< 0.01	0.07	0.05	
								Pulp	0.01	< 0.01	0.03	0.02	
								Whole fruit	0.02	< 0.01	0.05	0.03	
							2	Peel	0.05	< 0.01	0.06	0.05	
								Pulp	0.01	< 0.01	0.03	0.02	
								Whole fruit	0.03	< 0.01	0.04	0.03	
							3	Peel	0.02	< 0.01	0.07	0.05	
								Pulp	< 0.01	< 0.01	0.02	0.01	
								Whole fruit	0.01	< 0.01	0.03	0.03	
							7	Peel	0.04	< 0.01	0.10	0.07	
								Pulp	0.01	< 0.01	0.04	0.02	
								Whole fruit	0.02	< 0.01	0.06	0.04	
Poitou-Charentes, South France, 2004 (Cezanne)	WG	0.075–0.084	0.013	564–631	3	7–8	0	Peel	0.09	< 0.01	0.01	0.01	FA-22-04-04/01
								Pulp	< 0.01	< 0.01	0.01	< 0.01	
								Whole fruit	0.04	< 0.01	0.01	< 0.01	
							1	Peel	0.10	< 0.01	0.02	0.02	
								Pulp	< 0.01	< 0.01	0.01	< 0.01	
								Whole fruit	0.05	< 0.01	0.02	0.01	
							2	Peel	0.04	0.01	0.02	0.01	
								Pulp	< 0.01	< 0.01	0.01	< 0.01	
								Whole fruit	0.02	< 0.01	0.02	< 0.01	
							3	Peel	0.05	< 0.01	0.02	0.02	
								Pulp	< 0.01	< 0.01	0.01	0.01	
								Whole fruit	0.02	< 0.01	0.02	0.01	
							7	Peel	0.04	0.01	0.03	0.03	
								Pulp	< 0.01	< 0.01	0.02	0.01	
								Whole fruit	0.02	< 0.01	0.03	0.02	
Pyrenees	WG	0.049–	0.008	592–	3	7	0	Peel	0.03	Not	0.17	0.09	S-11-

Location, year (variety)	Application							Commodity	Residues (mg/kg)				Ref.
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Slovenia GAP	WG	0.05	0.005	1000	3	7	1						
Orientales, France, 2011 (Stellio)		0.050		610				Pulp	< 0.01	analysed	0.04	0.02	02600
								Whole fruit	0.02		0.09	0.05	
							1	Peel	< 0.01		0.14	0.08	
								Pulp	< 0.01		0.02	0.01	
								Whole fruit	< 0.01		0.07	0.04	
							3	Peel	0.03		0.16	0.14	
								Pulp	0.01		0.08	0.04	
								Whole fruit	0.02		0.11	0.08	
Emilia Romagna, Italy, 2011 (Bacir)	WG	0.047–0.049	0.006	780–805	3	7	0	Peel	0.02	Not analysed	0.08	0.02	
								Pulp	< 0.01		0.02	< 0.01	
								Whole fruit	< 0.01		0.05	0.01	
							1	Peel	0.03		0.08	0.03	
								Pulp	< 0.01		0.03	< 0.01	
								Whole fruit	0.01		0.05	0.02	
							3	Peel	0.01		0.11	0.04	
								Pulp	< 0.01		0.03	< 0.01	
								Whole fruit	< 0.01		0.06	0.02	
Castellon, Spain, 2011 (Sancho)	WG	0.048–0.050	0.006	760–805	3	7	0	Peel	0.13	Not analysed	0.11	0.10	
								Pulp	< 0.01		< 0.01	< 0.01	
								Whole fruit	0.07		0.06	0.05	
							1	Peel	0.08		0.13	0.14	
								Pulp	< 0.01		0.01	< 0.01	
								Whole fruit	0.04		0.07	0.08	
							3	Peel	0.05		0.13	0.14	
								Pulp	< 0.01		0.02	0.01	
								Whole fruit	0.02		0.07	0.07	
Albacete, Spain, 2011 (Piel de Sapo)	WG	0.050	0.006	798–806	3	7	0	Peel	0.01	Not analysed	0.09	0.03	
								Pulp	< 0.01		0.02	< 0.01	
								Whole fruit	0.04		0.05	0.02	
							1	Peel	0.10		0.02	0.05	
								Pulp	< 0.01		0.14	< 0.01	
								Whole fruit	0.04		0.07	0.02	
							3	Peel	0.02		0.13	0.05	
								Pulp	< 0.01		0.02	< 0.01	
								Whole fruit	< 0.01		0.07	0.03	

The experimental weight percentage ratio between peel and pulp was reported to be:

[a] 38.1% for peel and 61.9% for pulp

[b] 50.5% for peel and 49.5% for pulp

[c] 31.1 % for peel and 68.9% for pulpl

[d] 38.9 % for peel and 61.7% for pulp

[e] 41.6% for peel and 58.4% for pulp

Squash

Five independent trials were conducted on summer squash in the US during the 2001 growing season. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 6–7 days. Squash was harvested 0 DALT.

The analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

Three independent trials were also conducted on pumpkin in Australia during the 2010 and 2012 growing seasons. In all trials, three foliar spray applications of a WG formulation were made at 0.10 kg ai/ha or 0.20 kg ai/ha with a re-treatment interval of 7 days. Pumpkins were harvested 0, 1, 3 and 7 DALT.

The analytical method AATM-R-165 was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The Meeting also received four independent trials conducted on pumpkin in Hungary during the 2012 growing season. In all trials, two foliar spray applications of a WG formulation were made at 0.08 kg ai/ha with a re-treatment interval of 7 days. Pumpkins were harvested 0, 1, 3 and 7 DALT.

The analytical method SOP R 700 FEJ2 was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C for all trials was up to 351 days (12 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 55.

Table 55 Residues of Flonicamid in Summer Squash Following Foliar Spray with Flonicamid 50 WG Formulation from North American Regions, Australia and Hungary

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG	0.07–0.10	0.07–0.10	100	3	7	0					71512-9
North Rose NY, 2001 (Zucchini Select)	WG	0.10	0.04	234	3	7	0	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	IB-2001-MDG-007-00-01
Rose Hill NC, 2001 (Early Prolific Straight-neck)	WG	0.10	0.05	187	3	7	0	0.031 (0.031, 0.031)	< 0.01 (< 0.01, < 0.01)	0.053 (0.050, 0.055)	0.035 (0.033, 0.036)	
Hobe Sound FL, 2001 (Rogers Hybrid)	WG	0.10	0.05	187–196	3	6–7	0	0.032 (0.032, 0.031)	< 0.01 (< 0.01, < 0.01)	0.063 (0.064, 0.062)	0.039 (0.038, 0.039)	
Arkansaw WI, 2001 (Hybrid Monet)	WG	0.10	0.05	187–196	3	6–7	0	0.042 (0.040, 0.043)	< 0.01 (< 0.01, < 0.01)	0.073 (0.081, 0.065)	0.039 (0.043, 0.035)	
							1	0.025 (0.024, 0.026)	< 0.01 (< 0.01, < 0.01)	0.080 (0.075, 0.084)	0.031 (0.028, 0.033)	
							3	0.026 (0.023, 0.028)	< 0.01 (< 0.01, < 0.01)	0.083 (0.075, 0.091)	0.034 (0.030, 0.037)	
							7	0.016 (0.015, 0.017)	< 0.01 (< 0.01, < 0.01)	0.087 (0.081, 0.092)	0.027 (0.026, 0.028)	
Madera CA, USA, 2001 (Sundance)	WG	0.10	0.04	281	3	7	0	0.031 (0.033, 0.029)	< 0.01 (< 0.01, < 0.01)	0.065 (0.076, 0.053)	0.036 (0.042, 0.030)	
AUS GAP	WG	0.05–0.10	NS	NS	3		1					

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Ballandean, South Queensland, 2010 (Butternut Large)	WG	0.10	0.015	667	3	7	0	0.12	< 0.01	0.039	0.033	UPL-1003
							1	0.079	< 0.01	0.057	0.05	
							3	0.07	< 0.01	0.059	0.05	
							7	0.029	< 0.01	0.04	0.047	
		0.20	0.03	667	3	7	0	0.27	< 0.01	0.082	0.069	
							1	0.1	< 0.01	0.084	0.097	
							3	0.086	< 0.01	0.074	0.093	
							7	0.054	< 0.01	0.12	< 0.01	
Bowen,Queensland, 2012 (Ken's Special)	WG	0.10	0.02	638	3	7	0	0.01	< 0.01	< 0.01	< 0.01	UPL-1107
							1	< 0.01	< 0.01	< 0.01	< 0.01	
							3	< 0.01	< 0.01	< 0.01	< 0.01	
							7	< 0.01	< 0.01	< 0.01	< 0.01	
		0.20	0.03	638	3	7	0	0.039	< 0.01	< 0.01	< 0.01	
							1	< 0.01	< 0.01	< 0.01	< 0.01	
							3	< 0.01	< 0.01	< 0.01	< 0.01	
							7	0.013	< 0.01	< 0.01	< 0.01	
Bowen, Queensland, 2012 (Sunset QHI)	WG	0.10	0.025	401	3	7	0	0.026	< 0.01	< 0.01	0.017	
							1	0.013	< 0.01	0.013	0.021	
							3	0.042	< 0.01	0.012	0.021	
							7	0.017	< 0.01	0.023	0.031	
		0.20	0.05	401	3	7	0	0.068	< 0.01	0.032	0.057	
							1	0.11	< 0.01	0.023	0.03	
							3	0.063	< 0.01	0.022	0.031	
							7	0.043	< 0.01	0.02	0.043	

Location, year (variety)	Application						PHI, days	Residues (mg/kg)			Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA	TFNG	
Slovenia GAP	WG	0.05	0.005–0.012	400–1000	3	7	1				12 ISK AA 0701
Kapolnasnyék, Hungary, 2012 (NS)	WG	0.08	NS		2	7	0	< 0.01	< 0.01	< 0.01	
							1	< 0.01	< 0.01	< 0.01	
							3	< 0.01	< 0.01	< 0.01	
							7	< 0.01	< 0.01	< 0.01	
Füle, Hungary, 2012 (NS)	WG	0.08	NS		2	7	0	< 0.01	< 0.01	< 0.01	
							1	< 0.01	< 0.01	< 0.01	
							3	< 0.01	< 0.01	< 0.01	
							7	< 0.01	< 0.01	< 0.01	
Vereb, Hungary, 2012 (NS)	WG	0.08	NS		2	7	0	< 0.01	< 0.01	< 0.01	
							1	< 0.01	< 0.01	< 0.01	
							3	< 0.01	< 0.01	< 0.01	
							7	< 0.01	< 0.01	< 0.01	
Székesfehérvar-Csala, Hungary, 2012 (NS)	WG	0.08	NS		2	7	0	< 0.01	< 0.01	< 0.01	
							1	< 0.01	< 0.01	< 0.01	
							3	< 0.01	< 0.01	< 0.01	
							7	< 0.01	< 0.01	< 0.01	

Fruiting vegetables, other than cucurbits

Tomatoes

Twenty-six independent trials were conducted on field tomatoes in the US between 2001 and 2010. For 12 of the trials, three foliar spray applications of a WG formulation were made at 0.10 kg ai/ha with re-treatment intervals of 6–12 days. Fourteen additional independent trials on field tomatoes were conducted in the US between 2010 and 2011 where two foliar spray applications of a SG formulation were made at 0.15 kg ai/ha with 6–8 day retreatment intervals. Three independent trials were conducted on greenhouse tomatoes in Canada and the US between 2010 and 2011 where two

foliar sprays of a SG formulation were made at 0.15 kg ai/ha with 6–7 day retreatment intervals, In all trials, tomatoes were harvested 0 DALT.

The analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 382 days (19 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 56.

Table 56 Residues of Flonicamid in Field Tomatoes Following Foliar Spray with Flonicamid 50 WG and Beleaf 50SG and in Greenhouse Tomatoes Following Foliar Spray with Beleaf 50 SG in North American Regions

	Application							Residues (mg/kg)				
Location, year (variety)	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days	Flonicamid	TFNA-AM	TFNA	TFNG	Ref
Field tomatoes												
US GAP	WG/SG	0.10 0.15	0.10 0.15	100 100	3 2	7	0					
North Rose NY, 2001 (Floradade)	WG	0.10	0.04	234	3	7	0	0.022 (0.024, 0.019)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	IB-2001-MDG-006-00-1
							1	0.013 (0.035, 0.027)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
							3	0.033 (0.034, 0.032)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.013 (0.011, 0.014)	
							7	0.021 (0.023, 0.018)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Tifton GA, 2001 (5037)	WG	0.10	0.05	187	3	7	0	0.069 (0.057, 0.081)	< 0.01 (< 0.01, < 0.01)	0.014 (0.013, 0.015)	0.014 (0.010, 0.018)	
Hobe Sound FL, 2001 (Florida 47)	WG	0.10	0.02	496–514	3	7	0	0.048 (0.047, 0.045)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01 < 0.01)	0.011 (0.011, 0.010)	
Winter Garden FL, 2001 (Better Boy)	WG	0.10	0.04	271	3	6–7	0	0.093 (0.08, 0.105)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Northwood ND, 2001 (Sheyenne)	WG	0.10	0.10	187	3	7–12	0	0.056 (0.053, 0.058)	< 0.01 (< 0.01, < 0.01)	0.010 (< 0.01, 0.010)	0.013 (0.012, 0.014)	
Vacaville CA, 2001 (3155)	WG	0.10	0.04	234	3	7	0	0.077 (0.088, 0.066)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Davis CA [a], 2001 (Brigade)	WG	0.10	0.04	234	3	6–8	0	0.082 (0.079, 0.085)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Davis CA [a], 2001 (Brigade)	WG	0.10	0.04	224–243	3	6–8	0	0.086 (0.086, 0.086)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Chowchilla CA,	WG	0.10	0.04	281	3	7	0	0.143	< 0.01	0.013	< 0.01	

	Application							Residues (mg/kg)				
Location, year (variety)	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days	Flonicamid	TFNA-AM	TFNA	TFNG	Ref
2001 (US 99)								(0.154, 0.131)	(< 0.01, < 0.01)	(0.012, 0.013)	(< 0.01, < 0.01)	
Mader[a] CA, 2001 (Celebrity)	WG	0.10	0.04	271–281	3	7	0	0.217 (0.196, 0.238)	< 0.01 (< 0.01, < 0.01)	0.011 (< 0.01, 0.011)	< 0.01 (< 0.01, < 0.01)	
Fresno CA[b], 2001 (Super Roma)	WG	0.10	0.02	187–196	3	7	0	0.088 (0.091, 0.084)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Fresno CA[b], 2001 (Shady Lady)	WG	0.10	0.02	701–711	3	6–8	0	0.232 (0.272, 0.191)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.011 (0.012, 0.010)	
Jennings FL, 2003 (Florida 47)	WG	0.10	0.10	94–95	3	7	0	0.15 (0.15, 0.14)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	P-3695
							1	0.09 (0.08, 0.09)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
							3	0.06 (0.06, 0.05)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
							7	0.03 (0.03, 0.03)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.02 (0.02, 0.02)	
	SG w/o surfactant						0	0.13 (0.12, 0.014)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
							1	0.09 (0.11, 0.07)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
							3	0.06 (0.08, 0.04)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
							7	0.04 (0.04, 0.04)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.02 (0.02, 0.02)	
	SG with surfactant						0	0.12 (0.10, 0.13)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
							1	0.09 (0.08, 0.09)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
							3	0.08 (0.09, 0.06)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
							7	0.05 (0.04, 0.05)	< 0.01 (< 0.01,	< 0.01 (< 0.01,	0.03 (0.03, 0.03)	

	Application							Residues (mg/kg)				
Location, year (variety)	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days	Flonicamid	TFNA-AM	TFNA	TFNG	Ref
									< 0.01)	< 0.01)		
Maricopa AZ, 2010 (Round Red)	SG	0.16	0.05	290–299	2	7	0	0.063 (0.037, 0.088)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.01 (0.01, 0.01)	IR-4 PR No. 08556
Davis CA [c], 2010 (Sun 6366)	SG	0.15	0.05	299	2	8	0	0.118 (0.120, 0.115)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Davis CA [c], 2010 (Shady Lady)	SG	0.15	0.05	299	2	7	0	0.083 (0.078, 0.087)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Parlier CA [d], 2010 (H3155)	SG	0.16	0.04–0.08	196–206	2	7	0	0.066 (0.065, 0.067)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.011 (0.012, 0.010)	
Parlier CA [d], 2010 (Cherry Grande)	SG	0.15–0.16	0.04	383–393	2	7	0	0.103 (0.103, 0.103)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.017 (0.017, 0.017)	
Riverside [e] CA, 2010 (Sun 6788)	SG	0.15–0.16	0.04	374–383	2	7	0	0.191 (0.187, 0.194)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.014 (0.014, 0.014)	
Riverside [e] CA, 2010 (Celebrity)	SG	0.15–0.16	0.04	468–477	2	7	0	0.056 (0.057, 0.055)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Holtville CA, 2010 (Shady Lady)	SG	0.15	0.05	299–309	2	6	0	0.117 (0.116, 0.118)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.014 (0.014, 0.013)	
Holtville CA, 2011 (Hypeel 4S)	SG	0.15	0.04	337–346	2	8	0	0.110 (0.110, 0.109)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Citra FL, 2010 (BHN 602)	SG	0.16	0.04	383	2	8	0	0.048 (0.049, 0.047)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Tifton GA, 2010 (Sun Gold F1)	SG	0.15	0.04	393–402	2	6	0	0.102 (0.100, 0.103)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.019 (0.019, 0.019)	
Salisbury MD, 2010 (Sunbrite)	SG	0.15	0.05	309	2	6	0	0.131 (0.131, 0.130)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Clonton NC, 2010 (Supersweet 100)	SG	0.15	0.04	412	2	7	0	0.147 (0.148, 0.145)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.050 (0.050, 0.050)	
Las Cruces [f] NM, 2010 (Roma)	SG	0.15	0.03	468–486	2	6	0	0.078 (0.077, 0.078)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01 < 0.01)	
Las Cruces [f] NM, 2010 (Celebrity)	SG	0.15	0.03	187	2	6	0	0.074 (0.073, 0.074)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Freeville NY, 2010	SG	0.15–	0.03	552–561	2	7	0	0.050	< 0.01	< 0.01	< 0.01	

	Application							Residues (mg/kg)				
Location, year (variety)	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days	Flonicamid	TFNA-AM	TFNA	TFNG	Ref
(Marianna)		0.16						(0.049, 0.050)	(< 0.01, < 0.01)	(< 0.01, < 0.01)	(< 0.01, < 0.01)	
Fremont OH, 2010 (Mountain Pride)	SG	0.15	0.03	421–440	2	7	0	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Arlington OH, 2010 (Better Boy)	SG	0.16	0.08	187	2	7	0	0.070 (0.070, 0.071)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Greenhouse Tomatoes												
GAP	SG	0.10–0.15	0.10–0.15	100	2	7	0					
Parlier CA, 2010 (Trust)	SG	0.15	0.03	458–477	2	7	0	0.058 (0.060, 0.056)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Citra FL, 2011 (BHN 268)	SG	0.15	0.05	281	2	7	0	0.037 (0.037, 0.036)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.025 (0.025, 0.025)	
Harrow ON, CA, 2010 (Macarena)	SG	0.15	0.02	1000–1007	2	7	0	0.049 (0.053, 0.044)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.014 (0.013, 0.014)	
							3	0.050 (0.050, 0.050)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.023 (0.023, 0.022)	
							7	0.040 (0.036, 0.044)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.043 (0.043, 0.043)	
							10	0.041 (0.039, 0.043)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.069 (0.066, 0.071)	

[a] The last applications made at each site were on the same day and the varieties were the same, rendering the trials dependent

[b] The last applications made at each site were 19 days apart, therefore, trials were considered independent

[c] The last applications made at each site were 5 days apart, and varieties were not sufficiently different to render the trials independent

[d] The last applications made at each site were 8 days apart, and the tomato variety H3155 could not be identified, therefore, trials were considered dependent

[e] The last applications made at each site were 9 days apart, and varieties were not sufficiently different to render the trials independent

[f] The last applications were made on the same day and varieties were not sufficiently different to render the trials independent.

Bell peppers

Six independent trials were conducted on field bell peppers in the US in 2001 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–7 days. Bell peppers were harvested 0 DALT.

The analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 382 days (19 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 59.

Table 57 Residues of Flonicamid in Bell Peppers Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.10	0.10	100	3	7	0					
		0.15	0.15	100	2							
Rose Hill NC, 2001 (Jupiter)	WG	0.10	0.05	187	3	7	0	0.058 (0.056, 0.059)	< 0.01 (< 0.01, < 0.01)	0.070 (0.071, 0.068)	0.030 (0.029, 0.030)	IB-2001-MDG-006-00-01
Hobe Sound FL, 2001 (Wizard)	WG	0.10	0.02	571–589	3	6–7	0	0.057 (0.052, 0.062)	< 0.01 (< 0.01, < 0.01)	0.068 (0.064, 0.072)	0.031 (0.029, 0.032)	
Arkansaw WI, 2001 (Better Bell IMP.)	WG	0.10	0.05	187	3	6–7	0	0.056 (0.061, 0.051)	< 0.01 (< 0.01, < 0.01)	0.070 (0.077, 0.062)	0.031 (0.035, 0.027)	
East Bernard TX, 2001 (Capistrano)	WG	0.10	0.08–0.09	112–131	3	7	0	0.055 (0.056, 0.053)	< 0.01 (< 0.01, < 0.01)	0.034 (0.032, 0.035)	0.049 (0.043, 0.055)	
							1	0.113 (0.118, 0.108)	< 0.01 (< 0.01, < 0.01)	0.039 (0.040, 0.037)	0.079 (0.083, 0.074)	
							3	0.099 (0.105, 0.093)	< 0.01 (< 0.01, < 0.01)	0.047 (0.050, 0.044)	0.115 (0.122, 0.107)	
							7	0.051 (0.048, 0.054)	< 0.01 (< 0.01, < 0.01)	0.060 (0.054, 0.065)	0.144 (0.135, 0.153)	
Suisun CA, 2001 (variety not available)	WG	0.10	0.04	234	3	7	0	0.104 (0.107, 0.101)	< 0.01 (< 0.01, < 0.01)	0.045 (0.049, 0.041)	0.038 (0.039, 0.037)	
Fresno CA, 2001 (Jupiter)	WG	0.10	0.015	683–701	3	7	0	0.107 (0.105, 0.108)	< 0.01 (< 0.01, < 0.01)	0.037 (0.036, 0.038)	0.038 (0.037, 0.039)	

Non-bell Peppers

Two independent trials were conducted on field non-bell peppers in the US in 2001 where three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days. Non-bell peppers were harvested 0 DALT.

The analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 382 days (19 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 58.

Table 58 Residues of Flonicamid in Non-bell Peppers Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/ SG	0.10	0.10	100	3	7	0					
		0.15	0.15	100	2							
East Bernard TX, 2001 (Big Jim)	WG	0.10	0.08	122	3	7	0	0.219 (0.215, 0.223)	< 0.01 (< 0.01, < 0.01)	0.028 (0.029, 0.027)	0.041 (0.041, 0.040)	IB-2001-MDG-006-00-01
Suisun CA [a], 2001 (Anaheim)	WG	0.10	0.04	234	3	7–9	0	0.210 (0.204, 0.215)	< 0.01 (< 0.01, < 0.01)	0.030 (0.030, 0.030)	0.040 (0.039, 0.040)	
Suisun CA [a], 2001 (Anaheim)	WG	0.10	0.04	711–720	3	7	0	0.205 (0.208, 0.202)	< 0.01 (< 0.01, < 0.01)	0.031 (0.030, 0.031)	0.038 (0.036, 0.039)	

[a] The last applications were made on the same day and the varieties were the same, rendering the trials dependent.

Leafy vegetables (including Brassica leafy vegetables)

Head lettuce

Six independent trials were conducted on head lettuce in the US in 2002 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–8 days. Head lettuce was harvested 0 DALT.

Method P-3575, a modified version of analytical method P-3561, was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 147 days (ca. 5 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 59.

Table 59 Residues of Flonicamid in Head Lettuce Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Matrix	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/ SG	0.07–0.10	0.07–0.30	30–100	3	7	0						
Germansville PA, 2002 (Sun Devil)	WG	0.10	0.11	94	3	6–7	0	w/wrapper leaves	0.493 (0.392, 0.593)	< 0.01 (< 0.01, < 0.01)	0.021 (0.024, 0.018)	0.026 (0.026, 0.025)	Buser, J.W. and Chen, A.W., 2003
								w/out wrapper leaves	0.027 (0.028, 0.025)	< 0.01 (< 0.01, < 0.01)	0.012 (0.012, 0.011)	< 0.01 (< 0.01, < 0.01)	
Belle Glade FL, 2002 (Iceberg 35x110)	WG	0.10	0.10	92–98	3	7	0	w/wrapper leaves	0.617 (0.649, 0.584)	0.012 (0.013, < 0.01)	0.022 (0.020, 0.023)	0.025 (0.027, 0.023)	
								w/out wrapper leaves	0.024 (0.029, 0.019)	< 0.01 (< 0.01, < 0.01)	0.012 (0.013, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Lagurta AZ, 2002 (Desert Spring)	WG	0.10	0.11	94–96	3	7	0	w/wrapper leaves	0.518 (0.565, 0.471)	< 0.01 (< 0.01, < 0.01)	0.018 (0.021, 0.015)	0.023 (0.023, 0.022)	
								w/out wrapper	0.027 (0.026,	< 0.01 (< 0.01,	0.012 (0.012,	< 0.01 (< 0.01,	

Location, year (variety)	Application						DALT, days	Matrix	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
								leaves	0.028)	< 0.01)	0.011)	< 0.01)	
Visalia CA, 2002 (Great Lakes 659:)	WG	0.10	0.11	94	3	7–8	0	w/wrapper leaves	0.584 (0.723, 0.445)	< 0.01 (< 0.01, < 0.01)	0.023 (0.027, 0.018)	0.028 (0.031, 0.025)	
							0	w/out wrapper leaves	0.027 (0.028, 0.026)	< 0.01 (< 0.01, < 0.01)	0.014 (0.015, 0.013)	< 0.01 (< 0.01, < 0.01)	
							1	w/wrapper leaves	0.013 (0.038, 0.023)	< 0.01 (< 0.01, < 0.01)	0.028 (0.032, 0.024)	0.010 (0.010, < 0.01)	
							3		0.013 (0.012, 0.014)	< 0.01 (< 0.01, < 0.01)	0.014 (0.012, 0.015)	< 0.01 (< 0.01, < 0.01)	
							7		< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.022 (0.021, 0.023)	< 0.01 (< 0.01, < 0.01)	
Bard CA, 2002 (Green Lightning)	WG	0.10	0.11	93–95	3	7	0	w/wrapper leaves	0.431 (0.509, 0.353)	< 0.01 (< 0.01, < 0.01)	0.022 (0.026, 0.018)	0.026 (0.030, 0.022)	
								w/out wrapper leaves	0.033 (0.030, 0.035)	< 0.01 (< 0.01, < 0.01)	0.013 (0.012, 0.014)	< 0.01 (< 0.01, < 0.01)	
Greenfield CA, 2002 (Big Ben)	WG	0.10	0.11	93–97	3	7	0	w/wrapper leaves	0.394 (0.386, 0.402)	< 0.01 (< 0.01, < 0.01)	0.027 (0.034, 0.020)	0.032 (0.042, 0.021)	
								w/out wrapper leaves	0.028 (0.028, 0.027)	< 0.01 (< 0.01, < 0.01)	0.014 (0.013, 0.014)	< 0.01 (< 0.01, < 0.01)	

Leaf lettuce

Six independent trials were conducted on leaf lettuce in the US in 2002 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–7 days. Leaf lettuce was harvested 0 DALT. Side-by-side trials were also conducted in 2003 on Cos lettuce to compare the WG formulation to the SG formulation (with and without surfactant). The same use pattern was applied as that of the six trials.

A modified version of analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 172 days (ca. 6 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 60.

Table 60 Residues of Flonicamid in Leaf Lettuce Following Foliar Spray with Flonicamid 50 WG or Beleaf 50 SG in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.07–0.10	0.07–0.30	30–100	3	7	0					
Germansville PA, 2002 (New Fire)	WG	0.10	0.11	94	3	6	0	2.525 (2.741, 2.309)	0.015 (0.017, 0.013)	0.013 (0.014, 0.011)	0.036 (0.038, 0.034)	P-3575
Belle Glade FL, 2002 (Green Leaf Two Star)	WG	0.10	0.10	92–101	3	7	0	3.113 (3.211, 3.014)	0.017 (0.017, 0.016)	0.014 (0.014, 0.014)	0.042 (0.041, 0.042)	
Maricopa	WG	0.10	0.11	94	3	7	0	3.056	0.016	0.014	0.038	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
AZ, 2001 (Ventana)								(3.051, 3.061)	(0.015, 0.017)	(0.014, 0.014)	(0.037, 0.039)	
Visalia CA, 2002 (Salad Bowl)	WG	0.10	0.11	94	3	7	0	1.936 (1.738, 2.134)	0.023 (0.019, 0.026)	0.028 (0.024, 0.032)	0.100 (0.086, 0.113)	
							1	1.821 (1.764, 1.877)	0.029 (0.028, 0.030)	0.068 (0.087, 0.049)	0.115 (0.134, 0.095)	
							3	1.211 (1.058, 1.363)	0.028 (0.023, 0.033)	0.042 (0.039, 0.045)	0.065 (0.078, 0.051)	
							7	0.374 (0.348, 0.399)	0.013 (0.013, 0.013)	0.047 (0.053, 0.040)	0.061 (0.067, 0.054)	
Bard CA, 2001 (Marin)	WG	0.10	0.11	92–95	3	7	0	2.182 (2.713, 1.650)	0.017 (0.017, 0.016)	0.017 (0.021, 0.012)	0.039 (0.047, 0.031)	
Greenfield CA, 2001 (Green Towers)	WG	0.10	0.11	93–96	3	7	0	2.668 (2.257, 3.078)	0.018 (0.018, 0.018)	0.014 (0.016, 0.012)	0.040 (0.040, 0.040)	
Side-by-side trials												
Jennings FL, 2003 (Romain TA-11 Guzman)	WG	0.10	0.10	97	3	7	0	2.59 (2.56, 2.61)	0.04 (0.04, 0.03)	0.05 (0.05, 0.05)	0.06 (0.06, 0.06)	P-3695
							1	2.55 (2.50, 2.59)	0.06 (0.05, 0.06)	0.05 (0.05, 0.05)	0.08 (0.07, 0.08)	
							3	2.22 (1.90, 2.53)	0.04 (0.04, 0.04)	0.04 (0.03, 0.04)	0.05 (0.05, 0.05)	
							7	0.70 (0.71, 0.69)	0.08 (0.07, 0.08)	0.06 (0.06, 0.06)	0.14 (0.13, 0.014)	
Jennings FL, 2003 (Romain TA-11 Guzman)	SG	0.10	0.10	97	3	7	0	2.32 (2.41, 2.22)	0.02 (0.02, 0.02)	0.04 (0.04, 0.04)	0.04 (0.04, 0.04)	
							1	1.94 (1.92, 1.95)	0.10 (0.10, 0.10)	0.05 (0.05, 0.05)	0.13 (0.13, 0.12)	
							3	2.07 (1.84, 2.29)	0.04 (0.04, 0.04)	0.04 (0.03, 0.05)	0.07 (0.06, 0.08)	
							7	0.38 (0.35, 0.41)	0.04 (0.03, 0.04)	0.04 (0.04, 0.04)	0.06 (0.05, 0.07)	
Jennings FL, 2003 (Romain TA-11 Guzman)	SG (with surfactant)	0.10	0.10	97	3	7	0	2.71 (2.80, 2.61)	0.02 (0.02, 0.02)	0.05 (0.05, 0.04)	0.05 (0.05, 0.05)	
							1	1.82 (1.79, 1.84)	0.06 (0.06, 0.06)	0.04 (0.04, 0.04)	0.08 (0.09, 0.09)	
							3	1.55 (1.41, 1.68)	0.05 (0.05, 0.05)	0.04 (0.03, 0.04)	0.08 (0.08, 0.08)	
							7	0.50 (0.47, 0.53)	0.05 (0.04, 0.05)	0.05 (0.05, 0.05)	0.09 (0.09, 0.09)	

Spinach

Six independent trials were conducted on spinach in the US in 2001 and 2002 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–9 days. Plants were harvested 0 DALT.

Method P-3575, a modified version of analytical method P-3561M, was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 131 days (ca 4 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 61.

Table 61 Residues of Flonicamid in Spinach Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.07–0.10	0.07–0.30	30–100	3	7	0					
Baptistown NJ, 2002 (Tyee)	WG	0.10	0.11	94	3	6–9	0	6.967 (7.196, 6.737)	0.139 (0.134, 0.144)	0.402 (0.401, 0.402)	0.251 (0.247, 0.254)	P-3575
							1	3.062 (3.030, 3.094)	0.051 (0.053, 0.048)	0.218 (0.225, 0.210)	0.015 (0.118, 0.112)	
							3	2.049 (2.116, 1.981)	0.088 (0.089, 0.086)	0.314 (0.323, 0.304)	0.154 (0.159, 0.148)	
							7	0.580 (0.645, 0.514)	0.022 (0.028, 0.015)	0.181 (0.204, 0.158)	0.081 (0.092, 0.070)	
Suffolk VA, 2002 (Tyee)	WG	0.10	0.10	95–100	3	7	0	6.586 (6.073, 7.099)	0.150 (0.143, 0.156)	0.357 (0.353, 0.361)	0.262 (0.248, 0.275)	
Raymondville TX, 2002 (Skookum)	WG	0.10	0.11	94	3	7	0	4.820 (4.160, 5.480)	0.128 (0.116, 0.139)	0.296 (0.277, 0.315)	0.221 (0.204, 0.238)	
Wellington CO, 2002 (Unipack)	WG	0.10	0.11	94	3	7	0	4.855 (5.022, 4.687)	0.052 (0.054, 0.050)	0.132 (0.127, 0.136)	0.167 (0.163, 0.170)	
Yuma AZ, 2001 (RSP 6200)	WG	0.10	0.11	94	3	7	0	5.727 (6.000, 5.454)	0.149 (0.138, 0.160)	0.343 (0.332, 0.354)	0.251 (0.243, 0.259)	
San Ardo CA, 2001 (Bolero)	WG	0.10	0.11	94	3	7	0	5.713 (5.461, 5.965)	0.149 (0.149, 0.149)	0.332 (0.314, 0.350)	0.255 (0.280, 0.230)	

Radish leaves

Five independent trials were conducted on radish leaves in the US in 2003 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–9 days. Leaves were harvested 2 DALT.

The analytical method P-3561M was used to analyse all samples of radish roots and radish tops. The LOQ for radish leaves was determined to be 0.05 mg/kg/analyte.

The maximum period of sample storage at –20 °C was 517 days (ca. 17 months) for radish leaves. Concurrent storage stability data show that the residues are stable for up to 464 days (ca. 15 months). The results are summarized in Table 62.

Table 62 Residues of Flonicamid in Radish Leaves Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Matrix	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
GAP	WG	0.07–0.10	0.07–0.10	100	3	7	3						
Salinas CA, 2003 (Altaglobe)	WG	0.10	0.02	533–542	3	6–7	2	Tops	3.1 (3.2, 2.9)	0.068 (0.070, 0.066)	0.051 (0.05, 0.051)	0.20 (0.20, 0.20)	08753
Citra FL [a], 2003 (Cabernet F1)	WG	0.10	0.04	281	3	6–7	2	Tops	8.5 (8.8, 8.2)	0.47 (0.46, 0.48)	0.16 (0.14, 0.18)	0.70 (0.71, 0.68)	
Citra FL [a], 2003 (Cabernet F1)	WG	0.10	0.04	281–290	3	7–8	2	Tops	5.7 (6.2, 5.2)	0.30 (0.35, 0.25)	0.17 (0.22, 0.12)	0.33 (0.36, 0.29)	
Bridgeton NJ, 2003 (Rebel)	WG	0.10	0.04	243–262	3	8–9	2	Tops	5.4 (5.2, 5.6)	0.098 (0.096, 0.10)	< 0.05 (< 0.05, < 0.05)	0.12 (0.12, 0.12)	
Willard OH, 2003 (Cabernet)	WG	0.10	0.02–0.03	402–430	3	6–8	4	Tops	0.21 (0.23, 0.18)	< 0.05 (< 0.05, < 0.05)	< 0.05 (< 0.05, < 0.05)	0.069 (0.074, 0.063)	

[a] The last applications at each trial site were made 21 days apart, rendering the trials independent.

Mustard Greens

Eight trials were conducted on mustard greens in the US in 2003 and 2004 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–7 days. Mustard green leaves were harvested 0 DALT.

A modified version of analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 214 days (7 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 63.

Table 63 Residues of Flonicamid in Mustard Greens Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.07–0.10	0.07–0.30	100	3	7	0					
Goochland VA, 2003 (Southern Giant)	WG	0.10	0.10	101–107	3	6–8	0	6.873 (6.945, 6.801)	0.047 (0.047, 0.047)	0.411 (0.411, 0.411)	0.907 (0.911, 0.902)	
Senatobia MS, 2003 (Florida Broadleaf)	WG	0.10	0.10	93–94	3	7	0	8.307 (8.097, 8.517)	0.071 (0.064, 0.077)	0.136 (0.131, 0.141)	1.341 (1.304, 1.378)	P-3679
Ellendale MN, 2003 (Southern	WG	0.10	0.11	89–93	3	7	0	2.037 (2.147, 1.926)	< 0.010 (< 0.010, < 0.010)	0.044 (0.051, 0.037)	0.163 (0.182, 0.144)	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Giant Curled)												
Eakly OK, 2003 (Florida Broadleaf)	WG	0.10	0.10	94–95	3	6–7	0	3.965 (3.669, 4.260)	0.046 (0.043, 0.049)	0.184 (0.160, 0.207)	0.401 (0.361, 0.440)	
Visalia CA, 2003 (Florida Broadleaf)	WG	0.10	0.11	91–92	3	7	0	2.209 (1.813, 2.605)	0.031 (0.026, 0.035)	0.070 (0.060, 0.080)	0.418 (0.359, 0.477)	
							1	1.643 (1.598, 1.688)	0.033 (0.030, 0.035)	0.052 (0.049, 0.055)	0.340 (0.307, 0.373)	
							3	1.136 (0.989, 1.283)	0.040 (0.036, 0.044)	0.057 (0.055, 0.059)	0.417 (0.395, 0.438)	
							7	0.369 (0.388, 0.350)	0.018 (0.027, < 0.010)	0.082 (0.078, 0.086)	0.412 (0.425, 0.398)	
Chula GA, 2004 (Broadleaf)	WG	0.10	0.10	96–102	3	7	0	4.401 (4.468, 4.334)	< 0.010 (< 0.010, < 0.010)	0.041 (0.77, < 0.01)	0.448 (0.460, 0.435)	P-3764
Jennings FL, 2004 (Curly Leaf)	WG	0.10	0.10	94–102	3	6–7	0	4.778 (5.123, 4.433)	< 0.010 (< 0.010, < 0.010)	0.069 (0.066, 0.072)	0.416 (0.418, 0.413)	
Visalia CA, 2004 (Broadleaf)	WG	0.10	0.10	95	3	7	0	4.909 (5.042, 4.775)	< 0.010 (< 0.010, < 0.010)	0.084 (0.072, 0.096)	0.482 (0.496, 0.467)	

ND = Not detected

Root and tuber vegetables

Potato tubers

Sixteen independent trials were conducted on potatoes in the US in 2001 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–8 days. Potato tubers were harvested 0 DALT.

In Australia, four independent trials were conducted in 2010 and 2012 where two foliar spray applications of a WG formulation were made at 0.08 kg ai/ha or 0.16 kg ai/ha with 7–9 day re-treatment intervals. Potato tubers were harvested 14 DALT.

The analytical method P-3561M was used to analyse all samples collected from the US trials while method AATM-R-165 was used for the Australian trials. For both methods, the LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 315 days (ca 11 months). Storage stability data on high starch content commodities show that the residues are stable for at least 23 months. The results are summarized in Tables 64.

Table 64 Residues of Flonicamid in Potato Tubers Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions and with UPI-220 500 WG Formulation in Australia

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/ SG	0.07–0.10	0.07–0.30	30–100	3	7	7					
North Rose NY, 2001 (NY-79)	WG	0.12	0.044	234	3	7	0	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.014 (0.012, 0.015)	< 0.01 (< 0.01, < 0.01)	IB-2001-MDG-002-00-01
							1	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.017 (0.016, 0.017)	< 0.01 (< 0.01, < 0.01)	
							3	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.012 (0.012, 0.012)	< 0.01 (< 0.01, < 0.01)	
							7	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.033 (0.034, 0.032)	0.059 (0.060, 0.058)	
							14	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.022 (0.019, 0.025)	< 0.01 (< 0.01, < 0.01)	
Germansville PA, 2001 (Andover)	WG	0.10	0.04	281–290	3	7	7	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.035 (0.031, 0.039)	0.068 (0.055, 0.080)	
Suffolk VA, 2001 (Superior)	WG	0.10	0.10–0.11	94–103	3	7	7	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.026 (0.027, 0.025)	< 0.01 (< 0.01, < 0.01)	
Hobe Sound FL, 2001 (Red LaSoda)	WG	0.10	0.03	346–355	3	7	7	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.021 (0.022, 0.019)	0.014 (0.015, 0.013)	
Northwood ND, 2001 (Dark Red Norland)	WG	0.10	0.05	187	3	6–7	7	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.019 (0.020, 0.017)	0.015 (0.016, 0.013)	
Bygland MN, 2001 (Dark Red Norland)	WG	0.10	0.05	187–196	3	7	7	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.021 (0.020, 0.021)	0.014 (0.014, 0.014)	
Arkansaw WI, 2001 (Russet Burbank)	WG	0.10	0.06	187	3	7	7	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.023 (0.024, 0.022)	< 0.01 (< 0.01, < 0.01)	
Theilman MN, 2001 (Russet Norkotah)	WG	0.10	0.05–0.06	187–196	3	7–8	7	0.015 (0.020, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.049 (0.049, 0.049)	0.01 (0.01, < 0.01)	
Centre CO, 2001 (Norkotah)	WG	0.10	0.06	187	3	7	7	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.046 (0.039, 0.053)	0.010 (< 0.01, 0.010)	
Stockton CA, 2001 (Cal White)	WG	0.10	0.04	234	3	7	7	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.028 (0.025, 0.031)	0.016 (0.014, 0.017)	
Ephrata WA, 2001 (Russet Burbank)	WG	0.10–0.11	0.05–0.06	187–196	3	7	7	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.028 (0.028, 0.028)	0.016 (0.015, 0.016)	
Moses Lake WA, 2001 (Russet Burbank)	WG	0.10	0.11	94	3	7	7	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.035 (0.034, 0.036)	0.020 (0.020, 0.020)	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
American Falls ID, 2001 (Russet Burbank)	WG	0.10	0.05	196	3	6–7	7	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.034 (0.033, 0.035)	0.020 (0.020, 0.020)	
Minidoka ID, 2001 (Russet Burbank)	WG	0.10	0.06	159–168	3	7	0	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.013 (0.013, 0.013)	< 0.01 (< 0.01, < 0.01)	
							1	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.019 (0.015, 0.022)	< 0.01 (< 0.01, < 0.01)	
							3	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.013 (0.013, 0.013)	< 0.01 (< 0.01, < 0.01)	
							7	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.023 (0.021, 0.025)	< 0.01 (< 0.01, < 0.01)	
							14	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.019 (0.025, 0.012)	< 0.01 (< 0.01, < 0.01)	
Herminston OR, 2001 (Russet Burbank)	WG	0.10	0.04	281–290	3	7	7	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.037 (0.038, 0.036)	0.016 (0.016, 0.015)	
Jerome ID, 2001 (Russet Burbank)	WG	0.10	0.06	168–178	3	6–8	7	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.047 (0.042, 0.052)	0.023 (0.021, 0.025)	
AUS GAP	WG	0.07–0.10	NS	NS	2	14	14					
Gembrook, Victoria 2010 (Sebago)	WG	0.08	0.02	503–507	2	7	14	< 0.01	< 0.01	< 0.01	< 0.01	UPL-1001 and 1109
Gembrook Victoria, 2010 (Sebago)	WG	0.16	0.03	503–507	2	7	14	< 0.01	< 0.01	0.015	< 0.01	
Morgan South Australia, 2011 (Ruby Loo's)	WG	0.08	0.03	301–307	2	7	14	< 0.01	< 0.01	< 0.01	< 0.01	
Morgan South Australia, 2011 (Ruby Loo's)	WG	0.16	0.05	3 01–307	2	7	14	< 0.01	< 0.01	< 0.01	< 0.01	
Charleston South Carolina, 2011 (Coliban)	WG	0.08	0.02	402–407	2	7	14	< 0.01	< 0.01	< 0.01	< 0.01	
Charleston South Carolina, 2011 (Coliban)	WG	0.16	0.04	402–407	2	7	14	< 0.01	< 0.01	0.013	< 0.01	
Bundaberg Queensland, 2012 (Sebago)	WG	0.08	0.014	562–581	2	7	14	< 0.01	< 0.01	0.017	0.012	
Bundaberg	WG	0.16–0.17	0.03	581–599	2	7	14	< 0.01	< 0.01	0.026	0.016	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Queensland, 2012 (Sebago)												
Boneo Victoria, 2012 (Exton)	WG	0.08	0.01	603–609	2	9	14	< 0.01	< 0.01	< 0.01	< 0.01	
Boneo Victoria, 2012 (Exton)	WG	0.16	0.03	599–618	2	9	14	< 0.01	< 0.01	0.023	< 0.01	

Carrot roots

Eight independent trials were conducted on carrots in the US in 2003 where three foliar spray applications of a WG formulation with re-treatment intervals of 6–8 days. Carrot roots were harvested 6–8 DALT.

The analytical method P-3561M was used to analyse all samples. The LOQ of flonicamid was determined to be 0.02 mg/kg while the LOQ for all metabolites was determined to be 0.05 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 462 days (ca 15 months). Storage stability data on high starch content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 65.

Table 65 Residues of Flonicamid in Carrot Roots Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.07–0.10	0.07–0.30	30–100	3	7	3					
Salinas CA, 2003 (Mokum-Raw)	WG	0.09–0.10	0.020	449–542	3	7–8	7	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	< 0.050 (< 0.050, < 0.050)	0.060 (0.064, 0.056)	08754
Porterville CA, 2003 (Denver's Half Long 126)	WG	0.10	0.04	224–290	3	6–7	7	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	0.100 (0.122, 0.077)	< 0.050 (< 0.050, < 0.050)	
Parlier CA, 2003 (Denver's Half Long 126)	WG	0.10	0.04	234–243	3	7	1	0.022 (0.024, 0.020)	< 0.050 (< 0.050, < 0.050)	0.054 (0.052, 0.056)	0.050 (0.050, < 0.050)	
							3	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	0.071 (0.061, 0.080)	0.052 (< 0.050, 0.054)	
							6	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	0.092 (0.110, 0.074)	0.052 (0.054, < 0.050)	
							13	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	0.106 (0.099, 0.112)	0.070 (0.070, 0.070)	
Holtville CA, 2004 (Caropak)	WG	0.10	0.03	355–374	3	7	6	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	< 0.050 (< 0.050, < 0.050)	< 0.050 (< 0.050, < 0.050)	
Citra FL, 2003 (Triple Play 58	WG	0.10	0.04	281–299	3	7	7	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	0.051 (0.052, < 0.050)	< 0.050 (< 0.050, < 0.050)	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
SMS)												
Willard OH, 2003 (Scarlet Nantes)	WG	0.10	0.02–0.03	374–430	3	7	7	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	0.059 (0.058, 0.060)	< 0.050 (< 0.050, < 0.050)	
Weslaco TX, 2003 (Six Pence)	WG	0.10	0.04–0.05	206–224	3	6–7	1	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	< 0.05 (< 0.050, < 0.050)	0.086 (0.086, 0.086)	
							3	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	0.061 (0.059, 0.063)	0.091 (0.095, 0.086)	
							6	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	0.050 (0.050, < 0.050)	0.163 (0.178, 0.148)	
							13	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	< 0.050 (< 0.050, < 0.050)	0.124 (0.116, 0.132)	
Moxee WA, 2003 (Enterprise)	WG	0.10	0.03	299–327	3	6–7	8	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	0.072 (0.066, 0.077)	< 0.050 (< 0.050, < 0.050)	

Radish roots

Five independent trials were conducted on radish roots in the US in 2003 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–9 days. Radish roots were harvested 2 DALT.

The analytical method P-3561M was used to analyse all samples of radish roots. The LOQ for radish roots was determined to be 0.02 mg/kg/analyte.

The maximum period of sample storage at –20 °C was 434 days (ca 14 months) for radish roots. Concurrent storage stability data show that the residues are stable for up to 464 days (ca. 15 months). The results are summarized in Table 66.

Table 66 Residues of Flonicamid in Radish Roots Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Matrix	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG	0.07–0.10	0.07–0.10	100	3	7	3						
Salinas CA, 2003 (Altaglobe)	WG	0.10	0.02	533–542	3	6–7	2	Roots	0.13 (0.13, 0.13)	< 0.02 (< 0.02, < 0.02)	0.042 (0.044, 0.040)	< 0.02 (< 0.02, < 0.02)	08753
Citra FL, 2003 (Cabernet F1) [a]	WG	0.10	0.04	281	3	6–7	2	Roots	0.21 (0.25, 0.17)	< 0.02 (< 0.02, < 0.02)	0.078 (0.067, 0.088)	0.056 (0.066, 0.046)	
Citra FL, 2003 (Cabernet F1) [a]	WG	0.10	0.04	281–290	3	7–8	2	Roots	0.075 (0.080, 0.070)	< 0.02 (< 0.02, < 0.02)	0.034 (0.045, 0.022)	< 0.02 (< 0.02, < 0.02)	
Bridgeton NJ, 2003 (Rebel)	WG	0.10	0.04	243–262	3	8–9	2	Roots	0.099 (0.078, 0.12)	< 0.02 (< 0.02, < 0.02)	0.030 (0.030, 0.030)	< 0.02 (< 0.02, < 0.02)	
Willard OH, 2003 (Cabernet)	WG	0.10	0.02–0.03	402–430	3	6–8	4	Roots	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	0.022 (0.020, 0.024)	< 0.02 (< 0.02, < 0.02)	

[a] The last applications at each trial site were made 21 days apart, rendering the trials independent.

Celery

Six independent trials were conducted on celery in the US between 2001 and 2002 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 5–8 days. Celery was harvested 0 DALT. Celery stalks were cut at the soil level using hand clippers. Damaged leaves were removed when necessary.

A modified version of analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 198 days (ca. 7 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 67.

Table 67 Residues of Flonicamid in Celery Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.07–0.10	0.07–0.30	30–100	3	7	0					
Belle Glade FL, 2001 (Walts Pride)	WG	0.10	0.10–0.11	93–97	3	7	0	0.354 (0.391, 0.317)	< 0.01 (< 0.01, < 0.01)	0.013 (0.011, 0.015)	0.029 (0.019, 0.039)	P-3575
Laingsburg MI, 2002 (XP-266)	WG	0.10	0.10–0.11	94–103	3	7	0	0.450 (0.440, 0.459)	< 0.01 (< 0.01, < 0.01)	0.017 (0.015, 0.018)	0.037 (0.034, 0.040)	
Yuma AZ, 2001 (CUF 101)	WG	0.10	0.11	94	3	7–8	0	0.429 (0.459, 0.398)	< 0.01 (< 0.01, < 0.01)	< 0.01 (0.014, < 0.01)	0.026 (0.027, 0.024)	
Visalia CA, 2002 (Tall Utah 52-70)	WG	0.10	0.11	94	3	7	0	0.383 (0.435, 0.330)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.021 (0.025, 0.017)	
							1	0.931 (0.919, 0.942)	< 0.01 (< 0.01, < 0.01)	0.010 (< 0.01, 0.010)	0.032 (0.024, 0.039)	
							3	0.920 (0.956, 0.884)	< 0.01 (< 0.01, < 0.01)	0.010 (0.010, < 0.01)	0.034 (0.037, 0.030)	
							7	0.551 (0.578, 0.524)	< 0.01 (< 0.01, < 0.01)	0.011 (0.011, 0.010)	0.060 (0.057, 0.063)	
King City CA, 2002 (G-20)	WG	0.10	0.11	94–98	3	7	0	0.462 (0.457, 0.466)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.023 (0.025, 0.021)	
Camarillo CA, 2001 (Sonora)	WG	0.10–0.11	0.11	94–100	3	5–7	0	0.444 (0.423, 0.465)	< 0.01 (< 0.01, < 0.01)	0.010 (0.010,	0.029 (0.032,	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
										< 0.01)	0.026)	

Cereal grains

Wheat

The Meeting received information on fifteen independent trials on wheat in Northern and Southern EU between 2000 and 2001 with two foliar spray applications of a WG formulation and a re-treatment intervals of 16–28 days. Wheat grain was harvested 21–30 DALT.

The GC-MS analytical method A-22-00-02 was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte for grain.

The maximum period of sample storage at –20 °C was up to 433 days (ca. 15 months). Storage stability data on high starch content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 68.

Table 68 Residues of Flonicamid in Wheat grain Following Foliar Spray with a 50 WG Formulation of Flonicamid in European Regions

Location, year (variety)	Application						PHI, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Slovenia GAP	WG	0.07	Not specified		2	21	28					
Poggio Renatico, Ferrara, Italy, 2001 (Vayolet)	IBE 3894	0.07	0.018	407–417	2	22	28	< 0.01	< 0.01	0.07	0.7	
Emilia-Romagna, Italy, 2001 (Mieti)	IBE 3894	0.07	0.024	300	2	22	30	0.06 (0.10, < 0.01)	0.05 (0.09, < 0.01)	0.05 (0.09, < 0.01)	0.16 (0.29, 0.03)	
Italy, 2001 (Winter Wheat)	IBE 3894	0.07	0.022–0.023	300	2	22	28	< 0.01	< 0.01	0.05	0.43	
Minaya, Albacete, Spain, 2001 (Gazul) [a]	IBE-3894	0.07	0.02	357–363	2	21	27	< 0.01	< 0.01	0.02	0.09	
Minaya, Albacete, Spain, 2001 (Farak) [a]	IBE-3894	0.07	0.02	360–373	2	21	26	< 0.01	< 0.01	< 0.01	0.07	
Douzonville, North of France, 2001 (Soisson)	IBE-3894	0.07	0.035	205	2	21	21	< 0.01	< 0.01	< 0.01	0.13	
							28	< 0.01	< 0.01	< 0.01	0.12	
Thignonville, North of France, 2001 (Isengrains)	IBE-3894	0.07	0.035	198–200	2	21	28	< 0.01	< 0.01	0.01	0.14	
Rabastens, South of France, 2000 (Gascogne) [b]	IBE 3880	0.07	201	0.035	2	22	27	< 0.01	< 0.01	0.04	0.30	
Rabastens, South of France, 2000 (Gascogne) [b]	IBE 3894	0.07	203–208	0.035	2	22	27	< 0.01	< 0.01	0.06	0.53	
							28	< 0.01	< 0.01	0.03	0.55	
Rabastens, South of France, 2001 (Soisson) [b]	IBE-3894	0.07	0.035	208–211	2	16	28	0.02	< 0.01	0.03	0.16	

Location, year (variety)	Application						PHI, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Puycornet, South of France, 2001 (Soisson)	IBE-3894	0.07	0.035	207	2	19	21	< 0.01	< 0.01	< 0.01	0.07	
							28	< 0.01	< 0.01	< 0.01	0.02	
Stanton, Derbyshire, United Kingdom, 2001 (Consort)	IBE-3894	0.07	0.035	200	2	21	28	< 0.01	< 0.01	< 0.01	0.06	
Meckesheim,Germany, 2001 (Altos) [c] 070-i-d-01	IBE 3894	0.07	0.029	241–249	2	28	21	0.07	0.07	0.06	0.74	
							28	< 0.01	< 0.01	0.05	0.56	
Meckesheim, Germany, 2001 (Monopol) [c]	IBE - 3880	0.073–0.075	0.024	310–316	2	21	28	0.04	< 0.01	0.06	1.10	A-22-01-05
Meckesheim,Germany, 2001 (Bandit) [c]	IBE - 3880	0.066–0.074	278–314	0.024	2	22	28	0.02	< 0.01	0.06	0.28	
Audeville, North of France, 2000 (Tremie) [d]	IBE-3880	0.069	197–198	0.035	2	20	28	< 0.01	< 0.01	0.03	0.55	
Audeville, North of France, 2000 (Tremie) [d]	IBE-3894	0.07	200	0.035	2	20	21	0.01	< 0.01	0.06	0.78	
							28	< 0.01	< 0.01	0.01	0.46	
Puiselet-le-Marais, North of France, 2000 (Altria) [e]	IBE-3880	0.07	200–203	0.035	2	20	28	< 0.01	< 0.01	0.02	0.20	
Puiselet-le-Marais, North of France, 2000 (Altria) [e]	IBE-3894	0.07	204–208	0.035	2	20	28	< 0.01	< 0.01	0.05	0.49	
Meauzac, South of France, 2000 (Aztec) [f]	IBE-3880	0.07	200	0.035	2	20	28	< 0.01	< 0.01	0.07	0.46	
Meauzac, South of France, 2000 (Aztec) [f]	IBE-3894	0.07	198–200	0.035	2	20	21	< 0.01	< 0.01	0.06	0.51	
							28	< 0.01	< 0.01	0.06	0.36	
Hilgersmissen, Germany, 2000 (Brigadier) [g]	IBE 3880	0.07	196–206	0.035	2	22	28	< 0.01	< 0.01	0.01	0.15	A-22-01-10_VP00-1-9
Hilgersmissen,Germany, 2000 (Brigadier) [g]	IBE 3880	0.07	198–200	0.035	2	22	21	0.01	< 0.01	0.01	0.13	
							28	0.01	< 0.01	0.02	0.21	

Note: All trials identified with the same letter were considered dependent as they were conducted at the same location, the last applications were made on the same day at both sites and varieties were not determined to be sufficiently different

Barley

Eight independent trials were conducted on wheat in Germany and Denmark between 2011 and 2012 where a single foliar spray application of a WG formulation was made at 0.07 kg ai/ha. Barley grain was harvested 30–39 DALT.

The LC-MS/MS analytical method AGR/MOA/IKI220-1was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte for grain.

The maximum period of sample storage at –20 °C was up to 111 days (ca. 4 months). Storage stability data on high starch content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 69.

Table 69 Residues of Flonicamid in Barley Grain Following Foliar Spray with a 50 WG Formulation of Flonicamid in Denmark and Germany

Location, year (variety)	Application					PHI, days	Residues (mg/kg)			Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.		Flonicamid	TFNA	TFNG	
Middelfart, Fyn, Denmark, 2011 (Tamtam)	IBE 3894	0.069	0.035	198	1	38	< 0.01	0.01	0.13	S11-01987
Harndrup, Fyn, Denmark, 2011 (Quench)	IBE 3894	0.07	0.035	200	1	30	< 0.01	< 0.01	0.04	
Hygindvej, Ejby, Denmark, 2012 (Simba)	IBE 3894	0.067	0.035	192	1	33	0.02	< 0.01	0.07	S12-01930
Poppenhausen, Baden, Wurttemberg, Germany, 2012 (Grace)	IBE 3894	0.073	0.035	210	1	31	< 0.01	< 0.01	0.12	
Billeshavevej, Middelfart, Denmark, 2012 (Tamtam)	IBE 3894	0.073	0.035	210	1	39	< 0.01	0.01	0.13	
		0.21	0.10	200		39	< 0.01	0.04	0.52	
Tornhoj, Bogense, Denmark, 2012 (Quench)	IBE 3894	0.069	0.035	197	1	38	< 0.01	0.01	0.17	
Wiesentheid, Bavaria, Germany, 2012 (Marthe)	IBE 3894	0.071	0.035	203	1	31	0.01	< 0.01	0.08	
Main, Bavaria, Germany, 2012 (Quench)	IBE 3894	0.071	0.035	204	1	31	0.02	0.01	0.12	

Tree Nuts

Almonds

Five independent trials were conducted on almonds in the US between 1996 and 2008 where three foliar spray applications of a SG formulation were made with re-treatment intervals of 6–8 days. Almonds were harvested 39–42 DALT.

A modified version of analytical method P-3822 was used to analyse all almond nutmeat samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 196 days (ca. 7 months). Storage stability data on oil content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 70.

Table 70 Residues of Flonicamid in Almond Nutmeats Following Foliar Spray with Beleaf 50 SG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	SG	0.07–	0.01–	100–	3	7	40					IB-

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
		0.10	0.10	500								2011-JLW-014-01-01
Chico, CA, 2008 (Non-pareil)	SG	0.10	0.01	1029 – 1038	3	7–8	40	<u>≤ 0.01</u> (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.013 (0.014, 0.011)	< 0.01 (< 0.01, < 0.01)	
Orland, CA, 2004 (Non pareil)	SG	0.1	0.01	1169	3	7	20	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.011 (0.011, 0.010)	< 0.01 (< 0.01, < 0.01)	
							30	0.01 (0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.013 (0.014, 0.012)	< 0.01 (< 0.01, < 0.01)	
							40	<u>≤ 0.01</u> (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.022 (0.024, 0.020)	< 0.01 (< 0.01, < 0.01)	
							50	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.014 (0.014, 0.014)	< 0.01 (< 0.01, < 0.01)	
Wasco, CA, 1996 (Fritz)	SG	0.10	0.007	1459 – 1543	3	6–8	39	<u>≤ 0.01</u> (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Coalinga, CA, 2006 (Non-pareil)	SG	0.10	0.006 – 0.007	1534 – 1702	3	7	39	<u>≤ 0.01</u> (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Turlock, CA, 2007 (Butte)	SG	0.10	0.006 – 0.007	1487 – 1721	3	7	42	<u>≤ 0.01</u> (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.036 (0.034, 0.037)	< 0.01 (< 0.01, < 0.01)	

Pecans

Five independent trials were conducted on pecans in the US between 1983 and 2008 where three foliar spray applications of a SG formulation were made with re-treatment intervals of 7–8 days. Pecans were harvested 20–40 DALT.

A modified version of analytical method P-3822 was used to analyse all almond nutmeat and hulls samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 196 days (ca. 7 months). Storage stability data on oil content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 71.

Table 71 Residues of Flonicamid in Pecans Following Foliar Spray with Beleaf 50 SG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg) (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	SG	0.07-0.10	0.01-0.10	100-500	3	7	40					IB-2011-JLW-014-01-
Anton, TX, 1995	SG	0.10	0.01	1010-1038	3	7	40	<u>≤ 0.01</u> (< 0.0	< 0.01 (< 0.0	< 0.01 (< 0.0	< 0.01 (< 0.0	

Location, year (variety)	Application						DALT, days	Residues (mg/kg) (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
(Western Schley)								1, < 0.01)	1, < 0.01)	1, < 0.01)	1, < 0.01)	01
Pearsall, TX, 1983 (Cheyenne)	SG	0.10	0.007	1360-1375	3	7	39	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Opelousas, LA, 2000 (Native)	SG	0.10	0.009	1113-1141	3	7	39	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Bailey, NC, 1989 (Stuart)	SG	0.10	0.007-0.010	1048-1534	3	7	20	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Girard, GA, 1998 (Desirables)	SG	0.10	0.009	1150-1160	3	7-8	39	< 0.01 (< 0.01, < 0.01)	0.011 (0.011, 0.011)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	

Pistachios

Two independent trials were conducted on pistachios in the US in 2014 where three foliar spray applications of a SG formulation were made with re-treatment intervals of 6–7 days. Pistachios were harvested 40 DALT.

Analytical method P-3822 was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 24 days. As pistachio samples were analysed within 30 days of sampling, freezer storage stability information was not required. The results are summarized in Table 72.

Table 72 Residues of Flonicamid in Pistachios Following Foliar Spray with Beleaf 50 SG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg) (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	SG	0.07–0.10	0.01–0.10	100–500	3	7	40					IB-2014-JLW-015-01-01
Madera, CA, 2014 (Kerman)	SG	0.10	0.01	1219–1237	3	7	40	0.042 (0.042, 0.041)	< 0.01 (< 0.01, < 0.01)	0.064 (0.063, 0.065)	0.079 (0.080, 0.078)	
Terra Bella, CA, 2014 (Kerman)	SG	0.10	0.01	941–1393	3	6	40	0.018 (0.018, 0.017)	< 0.01 (< 0.01, < 0.01)	0.042 (0.043, 0.040)	0.069 (0.072, 0.066)	

Rape seed

Eight independent trials were conducted on canola in the US in 2007 where three foliar spray applications of a SG formulation were made with re-treatment intervals of 6–8 days. Canola seeds were harvested 6–8 DALT.

Analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.02 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 755 days (ca. 25 months). Concurrent storage stability data on canola seed showed that the residues of flonicamid and its associated metabolites are stable for 735 days (ca. 24 months). The results are summarized in Table 73.

Table 73 Residues of Flonicamid in Rape Seed Following Foliar Spray with Beleaf 50 SG Formulation in North American Regions

Location, year (variety)	Application							Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days	Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.10	0.1–0.3	30–100	3	6–8	7					
Kimberly, ID, 2007 (Sunrise Spring)	SG	0.10	0.07	140	3	6–7	6	0.083 (0.096, 0.070)	< 0.02 (< 0.02, < 0.02)	0.037 (0.039, 0.034)	0.084 (0.087, 0.081)	9783
Minot, ND, 2007 (5630)	SG	0.10	0.11	94	3	7–8	7	0.333 (0.339, 0.326)	0.033 (0.035, 0.031)	0.086 (0.087 0.086)	0.338 (0.385, 0.291)	
Velva, ND, 2007 (5550)	SG	0.10	0.07–0.08	131–140	3	7	7	0.021 (0.022, < 0.02)	< 0.02 (< 0.02, < 0.02)	0.049 (0.052, 0.045)	0.032 (0.032, 0.031)	
Bridgeton, NJ, 2007 (Sunrise)	SG	0.10	0.08	122	3	7	7	0.024 (0.025, 0.022)	< 0.02 (< 0.02, < 0.02)	0.021 (0.021, 0.020)	0.030 (0.039, 0.020)	
Brookings, SD, 2007 (Crosby)	SG	0.10	0.06	178	3	7–8	7	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	0.029 (0.026, 0.031)	0.042 (0.035, 0.048)	
Aurora, SD, 2007 (Crosby RR)	SG	0.10	0.06	168	3	6–8	6–7	0.092 (0.135, 0.048)	< 0.02 (< 0.02, < 0.02)	0.063 (0.050, 0.077)	0.161 (0.158, 0.164)	
Brookings, SD, 2008 (Hyclass 601)	SG	0.10	0.10	103	3	6	6	0.169 (0.087, 0.251)	0.068 (0.052, 0.084)	0.066 (0.049, 0.082)	0.136 (0.029, 0.243)	
Prosser, WA, 2007 (Raper)	SG	0.10	0.05–0.07	131–187	3	6–7	8	0.022 (0.023, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
Prosser, WA,	SG	0.1	0.08	122	3	6–8	6	0.045	< 0.02	< 0.02	< 0.02	

Location, year (variety)	Application							Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days	Flonicamid	TFNA-AM	TFNA	TFNG	
2008 (Raper)		0						(0.034, 0.056)	(< 0.02, < 0.02)	(< 0.02, < 0.02)	(< 0.02, < 0.02)	

Cotton

Twelve independent trials were conducted on cotton in the US in 2001 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–9 days. Seeds were collected 29–32 DALT, dried and cleaned followed by a stick extraction to remove the gin trash. The lint cotton was saw ginned to remove the majority of the lint from the cottonseed.

In Australia, ten independent trials were conducted on cotton in 2012 where one or two foliar spray applications were made at 0.10 kg ai/ha or 0.20 kg ai/ha at re-treatment intervals of 14–15 days. Cotton was picked from bolls 7–43 DALT and ginned to separate the fuzz (undelinted).

Method P-3567, a modified version of analytical method P-3561M was used to analyse all samples collected from the US trials while method AATM-R-165 was used to analyse all samples from the Australian trials. The LOQ was determined to be 0.02 mg/kg/analyte for P-3567. For method AATM-R-165, the LOQ was 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 470 days (ca. 16 months). Storage stability data on oil content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 74.

Table 74 Residues of Flonicamid in Undelinted Cottonseeds Following Foliar Spray with Flonicamid 50WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.05–0.10	0.02–0.05	30–50	3	7	30					
Elko, SC, 2001 (Delta Pine 451 B/RR)	WG	0.10	0.06	168	3	7	29	0.040 (0.042, 0.038)	< 0.02 (< 0.02, < 0.02)	0.050 (0.054, 0.046)	0.024 (0.028, 0.020)	IB-2001-MDG-004-00-01
West Memphis, AR, 2001 (Suregrow)	WG	0.10	0.07	150	3	7	0	0.104 (0.105, 0.102)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							10	0.029 (0.031, 0.026)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							21	0.028 (0.028, 0.027)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							30	0.042 (0.039, 0.045)	< 0.02 (< 0.02, < 0.02)	0.055 (0.051, 0.059)	0.024 (0.026, 0.021)	
							40	0.025 (0.026, 0.024)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							10	0.029 (0.025, 0.032)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							21	0.027 (0.028, 0.026)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
							30	0.036 (0.035, 0.036)	< 0.02 (< 0.02, < 0.02)	0.066 (0.063, 0.069)	0.026 (0.025, 0.027)	
							40	0.026 (0.022, 0.029)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
Tillar, AR, 2001 (Pay-master)	WG	0.10	0.10	94–103	3	6–9	30	0.031 (0.029, 0.033)	< 0.02 (< 0.02, < 0.02)	0.048 (0.048, 0.048)	0.021 (< 0.02, 0.021)	
Senatobia, MS, 2001 (DPL 451 Bt/RR)	WG	0.10	0.05	187–196	3	7	29	0.034 (0.032, 0.035)	< 0.02 (< 0.02, < 0.02)	0.049 (0.049, 0.048)	0.023 (0.023, 0.023)	
Eakly, OK, 2001 (PM 2280)	WG	0.10	0.05	187	3	6–8	30	0.035 (0.035, 0.035)	< 0.02 (< 0.02, < 0.02)	0.057 (0.057, 0.056)	0.026 (0.025, 0.027)	
Dill City, OK, 2001 (Pay-master 2326)	WG	0.10	0.05–0.06	168–206	3	7	31	0.048 (0.036, 0.059)	< 0.02 (< 0.02, < 0.02)	0.110 (0.113, 0.107)	0.094 (0.080, 0.107)	
Levelland, TX, 2001 (PM 2326 B6/RR)	WG	0.10	0.07	140	3	6–8	29	0.055 (0.050, 0.060)	< 0.02 (< 0.02, < 0.02)	0.117 (0.101, 0.133)	0.105 (0.094, 0.116)	
Uvalde, TX, 2001 (PM 2326 RR)	WG90	0.10	0.05	187–196	3	7	30	0.046 (0.057, 0.034)	< 0.02 (< 0.02, < 0.02)	0.124 (0.105, 0.143)	0.094 (0.079, 0.109)	
Edmonson, TX, 2001 (Pay-master HS 250)	WG	0.10	0.06–0.07	150–187	3	7–8	0	0.120 (0.143, 0.097)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							11	0.028 (0.028, 0.028)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							20	0.028 (0.026, 0.030)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							32	0.043 (0.050, 0.035)	< 0.02 (< 0.02, < 0.02)	0.120 (0.122, 0.118)	0.070 (0.071, 0.068)	
							43	0.025 (0.024, 0.026)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							11	0.033 (0.029, 0.037)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							20	0.038 (0.035, 0.040)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							32	0.035 (0.041, 0.028)	< 0.02 (< 0.02, < 0.02)	0.261 (0.305, 0.217)	0.149 (0.179, 0.118)	
							43	0.030 (0.032, 0.028)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
Stanfield, AZ, 2001 (DP458 B1RR)	WG	0.10	0.06	187	3	7	29	0.041 (0.041, 0.040)	< 0.02 (< 0.02, < 0.02)	0.125 (0.127, 0.123)	0.073 (0.074, 0.071)	
Mariopa, AZ, 2001 (DP451 B1RR)	WG	0.10	0.05	187	3	7	30	0.085 (0.083, 0.087)	< 0.02 (< 0.02, < 0.02)	0.133 (0.146, 0.119)	0.084 (0.087, 0.080)	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Madera, CA, 2001 (Acala Riata RR)	WG	0.10–0.11	0.04	281–290	3	7	29	0.085 (0.084, 0.086)	< 0.02 (< 0.02, < 0.02)	0.121 (0.133, 0.109)	0.108 (0.126, 0.089)	
AUS GAP	WG	0.07	NS	NS	2	NS	7					
Mywybilla, Queensland, (Sicot 71BRF)	WG	0.10	0.120	84	2	14	7	0.035	< 0.01	< 0.01	< 0.01	UPL GLP-10-07
		0.10	0.127	81			27	0.012	< 0.01	0.041	0.01	
		0.20	0.248	82			7	0.064	< 0.01	0.014	0.013	
		0.20	0.253	80			27	0.016	< 0.01	0.07	0.019	
		0.10	0.125	79		15	7	0.01 (< 0.01, 0.01)	0.01 (< 0.01, 0.01)	0.013 (0.015, 0.01)	0.01 (< 0.01, 0.01)	
		0.10	0.118	82		14	15	< 0.01	< 0.01	0.018	< 0.01	
		0.10	0.129	77		14	22	< 0.01	< 0.01	0.048	0.016	
		0.10	0.123	79		14	29	< 0.01	< 0.01	0.068	< 0.01	
		0.10	0.114	88		14	36	< 0.01	< 0.01	0.13	0.022	
		0.10	0.106	93		14	43	< 0.01	< 0.01	0.17	0.021	
		0.20	0.255	78		15	7	0.014	< 0.01	< 0.01	< 0.01	
		0.20	0.260	78		14	29	< 0.01	< 0.01	0.08	0.017	
Boggabilla, New South Wales (Sicot 71BRF)	WG	0.10	0.11	92	2	14	7	0.13	< 0.01	0.01	0.015	
		0.10	0.11	92		15	28	0.018	< 0.01	0.038	0.033	
		0.20	0.218	92		14	7	0.31	0.018	0.021	0.046	
		0.20	0.22	92		15	28	0.045	0.021	0.076	0.091	
Narrabi, New South Wales (Sicot 71BRF)	WG	0.10	0.112	89	2	14	7	0.094	< 0.01	< 0.01	< 0.01	
		0.10	0.11	92		15	13	0.074	< 0.01	< 0.01	< 0.01	
		0.10	0.109	92		14	21	0.016	< 0.01	0.05	< 0.01	
		0.10	0.111	91		13	28	0.024	< 0.01	0.12	0.023	
		0.10	0.114	88		14	35	< 0.01	< 0.01	0.1	0.014	
		0.10	0.105	97		14	41	0.012 (0.012, 0.011)	< 0.01 (< 0.01, < 0.01)	0.43 (0.47, 0.38)	0.069 (0.066, 0.071)	
		0.20	0.228	89		14	7	0.18	< 0.01	< 0.01	< 0.01	
		0.20	0.224	90		13	28	0.041	0.012	0.14	0.04	
Chinchilla, Queensland (Sicot 71BRF)	WG	0.10	0.10	105	2	14	7	0.022	< 0.01	< 0.01	< 0.01	
		0.10	0.10	110		14	28	0.011	< 0.01	< 0.01	0.011	
		0.10	0.10	101		14	49	< 0.01	< 0.01	0.01	0.015	
		0.10	0.10	102		14	63	< 0.01	< 0.01	0.063	0.04	
		0.20	0.19	106		14	7	0.085	< 0.01	< 0.01	< 0.01	
		0.20	0.18	110		14	28	0.025	< 0.01	< 0.01	0.018	
		0.20	0.20	101		14	49	< 0.01	< 0.01	0.021	0.026	
		0.20	0.20	102		14	63	< 0.01	< 0.01	0.09	0.077	
Condamine Plains, Queensland (Sicot71BRF)		0.10	0.09	110	2	14	7	< 0.01	< 0.01	< 0.01	< 0.01	UPL GLP 12 01-1
		0.10	0.10	100		15	20	< 0.01	< 0.01	0.037	< 0.01	
		0.10	0.10	101		14	27	0.012	< 0.01	0.056	0.014	
		0.10	0.10	103	1	NA	27	< 0.01	< 0.01	0.016	< 0.01	
		0.05	0.05	103	2	14	35	< 0.01	< 0.01	0.026	< 0.01	
	WG	0.10	0.10	101		14	41	< 0.01	< 0.01	0.096	0.027	
		0.10	0.09	106		13	49	< 0.01	< 0.01	0.11	0.014	
		0.10	0.09	109		14	55	< 0.01	< 0.01	0.19	0.06	
		0.20	0.18	111		13	7	< 0.01	< 0.01	< 0.01	< 0.01	
		0.20	0.20	102		14	27	0.02	< 0.01	0.056	0.022	
		0.20	0.20	101		14	41	0.014	< 0.01	0.16	0.053	
		0.20	0.18	110		14	55	< 0.01	< 0.01	0.34	0.099	
Narrabri, New South Wales (Sicot 71BRF)	WG	0.10	0.11	90–92	2	14	8	0.34	0.071	0.025	0.048	UPL GLP 12 01-1
		0.10	0.11	89–90	2	14	15	0.067	0.033	0.019	0.054	
		0.10	0.11	91–92	2	14	22	0.11	0.047	0.043	0.12	
		0.10	0.11	89–90	2	14	29	0.13	0.069	0.051	0.16	
		0.10	0.11	91	2	14	36	0.088	0.076	0.075	0.23	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
		0.10	0.11	88–91	2	14	43	0.032	0.029	0.064	0.12	
		0.10	0.11	90	2	14	50	0.025	0.028	0.098	0.16	
		0.10	0.11–0.12	84–86	2	14	57	< 0.01	< 0.01	0.1	0.12	
		0.23	0.22	92–93	2	14	8	0.48	0.072	0.026	0.049	
		0.21	0.22	92	2	14	29	0.11	0.082	0.053	0.17	
		0.20	0.22–0.24	85–92	2	14	43	0.096	0.097	0.13	0.33	
		0.20	0.24	85–86	2	14	57	0.022	0.025	0.12	0.19	
Narromine, New South Wales (Sicot 71BRF)	WG	0.10	0.08–0.09	114–125	2	14	7	0.16 (0.17, 0.14)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
		0.10	0.08–0.09	116–119	2	14	28	0.046	< 0.01	0.14	0.036	
		0.10	0.08	123–124	2	11	42	< 0.01	< 0.01	0.091	0.017	
		0.10	0.08–0.09	117–123	2	15	53	< 0.01	< 0.01	0.11	0.026	
		0.20	0.17–0.18	114–119	2	14	7	0.09 (0.088, 0.092)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
		0.21	0.17	122–124	2	14	28	0.056	0.1	0.13	0.034	
		0.21	0.17	119–123	2	11	42	0.012	< 0.01	0.17	0.037	
		0.21	0.17–0.18	118–123	2	15	53	< 0.01	< 0.01	0.18	0.045	

Mint

Three independent trials were conducted on fresh mint in the US in 2011 where three foliar spray applications of a SG formulation were made with re-treatment intervals of 13–15 days. Mint leaves were harvested 7 DALT.

Analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.02 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 372 days (ca. 12 months). Concurrent storage stability data on mint tops show that the residues are stable for at least 364 days (ca. 12 months). The results are summarized in Table 75.

Table 75 Residues of Flonicamid in Fresh Mint Tops Following Foliar Spray with Beleaf 50 SG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.07–0.10	0.04–0.05	100–200	3	14	7					
Bruneau, ID, 2011 (Black Mitcham Perppermint)	SG	0.10	0.04	271–281	3	14	7	2.36 (2.31, 2.41)	0.377 (0.376, 0.377)	0.105 (0.086, 0.125)	0.104 (0.146, 0.133)	9358
Prosser, WA, 2011 (Peppermint)	SG	0.10	0.04	243–262	3	13–14	7	1.70 (1.67, 1.73)	0.456 (0.451, 0.461)	0.234 (0.214, 0.254)	0.229 (0.222, 0.235)	
Endeavour,	SG	0.10	0.02	552–	3	13–	6	1.92	0.036	0.166	0.208	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
WI, 2011 (Scotch Spearmint)				561		15		(1.90, 1.93)	(0.337, 0.356)	(0.167, 0.164)	(0.211, 0.204)	

Dried hops

Four independent trials were conducted on hops in the US in 2003 and 2015 where three foliar spray applications of a WG or SG formulation were made with re-treatment intervals of 7–8 days. Green hop cones were sampled 9–11 DALT and dried to 8–10% moisture in a forced hot air dryer. Drying temperature was about 120–140 °F (49–64 °C).

The analytical methods P-3561M (2003 trial) or P-3822 (2015 trial) were used to analyse all samples. The LOQ for dried hop cones was determined to be 0.02 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 329 days (ca 11 months). Concurrent storage stability data on hops show that the residues are stable for at least 299 days (10 months). The results are summarized in Table 76.

Table 76 Residues of Flonicamid in Dried Hop Cones Following Foliar Spray with Flonicamid 50 WG and Beleaf 50SG Formulations in Northern America

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.06–0.10	0.01–0.02	500	3	7	10					
Parma ID, 2003 (Nugget)	WG	0.10	0.01	907–917	3	7–8	9	2.82 (2.85, 2.78)	0.717 (0.177, 0.165)	0.307 (0.312, 0.302)	0.104 (0.110, 0.098)	08706
Hubbard OR, 2003 (Nugget)	WG	0.10	0.01	795–945	3	7	9	1.15 (1.10, 1.20)	0.146 (0.139, 0.153)	0.456 (0.470, 0.442)	0.204 (0.204, 0.204)	
Prosser WA, 2003 (Nugget)	WG	0.10	0.01	1272–1347	3	7–8	11	0.563 (0.561, 0.565)	0.038 (0.038, 0.038)	0.335 (0.335, 0.334)	0.162 (0.156, 0.168)	
Ephrata WA, 2015 Cascade)	SG	0.10	0.01	945	3	7	10	9.33 (10.6, 8.06)	0.226 (0.269, 0.184)	0.727 (0.660, 0.794)	0.074 (0.076, 0.072)	IB-2014-JLW-014-01-01

Tea

Two independent trials were conducted on tea in Japan in 2001 where a single foliar spray application of a WG formulation was made. Leaves were harvested 7 DALT. On the day of harvest, leaves were processed according to standard procedure (steaming, cooling, primary drying and rolling, rolling, secondary drying and rolling, final drying and rolling and drying) prior to analysis

The analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.02 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 9 months. Storage stability data on various commodities show that the residues are stable for at least 23 months. The results are summarized in Table 77.

Table 77 Residues of Flonicamid in Tea Following Foliar Spray with Flonicamid 50 WG Formulation in Japan

Location, year (variety)	Application						DALT, days	Residues (mg/kg)			Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA	TFNG	
Japan GAP	DF	0.1	0.01	2000–4000	1	NA	7				
Tsukui, Kanagawa, Japan, 2001 (Yabukita)	DF	0.2	0.01	2000	1	NA	7	20.1 (22.7, 21.8, 18.0, 17.8)	0.31 (0.31, 0.26, 0.35, 0.32)	3.03 (3.32, 3.10, 2.85, 2.82)	Report No 13-79
Uji, Kyoto, Japan, 2001 (Yakibuta)	DF	0.2	0.01	2000	1	NA	7	15.7 (16.9, 16.5, 15.0, 14.5)	0.18 (0.18, 0.16, 0.20, 0.18)	1.82 (1.98, 1.97, 1.67, 1.64)	

Animal feeds

Wheat forage and straw

Fifteen independent supervised trials were conducted in EU on wheat in 2000 and 2001, where two foliar spray applications of a WG formulation were made with re-treatment intervals of 16–21 days. Green forage (green plant, rest of plant) was sampled 0–7 DALT, stems and ears 14–21 DALT and straw 21–30 DALT.

The analytical methods A-22-02 was used to analyse all samples. The LOQ for green forage and straw was 0.02 mg/kg/analyte and 0.01 mg/kg/analyte for stems and ears.

The maximum period of sample storage at –20 °C was up to days (ca. 14 months). Storage stability data on starch content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 78.

Table 78 Residues of Flonicamid in Wheat Forage (green plant, rest of plant), Straw, Ears and Stems Following Foliar Spray with 50 WG Formulations in Northern and Southern EU

Location, year (variety)	Application						PHI, days	Commodity	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
Slovenia GAP	WG	0.07			2	21	28						
Poggio Renatico, Ferrara, Italy, 2001 (Vayolet)	IBE 3894	0.07	0.018	407–417	2	22	28	Straw	< 0.02	< 0.02	< 0.02	0.17	
Emilia-Romagna, Italy, 2001 (Mieti)	IBE 3894	0.07	0.024	300	2	22	30	Straw	0.08	< 0.02	0.02	0.41	
Italy, 2001 (Winter Wheat)	IBE 3894	0.07	0.022–0.023	300	2	22	28	Straw	0.04	< 0.02	0.03	0.36	

Location, year (variety)	Application						PHI, days	Commodity	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
Minaya, Albacete, Spain, 2001 (Gazul)[1]	IBE-3894	0.07	0.02	357–363	2	21	27	Straw	0.11	< 0.02	< 0.02	0.15	
Minaya, Albacete, Spain, 2001 (Farak)[1]	IBE-3894	0.07	0.02	360–373	2	21	26	Straw	0.03	< 0.02	< 0.02	0.14	
Douzonville, North of France, 2001 (Soisson)	IBE-3894	0.07	0.035	205	2	21	0	Green Plant	0.66	< 0.02	< 0.02	0.14	
							7	Green Plant	0.99	< 0.02	< 0.02	0.21	
							14	Ears	0.03	< 0.02	< 0.02	0.03	
							14	Stem	0.06	< 0.02	< 0.02	0.04	
							21	Straw	< 0.02	< 0.02	< 0.02	< 0.02	
							28	Straw	< 0.02	< 0.02	< 0.02	< 0.02	
Thignonville, North of France, 2001 (Isengrains)	IBE-3894	0.07	0.035	198–200	2	21	28	Straw	< 0.02	< 0.02	< 0.02		
Rabastens, South of France, 2000 (Gascogne)[2]	IBE 3880	0.07	201	0.035	2	22	27	Straw	0.03	< 0.02	< 0.02	0.06	
Rabastens, South of France, 2000 (Gascogne)[2]	IBE 3894	0.07	203–208	0.035	2	22	27	Straw	0.02	< 0.02	< 0.02	0.06	
							28	Straw	0.05	< 0.02	< 0.02	0.10	
Rabastens, South of France, 2001 (Soisson)	IBE-3894	0.07	0.035	208–211	2	16	28	Straw	0.02	< 0.02	< 0.02	< 0.02	
Puycornet, South of France, 2001 (Soisson)	IBE-3894	0.07	0.035	207	2	19	0	Green Plant	0.48	< 0.02	< 0.02	0.19	
							7	Green Plant	0.99	< 0.02	< 0.02	0.23	
							14	Ears	0.04	< 0.02	< 0.02	< 0.02	
							14	Stem	0.06	< 0.02	< 0.02	0.05	
							21	Straw	0.07	< 0.02	< 0.02	0.04	
							28	Straw	< 0.02	< 0.02	< 0.02	< 0.02	
Stanton, Derbyshire, United Kingdom, 2001 (Consort)	IBE-3894	0.07	0.035	200	2	21	28	Straw	0.05	< 0.02	< 0.02	0.08	
Meckesheim, Germany, 2001 (Altos)[3]	IBE 3894	0.07	0.029	241–249	2	28	0	Green Plant	0.32	< 0.02	< 0.02	0.11	
							7	Green Plant	0.15	< 0.02	0.02	0.10	
							14	Ears	< 0.02	< 0.02	0.04	0.16	
							14	Stem	< 0.02	< 0.02	< 0.02	0.16	
							21	Straw	< 0.02	< 0.02	< 0.02	0.21	
							28	Straw	< 0.02	< 0.02	< 0.02	0.15	
Meckesheim, Germany, 2001 (Monopol)[3]	IBE - 3880	0.073–0.075	0.024	310–316	2	21	28	Straw	0.23	< 0.02	0.03	0.17	A-22-01-05
Meckesheim, Germany, 2001 (Bandit)[3]	IBE - 3880	0.066–0.074	278–314	0.024	2	22	0	Rest of Plant	0.88	< 0.02	< 0.02	0.16	
							7	Rest of Plant	0.47	< 0.02	< 0.02	0.13	

Location, year (variety)	Application						PHI, days	Commodity	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
							14	Ears	0.21	< 0.02	< 0.02	0.04	
							14	Stem	0.51	< 0.02	< 0.02	0.04	
							28	Straw	0.39	< 0.02	< 0.02	0.07	
Audeville, North of France, 2000 (Tremie)[4]	IBE-3880	0.069	197–198	0.035	2	20	0	Green Plant	0.53	< 0.02	< 0.02	0.07	
							28	Straw	0.05	< 0.02	< 0.02	0.10	
Audeville, North of France, 2000 (Tremie)[4]	IBE-3894	0.07	200	0.035	2	20	0	Green Plant	0.64	< 0.02	< 0.02	0.12	
							7	Green Plant	0.20	< 0.02	0.02	0.37	
							14	Ears	0.13	< 0.02	0.04	0.53	
							14	Stem	0.33	< 0.02	< 0.02	0.65	
							21	Straw	0.19	< 0.02	< 0.02	0.33	
							28	Straw	< 0.02	< 0.02	< 0.02	< 0.02	
Puiselet-le-Marais, North of France, 2000 (Altria)[5]	IBE-3880	0.07	200–203	0.035	2	20	28	Straw	0.04	< 0.02	< 0.02	0.05	
Puiselet-le-Marais, North of France, 2000 (Altria)[5]	IBE-3894	0.07	204–208	0.035	2	20	28	Straw	0.03	< 0.02	< 0.02	0.02	
Meauzac, South of France, 2000 (Aztec)[6]	IBE-3880	0.07	200	0.035	2	20	0	Green Plant	0.55	< 0.02	< 0.02	0.15	
							28	Straw	< 0.02	< 0.02	< 0.02	0.10	
Meauzac, South of France, 2000 (Aztec)[6]	IBE-3894	0.07	198–200	0.035	2	20	0	Green Plant	0.69	< 0.02	< 0.02	0.14	
							7	Green Plant	0.02	< 0.02	< 0.02	0.13	
							14	Ears	0.04	< 0.02	0.02	0.18	
							14	Stem	0.03	< 0.02	< 0.02	0.29	
							21	Straw	< 0.02	< 0.02	< 0.02	0.22	
							28	Straw	< 0.02	< 0.02	< 0.02	0.12	
Hilgersmissen ,Germany, 2000 (Brigadier)[7]	IBE 3880	0.07	196–206	0.035	2	22	0	Rest of Plant	0.83	< 0.02	< 0.02	0.08	A-22-01-10_VP00-1-9
							28	Straw	0.09	< 0.02	< 0.02	0.02	
Hilgersmissen ,Germany, 2000 (Brigadier)[7]	IBE 3880	0.07	198–200	0.035	2	22	0	Rest of Plant	0.67	< 0.02	< 0.02	0.06	
							7	Rest of Plant	0.65	< 0.02	< 0.02	0.06	
							14	Ears	0.79	< 0.02	< 0.02	0.08	
							14	Stem	1.58	0.03	0.05	0.30	
							21	Straw	0.11	< 0.02	< 0.02	0.03	
							28	Straw	0.07	< 0.02	< 0.02	0.02	

Barley ears

Four independent supervised trials were conducted in Germany and Denmark on barley ears in 2012, where a single foliar spray application of a WG formulation was made at 0.07 kg ai/ha and where ears were sampled 0–22 DALT.

The LC-MS/MS analytical method AGR/MOA/IKI220-1was used to analyse all samples. The LOQ for green forage and straw was 0.01 mg/kg/analyte for ears.

The maximum period of sample storage at –20 °C was up to 111 days (ca. 4 months). Storage stability data on high starch content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 79.

Table 79 Residues of Flonicamid in Barley Ears Following Foliar Spray with 50 WG Formulations in Denmark and Germany.

Location, year (variety)	Application					PHI, days	Residues (mg/kg)			
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.		Flonicamid	TFNA [a]	TFNG [a]	
Billeshavevej, Middelfart, Denmark, 2012 (Tamtam)	IBE 3894	0.073	0.035	210	1	0	1.18	< 0.01	0.02	S12-01930
						7	0.02	< 0.01	0.17	
						14	< 0.01	< 0.01	0.09	
						21	< 0.01	< 0.01	0.05	
Tornhoj, Bogense, Denmark, 2012 (Quench)	IBE 3894	0.069	0.035	197	1	0	0.49	< 0.01	0.02	
						6	0.06	0.01	0.18	
						14	0.01	0.01	0.15	
						21	< 0.01	< 0.01	0.09	
Wiesentheid, Bavaria, Germany, 2012 (Marthe)	IBE 3894	0.071	0.035	203	1	0	0.96	< 0.01	< 0.01	
						7	0.12	0.01	0.15	
						13	0.06	< 0.01	0.11	
						20	0.07	0.01	0.12	
Main, Bavaria, Germany, 2012 (Quench)	IBE 3894	0.071	0.035	204	1	0	1.0	< 0.01	0.02	
						8	0.14	0.01	0.13	
						15	0.05	< 0.01	0.11	
						22	0.04	0.01	0.12	

[a] Reported in flonicamid equivalents.

Alfalfa forage, seed and hay

Four independent trials were conducted on alfalfa in the US in 2009 where two foliar spray applications of a SG formulation were made with re-treatment intervals of 7–8 days. Because alfalfa is harvested differently in California compared to the Pacific Northwest, sample collection times varied between these two regions. In the Idaho and Washington trials, seed samples were collected 13–14 DALT in the summer, and forage and hay samples were harvested the following year, 265–293 DALT.

Analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.02 mg/kg/analyte.

The maximum period of sample storage at –20 °C was 432 days (ca. 14 months) for alfalfa seed, 490 days (ca. 16 months) for forage and 496 days (ca. 16 months) for hay. Concurrent storage stability data show that the residues of flonicamid and its metabolites are stable for 490 days in forage, 518 days in hay and 462 days in seed. The results are summarized in Table 80.

Table 80 Residues of Flonicamid in Alfalfa Following Foliar Spray with Beleaf 50 SG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Matrix	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
GAP (West of the US Rockies)	WG/SG	0.10	0.10–0.50	100–200	2	7	14 14 62	Seed Forage Hay					
Holtville, CA, 2009 (CUF 101)	SG	0.10	0.06–0.07	299–318	2	7	5	Forage	5.97 (5.76, 6.18)	0.074 (0.062, 0.085)	0.491 (0.479, 0.503)	2.012 (2.029, 1.996)	9943
					2	7	10		2.99 (2.57, 3.41)	0.046 (0.035, 0.057)	0.368 (0.371, 0.365)	1.725 (1.581, 1.868)	
					2	7	11		0.319 (0.303, 0.335)	< 0.02 (< 0.02, < 0.02)	0.077 (0.071, 0.083)	0.434 (0.417, 0.450)	
					2	7	19		0.256 (0.265,	< 0.02 (< 0.02,	0.032 (0.039,	0.247 (0.286,	

Location, year (variety)	Application						DALT, days	Matrix	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
									0.247)	< 0.02)	0.026)	0.208)	
					2	7	24		0.323 (0.321, 0.324)	< 0.02 (< 0.02, < 0.02)	0.108 (0.130, 0.085)	0.240 (0.258, 0.221)	
					2	7	62		< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	0.02 (< 0.02, 0.020)	
					2	7	62	Hay	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	0.021 (0.022, 0.020)	
					2	7	11	Seed	0.141 (0.131, 0.151)	0.03 (< 0.02, 0.030)	0.155 (0.116, 0.194)	0.011 (0.084, 0.127)	
Holtville, CA, 2009 (CUF 101)	SG	0.10	0.09	234	2	7	14	Forage	1.30 (0.981, 1.620)	0.077 (0.058, 0.096)	0.247 (0.197, 0.297)	1.495 (1.183, 1.806)	
					2	7	65		< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	0.035 (0.039, 0.031)	
					2	7	65	Hay	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	0.062 (0.062, 0.061)	
					2	7	14	Seed	0.106 (0.103, 0.108)	< 0.02 (< 0.02, < 0.02)	0.050 (0.040, 0.060)	0.031 (0.020, 0.041)	
Jerome, ID, 2009 (Rampage)	SG	0.10	0.07	281	2	7	293	Forage	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
					2	7	293	Hay	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
					2	7	13	Seed	0.138 (0.134, 0.142)	< 0.02 (< 0.02, < 0.02)	0.355 (0.357, 0.373)	0.045 (0.038, 0.051)	
Touchet, WA, 2009 (Forage Genetics 43M120)	SG	0.10	0.07	271–281	2	7	265	Forage	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	0.022 (< 0.02, 0.023)	< 0.02 (< 0.02, < 0.02)	
					2	7	282	Hay	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
					2	7	14	Seed	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	

Almond Hulls

Five independent trials were conducted on almonds in the US between 1996 and 2008 where three foliar spray applications of a SG formulation were made with re-treatment intervals of 6–8 days. Almonds were harvested 39–42 DALT.

A modified version of analytical method P-3822 was used to analyse all almond nutmeat samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 196 days (ca. 7 months). Storage stability data on oil content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 81.

Table 81 Residues of Flonicamid in Almond Hulls Following Foliar Spray with Beleaf 50 SG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Commodity[1]	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	SG	0.07–0.10	0.01–0.10	100–500	3	7	40						IB-2011-JLW-014-01-01
Chico, CA, 2008 (Non-pareil)	SG	0.10	0.01	1029–1038	3	7–8	40	Hulls (9.1%)	4.302 (4.411, 4.193)	0.053 (0.053, 0.053)	0.187 (0.199, 0.174)	0.257 (0.248, 0.265)	
							40	Hulls (dry weight)	4.731 (4.874, 4.588)	0.058 (0.058, 0.058)	0.205 (0.219, 0.191)	0.282 (0.273, 0.290)	
Orland, CA, 2004 (Non-pareil)	SG	0.10	0.01	1169	3	7	20	Hulls (11.1–75.2%)	4.107 (4.047, 4.167)	0.051 (0.052, 0.050)	0.133 (0.139, 0.126)	0.406 (0.402, 0.410)	
							30		0.734 (0.851, 0.616)	< 0.01 (< 0.01, < 0.01)	0.062 (0.067, 0.056)	0.143 (0.165, 0.120)	
							40		0.906 (0.916, 0.896)	0.01 (0.01, 0.01)	0.071 (0.063, 0.078)	0.140 (0.137, 0.143)	
							50		0.686 (0.613, 0.758)	0.01 (< 0.01, 0.013)	0.069 (0.070, 0.068)	0.120 (0.109, 0.130)	
							20	Hulls (dry weight)	16.58 (15.995, 17.147)	0.206 (0.207, 0.205)	0.535 (0.549, 0.520)	1.639 (1.589, 1.688)	
							30		2.032 (2.618, 1.896)	0.022 (0.025, 0.018)	0.190 (0.206, 0.173)	0.439 (0.508, 0.370)	
							40		1.089 (1.094, 1.083)	0.012 (0.012, 0.012)	0.085 (0.075, 0.094)	0.169 (0.164, 0.173)	
							50		0.771 (0.690, 0.851)	0.013 (0.010, 0.015)	0.078 (0.079, 0.076)	0.134 (0.122, 0.146)	
Wasco, CA, 1996 (Fritz)	SG	0.10	0.007	1459–1543	3	6–8	39	Hulls (20.2%)	1.442 (1.807, 1.076)	0.033 (0.034, 0.031)	0.032 (0.034, 0.029)	0.120 (0.133, 0.107)	
							39	Hulls (dry weight)	1.813 (2.297, 1.329)	0.041 (0.043, 0.039)	0.040 (0.044, 0.036)	0.151 (0.169, 0.133)	
Coalinga, CA, 2006 (Non-pareil)	SG	0.10	0.006–0.007	1534–1702	3	7	39	Hulls (24.1%)	0.700 (0.734, 0.665)	0.014 (0.015, 0.013)	0.079 (0.080, 0.078)	0.113 (0.123, 0.102)	
							39	Hulls (dry weight)	0.922 (0.981, 0.863)	0.019 (0.020, 0.017)	0.105 (0.107, 0.102)	0.148 (0.164, 0.132)	
Turlock, CA, 2007 (Butte)	SG	0.10	0.006–0.007	1487–1721	3	7	42	Hulls (60.0%)	1.095 (1.159, 1.030)	0.091 (0.093, 0.089)	0.305 (0.302, 0.308)	0.166 (0.167, 0.164)	
							42	Hulls (dry weight)	2.750 (2.912, 2.587)	0.229 (0.233, 0.225)	0.767 (0.759, 0.774)	0.415 (0.419, 0.411)	

Cotton seed by-products

Twelve independent trials were conducted on cotton in the US in 2001 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–9 days. Seeds were collected 29–32 DALT, dried and cleaned followed by a stick extraction to remove the gin trash. The

lint cotton was saw ginned to remove the majority of the lint from the cottonseed while the ginned seed was saw delinted to remove most of the remaining linters.

In Australia, ten independent trials were conducted on cotton in 2012 where one or two foliar spray applications were made at 0.10 kg ai/ha or 0.20 kg ai/ha at re-treatment intervals of 14–15 days. Cotton was picked from bolls 7–43 DALT and ginned to separate the fuzzy seed and lint. The simulated gin trash consisted of ground parts of the cotton plant including bracts, stems, leaves, immature or mummified bolls, flowers and raw cotton.

Method P-3567, a modified version of analytical method P-3561M was used to analyse all samples collected from the US trials while method AATM-R-165 was used to analyse all samples from the Australian trials. The LOQ was determined to be 0.02 mg/kg/analyte for P-3567. For method AATM-R-165, the LOQ was 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 470 days (ca. 16 months). Storage stability data on oil content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 82.

Table 82 Residues of Flonicamid in Delinted Seeds and Gin Trash Following Foliar Spray with Flonicamid 50WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Commodity	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.05–0.10	0.02–0.05	30–50	3	7	30						
Elko, SC, 2001 (Delta Pine 451 B/RR)	WG	0.10	0.06	168	3	7	29	Delinted cottonseed	0.050 (0.054, 0.046)	< 0.02 (< 0.02, < 0.02)	0.063 (0.064, 0.062)	0.030 (0.032, 0.028)	
West Memphis, AR, 2001 (Suregrow)	WG	0.10	0.07	150	3	7	0	Delinted cottonseed	0.077 (0.063, 0.090)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							10		0.029 (0.025, 0.032)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							21		0.027 (0.028, 0.026)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							30		0.036 (0.035, 0.036)	< 0.02 (< 0.02, < 0.02)	0.066 (0.063, 0.069)	0.026 (0.025, 0.027)	
							40		0.026 (0.022, 0.029)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
Tillar, AR, 2001 (Pay-master)	WG	0.10	0.10	94–103	3	6–9	30	Delinted cottonseed	0.049 (0.047, 0.051)	< 0.02 (< 0.02, < 0.02)	0.070 (0.066, 0.074)	0.032 (0.031, 0.032)	
								Gin trash	1.200 (1.048, 1.352)	0.343 (0.321, 0.364)	0.478 (0.453, 0.502)	1.258 (1.138, 1.377)	
Senatobia, MS, 2001 (DPL 451 Bt/RR)	WG	0.10	0.05	187–196	3	7	29	Delinted cottonseed	0.049 (0.050, 0.048)	< 0.02 (< 0.02, < 0.02)	0.059 (0.059, 0.059)	0.027 (0.027, 0.027)	
Eakly, OK, 2001 (PM 2280)	WG	0.10	0.05	187	3	6–8	30	Delinted cottonseed	0.050 (0.044, 0.055)	< 0.02 (< 0.02, < 0.02)	0.077 (0.075, 0.078)	0.031 (0.029, 0.032)	
Dill City, OK, 2001 (Pay-master 2326)	WG	0.10	0.05–0.06	168–206	3	7	31	Delinted cottonseed	0.027 (0.026, 0.027)	< 0.02 (< 0.02, < 0.02)	0.213 (0.226, 0.199)	0.125 (0.126, 0.123)	
								Gin trash	2.537 (2.550, 2.523)	0.470 (0.468, 0.471)	0.591 (0.620, 0.562)	1.297 (1.363, 1.230)	

Location, year (variety)	Application						DALT, days	Commodity	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
Levelland, TX, 2001 (PM 2326 B6/RR)	WG	0.10	0.07	140	3	6–8	29	Delinted cottonseed	0.026 (0.032, 0.020)	< 0.02 (< 0.02, < 0.02)	0.252 (0.244, 0.260)	0.144 (0.138, 0.149)	
								Gin trash	1.878 (2.093, 1.663)	0.231 (0.275, 0.186)	0.370 (0.446, 0.293)	0.726 (0.881, 0.570)	
Uvalde, TX, 2001 (PM 2326 RR)	WG	0.10	0.05	187–196	3	7	30	Delinted cottonseed	0.024 (< 0.02, 0.027)	< 0.02 (< 0.02, < 0.02)	0.160 (0.146, 0.174)	0.089 (0.084, 0.094)	
Edmonson, TX, 2001 (Paymaster HS 250)	WG	0.10	0.06–0.07	150–187	3	7–8	0	Delinted cottonseed	0.114 (0.112, 0.115)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							11		0.033 (0.029, 0.037)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							20		0.038 (0.035, 0.040)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							32		0.035 (0.041, 0.028)	< 0.02 (< 0.02, < 0.02)	0.261 (0.305, 0.217)	0.149 (0.179, 0.118)	
							43		0.030 (0.032, 0.028)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							32	Gin trash	2.191 (2.411, 1.970)	0.327 (0.370, 0.283)	0.498 (0.604, 0.392)	1.039 (1.220, 0.857)	
Stanfield, AZ, 2001 (DP458 B1RR)	WG	0.10	0.06	187	3	7	29	Delinted cottonseed	0.038 (0.030, 0.045)	< 0.02 (< 0.02, < 0.02)	0.249 (0.265, 0.232)	0.179 (0.212, 0.146)	
Mariopa, AZ, 2001 (DP451 B1RR)	WG	0.10	0.05	187	3	7	30	Delinted cottonseed	0.072 (0.073, 0.070)	< 0.02 (< 0.02, < 0.02)	0.262 (0.260, 0.264)	0.195 (0.204, 0.185)	
								Gin trash	1.223 (1.241, 1.204)	0.331 (0.334, 0.327)	0.464 (0.466, 0.461)	1.169 (1.196, 1.141)	
Madera, CA, 2001 (Acala Riata RR)	WG	0.10–0.11	0.04	281–290	3	7	29	Delinted cottonseed	0.089 (0.115, 0.063)	< 0.02 (< 0.02, < 0.02)	0.227 (0.202, 0.251)	0.149 (0.126, 0.171)	
								Gin trash	1.224 (1.212, 1.235)	0.325 (0.338, 0.312)	0.461 (0.505, 0.416)	1.171 (1.204, 1.137)	
AUS GAP	WG	0.07	NS	NS	2	NS	7						
Mywybilla, Queensland, (Sicot 71BRF)	WG	0.10	0.111	91	2	14	7	Gin trash	2.3	0.2	0.19	0.35	
		0.10	0.121	85			27	Gin trash	0.21	< 0.05	0.38	0.43	
		0.20	0.216	94			7	Gin trash	8.39	0.46	1.13	2.43	
		0.20	0.246	82			27	Gin trash	0.38	0.07	0.83	1.2	
		0.10	0.134	74		15	7	Gin trash	1.33	0.15	0.19	0.33	
		0.10	0.128	76		14	15	Gin trash	0.32	0.069	0.38	0.37	
		0.10	0.130	76		14	22	Gin trash	0.41	0.07	0.35	0.53	
		0.10	0.131	74		14	29	Gin trash	0.37 (0.51, 0.23)	0.108 (0.16, 0.055)	0.54 (0.59, 0.49)	0.83 (1.13, 0.53)	
		0.10	0.130	77		14	36	Gin trash	0.15	< 0.01	0.61	0.9	
		0.10	0.129	77		14	43	Gin trash	0.086	< 0.05	0.47	1.22	
		0.20	0.265	75		15	7	Gin trash	1.02	0.059	0.086	0.11	
		0.20	0.264	77		14	29	Gin trash	0.40 (0.36, 0.44)	0.08 (0.061, 0.10)	0.35 (0.31, 0.38)	0.53 (0.47, 0.59)	
Boggabilla	WG	0.10	0.111	91	2	14	7	Gin trash	2.75	0.068	0.069	0.12	

Location, year (variety)	Application						DALT, days	Commodity	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
, New South Wales (Sicot 71BRF)		0.10	0.112	90		15	28	Gin trash	0.42	0.089	0.43	0.45	
		0.20	0.223	90		14	7	Gin trash	5.19	0.19	0.16	0.31	
		0.20	0.224	90		15	28	Gin trash	1.42	0.28	0.7	0.95	
Narrabi, New South Wales (Sicot 71BRF)	WG	0.10	0.111	90	2	14	7	Gin trash	3.72	0.057	0.086	0.086	
		0.10	0.113	89		15	13	Gin trash	2.42	0.15	0.18	0.11	
		0.10	0.112	89		14	21	Gin trash	0.68	0.12	0.78	0.24	
		0.10	0.11	92		13	28	Gin trash	0.56	0.17	1.06	0.61	
		0.10	0.108	93		14	35	Gin trash	0.16	< 0.01	0.94	0.43	
		0.10	0.113	90		14	41	Gin trash	0.2	0.06	2.66	1.97	
		0.20	0.221	92		14	7	Gin trash	8.23	0.19	0.16	0.15	
		0.20	0.22	92		13	28	Gin trash	0.99	0.27	2.41	1.16	
Chinchilla, Queensland (Sicot 71BRF)	WG	0.10	0.10	98	2	14	7	Gin trash	3.0	0.13	0.23	0.36	UPL GLP 12 01-1
		0.10	0.10	103		14	28	Gin trash	0.18	< 0.01	0.75	0.61	
		0.10	0.10	103		14	49	Gin trash	< 0.05	< 0.05	0.7	0.65	
		0.10	0.10	102		14	63	Gin trash	0.05	< 0.05	0.49	0.89	
		0.20	0.20	98		14	7	Gin trash	6.8	0.24	0.38	0.53	
		0.20	0.20	99		14	28	Gin trash	0.67	< 0.05	0.82	0.92	
		0.20	0.19	104		14	49	Gin trash	0.14	< 0.05	1.1	1.4	
		0.20	0.20	102		14	63	Gin trash	< 0.05	< 0.05	1.4	1.5	
Condamine Plains, Queensland (Sicot71BRF)		0.10	0.10	100	2	14	7	Gin trash	1.7	0.47	0.23	0.55	
		0.10	0.09	111		15	20	Gin trash	1.1	0.29	0.36	0.52	
		0.10	0.10	103		14	27	Gin trash	1.1	0.16	0.29	0.38	
		0.10	0.10	103	1	NA	27	Gin trash	1.7	0.24	0.12	0.22	
		0.10	0.10	102	2	14	35	Gin trash	0.35	0.12	0.22	0.33	
	WG	0.10	0.10	103		14	41	Gin trash	0.27	0.15	0.74	1.2	
		0.05	0.05	103		13	49	Gin trash	0.05	< 0.05	0.4	0.48	
		0.10	0.10	102		14	55	Gin trash	0.11	< 0.05	0.91	2.2	
		0.20	0.20	100		13	7	Gin trash	1.4	0.25	0.11	0.27	
		0.20	0.19	103		14	27	Gin trash	1.3	0.36	0.57	0.9	
		0.20	0.20	103		14	41	Gin trash	0.63	0.12	1.1	1.7	
		0.20	0.20	102		14	55	Gin trash	0.17	0.14	1.3	3.3	
Moree, New South Wales (Sicot 71BRF)	WG	0.10	0.11	89–91	2	14	7	Gin trash	1.2	< 0.05	< 0.05	< 0.05	
		0.10	0.11	91		14	14	Gin trash	1.3	0.08	0.05	0.07	
		0.10	0.11	91–92		14	21	Gin trash	0.74	< 0.05	0.19	0.22	
		0.10	0.11	90–94		14	28	Gin trash	0.56	< 0.05	0.45	0.27	
		0.10	0.11	92		14	35	Gin trash	0.71	0.06	1.4	1.2	
		0.10	0.11	92–93		14	42	Gin trash	0.6	< 0.05	0.44	0.97	
		0.10	0.11	89–92		14	49	Gin trash	0.33	0.05	0.76	1.8	
		0.10	0.11	90–93		14	56	Gin trash	0.07	< 0.05	0.28	0.72	
		0.20	0.22–0.23	89–93		14	7	Gin trash	1.7	< 0.05	< 0.05	< 0.05	
		0.20	0.22	90–91		14	28	Gin trash	1.5	0.05	0.21	0.16	
		0.20	0.22–0.23	90–93		14	42	Gin trash	0.98	0.07	0.75	1.5	
		0.20	0.22–0.23	90–93		14	56	Gin trash	0.22	< 0.05	1	2.4	

Location, year (variety)	Application						DALT, days	Commodity	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
Narrabri, New South Wales (Sicot 71BRF)	WG	0.10	0.11	90–92	2	14	8	Gin trash	1.6	0.07	0.05	0.07	UPL GLP 12 01-1
		0.10	0.11	89-90	2	14	15	Gin trash	0.82	0.06	0.13	0.13	
		0.10	0.11	91–92	2	14	22	Gin trash	0.89	0.05	0.14	0.23	
		0.10	0.11	89–90	2	14	29	Gin trash	0.66	0.07	0.34	0.57	
		0.10	0.11	91	2	14	36	Gin trash	0.58	0.07	0.54	1.1	
		0.10	0.11	88–91	2	14	43	Gin trash	0.31	0.05	0.37	0.73	
		0.10	0.11	90	2	14	50	Gin trash	0.36	0.08	0.8	1.7	
		0.10	0.11–0.12	84–86	2	14	57	Gin trash	0.09	< 0.05	0.67	1.6	
		0.23	0.22	92–93	2	14	8	Gin trash	2.1	0.2	0.12	0.18	
		0.21	0.22	92	2	14	29	Gin trash	1.1	0.11	0.48	1	
		0.20	0.22–0.24	85–92	2	14	43	Gin trash	0.42	0.07	0.96	1.8	
		0.20	0.24	85–86	2	14	57	Gin trash	0.15	< 0.05	0.91	1.9	
Narromine, New South Wales (Sicot 71BRF)	WG	0.10	0.08–0.09	114–125	2	14	7	Gin trash	0.66	< 0.05	< 0.05	< 0.05	
		0.10	0.08–0.09	116–119	2	14	28	Gin trash	0.15	< 0.05	1.2	0.62	
		0.10	0.08	123–124	2	11	42	Gin trash	< 0.05	< 0.05	0.44	0.24	
		0.10	0.08–0.09	117–123	2	15	53	Gin trash	< 0.05	< 0.05	1	0.49	
		0.20	0.17–0.18	114–119	2	14	7	Gin trash	1.6	< 0.05	0.13	0.07	
		0.21	0.17	122–124	2	14	28	Gin trash	1.2	0.14	1.1	1	
		0.21	0.17	119–123	2	11	42	Gin trash	< 0.05	< 0.05	0.87	0.41	
		0.21	0.17–0.18	118–123	2	15	53	Gin trash	< 0.05	< 0.05	0.58	0.27	

FATE OF RESIDUES DURING PROCESSING

In processing-nature of residues

Hydrolysis of flonicamid, radio-labelled in the pyridine ring (specific activity 9.08 MBq/mg), at 1.0 mg ai/L, was investigated in aqueous buffer solutions (0.1 M sodium citrate-citric acid), at 90 °C and pH4 for 20 min (simulating pasteurisation), at 100 °C and pH 5 for 60 min (simulating baking, brewing and boiling), and at 120 °C and pH 6 for 20 min (simulating sterilisation).

Quantitative measurement of the radioactivity was carried out by LSC. Further analysis to quantify and identify the radio-labelled degradation products present in the test solutions was conducted using HPLC and TLC. Flonicamid was identified by HPLC co-chromatography with a certified standard. Selected samples were analysed by TLC to confirm the presence of flonicamid.

Table 83 Degradation of flonicamid under various hydrolysis conditions

Condition	Sampling Regime	Flonicamid [% AR]	Total Others [% AR]	Total Recovery [% AR]
pH 4, 90 °C	Heated	100.28	0.49	100.77
	Control	99.21	0.59	99.80
pH 5, 100 °C	Heated	96.87	0.98	97.85
	Control	97.19	0.57	97.76
pH 6, 120 °C	Heated	96.58	1.72	98.30

Condition	Sampling Regime	Flonicamid [% AR]	Total Others [% AR]	Total Recovery [% AR]
	Control	96.47	0.92	97.39

Overall good recovery of radioactivity was achieved for each of the processing conditions, ranging from 94.6 to 101.1% of the applied radioactivity (AR).

In all cases, flonicamid accounted for at least 96.5% of AR. Therefore, very limited degradation of flonicamid was observed in aqueous buffer solutions under all the conditions tested with no significant degradation product being formed.

In processing-effect on the residue level

The Meeting received information on the fate of flonicamid residues and its metabolites TFNA-AM, TFNA and TFNG during the processing of raw agricultural commodities (RAC) in apples to juice; peaches to canned peaches, juice, jam and puree; plums to dried prunes; tomatoes to paste; potatoes to chips and flakes; rape seed and cotton to refined oil and meal and mint to oil.

Processing of apples

One study was conducted in 2002 in Lyons, New York where apple trees were treated with three foliar spray applications, where the first two treatments were at 0.103 kg ai/ha and the third treatment was at 0.516 kg ai/ha, for a total of 0.722 kg ai/ha. The fruit was harvested 21 days after the last application and transported to the lab for processing into juice and pomace. 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 84 and 93. To make juice and wet pomace, apples were ground in a hammer-mill. The resulting wet mash was loaded in one or more cloth stacks on the hydraulic press. The cloth stacks were pressed for 5 minutes at 2200–3000 psi to remove the apple juice. The wet pomace sample was then taken from the cloth stacks and bagged.

Table 84 Residues of flonicamid in apples (RAC and processed fractions)

RAC/ Processed commodity	Residues (mg/kg)				Processing Factor				Reference
	Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
Apple fruit	0.032, 0.036 (0.034)	< 0.01	0.038, 0.041 (0.040)	< 0.01	–	–	–	–	IB-2001-MDG-003-00-01
Wet pomace	0.091, 0.101 (0.096)	< 0.01	0.049, 0.053 (0.051)	0.008, 0.008 (0.008)	2.82	NA	1.28	NA	
Juice	0.122, 0.127 (0.125)	< 0.01	0.139, 0.139 (0.139)	0.011, 0.011 (0.011)	3.67	NA	3.48	NA	

NA = Not applicable

Processing of peaches

Four processing trials were conducted in 2001 in Italy (two), Spain and Southern France where peach trees were treated with two applications of a WG formulation at a rate of 0.07 kg ai/ha/application for a total of 0.140 kg ai/ha. The peaches were harvested 14 days following the last application and processed into canned peaches, juice, jam and puree. 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 85 and 93. The information submitted on processing procedures is summarized as follows.

For each processed commodity, peaches were dipped in boiling water for a few minutes, peeled and stones removed.

Canned peaches

The fruits were cut in halves and placed in glass containers. Peaches were then covered with a 400 g/L sucrose solution. The containers were sealed and sterilized for 15 mins in a boiling water bath. The canned peaches were then cooled.

Juice

The fruits were cut into small pieces and weighed. The pulp was pressed through a sieve of 1 mm mesh size. The mixture was then centrifuged at 7500 rpm and filtered through a filter paper. The final volume of juice and sucrose content was measured and reported. Juice was transferred into a glass container, which was sealed and sterilized for 15 mins at 100 °C.

Jam

The fruits were cut in small pieces and weighed. A syrup solution was prepared by adding 50 mL of water to the same weight of sucrose as the quantity of peaches involved. The solution was cooked until complete dissolution of sucrose. The peaches were added to the syrup and cooked a few minutes before crushing. Pectin and citric acid were added to the mixture, corresponding to 0.5% and 0.6% in weight of sucrose added, respectively. The mixture was pressed through a sieve and the jam was cooked and controlled for sucrose concentration using a refractometer. Cooking was stopped as soon as 61% of sucrose concentration was achieved. The jam was transferred into a glass container, which was sealed and sterilized for 30 mins at 100 °C.

Purée

The fruits were cut into small pieces, weighed and transferred into a glass container. Sucrose was added equivalent to 10% of the weight of the peaches. The container was sealed and heated at 100 °C for 30 minutes. The syrup generated was removed and the volume was reported. The peaches were then crushed through a sieve. The weight of the resulting purée was reported and the sugar concentration was measured using a refractometer. The final sugar content was adjusted to 28% using sucrose. The purée was transferred into a glass container, which was sealed and sterilized for 15 min at 100 °C

Table 85 Residues of flonicamid in peaches (RAC and processed fractions)

Country, Year	RAC/Processed Commodity	Residues (mg/kg)				Processing Factor				Reference
		Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
Italy, 2001	Peaches	0.03	< 0.01	< 0.01	< 0.01	–	–	–	–	P-22-01-02
	Canned peaches	< 0.01	< 0.01	< 0.01	< 0.01	0.33	NA	NA	NA	
	Fruit juice	0.03	< 0.01	0.01	0.01	1	NA	NA	NA	
	Jam	0.01	< 0.01	0.02	0.04	0.33	1	2	4	
	Purée	0.02	< 0.01	< 0.01	< 0.01	0.67	NA	NA	NA	
	Peel	0.01	< 0.01	< 0.01	< 0.01	0.33	NA	NA	NA	
	Waste material out of purée	0.02	< 0.01	< 0.01	< 0.01	0.67	NA	NA	NA	
	Blanching water	0.03	< 0.01	0.01	0.01	1	NA	NA	NA	
	Waste material out of juice	0.02	< 0.01	< 0.01	< 0.01	0.67	NA	NA	NA	
Italy, 2001	Peaches	0.02	< 0.01	< 0.01	< 0.01	–	–	–	–	
	Canned peaches	< 0.01	< 0.01	< 0.01	< 0.01	0.5	NA	NA	NA	
	Fruit juice	0.02	< 0.01	< 0.01	< 0.01	1	NA	NA	NA	
	Jam	0.02	< 0.01	0.01	< 0.01	1	NA	NA	NA	

Country, Year	RAC/Processed Commodity	Residues (mg/kg)				Processing Factor				Reference
		Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
	Purée	0.02	< 0.01	< 0.01	< 0.01	1	NA	NA	NA	
Spain, 2001	Peaches	0.03	< 0.01	< 0.01	< 0.01	–	–	–	–	
	Canned peaches	0.01	< 0.01	< 0.01	< 0.01	0.33	NA	NA	NA	
	Fruit juice	0.01	< 0.01	< 0.01	< 0.01	0.33	NA	NA	NA	
	Jam	0.03	< 0.01	< 0.01	< 0.01	1	NA	NA	NA	
	Purée	0.03	< 0.01	< 0.01	< 0.01	1	NA	NA	NA	
South of France, 2001	Peaches	0.06	< 0.01	0.02	0.01	–	–	–	–	
	Canned peaches	0.10	< 0.01	0.02	0.01	1.67	NA	1	1	
	Fruit juice	0.03	< 0.01	0.03	< 0.01	0.5	NA	1.5	NA	
	Jam	0.01	< 0.01	0.03	< 0.01	0.17	NA	1.5	NA	
	Purée	0.05	< 0.01	0.03	< 0.01	0.83	NA	1.5	NA	

Processing of plums

One processing trial was conducted in 1992 in Fairfield, California where plum trees were treated with three foliar spray applications where the first two treatments were at 0.103 kg ai/ha and the third treatment was at 0.516 kg ai/ha, for a total of 0.722 kg ai/ha. The fruit was harvested 14 days after the last application and dried. 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 86 and 93. The information submitted on processing procedures is summarized as follows.

Dried prune

The plums were washed for five minutes in a tub of cold water. The washed plums were spread single layer on trays and dehydrated in a tray air dryer at 68–79 °C for 18–36 hours to reduce the moisture content to the desired range (19–29%).

Table 86 Residues of flonicamid in plums (RAC and processed fractions)

RAC/ Processed commodity	Residues (mg/kg)				Processing Factor				Reference
	Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
Plum	0.280 (0.275, 0.284)	0.024 (0.025, 0.023)	0.016, (0.016 0.016)	0.032 (0.033, 0.031)	–	–	–	–	IB-2001_MDG-004-00-01
Dried prune	0.278 (0.264, 0.287)	0.018 (0.017, 0.018)	0.024 (0.026, 0.021)	0.036 (0.038, 0.034)	1	0.75	1.5	1.13	

Processing of tomato

One study was conducted in 2001 in Davis, California where tomato plants were treated with three foliar spray applications where the first two treatments were at 0.102 kg ai/ha and the third treatment was at 0.506 kg ai/ha, for a total of 0.710 kg ai/ha. The fruit was harvested immediately after the last application and transported to the lab for processing into paste. The results and the calculated processing factors (residue in processed commodity/residue in RAC) for MRL setting and dietary intake purposes are presented in Table 93. The information submitted on processing procedures is summarized as follows.

Paste

The tomato fruit were batch rinsed using a high-pressure spray rinse at approximately 70–75 °C for 30 seconds per batch. The fruit was hand fed into the hammermill assembly of the Suntech Fruit Press for crushing. The crushed tomatoes were transferred to the Hubbert Steam Jacketed Kettle and rapidly heated to approximately 80–85 °C and held for 25–30 seconds. The hot break juice was hand fed into the Pulper Finisher for the separation of pomace and juice. The wet pomace recovered was pressed using the Suntech Fruit Press. The pressed wet pomace was discarded and the recovered press juice was returned to the finished juice.

The juice was then transferred to the Groen Vacuum Evaporator. The puree was removed from the evaporator when the desired Brix range was achieved. A portion of the puree was transferred to the 7.5 L Scrape Surface Vacuum Evaporator. The paste was removed from the evaporator when the desired Brix range was achieved. A portion of the paste was removed and 1% salt was added to adjust the Brix to the desired range of 24.0–30.0 °C. The paste was heated to 82–88 °C. The heated paste was packed in 3303 cans and sealed using the Dixie Electric Can Sealer. The sealed cans were then processed using an Open Atmospheric Water Bath Kettle for 15–20 minutes at 96–100 °C and then cooled under running tap water. A representative sample of the cooled canned puree was removed, packaged, labelled and placed in the freezer for the required sample fraction. The excess evaporated puree and paste was discarded.

Table 87 Residues of flonicamid in tomato (RAC and processed fractions)

RAC/ Processed commodity	Residues (mg/kg)				Processing Factor				Reference
	Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
Tomato	0.031 (0.029, 0.031, 0.033, 0.031)	< 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)	–	–	–	–	IB-2001-MDG-006-00-1
Paste	0.499 (0.494, 0.503)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.028, 0.029 (0.029)	16.1	NA	NA	2.8	

Processing of potato

One study was conducted in 2001 in Ephrata, Washington where potato plants were treated with three foliar spray applications where the first two treatments were at 0.10 kg ai/ha and the third treatment was at 1.0 kg ai/ha, for a total of 1.22 kg ai/ha. The fruit was harvested immediately after the last application and transported to the lab for processing into chips and flakes. 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 88 and 93. The information submitted on processing procedures is summarized as follows.

Chips

The potatoes are peeled for 25–35 seconds in batches using an abrasive peeler. A certain amount of peel is left on the tuber to produce a natural appearance to the finished product. The peel collected is weighed and discarded. The peeled potatoes are individually inspected and hand trimmed. The potatoes are cut using a restaurant style food cutter/slicer. The slices are placed in a tub of hot tap water to rinse the free starch from the surfaces of the slices. The slices are deep fried in a restaurant style deep fat fryer at approximately 163–191 °C for 60–90 seconds. Free oil is drained from the chips using a draining tray and salted by hand. The chips are inspected and undesirable chips are removed.

Flakes

Washed potatoes are sorted and scrubbed in batches using a restaurant style peeler fitted with a rubber scrubber for approximately 25–35 seconds. The peel is then hydraulically pressed to increase the solids content. Potatoes are then individually inspected, hand trimmed and cut into slabs using a restaurant style food cutter/slicer with a cutting blade set to approximately 1–1.3 cm. The potato slices are spray washed for approximately 30 seconds in cold water to rinse the free starch from the surface of the slices. The potato slices are precooked at approximately 70–77 °C for 202–22 minutes using a steam jacketed kettle and subsequently cooled down to less than 32 °C using cold running tap water in a 150 L steam jacketed kettle for 20–22 minutes. The cooled slices are steam cooked at 94–100 °C for 40–42 minutes using an atmospherically flowing steam batch style steam cooker and mashed using a restaurant style meat grinder without the grinding attachment. The mashed potatoes are placed in a bakery style mixer where an emulsion containing the additives are poured into the mashed potatoes and mixed for approximately 60 seconds. The potato mash is hand fed onto a laboratory single drum dryer where the potato mash is dried into a thin sheet. The dried potato sheet is broken into flakes. The large flakes are then hand fed into a hammermill for uniform sizing of the finished flakes. If moisture content of the potato flakes exceeds 9%, the flakes are dried on the fluidized bed dryer to less than or equal to 9% moisture.

Table 88 Residues of flonicamid in potatoes (RAC and processed fractions)

RAC/ Processed commodity	Residues (mg/kg)				Processing Factor				Reference
	Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
Potatoes	0.022 (0.022, 0.022)	< 0.01 (< 0.01, < 0.01)	0.041 (0.040, 0.041)	0.029 (0.030, 0.028)	–	–	–	–	IB-2001-MDG-002-00-01
Wet Peel	0.011 (0.010, 0.011)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.50	NA	0.24	0.3	
Chips	0.021 (0.021, 0.021)	< 0.01 (< 0.01, < 0.01)	0.072 (0.071, 0.072)	0.051 (0.049, 0.053)	0.95	NA	1.8	1.8	
Flakes	0.060 (0.059, 0.060)	< 0.01 (< 0.01, < 0.01)	0.122 (0.117, 0.126)	0.092 (0.089, 0.094)	2.73	NA	2.98	3.17	

The best estimates of the processing factors for parent residues (for MRL setting in case of residue increasing) and for the sum of flonicamid, TFNA-AM, TFNA AND TFNG (for dietary intake) are summarized in Table 93.

Rape seed

One study was conducted in 2007 in Prosser, Washington where rape seed plants were treated with three foliar spray applications of 0.30 kg ai/ha for a total of 0.90 kg ai/ha. The seeds were harvested 8 days following the last application and transported to the lab for processing into refined oil and meal. The results and the calculated processing factors (residue in processed commodity/residue in RAC) for MRL setting and dietary intake purposes are presented in Table 93. The information submitted on processing procedures is summarized as follows.

Refined Oil

Canola seeds were flaked in a flaking roll and flakes were heated to 85–100 °C and held for 10 to 15 minutes in the temperature range. Flakes were pressed (expelled) in an expeller to mechanically remove a portion of the crude oil. Residual crude oil remaining in the solid material (presscake) exiting the expeller was extracted with the hexane. The miscella (crude oil and hexane) was passed through a laboratory recovery unit to separate the crude oil and hexane. Crude oil was heated to 90–96 °C for hexane removal. Crude oil samples recovered from the expeller and solvent extraction were

filtered and combined. Percent free fatty acid (FFA) for the crude oil was determined. Crude canola oil was placed in a water bath and pre-treated with 85% phosphoric acid. Oil was mixed for 29–31 minutes at 40–45 °C. After the pre-treatment, an amount of 12°Baume sodium hydroxide was added to the oil. The samples were mixed for 19–21 minutes at 40–45 °C and then for 9–11 minutes at 65–70 °C. The neutralized oil was then centrifuged to separate the refined oil and soapstock. The refined oil was decanted and filtered. Soapstock was discarded. Resulting fraction of alkali refined oil was collected and frozen.

Meal

Presscake was placed in stainless steel batch extractors and submerged in 50–60 °C solvent (hexane). After 30 minutes, the hexane was drained and fresh hexane added to repeat the cycle two more times. The final two washes were for 15–30 minutes each. After the final draining, the extracted presscake (meal) was desolventized using warm air forced through the extracted presscake. Resulting fraction, canola meal was collected and placed into frozen storage.

Table 89 Residues of flonicamid in rape seed (RAC and processed fractions)

RAC/ Processed commodity	Residues (mg/kg)				Processing Factor				Reference
	Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
Whole seed	0.232	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	–	–	–	–	9783
Meal	< 0.02	< 0.02	< 0.02	< 0.02	< 0.1	NA	NA	NA	
Refined oil	< 0.02	< 0.02	< 0.02	< 0.02	< 0.1	NA	NA	NA	

Processing of cotton

Study 1: US

One processing trial was conducted in 2001 in Uvalde, Texas, where cotton was treated with three applications of a WG formulation where the first two treatments were made at a rate 0.10 kg ai/ha/application and the third treatment was made at a rate of 1.0 kg ai/ha/application for a total of 1.2 kg ai/ha. The undelinted cottonseed was harvested 30 days following the last application and processed into meal and oil. 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 90 and 93. The information submitted on processing procedures is summarized as follows.

Cottonseed hulls

Cotton seed was dried and cleaned followed stick extraction to remove the gin trash. The lint cotton was saw ginned to remove 85–89% of the lint from the cottonseed. The ginned seed was saw delinted to remove most of the remaining linters. Approximately 3% of the lint remained with the seed. A mill was used to crack the seed and the hulls were removed from the kernels and sampled for analysis.

Cottonseed oil and meal

The kernels were dried to < 12% water, heated to 80–90 ºC for 30 minutes, and flaked, followed by passage through an expander extruder to form collets. The collets were submerged in hexane at 50–60 ºC for 30 minutes and washed twice with fresh hexane to remove the cottonseed oil. Residual hexane was removed from the meal fractions with warm air and the meal was sampled for analysis. Hexane was removed from the oil with a vacuum extractor, NaOH was added to precipitate the soap stock, the remaining hexane was removed and refined oil was sampled for analysis.

Table 90 Residues of flonicamid in cotton (RAC and processed fractions)—US

RAC/	Residues (mg/kg)	Processing Factor	Reference

Processed commodity	Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
Seed at processing	0.084 (0.079, 0.088)	< 0.02 (< 0.02, < 0.02)	0.101 (0.092, 0.110)	0.080 (0.070, 0.090)	–	–	–	–	IB-2001-MDG-004-00-01
Hulls	0.071 (0.072, 0.069)	< 0.02 (< 0.02, < 0.02)	0.353 (0.351, 0.354)	0.210 (0.207, 0.212)	0.84	NA	3.5	2.6	
Meal	0.023 (0.023, 0.023)	< 0.02 (< 0.02, < 0.02)	0.899 (0.894, 0.883)	0.483 (0.489, 0.476)	0.27	NA	8.9	6.0	
Refined oil	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.24	NA	0.20	0.25	

Study 2—Australia

One processing trial was conducted in 2012 in Narrabri, New South Wales, where cotton was treated with two applications of a WG formulation at a rate 0.10 kg ai/ha/application or 0.20 kg ai/ha/application for a total of 0.2 kg ai/ha or 0.4 kg ai/ha, respectively. The undelinted cottonseed was harvested 8, 15, 22, 29, 36, 43, 50 and 57 DALA and processed into meal and oil. 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 91 and 93. The information submitted on processing procedures is summarized as follows.

Cottonseed hulls

The fuzzy seed was passed through a hand driven mechanical grinder to crack the hulls. The cracked fuzzy seed was sieved to separate the hulls from the unprocessed meal.

Cottonseed meal and oil

The unprocessed meal was placed in a small bolt apparatus which was screwed together to press the meal and extract the oil via pressure. Oil was collected during pressing from the drain hole on the apparatus using a syringe and collected in a plastic vial. The process was repeated until at least 1 m of oil was collected.

Table 91 Residues of flonicamid in cotton (RAC and processed fractions)—Australia

RAC/Processed Commodity	DALT	Residues (mg/kg)				Processing Factor				Reference
		Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
Total Application Rate of 0.20 kg ai/ha										
Seed at processing	8	0.034	0.071	0.025	0.048	–	–	–	–	
Hulls		0.13	< 0.02	< 0.02	< 0.02	3.8	0.3	0.8	0.4	
Meal		0.15	0.03	< 0.02	0.02	4.4	0.4	0.8	0.4	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.6	0.3	0.8	0.4	
Seed at processing	15	0.067	0.033	0.019	0.054	–	–	–	–	
Hulls		0.04	< 0.02	< 0.02	< 0.02	0.6	0.6	1.0	0.4	UPL GLP 12 01-1
Meal		< 0.02	< 0.02	< 0.02	< 0.02	0.3	0.6	1.0	0.4	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.3	0.6	1.0	0.4	
Seed at processing	22	0.11	0.047	0.043	0.12	–	–	–	–	
Hulls		0.05	< 0.02	< 0.02	0.02	0.4	0.4	0.5	0.2	
Meal		0.04	0.03	0.03	0.06	0.4	0.6	0.7	0.5	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.2	0.4	0.5	0.2	
Seed at processing	29	0.13	0.069	0.051	0.16	–	–	–	–	
Hulls		0.04	< 0.02	< 0.02	< 0.02	0.3	0.3	0.4	0.1	

RAC/Processed Commodity	DALT	Residues (mg/kg)				Processing Factor				Reference
		Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
Meal		0.05	0.05	0.08	0.12	0.4	0.7	1.6	0.8	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.2	0.3	0.4	0.1	
Seed at processing	36	0.088	0.076	0.075	0.23	–	–	–	–	
Hulls		0.10	< 0.02	0.04	0.02	1.1	0.3	0.5	0.09	
Meal		0.02	0.02	0.12	0.10	0.2	0.3	1.6	0.4	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.2	0.3	0.3	0.09	
Seed at processing	43	0.032	0.029	0.064	0.12	–	–	–	–	
Hulls		0.04	< 0.02	0.02	< 0.02	1.2	0.7	0.3	0.2	
Meal		< 0.02	0.02	0.06	0.07	0.6	0.7	0.9	0.6	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.6	0.7	0.3	0.2	
Seed at processing	50	0.025	0.028	0.098	0.16	–	–	–	–	
Hulls		0.06	< 0.02	0.04	0.02	2.4	0.7	0.4	0.1	
Meal		0.02	0.03	0.15	0.18	0.8	1.1	1.5	1.1	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.8	0.7	0.2	0.1	
Seed at processing	57	< 0.02	< 0.02	0.10	0.12	–	–	–	–	
Hulls		0.02	< 0.02	0.04	0.02	NA	NA	0.4	0.2	
Meal		< 0.02	< 0.02	0.11	0.09	NA	NA	1.1	0.8	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	NA	NA	0.2	0.2	
Total Application Rate of 0.40 kg ai/ha										
Seed at processing	8	0.48	0.72	0.26	0.049	–	–	–	–	
Hulls		0.26	< 0.02	< 0.02	< 0.02	0.54	0.03	0.08	0.41	
Meal		0.12	0.03	< 0.02	0.02	0.25	0.04	0.08	0.41	
Refined oil		0.02	< 0.02	< 0.02	< 0.02	0.04	0.03	0.08	0.41	
Seed at processing	29	0.11	0.082	0.053	0.17	–	–	–	–	
Hulls		0.08	< 0.02	0.02	< 0.02	0.7	0.2	0.4	0.1	
Meal		0.02	0.02	0.05	0.06	0.2	0.2	0.9	0.4	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.2	0.2	0.4	0.1	
Seed at processing	43	0.096	0.097	0.13	0.33	–	–	–	–	
Hulls		0.09	0.02	0.04	0.02	0.9	0.2	0.3	0.1	
Meal		0.03	0.03	0.13	0.12	0.3	0.3	1.0	0.4	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.2	0.2	0.2	0.1	
Seed at processing	57	0.022	0.025	0.12	0.19	–	–	–	–	
Hulls		0.03	< 0.02	0.05	0.02	1.4	0.8	0.4	0.1	
Meal		< 0.02	0.03	0.19	0.20	0.9	1.2	1.6	1.1	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.9	0.8	0.2	0.1	

Mint

Two processing trials were conducted in 2011 in Moxee, Washington and Endeavour and Wisconsin. Mint was treated with three applications of a SG formulation at a rate 0.10 kg ai/ha/application for a total of 0.3 kg ai/ha. The mint tops were harvested 7 days following the last application and processed into oil. 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 92 and 93. No information was submitted on processing procedures.

The maximum storage intervals for mint oil was 368 days. Concurrent storage stability samples were fortified with flonicamid and its metabolites at 0.2 ppm soon after the receipt of the samples by the analytical laboratory. The storage stability samples were held in frozen storage under similar conditions to the field generated samples. After 334 days of freezer storage for mint oil, the storage stability samples were analysed for flonicamid. The recoveries for the mint

oil storage stability samples were in the ranges 43–46% (flonicamid), 42–49% (TFNA), 46–53% (TFNA-AM), and 42–45% (TFNG). Concurrent recoveries for spikes analysed along with these storage stability samples were 95% (flonicamid), 100% (TFNA), 81% (TFNA-AM), and 89% (TFNG). These data indicate that flonicamid and its metabolites undergo about 50% degradation in mint oil under the conditions which the samples were held between harvest and analysis. However, even when correcting for in-storage dissipation, residues of flonicamid and its metabolites do not concentrate in mint oil.

Table 92 Residues of flonicamid in mint (RAC and processed fractions)

RAC/ Processed commodity	Residues (mg/kg)					Average Processing Factors					Reference
	Flonicamid	TFNA-AM	TFNA	TFNG	Sum	Flonicamid	TFNA-AM	TFNA	TFNG	Sum	
Mint tops	1.57 (1.55, 1.59)	0.339 (0.329, 0.349)	0.171 (0.170, 0.171)	0.193 (0.193, 0.193)	2.273	< 0.03 (< 0.01, < 0.04)	< 0.08 (< 0.06, < 0.09)	< 0.20 (< 0.12, < 0.27)	< 0.14 (< 0.10, < 0.18)	< 0.07 (< 0.04, < 0.09)	9358
Mint oil	< 0.02	< 0.02	< 0.02	< 0.02	< 0.08						
Mint tops	0.502 (0.500, 0.504)	0.222 (0.219, 0.225)	0.074 (0.072, 0.075)	0.108 (0.107, 0.108)	0.906						
Mint oil	< 0.02	< 0.02	< 0.02	< 0.02	< 0.08						

Table 93 Summary of processing factors for flonicamid residues

RAC	Processed Commodity	Calculated processing factors	Best estimate
		Flonicamid	
Apples	Juice	3.7	3.7
	Pomace	2.82	2.82
Peaches	Canned peaches	0.3, 0.5, 0.3, 1.7	0.7 (median)
	Juice	1.0, 1.0, 0.3, 0.5	0.8 (median)
	Jam	0.3, 1.0, 1.0, 0.2	0.7 (median)
	Puree	0.7, 1.0, 1.0, 0.8	0.9 (median)
Plums	Dried prunes	1.0	1.0
Tomato	Paste	16.1	16.1
Potato	Chips	0.95	0.95
	Flakes	2.7	2.7
Canola	Refined oil	< 0.1	0.1
	Meal	< 0.1	0.1
Cotton	Refined oil	< 0.24 (US); 0.6 and 0.04 (AUS)	0.32 (mean; AUS)
	Hulls	0.8 (US); 3.8, 0.5 (AUS)	2.2 (mean; AUS)
	Meal	0.3 (US); 4.4, 0.2 (AUS)	2.3 (mean; AUS)
Mint	Oil	< 0.03	0.03

Residues in animal commodities

Dairy Cattle

One cattle feeding study was conducted where twelve dairy cows (Red Holstein and Simmentaler Fleckvieh, 4–9 years old, 550–770 kg bw) were divided into three groups. Animals were treated twice daily with a 1/1 mixture of flonicamid/TFNG by means of gelatin capsules and using a balling gun. Treatments were made after the morning and evening milking for 28 consecutive days. One group of three cows served as a control group The actual average doses administered were 0.086, 0.252 and 0.839 mg/kg bw. Based on the actual average daily feed intake of 20.1–25.1 kg/day (or 3.0–4.4 kg/day/100 kg bw) during the acclimation period, the actual dosing levels, constituting a 1/1 mixture of flonicamid/TFNG, were 2.50 mg, 6.89 mg and 23.69 mg/kg feed. All cows were sacrificed after 28 days of dosing, within 24 hours after the last dose.

Milk samples were collected on 15 selected days throughout the administration period. All milk samples were frozen at –20 ºC and analysed within 30 days after sampling. Therefore, storage stability data are not necessary. In contrast, all tissue samples were analysed within 12 months of collection. Freezer storage stability studies, conducted concurrently with the feeding studies, demonstrated that flonicamid, TFNA, TFNA-AM, OH-TFNA-AM and TFNG were stable for 374 days in all tissues except fat. For fat, flonicamid and its metabolites were demonstrated to be stable for 315 days.

All samples were analysed for residues of flonicamid, TFNA, TFNA-AM, OH-TFNA-AM and TFNG using validated analytical methods. In general, the samples were homogenised, extracted and the supernatant was purified by means of liquid-liquid partition or gel permeation chromatography. Some of the solid residues were further subjected to acid hydrolysis. The concentration of flonicamid and its metabolites in the purified extracts were determined by HPLC MS/MS. The LOQ for flonicamid and each of its metabolites in milk and fat is 0.01 mg/kg and for muscle the LOQ is 0.025 mg/kg while for liver and kidney the LOQ is dependent on the method used (0.01 or 0.025 mg/kg).

In milk, no quantifiable (< LOQ) residues of flonicamid, TFNG and TFNA were detected in any test group. For TFNA-AM, the average residues increased from < LOQ in the low dose group to 0.02 mg/kg in the mid dose group and to 0.08 mg/kg in the high dose group. OH-TFNA-AM average residues were ⩽ LOQ in the low and mid-dose groups and increased to 0.015 mg/kg in the high dose group.

Table 94 Residues in whole milk following 28 days oral administration of flonicamid to dairy cows

Low Dose (2.5 mg/kg feed)			Mid dose (6.89 mg/kg feed)		High dose (23.69 mg/kg feed)	
Day	TFNA-AM (mg/kg)	OH-TFNA-AM (mg/kg)	TFNA-AM (mg/kg)	OH-TFNA-AM (mg/kg)	TFNA-AM (mg/kg)	OH-TFNA-AM (mg/kg)
1	< 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)	0.032 (0.035, 0.032, 0.027)	< 0.01 (< 0.01, < 0.01, < 0.01)
2	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.018 (0.019, 0.020, 0.014)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.080 (0.091,0.088, 0.060)	0.015 (0.021, 0.011, 0.011)
3	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.022 (0.024, 0.022, 0.018)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.086 (0.111, 0.093, 0.074)	0.016 (0.027, 0.013, 0.014)
4	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.023 0.025, 0.026, 0.019)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.093 (0.111, 0.093, 0.075)	0.018 (0.027, 0.013, 0.014)
5	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.030 (0.027, 0.042, 0.021)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.082 (0.109, 0.094, 0.042)	0.017 (0.027, 0.016, < 0.01)
6	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.026 (0.024, 0.030, 0.022)	0.0101 (0.0102, < 0.01, < 0.01)	0.092 (0.105, 0.093, 0.078)	0.019 (0.026, 0.015, 0.015)
7	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.024 (0.026, 0.027, 0.019)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.085 (0.094, 0.081, 0.081)	0.016 (0.021, 0.013, 0.014)
8	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.020 (0.024, 0.022, 0.012)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.069 (0.085, 0.062, 0.0601)	0.013 (0.018, < 0.01, < 0.01)
10	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.018 (0.016, 0.022, 0.017)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.049 (0.019, 0.069, 0.058)	0.013 (0.016, < 0.01, 0.012)
14	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.018 (0.018, 0.019, 0.016)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.068 (0.074, 0.075, 0.056)	0.013 (0.018, 0.010, 0.011)

Low Dose (2.5 mg/kg feed)			Mid dose (6.89 mg/kg feed)		High dose (23.69 mg/kg feed)	
17	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.017 (0.017, 0.019, 0.014)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.079 (0.077, 0.090, 0.069)	0.014 (0.019, 0.012, 0.010)
21	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.019 (0.019, 0.023, 0.016)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.076 (0.079, 0.092, 0.058)	0.014 (0.020, 0.012, 0.011)
24	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.023 (0.024, 0.024, 0.022)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.089 (0.097, 0.099, 0.071)	0.018 (0.023, 0.014, 0.015)
27	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.022 (0.022, 0.023, 0.020)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.077 (0.090, 0.088, 0.054)	0.013 (0.018, 0.011, 0.010)
29	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.021 (0.020, 0.023, 0.021)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.086 (0.101, 0.088, 0.068)	0.013 (0.018, < 0.01, < 0.01)

In liver, TFNA-AM and OH-TFNA-AM were detected in the mid and high dose groups above the LOQ using two different analytical methods (FMC-P-3580 / RCC 844743) with different LOQ (0.025/0.01 mg/kg). TFNA-AM levels increased from less than LOQ in the low dose group to 0.039/0.015 mg/kg in the mid dose group and 0.113/0.053 mg/kg in the high dose group. OH-TFNA-AM levels increased from levels below LOQ (0.025/0.01 mg/kg) in the low dose group to levels slightly above the LOQ (< 0.025/0.010 mg/kg) in the mid dose group and 0.030/0.037 mg/kg in the high dose group.

In kidney, TFNA and TFNA-AM were detected in the medium and high dose groups above the LOQ using the same analytical methods as those used for kidney. OH-TFNA-AM and TFNG were only detected above the LOQ (0.025/0.01 mg/kg) in the high dose group. TFNA levels increased from levels below LOQ in the low dose group to 0.043/0.038 mg/kg in the mid dose group and 0.142/0.135 mg/kg in the high dose group. TFNA-AM levels increased from levels below LOQ in the low dose group to 0.031/0.023 mg/kg in the mid dose group and 0.105/0.088 mg/kg in the high dose group. OH-TFNA-AM levels increased from levels below LOQ in the low and mid dose group to 0.025/0.027 mg/kg in the high dose group. TFNG levels increased from levels below LOQ in the low and mid dose group to 0.010 mg/kg in the high dose group.

Table 95 Residues in liver and kidney following 28 days oral administration to dairy cows

	Liver				Kidney							
	Solvent extraction		Hydrolysis		Solvent extraction				Hydrolysis			
Dose (mg/kg feed)	TFNA-AM	OH-TFNA-AM	TFNA-AM	OH-TFNA-AM	TFNA-AM	OH-TFNA-AM	TFNG	TFNA	TFNA-AM	OH-TFNA-AM	TFNG	TFNA
2.5	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.025 (< 0.025, < 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025, < 0.025)	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.016 (0.014, 0.019, 0.014)	< 0.025 (< 0.025, < 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025, < 0.025)
6.89	0.015 (0.010, 0.019, 0.015)	0.010 (< 0.01, < 0.01, 0.011)	0.039 (0.042, 0.040, 0.034)	< 0.025 (< 0.025, < 0.025, < 0.025)	0.023 (0.020, 0.024, 0.025)	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.038 (0.032, 0.041, 0.041)	0.031 (0.027, 0.034, 0.032)	< 0.025 (< 0.025, < 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025, < 0.025)	0.043 (0.039, 0.047, 0.045)

	Liver				Kidney							
	Solvent extraction		Hydrolysis		Solvent extraction				Hydrolysis			
23.69	0.053 (0.056, 0.052, 0.051)	0.037 (0.051, 0.034, 0.026)	0.113 (0.124, 0.124, 0.091)	0.028 (0.035, < 0.025, < 0.025)	0.088 (0.112, 0.083, 0.070)	0.027 (0.038, 0.021, 0.022)	0.01 (0.01, < 0.01, < 0.01)	0.135 (0.166, 0.108, 0.132)	0.105 (0.124, 0.092, 0.099)	0..025 (0.025, < 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025, < 0.025)	0.142 (0.173, 0.107, 0.146)

In muscle, only TFNA-AM was found. The level increased from below LOQ (0.025 mg/kg) in the low dose group to 0.027 mg/kg in the mid dose group and 0.088 mg/kg in the high dose group.

Similarly, only TFNA-AM was measurable in fat and only at the high dose level (0.015 mg/kg).

Table 96 TFNA-AM residues in fat and muscle following 28 days oral administration to dairy cows

	TFNA-AM (mg/kg)	
Dose (mg/kg feed)	Fat	Muscle
2.5	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.025 (< 0.025, < 0.025, < 0.025)
6.89	< 0.01 (< 0.01, < 0.01, < 0.01)	0.027 (< 0.025, 0.027, 0.030)
23.69	0.015 (0.021, 0.013, 0.011)	0.057 (0.010, 0.088, 0.072)

Poultry

Flonicamid and TFNG were dosed in a 1:1 mixture to 50, 22 week-old, laying hens (white leghorn hybrids) weighing on average 1.54 kg with an egg production of at least 0.8 eggs per day. The hens were randomly assigned to five groups, one of which served as control group. Each group was separated into three subgroups of three to four animals. After the acclimation period, the test substance was orally administered, once daily in the afternoon by means of gelatin capsules (after egg sampling). for 28 days. Based on the actual average daily feed intake of 0.108–0.116 kg/hen during the 4-week acclimation period, the actual dose levels were equivalent to average potential concentrations in the feed of 0.26, 2.51, 7.47 and 25.83 mg flonicamid/TFNG per kg feed.

Eggs were collected once daily and pooled per subgroup of three or four hens, resulting in three unique samples of eggs for each dose level. The egg pools were stored at –20 °C until analysis. The animals were sacrificed for tissue sampling the day after the last administration, 24 hours after the last dosing. Liver fat (composite of skin fat) and muscle were excised. Tissue samples were rinsed, weighed and labelled. Tissues were pooled per subgroup of three or four hens, homogenised and stored deep-frozen until analysis. Egg and tissue samples were stored for greater than 30 days. Freezer storage stability studies, conducted concurrently with the feeding studies, demonstrated that flonicamid, TFNA, TFNA-AM, OH-TFNA-AM and TFNG were stable for 8–10 months in eggs and tissues.

All samples were analysed for residues of flonicamid, TFNA, TFNA-AM, OH-TFNA-AM and TFNG using a validated analytical method. The samples were homogenised, extracted and the supernatant was purified by means of gel permeation chromatography. The concentration of flonicamid and its metabolites in the purified extracts were determined by MS/MS detection using HPLC for separation.

In eggs, no quantifiable (< LOQ) residues of TFNA, OH-TFNA-AM and TFNG were detected in any test group. For flonicamid, the average residues increased from < LOQ in the very low and low dose groups to 0.02 mg/kg in the mid dose group and to 0.08 mg/kg in the high

dose group. TFNA-AM average residues increased from < LOQ in the very low and low dose groups, to 0.27 mg/kg in the mid dose group and 0.95 mg/kg in the high dose group.

Table 97 Residues in eggs following 28 days oral administration to laying hens

Day	Very Low Dose (0.26 mg/kg feed)		Day	Low Dose (2.51 mg/kg feed)		Day	Mid Dose (7.47 mg/kg feed)		Day	High Dose (25.83 mg/kg feed)	
	Flonicamid (mg/kg)	TFNA-AM (mg/kg)		Flonicamid (mg/kg)	TFNA-AM (mg/kg)		Flonicamid (mg/kg)	TFNA-AM (mg/kg)		Flonicamid (mg/kg)	TFNA-AM (mg/kg)
1	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	1	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	1	< 0.01 (< 0.01, < 0.01, < 0.01)	0.014 (0.022, < 0.01, < 0.01)	1	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)
2	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	2	< 0.01 (< 0.01, < 0.01, < 0.01)	0.034 (0.038, 0.028, 0.038)	2	0.013 (< 0.01, 0.013, 0.013)	0.136 (0.165, 0.124, 0.119)	2	0.052 (0.063, 0.041, 0.051)	0.450 (0.429, 0.430, 0.492)
3	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	3	< 0.01 (< 0.01, < 0.01, < 0.01)	0.053 (0.054, 0.044, 0.061)	3	0.014 (0.010, 0.016, 0.014)	0.190 (0.220, 0.180, 0.169)	3	0.056 (0.064, 0.052, 0.055)	0.691 (0.746, 0.564, 0.764)
4	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	4	< 0.01 (< 0.01, < 0.01, < 0.01)	0.083 (0.075, 0.088, 0.087)	4	0.017 (0.012, 0.024, 0.014)	0.260 (0.274, 0.295, 0.210)	4	0.067 (0.071, 0.076, 0.055)	0.837 (0.843, 0.776, 0.893)
5	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	5	< 0.01 (< 0.01, < 0.01, < 0.01)	0.078 (0.071, 0.077, 0.086)	5	0.014 (< 0.01, 0.014, 0.014)	0.263 (0.281, 0.268, 0.240)	5	0.056 (0.062, 0.064, 0.044)	0.895 (0.950, 0.791, 0.945)
6	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	6	< 0.01 (< 0.01, < 0.01, < 0.01)	0.073 (0.070, 0.058, 0.092)	6	0.012 (0.011, 0.014, 0.011)	0.250 (0.254, 0.286, 0.211)	6	0.046 (0.050, 0.047, 0.043)	1.007 (1.052, 0.968, 1.001)
7	< 0.01 (< 0.01, < 0.01, < 0.01)	0.010 (0.011, < 0.01, < 0.01)	7	< 0.01 (< 0.01, < 0.01, < 0.01)	0.079 (0.074, 0.065, 0.099)	7	0.019 (0.018, 0.019, 0.019)	0.271 (0296, 0.309, 0.208)	7	0.058 (0.064, 0.057, 0.054)	0.874 (0.773, 0.982, 0.867)
8	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	8	< 0.01 (< 0.01, < 0.01, < 0.01)	0.079 (0.070, 0.062, 0.106)	8	0.016 (0.015, 0.015, 0.016)	0.244 (0.252, 0.258, 0.223)	8	0.068 (0.072, 0.058, 0.074)	0.820 (0.907, 0.718, 0.836)
10	< 0.01 (< 0.01, < 0.01, < 0.01)	0.010 (0.011, < 0.01, < 0.01)	9	< 0.01 (< 0.01, < 0.01, < 0.01)	0.079 (0.080, 0.063, 0.093)	10	0.014 (0.014, 0.014, 0.013)	0.254 (0.306, 0.264, 0.193)	9	0.062 (0.061, 0.052, 0.073)	0.963 (1.110, 0.866, 0.912)
14	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	13	< 0.01 (< 0.01, < 0.01, < 0.01)	0.079 (0.073, 0.059, 0.102)	14	0.012 (0.012, 0.015, 0.011)	0.246 (0.266, 0.296, 0.176)	13	0.048 (0.054, 0.054, 0.037)	0.985 (1.010, 0.831, 1.114)
17	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	16	< 0.01 (< 0.01, < 0.01, < 0.01)	0.084 (0.073, 0.059, 0.102)	17	0.017 (0.012, 0.015, 0.011)	0.311 (0.340, 0.357, 0.236)	16	0.075 (0.054, 0.054, 0.037)	0.865 (1.010, 0.831, 1.114)
21	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	20	< 0.01 (< 0.01, < 0.01, < 0.01)	0.091 (0.067, 0.087, 0.118)	21	0.013 (0.012, 0.015, 0.012)	0.295 (0.312, 0.370, 0.202)	20	0.050 (0.048, 0.051, 0.051)	1.023 (0.956, 0.935, 1.177)
24	< 0.01 (< 0.01, < 0.01, < 0.01)	0.0115 (0.014, 0.010, 0.010)	23	< 0.01 (< 0.01, < 0.01, < 0.01)	0.087 (0.096, 0.076, 0.088)	24	0.014 (< 0.01, 0.015, 0.012)	0.226 (0.196, 0.276, 0.206)	23	0.053 (0.053, 0.062, 0.045)	1.041 (1.071, 0.961, 1.090)
27	< 0.01 (< 0.01, < 0.01,	< 0.01 (< 0.01, < 0.01,	26	< 0.01 (< 0.01, < 0.01,	0.098 (0.124, 0.082,	27	0.013 (0.011, 0.016,	0.310 (0.330, 0.349,	26	0.052 (0.058, 0.054,	1.119 (1.076, 1.068,

Day	Very Low Dose (0.26 mg/kg feed)		Day	Low Dose (2.51 mg/kg feed)		Day	Mid Dose (7.47 mg/kg feed)		Day	High Dose (25.83 mg/kg feed)	
	Flonicamid (mg/kg)	TFNA-AM (mg/kg)		Flonicamid (mg/kg)	TFNA-AM (mg/kg)		Flonicamid (mg/kg)	TFNA-AM (mg/kg)		Flonicamid (mg/kg)	TFNA-AM (mg/kg)
	< 0.01)	< 0.01)		< 0.01)	0.089)		0.012)	0.252)		0.044)	1.214)
28	< 0.01 (< 0.01, < 0.01, < 0.01)	0.0083 (< 0.01, < 0.01, < 0.01)	28	< 0.01 (< 0.01, < 0.01, < 0.01)	0.080 (0.078, 0.064, 0.099)	28	0.017 (0.018, 0.018, 0.016)	0.321 (0.333, 0.365, 0.265)	28	0.074 (0.093, 0.081, 0.048)	0.993 (1.067, 0.891, 1.020)

No quantifiable residues (< LOQ) of flonicamid, TFNA and TFNG were found in muscle in any treatment group. No quantifiable residues (< LOQ) of TFNA-AM was measurable in muscle at the very low dose group, but there appeared to be a dose response relationship at all other dose levels; 0.049 mg/kg in the low dose group, 0.168 mg/kg in the mid dose group and 0.654 mg/kg in the high dose group. OH-TFNA-AM was measurable only at the high dose level (0.014 mg/kg).

In liver and fat, no quantifiable residues (< LOQ) of flonicamid, TFNA, OH-TFNA-AM and TFNG were found at any dosing level. For liver, TFNA-AM residues increased from < 0.01 mg/kg (very low) to 0.054 mg/kg (low) to 0.166 mg/kg (mid) and 0.706 mg/kg (high) while for fat, TFNA-AM residues increased from 0.01 mg/kg (very low) to 0.022 mg/kg (low) to 0.062 mg/kg (mid) and 0.286 mg/kg (high).

Table 98 Residues in muscle, liver and fat following 28 days oral administration to laying hens

Dose Level (mg/kg feed)	Muscle		Liver	Fat		
	OH-TFNA-AM	TFNA-AM	TFNA-AM	OH-TFNA-AM	TFNA-AM	TFNA
0.259	< 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)
2.51	< 0.01 (< 0.01, < 0.01, < 0.01)	0.049 (0.050, 0.035, 0.062)	0.054 (0.057, 0.040, 0.065)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.022 (0.018, 0.016, 0.031)	< 0.01 (< 0.01, < 0.01, < 0.01)
7.47	< 0.01 (< 0.01, < 0.01, < 0.01)	0.168 (0.169, 0.187, 0.149)	0.166 (0.178, 0.187, 0.134)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.062 (0.061, 0.080, 0.046)	< 0.01 (< 0.01, < 0.01, < 0.01)
29.83	0.014 (0.014, 0.013, 0.016)	0.654 (0.654, 0.590, 0.718)	0.706 (0.786, 0.606, 0.671)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.286 (0.353, 0.265, 0.242)	< 0.01 (< 0.01, < 0.01, < 0.01)

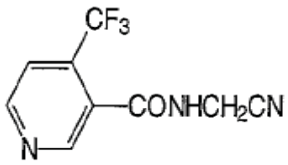
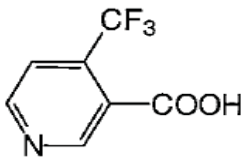
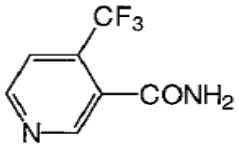
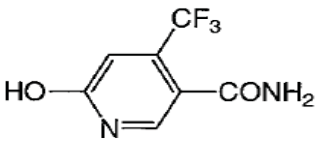
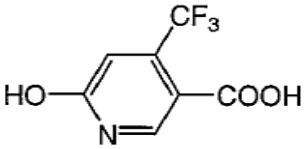
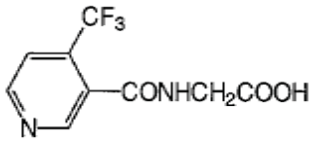
APPRAISAL

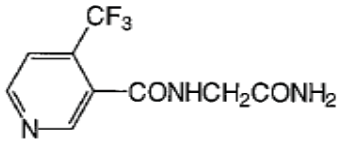
Flonicamid is a new insecticide for control of aphids and other sucking insects. It belongs to a new class of chemistry known as pyridinecarboxamide. At the Forty-sixth Session of the CCPR, flonicamid was scheduled for evaluation, for both toxicology and residues, as a new compound by the 2015 JMPR.

The Meeting received information on the metabolism of flonicamid in peaches, bell peppers, potatoes, wheat, lactating goats, laying hens and rotational crops, environmental fate, methods of residue analysis, freezer storage stability, GAP, supervised residue trials on various fruits, vegetables, tree nuts, oil seeds, dried hops, mint and tea, processing studies as well as livestock feeding studies.

In this document, the code names and chemical structures of the metabolites were as follows:

Flonicamid is *N*-cyanomethyl-4-(trifluoromethyl)nicotinamide (IUPAC).

Code Name	Structure	Chemical Name
Flonicamid IKI-220		N-cyanomethyl-4-(trifluoromethyl)nicotinamide
TFNA		4-trifluoromethylnicotinic acid
TFNA-AM		4-trifluoromethylnicotinamide
OH-TFNA-AM		6-hydroxy-4-trifluoromethylnicotinamide
TFNA-OH		6-hydroxy-4-trifluoromethylnicotinic acid
TFNG		N-(4-trifluoromethylnicotinoyl)glycine

TFNG-AM		N-(4-trifluoromethylnicotinoyl)glycinamide

Environmental fate in soil

The FAO Manual (FAO, 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 flonicamid, supervised residue trials were received for foliar spray on permanent crops and on annual crops. Therefore, according to the FAO manual, only studies on aerobic degradation, photolysis and rotational crops (confined, field) were evaluated.

Degradation

The route of degradation of [^{14}C]flonicamid in soil under aerobic conditions was investigated in a biologically active loamy sand soil collected from Madison, Ohio, USA and stored in a greenhouse. Flonicamid rapidly declined from 99.3% of the applied radioactivity (AR) at Day 0 to 2.3% by Day 30, resulting in a DT_{50} of 1 day and a DT_{90} of 3.4 days. TFNA and TFNA-OH were major components of the residue with TFNG, TFNG-AM and TFNA-AM all identified as minor metabolites.

The rate of aerobic degradation of [^{14}C]flonicamid, radiolabelled at the 3 position of the pyridine was investigated in three biologically active soils (sandy loam and sand at 10 °C and/or 20 °C)

For the soils incubated at 20 °C the DT_{50} and DT_{90} values for flonicamid ranged from 0.7 to 1.8 days and 2.3 to 6.0 days, respectively. For the soil incubated at 10 °C, the DT_{50} and DT_{90} values for flonicamid were 2.4 days and 7.9 days, respectively. TFNA, TFNA-OH and TFNG-AM were the major degradates in all soils over the course of the study. Minor degradates TFNG and TFNA-AM were detected at all sampling points over the course of the study. All of the degradates were metabolised and mineralised to carbon dioxide and immobilised as soil-bound residue.

Photolysis

The photochemical degradation of [pyridyl-^{14}C]flonicamid was investigated in a loamy sand under laboratory conditions.

[^{14}C]Flonicamid decreased to 60% of the applied radioactivity (AR) after 15 days of continuous illumination, resulting in a DT_{50} of 22 days. Concurrently, the major metabolite TFNG-AM was detected in Day 1 sample extracts and increased by Day 15. TFNA-AM and TFNG were also detected as minor metabolites in the illuminated soils, reaching maximum concentrations of 5% AR (Day 11 and Day 15) and 2% AR (Day 15), respectively.

In summary, based on the results of the environmental fate studies, flonicamid as well as its metabolites are likely to readily degrade and not persist in aerobic soil environments.

Plant metabolism

The metabolism of flonicamid was studied in peaches, bell peppers, potatoes and wheat.

Peach

Flonicamid, radiolabelled at the 3 position of the pyridine ring and formulated as a wettable granule, was applied twice to peach trees grown outdoor, with a 14-day re-treatment interval, at rates of 100 g ai/ha (low rate) or 500 g ai/ha (high rate) per application. Mature fruits and leaves were

harvested 21 days after the last treatment (DALA). Overall total radioactive residues (TRRs) in fruits at the low rate and the high rate were 0.10 mg eq/kg and 0.32 mg eq/kg, respectively, while in the leaves, TRRs followed the same trend, where residues were lower at the lower application rate (6.2 mg eq/kg) compared to those at the higher treatment rate (24 mg eq/kg).

The peaches were subjected to a surface wash using deionised water which removed very little radioactivity (≤ 15% TRRs), evidence of limited penetration. The majority of the TRRs were partitioned into the juice fraction (64–73% of the TRR) and to a lesser extent into the pulp (21% TRRs). While juice was not further extracted with organic solvents, extraction of the pulp with acetonitrile:water:phosphoric acid recovered 92% TRR. At both treatment rates, flonicamid (30–60% TRRs) and TFNA (17–49% TRRs) were the predominant residues in juice and pulp. All other metabolites, TFNG, TFNG-AM and TFNA- AM were ≤ 6% TRRs.

Bell pepper

A single application of flonicamid, radiolabelled at the 3 position of the pyridine ring, formulated as a 50% wettable granule formulation, was made to greenhouse grown bell pepper plants at 100 g ai/ha. Fruits and leaves were harvested 7 days and 14 days after treatment (DAT).

The TRRs in fruits decreased insignificantly from 0.17 mg eq/kg (7 DAT) to 0.11 mg eq/kg (14 DAT) while TRRs in leaves decreased from 2.2 mg eq/kg, when harvested 7 days after treatment to 1.4 mg eq/kg at 14 DAT.

The %TRR in the methanol:water surface wash for both leaves and fruits decreased as the corresponding extracted TRRs (61–81% TRRs) and those in the post-extraction solids (PES) increased with increasing DAT. This trend demonstrated the penetration of the radioactivity from the surface into the leaves and fruits.

Flonicamid and TFNG were the predominant residues in leaves (47–74% TRRs and 12–28% TRRs, respectively) while only flonicamid was the predominant residue in fruits (77–91% TRRs) at both harvest intervals. All identified metabolites (TFNA, TFNA-AM and TFNG-AM) were either not detected or were ≤ 12% TRRs.

Potato

Potato plants maintained outdoor were treated, either at the lower rate of 100 g ai/ha or the higher rate of 500 g ai/ha, with flonicamid radiolabelled at the 3 position of the pyridine ring and formulated as a 50% wettable powder. Both treatments were repeated at a two-week interval and potato tubers and foliage were harvested 14 days after the last application.

Overall TRRs in tubers at the low rate and the high rate were 0.11 mg eq/kg and 0.20 mg eq/kg, respectively, while those in mature foliage were higher than those in tubers; 1.5 mg eq/kg at the low rate and 7.7 mg eq/kg at the high rate. Considering the applications were made to the foliage of the potato plants, the presence of measurable TRRs in the tubers is evidence of translocation of the radioactivity from the foliage to the tubers. Furthermore, while the TRRs in tubers and foliage increased with increased application, the distribution of TRRs was relatively the same irrespective of the treatment rate.

Extraction of the potato tubers with ACN and ACN:water recovered up to 93% TRRs while extraction of potato foliage with ACN:water:acetic acid recovered up to 90% TRRs. Analysis of each of the extracted fractions of potato tubers and foliage from the low and high rate demonstrated that the major components of the residue, TFNA and TFNG, accounted for a significant portion of the TRRs. Moreover, TFNA accounted for 34% TRRs in tubers and 12–17% TRRs in foliage at both rates while TFNG accounted for 25–39% TRRs in tubers and 28–36% TRRs in leaves at both rates. The parent, flonicamid, accounted for 6–19% TRRs in potato tubers and 10–25% TRRs in foliage. Each of the other identified metabolites (TFNA conjugate, TFNG-AM, TFNA-AM, PM-1a, PM-1b and PM-3a) accounted for < 10% TRRs in tubers and in foliage. Overall, the general metabolic profile in foliage was similar to that in tubers.

Spring wheat

Spring wheat plants grown outdoor were treated with flonicamid, radiolabelled at the 3 position of the pyridine ring and formulated as a wettable powder, at a single application rate of 100 g ai/ha or 2 applications at 100 g ai/ha with a re-treatment interval of 7 days. Forage and hay were harvested 14 days and 42 days, respectively, after the single application. Approximately 95 days after the second treatment (200 g ai/ha/season), mature plants were harvested and separated into straw, chaff and grain.

Overall residues were lower in the wheat grain sample (2.6 mg eq/kg) than the straw (5.6 mg eq/kg) or chaff (6.6 mg eq/kg). The TRRs in the chaff were higher compared to straw potentially because of tissue size differences (higher surface area to weight ratio) assuming a uniform application. Further to this, considering the timing of application of the test material and the measurable TRRs in grain, chaff and straw at maturity, there appears to have been translocation of the radioactivity from the site of application to the mature plant parts.

Only forage and hay were analysed to elucidate the nature of the flonicamid residues. Extraction of these matrices with ACN:water:acetic acid recovered 96% TRRs. The analysis of forage and hay samples demonstrated that the nature and distribution of metabolites were similar in both matrices. The parent compound, flonicamid, and the TFNG metabolite represented the majority of the TRRs in both the forage (flonicamid: 43% TRRs; TFNG: 33% TRRs) and hay (flonicamid: 22%TRRs; TFNG: 53% TRRs). Metabolites TFNG-AM, TFNA and TFNA-AM were present at ≤ 13% TRRs.

In a second spring wheat metabolism study, plants grown outdoor were treated with flonicamid, radiolabelled at the 3 position of the pyridine ring and formulated as a wettable granule, at rates equivalent to 100 g ai/ha or 500 g ai/ha. The wheat plants were harvested 21 DAT and separated into straw (leaves and stem), chaff and grain (with hulls attached).

The TRRs in wheat straw, chaff and grain increased with increased application rate with the highest TRRs observed in chaff, followed by straw and grain, irrespective of the application rate. The distribution of TRRs was relatively the same irrespective of the treatment rate.

Similar to the first wheat metabolism study, extraction with ACN:water:acetic acid recovered 80–94%TRRs with flonicamid (24–50%TRRs) and TFNG (17–44% TRRs) representing the predominant residues at both treatment rates. All identified metabolites (TFNA, TFNG-AM, TFNA-AM and N-oxide TFNA AM) were either not detected or were each ≤ 10% TRR.

In summary, the Meeting determined that the degree of metabolism in all crops tested, following foliar application, was qualitatively similar, with the parent compound as the predominant residue. The major metabolic pathway of flonicamid in plants involved hydrolysis of the cyano group and the amide groups.

Rotational crops

In the confined rotational crop study, flonicamid, radiolabelled at the 3 position of the pyridine ring and formulated as a wettable granule was applied twice to loamy sand soil at a rate equivalent to 100 g ai/ha at an interval of two weeks. After the appropriate plant-back intervals (PBIs) of 30, 120 or 360 days, the rotational crops, representative of the root vegetable (carrot), small grain (wheat), and leafy vegetable (lettuce) crop groups, were planted.

TRRs in all raw agricultural commodities (RACs) declined with prolonged PBIs such that, at the 120-day PBI, no further characterization/identification of the TRRs was performed for immature and mature lettuce and mature carrot roots due to the low total radioactivity. Further to this, at the 360-day PBI, none of the TRRs from any of the crop parts were further subjected to characterization/identification as these were also too low.

Analysis of the harvested crop samples demonstrated very little uptake of ^{14}C-residues. Of the radioactivity taken up by plants, only limited amounts of flonicamid were detected (≤ 13% TRRs). TFNG and TFNG-AM were identified at > 10% TRRs in most RACs. In addition to

TFNG, other identified metabolites accounting for > 10% TRRs in wheat matrices and mature carrot root included TFNA-AM and TFNA-OH.

Conversely, in the field accumulation study, no quantifiable residues of flonicamid or its metabolites TFNG, TFNA, and TFNA-AM were detected in wheat (forage, straw and grain) and turnip (tops and roots) planted at either 30 or 60 days after the last application of flonicamid to the primary crop, cotton.

Based on the findings of the field crop rotation studies, the Meeting concluded that the uptake of quantifiable residues of flonicamid or its associated metabolites in secondary crops is unlikely.

Animal metabolism

Metabolism studies in rats reviewed by the 2015 JMPR and conducted using [^{14}C]flonicamid labelled at the 3-nicotinamide position, indicated that flonicamid was rapidly absorbed and quickly excreted. The majority of administered radioactivity was excreted in the urine and within the first 24 hours. There was no evidence of bioaccumulation following repeat doses. Distribution into the tissues was extensive with levels similar to blood concentrations; however, slightly higher concentrations were seen in the liver, kidneys, adrenals, thyroid and ovaries following single or repeat dosing and in the lungs following repeat dosing in males.

The main urinary residue was unchanged parent, followed by TFNA-AM, which was also the predominant metabolite in the faeces and bile. Other metabolites were TFNA in the faeces of low-dose animals, TFNA-AM N-oxide conjugate in the high-dose animals, TFNG-AM in the bile of high-dose animals and TFNG and TFNA-AM in the liver.

Metabolism studies were conducted in lactating goats where they were dosed orally once daily for 5 consecutive days with 3-pyridine-^{14}C-labelled flonicamid at a dose level equivalent to 10 ppm in feed. The major route of elimination of the radioactivity was via the urine which accounted for 49% of the administered dose (AD), while faeces accounted for up to 21% of the AD and milk accounted for 1% of the AD. Overall, the tissue burden was low, accounting for < 10% of the AD. The TRRs were highest in liver (1.2 mg eq/kg) followed by kidney (0.70 mg eq/kg), muscle (0.30–0.40 mg eq/kg) and fat (0.05–0.14 mg eq/kg).

Extraction of milk, using ethanol and ethanol:water recovered 97% TRRs and extraction of tissues and organs using ACN and ACN:water containing 1% acetic acid recovered greater than 42% TRRs. Flonicamid was rapidly metabolised in lactating goats, representing less than 6% TRRs in tissues and organs. TFNA-AM was the major component of the residue in organs (29% TRRs in liver, 31–41% TRRs in kidney), tissues (74% TRRs in fat, 42–50% TRRs in muscle) and milk (97% TRRs). The minor metabolites TFNA and 6-OH-TFNA-AM each accounted for ≤ 7% TRRs in liver, kidney, muscle and milk.

Leghorn laying hens were dosed orally once daily for 5 consecutive days with 3-pyridine-^{14}C-labelled flonicamid at a dose level equivalent to 10 ppm in feed. Approximately 91% of AD including 6% of AD from the gastrointestinal tract and its contents was recovered. Most of the AD (72%) was excreta-related. TRRs in egg white and egg yolk accounted for about 2.4% of AD (1.8% AD in egg white plus 0.6% AD in yolk). The tissue burden was low (< 6% of the AD) with highest concentrations of ^{14}C-residues found in kidney (1.4 mg eq/kg) followed by liver (1.2 mg eq/kg), muscle (evenly distributed between breast and thigh muscle; 1.0 mg eq/kg each), skin (0.70 mg eq/kg) and fat (0.15 mg eq/kg).

Extraction of eggs, tissues and organs with ACN and ACN:water containing 1% acetic acid recovered more than 81% TRR. Flonicamid accounted for only a very small percentage of the TRRs in eggs (2–4% TRRs), tissues (< 1% TRRs) and organs (< 0.5% TRRs). TFNA-AM was the predominant component of the residue in egg whites and egg yolks (≤ 96%TRRs), liver (93%TRRs), kidney (76%TRRs) and tissues (97%TRRs in both breast muscle and thigh muscle, 96%TRRs in skin and 95%TRRs in fat). Other metabolites identified in organs and tissues were

OH-TFNA-AM and TFNG-AM, however, neither of these accounted for greater than 5% of TRR.

The Meeting concluded that, in all species investigated, the total administered radioactivity was quickly and almost completely eliminated in excreta. The metabolic profiles differed quantitatively between the species, but qualitatively there were no major differences. The routes and products of metabolism in animals were consistent across the studies resulting from the hydrolysis of the cyano function to the amide function as well as ring hydroxylation. Moreover, TFNA-AM was the major component of the residue in all tissues, organs, milk and eggs of livestock animals.

While the overall metabolism in plants, livestock and rats is similar, the metabolism of flonicamid in animals is more extensive with hydrolysis of flonicamid to the major amide metabolite TFNA-AM.

Methods of analysis

The Meeting received descriptions and validation data for analytical methods for residues of flonicamid and its relevant metabolites TFNA-AM, TFNA and TFNG in plant commodities and for flonicamid, TFNA-AM, TFNA, TFNG and OH-TFNA-AM in animal commodities. Residue analytical methods rely on LC/MS-MS. Typical limits of quantitation (LOQs) achieved for plant commodities fell in the range of 0.01–0.02 mg/kg for each analyte. The LOQs for milk and animal products (liver, kidney, muscle and eggs) were 0.01 mg/kg for each analyte. Methods were successfully subjected to independent laboratory validation.

The acid version (addition of 1% formic acid to the acetonitrile extraction solvent) of the QuEChERS multi residue LC-MS/MS method was used for flonicamid, TFNA, TFNG and TFNA-AM in plant matrices with LOQs of 0.01 mg/kg for each analyte.

The Meeting determined that suitable methods are available for the analysis of flonicamid and its relevant metabolites in plant and animal commodities.

Stability of residues in stored analytical samples

The Meeting received storage stability studies under freezer conditions at –17 °C for flonicamid and its relevant metabolites TFNA-AM, TFNA and TFNG for the duration of the storage of 18 to 23 months in a wide range of raw and processed crop matrices, including high-water, high-starch and high-oil crops. The Meeting concluded that residues of flonicamid, TFNA-AM, TFNA and TFNG are stable for at least 18 months. Freezer storage stability studies were also conducted concurrently with several of the crop field trials, demonstrating similar results.

All milk samples from the feeding studies were frozen at –20 ºC and analysed within 30 days after sampling. Therefore, storage stability data are not necessary. In contrast, all tissue samples were analysed within 12 months of collection. Freezer storage stability studies, conducted concurrently with the feeding studies, demonstrated that flonicamid, TFNA, TFNA-AM, OH-TFNA-AM and TFNG were stable for 374 days in all tissues except fat. For fat, flonicamid and its metabolites were demonstrated to be stable for 315 days.

Definition of the residue

In primary crops, the parent compound represented the majority of the residue accounting for up to 61% TRRs in peach fruits, 91%TRRs in bell pepper fruits, 19% TRRs in potato and up to 50% TRRs in wheat forage, hay, straw, chaff and grain. Metabolites TFNA and TFNG were identified as predominant metabolites (> 10%TRRs) in all crop commodities. In the crop field trials, residues of TFNA and TFNG were measurable in all crops, the magnitude of which was crop-dependent. However, both the TFNA and TFNG were seen in the rat metabolism study and considered to be up to 10-fold less toxic than the parent flonicamid based on toxicity studies reviewed by the 2015 WHO.

In the field accumulation study no measurable residues of parent or any of its associated metabolites were observed in secondary crops.

In light of the above, the Meeting decided to define the residue for enforcement/monitoring and for risk assessment for plant commodities as parent only.

In the farm animal metabolism studies, the parent, flonicamid, was rapidly degraded in ruminants and poultry, accounting for ≤ 6% TRRs in all tissues, milk and eggs. Conversely, the metabolite TFNA-AM accounted for the majority of the radioactivity in goat tissues (29–74% TRRs) and milk (92–97%TRRs) and laying hen tissues (76–97% TRRs) and eggs (ca. 95%TRRs).

Similar findings were observed in the livestock feeding studies whereby flonicamid was present at very low levels in all animal commodities with the metabolite TFNA-AM representing the majority of the residues in tissues, milk and eggs. Therefore, TFNA-AM will be included in the residue definition for enforcement as a marker compound. Since the method of analysis is capable of analysing both flonicamid and TFNA-AM, the Meeting agreed to define the residue for enforcement/monitoring as flonicamid and TFNA-AM.

The log K_{ow} for flonicamid is 0.3. In the metabolism studies there was no evidence of the parent compound and TFNA-AM partitioning into fatty matrices (fat, milk and egg yolks) as the total residues were present at comparable concentrations in all livestock matrices. In the dairy cattle and poultry feeding studies, there was no evidence of the total residues of flonicamid and TFNA-AM sequestering into milk, eggs or fat. Therefore, the Meeting did not consider the residue fat soluble.

As TFNA-AM was the major component of the residue in all animal matrices in both the metabolism and feeding studies, the Meeting decided to define the residue for dietary risk assessment for animal commodities as parent and TFNA-AM.

Based on the above, the Meeting recommended that 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 plant commodities: *Flonicamid*

Definition of the residue for compliance with MRL and estimation of dietary intake for animal commodities: Flonicamid and the metabolite TFNA-AM, expressed as parent.

The residue is not fat soluble.

Results of supervised residue trials on crops

Pome fruits

Results from supervised field trials on apples and pears conducted in the US were provided to the Meeting, including apple and pear data from Australia.

A total of 16 independent supervised trials were conducted in the US on apples (12) and pears (4). The GAP in the US for pome fruits allows three applications at a maximum rate of 0.1 kg ai/ha with a PHI of 21 days.

Flonicamid residues from 12 apple trials matching the US GAP were: 0.02, 0.04 (3), 0.05 (4), 0.06, 0.07, 0.10 and 0.11 mg/kg.

Flonicamid residues from four pear trials matching the US GAP were: 0.01 (3) and 0.02 mg/kg.

A total of seven independent supervised trials were also conducted on apples in Australia according to the Australian GAP which allows three applications at a maximum rate of 0.01 kg ai/hL with a PHI of 21 days. Nine supervised trials were conducted on pears in Australia, however, in the absence of an Australian GAP, these trials were not considered.

Flonicamid residues from seven apple trials matching the Australia GAP were 0.09, 0.12 (2), 0.13, 0.15, 0.22 and 0.24 mg/kg.

The Meeting noted that in the US a group GAP for pome fruit exists and decided to explore the possibility of setting a group maximum residue level. As the supervised trials on apples conducted in Australia in accordance with the Australian GAP lead to the higher residues, the Meeting recommended that the group maximum residue level be based on the dataset from Australia.

Based on the Australian residue data for apples, the Meeting estimated a maximum residue level for pome fruits of 0.8 mg/kg and an STMR of 0.13 mg/kg.

Stone fruits

Results from supervised field trials on peaches, cherries and plums conducted in the US were provided to the Meeting.

A total of 19 independent supervised trials were conducted in the US on peaches (8), cherries (6) and plums (5) according to the US GAP on stone fruits which allows three applications at a maximum rate of 0.1 kg ai/ha with a 14-day PHI.

Residues of flonicamid from eight peach trials matching the US GAP for stone fruits were: 0.09 (2), 0.10, 0.13, 0.15, 0.22 (2) and 0.42 mg/kg.

Residues of flonicamid from six cherry trials matching the US GAP for stone fruits were: 0.26, 0.27 (2), 0.28 (2) and 0.36 mg/kg.

Residues of flonicamid from five plum trials matching the GAP for stone fruits were: 0.01, 0.02, 0.03 and 0.04 (2) mg/kg.

The Meeting noted that in the US a group GAP for stone fruits exists and decided to explore the possibility of setting a group maximum residue level. Since median residues among the representative commodities were not within a 5-fold range (0.14 mg/kg vs. 0.28 mg/kg vs. 0.03 mg/kg), the Meeting decided to estimate maximum residue levels for each subgroup based on the individual dataset for each representative commodity.

The Meeting estimated a maximum residue level of 0.9 mg/kg and an STMR of 0.28 mg/kg for cherries subgroup.

The Meeting estimated a maximum residue level of 0.7 mg/kg and an STMR of 0.14 mg/kg for peaches subgroup.

The Meeting estimated a maximum residue level of 0.1 mg/kg and an STMR of 0.03 mg/kg for plums subgroup.

Strawberries

Results from supervised field trials on strawberries conducted in the US were provided to the Meeting.

A total of eight independent supervised trials were conducted in the US on strawberries according to the US GAP for low growing berries, which allows three applications at a maximum rate of 0.1 kg ai/ha with a 0-day PHI.

Residues of flonicamid matching the US GAP were: 0.13, 0.19, 0.27, 0.33, 0.41, 0.47, 0.54 and 0.59 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg and an STMR of 0.37 mg/kg for low growing berries.

Brassica (Cole or cabbage) vegetables, Head cabbages, Flowerhead brassicas

Results from supervised field trials on cabbage and broccoli conducted in the US were provided to the Meeting.

A total of 12 independent supervised trials were conducted in the US on broccoli (6) and cabbage (6) according to the US GAP on Brassica (Cole) Leafy Vegetables which allows three applications at a maximum rate of 0.1 kg ai/ha with a 0-day PHI.

Residues of flonicamid from six broccoli trials matching the US GAP for Brassica (Cole) leafy vegetables were: 0.250, 0.428, 0.432, 0.462, 0.499 and 0.553 mg/kg.

Residues of flonicamid from six trials on cabbage (with wrapper leaves) matching the US GAP for Brassica (Cole) Leafy Vegetables were: < 0.025, 0.025, 0.062, 0.205, 0.288 and 1.262 mg/kg.

Residues of flonicamid from six trials on cabbage (without wrapper leaves) matching the US GAP for Brassica (Cole) Leafy Vegetables were: < 0.025 (6) mg/kg.

The Meeting noted that in the US a group GAP for Brassica (Cole) leafy vegetables exists and decided to explore the possibility of setting a group maximum residue level. Since median residues among the representative crops were within a 5-fold range (0.45 mg/kg vs. 0.134 mg/kg) and the Mann-Whitney test indicated that the residues were not statistically different, the Meeting decided to estimate a group maximum residue level based on the following combined residues: < 0.025(7), 0.025, 0.062, 0.205, 0.288 and 1.262 mg/kg.

The Meeting estimated a maximum residue level of 2.0 mg/kg and an STMR of 0.358 mg/kg for Brassica (Cole or cabbage) vegetables, head cabbages and flowerhead Brassicas.

The Meeting estimated an STMR of 0.02 mg/kg for cabbage (without wrapper leaves).

Fruiting vegetables, Cucurbits

Supervised field trials on field- and greenhouse-grown melons conducted in Southern EU and on field-grown pumpkins conducted in Hungary were provided to the Meeting. However, only four trials on melons and four trials on pumpkins matched the critical GAP of Slovenia which allows three foliar spray applications of a WG formulation at 0.05 kg ai/ha with a re-treatment interval of 7 days and a PHI of 1 day. Therefore, in the absence of a sufficient number of trials matching the Slovenia critical GAP, these trials were not considered further.

A total of 17 independent supervised trials, conducted in the US on cucumber (6), melon (6) and summer squash (5) according to the US GAP on cucurbit vegetables, which allows three applications at a maximum rate of 0.1 kg ai/ha with a 0-day PHI, were provided to the Meeting. In addition, the Meeting received four greenhouse cucumber trials conducted in Canada and the US according to the US critical GAP which allows two foliar spray or soil applications at a maximum rate 0.15 kg ai/ha with a re-treatment interval of 6–7 days and a 0-day PHI.

Residues of flonicamid from six field cucumber trials matching the US GAP for cucurbit vegetables were: 0.04, 0.06 (3), 0.07 and 0.12 mg/kg.

Residues of flonicamid from six melon trials matching the US GAP for cucurbit vegetables were: 0.020, 0.03, 0.04, 0.05, 0.06 and 0.09 mg/kg.

Residues of flonicamid from five summer squash trials matching the US GAP for cucurbit vegetables were: 0.01, 0.03 (3) and 0.04 mg/kg.

Residues of flonicamid from the greenhouse cucumber trials matching the US GAP for the foliar spray application were: 0.05, 0.06, 0.14 and 0.54 mg/kg.

Residues of flonicamid from the greenhouse cucumber trials where the growth media was treated were: 0.09, 0.13, 0.16 and 0.20 mg/kg.

For greenhouse cucumbers, as there is an insufficient number of supervised trials conducted in accordance with the US critical GAP, the Meeting did not consider these trials further.

In addition to the US trials, the Meeting received 10 independent supervised field trials conducted in Australia on cucumbers (2), melons (5) and summer squash (3) according to the

Australian GAP on cucurbit vegetables which allows three applications at a maximum rate of 0.1 kg ai/ha with a 1-day PHI.

Residues of flonicamid from two field cucumber trials matching the Australian GAP for Cucurbit Vegetables were: 0.03 (2) mg/kg.

Residues of flonicamid from five melon trials matching the Australian GAP for Cucurbit Vegetables were: 0.03, 0.05 (2), 0.09 and 0.17 mg/kg.

Residues of flonicamid from three summer squash trials matching the Australian GAP for Cucurbit Vegetables were: 0.01, 0.04 and 0.08 mg/kg.

Since the use of flonicamid on the cucurbits crop group is registered in Australia, the residue decline trials demonstrated limited dissipation of flonicamid residues with increasing PHI and that there are an insufficient number of Australian trials at the critical GAP, the Meeting compared the US field trials against the Australian GAP and combined them as follows:

Residues of flonicamid in field cucumbers from eight trials were: 0.03 (2), 0.04, 0.06 (3), 0.07 and 0.12 mg/kg.

Residues of flonicamid in melons from 11 trials were: 0.02, 0.03(2), 0.04 (2), 0.05 (2), 0.06, 0.09 (2) and 0.17 mg/kg.

Residues of flonicamid in summer squash from eight trials were: 0.01 (2), 0.03 (3), 0.04 (2) and 0.08 mg/kg.

The median residues among the representative crops were within a 5-fold range (0.06 mg/kg vs. 0.05 vs 0.03 mg/kg) and the Kruskall-Wallis test indicated that the residues were not statistically different, therefore, the Meeting decided to combine the dataset as follows: 0.01 (2), 0.02, 0.03 (7), 0.04 (5), 0.05 (2) 0.06 (4), 0.07, 0.08, 0.09 (2), 0.12 and 0.17 mg/kg.

The Meeting estimated a maximum residue level for fruiting vegetables, cucurbits, of 0.2 mg/kg and an STMR of 0.04 mg/kg.

Fruiting vegetables, other than cucurbits

Results from supervised field trials on tomatoes, bell peppers and non-bell peppers were conducted in the US as well as supervised trials on greenhouse tomatoes conducted in Canada and the US were provided to the Meeting.

A total of 34 independent supervised trials were conducted in the US on field tomatoes (26), bell peppers (6) and non-bell peppers (2) according to the US GAP on fruiting vegetables, which allows three foliar spray applications of a WG formulation at a maximum rate of 0.1 kg ai/ha or two applications of a SG formulation at a maximum rate of 0.15 kg ai/ha. For both formulations, the crops may be harvested at a 0-day PHI.

Three additional trials were conducted in Canada and the US on greenhouse tomatoes where treatments were conducted according to the US GAP which allows two foliar spray applications at a maximum rate of 0.15 kg ai/ha with a 0-day PHI.

Only field tomato trials were conducted with both the WG and SG formulations, however, it was not clear which formulation resulted in the critical GAP:

Residues of flonicamid from 12 field tomato trials where the WG formulation was applied according to the US critical GAP for fruiting vegetables were: 0.03, 0.05, 0.06, 0.07, 0.08, 0.09 (3), 0.14, 0.15, 0.22 and 0.23 mg/kg.

Residues of flonicamid from 14 field tomato trials where the SG formulation was applied according to the US critical GAP for fruiting vegetables were: < 0.01, 0.05(2), 0.06, 0.07, 0.08, 0.10 (2), 0.11, 0.12 (2), 0.13, 0.15 and 0.19, mg/kg.

As highest residues in tomatoes were observed following treatment with the WG formulation, only these were considered when estimating the maximum residue level.

Residues of flonicamid from six bell pepper trials matching the US critical GAP for fruiting vegetables were: 0.06 (3), 0.10 and 0.11 (2) mg/kg.

Residues of flonicamid from two non-bell pepper trials matching the US critical GAP for fruiting vegetables, other than cucurbits were: 0.21 and 0.22 mg/kg.

As the GAP in the US is for the fruiting vegetables crop group, the median values from the trials conducted in the US on tomatoes, bell peppers and non-bell peppers were within 5-fold (0.09 mg/kg vs 0.08 mg/kg vs 0.21 mg/kg) and the Kruskall-Wallis test indicated that the residues from field trials were not statistically different, the Meeting decided to estimate a group maximum residue level. The residues in tomatoes, bell peppers and non-bell peppers were combined as follows: 0.03, 0.05, 0.06 (4), 0.07, 0.08, 0.09 (3), 0.10, 0.11 (2), 0.14, 0.15, 0.21, 0.22 (2) and 0.23 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg and an STMR of 0.09 mg/kg for fruiting vegetables, other than cucurbits, excluding mushrooms and sweet corn.

Leafy vegetables

Leafy vegetables (excluding Brassica leafy vegetables)

Results from supervised field trials on head lettuce, leaf lettuce, spinach and radish leaves conducted in the US were provided to the Meeting.

A total of 18 independent supervised trials were conducted in the US on head lettuce (6), leaf lettuce (6) and spinach (6) according to the US GAP on leafy vegetables which allows three applications at a maximum rate of 0.1 kg ai/ha with a 0-day PHI.

A total of five independent supervised trials were conducted in the US on radish leaves according to the US GAP on root and tuber vegetables which allows three applications at a maximum rate of 0.1 kg ai/ha with a 3-day PHI.

Residues of flonicamid from six head lettuce (with wrapper leaves) trials matching the US GAP for leafy vegetables were: 0.39, 0.43, 0.49, 0.52, 0.58 and 0.62 mg/kg.

Residues of flonicamid from six trials on leaf lettuce matching the US GAP for leafy vegetables (except Brassica) were: 1.94, 2.18, 2.52, 2.67, 2.71, 3.06 and 3.11 mg/kg.

Side-by-side trials were conducted on cos lettuce comparing the WG formulation with the SG formulation with and without surfactant. These trials were not considered further in the estimation of the maximum residue level.

Residues of flonicamid from six trials on spinach matching the US GAP for leafy vegetables were: 4.82, 4.86, 5.71, 5.73, 6.59 and 6.97 mg/kg.

Residues of flonicamid from five trials on radish leaves matching the US GAP for root and tuber vegetables were: 0.21, 3.1, 5.4, 5.7 and 8.5 mg/kg.

As the GAP in the US is established for the leafy vegetables crop group, the Meeting decided to explore the possibility of setting a group MRL. The median residues in head lettuce, leaf lettuce and spinach, which are the representative commodities for this subgroup, differed by more than 5-fold (0.51 mg/kg vs 2.67 mg/kg vs 5.72 mg/kg). In addition, as the GAP for radish leaves differs from that of the other leafy vegetables, the Meeting decided to estimate maximum residue levels for each commodity based on the individual datasets without extrapolation to the entire subgroup.

The Meeting estimated a maximum residue level of 1.5 mg/kg and an STMR of 0.51 mg/kg for head lettuce with wrapper leaves.

For leaf lettuce, the Meeting estimated a maximum residue level of 8 mg/kg and an STMR of 2.67 mg/kg

The Meeting estimated a maximum residue level of 20 mg/kg and an STMR of 5.72 mg/kg for spinach.

The Meeting estimated a maximum residue level of 20 mg/kg and an STMR of 8.50 mg/kg for radish leaves.

Brassica leafy vegetables

Results from supervised field trials on mustard greens conducted in the US were provided to the Meeting.

A total of eight independent supervised trials were conducted in the US on mustard greens according to the US GAP on Brassica (Cole) leafy vegetables which allows three applications at a maximum rate of 0.1 kg ai/ha with a 0-day PHI.

Residues of flonicamid from eight trials on mustard greens matching the US GAP for Brassica (Cole) leafy vegetables were: 2.04, 2.21, 3.96, 4.40, 4.78, 4.92, 6.87 and 8.31 mg/kg.

The Meeting estimated a maximum residue level of 15 mg/kg and an STMR of 8.31 mg/kg for the Brassica leafy vegetables subgroup.

Root and tuber vegetables

Results from supervised field trials on potatoes, carrots and radish roots conducted in the US and Australia (potatoes only) were provided to the Meeting.

A total of 23 independent supervised trials were conducted in the US on potatoes (16), carrots (2) and radishes (5) according to the critical GAP in the US which allows three applications at a maximum rate of 0.1 kg ai/ha with a 7-day PHI for potatoes and a 3-day PHI for carrots and radishes.

Residues of flonicamid from 16 potato trials matching the US GAP were: < 0.01 (15) and 0.015 mg/kg.

Residues of flonicamid from two carrot trials matching the US GAP were: 0.02 (2) mg/kg.

Residues of flonicamid from five radish trials matching the US GAP were: 0.02, 0.07, 0.10, 0.13 and 0.21 mg/kg.

The Meeting noted that in the US, group GAPs for root and tuber vegetables and tuberous and corm vegetables exist; however, as these GAPs are different for each crop group and there is an insufficient number of supervised residue trials provided for carrots, the Meeting decided to estimate individual maximum residue levels for potato and radish roots only.

For potatoes, the Meeting estimated a maximum residue level of 0.015 mg/kg and an STMR of 0.01 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg and an STMR of 0.10 mg/kg for radish roots.

Celery

Results from supervised field trials on celery conducted in the US were provided to the Meeting.

A total of six independent supervised trials were conducted in the US on celery according to the US GAP for leafy vegetables, except Brassica vegetables, which includes the leaf petioles subgroup, and allows three applications at a maximum rate of 0.1 kg ai/ha with a 0 PHI.

Residues of flonicamid matching the US GAP were: 0.35, 0.43, 0.44, 0.45, 0.46, 0.93 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg and an STMR of 0.45 mg/kg for celery.

Cereal grains

Results from supervised trials on wheat and barley conducted in Northern and Southern EU were provided to the meeting.

A total of 23 independent supervised trials were conducted in EU on wheat (15) and barley (8). The wheat trials were conducted according to the Slovenia GAP which allows two applications at a maximum rate of 0.07 kg ai/ha with a 28-day PHI.

As there is no GAP for barley, these trials were not considered further.

Residues of flonicamid in wheat grain matching the Slovenia GAP were: < 0.01 (11), 0.01, 0.02, 0.04 and 0.06 mg/kg.

The Meeting estimated a maximum residue level of 0.08 mg/kg and an STMR of 0.01 mg/kg for wheat.

Tree nuts

Results from supervised field trials on almonds, pecans and pistachios conducted in the US were provided to the Meeting.

A total of 12 independent supervised trials were conducted in the US on almonds (5), pecans (5) and pistachios (2) according to the US GAP which allows three applications at a maximum rate of 0.1 kg ai/ha with a 40-day PHI.

Residues of flonicamid in almond nutmeats matching the US GAP were: < 0.01 (5) mg/kg.

Residues of flonicamid in pecan nutmeats matching the US GAP were: < 0.01 (5) mg/kg.

Residues of flonicamid in pistachios matching the US GAP were 0.02 and 0.04 mg/kg.

As the Meeting could not conclude that there are no measurable residues in all tree nuts in the tree nut crop group and considering the insufficient number of supervised residue trials for pistachios, the Meeting agreed to estimate individual maximum residue levels for almonds and pecans at 0.01* mg/kg with an STMR of 0.01 mg/kg.

Oilseeds

Rape seed

Results from supervised field trials on rape seed conducted in the US were provided to the Meeting.

A total of nine independent supervised trials were conducted in the US on rape seed according to the US GAP which allows three applications at a maximum rate of 0.1 kg ai/ha with a 7-day PHI.

Residues of flonicamid matching the US GAP were: < 0.02, 0.02 (3), 0.04, 0.08, 0.09, 0.17 and 0.33 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.04 mg/kg for rape seed.

Cotton seed

Results from supervised field trials on cotton conducted in the US and Australia were provided to the Meeting.

The GAP in the US is three applications at a maximum rate of 0.1 kg ai/ha with a 30-day PHI while the GAP in Australia is three applications at a maximum rate of 0.07 kg ai/ha with a 7-day PHI.

As the critical GAP is in Australia, only the Australian trials were considered.

Residues of flonicamid in cottonseed from eight independent supervised residue trials matching the Australian critical GAP were: 0.01 (2), 0.02, 0.04, 0.09, 0.13, 0.16 and 0.34 mg/kg.

The Meeting estimated a maximum residue level of 0.6 mg/kg and an STMR of 0.06 mg/kg for cottonseed.

Mint

Results from supervised field trials on fresh mint leaves conducted in the US were provided to the Meeting.

A total of three independent supervised trials were conducted in the US on mint according to the US GAP which allows three applications at a maximum rate of 0.1 kg ai/ha with a 7-day PHI.

Residues of flonicamid matching the US GAP were: 1.70, 1.92 and 2.36 mg/kg.

The Meeting estimated a maximum residue level of 6 mg/kg and an STMR of 1.92 mg/kg for mint.

Dried hops

Results from supervised field trials on dried hops conducted in the US were provided to the Meeting.

A total of four independent supervised trials were conducted in the US on dried hops according to the US GAP which allows three applications at a maximum rate of 0.1 kg ai/ha with a 10-day PHI.

Residues of flonicamid matching the US GAP were: 0.56, 1.15, 2.82 and 9.33 mg/kg.

The Meeting estimated a maximum residue level of 20 mg/kg and an STMR of 1.98 mg/kg for dried hops.

Teas

Results from supervised field trials on tea conducted in Japan were provided to the Meeting.

A total of two independent supervised trials were conducted in Japan on tea according to the Japanese GAP which allows one application at a maximum rate of 0.1 kg ai/ha with a 7-day PHI.

Residues of flonicamid in green tea leaves matching the Japanese GAP were: 15.7 and 20.1 mg/kg.

There is insufficient data for the Meeting to estimate a maximum residue level.

Animal feeds

Straw, fodder and forage of cereal grains and grasses including buckwheat fodder forage

Wheat

Results from supervised trials on wheat and barley conducted in Northern and Southern EU were provided to the meeting.

A total of 23 independent supervised trials were conducted in EU on wheat (15) and barley (8). The wheat trials were conducted according to the Slovenia GAP which allows two applications at a maximum rate of 0.07 kg ai/ha with a 28-day PHI.

As there is no GAP for barley, these trials were not considered further.

Residues of flonicamid in wheat forage matching the Slovenia Gap were: 0.64, 0.69, 0.83, 0.88 and 0.99 (2).

The Meeting estimated a maximum residue level of 3.0 mg/kg and a median of 0.86 mg/kg for wheat forage.

Residues of flonicamid in wheat straw matching the Slovenia GAP were: < 0.02 (5), 0.02, 0.04 (2), 0.05 (3), 0.08, 0.09, 0.11 and 0.23 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg and a median of 0.04 mg/kg for wheat straw and fodder, dry.

Alfalfa

Results from six independent supervised field trials on alfalfa (4) and clover (2) conducted in the US were provided to the Meeting.

The US GAP for alfalfa grown west of the Rockies allows two applications at a maximum rate of 0.10 kg ai/ha with PHIs of 14 days for seed and forage and 60 days for hay.

Two supervised trials were conducted on clover in the US, however, in the absence of a US GAP, these trials were not considered.

Four trials were conducted on alfalfa in the US, of which only two were conducted according to the US GAP. In the absence of a sufficient number of trials, the Meeting could not estimate a maximum residue level or a median residue for alfalfa seed, forage and hay.

Miscellaneous fodder and forage crops (fodder)

Almond hulls

Results from supervised field trials on almonds conducted in the US were provided to the Meeting.

Five independent trials were conducted on almonds in the US. The GAP in the US allows three applications at a maximum rate of 0.10 kg ai/ha with a PHI of 40 days.

Residues in almond hulls (dry weight) from five trials matching US GAP were: 0.92, 1.09, 1.81, 2.75 and 4.73 mg/kg. The meeting estimated a maximum residue level of 9 mg/kg and a median residue of 1.8 mg/kg.

Cotton gin trash

Results from supervised field trials on cotton conducted in the US and Australia were provided to the Meeting.

The GAP in the US is three applications at a maximum rate of 0.1 kg ai/ha with a 30-day PHI while the GAP in Australia is three applications at a maximum rate of 0.07 kg ai/ha with a 7-day PHI.

As the critical GAP is in Australia, only the Australian trials were considered.

The residues of flonicamid in cotton gin trash from eight independent supervised trials matching the Australian critical GAP were: 0.66, 1.20, 1.33, 1.60, 1.70, 2.30, 2.75, 3.00 and 3.72 mg/kg.

The Meeting estimated a median residue of 1.7 mg/kg.

Fate of residues during processing

High temperature hydrolysis

To simulate the degradation of flonicamid during pasteurization, baking, brewing, boiling and sterilisation, the hydrolysis of radio-labelled flonicamid 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. More specifically, flonicamid accounted

for at least 96% of the applied radioactivity. Therefore, very limited degradation of flonicamid was observed in aqueous buffer solutions under all the conditions tested with no significant degradation product being formed.

Processing

The Meeting received information on the fate of flonicamid residues and its metabolites TFNA-AM, TFNA and TFNG during the processing of raw agricultural commodities (RAC) like apples, peaches, plums, tomatoes, potatoes, rape seed, cotton and mint.

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 for which maximum residue levels were estimated.

RAC	Processed Commodity	Calculated processing factors	Best estimate	STMR-P
		Flonicamid		
Peaches	Canned peaches	0.3, 0.5, 0.3, 3.3	0.3 (median)	0.08
	Juice	1.0, 1.0, 0.3, 0.5	0.8 (median)	1.8
	Jam	0.3, 1.0, 1.0, 0.2	0.7 (median)	0.16
	Puree	0.7, 1.0, 1.0, 0.8	0.9 (median)	0.21
Plums	Dried prunes	1.0	1.0	0.04
Tomato	Paste	16.1	16.1	1.45
Potato	Chips	0.95	0.95	0.01
	Flakes	2.7	2.7	0.03
Canola	Refined oil	< 0.1	0.1	0.004
Cotton	Refined oil	< 0.24 (US), 0.6 and 0.04 (AUS)	0.32 (mean; AUS)	0.02
Mint	Oil	< 0.03	0.03	0.06

As the residue concentration in both apple juice and apple pomace were higher than in fresh apple which is not physically possible, the Meeting determined that the apple processing study was not reliable and did not calculate a processing factor for juice.

As the residue concentration is higher in tomato paste than in fresh tomato, the Meeting estimated a maximum residue level of 7.0 mg/kg by multiplying the maximum residue level for fruiting vegetables, other than cucurbits, (0.4 mg/kg) by 16.1.

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 2.50, 6.89 and 23.7 ppm of a 1:1 mixture of flonicamid:TFNG for 28 consecutive days. As demonstrated in the metabolism studies, residues of TFNG present in feed items may be converted to TFNA-AM. Therefore, the Meeting concluded that the test material used in the feeding studies was appropriate.

In milk, no quantifiable (< LOQ) residues of flonicamid were detected in any test group. For TFNA-AM, the average residues increased from < LOQ in the low dose group to 0.02 mg/kg in the mid dose group and to 0.08 mg/kg in the high dose group.

In liver, no quantifiable residues of flonicamid were detected. For TFNA-AM, residues were detected in the mid and high dose groups above the LOQ using two different analytical methods (FMC-P-3580/RCC 844743) with different LOQ (0.025/0.01 mg/kg). TFNA-AM levels increased from less than LOQ in the low dose group to 0.039/0.02 mg/kg in the mid dose group and 0.113/0.05 mg/kg in the high dose group.

In kidney, TFNA-AM was detected in the medium and high dose groups above the LOQ using the same analytical methods as those used for kidney. TFNA-AM levels increased from

levels below LOQ in the low dose group to 0.031/0.02 mg/kg in the mid dose group and 0.105/0.09 mg/kg in the high dose group.

In muscle, only TFNA-AM was found. The level increased from below LOQ (0.025 mg/kg) in the low dose group to 0.027 mg/kg in the mid dose group and 0.088 mg/kg in the high dose group. Similarly, only TFNA-AM was measurable in fat and only at the high dose level (0.015 mg/kg).

The Meeting also received information on the residue levels arising in tissues and eggs when groups of laying hens were fed with a diet containing 0.26, 2.51, 7.47 and 25.83 ppm of a 1:1 mixture of flonicamid:TFNG for 28 consecutive days.

The average flonicamid residues in eggs increased from < LOQ in the very low and low dose groups to 0.02 mg/kg in the mid dose group and to 0.08 mg/kg in the high dose group. Average residues of TFNA-AM increased from < LOQ in the very low and low dose groups to 0.27 mg/kg in the mid dose group and 0.95 mg/kg in the high dose group.

No quantifiable residues (< LOQ) of flonicamid were found in muscle in any treatment group. No quantifiable residues (< LOQ) of TFNA-AM was measurable in muscle at the very low dose group, but there appeared to be a dose response relationship at all other dose levels; 0.049 mg/kg in the low dose group, 0.168 mg/kg in the mid dose group and 0.654 mg/kg in the high dose group.

In liver and fat, no quantifiable residues (< LOQ) of flonicamid were found at any dosing level. For liver, TFNA-AM residues increased from < 0.01 mg/kg (very low) to 0.05 mg/kg (low) to 0.17 mg/kg (mid) and 0.71 mg/kg (high) while for fat, TFNA-AM residues increased from 0.01 mg/kg (very low) to 0.02 mg/kg (low) to 0.06 mg/kg (mid) and 0.29 mg/kg (high).

Estimated dietary burdens of farm animals

Maximum and mean dietary burden calculations for flonicamid 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.

The foliar application of flonicamid to apples, cabbage, potato, almonds, rape seed, cotton and wheat resulted in residues of flonicamid in the following feed items: wet apple pomace, head cabbage with wrapper leaves, potato culls, almond hulls, rape seed meal, undelinted cottonseed, cotton seed hulls, cottonseed meal, gin trash, wheat forage, grain and straw. Based on the named feed items, the calculated maximum animal dietary burden for dairy or beef cattle was in Australia (3.96 ppm), followed by EU (1.39 ppm) and US-Canada (0.29 ppm).

	Livestock dietary burden, flonicamid, ppm of dry matter							
	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0.27	0.13	1.39	1.02	3.96 a	3.44 c	0.003	0.003
Dairy cattle	0.81	0.70	0.82	0.71	2.38 b	2.07 d	0.002	0.002
Poultry—broiler	0.03	0.03	0.01	0.01	0.02	0.02	0	0
Poultry—layer	0	0	0.40 e	0.34 f	0	0	0	0

[a] Suitable for MRL estimates for mammalian meat, fat and edible offal

[b]. Suitable for MRL estimates for milk

[c] Suitable for STMR estimates for mammalian meat, edible offal

[d] Suitable for STMR estimate for milk

[e] Suitable for MRL estimates for eggs, meat, fat and edible offal of poultry

[f] Suitable for STMR estimates for eggs, meat, fat and edible offal of poultry

Animal commodities maximum residue level estimation

As all dietary burdens were lower than the lowest feeding levels from the dairy cow and laying hen feeding studies and since all residues of flonicamid and TFNA-AM were below the limit of quantitation at the lowest feeding levels, there is no expectation of any measurable transfer of residues from the feed items into the livestock commodities (see tables below).

	Feed level (ppm) for milk residues	Total flonicamid and TFNA-AM residues in milk (mg/kg)	Feed level for tissue residues (ppm)	Flonicamid and TFNA-AM Residues			
				Muscle	Liver	Kidney	Fat
Maximum residue level—beef or dairy cattle							
Feeding study	2.50	0.043	2.50 6.89	< 0.045 0.050	< 0.045 0.062	< 0.045 0.054	< 0.02 < 0.02
Dietary burden and residue estimate	2.38	0.04	3.96	0.047	0.051	0.048	< 0.02
STMR—beef or dairy cattle							
Feeding study	2.50	0.041	2.50 6.89	< 0.045 0.047	< 0.045 0.059	< 0.045 0.051	< 0.02 < 0.02
Dietary burden and residue estimate	2.07	0.04	3.44	0.045	0.048	0.046	< 0.02

	Feed level (ppm) for egg residues	Total flonicamid and TFNA-AM residues in eggs (mg/kg)	Feed level for tissue residues (ppm)	Flonicamid and TFNA-AM Residues		
				Muscle	Liver	Fat
Maximum residue level—poultry broiler or layer						
Feeding study	0.26 2.51	0.02 0.11	0.26 2.51	< 0.02 0.07	< 0.02 0.08	< 0.02 0.04
Dietary burden and residue estimate	0.40	0.03	0.40	0.02	0.02	0.02
STMR—poultry broiler or layer						
Feeding study	0.26 2.51	0.02 0.09	0.26 2.51	< 0.02 0.06	< 0.02 0.06	< 0.02 0.03
Dietary burden and residue estimate	0.34	0.02	0.34	0.02	0.02	0.02

The Meeting estimated maximum residue levels of 0.02* mg/kg for mammalian fats, 0.04 mg/kg for milks and 0.05 mg/kg for meat from mammals other than marine mammals and 0.06 mg/kg for edible offal (mammalian). The STMRs for mammalian fats, milks, meat from mammals other than marine mammals and edible offal (mammalian) are 0.02 mg/kg, 0.04 mg/kg. 0.047 mg/kg and 0.051 mg/kg, respectively.

In addition, the Meeting estimated maximum residue levels of 0.02* mg/kg for poultry meat (including Pigeon meat), poultry fats and edible offal of poultry and 0.03 mg/kg for eggs. The STMRs were 0.02 mg/kg, 0.02 mg/kg, 0.02 mg/kg and 0.02 mg/kg for meat, edible offal, fat and eggs, respectively.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed in Annex 1 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 commodities: *Flonicamid*

Definition of the residue for compliance with the MRL and for estimation of dietary intake for animal commodities: Sum of flonicamid, N-cyanomethyl-4-(trifluoromethyl)nicotinamide and the metabolite TFNA-AM, 4-(trifluoromethyl)nicotinamide.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
TN 0660	Almonds	0.01*		0.01	
VB 0040	Brassica (cole or cabbage) vegetables, Head cabbages, Flowerhead brassicas	2		0.36	
VL 0054	Brassica leafy vegetables	15		8.31	
VS 0624	Celery	1.5		0.45	
FS 0013	Cherries	0.9		0.28	
SO 0691	Cotton seed	0.6		0.06	
MM 032	Edible offal (mammalian)	0.06		0.05	
PE 039	Eggs	0.03		0.02	
VC 0045	Fruiting vegetables, Cucurbits	0.2		0.04	
VO 0050	Fruiting vegetables, other than Cucurbits (except mushrooms and sweet corn)	0.4		0.09	
DH 1100	Hops, dry	20		1.98	
VL 0482	Lettuce, Head	1.5		0.51	
VL 0483	Lettuce, Leaf	8		2.67	
FB 2009	Low growing berries	1.5		0.37	
MM 031	Mammalian fats	0.02		0.02	
MM 030	Meat (from mammals other than marine mammals)	0.05		0.04	
MM 033	Milks	0.04		0.04	
HH 0738	Mints	6		1.92	
AM 0660	Miscellaneous fodder and forage crops (fodder)	9		1.81	
FS 2001	Peaches (including Nectarine and Apricot)	0.7		0.14	
TN 0672	Pecan	0.01*		0.01	
FS 0014	Plums (including Prunes)	0.1		0.03	
FP 0009	Pome fruits	0.8		0.13	
VR 0589	Potatoes	0.015		0.01	
PF 037	Poultry fats	0.02		0.02	
PM 036	Poultry meat (including Pigeon meat)	0.02		0.02	
PO 038	Poultry, Edible offal of	0.02		0.02	
VR 0494	Radish	0.4		0.1	
VL 0494	Radish leaves	20		8.5	
SO 0495	Rape seed	0.5		0.04	
VL 0502	Spinach	20		5.72	
AF 051	Straw, fodder and forage of cereal grains and grasses (including buckwheat fodder) (forage)	3		0.86	
AS 051	Straw, fodder and forage of cereal grains and grasses (including buckwheat fodder) (straws and fodders dry)	0.3		0.04	
VW 0448	Tomato paste	7		1.45	
GC 0654	Wheat	0.08		0.01	
	Canned peaches			0.1	
OC 0691	Cotton seed oil, crude			0.02	

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
DF 0014	Prunes			0.04	
	Head cabbage without wrapper leaves			0.025	
	Mint oil			0.06	
	Peach Jam			0.16	
	Peach Juice			1.8	
	Peach Puree			0.21	
	Potato chips			0.01	
	Potato flakes			0.03	
OR 0495	Rape seed oil, edible			0.004	
AB 0691	Cotton seed hulls			0.13	
AB 1203	Cotton seed meal			0.14	
	Rape seed meal			0.004	

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Dietary Intakes (IEDIs) of flonicamid were calculated for the 17 GEMS/Food cluster diets using STMRs and STMR-Ps estimated by the current Meeting (Annex 3 to the 2015 Report) estimated by the current Meeting (Annex 3). The ADI is 0–0.07 mg/kg bw and the calculated IEDIs were 1–10% of the maximum ADI. The Meeting concluded that the long-term intake of residues of flonicamid resulting from the uses considered by the current JMPR is unlikely to present a public health concern.

Short-term intake

The Meeting decided that an ARfD is unnecessary and concluded that the short-term intake of residues resulting from the use of flonicamid, considered by the present Meeting, is unlikely to present a public health concern.

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A-22-01-10 AF/5174/IB	Ginzburg, N	2002j	Determination of Residues of IKI-220 And its Metabolites TFNG, TFNA and TFNA-AM In Winter Wheat after two treatments of IKI-220 50% WG (IBE 3880 or IBE 3894) (North and South of France and Germany—Season 2000) Batelle, Geneva Research Centres; GLP, unpublished Field part: Anthony, S, 2000. To generate crop specimens for analysis of IKI-220 residues in the RAC winter wheat and processed fractions resulting from a sequential application of IBE 3894 or IBE 3880 in S. France and N. France during 2000 Agrisearch, report no, November 24, 2000 GLP, unpublished Trial—220/TRAZW 06/F/00
A-22-01-16 20015002/I1-FPWW,	Ginzburg, N	2002k	Determination of Residues of IKI-220 And its Metabolites TFNG, TFNA and TFNA-AM In Winter Wheat after two treatments of IKI-220 50% WG (IBE 3894) (Italy, Spain, North and South of France, Germany and United Kingdom—Season 2001) Batelle, Geneva Research Centres; GLP, unpublished Field part: Miserocchi, G 2001. Generation of samples for the determination of residues of IKI-220 (code IBE 3894) on winter wheat at 1 site in Italy, 2001 S.P.F. GAB, report no October 22, 2001 GLP, unpublished Trial—220/TRAZW 07/I/01
A-22-01-16 E/789/S/01	Ginzburg, N	2002l	Determination of Residues of IKI-220 And its Metabolites TFNG, TFNA and TFNA-AM In Winter Wheat after two treatments of IKI-220 50% WG (IBE 3894) (Italy, Spain, North and South of France, Germany and United Kingdom—Season 2001) Batelle, Geneva Research Centres; GLP, unpublished Field part: Valli, F 2001. Production of samples for residue analysis in wheat after 2 foliar

Code	Author(s)	Year	Title, Institute, Report reference
			applications of IKI-220 Agri 2000, December 5, 2001 GLP, unpublished Trial—220/TRAZW 08/I/01
A-22-01-16 E/789/S/01	Ginzburg, N	2002m	Determination of Residues of IKI-220 And its Metabolites TFNG, TFNA and TFNA-AM In Winter Wheat after two treatments of IKI-220 50% WG (IBE 3894) (Italy, Spain, North and South of France, Germany and United Kingdom—Season 2001) Batelle, Geneva Research Centres; GLP, unpublished Field part: Valli, F 2001. Production of samples for residue analysis in wheat after 2 foliar applications of IKI-220 Agri 2000, December 5, 2001 GLP, unpublished Trial—220/TRAZW 09/I/01
A-22-01-16 AF/5731/IB	Ginzburg, N	2002n	Determination of Residues of IKI-220 And its Metabolites TFNG, TFNA and TFNA-AM in Winter Wheat after two treatments of IKI-220 50% WG (IBE 3894) (Italy, Spain, North and South of France, Germany and United Kingdom—Season 2001) Batelle, Geneva Research Centres; GLP, unpublished Field part: Anthony, S, 2002. To generate crop specimens for analysis of IKI-220 residues in the RAC winter wheat resulting from a sequential application of IBE 3894 in S France. N France and UK during 2001 Agrisearch, report no, August 6, 2002 GLP, unpublished Trial—220/TRAZW 14/F/01
A-22-01-16 AF/5731/IB	Ginzburg, N	2002o	Determination of Residues of IKI-220 And its Metabolites TFNG, TFNA and TFNA-AM In Winter Wheat after two treatments of IKI-220 50% WG (IBE 3894) (Italy, Spain, North and South of France, Germany and United Kingdom—Season 2001) Batelle, Geneva Research Centres; GLP, unpublished Field part: Anthony, S, 2002. To generate crop specimens for analysis of IKI-220 residues in the RAC winter wheat resulting from a sequential application of IBE 3894 in S France. N France and UK during 2001 Agrisearch, report no, August 6, 2002 GLP, unpublished Trial—220/TRAZW 15/F/01
A-22-01-16 016-01-IK-I/G	Ginzburg, N	2002p	Determination of Residues of IKI-220 And its Metabolites TFNG, TFNA and TFNA-AM In Winter Wheat after two treatments of IKI-220 50% WG (IBE 3894) (Italy, Spain, North and South of France, Germany and United Kingdom—Season 2001) Batelle, Geneva Research Centres; GLP, unpublished Field part: Corts, V. 2001: Generation of specimens of wheat RAC following a program of foliar sprays of an IBE 3894 WG formulation for the purpose of quantifying residues of the ai. Trial to generate a single sampling. Recerca agrícola; November 22, 2001 GLP, unpublished Trial—220/TRAZW 10/E/01
A-22-01-16 043-01-IK-I/G	Ginzburg, N	2002q	Determination of Residues of IKI-220 And its Metabolites TFNG, TFNA and TFNA-AM In Winter Wheat after two treatments of IKI-220 50% WG (IBE 3894) (Italy, Spain, North and South of France, Germany and United Kingdom—Season 2001) Batelle, Geneva Research Centres; GLP, unpublished Field part: Corts, V. 2001: Generation of specimens of wheat RAC following a program of foliar sprays of an IBE 3894 WG formulation for the purpose of quantifying residues of the ai. Trial to generate a single sampling. Recerca agrícola; November 22, 2001 GLP, unpublished Trial—220/TRAZW 11/E/01
P-22-01-02	Ginzburg, N	2003b	Processing study for determination of IKI-220 and its metabolites TFNG, TFNA and TFNA-AM on peaches after two treatments of IKI-220 50% WG (IBE 3894) (Southern Europe—Season-2001) Battelle, GLP, unpublished
A-22-00-03	Ginzburg, N	2003a	Freezer storage stability of IKI-220 and its metabolites TFNG, TFNA and TFNA-AM on various crops. Battelle, Carouge/Geneva, Switzerland; GLP, unpublished
010424-1	Gupta, KS	2002	Metabolism of [^{14}C]IKI-220 by potato. Ricerca LLC, Ohio, USA; Report No, March 5, 2002 GLP, unpublished
011750-1	Gupta, KS & Bassett, J	2002	Metabolism of [^{14}C]IKI-220 in laying hens. Ricerca LLC, Ohio, USA; Report No; May 17, 2002; amendment no. 1 of March 25, 2004 and amendment no. 2 of September 27, 2004 GLP, unpublished
010416-1	Gupta, KS & Kaman, RA	2002	Metabolism of [^{14}C]IKI-220 by wheat. Ricerca LLC, Ohio, USA; March 5, 2002 GLP, unpublished
011048-1	Gupta, KS & Savides, MC	2002	Metabolism of [^{14}C]IKI-220 in lactating goats. Ricerca LLC, Ohio, USA; April 17, 2002; amendment no. 1 of March 25, 2004 GLP, unpublished
6933-96-0186-EF-001-001	Hatzenbeler, CJ & Herczog, KJS	2002	An Aerobic Soil Metabolism Study with [^{14}C]IKI-220. Ricerca, LLC, amended GLP, unpublished
12 ISK AA 0701	Kiss, Z	2013	Residue Analysis of the Active Ingredient Flonicamid and its

Code	Author(s)	Year	Title, Institute, Report reference
			Metabolites (TFNA and TFNG) of IBE3894/TEPPEKI (Teppeki 50% Flonicamid WG) Insecticide in Pumpkin According to GLP Quality Control System Pesticide Analytical Laboratory, Velence, GLP, unpublished
I-329	Kiyuna, C, Sakai, A, Tada, Y & Kanza, T	2008	Metabolism of [^{14}C]IKI-220 in bell peppers Ishihara Sangyo Kaisha Ltd., Japan; August 25, 2008 non-GLP, unpublished
842993	Krainz, A	2002	Validation of a residue analytical method for IKI-220 and its metabolites TFNA, TFNA-AM, OH-TFNA-AM and TFNG in milk. RCC Ltd, Itingen, Switzerland, August 22, 2002 GLP, unpublished
844743	Krainz, A	2003	Development and validation of a residue analytical method for IKI-220 and its metabolites OH-TFNA-AM, TFNA-AM, TFNG and TFNA in animal tissue. RCC Ltd, Itingen, Switzerland, January 13, 2003 GLP, unpublished
013066-1	Lentz, NR	2002	Rate of Degradation of [^{14}C]IKI-220 in Soil. Ricerca LLC, GLP, unpublished
UPL/GLP/10/07-1	Litzow, D	2013a	Residues of UPI-220 in Cotton Australia, 2011 Agrisearch Services Pty Ltd, GLP, unpublished
UPL/GLP/12/01-1	Litzow, D	2013b	Residues of UPI-220 in Cotton Australia, 2012 Agrisearch Services Pty Ltd, GLP, unpublished
UPL/GLP/12/01-1	Litzow, D	2013b	Residues of UPI-220 in Cotton Australia, 2012 Agrisearch Services Pty Ltd, GLP, unpublished
010250-1	O`Donnell, RT	1999b	IKI-220, PAI (Lot #9803)—Organic Solvent Solubility Ricerca, Inc.; Report No GLP, unpublished
010250-1	O`Donnell, RT	1999b	IKI-220, PAI (Lot #9803)—Organic Solvent Solubility Ricerca, Inc.; GLP, unpublished
010251-1	O´Donnell, RT	1999a	IKI-220, PAI (Lot #9803)—Water Solubility Ricerca, Inc.; GLP, unpublished
011586-1	Panthani, AM, Baker, MC & Sandacz Herczog, KJ	2002	Metabolism of [^{14}C]IKI-220 in peaches. Ricerca LLC, Ohio, USA; March 5, 2002 GLP, unpublished
014121-1	Panthani, AM, Findak, D & Herczog, KJS	2003	Metabolism of [^{14}C]IKI-220 by wheat forage and hay. Ricerca LLC, Ohio, USA; January 7, 2003 GLP, unpublished
012575-1	Pelton, JA	2000	IKI-220 TGAI—Appearance, pH, and Relative Density Ricerca, Inc.; GLP, unpublished
012575-1	Pelton, JA	2000	IKI-220 TGAI—Appearance, pH, and Relative Density Ricerca, Inc.; GLP, unpublished
ADPEN-2K2-1126-FMC-ISK	Perez, R	2003	Independent laboratory validation of FMC Corporation for the analysis of IKI-220 and degradates in/on cow muscle, kidney and liver. ADPEN Laboratories, Florida, USA, January 14, 2003 GLP, unpublished
P 2960 G	Richter, S	2013	Independent Laboratory Validation (ILV) of a Residue Analytical Method for the Determination of Flonicamid and Its Metabolite TFNA-AM in Foodstuff of Animal Origin PTRL Europe, GLP, unpublished
ISK/IKI/06001	Royer, A	2008	IKI-220 and its major metabolites Validation of an LC-MS/MS analytical method for the active substance IKI-220 and its 3 major metabolites (TFNA-AM, TFNA and TFNG) in lemon, potato, oil-seed rape and wheat grain, plum and prune ADME Bioanalyses France; GLP, unpublished
IR-4 PR No. 09604	Samoil, KS	2010	Flonicamid: Magnitude of the Residue on Strawberry IR-4 Project, GLP, unpublished
IR-4 PR No. 08551	Samoil, KS	2011a	Flonicamid: Magnitude of the Residue on Cucumber (Greenhouse) IR-4 Project, GLP, unpublished
IR-4 PR No. 08556	Samoil, KS	2012a	Flonicamid: Magnitude of the Residue on Tomato (Field and Greenhouse) IR-4 Project, GLP, unpublished
IR-4 PR No. 08754	Samoil, KS	2006a	Flonicamid: Magnitude of the Residue on Carrot IR-4 Project, GLP, unpublished
IR-4 PR No. 08753	Samoil, KS	2006b	Flonicamid: Magnitude of the Residue on Radish IR-4 Project, GLP, unpublished
IR-4 PR No. 09783	Samoil, KS	2011b	Flonicamid: Magnitude of the Residue on Canola IR-4 Project, GLP, unpublished
IR-4 PR No. 09358	Samoil, KS	2012b	Flonicamid: Magnitude of the Residue on Mint IR-4 Project, GLP, unpublished
IR-4 PR No. 09943	Samoil, KS	2012c	Flonicamid: Magnitude of the Residue on Alfalfa and Crimson Clover IR-4 Project, GLP, unpublished

Code	Author(s)	Year	Title, Institute, Report reference
IR-4 PR No. 08706	Samoil, KS	2005	Flonicamid: Magnitude of the Residue on Hops IR-4 Project, GLP, unpublished
IR-4 PR No. 09783	Samoil, KS	2011b	Flonicamid: Magnitude of the Residue on Canola IR-4 Project, GLP, unpublished
IR-4 PR No. 09358	Samoil, KS	2012b	Flonicamid: Magnitude of the Residue on Mint IR-4 Project, Report No GLP, unpublished
IR-4 PR No. 09604	Samoil, KS	2010	Flonicamid: Magnitude of the Residue on Strawberry IR-4 Project, Report No GLP, unpublished
IR-4 PR No. 08551	Samoil, KS	2011a	Flonicamid: Magnitude of the Residue on Cucumber (Greenhouse) IR-4 Project, Report No GLP, unpublished
010341-1	Schetter, JE	1999	IKI-220—Vapor Pressure Ricerca, Inc.; GLP, unpublished
834028	Schmiedel, U	2001	Expert Statement on the Explosive Properties of IKI-220 Technical RCC Ltd, GLP, unpublished
PL/11/002	Simmonds, R	2011	[^{14}C]Flonicamid: Nature of residues in processed commodities—High temperature hydrolysis Battelle UK Ltd., GLP, unpublished
01053-1	Sweetapple, GG	1999	IKI-220 PAI—Melting Point, Relative Density, Physical State, Color, and Odor Ricerca, Inc.; GLP, unpublished
01053-1	Sweetapple, GG	1999	IKI-220 PAI—Melting Point, Relative Density, Physical State, Color, and Odor Ricerca, Inc.; GLP, unpublished
01053-1	Sweetapple, GG	1999	IKI-220 PAI—Melting Point, Relative Density, Physical State, Color, and Odor Ricerca, Inc.; GLP, unpublished
S11-02600	Tessier, V	2012	Flonicamid IKI-220 (IBE 3894)—Residue study in melon fruits (pulp and peel) after foliar applications of IBE 3894 in Spain, Italy and Southern France in 2011 Eurofins ADME Bioanalyses, GLP, unpublished
842001	Tognucci, A	2002	Determination of the boiling point/boiling range of IKI-220 PAI RCC Ltd, RCC GLP, unpublished
826154	Van Dijk, A	2003	IKI-220 ruminant feeding study: Residues of IKI-220 in milk and edible tissues of cattle. RCC Ltd., Switzerland; January 14, 2003 GLP, unpublished
011049-1	Walsh, KJ	2002c	A Confined Rotational Crop Study with [^{14}C]IKI-220. Ricerca, LLC, GLP, unpublished
011298-1	Walsh, KJ	2002b	Photochemical Degradation of [^{14}C]IKI-220 in Soil. Ricerca LLC, GLP, unpublished
011050-1	Walsh, KJ	2002a	A photolysis study of [^{14}C]IKI-220 in water. Ricerca, LLC; March 6, 2002 GLP, unpublished
008076-2	Walsh, KJ & Murray, MD	2000	A Hydrolysis Study of [^{14}C]IKI-220 in Water Ricerca, Inc.; GPL, unpublished
008076-2	Walsh, KJ & Murray, MD	2000	A hydrolysis study of [^{14}C]IKI-220 in water. Ricerca, LLC; Report No GLP, unpublished
011050-1	Walsh, KJ	2002a	A Photolysis Study of [^{14}C]IKI-220 in Water Ricerca, Inc.; GLP, unpublished
EASSM No. S09-01231	Weber, H	2010	Independent Laboratory Validation of an LC-MS/MS analytical method for the active substance IKI-220 and its 3 major metabolites (TFNA-AM, TFNA and TFNG) in lemon, potato, oil-seed rape, wheat (grain) and plum Eurofins Dr. Specht GLP GmbH; GLP, unpublished
IB-2001-MDG-003-00-01	Wiedman, JL	2002a	Magnitude of Residues of IKI-220 on Pome Fruit—USA in 2001 ISK Biosciences, GLP, unpublished
IB-2001-MDG-005-00-01	Wiedman, JL	2002b	Magnitude of Residues of IKI-220 on Stone Fruit—USA in 2001 ISK Biosciences, GLP, unpublished
IB-2001-MDG-007-00-01	Wiedman, JL	2002c	Magnitude of Residues of IKI-220 on Cucurbits—USA in 2001 ISK Biosciences, GLP, unpublished
IB-2001-MDG-006-00-01	Wiedman, JL	2003a	Magnitude of Residues of IKI-220 on Fruiting Vegetables—USA in 2001 ISK Biosciences, GLP, unpublished
IB-2001-MDG-006-00-01	Wiedman, JL	2003a	Magnitude of Residues of IKI-220 on Fruiting Vegetables—USA in 2001 ISK Biosciences, GLP, unpublished
IB-2001-MDG-002-00-01	Wiedman, JL	2003b	Magnitude of Residues of IKI-220 on Potatoes—USA in 2001 ISK Biosciences, GLP, unpublished
IB-2011-JLW-014-01-01	Wiedman, JL	2012	Magnitude of Residues of Flonicamid on Almonds and Pecans—USA in 2011 ISK Biosciences, Report No GLP, unpublished
IB-2001-MDG-004-00-01	Wiedman, JL	2002d	Magnitude of Residues of IKI-220 on Cotton—USA in 2001 ISK Biosciences, GLP, unpublished
IB-2001-MDG-003-00-01	Wiedman, JL	2002a	Magnitude of Residues of IKI-220 on Pome Fruit—USA in 2001 ISK Biosciences, GLP, unpublished
IB-2001-MDG-005-00-01	Wiedman, JL	2002b	Magnitude of Residues of IKI-220 on Stone Fruit—USA in 2001 ISK Biosciences, GLP, unpublished

Code	Author(s)	Year	Title, Institute, Report reference
IB-2001-MDG-006-00-01	Wiedman, JL	2003a	Magnitude of Residues of IKI-220 on Fruiting Vegetables—USA in 2001 ISK Biosciences, GLP, unpublished
IB-2001-MDG-002-00-01	Wiedman, JL	2003b	Magnitude of Residues of IKI-220 on Potatoes—USA in 2001 ISK Biosciences, GLP, unpublished
IB-2001-MDG-004-00-01	Wiedman, JL	2002d	Magnitude of Residues of IKI-220 on Cotton—USA in 2001 ISK Biosciences, GLP, unpublished
IB-2001-JLW-001-00-01	Wiedman, JL	2003c	Field Accumulation of IKI-220 (Flonicamid) in Rotational Crops—USA in 2001 ISK Biosciences Corporation, USA; GLP, unpublished

FLUMIOXAZIN (284)

The first draft was prepared by Mr David Lunn, Plants, Food & Environment Directorate, Ministry for Primary Industries, Wellington, New Zealand

EXPLANATION

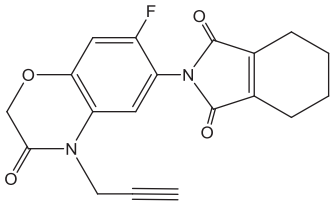
Flumioxazin (S-53482) is variously described as a dicarboxamide, diphenyl-ether or a phenyl-phthalimide herbicide, used for pre-emergent and post-emergent control of a range of broad-leaf weeds and suppression of some grass weed species in a range of fruit, vegetable and field crops. It is non-systemic but is readily absorbed by the foliage of susceptible plants. In the presence of oxygen and light flumioxazin inhibits protoporphyrinogen oxidase resulting in accumulation of porphyrins. The photosensitising action of the accumulated porphyrins enhances peroxidation of membrane lipids and this leads to irreversible damage to the membrane function and structure.

Authorisations exist for the use of flumioxazin as pre-emergence or early post-emergence broadcast treatments, as directed inter-row band soil treatments and as a pre-harvest desiccant (harvest aid) treatment in North America, Europe, Latin America, Australia and some Asian countries.

Flumioxazin was scheduled by the 46th Session of the CCPR as a new compound for consideration by the 2015 JMPR. Residue and analytical aspects of flumioxazin 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 and environmental fate in soil.

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:	Flumioxazin
Code number	S-53482, V-53482
IUPAC name:	*N*-(7-fluoro-3,4-dihydro-3-oxo-4-prop-2-ynyl-2*H*-1,4-benzoxazin-6-yl)cyclohex-1-ene-1,2-dicarboximide
Chemical Abstracts name:	2-[7-fluoro-3,4-dihydro-3-oxo-4-(2-propynyl)-2*H*-1,4-benzoxazin-6-yl]-4,5,6,7-tetrahydro-1*H*-isoindole-1,3(2*H*)-dione
CAS number	103361-09-7
CIPAC number	578
Molecular mass:	354.3
Molecular formula	$C_{19}H_{15}FN_2O_4$
Structural formula:	

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

A detailed chemical and physical characterisation of the active ingredient is given in the following table.

Test or Study & Annex point	Test material purity and specification	Findings and comments	Reference
Melting point	Pure ai (99.6%)	203.51–209.74 °C	SBP-0056
Boiling point	Pure ai (99.6%)	No boiling point measured decomposition at *ca.* 273 °C	SBP-0056
Relative density	Pure ai (99.6%)	1.4157 (20.1 °C)	SBP-0056
Vapour pressure	Pure ai (99.5%)	3.2×10^{-4} Pa at 22 °C	SBP-0010
pH	Technical (97.6%)	7.29 (25 °C) in saturated solution	SBP-0009
Henry's law constant	calculated	K_H = 0.145 Pa m³ mol^{-1} (20–22 °C)	SBH-059
Appearance	Pure ai (99.5%) Technical (97.6%)	White odourless powdery solid Yellowish brown odourless powder	SBP-0011
Solubility in water	Pure ai (99.6%)	0.786 ± 0.1081 mg/L (20 °C) in distilled water pH effect not investigated because of the neutral properties of flumioxazin	SBP-0057
Solubility in organic solvents (g/L, 25 °C)	Technical (97.6%)	Dichloromethane: 191 Tetrahydrofuran: 53.8 Acetonitrile: 32.3 Ethyl acetate: 17.8 Acetone: 17 Methanol: 1.56 n-Octanol 0.163 Hexane: 0.0247	SBP-0011
Octanol/water partition coefficient	Pure ai (99.9%)	Log P_{OW} 2.55 (20 °C, pH 5.92–5.98)	SBP-0001
Hydrolysis (sterile buffer in the dark, 25 °C)	^{14}C labelled pure ai (> 99%)	THP-label / Phenyl-label DT_{50} (pH 5): 3.4 days / 5 days DT_{50} (pH 7): 19–24 hours / 23–26 hours DT_{50} (pH 9): 14–15 minutes / 21–23 minutes	SBM-0006 SBM-0005
Photolysis characteristics	^{14}C labelled pure ai (> 99%)	DT_{50} (pH 5, 25 °C):20.94 hrs (phenyl-label) DT_{50} (pH 5, 25 °C):26.31 hrs (THP-label) under artificial sunlight conditions Degradates: THPA, APF and 482-PHO	JMPS 578

Formulations

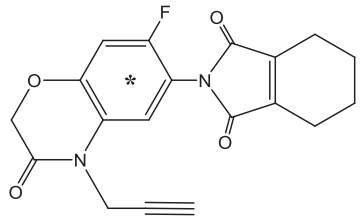
Formulations of flumioxazin are available for use as pre-emergent or post-emergent broadcast or banded soil applications, directed inter-row band sprays in established crops and pre-harvest desiccants, both as solo products or co-formulated or tank-mixed with other herbicides.

Specifications for flumioxazin technical material have been established by the JMPS (2015) and published as FAO Specification 578, available on the FAO Website.

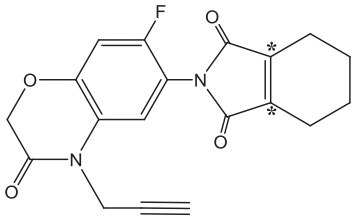
FORMULATION TYPE	FLUMIOXAZIN CONTENT	OTHER ACTIVE INGREDIENTS
WG (Water dispersible granule)	510 g/kg 500 g/kg 400 g/kg	chlorimuron ethyl
GR (Granule)	2.5 g/kg	

METABOLISM AND ENVIRONMENTAL FATE

The Meeting received flumioxazin metabolism studies on plants (soya beans, grapes, sugar cane, apples and peanuts), animals (rats, lactating goats and laying hens) and rotational crops (lettuce, carrots and wheat). Flumioxazin radio-labelled on the phenyl ring or the tetrahydrophthaloyl (THP) ring were used in these studies. The label positions (*) are shown below:

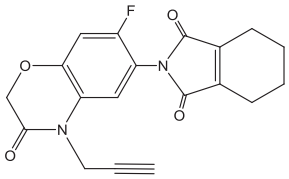
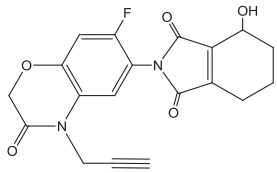
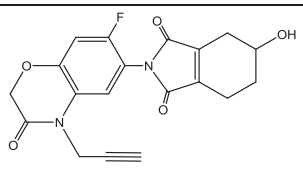


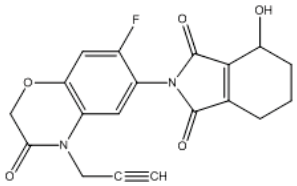
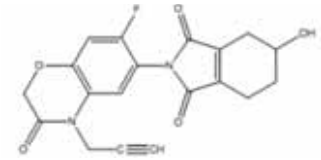
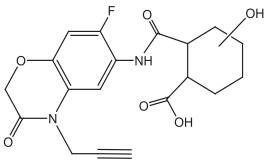
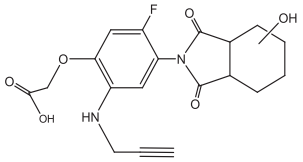
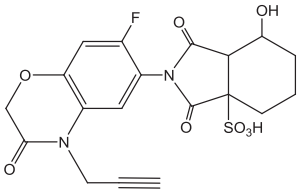
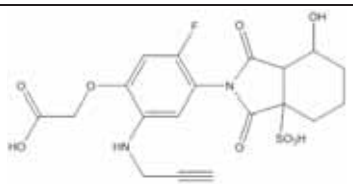
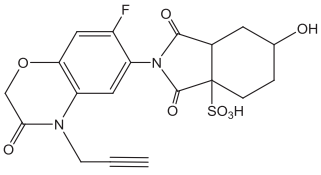
[phenyl-^{14}C]-flumioxazin

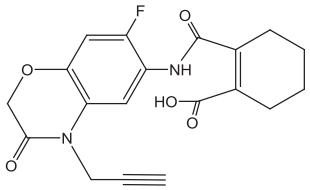
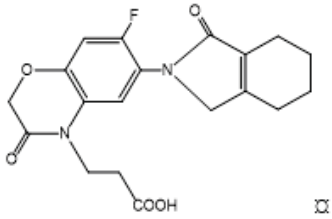
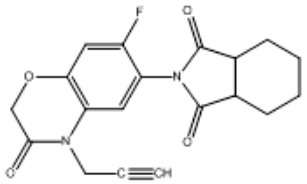
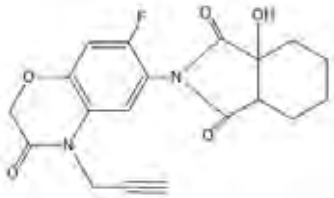
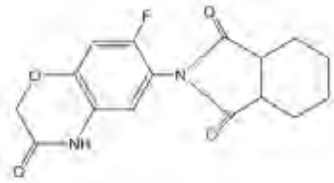
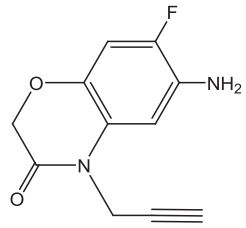
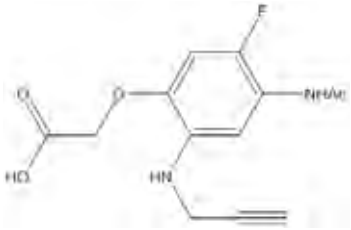


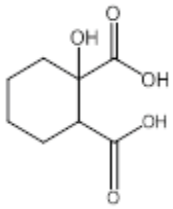
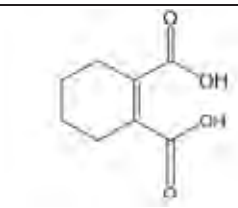
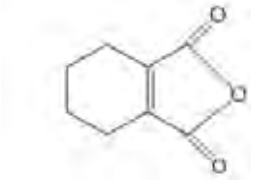
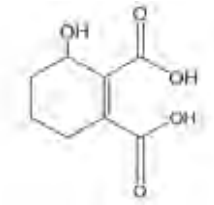
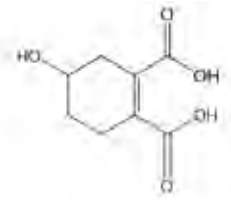
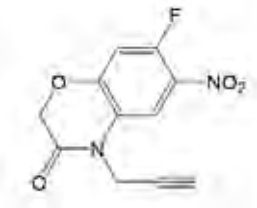
[THP-^{14}C]-flumioxazin

Major metabolites identified in these studies and discussed in this evaluation are listed below.

Compound Name/Code	Structure		Matrices
Flumioxazin (S-53482) (V-53482)		*N*-(7-fluoro-3,4-dihydro-3-oxo-4-prop-2-ynyl-2*H*-1,4-benzoxazin-6-yl)cyclohex-1-ene-1,2-dicarboxamide	Plants Goat Hen Rat Soil Photolysis
3-OH-Flumioxazin		7-fluoro-6-(3-hydroxy-3,4,5,6-tetrahydrophthalimido)-4-(2-propynyl)-2H-1,4-benzoxazin -3(4H)-one	Goat Hen Rat
4-OH-Flumioxazin		7-fluoro-6-(4-hydroxy-3,4,5,6-tetrahydrophthalimido)-4-(2-propynyl)-2H-1,4-benzoxazin-3(4H)-one	Goat Hen Rat

Compound Name/Code	Structure		Matrices
Metabolite B or metabolite F 3-OH-SAT-482		7-fluoro-6-(3-hydroxy-1,2-cyclohexanedicarboximido)-4-(2-propynyl)-2*H*-1,4-benzoxazin-3(4*H*)-one	Goat Rat
4-OH-SAT-482 Exponent asked for revised structures		7-fluoro-6-(4-hydroxy-1,2-cyclohexanedicarboximido)-4-(2-propynyl)-2*H*-1,4-benzoxazin-3(4*H*)-one	
Metabolite C	 OR 	not available	Goat
3-OH-Flumioxazin-SA		7-fluoro-6-(1-sulfo-3-hydoxy-1,2-cyclohexanedicarboximido)-4-(2-propynyl)-2H-1,4-benzoxazin-3(4H)-one	Goat Hen Rat
3-OH-Flumioxazin-ASA		5-fluoro-2-(2-propynylamino-4-(1-sulfo-3-hydroxy-1,2-cyclohexanedicarboximide)phenoxyacetic acid	Rat
4-OH-Flumioxazin-SA		7-fluoro-6-(1-sulfo-4-hydroxy-1,2-cyclohexanedicarboximido)-4-(2-propynyl)-2H-1,4 benzoxazin-3(4H)-one	Goat Hen Rat

Compound Name/Code	Structure		Matrices
482-HA		N-(7-fluoro-3,4-dihdyro-3-oxo-4-prop-2-ynyl-2H- 1,4-benzoxazin-6-yl)cyclohex-1-ene-1-carboxamide-2-carboxylic acid	Plants (rotational) Goat Rat Soil Photolysis
482-CA		2-[7-fluoro-3-oxo-6-(3,4,5,6-tetrahydrophthalimido)-2H-1,4-benzoxazin-4-yl] propionic acid	Plants (rotational) Soil
SAT-482		6-(cis-1,2-cyclohexanedicarboximido)-7-fluoro-4-(2-propynyl)2H-1,4-benzoxazin-3(4H)-one	Goat Rat
1-OH-SAT-482		not available	Plants (rotational)
IMOXA		2-[7-fluoro-3,4-dihydro-3-oxo-2H-1,4-benzoxazin-6-yl]-4,5,6,7-tetrahydro-1H-isoindole-1,3(2H)-dione	Plants (rotational) Soil Photolysis
APF		6-amino-7-fluoro-4-(2-propenyl)-2H-1,4-benzoxazin-3(4H)-one	Plants Goat Hen Rat Soil Photolysis
Ac-APFA		4-acetylamino-5-fluoro-2-(2-propynylamino)phenoxyacetic acid	Rat

Compound Name/Code	Structure		Matrices
1-OH-HPA		1-hydroxy-*trans*-1,2-cyclohexanedicarboxylic acid	Plants Rat Photolysis
THPA		3,4,5,6-tetrahydrophthalic acid	Plants Goat Hen Rat Soil Photolysis
Δ^1-TPA		3,4,5,6-tetrahydrophthalic anhydride	Plants (rotational) Hen Soil Photolysis
3-OH-THPA		3-hydroxy-1-cyclohexene-1,2-dicarboxylic acid	Hen
4-OH-THPA		4-hydroxy-1-cyclohexene-1,2-dicarboxylic acid	Goat Hen
PNF		7-fluoro-6-nitro-4-(2-propynyl)-2*H*-1,4-benzoxazin-3(4*H*)-one	Plants

Environmental fate

The Meeting received information the environmental fate and behaviour of flumioxazin, including hydrolytic stability, photochemical degradation in soils and aerobic metabolism studies.

Hydrolysis

The hydrolytic degradation of flumioxazin was investigated at pH 5, 7 and 9 using either [phenyl-^{14}C]-flumioxazin or [THP-^{14}C]-flumioxazin and reported by Katagi, 1990 [Ref: SBM-0005 and Ref: SBM-0006].

Radio-labelled flumioxazin (0.1 mg/L) was incubated in the dark in sterile aqueous buffered solutions at pH 5, 7, and 9 for up to 30 days at 25 °C. Samples were taken at regular intervals throughout the study and were analysed for total radioactivity by LSC. HPLC was used

to determine the hydrolysis rate and identify the degradation products. Further characterization of degradation products was carried out by two-dimensional TLC with reference standards. The hydrolytic half-lives at each pH were calculated from the analytical data.

In both studies, 94–105% of the applied radioactivity was recovered in all samples analysed. Flumioxazin was rapidly hydrolysed in all three buffered solutions and the half-lives were calculated to be about 3.4–5 days at pH 5, 19–26 hours at pH 7 and 14–23 minutes at pH 9.

The major degradation products after 30 days of incubation in the phenyl-label study were APF (87%) at pH 5; APF (80%) and 482-HA (8–10%) at pH 7; and 482-HA (99%) at pH 9. In the THP-label study, the major degradation products were THPA (96%) and Δ^1-TPA (2.5%) at pH 5; THPA (84%), Δ^1-TPA (6%) and 482-HA (8%) at pH 7; and 482-HA (96%) at pH 9.

Table 1 Major degradation products in aqueous solutions containing [^{14}C]flumioxazin after incubation in the dark at 25 °C for 30 days

DEGRADATION PRODUCTS	% APPLIED RADIOACTIVITY					
	PH 5		PH 7		PH 9	
	PHENYL-LABEL	THP-LABEL	PHENYL-LABEL	THP-LABEL	PHENYL-LABEL	THP-LABEL
Flumioxazin						
1 hr					15	5.5
2 hrs			92	94		
8 hrs	91	89	80	77		
1 days	81	75	41	32	< 0.1	< 0.1
3 days	57	51	25	20	< 0.1	< 0.1
7 days	31	23	20	16	< 0.1	< 0.1
30 days	< 0.1	< 0.1	5.8	3.5	< 0.1	< 0.1
482-HA						
1 hr					84	95
2 hrs			5.1	6.2		
8 hrs	5.3	5.9	19	24		
1 days	4.7	4.2	53	63	99	98
3 days	3.5	2.9	59	68	100	101
7 days	2.8	< 0.1	46	50	99	102
30 days	< 0.1	< 0.1	10	8.1	99	96
THPA						
1 hr						< 0.1
2 hrs				< 0.1		
8 hrs		5.4		< 0.1		
1 days		18		3.3		< 0.1
3 days		47		13		< 0.1
7 days		76		34		< 0.1
30 days		96		84		< 0.1
Δ^1-TPA						
1 hr						< 0.1
2 hrs				< 0.1		
8 hrs		< 0.1		< 0.1		
1 days		< 0.1		< 0.1		< 0.1
3 days		< 0.1		< 0.1		< 0.1
7 days		1.5		0.2		< 0.1
30 days		2.5		6.0		< 0.1

DEGRADATION PRODUCTS	% APPLIED RADIOACTIVITY					
	PH 5		PH 7		PH 9	
	PHENYL-LABEL	THP-LABEL	PHENYL-LABEL	THP-LABEL	PHENYL-LABEL	THP-LABEL
APF						
1 hr						
2 hrs			< 0.1		< 0.1	
8 hrs	4.7		< 0.1			
1 days	13		3.8			
3 days	39		15		< 0.1	
7 days	64		33		< 0.1	
30 days	87		80		< 0.1	

The proposed degradation pathway involves hydrolysis to the amide 482-HA, with further cleavage of the amide link occurring at pH 7 or below, forming THPA (and its anhydride Δ^1-TPA) and APF.

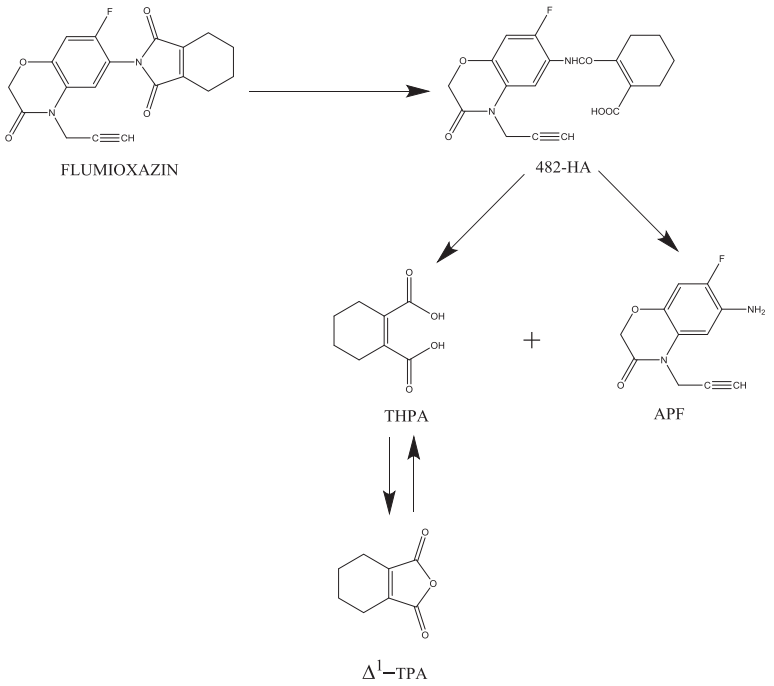


Fig 1 Proposed degradation pathway of flumioxazin in aqueous solutions

Photochemical degradation in soil

Artificial sunlight photo-degradation of [phenyl-^{14}C]-flumioxazin and [THP-^{14}C]-flumioxazin in sandy loam soils was investigated in two studies reported by Fathulla, 1993 [Ref: SBM-0029 and Ref: SBM-0035], respectively.

In these studies, the radio-labelled flumioxazin was applied in acetonitrile to thin layers (1–2 mm thick) of similar Californian sandy loam soils (61–63% sand, 29–30% silt, 8–9% clay, 0.9–1.4% O.M., pH 7.6–7.9) and the soil moisture was adjusted to 75% FC. Samples were irradiated (xenon lamp) for about 12 hours/day at 25–28 °C and duplicate samples were analysed immediately after fortification (Day 0) and intervals for the next 6–14 days.

Soil samples were extracted with acetone:water (5:1, v/v) and then with acidified (pH 1) acetone:water, and analysed using thin-layer chromatography. In the phenyl-label study, the post-

extracted samples containing more than 10% AR were more exhaustively extracted by acid then base refluxing in methanol or by refluxing in dimethylformamide/oxalic acid then basic methanol.

The mean recovery of the applied radioactivity in both studies ranged from 89% to 108%. Volatiles did not exceed 0.5% of the applied radiocarbon for the irradiated samples or 0.2% for the dark controls.

In the phenyl-label study, the acetone extracts from the Day-0 samples contained 102% AR and this decreased in the Day-6 irradiated samples to 48% AR (86% AR in the dark control samples). The more aggressive reflux treatments were able to extract most of the remaining residue, leaving less than 10% AR unextracted.

In the THP-label study, the radioactivity in the combined acetone:water extracts decreased from an initial 99% AR to 83% AR (irradiated) and 87% AR (dark controls) by the end of the 14-day study period, with an increase in the amount of ^{14}C bound to soil, up to 9.3% AR in the irradiated samples and 5% AR in the dark controls.

Flumioxazin accounted for 97–99% AR in the Day-0 samples, decreasing in the irradiated samples to 29% (Day 6—phenyl-label) and 82% AR (Day 7—THP-label) and to 37% AR in the THP-label samples on Day 14. No other TLC areas of radioactivity were more than 10% AR except for Δ^1-TPA and THPA.

Levels of Δ^1-TPA peaked at 22% AR on Day 9 in the irradiated samples, but were < 10% AR in all other sampling times (and in all dark control samples). THPA reached a maximum of about 13% AR (10% AR in the dark control samples). Other minor components were identified as IMOXA and 1-OH-HPA, both measured at < 4% AR in the irradiated samples.

Table 2 Photochemical degradation on soil of [^{14}C]flumioxazin in a Californian sandy loam soil at 25 °C

COMPONENT	% APPLIED RADIOACTIVITY						
	DAY 0	DAY 6–7 [A]		DAY 9		DAY 14	
		IRRADIATED	DARK	IRRADIATED	DARK	IRRADIATED	DARK
Phenyl-label							
Flumioxazin	96.9	29.1	68.4				
IMOXA	0.8	3.1	3.8				
APF + 482-HA	1.4	0.6	ND				
CO_2	< 0.1	0.5	0.2				
Unextracted	3.0	43.3	17.1				
Recovery	105.1	92.3	103.2				
THP-label							
Flumioxazin	99.2	82.2	89.4	36.9	81.5	37.0	51.7
Δ^1-TPA	ND	5.2	3.8	21.6	2.6	8.6	9.0
THPA	ND	1.7	0.2	7.4	10.2	12.9	7.7
1-OH-HPA	ND	ND	ND	3.0	1.5	4.4	8.3
CO_2	ND	ND	ND	ND	ND	ND	ND
Unextracted	1.7	3.1	1.4	4.7	2.3	9.3	5.0
Recovery	100.9	98.3	99.2	93.9	100.5	92.4	92.1

[a] Samples taken on Day 6 in the phenyl-label study and Day 7 in the THP-label study

Radio-labelled flumioxazin degraded more rapidly on irradiated soil than on dark soil, with the amount of ^{14}C bound to soil increasing over time. The calculated soil degradation half-lives were 3.2 days (phenyl-label study) and 8.4 days (THP-label study) and were 12–16 days in non-irradiated samples.

The proposed degradation pathways include hydrolysis of the parent to the amide 482-HA, with further cleavage of the amide link, forming THPA (and its anhydride Δ^1-TPA) and then 1-OH-HPA and also the dealkylation of flumioxazin to form IMOXA.

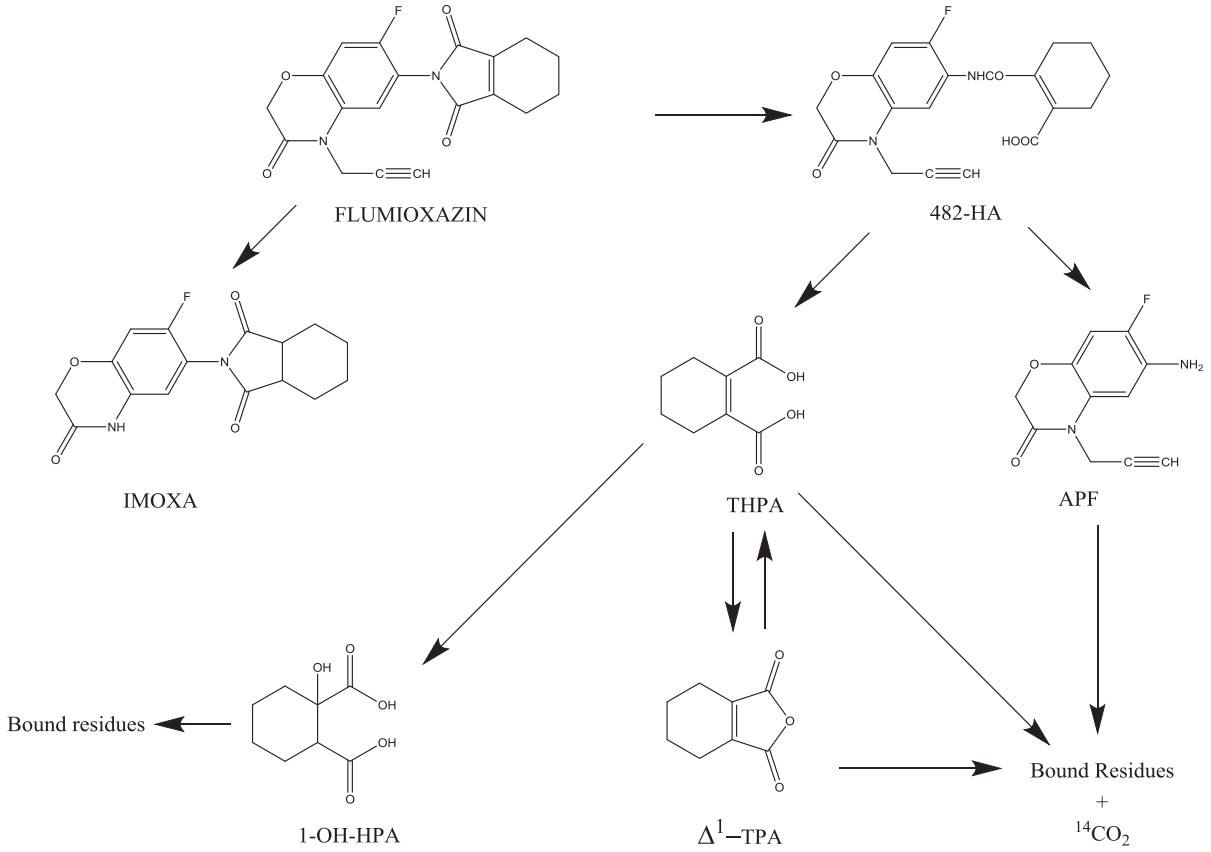


Fig 2 Metabolic pathway proposed for photochemical degradation of flumioxazin on soil

Aerobic soil metabolism

The degradation of flumioxazin in soil was investigated under aerobic conditions using phenyl-labelled flumioxazin (Fathulla, 1991 [Ref: SBM-0012]) and using THP-labelled flumioxazin ((Fathulla, 1993 [Ref: SBM-0030]). In these studies, radio-labelled flumioxazin was applied to sieved, sandy loam soils at a rate of 0.25–0.26 mg/kg, equivalent to about 0.3 kg ai/ha (7.6 cm depth). The characteristics of the soils are summarized below.

Table 3 Characteristics of the soils used in the flumioxazin aerobic soil metabolism studies

SOIL CHARACTERISTICS	PHENYL-LABEL STUDY	THP-LABEL STUDY
Soil type	Sandy Loam	Sandy Loam
Sand	67%	61.2%
Silt	29%	30%
Clay	4%	8.8%
Organic matter	1.2%	1.4%
Cation exchange capacity	18 meq/100 g	6.4 meq/100 g
pH (H_2O)	7.8	7.9
Field moisture capacity	8.9% (at 0.33 bar)	13.4

The soil samples were incubated at 25 °C in glass chambers maintained in a dark, temperature-controlled room for up to 181 days in the phenyl-label study and up to 91 days in the

THP-label study. The glass chambers were connected to traps containing charcoal, ethylene glycol and 2-ethoxyethanol:ethanolamine (1:1, v/v) for collection of volatile organic components and carbon dioxide. Samples were collected for analysis of radioactivity on Day 0 and at various intervals throughout the study periods and extracted with acetone:water (5:1, v/v) and acetone: 0.1 N HCl (9:1, v/v). The combined extracts were analysed by LSC and the distribution of radioactivity in the samples was determined by two-dimensional TLC, HPLC, and comparison with reference standards. The radioactivity remaining in the soil was determined by combustion and LSC. Residues were further extracted with acetonitrile: 0.25 N HCl (4:1, v/v) then 0.5 N sodium hydroxide in the phenyl-label study and acetonitrile: methanol:0.1 N HCl (25:15:10) followed by 0.5 N sodium hydroxide in the THP-label study, with analysis by TLC or LSC. Extraction efficiencies ranged from 94–102% in the two studies.

Radioactivity was distributed primarily among unchanged flumioxazin, CO_2 and soil-bound residues with minor identified components being 482-HA, 482-CA, APF, Δ^1-TPA, THPA and IMOXA. None of these individually exceeded 8% AR. Radioactivity recovered as CO_2 accounted for 12% of the applied radioactivity by Day 181 in the phenyl-label study and accounted for 55% AR at the end of the 91-day TPH-label study period.

Flumioxazin accounted for about 3.5% of the applied radioactivity in phenyl-label soils incubated for 89–181 days, and about 12% AR in the THP-label soils incubated for 90 days and the calculated half-lives in the respective studies were 12 days and 17.5 days. Calculated DT_{90} values (FOMC) were about 51 days (phenyl-label) and 95 days (THP-label).

Table 4 Aerobic degradation of [^{14}C]flumioxazin in a Californian sandy loam soil at 25 °C

COMPONENT	% APPLIED RADIOACTIVITY (0.25-0.26 MG/KG) [A]								
	DAY 0	DAY 3	DAY 7	DAY 14	DAY 28–30	DAY 59–63	DAY 89–91	DAY 120	DAY 181
Phenyl-label									
Flumioxazin	92.9	68.4	60	36.3	18.0	7.6	3.2	3.5	3.7
Origin		1.3	2.4	4.1	8.1	3.9	2.5	2.4	2.8
Region 1			0.4	0.3		2.3 [b]	0.5		
Region 2			0.3	0.3		2.2 [c]	0.1		
Region 3					4.6		5.1	5.5	1.4
Unresolved	6.5	10.8	8.8	17.5	9.2	5.8	4.6	1.1	1.9
Total extracted	99.4	80.5	71.9	58.5	39.9	21.8	16	12.5	9.8
Unextracted	0.7	16.9	25.8	43.0	52.7	71.3	70.0	73.9	73.6
CO_2	–	0.1	0.2	0.6	2.3	5.6	7.7	9.2	11.5
Recovery	100.1	97.4	97.8	102.1	94.9	98.7	94.2	95.8	95.4
THP-label									
Flumioxazin	97.3	78.4	63.6	51.4	28.9	12.3	11.8	–	–
THPA		6.6	5.7	1.0		0.7		–	–
Δ^1-TPA		4.6	5.1	4.8	2.1	0.3		–	–
IMOXA				1.6	2.7	3.0	2.0	–	–
Unresolved		4.2	7.1	2.4	7.0	8.4	1.7	–	–
Total extracted	97.3	93.8	81.5	61.2	40.7	24.7	15.5	–	–
CO_2	-	1.5	7.7	18.4	33.9	48.9	54.9	–	–
Unextracted	2.7	3.9	12.1	16.5	20.0	23.7	29.0	–	–
Recovery	100	99.6	101.3	97.9	96.4	97.5	100.1	–	–

[a] Mean of duplicate samples

[b] Identified as IMOXA plus an unknown component

[c] Identified as 482-HA and 482-CA

Flumioxazin degrades in aerobic soil with calculated half-lives of 12–18 days, with degradation products being CO_2 and a number of minor soil-bound degradates. The proposed metabolic pathways include hydrolysis of the parent compound to 482-HA, oxidation to 482-CA, and by dealkylation to IMOXA. Both IMOXA and 482-HA hydrolyse to THPA, which would be in equilibrium with Δ^1-TPA. THPA appears to be an end product that is incorporated into soil organic components or oxidized to CO_2.

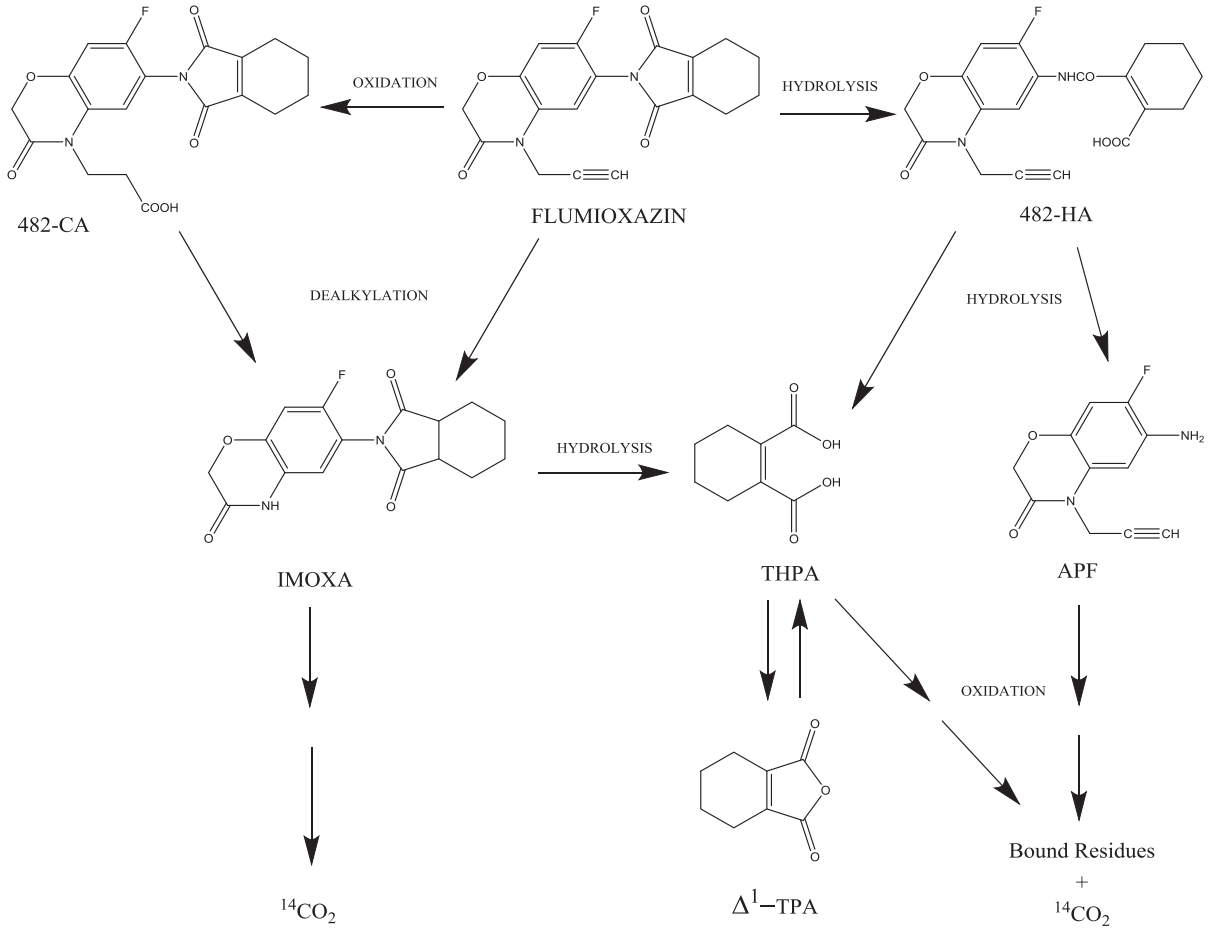


Fig 3 Metabolic Pathway for Aerobic Degradation of Flumioxazin in Soil

Flumioxazin is rapidly hydrolysed in aqueous solutions with average half-lives of 4–5 days (pH 5) decreasing to about 20 minutes at pH 9. Degradation products include 482-HA, THPA (and its anhydride Δ^1-TPA) and APF. The compound 482-HA was the predominant degradate (> 97%) in the pH 9 solution and the cleavage compounds APF and THPA were the major components in the pH 5 and 7 solutions.

Radio-labelled flumioxazin degraded more rapidly on irradiated soil than on dark soil, with the amount of ^{14}C bound to soil increasing over time. THPA and its anhydride Δ^1-TPA together accounted for up to 29% AR in irradiated samples (up to 17% in dark samples). The calculated soil degradation half-lives were 3.2 days (phenyl-label study) and 8.4 days (THP-label study) and were 12–16 days in non-irradiated samples.

In aerobic soil, calculated half-lives for flumioxazin are 12–18 days, with degradates 482-HA, 482-CA and IMOXA, each accounting for less than 7% of the applied radioactivity and generally present at < 0.01 mg/kg. The parent compound accounted for the majority of extractable radioactivity in almost all samples examined.

Plant metabolism

The Meeting received plant metabolism studies on soya beans, grapes, sugar cane, apples, peanuts and rotational crops following treatments with flumioxazin radio-labelled in the phenyl ring or the tetrahydrophthaloyl (THP) ring.

Grape

In a confined metabolism study on grape vines reported by Goodyear, 1998 [Ref: SBM-0064], flumioxazin, radio-labelled in the phenyl ring or the THP ring, was applied to soil surrounding grape vines at a rate equivalent to 0.6 kg ai/ha, the vines were grown to maturity in a glass house and at maturity (91 DAT), samples of grapes and shoots were extracted with acetone:water (1:1, v/v) and radioactivity in the extracts was measured by liquid scintillation counting (LSC) and by combustion analysis in the post-extraction solids.

Total radioactivity in the mature grapes and shoots were extremely low. The mean levels of radioactivity in grapes were 0.0021 mg/kg (-phenyl label) and 0.0054 mg/kg (THP-label) and in the shoots, radioactivity measured 0.014 mg/kg (-phenyl label) and 0.04 mg/kg (THP-label).

The majority of the residue (78–92%) was extracted into acetone or acetone:water with 9–21% of the residue remaining "bound" to the plant material. HPLC analysis of the aqueous extracts indicated the presence of a number of metabolites, the majority of which were polar in nature and were not retained on the column under the chromatographic conditions used. The polar fraction contained about 58% TRR, one other metabolite was present at about 11–14% TRR and eight other components were each present at < 6% TRR. Co-chromatography of the radioactivity with the known standards was not possible due to the high levels of UV-absorbing co-extracted samples.

Apple

In a metabolism study on apples reported by Jalal, 2003 [Ref: SBM-0073], flumioxazin, radio-labelled in the phenyl ring or the THP ring, was applied twice as broadcast sprays to bare soil (1.2 m × 1.2 m loamy sand plots) surrounding 4 year-old trees, with about 30 cm of tree trunk receiving direct spray. Treatments equivalent to 0.47 kg ai/ha were applied 47 days before fruit thinning and 60 days later (about 60 days before fruit maturity).

Apples were sampled and analysed at tree thinning (immature apples) and at harvest (mature apples. Combustion analysis was validated using spiked control apples, with a recovery rate of about 95%.

Total radioactive residues (TRR) were 0.002 mg/kg in immature apples from either the [phenyl-^{14}C]flumioxazin treated plot or from the [THP-^{14}C]flumioxazin treated plot. TRRs were 0.001 mg/kg in the mature apples from the [phenyl-^{14}C]flumioxazin treated plot and 0.003 mg/kg in apples from the [THP-^{14}C]flumioxazin treated plot. Since these residue levels were extremely low, further characterization or identification of the residues could not be conducted.

Table 5 Radioactive residues in apples following 1–2 soil/trunk applications of [^{14}C]flumioxazin at rates equivalent to 0.47 kg ai/ha

TREATMENT	TOTAL RADIOACTIVITY (MG/KG)	
	Immature apple (47 days after 1st application)	Mature apple (60 days after 2nd application)
Control	< 0.001	< 0.001
Phenyl-label	0.002	0.001
THP-label	0.002	0.003

Peanut

In a metabolism study on peanuts reported by Comezoglu, 1994 [Ref: SBM-0044], flumioxazin radio-labelled in the phenyl ring or the THP ring was applied once as a pre-emergent broadcast soil treatment at rates equivalent to 0.11 kg ai/ha (3 days after sowing) or 0.33 kg ai/ha as a pre-plant treatment, 32 day before sowing (treated plots were re-sown following poor initial crop emergence). Treatments were made by mixing the labelled flumioxazin into soil (sandy loam) taken from each plot and adding the treated soil back to the tops of the respective plots.

Samples of mature foliage and whole peanuts were harvested from the 0.11 kg ai/ha plots 194 days after treatment (DAT) and from the 0.33 kg ai/ha plots 245 days after resowing (277 DAT). Samples of foliage (vines) were frozen immediately after sampling. Whole peanuts were washed to remove adhering soil and separated into hulls, seed coats and nutmeats. Samples were frozen and shipped on dry ice by overnight courier to the analytical laboratory where samples were stored at < –10 °C prior to analysis.

Samples were homogenized with dry ice and total radioactive residues (TRR) were measured by combustion and LSC analysis.

Total radioactive residues (TRR) in all matrices from the 0.11 kg ai/ha pre-plant treatment were < 0.04 mg/kg, with ^{14}C-residues being lower in the phenyl-label samples. TRRs in samples from the 0.33 kg ai/ha pre-plant treatment were ca.3× higher than those from the 0.11 kg ai/ha treatment except for the phenyl-label hulls and the THP-label vines. Radioactive residues were generally lowest in vines (0.009–0.027 mg/kg) and highest in hulls (0.019–0.166 mg/kg).

Table 6 Radioactive residues in peanut matrices following single pre-plant or pre-emergence soil treatments of [^{14}C]flumioxazin

MATRIX	TOTAL RADIOACTIVITY (MG/KG FLUMIOXAZIN EQUIVALENTS)			
	PRE-EMERGENT TREATMENT (0.11 KG AI/HA) 3 DAYS AFTER SOWING, SAMPLED 194 DAT		PRE-PLANT TREATMENT (0.33 KG AI/HA) 32 DAYS BEFORE SOWING, SAMPLED 277 DAT	
	PHENYL-LABEL	THP-LABEL	PHENYL-LABEL	THP-LABEL
Nutmeats	0.012	0.031	0.044	0.085
Hulls	0.019	0.02	0.166	0.097
Vines	0.009	0.021	0.027	0.023
Seed coats	0.013	0.036	0.045	0.093

Samples were also extracted with acetone:water (4:1) and partitioned with hexane, with total radioactivity in the extracts and the post-extraction solids being measured by combustion and LSC analysis. Radioactivity in the hexane fraction from vines and hulls was too low (≤ 0.002 mg/kg) to permit further characterisation or identification.

The hexane fractions from the nutmeat samples were further partitioned between hexane:acetonitrile (1:1), with essentially all the radioactivity remaining in the hexane phase. Attempts to separate this radioactivity from the oil fraction by freezing to precipitate fats or by chromatography using a silica gel, C_{18}, or gel permeation columns were not successful. However, data from extraction of control nutmeat samples fortified with flumioxazin indicated that parent is unlikely to be present in this fraction.

The aqueous fractions from all samples (except the vines from the pre-plant treatment) were acidified to pH 2–3 and partitioned with ethyl acetate (EtOAc), and selected fractions from various samples were then analysed by reverse-phase HPLC.

Following solvent extraction, the majority of ^{14}C-residues in nutmeats (67–83% TRR), hulls (62–69% TRR) and vines (51–59% TRR) remained in the post extraction solids. To further characterize these residues, the post-extraction solids (PES) fractions from the pre-emergence treatment samples were subjected to sequential enzymatic (cellulase), acid (2 N HCl) and base

(2 N NaOH) hydrolyses. Radioactive residues remaining in the final PES fractions accounted for 23–35% TRR (0.003–0.01 mg/kg) in nutmeats and hulls and 6.4–8.6% TRR (0.001–0.002 mg/kg) in vines.

Radioactive residues in selected aqueous, organic and hydrolysate fractions containing ≥ 10% of the TRR were analysed by reverse phase HPLC using a C_{18} column. Radioactive residues were detected and quantified by LSC and reference standards were detected using a UV absorbance detector (220 nm). Peak retention times for ^{14}C-residues were compared to retention times of reference standards. HPLC peaks containing significant amounts of radioactivity were also analysed by TLC using silica gel plates with a variety of solvent systems.

Flumioxazin residues were measured at levels of < 1% TRR (< 0.001 mg/kg) in the ethyl acetate fractions from hulls and vines. The majority of ^{14}C-residues in solvent and hydrolysate fractions was generally comprised of four regions (A, B, C, and D). Regions A and B were polar in nature and did not correspond to any of the reference standards used in the study. Region C was typically a broad peak, suggesting multiple components, such as 1-OH-HPA, THPA, APF, and 482-HA. Region D was a minor peak with peaks similar to the standards IMOXA, PNF, and 482-CA.

In the nutmeats and vine extracts, each of these general regions accounted for ≤ 0.01 mg/kg in each fraction analysed by HPLC. These regions also each accounted for ≤ 0.005 mg/kg in fractions from hulls, with the exception of Region C which accounted for 0.025–0.038 mg/kg in solvent extracts from the pre-plant (0.33 kg ai/ha) hulls. Subsequent TLC analyses suggested that this region contained minor levels of 1-OH-HPA (≤ 4% TRR, ≤ 0.006 mg/kg) and THPA (≤ 2% TRR, ≤ 0.004 mg/kg) in hulls and vines from the pre-plant samples, however, the majority of ^{14}C-residues in Region C were multiple unknown polar components.

Table 7 Distribution of radioactive residues in peanut nutmeat following one pre-plant or pre-emergent soil application of [^{14}C]flumioxazin

		TOTAL RADIOACTIVITY (MG/KG FLUMIOXAZIN EQUIVALENTS)							
FRACTION	HPLC	PHENYL-LABEL				THP-LABEL			
		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)	
		%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
Acetone/water		24.2	0.002	32.8	0.014	16.9	0.005	29.3	0.027
1st hexane		12.2	0.001	23.0	0.01	6.4	0.002	16.5	0.015
2nd hexane		12.0	0.001	22.7	0.01			16.4	0.015
Acetonitrile		0.14	< 0.001	0.33	< 0.001			0.07	< 0.001
1st aqueous		12.0	0.001	9.8	0.004	10.6	0.003	12.9	0.012
	Region A			5.8	0.002				
	Region B			ND	ND				
	Region C			4.0	0.002				
	Others			ND	ND				
2nd aqueous		8.4	0.001			7.1	0.002	5.7	0.005
	Region A							3.5	0.003
	Region B							0.5	< 0.001
	Region C							1.7	0.002
	Others							ND	ND
Ethyl acetate		3.5	< 0.001			3.5	0.001	7.2	0.007
PES-1		75.9	0.009	67.2	0.03	83.1	0.026	70.7	0.066
Enzyme filtrate		13.9	0.002			15.6	0.005		
	Region A	5.4	< 0.001			7.8	0.002		
	Region B	ND	ND			ND	ND		
	Region C	7.7	0.001			7.7	0.002		

		TOTAL RADIOACTIVITY (MG/KG FLUMIOXAZIN EQUIVALENTS)							
FRACTION	HPLC	PHENYL-LABEL				THP-LABEL			
		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)	
		%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
	Others	0.86 (4)	< 0.001			0.07 (1)	< 0.001		
PES-enzyme		61.9	0.007			67.4	0.021		
Acid-aqueous		22.9	0.003			23.2	0.007		
	Region A	14.4	0.002			16.3	0.005		
	Region B	0.5	< 0.001			ND	ND		
	Region C	1.2	< 0.001			1.5	< 0.001		
	Others	0.4 (5)	< 0.001			0.1 (1)	< 0.001		
	MeOH eluate	6.4	0.001			5.2	0.002		
Acid-EtOAc		5.8	0.001			7.5	0.002		
PES-acid		33.2	0.004			36.8	0.011		
Base-aqueous		8.9	0.001			3.8	0.001		
Base-EtOAc									
PES-base		24.3	0.003			32.9	0.01		

Fractions indicated in bold were analysed by HPLC.

Numbers of other peaks listed in brackets

Regions A and B were polar in nature, not corresponding to any reference standards

Region C, a broad peak possibly including 1-OH-HPA, THPA, APF, and 482-HA

Table 8 Distribution of radioactive residues in peanut hulls following one pre-plant or pre-emergent soil application of [^{14}C]flumioxazin

	TOTAL RADIOACTIVITY (MG/KG FLUMIOXAZIN EQUIVALENTS)								
FRACTION		PHENYL-LABEL				THP-LABEL			
		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)	
		%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
Acetone/water		34.5	0.006	30.9	0.051	31.2	0.006	38.2	0.037
1st hexane		1.0	< 0.001	0.75	0.001	1.2	< 0.001	1.1	0.001
1st aqueous		33.5	0.006	30.2	0.05	30.0	0.006	37.1	0.036
2nd aqueous		18.5	0.004	10.8	0.018	17.0	0.003	13.1	0.013
	Region A	5.2	< 0.001	1.7	0.003	3.8	< 0.001	2.2	0.001
	Region B	ND	ND	0.18	< 0.001	ND	ND	1.1	< 0.001
	Region C	13.3	0.003	8.6	0.014	13.1	0.002	9.8	0.009
	Others	ND	ND	0.36 (2)	0.001	0.08 (1)	< 0.001	ND	ND
Ethyl acetate		15.0	0.003	19.4	0.032	13.1	0.003	24.0	0.023
	Region A	0.54	< 0.001	1.8	0.003	ND	ND	4.3	0.004
	Region B	ND	ND	ND	ND	1.4	< 0.001	2.0	0.002
	Region C	12.1	0.002	14.8	0.024	9.7	0.002	16.3	0.016
	Region D	0.99	< 0.001	0.54	< 0.001	0.71	< 0.001	0.57	< 0.001
	Flumioxazin	0.55	< 0.001	0.68	< 0.001	0.48	< 0.001	0.67	< 0.001
	Others	0.88 (3)	< 0.001	1.6 (3)	< 0.001	0.77 (3)	< 0.001	0.18 (1)	< 0.001
PES-1		65.5	0.012	69.1	0.115	68.8	0.014	61.8	0.06
Enzyme filtrate		10.3	0.002			7.7	0.002		
	Region A	4.5	< 0.001						

	TOTAL RADIOACTIVITY (MG/KG FLUMIOXAZIN EQUIVALENTS)								
FRACTION		PHENYL-LABEL				THP-LABEL			
		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)	
		%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
	Region B	2.3	< 0.001						
	Region C	3.2	< 0.001						
	Region D	ND	ND						
	Others	0.31 (2)	< 0.001						
PES-enzyme		55.2	0.01			61.2	0.012		
Acid-aqueous		14.8	0.003			11.9	0.002		
	Region A	7.7	0.001			8.4	0.002		
	Region B	ND	ND			ND	ND		
	Region C	1.0	< 0.001			0.73	< 0.001		
	Region D	ND	ND			ND	ND		
	Others	2.7 (3)	< 0.001			ND	ND		
	MeOH eluate	3.47	0.001			2.8	< 0.001		
Acid-EtOAc		7.0	0.001			4.6	0.001		
PES-acid		33.4	0.006			44.7	0.009		
Base-aqueous		5.0	0.001			4.0	0.001		
Base-EtOAc		5.2	0.001			5.5	0.001		
PES-base		23.2	0.004			35.3	0.007		

Fractions indicated in bold were analysed by HPLC.

Numbers of other peaks listed in brackets

Regions A and B were polar in nature, not corresponding to any reference standards

Region C, a broad peak possibly including 1-OH-HPA, THPA, APF, and 482-HA

Region D, a minor peak possibly including IMOXA, PNF, and 482-CA.

Table 9 Distribution of radioactive residues in peanut vines following one pre-plant or pre-emergent soil application of [^{14}C]flumioxazin

		TOTAL RADIOACTIVITY (MG/KG FLUMIOXAZIN EQUIVALENTS)							
FRACTION		PHENYL-LABEL				THP-LABEL			
		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)	
		%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
Acetone/water		47.0	0.004	49.5	0.013	41.1	0.009	47.2	0.011
1st hexane		0.25	< 0.001	4.5	0.001	3.5	0.001	8.8	0.002
1st aqueous		46.7	0.004	45.1	0.012	37.6	0.008	38.4	0.009
	Region A			9.5	0.002			8.2	0.002
	Region B			ND	ND			3.4	< 0.001
	Region C			35.0	0.009			26.1	0.006
	Others			0.52 (1)	< 0.001			0.79 (3)	< 0.001
2nd aqueous		25.4	0.002			23.8	0.005		
	Region A	9.9	< 0.001			9.5	0.002		
	Region B	0.8	< 0.001			0.36	< 0.001		
	Region C	14.6	0.001			13.9	0.003		
	Others	0.11 (1)	< 0.001			0.05 (1)	< 0.001		
Ethyl acetate		21.3	0.002			13.8	0.003		

		TOTAL RADIOACTIVITY (MG/KG FLUMIOXAZIN EQUIVALENTS)							
FRACTION		PHENYL-LABEL				THP-LABEL			
		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)	
		%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
	Region A	0.27	< 0.001			ND	ND		
	Region B	2.0	< 0.001			1.3	< 0.001		
	Region C	14.9	0.001			9.8	0.002		
	Region D	0.63	< 0.001			0.33	< 0.001		
	Flumioxazin	0.16	< 0.001			ND	ND		
	Others	3.3 (3)	< 0.001			2.4 (5)	< 0.001		
PES-1		53.0	0.005	50.5	0.014	58.9	0.012	52.8	0.012
Enzyme filtrate		11.2	0.001			13.9	0.003		
	Region A	ND	ND			7.7	0.002		
	Region B	2.8	< 0.001			ND	ND		
	Region C	7.5	< 0.001			6.25	0.001		
	Region D	0.3	< 0.001			ND	ND		
	Others	0.56 (1)	< 0.001			ND	ND		
PES-enzyme		41.9	0.004			45.0	0.009		
Acid-aqueous		17.8	0.002			18.1	0.004		
	Region A	9.8	< 0.001			14.8	0.003		
	Region B	ND	ND			ND	ND		
	Region C	1.1	< 0.001			0.95	< 0.001		
	Region D	ND	ND			ND	ND		
	Others	0.07 (1)	< 0.001			0.04	< 0.001		
	MeOH eluate	6.9	0.001			2.4 (1)	0.001		
Acid-EtOAc		5.7	0.001			8.9	0.002		
PES-acid		18.3	0.002			18.0	0.004		
Base-aqueous		6.9	0.001			5.5	0.001		
Base-EtOAc		5.0	0.001			3.9	0.001		
PES-base		6.4	0.001			8.6	0.002		

Fractions indicated in bold were analysed by HPLC.

Numbers of other peaks listed in brackets

Regions A and B were polar in nature, not corresponding to any reference standards

Region C, a broad peak possibly including 1-OH-HPA, THPA, APF, and 482-HA

Region D, a minor peak possibly including IMOXA, PNF, and 482-CA.

Soya bean—Study 1

In a confined metabolism study on soya beans reported by Hubert, 1992 [Ref: SBM-0021], flumioxazin, radio-labelled in the phenyl ring or the THP ring, was applied to soil (sandy loam) three days after sowing at rates equivalent to 0.1 kg ai/ha or 0.2 kg ai/ha. Forage and root samples were taken 70 days after treatment and samples of plants (without pods), pods, seeds and roots were harvested at maturity, 100 days after treatment.

Soya bean forage and seed samples were extracted with acetone:water (4:1) followed by acetone:0.1 M HCl (4:1). The concentrated extracts were partitioned with ethyl acetate and the radioactivity quantified by LSC. The radioactivity in the post-extraction solids was determined by oxidation and LSC. In order to liberate further amounts of radioactivity, successive hydrolysis with 2 N HCl and 2 N sodium hydroxide under reflux (2 hours) was carried out. Following hydrolysis, the aqueous phases were acidified (pH 2–3), extracted with ethyl acetate and

radioactivity in the extracts and post-extraction solids was measured by liquid scintillation counting (LSC). Residues in the post-extraction solids were also analysed by HPLC.

Analysis of the radioactivity in the immature forage and mature plants, pods and seeds indicated preferential uptake from the ^{14}C-THP-radio-labelled material. Total radioactive residues in immature forage were 0.03 mg/kg and 0.06 mg/kg flumioxazin equivalents for the low and high rates of ^{14}C-phenyl-labelled material, respectively. The corresponding values for ^{14}C-THP-labelled treatments were 0.12 mg/kg and 0.14 mg/kg flumioxazin equivalents. Hay from immature forage (dried for 3–7 days to achieve a moisture content of 8.5–12%) contained 0.19 and 0.29 mg/kg flumioxazin equivalents for the high rate ^{14}C-phenyl and ^{14}C-THP treatments, respectively. In pods and seeds, radioactivity levels were 0.02–0.03 mg/kg (phenyl-label) and 0.23–0.36 mg/kg and 0.12–0.18 mg/kg respectively in the THP-label treatments.

Table 10 Radioactive residues in soya bean forage, pods and seeds following a pre-emergent soil application of [^{14}C]flumioxazin

MATRIX	DOSE (KG AI/HA)	RADIOACTIVE RESIDUES (MG/KG FLUMIOXAZIN EQUIVALENTS)			
		70 DAT		100 DAT	
		[PHENYL-^{14}C]-FLUMIOXAZIN	[THP-^{14}C]]-FLUMIOXAZIN	[PHENYL-^{14}C]]-FLUMIOXAZIN	[THP-^{14}C]]-FLUMIOXAZIN
Forage (immature)	0.1	0.03, 0.03	0.13, 0.11		
	0.2	0.07, 0.05	0.12, 0.16		
Hay (immature)	0.1	–	–		–
	0.2	0.19	0.29	–	–
Plants (without pods)	0.1			0.05, 0.04	0.22, 0.3
	0.2			0.06, 0.08	0.29, 0.4
Pods	0.1			0.03, 0.02	0.2, 0.26
	0.2			0.03, 0.02	0.29, 0.42
Seeds	0.1			0.03, 0.02	0.12, 0.13
	0.2			0.03, 0.03	0.17, 0.18

Sequential acetone:water and acetone:HCl extractions were able to extract close to 60% TRR in hay and when followed by acid and base hydrolysis in the case of the forage and seed, was able to extract more than 90% TRR in immature forage and more than 95% TRR in seed.

Table 11 Distribution of radioactive residues in soya bean forage, hay and seeds following one pre-emergent soil application equivalent to 0.2 kg ai [^{14}C]flumioxazin/ha

MATRIX	FORAGE (70 DAY)		SEED (100 DAY)		HAY (70 DAY)	
	% TRR	MG/KG	% TRR	MG/KG	% TRR	MG/KG
[phenyl-^{14}C]Flumioxazin						
Acetone:water	49	0.03	20.3	0.005	46.2	0.2
Acetone:HCl	16.7	0.01	4.7	0.001	12.6	0.02
Residue	29.3	0.02	68.9	0.02	34.5	0.07
Acid hydrolysis	17.7	0.01	49.1	0.009		
Base hydrolysis	3.5	0.002	8.6	0.002		
Unextracted residue	9.4	0.007	4.4	0.0008		
[THP-^{14}C]Flumioxazin						
Acetone:water	43.2	0.07	48.4	0.09		
Acetone:HCl	33.5	0.05	4.7	0.008		

MATRIX	FORAGE (70 DAY)		SEED (100 DAY)		HAY (70 DAY)	
	% TRR	MG/KG	% TRR	MG/KG	% TRR	MG/KG
Residue	27.4	0.04	49.1	0.09		
Acid hydrolysis	15.5	0.03	34.6	0.03		
Base hydrolysis	3.5	0.006	6.5	0.006		
Unextracted residue	6.6	0.01	3.3	0.003		

None of the radioactivity measured in forage, mature seeds or hay from immature forage could be identified as either the parent flumioxazin or any of the available reference standards. Analysis of some of the solubilized forage fractions indicated the presence of 10–16 unknown components that together accounted for 59–89% (0.017–0.086 mg/kg) TRR.

Soya bean—Study 2

In a further confined metabolism study on soya beans reported by Miyashita & Nambu, 1993 [Ref: SBM-0031], flumioxazin, radio-labelled in the phenyl ring or tetrahydrophthaloyl (THP) ring, was applied to sandy loam soil three days after sowing, at rates of about 0.1 kg ai/ha and 0.2 kg ai/ha. Samples of immature whole plants (forage) were taken 53 days after soil treatment and dried to prepare forage hay. Samples of seeds, pods and straw were harvested at maturity, 138 days after treatment.

Forage, hay and seed samples were extracted three times with acetone/water (4:1). The combined acetone/water extracts were concentrated and the aqueous remainder was partitioned three times with hexane. The aqueous remainder was adjusted to pH 2 with hydrochloric acid and partitioned three times into ethyl acetate. Finally the aqueous remainder was neutralised with sodium hydrogen carbonate. In each fraction, radioactivity was quantified by LSC and characterized by HPLC and TLC. The post-extraction solids were further extracted using cellulase digestion, acid and base hydrolysis with the liberated radioactivity being partitioned into ethyl acetate and quantified by LSC.

Total radioactive residues in immature forage (53 DAT) did not exceed 0.7% of the applied radioactivity, indicating that the radioactivity applied to the soil surface did not tend to translocate into the soya bean plants until a later stage. In the phenyl-label study, TRRs in forage from the 0.1 kg ai/ha and 0.2 kg ai/ha plots were about 0.06 mg eq/kg and 0.11 mg eq/kg respectively. Higher levels were present in the forage in the THP-label study (about 0.07 mg eq/kg and 0.2 mg eq/kg in the low and high rate plots). The TRR levels in hay were approximately three to four times higher compared to forage reflecting a concentration of residues due to the loss of water.

In mature soya bean seeds (138 DAT) in the phenyl-label study, TRRs were about 0.03 mg eq/kg (low rate) and about 0.06 mg eq/kg (high rate), and significantly higher in seeds from the equivalent plots in the THP-label study (about 0.25 mg eq/kg and 0.18 mg eq/kg respectively), indicating a preferential uptake of the THP-label.

Table 12 Total radioactive residues in soya bean forage, hay, seeds, pods and straw following a pre-emergent soil application of [^{14}C]flumioxazin

Matrix	Dose (kg ai/ha)	Radioactive residues (mg eq/kg flumioxazin)							
		53 DAT				138 DAT			
		[phenyl-^{14}C]-flumioxazin		[THP-^{14}C]-flumioxazin		[phenyl-^{14}C]-flumioxazin		[THP-^{14}C]-flumioxazin	
		mg eq/kg	%AR	mg eq/kg	%AR	mg eq/kg	%AR	mg eq/kg	%AR
Forage (immature)	0.1	0.055	0.6	0.069	0.7				
	0.2	0.108	0.7	0.196	0.5				
Hay (immature)	0.1	0.155		0.257					
	0.2	0.348		0.617					

Matrix	Dose (kg ai/ha)	Radioactive residues (mg eq/kg flumioxazin)							
		53 DAT				138 DAT			
Seeds	0.1					0.033	0.1	0.245	0.7
	0.2					0.055	0.1	0.177	0.9
Pods	0.1					0.06	0.1	0.326	1.7
	0.2					0.118	0.1	0.551	0.3
Straw	0.1					0.152	0.6	0.207	0.8
	0.2					0.176	0.3	0.254	0.6

Acetone:water extraction was able to retrieve 61–71% TRR from immature forage and hay and 36–66% TRR from seeds. Further partitioning and more aggressive cellulase digestion, acid and base hydrolysis was able to extract most of the remaining radioactivity, with about 1–4% TRR remaining in the post extraction solids.

Flumioxazin made up < 1.8–6.1% TRR in forage and hay, at levels of < 0.01 mg/kg in forage and up to 0.03 mg/kg in hay with trace levels (< 2.3% TRR, < 0.004 mg/kg) reported only in seed from the 0.2 kg ai/ha treatment in the THPA-label study.

The major component of the residue was metabolite 1-OH-HPA (free or partly cellulose conjugated), making up about 15–31% of the TRR in immature forage and hay and about 38–42% TRR (0.06–0.09 mg/kg) in seed. Minor metabolites included THPA (up to 8.6% TRR in forage and hay and < 3.2% TRR, < 0.007 mg/kg) in seeds) and 482-HA and APF found at trace amounts (< 1.8% TRR) in the immature commodities forage and hay.

Table 13 Characterization and identification of residues in soya bean forage 53 days after pre-emergence soil surface application with [^{14}C]flumioxazin

Metabolite	[Phenyl-^{14}C]Flumioxazin				[THP-^{14}C]Flumioxazin			
	100 g ai/ha		200 g ai/ha		100 g ai/ha		200 g ai/ha	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Acetone:water	0.039	69.2	0.081	70.5	0.044	61.2	0.124	70.8
1st Partition								
Hexane phase	0.007	12.7	0.012	10.7	0.006	7.9	0.017	9.5
Flumioxazin	0.004	6.1	0.006	5.5	< 0.001	< 1.8	0.008	4.4
APF	< 0.001	< 1.8	ND	ND				
Single unidentified	< 0.001	< 1.8	0.003	2.7	0.001	1.5	0003	1.4
Others (max & number)	< 0.001 (3)		0.002 (5)		< 0.001 (2)		0.002 (2)	
Others (total)	0.003	6.6	0.003	2.5	0.005	6.4	0.006	3.7
2nd Partition								
Ethyl acetate phase	0.020	35.3	0.043	37.4	0.032	43.7	0.087	49.7
Flumioxazin	< 0.001	< 1.0			< 0.001	< 1.6	< 0.002	< 1.4
482-HA	< 0.001	< 1.0	0.001	0.7	ND	ND	ND	ND
APF	ND	ND	< 0.001	< 0.5				
THPA					0.002	2.6	0.007	4.2
1-OH-HPA					0.011	15.3	0.028	15.8
Single unidentified	0.015	25.8	0.033	28.9	0.012	15.9	0.030	17.0
Others (max & number)	0.003 (22)		0.004 (32)		0.002 (12)		0.006 (11)	
Others (total)	0.005	9.5	0.009	7.8	0.007	9.9	0.022	12.7
Aqueous phase	0.012	21.2	0.026	22.4	0.006	9.6	0.020	11.6
Single unidentified	0.005	9.5	0.017	14.7	Not analysed		0.020	5.6
Others (max & number)	0.002 (7)		0.006 (12)				0.008 (3)	
Others (total)	0.007	11.7	0.09	7.7			0.010	6.0
Cellulase treatment								
Extract	0.006	9.9	0.008	7.2	0.011	15.6	0.029	16.8
1-OH-HPA	Not analysed				< 0.003	< 4.0	0.017	9.4
Single unidentified					0.006	7.9	0.005	2.8
Others (max & number)					< 0.003 (7)		0.004 (3)	
Others (total)					0.005	7.7	0.007	4.6
Acid hydrolysis								
Extract	0.006	9.9	0.013	11.7	0.010	13.9	0.015	8.8
Ethyl acetate phase	Not analysed		0.003	3.0	0.005	7.5	0.007	4.0

Metabolite	[Phenyl-^{14}C]Flumioxazin				[THP-^{14}C]Flumioxazin			
	100 g ai/ha		200 g ai/ha		100 g ai/ha		200 g ai/ha	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Aqueous phase			0.010	8.7	0.005	6.4	0.008	4.8
Alkaline hydrolysis								
Extract	0.004	6.8	0.008	7.4	0.005	7.3	0.005	2.7
PES	0.002	4.2	0.004	3.2	0.003	2.0	0.002	0.9
Total	0.057	100	0.114	100	0.073	100	0.175	100

ND = Non detectable

Table 14 Characterization and identification of residues in soya bean forage hay 53 days after pre-emergence soil surface application with [^{14}C]flumioxazin

Metabolite	[Phenyl-^{14}C]Flumioxazin				[THP-^{14}C]Flumioxazin			
	100 g ai/ha		200 g ai/ha		100 g ai/ha		200 g ai/ha	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Acetone:water	0.107	64.7	0.230	68.9	0.161	60.1	0.354	61.3
1st Partition								
Hexane phase	0.014	8.2	0.028	8.3	0.013	5.0	0.040	6.9
Flumioxazin	0.007	4.4	0.017	5.2	0.006	2.2	0.030	5.1
THPA					< 0.004	< 1.5	ND	ND
Single unidentified	< 0.002	< 1.0	0.005	1.5	ND	ND	0.010	1.8
Others (max & number)	< 0.002 (1)		0.003 (2)		ND		0.006 (2)	
Others (total)	0.007	3.8	0.006	1.6	0.007	2.8	< 0.004	< 0.6
2nd Partition								
Ethyl acetate phase	0.066	39.7	0.134	40.0	0.117	43.7	0.265	45.8
Flumioxazin	ND	ND	ND	ND	ND	ND	ND	ND
482-HA	ND	ND	< 0.003	< 1.0	ND	ND	ND	ND
APF	< 0.003	< 1.6	< 0.003	< 1.0				
THPA					0.010	3.6	0.027	4.7
1-OH-HPA					0.043	15.9	0.068	11.7
Single unidentified	0.048	28.7	0.102	30.6	0.040	14.8	0.110	19.0
Others (max & number).	0.014 (14)		0.027 (19)		0.012 (6)		0.027 (14)	
Others (total)	0.018	11.0	0.032	9.4	0.024	9.4	0.060	10.4
Aqueous phase	0.027	16.8	0.068	20.6	0.031	11.4	0.049	8.6
THPA					0.003	1.2	0.004	0.8
Single unidentified	0.021	12.9	0.061	18.2	0.012	4.6	0.029	5.0
Others (max & number)	0.003 (26)		0.006 (37)		0.003 (7)		0.004 (18)	
Others (total)	0.006	3.9	0.007	2.4	0.016	5.6	0.016	2.8
Cellulase treatment								
Extract	0.021	12.5	0.048	14.3	0.082	30.7	0.172	29.7
THPA					< 0.005	< 1.9	0.018	3.1
1-OH-HPA					0.042	15.6	0.082	14.1
Single unidentified	0.009	5.4	0.024	7.0	0.009	3.5	0.016	2.8
Others (max & number).	< 0.004 (6)		< 0.004 (10)		0.007 (2)		< 0.014 (2)	
Others (total)	0.012	7.1	0.024	7.3	0.031	11.6	0.056	9.7
Acid hydrolysis								
Extract	0.012	7.3	0.032	9.6	0.013	4.9	0.039	6.7
Ethyl acetate phase	0.003	1.9	0.008	2.5	0.013	4.9	0.018	3.1
Aqueous phase	0.009	5.4	0.024	7.1	not analysed		0.021	3.6
Alkaline hydrolysis								
Extract	0.020	12.2	0.012	3.5	0.007	2.7	0.007	1.2
Ethyl acetate phase	0.005	3.1	0.012	3.5	0.007	2.7	0.007	1.2
Aqueous phase	0.015	9.1	not analysed		not analysed		not analysed	
PES	0.006	3.3	0.012	3.7	0.004	1.6	0.006	1.1
Total	0.166	100	0.334	100	0.267	100	0.578	100

ND = non detectable

Table 15 Characterization and identification of residues in soya bean seeds 138 days after pre-emergence soil surface application with [^{14}C]flumioxazin

Metabolite	[Phenyl-^{14}C]Flumioxazin				[THP-^{14}C]Flumioxazin			
	100 g ai/ha		200 g ai/ha		100 g ai/ha		200 g ai/ha	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Acetone:water	0.012	35.9	0.022	45.0	0.145	66.5	0.108	66.3
1st Partition								
Hexane phase	not analysed		not analysed		not analysed		not analysed	
2nd Partition								
Ethyl acetate phase	0.008	24.0	0.014	27.4	0.126	57.7	0..093	56.9
Flumioxazin	ND	ND	ND	ND	< 0.004	< 1.8	< 0.003	< 2.3
THPA					0.013	6.0	0.006	4.0
1-OH-HPA					0.070	32.0	0.055	32.9
Single unidentified	0.003	10.0	0.007	14.1	0.028	13.0	0.017	9.7
Others (max & number)	< 0.002 (5)		0.005 (3)		0.013 (7)		0.015 (7)	
Others (total)	0.005	14.0	0.007	13.3	0.015	6.7	0.015	10.3
Aqueous phase	0.004	11.9	0.008	17.6	0.019	8.8	0.015	9.4
Single unidentified	not analysed				0.011	5.1	0.010	6.6
Others (max & number)					0.003 (12)		0.003 (8)	
Others (total)					0.008	3.7	0.005	2.8
Cellulase treatment								
Extract	0.010	30.4	0.013	26.2	0.061	28.5	0.026	15.9
THPA					< 0.007	< 3.2	< 0.003	< 2.1
1-OH-HPA					0.022	10.2	0.008	5.0
Single unidentified	0.007	20.8	0.006	12.6	0.018	8.4	0.004	2.8
Others (max & number)	< 0.002 (6)		0.004 (5)		0.014 (4)		0.003 (3)	
Others (total)	0.003	9.6	0.007	13.6	0.021	9.9	0.014	8.1
Acid hydrolysis								
Extract	0.010	28.9	0.012	22.3	0.011	4.9	0.018	12.4
Ethyl acetate phase	0.002	4.8	0.001	2.0	0.002	0.8	0.006	3.5
Aqueous phase	0.008	24.1	0.011	20.3	0.009	4.1	0.012	8.9
Alkaline hydrolysis								
Extract	0.001	3.7	0.003	5.9	not analysed		0.007	5.1
PES	0.002	1.1	0.001	0.6	< 0.001	0.1	< 0.001	0.3
Total	0.035	100	0.051	100	0.217	100	0.159	100

ND = non detectable

Table 16 Identified main metabolites in soya bean forage, hay and seeds after pre-emergence soil surface application with [^{14}C]flumioxazin

Metabolite	[Phenyl-^{14}C]Flumioxazin				[THP-^{14}C]Flumioxazin			
	100 g ai/ha		200 g ai/ha		100 g ai/ha		200 g ai/ha	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Forage								
Flumioxazin	0.004	6.1	0.006	5.5	< 0.001	< 1.8	0.008	4.4
482-HA	< 0.001	< 1.0	0.001	0.7	ND	ND	ND	ND
APF	< 0.001	< 1.8	< 0.001	< 0.5				
THPA					0.002	2.6	0.007	4.2
1-OH-HPA (free and conj.)					0.011	15.3	0.045	25.2
1-OH-HPA (conjugated)					< 0.003	< 4.0	0.017	9.4
Hay								
Flumioxazin	0.007	4.4	0.017	5.2	0.006	2.2	0.030	5.1
482-HA	ND	ND	< 0.003	< 1.0	ND	ND	ND	ND
APF	< 0.003	< 1.6	< 0.003	< 1.0				
THPA					0.013	4.8	0.049	8.6
1-OH-HPA (free and conj.)					0.085	31.5	0.150	25.8
1-OH-HPA (conjugated)					0.042	15.6	0.082	14.1
Seeds								
Flumioxazin	ND	ND	ND	ND	< 0.004	< 1.8	< 0.003	< 2.3
482-HA	ND	ND	ND	ND	ND	ND	ND	ND

Metabolite	[Phenyl-^{14}C]Flumioxazin				[THP-^{14}C]Flumioxazin			
	100 g ai/ha		200 g ai/ha		100 g ai/ha		200 g ai/ha	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
APF	ND	ND	ND	ND				
THPA					< 0.007	< 3.2	< 0.003	< 2.1
1-OH-HPA (free and conj.)					0.092	42.2	0.063	37.9
1-OH-HPA (conjugated)					0.022	10.2	0.008	5.0

ND = Non detectable

Sugar cane

In a metabolism study on sugar cane reported by Jalal, 2003 [Ref: SBM-0074], flumioxazin, radio-labelled in the phenyl ring or the THP ring, was applied at a rate equivalent to 0.48 kg ai/ha as a directed spray to 1.5–2 m high sugar cane prior to stem elongation, at the 6–10 leaf stage, with up to 1 m of the cane receiving direct spray. Immature sugarcane forage (leaves and cane) were sampled about a month after the application and mature canes and leaves (3–3.6 m high) were also sampled at maturity, 90 days after treatment, when the canes were 5 cm in diameter.

Samples were homogenized with dry ice and combusted to determine the total radioactive residue (TRR) and were also sequentially extracted with acetonitrile and water with total radioactivity in the extracts and the post-extraction solids being measured by combustion and LSC analysis.

The total radioactive residues determined by combustion analysis were 0.001–0.004 mg/kg in mature cane, 0.23–0.89 mg/kg in immature forage and 0.5–0.52 mg/kg in mature leaves. Acetonitrile and water extraction was able to retrieve more than 90% TRR in immature forage and mature canes. Higher levels (0.53–1.0 mg/kg) were reported in the extracted mature leaf samples (with larger aliquots of a more homogenous mixture of vascular and non-vascular tissues).

Table 17 Total radioactive residues in sugarcane immature forage, mature leaves and canes after a directed foliar application equivalent to 0.48 kg ai [^{14}C]flumioxazin/ha

DETERMINATION METHOD		TOTAL RADIOACTIVITY (MG/KG FLUMIOXAZIN EQUIVALENTS)		
		IMMATURE FORAGE (30 DAT)	MATURE LEAVES (90 DAT)	MATURE CANE (90 DAT)
Combustion:	Phenyl-label	0.227	0.517	0.001
Extraction:	Phenyl-label	0.209	1.046	0.002
Combustion:	THP-label	0.889	0.496	0.004
Extraction:	THP-label	0.888	0.526	0.004

The acetonitrile extracts of the forage, mature leaves and cane were analysed by HPLC and TLC. The post-extraction solids from the mature leaf samples were hydrolysed by refluxing in 2 M HCl for 2 hours, and after ethyl acetate partitioning, the remaining solid fractions were then refluxed in 2 M NaOH for approximately 2 hours and the base hydrolysate was adjusted to pH 1 to precipitate and centrifuge out the insoluble lignin fraction.

More than 90% TRR was able to be solvent-extracted, with the more aggressive extraction methods able to retrieve all but 2% of the remaining TRR.

Flumioxazin was the predominant residue in immature forage (leaves and canes) accounting for 90–93% TRR (0.19 mg/kg—phenyl-label, 0.83 mg/kg—THP-label). Among the minor components, one polar constituent made up 2.8–3.8% of TRR (0.008–0.025 mg/kg). The unextracted residue in the post-extraction solids accounted for 2.3–4.7% of the TRR (0.01–0.02 mg/kg).

Flumioxazin was also the predominant residue in mature leaves, making up 81–88% of TRR (0.92 mg/kg—phenyl-label, 0.427 mg/kg—THP-label). Among the minor components, a polar constituent was found in various extract fractions, making up a total of 5.1–8.7% of TRR (0.046–0.053 mg/kg). In the post-extraction solids, radioactivity was distributed into all plant constituents including the starch, cellulose, lignin, lipids and proteins, but did not exceed 0.03 mg/kg in any individual PES sub-fraction, with none of the individual TLC bands containing significant residue and none corresponded to any of the reference standards.

Flumioxazin also accounted for most of the mature cane residue (68–75% of TRR, 0.001–0.003 mg/kg), with the aqueous extract and PES contained only a trace level (≤ 0.001 mg/kg) of the radioactivity

Table 18 Characterisation and identification of residues in sugar cane matrices following one directed foliar application equivalent to 0.48 kg ai [^{14}C]flumioxazin/ha

Matrix	Immature forage (30 DAT)				Mature leaves (90 DAT)				Mature cane (90 DAT)			
[^{14}C]-label	Phenyl-label		THP-label		Phenyl-label		THP-label		Phenyl-label		THP-label	
TRR mg/kg	0.209		0.888		1.046		0.526		0.002		0.004	
%TRR extracted	95.3		97.7		93.7		90.5		90.0		92.0	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Flumioxazin	0.189	90.3	0.825	92.9	0.922	88.2	0.427	81.1	0.001	74.7	0.003	68.3
Polar	0.008	3.8	0.025	2.8	0.053	5.1	0.046	8.7	< 0.001	6.7	< 0.001	8.9
Others	0.002	1.1	0.017	2.0	0.005	0.5	0.004	0.7	< 0.001	18.6	< 0.001	22.8
Extracted-Acetonitrile	0.189	90.2	0.843	94.9	0.921	88.1	0.427	81.1	0.002	81.4	0.003	77.1
Extracted-Aqueous	0.011	5.1	0.025	2.8	0.059	5.6	0.049	9.3	< 0.001	8.6	0.001	14.8
Unextracted (PES)	0.01	4.7	0.02	2.3	0.065	6.3	0.05	9.5	< 0.001	10.0	< 0.001	8.0
PES lipid/phenol fraction					0.011	1.1	0.012	2.2				
PES starch fraction					0.026	2.5	0.019	3.6				
PES protein fraction					0.007	0.7	0.007	1.3				
PES lignin fraction					0.018	1.8	0.011	2.1				
PES cellulose fraction					0.002	0.2	0.002	0.3				
PES acid hydrolysis - EtOAc fraction					0.011	1.1	0.012	2.2				
PES acid hydrolysis - aqueous fraction					0.026	2.5	0.019	3.6				
Base hydrolysis - acid soluble					0.007	0.7	0.007	1.3				
base hydrolysis - acid insolubles					0.018	1.8	0.011	2.1				
Total	0.209	100	0.888	100	1.046	100	0.526	100	0.002	100	0.004	100

When applied to soil prior to crop emergence or as directed treatments to soil surrounding established plants, flumioxazin does not translocate or accumulate in significant concentrations in plant matrices. In general, no parent or identifiable metabolites were found in the plant matrices analysed although very low levels of metabolites (most also identified as rat metabolites) were identified in peanut plant samples.

Following directed foliar applications, flumioxazin is not translocated, with the majority of the residue remaining as the parent, with some incorporation into natural plant constituents.

Rotational crop metabolism

The Meeting received information on the metabolism of flumioxazin in lettuce, carrot and wheat grown as rotational crops in flumioxazin-treated soil.

Two confined rotational crop studies using lettuce, carrots and wheat were conducted with flumioxazin labelled in the phenyl ring (Patrick, 1993 [Ref: SBM-0034]) or in the THP ring (Patrick, 1993 [Ref: SBM-0048]). In both studies, the radio-label was applied to bare sandy loam soil plots at rates equivalent to 0.11 kg ai/ha or 0.21 kg ai/ha and the rotational crops were planted 30 days after treatment in all plots and 120, 180 and 365 days after treatment in the higher treatment plots. Fallowed plots were maintained outdoors, and except during periods of heavy rainfall when they were covered to prevent flooding, they were exposed to the environment. Planted crops were maintained in screen houses. Phytotoxicity was observed in most of the rotational crops, particularly in lettuce and carrots, with some plots being replanted because of crop failure (See footnotes to the following Tables).

Radioactive residues in soil and plant materials were determined by combustion followed by Liquid Scintillation Counting and extracted residues were characterized and identified by HPLC or TLC using known reference standards. The stability of stored analytical samples was established by analysis of samples at the beginning and at the end of the study, with no significant degradation being observed.

Soils core samples were taken at each planting and sampling date and segmented into 0–10 cm and 10–20 cm samples prior to combustion analysis and in the phenyl-label study, extracted with acetone:water and acetone:aqueous 0.1 N HCl (5:1) prior to HPLC analysis.

In both studies, results showed that crops assimilated only very small amounts of radioactivity when grown in soil treated with radiolabelled flumioxazin. In the phenyl-label study (0.21 kg ai/ha treatment), TRRs above 0.01 mg/kg were found in wheat straw and chaff at all PBIs, in carrot tops (120 day PBI) and in wheat grain from the 30 day PBI plot. Highest residues were 0.02–0.03 mg/kg eq. in wheat straw.

In the THP-label study, TRRs above 0.01 mg/kg eq were found in carrot tops, wheat straw, chaff and grain from the 0.11 kg ai/ha 30 day PBI plots. TRRs increased in some commodities at the 120-day and 180-day plant-back intervals, suggesting that THP-derived cleavage products in soil are either more readily assimilated by the plants or less tightly bound to soil than those from the phenyl label. In the 0.21 kg ai/ha treated plots, highest residues were 0.015 mg/kg eq in wheat forage (180 day PBI), 0.012 mg/kg eq. in lettuce (180 day PBI), 0.045 mg/kg eq. in carrot tops (30 day PBI), 0.022 mg/kg eq. in carrot roots (30 day PBI), 0.13 mg/kg eq. in wheat straw (120 day PBI), 0.043 mg/kg eq. in wheat chaff (120 day PBI) and 0.023 mg/kg eq. in wheat grain (120 day PBI).

Table 19 Total radioactive residues (mg/kg eq) in rotational crops planted 30 days after soil application of [^{14}C]flumioxazin

Crop	Matrix	Total radioactive residues			
		30-day PBI			
		0.11 kg ai/ha		0.21 kg ai/ha	
phenyl-label (mg eq/kg)					
		DAT	mg eq/kg	DAT	mg eq/kg
Wheat forage	Foliage	84	0.002	84	0.006

Crop	Matrix	Total radioactive residues			
		30-day PBI			
		0.11 kg ai/ha		0.21 kg ai/ha	
Lettuce	Foliage	122	0.002 [a]	180	0.005 [b]
Carrot	Foliage	132	0.002	132	0.01
	Root	132	0.001	132	0.005
Wheat	Straw	176	0.013	176	0.029
	Chaff	176	0.005	176	0.011
	Grain	176	0.006	176	0.011
THP-label (mg eq/kg)					
		DAT	mg eq/kg	DAT	mg eq/kg
Wheat forage	Foliage	95	0.006	95	0.008
Lettuce	Foliage	172	0.004 [a]	172	0.003 [b]
Carrot	Foliage	175	0.028	175	0.045 [a]
	Root	175	0.01	175	0.022
Wheat	Straw	159	0.057	159	0.072
	Chaff	159	0.026	159	0.033
	Grain	159	0.013	159	0.017

PBI = Plant-back interval

[a] 60–61 day Plant-back intervals (crop failure)

[b] 90 day Plant-back interval (crop failure)

Table 20 Total radioactive residues (mg/kg) in rotational crops planted 120–365 days after soil application of [^{14}C]flumioxazin (0.21 kg ai/ha)

Crop	Matrix			Total radioactive residues			
		120-day PBI		180-day PBI		365-day PBI	
phenyl-label							
		DAT	mg eq/kg	DAT	mg eq/kg	DAT	mg eq/kg
Wheat forage	Foliage	180	0.003	261	0.003	412	0.001
Lettuce	Foliage	226	0.007 [a]	254	0.002	440	0.002
Carrot	Foliage	281	0.011	330	0.004	462	0.004
	Root	281	0.005	330	0.005	462	0.001
Wheat	Straw	295	0.02	364	0.028	492	0.009
	Chaff	295	0.016	364	0.013	492	0.003
	Grain	295	0.013	364	0.006	492	0.002
THP-label							
		DAT	mg eq/kg	DAT	mg eq/kg	DAT	mg eq/kg
Wheat forage	Foliage	195	0.011	238	0.015	431	0.004
Lettuce	Foliage	195	0.006	253	0.012	431	0.004
Carrot	Foliage	253	0.026	294	0.013	494	0.013
	Root	253	0.01	294	0.004	494	0.005
Wheat	Straw	253	0.131	308	0.062	494	0.049
	Chaff	253	0.043	308	0.027	494	0.016
	Grain	253	0.023	308	0.008	494	0.005

[a] 149 day Plant-back interval (crop failure)

In soil, the extractable radiocarbon showed a slow decrease over time, decreasing to about 50% during the second half of the study period and remained mostly in the top 0–10 cm layer. The major component was flumioxazin, with minor components (each < 0.01 mg/kg) being tentatively identified as 482-HA, 482-CA, IMOXA and APF based on their retention times.

Table 21 Total radioactive residues and flumioxazin residues in soil (0–10 cm layer) following soil applications of [^{14}C]-flumioxazin

Residues in soil (TRR and extracted flumioxazin)						
0.11 kg ai/ha			0.21 kg ai/ha			Sampling point
DAT		Flumioxazin	DAT	TRR	Flumioxazin	

Residues in soil (TRR and extracted flumioxazin)						
0.11 kg ai/ha			0.21 kg ai/ha			Sampling point
DAT		Flumioxazin	DAT	TRR	Flumioxazin	
phenyl-label						
DAT	TRR	Flumioxazin	DAT	TRR	Flumioxazin	
0	0.129		0	0.259	0.305	Application
30	0.113	0.078	30	0.212	0.154	30 d PBI planting
61	0.152	0.082	61	0.239	0.143	61 d PBI replanting (lettuce, carrot)
84	0.149	0.057	84	0.208	0.09	30 d PBI wheat forage sampling
			90	0.155	0.074	90 d PBI replanting (lettuce)
			120	0.208	0.091	120 d PBI planting
122	0.068	0.017				61 d PBI lettuce sampling
132	0.083	0.029				30 d PBI carrot sampling
			132	0.184	0.063	61 d PBI carrot sampling
			149	0.11	0.055	149 d PBI lettuce planting
176	0.086	0.028	176	0.188	0.058	30 d PBI wheat sampling
			180	0.16	0.07	180 d PBI planting
			180	0.138	0.029	149 d PBI lettuce sampling
			180	0.099	0.02	120 d PBI wheat forage sampling
			226	0.207	0.059	120 d PBI lettuce sampling
			261	0.165	0.056	180 d PBI wheat forage sampling
			254	0.173	0.056	180 d PBI lettuce sampling
			281	0.256	0.078	120 d PBI carrot sampling
			295	0.05	0.007	120 d PBI wheat sampling
			330	0.122	0.028	180 d PBI carrot sampling
			364	0.158	0.031	180 d PBI wheat sampling
			365	0.129	0.037	365 d PBI planting
			412	0.061	0.003	365 d PBI wheat forage
			440	0.121	0.009	365 d PBI lettuce sampling
			462	0.148	0.015	365 d PBI carrot sampling

Residues in soil (TRR and extracted flumioxazin)						
0.11 kg ai/ha			0.21 kg ai/ha			Sampling point
DAT		Flumioxazin	DAT	TRR	Flumioxazin	
			492	0.059	0.006	365 d PBI wheat sampling
THP-label						
DAT	TRR	Flumioxazin	DAT	TRR	Flumioxazin	
0	0.1		0	0.194		Application
30	0.111		30	0.144		30 d PBI planting
60	0.096		60	0.131		30 d PBI replanting (lettuce)
			60	0.209		30 d PBI replanting (carrot)
			90	0.138		
95	0.089		95	0.177		30 d PBI wheat forage sampling
			120	0.138		120 d PBI planting
159	0.067		159	0.081		30 d PBI wheat sampling
172	0.062		172	0.095		60 d PBI lettuce sampling
175	0.064					30 d PBI carrot sampling
			175	0.17		60 d PBI carrot sampling
			180	0.118		180 d PBI planting
			195	0.115		120 d PBI wheat forage sampling
			195	0.125		120 d PBI lettuce sampling
			238	0.108		180 d PBI wheat forage sampling
			253	0.132		180 d PBI lettuce sampling
			253	0.118		120 d PBI carrot sampling
			253	0.113		120 d PBI wheat sampling
			294	0.093		180 d PBI carrot sampling
			308	0.106		180 d PBI wheat sampling
			365	0.108		365 d PBI planting
			431	0.122		365 d PBI wheat forage sampling
			431	0.097		365 d PBI lettuce sampling
			494	0.074		365 d PBI carrot sampling

Residues in soil (TRR and extracted flumioxazin)						
0.11 kg ai/ha			0.21 kg ai/ha			Sampling point
DAT		Flumioxazin	DAT	TRR	Flumioxazin	
			494	0.1		365 d PBI wheat sampling

Sequential solvent extractions of samples containing more than 0.01 mg/kg using acetone:water (4:1), acetone:0.1 N HCl (4:1) and refluxing with acetonitrile:0.25 N HCl was able to extract 62–85.5% TRR in wheat straw and chaff from the 30 day PBI plots and 36–61% TRR in the 120 day and 180 day PBI plots in the phenyl-label study and in the THP-label study, extraction efficiencies were 61–84% in wheat straw, chaff and carrot roots, 75–69% in carrot tops at PBIs of 30 days and 120 days, decreasing to 59% (180 day PBI) and 47% in the 365 day PBI samples. Some plant samples containing < 0.01 mg/kg were also analysed and the similar metabolic profile was confirmed.

In wheat grain, 5–13% TRR was able to be extracted in the 30 day and 120 day PBI plots with a further 22–26% TRR being extracted after cellulase incubation for 24 hours at 37 °C in the phenyl-label study and in the THP-label study, more aggressive digestion and fractionation was able to show that 12–21% TRR was present in cellulose, hemicellulose and starch fractions and 3–9% TRR was found in the protein, lignin and pectin fractions.

HPLC analysis of the acetone:water extracts containing more than 0.01 mg/kg TRR from the phenyl-label study identified the presence of flumioxazin and the metabolites 482-HA, IMOXA, and 482-CA, with wheat straw also containing low levels of 1-OH-SAT-482, 1-OH-HPA, THPA, and TPA, all at < 0.01 mg/kg eq. Flumioxazin residues above 0.01 mg/kg were only found in wheat straw (0.03 mg/kg) from the 120-day plant-back treatment.

Table 22 Characterisation and identification of radioactive residues in rotational crops planted after soil application of 0.21 kg ai/ha [^{14}C-THP]flumioxazin

Matrix	PBI	Treatment (kg ai/ha)	Hvst DAT	TRR (mg/kg eq)	Component					
					mg/kg (%TRR)					
					Polar	Flumioxazin	482-HA	IMOXA	482-CA	Others
Wheat straw	30	0.11	159	0.03	0.015 (50%)	0.002 (5.9%)	< 0.001 (2.5%)	< 0.001 (0.87%)	–	–
Wheat chaff	30	0.11	159	0.012	0.007 (59.6%)	< 0.001 (2.3%)	< 0.001 (1.4%)	–	–	–
Carrot foliage	30	0.11	175	0.017	0.008 (48.6%)	0.003 (17.5%)	< 0.001 (5.5%)	–	< 0.001 (2.1%)	–
Wheat straw	30	0.21	159	0.034	0.012 (34%)	0.003 (8.6%)	0.001 (3.3%)	< 0.001 (1.6%)	< 0.001 (1.6%)	–
	120	0.21	253	0.08	0.012 (15.2%)	0.033 (40.7%)	< 0.001 (2.2%)	–	–	[a]
	180	0.21	308	0.027	0.002 (6.4%)	0.009 (35.1%)	–	–	–	[b]

Matrix	PBI	Treatment (kg ai/ha)	Hvst DAT	TRR (mg/kg eq)	Component					
					mg/kg (%TRR)					
					Polar	Flumioxazin	482-HA	IMOXA	482-CA	Others
	365	0.21	494	0.04	0.005 (13.2%)	0.007 (16.3%)	–	–	–	
Wheat chaff	30	0.21	159	0.012	0.007 (54.9%)	< 0.001 (0.99%)	< 0.001 (3.7%)	–	–	–
	120	0.21	253	0.026	0.015 (56.6%)	0.002 (8.8%)	0.002 (6.1%)	< 0.001 (0.56%)	< 0.001 (2.1%)	–
	180	0.21	308	0.011	0.005 (49.2%)	0.002 (10.7%)	< 0.001 (5.3%)	< 0.001 (1.9%)	–	–
	365	0.21	494	0.011	0.005 (41.1%)	0.001 (20.3%)	–	–	–	–
Carrot foliage	60	0.21	175	0.022	0.013 (58.5%)	0.002 (10.4%)	0.001 (6.6%)	< 0.001 (0.48%)	< 0.001 (1.7%)	
	120	0.21	253	0.016	0.006 (39.2%)	0.007 (42.8%)	< 0.001 (1.7%)	< 0.001 (1.6%)	< 0.001 (0.84%)	–
Carrot roots	60	0.21	175	0.013	0.007 (57.3%)	0.005 (38.8%)	< 0.001 (1.2%)	< 0.001 (0.66%)	–	–

[a] 1-OH-SAT (0.008 mg/kg eq, 9.7% TRR)

[b] 1-OH-HPA (0.004 mg/kg eq, 13.4% TRR)

THPA (0.004 mg/kg eq, 15.3% TRR)

TPA (0.004 mg/kg eq, 15.2% TRR)

Radioactive residues in rotational crops planted 30–365 days after bare soil treatments with [^{14}C]flumioxazin were low, generally less than 0.01 mg/kg and less than 0.05 mg/kg in all matrices except wheat straw, where up to 0.13 mg/kg were found in the THP-label study. The only significant residue identified in rotated crop matrices above 0.01 mg/kg was the parent, flumioxazin, in wheat straw (0.013 mg/kg). Low levels of 482-HA, IMOXA and 482-CA were found in most crop matrices, up to 6.6% TRR (< 0.002 mg/kg) with wheat straw also containing low levels of 1-OH-SAT-482, 1-OH-HPA, THPA, and TPA, all < 0.01 mg/kg.

Animal metabolism

The Meeting received animal metabolism studies on rats, lactating goats and laying hens where animals were dosed with flumioxazin radio-labelled in the phenyl ring or the tetrahydrophthaloyl (THP) ring.

Rats

The metabolism of flumioxazin in rats was evaluated by the WHO Core Assessment Group of the 2015 JMPR. Studies were carried out to investigate the metabolism of phenyl-label and THP-label flumioxazin in rats. Excretion of radioactivity was rapid, with 69–87% being eliminated in urine and faeces within 24 hours with the reminder found mainly in excretory organs. Flumioxazin was extensively metabolized (29–35 metabolites detected and quantified), with 7–10 of these being identified. Flumioxazin accounted for 47–66% of the administered dose in the 100 mg/kg bw and 0.3–2% in the 1 mg/kg bw dose group. Metabolites found at more than 5% of the administered dose were

3-OH-flumioxazin, 3-OH-flumioxazin-SA, 4-OH-flumioxazin and 4-OH-flumioxazin-SA. The proposed metabolic pathways included hydroxylation of the cyclohexene ring, cleavage of the imide linkage, cleavage of the amide linkage in the benzoxadine ring, reduction of the double bond in the THP ring, acetylation of the amino group of the aniline derivative and the addition of a sulphonic acid group to the THP ring.

Lactating goats

Two studies were carried out to investigate the absorption and deposition of phenyl-label and THP-label flumioxazin in lactating goats. In the first study, reported by Sharp, 1993 [Ref: SBM-0026], two lactating goats (average body-weight of 48 kg) were dosed orally for 5 days with capsules containing [^{14}C-phenyl] flumioxazin at the rate equivalent to 11.8 ppm in the diet (based on an average feed consumption of 2.1 kg/goat/day and a total dose of 2.63 mg/kg bw). Milk, urine and faeces were collected twice daily and liver, fat, muscle, blood, gastrointestinal tract and contents were collected at sacrifice, about 6 hours after the last dose.

The second study, reported by Panthani, 1994 [Ref: SBM-0040] used a similar protocol involving two goats (average body-weight of 45 kg) but with [^{14}C-THP] flumioxazin at a dose equivalent to 7.2 ppm in the diet (based on an average feed consumption of 2 kg/goat/day and a total dose of 1.44 mg/kg bw).

Radioactivity was quantified by LSC. Samples of liver, kidney, muscle, and fat were initially homogenized in dry ice, and then subjected to combustion/LSC.

The average total recoveries of radioactivity were 81% and 94% of the administered radioactivity (AR) in the phenyl-label and the THP-label studies respectively, mostly found in faeces, urine and the GI tract contents (80–93% AR). Tissues and milk contained relatively small amounts of radioactivity (< 1% and 0.22% AR respectively).

Average concentrations of radioactivity were low in muscle and fat, up to 0.014 mg/kg (phenyl-label) and 0.028 mg/kg (THP-label), but were higher in liver, up to 0.21 mg/kg (phenyl-label) and 0.33 mg/kg (THP-label). In kidney the radioactive residues were up to 0.18 mg/kg (phenyl-label) and 0.24 mg/kg (THP-label). The average total radioactivity concentration in milk plateaued around Day 3 at about 0.03 mg/kg (phenyl-label) and about 0.06 mg/kg in the THP-label study.

Table 23 Distribution of radioactive residues in tissues, excreta and milk of lactating goats following 5 daily doses of [^{14}C]flumioxazin

MATRIX	RADIOACTIVE RESIDUES (MG FLUMIOXIN EQUIVALENTS/KG)							
	[^{14}C-PHENYL] FLUMIOXAZIN (11.8 PPM IN THE DIET)				[^{14}C-THP] FLUMIOXAZIN (7.2 PPM IN THE DIET)			
	GOAT 2		GOAT 3		GOAT 500090		GOAT 500092	
	%AD	MG/KG	%AD	MG/KG	%AD	MG/KG	%AD	MG/KG
Fat (omental)	< 0.01	0.006	< 0.01	0.005	0.01	0.006	0.01	0.01
Fat (perirenal)	< 0.01	0.006	< 0.01	0.004	0.01	0.008	0.01	0.008
Kidneys	0.02	0.182	0.01	0.11	0.05	0.189	0.04	0.238
Liver	0.19	0.209	0.12	0.165	0.44	0.286	0.40	0.33
Muscle (rear leg)	0.01	0.014	0.01	0.013	0.02	0.023	0.02	0.028
Muscle (loin)	0.01	0.014	0.01	0.012	0.02	0.022	0.03	0.025
Total tissues	0.25	0.43	0.17	0.31	0.55	0.53	0.51	0.64
Milk day 1 (pm)		0.019		0.023		0.033		0.049
Milk day 2 (am)		0.005		0.005		0.005		0.010
Milk day 2 (pm)		0.023		0.026		0.041		0.053
Milk day 3 (am)		0.007		0.007		0.007		0.009
Milk day 3 (pm)		0.026		0.032		0.042		0.046
Milk day 4 (am)		0.007		0.007		0.007		0.012

MATRIX	RADIOACTIVE RESIDUES (MG FLUMIOXIN EQUIVALENTS/KG)							
	[^{14}C-PHENYL] FLUMIOXAZIN (11.8 PPM IN THE DIET)				[^{14}C-THP] FLUMIOXAZIN (7.2 PPM IN THE DIET)			
	GOAT 2		GOAT 3		GOAT 500090		GOAT 500092	
	%AD	MG/KG	%AD	MG/KG	%AD	MG/KG	%AD	MG/KG
Milk day 4 (pm)		0.025		0.03		0.046		0.055
Milk day 5 (am)		0.007		0.006		0.006		0.011
Milk day 5 (pm)		0.028		0.031		0.043		0.05
Total milk	0.05		0.17		0.22		0.2	
Blood	< 0.01	0.019	< 0.01	0.025	–	0.061	–	0.068
Urine	14.5		15.4		33.8		27.1	
Faeces	50.3		50.2		44.6		45.5	
GI tract	15.0 [a]	2.01	15.0 [a]	2.26	14.9 [a]		18.8 [a]	
Pan rinse	0.08		0.29		0.45		0.61	
Total	80.2		81.2		94. 5		92.7	

%AD = % administered dose

[a] Includes GI tract contents

In the first study, milk and tissue samples (except fat) from goats dosed with the phenyl-label were solvent-extracted and subjected to protease digestion (liver and kidney) to characterize and identify residues. Milk samples were extracted with hexane and the extracted residues were triple-extracted into methanol, filtered, evaporated and redissolved in methanol for LSC and HPLC analysis. Muscle samples were extracted with acetonitrile and after evaporation and reconstitution in methylene chloride (to solubilise the lipids), the supernatants were partitioned between acetonitrile and hexane for analysis. The remaining solid phases from the acetonitrile extractions were also subjected to an additional sodium bicarbonate extraction step and aliquots were subjected to overnight enzyme digestion (protease) prior to sequential extraction with water, methanol and acetonitrile and analysis.

In the second study, milk and tissue samples (except fat) from goats dosed with the THP-label were solvent-extracted to characterize and identify residues. Milk samples were mixed with ethanol, filtered, concentrated and partitioned with hexane. The aqueous phases were concentrated, additional ethanol was added, and after centrifuging, the supernatants were concentrated for TLC and HPLC analysis. Tissue samples were extracted with acetonitrile and acetonitrile:water with 1%HOAc (1:1), filtered and the supernatants were concentrated for TLC and HPLC analysis. The solids fractions were dried and combusted to quantitate the unextracted radioactivity.

About 80–94% TRR in milk was able to be extracted with methanol or ethanol, and acetonitrile was able to extract 58–74% TRR from muscle (45–53% in the THP-label samples, with a further 30% extracted in acetonitrile:water). In the phenyl-label liver and kidney samples, sequential extractions with acetonitrile and bicarbonate were able to extract more than 90% TRR and further enzyme extraction released an additional 10% TRR. In the THP-label liver and kidney samples, sequential acetonitrile and acetonitrile:water extractions were able to extract 80–87% TRR.

Table 24 Total radioactive residues recovered in tissues and milk of lactating goats following five daily doses of [^{14}C]flumioxazin

EXTRACT	LIVER		KIDNEY		LOIN MUSCLE		REAR LEG MUSCLE		MILK	
	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
Phenyl-label (11.8 ppm in the diet)										
Acetonitrile	42.3	0.88	55.8	0.1	73.7	0.009	58.3	0.008		
Methanol									80.4	0.02
Bicarbonate	47.7	0.1	44.3	0.081						

EXTRACT	LIVER		KIDNEY		LOIN MUSCLE		REAR LEG MUSCLE		MILK	
	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
Protease	9.2	0.019	11.2	0.02						
Post-extraction solids	5.1	0.011	4.5	0.008	37.8	0.005	49.6	0.006	10.2	0.003
%TRR	104		116		112		108		90.6	
THP-label (7.2 ppm in the diet)										
Acetonitrile	55.7	0.159	49.9	0.094	52.9	0.012	45.3	0.011		
Ethanol									94.1	0.024
Acetonitrile:water	31.1	0.089	29.7	0.056	20.4	0.005	26.9	0.006		
Post-extraction solids	7.6	0.022	11.7	0.022	19.5	0.004	21.4	0.005	11.4	0.003
%TRR	94		91		93		94		105	

Metabolites were characterized and identified following sample extraction and co-chromatography with known reference materials using thin-layer chromatography and HPLC with uv detection high-performance liquid chromatography within 4–6 months of sampling. Concurrent analysis of stored analytical samples indicated that residues were stable over the storage intervals in the studies.

Flumioxazin was extensively metabolized, with residues above 0.001 mg/kg found only in liver (up to 0.01 mg/kg and < 5% TRR).

The only identified metabolite present at more than 10% TRR was the 4-OH-flumioxazin, accounting for up to 14% TRR in kidney (up to 0.025 mg/kg) and muscle (up to 0.003 mg/kg). In liver, 4-OH-flumioxazin residues did not exceed 0.025 mg/kg (9.4% TRR) and were up to 0.002 mg/kg (8.6% TRR) in milk.

Other identified metabolites found at more than 5% TRR were 482-HA, found in liver and kidney (close to 10% TRR, 0.02 mg/kg), 3-OH-flumioxazin in liver (up to 8.6% TRR, 0.023 mg/kg) and kidney (up to 6% TRR, 0.011 mg/kg), APF in kidney (5.8% TRR, 0.011 mg/kg) and SAT-482 in liver and kidney (5–6% TRR, up to 0.013 mg/kg).

In kidney, metabolite B, tentatively identified as 3- or 4-OH-SAT-482, made up about 14% TRR (0.024 mg/kg) and residues of metabolite C was measured at 0.015 mg/kg (8.5% TRR).

In liver, metabolite F, tentatively identified as an isomer of 3- or 4-OH-SAT-482, made up about 11% TRR (0.03 mg/kg) and residues of metabolite D were measured at 0.013 mg/kg (4.9% TRR).

In muscle, metabolite C accounted for 20-23% TRR and in milk, metabolites B and C were found at 12–18% TRR. However, absolute levels were all 0.005 mg/kg or less.

Table 25 Characterisation and identification of radioactive residues in goat tissues and milk following five daily doses of [^{14}C-phenyl]-flumioxazin (11.8 ppm in the diet)

METABOLITE	LIVER		KIDNEY		LOIN MUSCLE		REAR LEG MUSCLE		MILK	
	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
Flumioxazin	4.7	0.01	0.2	< 0.001	1.2	< 0.001	0.7	< 0.001	ND	< 0.001
3-OH-flumioxazin	4.2	0.009	6.2	0.011	1.2	< 0.001	ND	< 0.001	1.8	< 0.001
4-OH-flumioxazin	6.5	0.014	13.7	0.025	1.6	< 0.001	2.7	< 0.001	1.5	< 0.001
3-OH-flumioxazin-SA+ 4-OH-flumioxazin-SA	1.8	0.004	ND	< 0.001	ND	< 0.001	ND	< 0.001	6.5	0.002
482-HA	9.8	0.02	8.7	0.016	4.2	< 0.001	5.1	< 0.001	14.4	0.004
APF	3.8	0.008	5.8	0.011	3.5	< 0.001	ND	< 0.001	0.2	< 0.001
Maximum single other metabolite	7.6	0.016	18.1	0.033	7.4	0.001	13.9	0.002	11.5	0.003
Total identified	30.8		34.6		11.7		8.5		24.4	

ND = non detectable

Table 26 Characterisation and identification of radioactive residues in goat tissues and milk following five daily doses of [^{14}C-THP]-flumioxazin (7.2 ppm in the diet)

METABOLITES	LIVER		KIDNEY		LOIN MUSCLE		REAR LEG MUSCLE		MILK	
	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
Flumioxazin	1.4	0.004	–	–	1.4	0.0003	1.8	0.0004	–	–
3-OH-flumioxazin	8.6	0.023	4.5	0.008	–	–	–	–	–	–
4-OH-flumioxazin	9.4	0.025	7.9	0.014	11.7	0.002	13.2	0.003	8.6	0.002
4-OH-THPA	0.9	0.003	3.8	0.007	6.9	0.001	6.8	0.002	6.0	0.002
SAT-482	4.7	0.013	5.5	0.01	–	–	–	–	–	–
THPA	3.2	0.009	1.2	0.002	–	–	–	–	–	–
Metabolite B	1.6	0.004	14.0	0.024	–	–	–	–	17.9	0.005
Metabolite C	–	–	8.5	0.015	23.3	0.005	19.6	0.004	12.3	0.003
Metabolite D	4.9	0.013	3.7	0.006	–	–	–	–	1.5	0.0004
Metabolite E	1.5	0.004	0.9	0.002	–	–	–	–	–	–
Metabolite F	11.4	0.031	–	–	–	–	–	–	–	–
Unknowns	38.2	0.103	26.3	0.057	28.5	0.006	30.5	0.007	39.5	0.011
Maximum single other metabolite	8.5	0.023	8.5	0.015	19.4	0.004	19.6	0.004	11.2	0.003
Nonextractable	8.0	0.022	12.8	0.022	20.8	0.004	22.8	0.005	10.8	0.003

The major metabolic pathways proposed for flumioxazin in goats include: the hydroxylation of the parent to 3-OH-flumioxazin and the subsequent incorporation of a sulfonic group to form 3-OH-flumioxazin-SA; the reduction of the parent molecule and subsequent hydroxylation to SAT-482; and the cleavage of the imide and amide linkages of the parent molecule to THPA and APF.

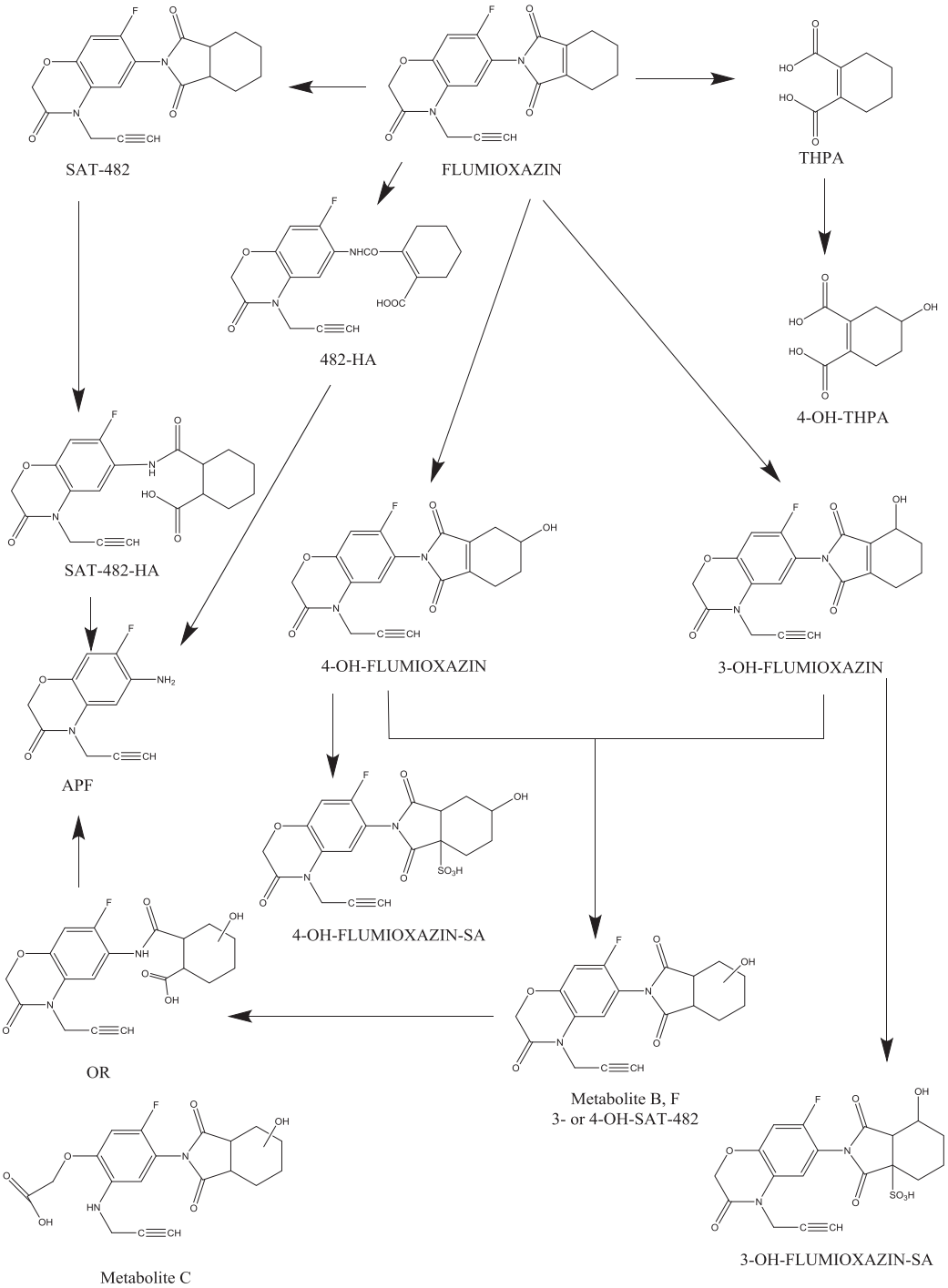


Fig 4 Metabolic Pathway of Flumioxazin in Lactating Goats

Laying Hens

Two studies were carried out to investigate the absorption and deposition of phenyl-label and THP-label flumioxazin in laying hens. In the first study, reported by Sharp, 1993 [Ref: SBM-0027], ten laying hens (average body-weight of 1.65 kg) were dosed orally for 14 days with capsules containing [^{14}C-phenyl] flumioxazin at the rate equivalent to 10 ppm in the diet (based on an average feed consumption of 0.122 kg/hen/day and an average daily dose of 0.683 mg/kg bw/day).

The second study, reported by Panthani, 1994 [Ref: SBM-0039] used a similar protocol involving 10 hens (1.3–1.9 kg bodyweight) and [^{14}C-THP] flumioxazin at a dose equivalent to 10 ppm in the diet (based on an average feed consumption of 0.127 kg/hen/day).

Eggs were collected twice daily. Eggs collected on the same day were pooled and then separated into yolks and whites. Samples of excreta were collected daily. The hens were sacrificed 4 hours after the last dose and the following samples were collected: kidney, heart, liver, muscle (breast and thigh), abdominal fat, skin with fat, gizzard, reproductive organs, and GI tract and contents.

Samples of kidney, liver, muscle, fat, and skin with fat were homogenized in dry ice, and then subjected to combustion/LSC. Egg yolk and white samples were blended and then subjected to combustion/LSC. Samples of excreta and cage washings were also collected and were analysed for TRR.

The average total recoveries of radioactivity in the samples collected for analysis from the treated animals were 95% of the administered dose (phenyl-label study) and 87% in the THP-label study. Most of the radioactivity was found in the excreta, GI tract contents and cage wash, which, together accounted for 94% and 83% of the doses in the respective studies. Liver, kidney, muscle, fat, skin and eggs contained relatively small amounts of radioactivity (totalling < 0.6% and < 0.9% of the administered dose, respectively).

Radioactivity in egg yolks accounted for 0.35–0.36% AR, with < 0.01% AR in the corresponding egg whites and liver contained 0.08% AR in the phenyl-label study and 0.27% AR in the THP-label study (0.24 mg/kg eq and 1.14 mg/kg eq respectively. In egg yolks, residues reached a plateau of 0.4–0.6 mg/kg eq by Day 10 or 11 in the two studies.

Table 27 Distribution of radioactive residues in tissues, excreta and eggs of laying hens following 14 daily doses of [^{14}C]flumioxazin

MATRIX	RADIOACTIVE RESIDUES (MG FLUMIOXIN EQUIVALENTS/KG)					
	[^{14}C-PHENYL] FLUMIOXAZIN (10 PPM IN THE DIET)			[^{14}C-THP] FLUMIOXAZIN (10 PPM IN THE DIET)		
	% ADMINISTERED DOSE	MG/KG		% ADMINISTERED DOSE	MG/KG	
Liver	0.08	0.237		0.27	1.137	
Kidney	0.02	0.272		0.06	0.887	
Breast muscle	0.04	0.040		0.05	0.138	
Thigh muscle	0.03	0.050		0.06	0.175	
Fat	0.02	0.074		0.01	0.226	
Skin with fat	0.04	0.143		0.02	0.667	
Total tissues	0.23			0.47		
Eggs		Yolk	White		Yolk	White
Day 1		ND	ND		0.009	0.029
Day 2		0.01	0.017		0.034	0.033
Day 3		0.036	0.012		0.119	0.025
Day 4		0.099	0.015		0.154	0.041
Day 5		0.178	0.018		0.240	0.037
Day 6		0.237	0.017		0.338	0.03
Day 7		0.323	0.018		0.414	0.036
Day 8		0.349	0.015		0.467	0.034
Day 9		0.407	0.01		0.531	0.03
Day 10		0.425	0.008		0.57	0.036
Day 11		0.437	0.008		0.638	0.027
Day 12		0.422	0.01		0.64	0.025
Day 13		0.409	0.005		0.63	0.024

MATRIX	RADIOACTIVE RESIDUES (MG FLUMIOXIN EQUIVALENTS/KG)					
	[^{14}C-PHENYL] FLUMIOXAZIN (10 PPM IN THE DIET)			[^{14}C-THP] FLUMIOXAZIN (10 PPM IN THE DIET)		
	% ADMINISTERED DOSE	MG/KG		% ADMINISTERED DOSE	MG/KG	
Day 14		0.382	0.007		0.76	0.032
Total eggs	0.35			0.43		
Heart	< 0.01	0.161		0.04	0.761	
Gizzard	0.02	0.104		1.14	5.253	
GI tract & contents	1.43 [a]	0.62		4.67 [a]	6.018 [a]	
Blood	0.03	0.603		1.53	1.326	
Cage wash	0.5	–		2.89	–	
Reproductive organs	0.23	0.25		0.35	0.483	
Excreta	92.1	–		75.36	–	
Total	94.9			86.87		

[a] Includes GI tract contents

Egg yolk, egg white, and tissues samples were extracted with various solvents and the solvent-extracted radio-labelled residues were analysed by HPLC and TLC to characterize and identify the major metabolites. Identification was made by co-chromatography of extracts with known standards. The unextracted radioactive residues were characterized by acid or base hydrolysis, or by enzyme digestion. Additional metabolites were isolated from excreta extracts for mass spectral analysis to confirm the identity of the structures of the metabolites.

In the first study, egg (Day 7 and 13) and tissue samples from hens dosed with the phenyl-label were solvent-extracted and subjected to enzyme digestion to characterize and identify residues. Egg yolk samples were extracted with acetonitrile, the supernatant partitioned with hexane and the remaining residue subjected to enzyme (lipase) digestion and sodium bicarbonate extraction of the insoluble fraction with sequential extractions/elutions in hexane, methanol and acetonitrile. Egg white samples were extracted in acetonitrile. Liver samples were extracted with acetonitrile, the supernatant partitioned between acetonitrile and hexane and the remaining residue further extracted with sodium bicarbonate and subjected to enzyme (protease) digestion with the various fractions being sequentially extracted or eluted with water, methanol and acetonitrile. Kidney and muscle samples were extracted with acetonitrile, the supernatants partitioned between acetonitrile and hexane and the remaining residue further extracted with water (except breast muscle) and subjected to enzyme (protease) digestion and acid hydrolysis (6 N HCl), with the various fractions being sequentially extracted or eluted with water, hexane, methanol, dichloromethane and acetonitrile. Skin + fat and fat samples were extracted with chloroform:methanol (2:1) with the chloroform phase being partitioned between acetonitrile and hexane. The remaining residue from the skin + fat samples were subjected to enzyme (protease) digestion with the various fractions being sequentially extracted or eluted with water, methanol and acetonitrile.

In the second study, egg (Day 13) and tissue samples from hens dosed with the THP-label were extracted with acetonitrile and acetonitrile:water with 1% HOAc (1:1), filtered and the supernatants were concentrated for TLC and HPLC analysis. The post-extraction solids were also subjected to pronase hydrolysis. The remaining solids fractions were dried and combusted to quantitate the unextracted radioactivity.

In the THP-label and the phenyl-label studies respectively, highest concentrations of radioactivity were in liver (1.1 mg/kg and 0.24 mg/kg) and kidney (0.89 mg/kg and 0.27 mg/kg) with lower levels in egg yolks (0.63 mg/kg and 0.41 mg/kg). Radioactivity in fat and skin + fat were 0.23–0.67 mg/kg (THP-label) and 0.07–0.14 mg/kg (phenyl-label) respectively. In muscle, radioactive residues were 0.14–0.18 mg/kg (THP-label) and 0.04–0.05 mg/kg (phenyl-label) and egg white contained about 0.02 mg/kg in both studies.

More than 87% TRR in eggs was able to be extracted, and acetonitrile was able to extract 37–67% TRR from muscle. In the phenyl-label liver and kidney samples, sequential extractions with acetonitrile and bicarbonate were able to extract more than 60% TRR and further enzyme extraction released an additional 30% TRR. In the THP-label liver and kidney samples, sequential acetonitrile and acetonitrile:water extractions were able to extract 75–78% TRR. In fat and fat + skin, extraction efficiencies were 76–91% TRR and 54–95% respectively.

Table 28 Total radioactive residues recovered in tissues and eggs of laying hens following 14 daily doses of [^{14}C]flumioxazin (10 ppm in the diet)

EXTRACT	EGG WHITE	EGG YOLK	LIVER	KIDNEY	THIGH	BREAST	FAT	SKIN + FAT
	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)
Phenyl-label								
TRR (mg/kg)	0.018	0.409	0.237	0.272	0.05	0.04	0.074	0.143
Acetonitrile	100.0 (0.018)	42.9 (0.175)	45.9 (0.109)	53.0 (0.144)	36.7 (0.018)	42.3 (0.017)		
Water				13.6 (0.037)				
Bicarbonate		10.6 (0.043)	14.9 (0.035)					
Enzyme		40.3 (0.165)	31.3 (0.074)	20.1 (0.055)	39.4 (0.02)	27.2 (0.011)		13.7 (0.02)
Acid hydrolysate				9.8 (0.027)	14.8 (0.007)	29.8 (0.012)		
MeOH/$CHCl_3$ (organic)							54.0 (0.040)	48.7 (0.07)
MeOH/$CHCl_3$ (aqueous)							21.5 (0.016)	32.9 (0.047)
%Total extracted	100	93.8	92.1	96.5	90.9	99.3	75.5	95.3
Post-extraction solids	10.0 (0.002)	4.8 (0.02)	11.1 (0.026)	2.7 (0.007)	5.2 (0.003)	6.6 (0.003)	9.9 (0.007)	0.81 (0.001)
%TRR	110	98.6	103	99.2	96.1	106	85.4	96.1
THP-label								
TRR (mg/kg)	0.024	0.63	1.137	0.887	0.175	0.138	0.226	0.667
Acetonitrile	79.4 (0.017)	20.0 (0.184)	48.9 (0.45)	52.6 (0.101)	55.7 (0.101)	62.9 (0.086)	69.0 (0.179)	36.6 (0.229)
Acetonitrile:water	13.2 0.003	64.5 (0.515)	28.9 (0.325)	22.2 (0.19)	9.0 (0.016)	4.2 (0.006)	22.0 (0.057)	17.8 (0.111)
%Total extracted	92.6	87.5	77.8	74.8	64.7	67.0	91.0	54.4
Post-extraction solids	7.4 (0.002	12.5 (0.1)	22.2 (0.25)	25.2 (0.217)	35.3 (0.064)	33.0 (0.045)	9.0 (0.023)	45.5 (0.284)
%TRR	87.5	126.8	99.1	96.7	103.6	98.8	114.9	93.5

Values reported for eggs are from Day 13 Samples

Metabolites were characterized and identified following solvent extraction and co-chromatography with known reference materials using thin-layer chromatography and HPLC with uv detection high-performance liquid chromatography within 4.5 months of sampling. Concurrent analysis of stored analytical samples indicated that residues were stable (more than 93% recovery from spiked samples) over the storage intervals in the studies.

In the phenyl-label study, flumioxazin was the predominant residue in fat (49% TRR), skin + fat (25% TRR), muscle (10–14% TRR), liver (9.1% TRR) and kidney (6.9% TRR), made up about 3.8% TRR in egg yolk and was not detected in egg white. Absolute levels of

flumioxazin were < 0.05 mg/kg in skin + fat and fat, < 0.02 mg/kg in liver, kidney and egg yolk and about 0.005 mg/kg in muscle.

The major identified metabolites, present at more than 10% TRR were APF and 482-HA. The APF metabolite accounted for 20% TRR in egg white and 10% TRR in muscle (but both at absolute levels of < 0.005 mg/kg) and 482-HA made up about 20% of the TRR in egg white. All other identified metabolites were found at < 8% TRR and the highest level of any single unidentified metabolite was measured in liver, at 12% TRR.

Table 29 Characterisation and identification of radioactive residues in hen tissues and eggs following 14 daily doses of [^{14}C-phenyl]-flumioxazin (10 ppm in the diet)

METABOLITE	EGG WHITE	EGG YOLK	LIVER	KIDNEY	THIGH	BREAST	FAT	SKIN + FAT
	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)
Flumioxazin	ND	3.8 (0.016)	9.1 (0.022)	6.9 (0.019)	9.9 (0.005)	13.9 (0.006)	48.8 (0.046)	24.7 (0.035)
3-OH- flumioxazin SA	ND	0.2 (ND)	0.7 (0.002)	1.3 (0.004)	0.7 (ND)	0.5 (ND)	1.2 (ND)	0.3 (ND)
4-OH-flumioxazin SA	ND	0.1 (ND)	ND	1.4 (0.004)	3.3 (0.002)	0.6 (ND)	ND	ND
482-HA	20.0 (0.004)	0.6 (0.002)	1.2 (0.003)	0.1 (ND)	5.5 (0.003)	1.2 (ND)	ND	6.9 (0.01)
APF	23.2 (0.004)	3.5 (0.015)	3.1 (0.007)	4.8 (0.013)	7.7 (0.004)	10.4 (0.004)	ND	1.1 (0.001)
4-OH flumioxazin	ND	1.1 (0.004)	3.9 (0.009)	7.2 (0.02)	6.8 (0.003)	7.7 (0.003)	3.7 (0.003)	1.6 (0.002)
3-OH flumioxazin	ND	0.5 (0.002)	2.6 (0.006)	3.1 (0.008)	5.6 (0.003)	6.7 (0.003)	1.0 (ND)	2.6 (0.004)
Maximum other single metabolite	8.4	4.3	11.9	5.0	8.2	5.1	2.3	10.3
Total of identified metabolites	43.2 (0.008)	9.8 (0.039)	20.6 (0.049)	24.8 0.068)	39.5 (0.021)	41.0 (0.016)	54.7 (0.049)	37.2 (0.052)

ND = non detectable

Values reported for eggs are from Day 13 Samples

In the THP-label study, flumioxazin was the predominant residue in fat (49% TRR), skin + fat (12% TRR) and muscle (11% TRR) and was found at 7% TRR in liver, kidney and 9% TRR in egg yolk. Absolute levels of flumioxazin were 0.07–0.13 mg/kg in skin + fat and fat, < 0.08 mg/kg in liver and kidney, < 0.04 mg/kg in egg yolk and < 0.02 mg/kg in muscle.

The major identified metabolites, present at more than 10% TRR in tissues were 4-OH-flumioxazin, 3-OH-flumioxazin and 4-OH-THPA. The 4-OH-flumioxazin accounted for 9–12% TRR in all tissues (< 0.03 mg/kg in muscle and fat, < 0.08 mg/kg in kidney and skin + fat, 0.12 mg/kg in liver) while the 3-OH-flumioxazin accounted for 8–12% TRR (0.015 mg/kg) in muscle. The 4-OH-THPA metabolite made up 10% TRR (0.09 mg/kg) in kidney.

In eggs, metabolites present at more than 10% TRR were 4-OH-flumioxazin-SA in egg yolk (32% TRR, 0.14 mg/kg) and in egg white, THPA and 4-OH-THPA each accounted for 23–26% TRR and (but < 0.01 mg/kg), with TPA and 3-OH-THPA each present at 16–17% TRR. Absolute levels of these metabolites in egg white were all < 0.01 mg/kg.

Table 30 Characterisation and identification of radioactive residues in hen tissues and eggs following 14 daily doses of [^{14}C-THP]-flumioxazin (10 ppm in the diet)

METABOLITE	EGG WHITE	EGG YOLK	LIVER	KIDNEY	THIGH	BREAST	FAT	SKIN + FAT

	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)
Flumioxazin	0.65 (0.000)	8.9 (0.039)	6.7 (0.076)	7.2 (0.062)	11.0 (0.020)	10.8 (0.015)	49.0 (0.128)	11.6 (0.072)
THPA	22.7 (0.009)	6.8 (0.029)	9.7 (0.109)	9.8 (0.084)	9.4 (0.017)	8.4 (0.011)	2.3 (0.006)	4.5 (0.028)
TPA	16.4 (0.006)	ND	ND	ND	ND	ND	ND	ND
3-OH-flumioxazin-SA	ND	1.1 (0.005)	6.0 [b] (0.067)	4.3 [b] (0.037)	ND	ND	ND	ND
4-OH-flumioxazin-SA	ND	31.8 (0.139)			ND	ND	ND	ND
4-OH-flumioxazin	ND	5.4 (0.024)	10.8 (0.121)	8.7 (0.075)	10.2 (0.018)	12.3 (0.016)	11.4 (0.030)	10.9 (0.068)
3-OH-flumioxazin	ND	3.6 (0.016)	7.0 (0.079)	7.1 (0.061)	7.7 (0.014)	11.7 (0.016)	9.6 (0.025)	6.4 (0.040)
4-OH-THPA	25.8 (0.009)	7.8 (0.034)	4.4 (0.05)	10.3 (0.088)	7.0 (0.013)	6.4 (0.009)	2.9 (0.008)	3.2 (0.020)
3-OH-THPA	16.7 (0.006)	ND	ND	ND	ND	ND	ND	ND
OH-flumioxazin [a]	0.47 (0.000)	4.9 (0.021)	2.7 (0.030)	3.2 (0.027)	3.7 (0.007)	3.4 (0.004)	5.0 (0.013)	3.3 (0.020)
Maximum single other metabolite	< 1%	< 5%	< 5%	< 5%	< 5%	< 5%	< 5%	–
Unknown	11.1 (0.004)	19.4 (0.085)	29.8 (0.336)	23.4 (0.201)	14.6 (0.026)	13.1 (0.018)	10.0 (0.026)	13.8 (0.087)

Values reported for eggs are from Day 7 Samples

[a] Exact position of hydroxylation not determined

[b] Mixture of two metabolites

The major metabolic pathways proposed for flumioxazin in hens include the hydroxylation of the parent and the subsequent incorporation of sulfonic groups to form 3-OH-flumioxazin-SA and 4-OH-flumioxazin-SA and the cleavage of the imide and amide linkages of the parent molecule to THPA and APF.

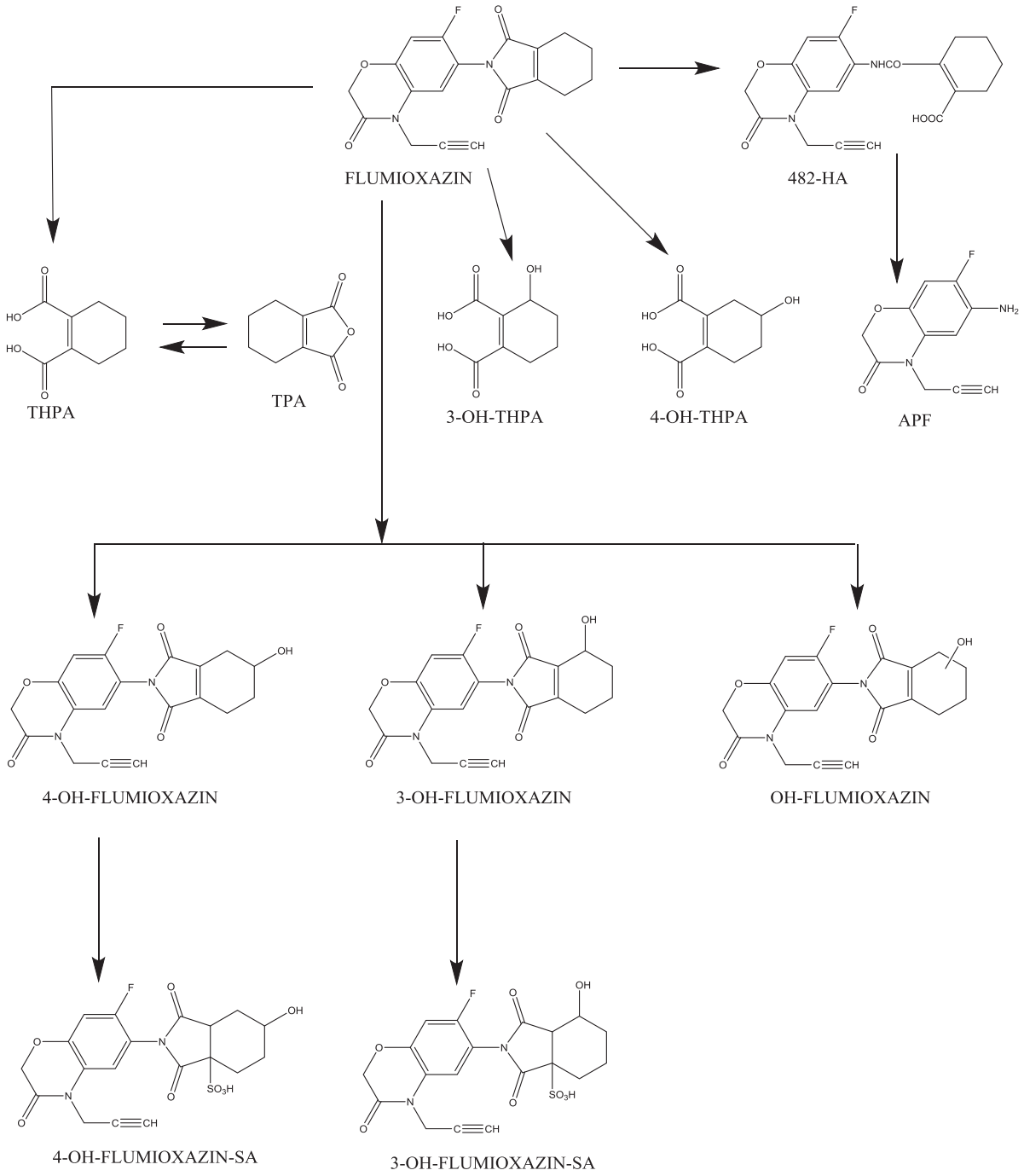


Fig 5 Metabolic Pathway of Flumioxazin in Poultry

Flumioxazin is extensively metabolized with limited absorption into tissues, eggs or milk (less than 0.5% of the administered dose). Flumioxazin was not found at levels above 0.01 mg/kg in goat milk or tissues and in poultry, highest residues found were in fat (0.13 mg/kg, 49% TRR), with lower levels (up to 0.08 mg/kg) in other tissues and egg yolks. The major identified metabolites found above 10% TRR and above 0.01 mg/kg in various matrices were 4-OH-flumioxazin, 4-OH-flumioxazin-SA, 3-OH-flumioxazin and 4-OH-THPA.

METHODS OF RESIDUE ANALYSIS

Analytical methods

The Meeting received analytical method descriptions and validation data for flumioxazin in plant and animal matrices and these are summarized below.

Table 31 Summary of analytical methods for flumioxazin and its 1-OH-HPA metabolite, developed for plant and animal matrices

Matrix	Analyte	Method	Principle	LOQ (mg/kg)	Reference
Fresh and processed plant matrices	Flumioxazin	RM 30A (RM 30A-1) (RM 30A-2) (RM 30A-3)	Acetone/water extraction Dichloromethane partition Hexane/acetonitrile partition Florisil column clean-up GC-MS analysis * RM 30A-1 includes minor modifications to the sample grinding/preparation steps * RM 30A-2 includes minor equipment and text modifications * RM 30A-3 adds confirmatory GC/MS conditions	0.02	SBR-0003
Processed plant oils	Flumioxazin	RM 30B	Hexane/acetonitrile extraction Acetonitrile partition Florisil column clean-up GC-MS analysis	0.02	SBR0019
Eggs Animal tissues Milk	Flumioxazin	ER-MT-9403	Acetone extraction Hexane/acetonitrile partition Florisil column clean-up GC-MS analysis	0.02	SBA-0037
Eggs Animal tissues Milk	Flumioxazin 3-OH-flumioxazin 4-OH-flumioxazin	RM-30T RM-30MK	Acetonitrile & acetonitrile:water extraction (Acetone extraction for milk) Dichloromethane & hexane/acetonitrile partition HPLC-MS/MS analysis	0.02	SBR-0138
Animal feeds	1-OH-HPA including conjugates	RM 30M	Acid hydrolysis extraction Ethyl acetate partition Methylation (dimethyl sulphate) Water/hexane partition Florisil column clean-up CG-MSD analysis (1-OH-HPA-dimethyl ester)	0.02	SBR-0019
Processed plant oils	1-OH-HPA including conjugates	RM 30P	Acid hydrolysis and hexane extraction SPE (ethyl acetate) extraction Methylation (dimethyl sulphate) Water/hexane partition Florisil column clean-up CG-MSD analysis (1-OH-HPA-dimethyl ester)	0.02	SBR-0019

Data collection methods

Method RM 30A (Flumioxazin—fresh and processed plant matrices)

Method 30A, used with minor equipment modifications and sample preparation steps to measure residues of flumioxazin in fresh plant commodities and their processed fractions was first reported by Pensyl, 1992 [Ref: SBR-0003].

In this method, homogenised samples are double-extracted with acetone:water (4:1), partitioned into dichloromethane and after evaporation to dryness, dissolved in hexane:acetonitrile (30:1) then shaken with acetonitrile:hexane (5:1), After separation, the combined acetonitrile extracts are evaporated to dryness, redissolved in ethyl acetate and diluted with hexane and purified using Florisil columns eluted with hexane:ethyl acetate (2:1 v:v). Residues in the eluate are concentrated, reconstituted in acetone, and then analysed by GC/NPD or in some cases, GC/MSD, using an external standard. Validation studies were conducted in parallel with some of the supervised field trials. The LOQ for this method is 0.02 mg/kg for all matrices except grapes, almonds and cotton seed, where acceptable recovery rates were achieved at a lower fortification level of 0.01 mg/kg.

For some commodities, method validation was conducted prior to analysing the samples from the supervised field trials. Recovery validation data from these trials are summarized in the table below.

Table 32 Flumioxazin analytical method (GC-MS) validation recovery rates in plant matrices

COMMODITY	FORTIFICATION (MG/KG)	N	%RECOVERY			METHOD	REFERENCE
			RANGE	MEAN	RSD		
Artichoke	0.02–0.2	6	80–115	96	14.5	RM 30A-1	SBR-0128
Asparagus	0.02–0.2	6	95–102	99	3	RM 30A-3	SBR-0116
Blueberries	0.02–0.2	6	95–109	103	5	RM 30A-3	SBR-0115
Cantaloupe	0.02–0.2	6	92–106	99	7	RM 30A-3	SBR-0112
Peanut hay	0.02–0.10	6	83–86	85	2.9	RM 30A	SBR-0018
Peanut hulls	0.02–0.10	6	90–101	96	2.1	RM 30A	SBR-0018
Peanut nutmeat	0.02–0.10	11	89–91	90	3.3	RM 30A	SBR-0018
Peanut oil	0.02–0.10	6	88–99	94	4.3	RM 30B	SBR-0018
Peanut vines	0.02–0.10	6	90–95	92	4.8	RM 30A-3	SBR-0018
Soya bean forage	0.1	3	90–98	94	4.4	RM 30A-3	SBR-0003
Soya bean hay	0.1	3	95–101	97	2.4	RM 30A-3	SBR-0003
Soya bean	0.1	3	81–84	80	2.5	RM 30A-3	SBR-0003

Method RM 30B (Flumioxazin—processed plant oils)

This method, similar to method RM 30A but without the initial acetone extraction and dichloromethane partitioning steps, used for the determination of flumioxazin in processed oils (maize oil, cottonseed oil and peanut oil) was first reported by Pensyl, 1994 [Ref: SBR-0019]. Samples are dissolved in hexane:acetonitrile (30:1), shaken with acetonitrile:hexane (5:1) and after separation, the acetonitrile extracts are evaporated and samples are cleaned-up using Florisil columns eluted with hexane:ethyl acetate (2:1 v:v). Residues in the eluate are concentrated, reconstituted in acetone, and then analysed by GC/NPD using an external standard. The LOQ of flumioxazin in oil matrices by this method is 0.02 mg/kg.

Method RM 30M (Metabolite 1-OH-HPA—animal feeds)

This method, developed for the determination of residues of the plant metabolite, 1-OH-HPA in animal feed commodities (almond hulls, peanut and soya bean forage/hay, cotton gin trash and sugar matrices) was first reported by Pensyl, 1994 [Ref: SBR-0019]. The metabolite, 1-OH-HPA is extracted from homogenized samples using acid hydrolysis (refluxing for 3 hours in 2.5 N HCl) prior to washing with hexane and partitioning into ethyl acetate. The concentrated extract is refluxed for 30 minutes with acetone, triisopropanolamine and dimethyl sulfate to convert the 1-OH-HPA to its dimethyl ester. The samples are then shaken with water and hexane, and after separation, the hexane extracts are cleaned-up using Florisil columns eluted with hexane:ether (1:2 v:v). Residues in the eluate are concentrated, reconstituted in acetone, and analysed by GC/MSD (m/z 157.2—

quantification and m/z 125.1—qualifier). The LOQs for the method are 0.02 mg/kg (peanut and soya bean forage/hay and sugar matrices) and 0.1 mg/kg (almond hulls and gin trash).

Method RM 30P (Metabolite 1-OH-HPA—processed plant oils)

This method, a modification of Method RM 30M (with an additional hexane partitioning step) to determine 1-OH-HPA in peanut and soya bean oils was reported by Pensyl, 1994 [Ref: SBR-0019]. Samples are hydrolysed in 2.5 N HCl and then partitioned with hexane to remove oils. The 1-OH-HPA is then extracted from the aqueous phase using ethyl acetate via solid phase extraction. The concentrated extract is re-dissolved in acetone and refluxed for 30 minutes with acetone, triisopropanolamine and dimethyl sulphate to convert the 1-OH-HPA to its dimethyl ester. The samples are then shaken with water and hexane, and after separation, are cleaned-up using Florisil columns eluted with hexane:ether (1:2 v:v). Residues in the eluate are concentrated, reconstituted in acetone, and analysed by GC/MSD. The LOQ for the method is 0.02 mg/kg.

Method ER-MT-9403 (Flumioxazin—animal matrices)

A method for determining residues of flumioxazin in milk, eggs and animal tissues was developed by Oishni, 1994 [Ref: SBA-0037]. Homogenised samples are double-extracted with acetone, partitioned into dichloromethane, evaporated to dryness, redissolved in ethyl acetate, diluted with hexane and purified using Florisil columns eluted with hexane:ethyl acetate (2:1 v:v for all tissues except chicken liver, where a 3:1 ratio is used). Meat and fat extracts also undergo an additional partitioning step before the Florisil clean-up, with samples being dissolved in hexane:acetonitrile (30:1), shaken with acetonitrile:hexane (5:1) and after separation, the combined acetonitrile extracts being evaporated to dryness. Residues in the eluate are concentrated, reconstituted in acetone, and then analysed by GC/NPD with a validated LOQ of 0.02 mg/kg for each analyte. Recovery data are summarized in the following table.

Table 33 Flumioxazin analytical method ER-MT-9403 (GC-MS) recovery rates in animal matrices [Ref SBA-0037]

Commodity	%Recovery 0.02 mg/kg fortification		%Recovery 0.1 mg/kg fortification		%Recovery 1.0 mg/kg fortification	
	%Recovery	%Mean	%Recovery	%Mean	%Recovery	%Mean
Meat	97, 96	96	102, 101	102	98, 96	97
Fat	101, 92	97	96, 93	95	94, 92	93
Liver	108, 99	103	100, 96	98	97, 96	96
Kidney	107, 107	107	95, 95	95	101, 99	100
Milk	105, 103	104	101, 98	100	94, 92	93
Poultry meat	96, 100	98	97, 97	97	101, 97	99
Poultry fat	96, 99	98	101, 98	100	98, 96	97
Poultry liver	87, 90	88	91, 88	90	92, 89	91
Poultry gizzard	89, 91	90	96, 96	96	97, 98	97
Eggs	97, 98	98	91, 89	90	96, 96	96

Methods RM- 30T, RM-30MK (Flumioxazin, 3-OH-flumioxazin, 4-OH-flumioxazin—animal matrices)

A method (RM-30T) for determining residues of flumioxazin and the 3-OH and 4-OH metabolites in animal tissues and a modified version (RM-30MK) were reported by Kowalsky, 2006 [Ref: SBR-0138] in an dairy cattle feeding study. Tissue samples are homogenised in acetonitrile, extracted in acetonitrile:water (50:50) acidified with 1% acetic acid. Milk samples are extracted with acetone. Sample extracts are partitioned into dichloromethane, evaporated to dryness, then dissolved in hexane:acetonitrile (30:1), shaken with acetonitrile:hexane (5:1) and after separation, the combined acetonitrile extracts being evaporated to dryness. Residues in the eluate are concentrated, reconstituted

in methanol:water and analysed by LC-MS/MS ((flumioxazin: m/z 355MS/MS 3-OH-flumioxazin: m/z 371OH-flumi and 4-OH-flumioxazin: m/z 371 →and 4-OH with an LOQ of 0.02 mg/kg for each analyte. Recovery data are summarized in the following table.

Table 34 Flumioxazin analytical methods RM-30T, RM-30MK recovery rates in animal matrices [Ref SBR-0138]

	Flumioxazin %Recovery (mean)		3-OH-flumioxazin %Recovery (mean)		4-OH-flumioxazin %Recovery (mean)	
Fortification	0.02 mg/kg	0.1 mg/kg	0.02 mg/kg	0.1 mg/kg	0.02 mg/kg	0.1 mg/kg
Muscle (concurrent)	82, 87, 88 (86)	82	72, 116, 124 (104)	84	83, 100, 108 (97)	83
Fat (concurrent)	77, 94, 103 (91)	79	75, 120, 126 (107)	87	83, 116, 119 (106)	83
Liver (validation)	77, 78, 82 (79)	85, 86, 90, 90, 90, 92 (92)	116, 117, 120 (118)	87, 89, 91, 91, 92, 93 (91)	110, 111, 111 (111)	96, 97, 101, 102, 102, 107 (101)
Liver (concurrent)	84	70	93	76	114	89
Kidney (concurrent)	81, 83, 88 (84)	82	73, 117, 120 (103)	90	81, 113, 114 103)	87
Milk (validation)	88, 89, 89 (89)	74, 78, 79,79,82, 84 (79)	80, 92, 103 (92)	81, 82, 90, 92, 93, 97 (89)	79, 87, 87 (84)	81, 83, 85, 86, 87, 90 (85)
Milk (concurrent)	77, 90, 92 (86)	78, 82, 85 (82)	96, 101, 106 (101)	78, 82, 85 (82)	92, 94, 99 (95)	83, 86, 94 (88)
Cream (concurrent)	84, 85 (85)	83	96, 98 (97)	78	89, 90 (90)	72
Skim milk (concurrent)	98	85	95	83	105	80

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 flumioxazin at 0.01 mg/kg or 0.02 mg/kg and at higher levels that generally reflected the range of expected residues. For each study, average recoveries per fortification level generally fell within the 70–120% range, with a relative standard deviation of 20% or less. A summary of recovery data from the methods used for plant commodities evaluated by the Meeting where one or more individual recovery values were outside the above criteria are presented in the table below.

Table 35 Flumioxazin analytical concurrent recovery rates in studies where one or more individual recovery values were outside the 70–120% range

Commodity	Fortification (mg/kg)	n	%Recovery range	%Recovery mean	%RSD	Method	Determina-tion	Study reference
Alfalfa forage	0.02–0.1	47	78–122	101	10.6	RM 30A-3	GC-MS	SBR-0111
Celery	0.02–0.2	13	90–150	113	17	RM 30A-1	GC-MS	SBR-0122
Cottonseed meal	0.01–0.05	3	101–135	113	16.6	RM 30A-1	GC-MS	SBR-0026
Grapes	0.01–0.05	16	82–123	107	9.6	RM 30A-1	GC-MS	SBR-0025
Maize grain	0.02–0.1	14	85–122	96	9.2	NCL 293	LC/MS/MS	SBR-0078
Olives	0.02–0.2	6	76–122	103	15	RM 30A-3	GC-MS	SBR-0130
Peanut hay	0.02	5	63–79	71	10	RM 30A	GC-MS	SBR-0019
Peppers	0.02–0.2	7	68–117	91	18.4	RM 30A-1	GC-MS	SBR-0118
Soya bean forage	0.02	29	67–120	92	15.3	RM 30A	GC-MS	SBR-0021
Soya bean hay	0.02	19	73–130	89	19.5	RM 30A	GC-MS	SBR-0021
Sugar cane	0.01–0.5	12	67–113	89	16	RM 30A-1	GC-MS	SBR-0022

Commodity	Fortification (mg/kg)	n	%Recovery range	%Recovery mean	%RSD	Method	Determina-tion	Study reference
Wheat grain	0.02–0.5	34	70–122	103	12.9	RM 30A-3	GC-MS	SBR-0092

In some supervised trials, residues of the 1-OH-HPA were also measured, together with analytical recovery rates in control samples fortified with 0.02–0.5 mg/kg 1-OH-HPA. For each study, average recoveries per fortification level generally fell within the 70–120% range, with a relative standard deviation of 20% or less. A summary of recovery data from the methods used for plant commodities evaluated by the Meeting are presented in the table below.

Table 36 Analytical concurrent recovery rates for 1-OH-HPA in plant matrices

Commodity	Fortification (mg/kg)	n	%Recovery range	%Recovery mean	%RSD	Method	Determina-tion	Study reference
Almond hulls	0.1–0.5	10	81–98	90	6.0	RM 30M	GC-MS	SBR-0024
Gin trash	0.1–0.5	14	81–121	99	9.4	RM 30M	GC-MS	SBR-0026
Molasses	0.02, 0.1	2	78, 114	96	–	RM 30M	GC-MS	SBR-0022
Peanut soapstock	0.02–0.1	9	69–87	74	11.6	RM 30P	GC-MS	SBR-0021
Soya bean oil	0.02–0.1	9	85–88	86	8.4	RM 30P	GC-MS	SBR-0021
Soya bean seeds	0.02	14	71–100	81	9.6	RM 30M	GC-MS	SBR-0021
Sugar	0.02, 0.1	2	80, 111	96	–	RM 30M	GC-MS	SBR-0022
Sugar cane	0.02–0.2	10	70–114	96	16.4	RM 30M	GC-MS	SBR-0022

Enforcement methods

FDA Multi-residue method

Nandihalli, 1996 [Ref: SBA-0040] evaluated the suitability of the FDA PAM Multi-residue methods for measuring residues of flumioxazin. Testing according to Protocols A, C and F showed that retention times and sensitivity criteria were not met, and that none of the FDA multi-residue method test procedures are suitable for the regulatory analysis of flumioxazin.

Multi-residue method DFG S19 (plant matrices)

The multi-residue method DFG S19 (revised) was investigated and validated for the determination of flumioxazin in cereals and other dry crops (Rzepka, 2004; SBA-0048), potato (Rzepka and Klimmek, 2006; SBA-0051), and oily crops such as sunflower seeds (Class and Merdian, 2010; SBA-0064). Samples are extracted with acetone:water (2:1 v/v) and the extracts partitioned with 1:1 v/v ethyl acetate:cyclohexane (Module E 2). The organic phase is cleaned up by gel permeation chromatography using ethyl acetate:cyclohexane (1:1, v/v) as the eluent and after concentration, flumioxazin residues are determined by GC-MS (Module D4). The fragment ion m/z 354 was used for quantitation and m/z 287 and m/z 259 were used for confirmation. The LOQ was 0.02 mg/kg for all matrices tested.

The method showed good linearity (correlation coefficients > 0.997 and no significant interferences were detected at the retention time corresponding to flumioxazin in any control samples, although confirmatory analysis of wheat straw samples yielded chromatographic interferences. These were removed by an additional clean-up step using silica gel mini-columns. The mean recoveries for all matrices tested and at all fortification levels ranged from 70 and 110%, within the acceptable range, with relative standard deviations of 20% or less.

Table 37 Multi-residue method DFG S19 analytical recovery rates for flumioxazin

Commodity	Fortification (mg/kg)	Fragment ion (m/z)	% Recovery	%Recovery mean	SD	Study reference

Commodity	Fortification (mg/kg)	Fragment ion (m/z)	% Recovery	%Recovery mean	SD	Study reference
Wheat grain	0.02 0.2	354 354	80, 106, 101, 117, 126 107, 112, 109, 112, 95	106 107	18 7	SBA-0048
Wheat grain	0.02 0.2	287 287	83, 103, 108, 118, 122 107, 113, 109, 112, 95	107 107	15 7.2	SBA-0048
Wheat grain	0.02 0.2	259 259	101, 102, 94, 100, 105 108, 113, 106, 109, 98	100 107	4 5.5	SBA-0048
Wheat straw	0.05 0.5	354 354	77, 71, 62, 73, 66 75, 77, 70, 80, 76	70 76	5.9 3.6	SBA-0048
Wheat straw	0.05 [a] 0.05 [a] 0.05 [a]	354 287 259	95, 98, 103 92, 93, 102 90, 91, 96	99 96 92	4 5.5 3.2	SBA-0048
Potato	0.02 0.2	354 354	107, 110, 112, 113, 102 107, 114, 112, 108	109 110	4.4 3.3	SBA-0051
Potato	0.02 0.2	287 287	100, 93, 108, 100, 103 108, 111, 109, 106	101 109	5.4 2.1	SBA-0051
Potato	0.02 0.2	259 259	97, 83, 112, 101, 110 106, 112, 109, 106, 74	101 101	12 16	SBA-0051
Sunflower seed	0.05 0.5	354 354	99, 102, 100, 101, 101 113, 111, 104, 101, 104	101 107	1 5	SBA-0064
Sunflower seed	0.05 0.5	287 287	99, 102, 100, 100, 101 110, 111, 101, 102, 103	100 105	1 4	SBA-0064
Sunflower seed	0.05 0.5	259 259	102, 103, 101, 101, 98 111, 110, 101, 100, 102	101 105	2 5	SBA-0064

[a] With an additional silica gel mini-column clean-up step

Stability of residues in stored analytical samples

The Meeting received information on the stability of residues of flumioxazin in a wide range of fresh and processed commodities with high water, starch, protein, oil and acid contents, stored at freezer temperatures of –20 °C (or below) for various intervals. Several studies were also provided on the stability of the 1-OH-HPA metabolite. Most of these studies were conducted concurrently with the supervised field trials, and the longest storage intervals reflected those used in the field trials.

Table 38 Stability of flumioxazin residues in a range of fresh and processed plant matrices with high water content, spiked at 0.1–0.5 mg/kg and stored at –20 °C or below

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Alfalfa forage (0.1 mg/kg)	0 131 305 929	105, 107, 106 83, 88 94,96 80, 85	– 86 95 83	106 93 101 100	RM 30A-3	SBR-0111
Alfalfa hay (0.1 mg/kg)	0 131 305 929	95, 99, 111 87, 77 91, 96 67, 73	– 82 94 70	102 79 97 70	RM 30A-3	SBR-0111
Apple juice (0.5 mg/kg)	0 60 119 196 265	88, 101, 92 85, 97, 91 85, 93 101, 102 59, 60, 63	– 91 89 102 61	94 109 93 94 81	RM 30A-3	SBR-0031
Apple wet pomace (0.5 mg/kg)	0 69 197 267	98, 98, 115 92, 98 89, 87 82, 79	– 95 88 81	104 105 87.4 79	RM 30A-3	SBR-0031

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Artichoke (0.2 mg/kg)	252	85, 90, 120	98 [a]	107	RM 30A-1	SBR-0128
Asparagus (0.1 mg/kg)	217	98, 94, 86	93 [a]	105	RM 30A-3	SBR-0116
Cabbage (0.2 mg/kg)	243	120, 120, 105	115 [a]	110	RM 30A-1	SBR-0129
Cantaloupe (0.2 mg/kg)	125	95, 94, 92	94 [a]	106	RM 30A-3	SBR-0112
Celery (0.2 mg/kg)	298	100, 90, 90	93 [a]	108	RM 30A-1	SBR-0122
Cherries (0.1 mg/kg)	0 112 316 354	104, 99, 101 101, 103 88, 92 79, 92	– 102 90 86	101 92 94 86	RM 30A-3	SBR-0027
Cucumber (0.2 mg/kg)	203	70, 85, 70	75 [a]	80	RM 30A-1	SBR-0121
Maize forage (0.1 mg/kg)	0 162 293 417	87, 95, 99 74, 90 93, 84 72, 76	– 82 89 74	94 98 95 76	RM 30A-3	SBR-0078
Maize stover (0.1 mg/kg)	0 165 293 404	98, 103, 105 77, 75 88, 90 73, 75	– 76 89 74	102 85 101 79	RM 30A-3	SBR-0078
Non-bell pepper (0.2 mg/kg)	786	77, 77, 76, 73, 77, 75	76 [a]	111	RM 30A-1	SBR-0118
Onion bulb (0.1 mg/kg)	124	92, 78, 80	83 [a]	80	RM 30A-1	SBR-0083
Peanut hay (0.1 mg/kg)	0 20 41 142 296	92, 94, 101 100, 101 93, 96 117, 128 74, 92	– 101 95 123 83	96 95 84 112 73	RM 30A-1	SBR-0018
Peanut vines (0.1 mg/kg)	0 20 40 147 300	95, 96, 99 97, 97 100, 105 110, 111 92, 100	– 97 103 111 96	97 99 103 100 100	RM 30A-1	SBR-0018
Soya bean forage (0.1 mg/kg)	0 30 92 190 240 360	102, 102, 103 87, 88 77, 79 83, 86 95, 96 112, 112	– 88 78 85 96 112	102 89 81 95 95 121	RM 30A-3	SBR-0003
Soya bean hay (0.1 mg/kg)	0 31 87 182 240 297 360	79, 79, 80 91, 92 76, 90 78, 78 66, 67 91, 92 87, 91	– 92 83 78 67 92 89	79 97 89 87 80 97 90	RM 30A-3	SBR-0003
Sugar cane (0.1 mg/kg)	0 29 64	94, 94, 99 93, 100 100, 99	– 97 100	96 86 92	RM 30A-1	SBR-0022
Sugar (0.1 mg/kg)	0 32 54	90, 97, 99 83, 96 82, 76	– 90 79	95 94 101	RM 30C	SBR-0022

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Summer squash (0.2 mg/kg)	477/479	65, 80, 80	75 [a]	108	RM 30A-1	SBR-0120
Tomato (0.2 mg/kg)	218	110, 115, 115, 125	116 [a]	100	RM 30A-1	SBR-0117

[a] % nominal residue remaining. No analysis of Day-0 sample

Table 39 Stability of flumioxazin residues in a range of of fresh and processed plant matrices matrices with high oil content, spiked at 0.05–1.0 mg/kg and stored at –20 °C or below

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Almond nutmeat (0.05 mg/kg)	0 29 60 92 186	98, 99, 101 117, 116 94, 100 123, 130 83, 79	– 117 97 127 81	99 115 95 119 99	RM 30A-1	SBR-0024
Almond hulls (0.05 mg/kg)	0 29 60 92 186	91, 91, 94 100, 112 89, 88 93, 96 92, 102	– 106 89 95 97	92 103 95 101 78	RM 30A-1	SBR-0024
Pecan (0.1 mg/kg)	0 135	88, 83, 84 100, 96, 90	– 95	85 103	RM 30A-3	SBR-0062
Cotton seed (0.1 mg/kg)	0 90 197 273	121, 126, 129 104, 104 117, 120 76, 87	– 104 119 82	125 95 120 78	RM 30A-1	SBR-0011
Cotton seed (1.0 mg/kg)	0 90 197 273	93, 100, 107 90, 117 104, 104 99, 99	– 104 104 99	100 103 111 85	RM 30A-1	SBR-0011
Cotton seed (0.05 mg/kg)	0 36 61 90 183	76, 82, 83 85, 111 81, 80 82,74 70, 77	– 98 81 78 74	80 114 77 81 97	RM 30A-1	SBR-0026
Cotton gin trash (0.05 mg/kg)	0 34 59 88	70, 70, 70 92, 84 96, 93 101, 85	– 88 95 93	70 83 93 92	RM 30A-1	SBR-0026
Cottonseed hulls (0.05 mg/kg)	0 33 61 93	93, 97, 98 111, 108 119, 111 103, 96	– 110 115 100	96 109 108 94	RM 30A-1	SBR-0026
Cottonseed meal (0.05 mg/kg)	0 33 61 93	106, 107, 108 98, 102 110, 128 113, 95	– 100 119 104	107 67 115 92	RM 30A-1	SBR-0026
Peanut nutmeat (0.1 mg/kg)	0 20 40 147 300	85, 87, 89 84, 86 92, 105 74, 86 93, 92	– 85 99 80 93	87 94 102 105 77	RM 30A-1	SBR-0018

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Peanut hull (0.1 mg/kg)	0 20 41 142 296	88, 89, 105 89, 98 91, 97 91, 93 92, 124	– 94 94 92 108	94 92 95 100 75	RM 30A-1	SBR-0018
Peanut presscake (0.1 mg/kg)	0 30	107, 108, 111 119, 119	– 119	109 96	RM 30A-1	SBR-0018
Peanut soapstock (0.1 mg/kg)	0 15 30 31	96, 98, 104, 108, 109 64, 67 37, 57 44, 44	– 66 47 44	103 111 93 97	RM 30A-1	SBR-0018
Peanut oil (crude) (0.1 mg/kg)	0 31	115, 119, 114 123, 133	– 128	116 98	RM 30B	SBR-0018
Olives (0.2 mg/kg)	526	91, 95, 105	97 [a]	89	RM 30A-3	SBR-0130
Olive oil (0.2 mg/kg)	479	99, 105, 107	104 [a]	109	RM 30A-3	SBR-0130
Mint tops (0.2 mg/kg)	0 82 354	98, 102, 104 99, 93 89, 94	– 96 92	101 98 103	RM 30A-2	SBR-0136
Mint oil (0.2 mg/kg)	0 267	77, 88, 89 84, 83	– 84	85 82	RM 30A-2	SBR-0136

[a] % nominal residue remaining. No analysis of Day-0 sample

Table 40 Stability of flumioxazin residues in soya bean seed (high protein content), spiked at 0.1 mg/kg and stored at -20 °C or below

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Soya bean seed (0.1 mg/kg)	0 30 91 178 240 357	85, 86, 88 97, 103 100, 107 91, 91 99, 101 104, 105	– 100 104 91 100 105	86 96 99 87 93 96	RM 30A-3	SBR-0003

Table 41 Stability of flumioxazin residues in a range of of fresh and processed plant matrices with high starch content, spiked at 0.1–1.0 mg/kg and stored at –20 °C or below

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Maize grain (0.1 mg/kg)	0 162 293 404	108, 110, 103 87, 87 89, 72 85, 82	– 87 81 84	107 87 92 75	RM 30A-3	SBR-0078
Potato tubers (0.1 mg/kg)	0 92 196 274	109, 116, 118 83, 117 92, 92 93, 104	– 100 92 99	114 106 96 111	RM 30A-3	SBR-0011
Potato tubers (1.0 mg/kg)	0 92 196 274	86, 88, 99 88, 89 83, 87 80, 80	– 89 85 80	91 97 113 100	RM 30A-3	SBR-0011

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Potato tubers (0.2 mg/kg)	0 218 279	92, 94, 95 113, 114 89, 93	– 114 91	94 118 104	RM 30A-2	SBR-0091
Potato chips (0.2 mg/kg)	0 279	95, 96, 99 94, 95	– 95	97 98	RM 30A-2	SBR-0091
Potato flakes (0.2 mg/kg)	0 279	91, 89, 91 92, 93	– 93	90 104	RM 30A-2	SBR-0091

Table 42 Stability of flumioxazin residues in a range of of fresh and processed plant matrices with high acid content, spiked at 0.05–0.2 mg/kg and stored at –20 °C or below

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Blueberry (0.1 mg/kg)	0 176	88, 82, 81 100, 102, 102	– 101	84 102	RM 30A-3	SBR-0115
Strawberry (0.2 mg/kg)	252 254	90, 100 100, 100	95 [a] 100 [a]	115 70	RM 30A-1	SBR-0109
Grape (0.05 mg/kg)	0 29 93 198	98, 101, 105 129, 115 93, 93 74, 100	– 122 93 87	101 116 103 95	RM 30A-1	SBR-0025
Grape juice (0.05 mg/kg)	0 30 68	94, 100, 102 111, 113 105, 92	– 112 99	99 99 101	RM 30A-1	SBR-0025
Dried grapes (0.05 mg/kg)	0 30 90 188	105, 114, 114 88, 104 106, 96 94, 83	– 96 101 89	111 99 118 114	RM 30A-1	SBR-0025

[a] % nominal residue remaining. No analysis of Day-0 sample

Table 43 Stability of 1-OH-HPA (flumioxazin metabolite) residues in a range of plant matrices spiked at 0.05–0.5 mg/kg and stored at –20 °C or below

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Sugar cane (0.1 mg/kg)	0 29 65 93 393	98, 97, 84 95, 99 99, 108 99, 92 82, 87	– 97 104 96 85	93 97 108 101 95	RM 30M	SBR-0023
Sugar (0.1 mg/kg)	0 14 35 78	106, 106, 102 86, 93 78, 69 79, 95	– 90 74 87	105 94 76 94	RM 30M	SBR-0022
Almond hulls (0.5 mg/kg)	0 27 55 131 263	88, 89, 90 94, 97 77, 81 77, 80 70, 72	– 96 79 79 71	89 94 80 76 70	RM 30M	SBR-0024
Cottonseed gin trash (0.05 mg/kg)	0 34 64 140 247	95, 97, 103 113, 114 84, 91 72, 80 104, 106	– 114 88 76 105	98 116 88 78 99	RM-30M	SBR-0026

USE PATTERNS

Information on GAP in the USA was provided to the Meeting on the use of flumioxazin, available as WG, SC or WP formulations, often co-formulated with other herbicides. The Meeting also noted that flumioxazin registrations existed in Australia, Europe, Canada, Latin America and some countries in Asia.

The following table summarizes the representative critical GAPs in the USA for crops relevant to the available residue field trials.

Table 44 Representative registered uses of flumioxazin (510 g ai/kg WG formulations)

Crop	Country	Application (max)		Max/season		PHI (days)	Comments
		kg ai/ha	water L/ha	no	kg ai/ha		
Pome fruit	USA	0.42	140–280		0.84	60	Directed inter-row band sprays, up to pink bud or bud-burst, min 30 day RTI
Stone fruit	USA	0.42	140–280		0.84	60	Directed inter-row band sprays, up to bud break, min 30 day RTI
Bush berries	USA	0.42	140–280		0.42	7	Directed inter-row band sprays. Min 30 day RTI
Grapes	USA	0.42	140–280		0.84	60	Directed inter-row band sprays. Min 30 day RTI. Not after bud-break on table grapes
Strawberries	USA	0.105	140–280		0.105		Pre-plant (at least 30 days before transplanting)
	USA	0.105	140–280		0.105		Broadcast to dormant plants
	USA	0.105	140–280		0.105		Directed inter-row band application up to fruit-set
Olives	USA	0.42	140–280		0.84	60	Directed inter-row band sprays. Min 30 day RTI
Pomegranates	USA	0.42	140–280		0.84	60	Directed inter-row band sprays. Min 30 day RTI
Garlic	USA	0.21	140–280		0.21		Pre-emergent application up to 3 days after planting
Onion, bulb	USA	0.07	140–180		0.105	45	Apply from 2-leaf and 6-leaf stage (BBCH12–16). Min 14 day RTI
Cabbage, head	USA	0.14	140–280		0.28		Pre-plant directed inter-row application (between raised plastic mulched beds)
Cucurbit vegetables	USA	0.14	140–280		0.28		Pre-plant directed inter-row applications (between raised plastic mulched beds), up to 14 days before planting
	USA	0.14	140–280		0.28		Directed inter-row band application up to 21 days after transplanting/emergence, not after start of flowering
Fruiting	USA	0.14	140–280		0.28		Pre-plant directed inter-row applications (between raised plastic mulched beds), up to 14 days before

Crop	Country	Application (max)		Max/season		PHI (days)	Comments
		kg ai/ha	water L/ha	no	kg ai/ha		
vegetables							planting
	USA	0.14	140–280		0.28		Directed inter-row band application up to 21 days after transplanting/emergence. Not after start of flowering
Beans, dry (incl lentils)	USA	0.07	140–280		0.07		Pre-plant or pre-emergent (up to 2 days after sowing)
	USA	0.105	140–560		0.105	5	Apply when crop is mature and at least 80% of pods are yellowing (BBCH 87–89)
Field peas	USA	0.07	140–280		0.07		Pre-plant or pre-emergent (up to 2 days after sowing)
	USA	0.105	140–560		0.105	5	Apply when crop is mature and at least 80% of pods are yellowing (BBCH 87–89)
Soya bean	USA	0.105	140–280		0.105		Pre-plant or pre-emergent (up to 3 days after sowing). No grazing or use for stock feed
Potato	USA	0.053	140–280		0.053		Pre-emergent after hilling or to soil-covered potatoes
Sweet potato	USA	0.105	140–280		0.105		Pre-plant
Artichoke, Globe	USA	0.21	94–280		0.21		Pre-plant (annual varieties) or pre-emergence (perennial varieties)
Asparagus	USA	0.21	140–280		0.21		Broadcast application min 14 days prior to spear emergence (perennial varieties) or fern emergence (annual varieties)
Celery	USA	0.105	140–280		0.105		Pre-transplant
	USA	0.105	140–280		0.105		Broadcast application, 3–7 days after transplanting
Maize	USA	0.105	140–280		0.105		Broadcast application 14–30 days prior to sowing
Wheat	USA	0.07	140–280		0.07		Pre-plant or pre-emergent (up to 2 days after sowing) in minimum tillage fields. No grazing until wheat is 13 cm high
	USA	0.07	min 93 air 47		0.07	10	Apply when crop reaches BBCH 87 (hard dough stage, grain 70% DM)
Sugar cane	USA	0.28	140–280		0.42		Broadcast up to 14 days before planting or broadcast pre-emergent
	USA	0.14	140–280		0.42	90	Directed inter-row band applications after canes are 60 cm height or at layby (canes > 76 cm height). Min 14 day application interval

Crop	Country	Application (max)		Max/season		PHI (days)	Comments
		kg ai/ha	water L/ha	no	kg ai/ha		
Tree nuts	USA	0.42	140–280		0.84	60	Directed inter-row band sprays. Min 60 day RTI
Cotton	USA	0.07	140–280		0.14		Autumn or spring burndown, up to 21 days before planting
	USA	0.07	140–280		0.14	60	Directed inter-row band applications after cotton is 15 cm height or at layby (cotton > 40 cm height). Min 30 day application interval. Use with non-ionic adjuvant
Linseed (flax)	USA	0.105	140–560		0.105	5	Apply when crop is mature and at least 75% of the seed heads are brown in colour (BBCH 87–89). Mix with MSO adjuvant
Sunflower seed Safflower seed	USA	0.105	140–560		0.105	5	Apply when crop is mature (BBCH 86–87—seedheads yellowing and the bracts turning brown). Mix with MSO adjuvant
Peanut	USA	0.105	140		0.105		Pre-plant or pre-emergent (up to 2 days after sowing). With adjuvant. No grazing or use for stock feed
Mints (spearmint, peppermint)	USA	0.14	140–180		0.28	80	Autumn-spring applications to established dormant plants. At least 60 days between applications
Alfalfa	USA	0.14	94–280		0.28	25	After last cut (Autumn) and/or after 1st cut, before crop reaches 15 cm height. PHI is for cutting and grazing

Pome fruit = apple, crabapple, loquat, mayhaw, pear, pear (oriental) and quince

Stone Fruit = apricot, cherries (sweet and tart), nectarine, peach, plum (chickasaw, damson, japanese), plumcot and prune

Bushberries = aronia berry, black currant, blueberry (highbush, rabbit-eye and lowbush), buffalo currant, chilean guava, cranberry (highbush), elderberry, european barberry, gooseberry, honeysuckle (edible), huckleberry, jostaberry, juneberry, lingonberry, native currant, red currant, salal and sea buckthorn

Cucurbits = chayote (fruit); chinese waxgourd (chinese preserving melon); citron melon; cucumber; gherkin; gourd, edible (includes hyotan, cucuzza, hechima, chinese okra); Momordica spp. (includes balsam apple, balsam pear, bittermelon, chinese cucumber); muskmelon (includes cantaloupe); pumpkin; squash, summer; squash, winter (includes butternut squash, calabaza, hubbard squash, acorn squash, spaghetti squash) and watermelon

Fruiting vegetables = eggplant, groundcherry (Physalis spp), okra, pepino, peppers (Capsicum spp incl bell, chili, cooking, pimento & sweet), tomatillo and tomato

Tree nuts = almond, beechnut, betelnut, black walnut, brazil nut, butternut, cashew, chestnut, chinquapin, coconut, english walnut, filbert (hazelnut), ginkgo, heartnut, hickory nut, macadamia nut, oak, pecan, pili nut, pine nut, pistachio and tropical almond

Dry beans = Dried cultivars of bean (Lupinus), bean (Phaseolus) (incl field bean, kidney bean, lima bean (dry), navy bean, pinto bean, tepary bean); bean (Vigna) (incl adzuki bean, blackeyed pea, catjang, cowpea, crowder pea, moth bean, mung bean, rice bean, southern pea, urd bean); broad bean (dry); chickpea; guar; lablab bean, lentil

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials involving soil or foliar treatments of flumioxazin to the following crops.

Group	Crop	Countries	Table no
Pome fruits	Apple, Pear	USA	45
Stone fruits	Cherry, Peach Plum	USA	46
Berries and other small fruit	Blueberry	USA	47
	Grape	USA	48
	Strawberries	USA	49
Assorted tropical and sub-tropical fruits	Olives	USA	50
	Pomegranate	USA	51
Bulb vegetables	Onion, dry bulb	USA	52
Brassica vegetables	Cabbage	USA	53
Fruiting vegetables, Cucurbits	Cucumber	USA	54
	Melons	USA	55
	Summer squash	USA	56
Fruiting vegetables, other than Cucurbits	Peppers	USA	57
	Tomato	USA	58
Pulses	Beans (dry)	USA	59
	Peas (dry)	USA	60
	Soya bean (dry)	USA	61
Root and tuber vegetables	Potato	USA	62
Stalk and stem vegetables	Artichoke, Globe	USA	63
	Asparagus	USA	64
	Celery	USA	65
Cereal grains	Maize	North America	66
	Wheat	USA	67
Grasses for sugar or syrup production	Sugar cane	USA	68
Tree nuts	Almond	USA	69
	Pecan	USA	70
Oilseed	Cottonseed	USA	71
	Rape seed	USA	72
	Peanut	USA	73
	Sunflower seed	USA	74
Herbs	Mint leaves and oil	USA	75
Legume animal feeds	Alfalfa forage and fodder	USA	76, 77
	Peanut vines and fodder	USA	78
	Soya bean forage and fodder	USA	79
Straw, forage, fodder of cereal grains	Maize forage and fodder	USA	80
	Wheat forage, hay and straw	USA	81, 82

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. 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.

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.

Pome fruits

In supervised trials on pome fruit (12 on apples and six on pears) conducted in the USA during 2002–2003, two inter-row/berm soil treatments of 0.42–0.45 kg ai flumioxazin/ha (WG or SC formulations) were applied using tractor-mounted boom sprayers or back-pack sprayers with hand-held booms. Treatments were applied about 60 days apart, with the last application about 60 days before harvest.

Duplicate samples of mature fruit (min 2 kg or 24 fruit) were frozen within 2 hours and analysed for flumioxazin within 1 year of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 80–115% and the validated LOQ was 0.02 mg/kg.

Table 45 Residues in pome fruit from supervised trials in the USA involving two directed inter-row soil applications of flumioxazin (SC or WG formulations)

POME FRUIT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
PEAR									
USA, 2002 Orefield, PA (Bartlett)	2	0.43 0.44	354 362	0.87	whole fruit	59	< 0.02, < 0.02	< 0.02	SBR-0029 V-24678-02-C
USA, 2002 Soap Lake, WA (Anjou)	2	0.445 0.429	164 205	0.874	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0029 V-24678-02-A
USA, 2002 Ukiah, CA (Bosc)	2	0.427 0.436	186 189	0.863	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0029 V-24678-02-B
USA, 2003 Hood River, OR (Bosc)	2	0.419 0.434	270 311	0.853	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0029 V-24678-03-F
USA, 2003 Ukiah, CA (Bosc)	2	0.434 0.434	189 189	0.868	whole fruit	61	< 0.02, < 0.02	< 0.02	SBR-0029 V-24678-03-D
USA, 2003 White Salmon, WA (Bosc)	2	0.434 0.439	282 314	0.873	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0029 V-24678-03-E
APPLE									

POME FRUIT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2002 Conklin, MI (Red Delicious)	2	0.40 0.431	260 266	0.861	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-C
USA, 2002 Eckert, CO (Yellow Delicious)	2	0.434 0.445	163 167	0.879	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-E
USA, 2002 Ephrata, WA (Rome)	2	0.431 0.432	200 201	0.863	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-F
USA, 2002 Hood River, OR (Jonagold)	2	0.445 0.441	293 294	0.886	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-D
USA, 2002 Monetta, SC (Gala)	2	0.43 0.431	259 255	0.861	whole fruit	56	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-B
USA, 2002 Orefield, PA (Rome)	2	0.43 0.429	354 353	0.859	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-A
USA, 2003 Conklin, MI (Red Delicious)	2	0.431 0.432	270 259	0.863	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-J
USA, 2003 North Rose, NY Golden Delicious)	2	0.441 0.432	287 282	0.873	whole fruit	61	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-H
USA, 2003 Orefield, PA (Rome)	2	0.429 0.445	355 368	0.874	whole fruit	58	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-I
USA, 2003 Parkdale, OR (Jonagold)	2	0.441 0.438	285 314	0.879	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-03-M
USA, 2003 Payette, ID (Rome)	2	0.426 0.423	279 276	0.849	whole fruit	61	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-03-L
USA, 2003 Santa Maria, CA (Fuji)	2	0.424 0.421	276 275	0.845	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-K

Stone fruits

Cherry, peach, and plum

In supervised trials on stone fruit (six on cherries, nine on peaches, and six on plums) conducted in the USA during 2002–2003, two inter-row/berm soil treatments of 0.42–0.45 kg ai flumioxazin/ha (WG or SC formulations) were applied using tractor-mounted boom sprayers or back-pack sprayers with hand-held booms. Treatments were applied 50–60 days apart (except in two trials with shorter intervals of 34 and 15 days) with the last application about 60 days before harvest.

Duplicate samples of mature fruit (min 1 kg cherries, 2 kg peaches, plums) were frozen within 2 hours and after removing the stones, were analysed for flumioxazin within 10 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 75–120% and the validated LOQ was 0.02 mg/kg.

Table 46 Residues in stone fruit from supervised trials in the USA involving two directed inter-row soil applications of flumioxazin (SC or WG formulations)

STONE FRUIT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
CHERRY									
USA, 2002 Casnovia, MI (Montmorency)	2	0.432 0.427	256 253	0.859	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0027 V-24694-B
USA, 2002 Conklin, MI (Montmorency)	2	0.427 0.427	261 255	0.854	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0027 V-24694-A
USA, 2002 Ephrata, WA (Van)	2	0.425 0.427	186 187	0.852	fruit without stone	61	< 0.02, < 0.02	< 0.02	SBR-0027 V-24694-F
USA, 2002 Madera, CA (Brooks)	2	0.42 0.425	324 326	0.845	fruit without stone	59	< 0.02, < 0.02	< 0.02	SBR-0027 V-24694-D 34 day RTI
USA, 2002 Orefield, PA (Montmorency)	2	0.435 0.425	356 349	0.86	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0027 V-24694-C 15 day RTI
USA, 2002 Parkdale, OR (Bing)	2	0.435 0.413	321 344	0.848	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0027 V-24694-E
PEACH									
USA, 2002 Athens, GA (Contender)	2	0.423 0.432	319 329	0.855	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0028 V-24686-B
USA, 2002 Conklin, MI (Red Heaven)	2	0.435 0.43	245 255	0.865	fruit without stone	59	< 0.02, < 0.02	< 0.02	SBR-0028 V-24686-D
USA, 2002 Mexia, TX (Redskins)	2	0.425 0.43	326 330	0.855	fruit without stone	55	< 0.02, < 0.02	< 0.02	SBR-0028 V-24686-C
USA, 2002 Orefield, PA (Suncrest)	2	0.445 0.428	365 352	0.873	fruit without stone	59	< 0.02, < 0.02	< 0.02	SBR-0028 V-24686-A
USA, 2002 Selma, CA (September Sun)	2	0.445 0.437	192 189	0.882	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0028 V-24686-E
USA, 2003 Athens, GA (Contender)	2	0.435 0.435	280 279	0.87	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0028 V-24686-F
USA, 2003 Batesburg, SC (Monroe)	2	0.432 0.437	247 254	0.869	fruit without stone	53	< 0.02, < 0.02	< 0.02	SBR-0028 V-24686-G
USA, 2003 Gridley, CA (Starn)	2	0.43 0.43	234 234	0.86	fruit without stone	59	< 0.02, < 0.02	< 0.02	SBR-0028 V-24686-I
USA, 2003 Selma, CA (September Sun)	2	0.437 0.43	190 187	0.867	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0028 V-24686-H
PLUM									

STONE FRUIT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2002 Conklin, MI (Vision)	2	0.437 0.43	262 249	0.867	fruit without stone	59	< 0.02, < 0.02	< 0.02	SBR-0030 V-24539-B
USA, 2002 Hughson, CA (French)	2	0.428 0.428	375 375	0.856	fruit without stone	46 53 60 68 75	< 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	SBR-0030 V-24539-F
USA, 2002 Madera, CA (Fortune)	2	0.432 0.423	333 326	0.855	fruit without stone	59	< 0.02, < 0.02	< 0.02	SBR-0030 V-24539-D
USA, 2002 Porterville, CA (Angelino)	2	0.440 0.435	308 325	0.875	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0030 V-24539-C
USA, 2002 Yuba City, CA (French)	2	0.43 0.43	188 187	0.86	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0030 V-24539-E
USA, 2003 Zillah, WA (Autumn Sweet)	2	0.42 0.423	310 314	0.843	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0030 V-24539-H

Berries and other small fruits

Blueberries

In supervised trials on blueberries (six) conducted in the USA during 2003, two inter-row/berm soil treatments of 0.41–0.45 kg ai flumioxazin/ha (WG formulations) were applied using back-pack sprayers with 1–4 nozzle hand-held booms. Treatments were applied 50–113 days apart with the last application 6–8 days before harvest (except in one lowbush trial where a single application was made to dormant bushes).

Duplicate samples of mature fruit (min 1 kg except at two sites) were frozen within 4 hours and analysed for flumioxazin within 6 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 74–113% and the validated LOQ was 0.02 mg/kg.

Table 47 Residues in blueberries from supervised trials in the USA involving 1–2 directed inter-row soil applications of flumioxazin (WG formulations)

BLUEBERRY COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2003 Aurora, OR (Bluecrop)	2	0.44 0.45	287 299	0.89	berries	7	< 0.02, < 0.02	< 0.02	SBR-0115 OR11
USA, 2003 Bridgeton, NJ, (Duke)	2	0.45 0.44	238 231	0.89	berries	7	< 0.02, < 0.02	< 0.02	SBR-0115 NJ16
USA, 2003 Castle Hayne, NC (Croatan)	2	0.42 0.41	274 270	0.83	berries	6	< 0.02, < 0.02	< 0.02	SBR-0115 NC15

BLUEBERRY COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2003 Holt, MI (Jersey)	2	0.43 0.45	192 203	0.88	berries	8	< 0.02, < 0.02	< 0.02	SBR-0115 MI21 56g sample size
USA, 2003 Jonesboro, ME (Wild blueberries) Lowbush	1	0.45	194	0.45	berries	99	< 0.02, < 0.02	< 0.02	SBR-0115 ME02 Dormant bushes
USA, 2003 Onondaga, MI (Bluecrop)	2	0.45 0.44	200 197	0.89	berries	8	< 0.02, < 0.02	< 0.02	SBR-0115 MI22 227g sample size

Grapes

In supervised trials on grapes (12) conducted in the USA during 1999, two directed inter-row/berm soil treatments of 0.4–0.43 kg ai flumioxazin/ha (WG formulations) with added crop oil were applied using tractor-mounted boom sprayers or back-pack sprayers with hand-held booms. Treatments were applied about 60 days apart with the last application about 60 days before harvest.

Duplicate samples of grapes (min 12 bunches or 1 kg) were frozen within 4 hours and analysed for flumioxazin within 6 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.01 and 0.05 mg/kg ranged from 82–123% and the LOQ was 0.01 mg/kg.

Table 48 Residues in grapes from supervised trials in the USA involving two inter-row soil applications of flumioxazin (WG formulations)

GRAPES COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1999 Breinigsville, PA (Vidal 256)	2	0.421 0.419	187 187	0.84	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-B
USA, 1999 Dundee, NY (Delaware)	2	0.416 0.408	185 181	0.824	bunches	59	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-A
USA, 1999 Dunnigan, CA (Symphony)	2	0.419 0.418	186 186	0.837	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-C
USA, 1999 Hughson, CA (Thompson seedless)	2	0.421 0.427	234 238	0.848	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-N
USA, 1999 Hughson, CA (Thompson seedless)	2	0.86 0.844	240 235	1.704	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-N 2x
USA, 1999 Kerman, CA (Thompson seedless)	2	0.42 0.425	184 187	0.845	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-L

GRAPES COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1999 Madera, CA (Thompson seedless)	2	0.418 0.423	186 188	0.841	bunches	59	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-D
USA, 1999 Orland, CA (Zinfindel)	2	0.422 0.42	218 223	0.842	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-G
USA, 1999 Orland, CA (Zinfindel)	2	0.83 0.826	214 219	1.656	bunches	60	< 0.01, < 0.01	0.01	SBR-0025 V-20108-G 2x
USA, 1999 Poplar, CA (Thompson seedless)	2	0.42 0.422	186 187	0.842	bunches	59	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-E
USA, 1999 San Luis Obispo, CA (Chardonnay)	2	0.426 0.42	238 234	0.846	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-H
USA, 1999 Temecula, CA (Merlot)	2	0.424 0.434	189 191	0.858	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-I
USA, 1999 Trinidad, W (Gamay Noir)	2	0.422 0.419	188 187	0.841	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-J
USA, 1999 Watsonville, CA (Pinot Noir)	2	0.398 0.428	209 224	0.826	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-F
USA, 1999 Watsonville, CA (Pinot Noir)	2	0.836 0.828	219 217	1.664	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-F 2x

Strawberry

In supervised trials on strawberries (five) conducted in the USA during 2002, one inter-row soil treatment of 0.1–0.11 kg ai flumioxazin/ha (WG formulations) was applied 1–2 days before harvest using shielded back-pack sprayers with 1–4 nozzle mini-booms. In three additional trials, two applications of 0.1 kg ai/ha flumioxazin were made, the first being a broadcast application to dormant strawberries and the second as an inter-row shielded application 1–2 days before harvest.

Duplicate samples of at least 1 kg mature fruit (with sepals removed) were frozen within 4 hours and analysed for flumioxazin within 7 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 100–120% and the validated LOQ was 0.02 mg/kg.

Table 49 Residues in strawberries from supervised trials in the USA involving 1–2 inter-row soil applications of flumioxazin (WG formulations)

STRAWBERRIES COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2002 Bridgeton, NJ (Early Glow)	2	0.109 0.104	215 253	0.213	berries	1	< 0.02, < 0.02	< 0.02	SBR-0109 08063.02-NJ04

STRAWBERRIES COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2002 Clinton, NC (Camarosa)	1	0.108	187	0.108	berries	1	< 0.02, < 0.02	< 0.02	SBR-0109 08063.02-NC06
USA, 2002 Holt, MI (Mira)	2	0.104 0.110	178 187	0.214	berries	1	0.034, 0.021	0.03	SBR-0109 08063.02-MI04
USA, 2002 Live Oak, FL (Sweet Charlie)	1	0.108	187	0.108	berries	1	< 0.02, < 0.02	< 0.02	SBR-0109 08063.02-FL08
USA, 2002 Madera, CA (Hecker)	1	0.106	281	0.106	berries	1	< 0.02, < 0.02	< 0.02	SBR-0109 08063.02-CA26
USA, 2002 Mt. Vernon, WA (Totem)	2	0.108 0.113	187 196	0.221	berries	1	< 0.02, < 0.02	< 0.02	SBR-0109 08063.02-WA36
USA, 2002 Salinas, CA (Diamonte)	1	0.108	327	0.108	berries	2	0.034, 0.036	0.04	SBR-0109 08063.02-CA*24
USA, 2002 Watsonville, CA (Camarosa)	1	0.105	346	0.105	berries	1	0.036, 0.05	0.04	SBR-0109 08063.02-CA*25

Assorted tropical and sub-tropical fruits

Olives

In supervised trials on olives (five) conducted in the USA during 2008, two directed inter-row/berm soil treatments of 0.4–0.43 kg ai flumioxazin/ha (WG formulations) with added crop oil were applied using back-pack sprayers with hand-held 3-nozzle minibooms. Treatments were applied about 60 days apart with the last application 56–59 days before harvest.

Duplicate samples of olives were stored refrigerated for up to 2 days before pitting, with the pitted olives (min 0.5 kg) frozen within 2.5 hours and analysed for flumioxazin within 18 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 76–122% and the validated LOQ was 0.02 mg/kg.

Table 50 Residues in olives from supervised trials in the USA involving two inter-row soil applications of flumioxazin (WG formulations)

OLIVE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2008 Orange Cove, CA (Manzanillo)	2	0.415 0.421	326 330	0.841	fruit without pits	59	< 0.02, < 0.02	< 0.02	SBR-0130 CA94
USA, 2008 Orange Cove, CA (Manzanillo)	2	0.424 0.423	232 240	0.852	fruit without pits	59	< 0.02, < 0.02	< 0.02	SBR-0130 CA95 not independent

OLIVE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2008 Glenn, CA (Korondiki 1-38 clone)	2	0.435 0.437	224 224	0.874	fruit without pits	57	< 0.02, < 0.02	< 0.02	SBR-0130 CA92
USA, 2008 Glenn, CA (Arbosama 1-43 line)	2	0.424 0.408	218 210	0.829	fruit without pits	57	< 0.02, < 0.02	< 0.02	SBR-0130 CA93 not independent
USA, 2008 Glenn, CA (Arbegnina 1-18 clone)	2	0.423 0.432	217 222	0.852	fruit without pits	56	< 0.02, < 0.02	< 0.02	SBR-0130 CA91 not independent

Pomegranate

In supervised trials on pomegranates (three) conducted in the USA during 2008, two directed inter-row/berm soil treatments of 0.4–0.43 kg ai flumioxazin/ha (WG formulations) with adjuvant were applied using back-pack sprayers with hand-held 3-nozzle minibooms. Treatments were applied about 60 days apart with the last application 57–59 days before harvest.

Duplicate samples of fruit (min 24 fruit, 6 kg) were frozen within 4 hours and analysed for flumioxazin within 17 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 80–103% and the validated LOQ was 0.02 mg/kg.

Table 51 Residues in pomegranates from supervised trials in the USA involving two inter-row soil of flumioxazin (WG formulations)

POMEGRANATE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
GAP:USA	2	0.42	140–280	0.84		60	Directed inter-row soil sprays, min 30 day RTI		
USA, 2008 Kettleman City, CA (Wonderful)	2	0.42 0.423	305 333	0.841	whole fruit	59	< 0.02, < 0.02	< 0.02	SBR-0131 CA82
USA, 2008 Kettleman City, CA (Wonderful)	2	0.429 0.427	240 239	0.852	whole fruit	59	< 0.02, < 0.02	< 0.02	SBR-0131 CA83 not independent
USA, 2008 Gridley, CA (Wonderful)	2	0.407 0.411	291 294	0.818	whole fruit	57	< 0.02, < 0.02	< 0.02	SBR-0131 CA96

Bulb vegetables

Onion, dry bulb

In supervised trials on bulb onions (nine) conducted in the USA during 2001, two foliar broadcast sprays of 0.1–0.11 kg ai flumioxazin/ha (WG formulations) with added adjuvant were applied using tractor-mounted, wheeled or back-pack sprayers with 3–6 nozzle minibooms. The first applications were made when the onions were at or about the 2-leaf stage, re-treatment intervals were 29–78 days and the last applications were 42–49 days before harvest. Phytotoxicity was observed in most trials.

Duplicate samples of topped and trimmed dry onion bulbs (min 12 bulbs, 1.3 kg) were frozen within 1 hour and analysed for flumioxazin within 2 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 70–120% and the validated LOQ was 0.02 mg/kg.

Table 52 Residues in onion bulbs from supervised trials in the USA involving two broadcast foliar applications of flumioxazin (WG formulations)

ONION, BULB COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2001 Bridgeton, NJ (Santana)	2	0.11 0.102	250 272	0.212	bulb	42	< 0.02, < 0.02	< 0.02	SBR-0083 07389.01-NJ02 RTI 62 days
USA, 2001 Celeryville, OH (Burgos)	2	0.102 0.108	317 365	0.21	bulb	42	< 0.02, < 0.02	< 0.02	SBR-0083 07389.01-OH*02 RTI 37 days
USA, 2001 Fort Collins, CO (Vision)	2	0.115 0.101	206 178	0.216	bulb	43	< 0.02, < 0.02	< 0.02	SBR-0083 07389.01-CO01 RTI 64 days
USA, 2001 Freeville, NY (F1 Candy)	2	0.109 0.108	285 285	0.216	bulb	44	< 0.02, < 0.02	< 0.02	SBR-0083 07389.01-NY01 RTI 51 days Includes 11 d field drying
USA, 2001 Fresno, CA (Cimarron)	2	0.114 0.11	391 304	0.224	bulb	44	< 0.02, < 0.02	< 0.02	SBR-0083 07389.01-CA128 RTI 72 days
USA, 2001 Laingsburg, MI USA, 2001 (Hustler F1)	2	0.11 0.109	191 194	0.219	bulb	44	< 0.02, < 0.02	< 0.02	SBR-0083 07389.01-MI02 RTI 33 days
USA, 2001 Prosser, WA (Teton)	2	0.104 0.103	148 147	0.207	bulb	45	< 0.02, < 0.02	< 0.02	SBR-0083 07389.01-WA*04 RTI 33 days
USA, 2001 Salinas, CA (Tahoe)	2	0.112 0.105	325 312	0.217	bulb	49	< 0.02, < 0.02	< 0.02	SBR-0083 07389.01-CA*03 RTI 29 days Includes 9 d field drying
USA, 2001 Weslaco, TX (Cougar)	2	0.106 0.105	208 219	0.212	bulb	48	< 0.02, < 0.02	< 0.02	SBR-0083 07389.01-TX01 RTI 78 days

Brassica vegetables

Cabbage

In supervised trials on cabbage (eight) conducted in the USA during 2006, one pre-plant broadcast soil treatment of 0.05 kg ai/ha or 0.1–0.11 kg ai flumioxazin/ha (WG formulations) were applied using tractor-mounted or back-pack sprayers with 3–8 nozzle booms. Phytotoxicity was observed in most trials, particularly in the plots treated at the higher rate.

Duplicate samples of mature cabbage heads with wrapper leaves (min 12 heads) from most plots were quartered or halved in the field (to give sample sizes of at least 1.8 kg). In three trials, smaller sample sizes were taken because of reduced plant numbers. Samples were frozen within 2 hours and analysed for flumioxazin within 8 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 85–90% and the validated LOQ was 0.02 mg/kg.

Table 53 Residues in cabbage heads (with wrapper leaves) from supervised trials in the USA involving one pre-plant broadcast soil application of flumioxazin (WG formulation)

CABBAGE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2006 Freeville, NY (Bobcat)	1	0.052	277	0.052	head with wrapper leaves	94	< 0.02, < 0.02	< 0.02	SBR-0129 NY08 Subsampled in the field
	1	0.107	286	0.107	head with wrapper leaves	94	< 0.02, < 0.02	< 0.02	
USA, 2006 Bridgeton, NJ (Wisconsin Golden Acre)	1	0.051	327	0.051	head with wrapper leaves	55	< 0.02, < 0.02	< 0.02	SBR-0129 NJ15 Subsampled in the field
	1	0.107	337	0.107	head with wrapper leaves	55	< 0.02, < 0.02	< 0.02	
USA, 2006 Clinton, NC (Bravo)	1	0.053	326	0.053	head with wrapper leaves	67	< 0.02, < 0.02	< 0.02	SBR-0129 NC10 Subsampled in the field
	1	0.106	326	0.106	head with wrapper leaves	67	< 0.02, < 0.02	< 0.02	
USA, 2006 Citra, FL (Bravo)	1	0.054	286	0.054	head with wrapper leaves	67	< 0.02, < 0.02	< 0.02	SBR-0129 FL24 Subsampled in the field
	1	0.108	286	0.108	head with wrapper leaves	67	< 0.02, < 0.02	< 0.02	
USA, 2006 Arlington, WI (Blue Vantage)	1	0.053	271	0.053	head with wrapper leaves	87	< 0.02, < 0.02	< 0.02	SBR-0129 WI15 Subsampled in the field
	1	0.106	271	0.106	head with wrapper leaves	87	< 0.02, < 0.02	< 0.02	
USA, 2006 Wesalco, TX (Blue Vantage)	1	0.053	284	0.053	head with wrapper leaves	98	< 0.02, < 0.02	< 0.02	SBR-0129 TX*26 Subsampled in the field
	1	0.107	284	0.107	head with wrapper leaves	98	< 0.02, < 0.02	< 0.02	
USA, 2006 Brighton, CO (Blue Dynasty)	1	0.054	190	0.054	head with wrapper leaves	84	< 0.02, < 0.02	< 0.02	SBR-0129 CO06
	1	0.106	187	0.106	whole plants [a]	84	< 0.02, < 0.02	< 0.02	
USA, 2006 Holtville, CA (Grenadier)	1	0.054	240	0.054	head with wrapper leaves	104	< 0.02, < 0.02	< 0.02	SBR-0129 CA61 Subsampled in the field
	1	0.108	242	0.108	head with wrapper leaves	104	< 0.02, < 0.02	< 0.02	

[a] Reduced sample size—only two whole plants able to be collected

Fruiting vegetables, cucurbits

Supervised trials on fruiting vegetables, cucurbits were conducted in the USA between 2003 and 2005.

Cucumber

In eight trials on cucumbers, two treatments of 0.14–0.17 kg ai flumioxazin/ha (WG formulations) were applied as inter-row shielded applications, the first application about 14 days before crop emergence or before transplanting and the second application about 21 days after transplanting or 28 days after the crop emergence (at or before flowering). Treatments were made using tractor-mounted or backpack sprayers with 1–4 shielded nozzle minibooms.

Duplicate samples of mature fruit (min 12 units, 1.8 kg). In two trials, samples were quartered or halved in the field. Samples were frozen within 2.5 hours and analysed for flumioxazin within 5 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 70–120% and the validated LOQ was 0.02 mg/kg.

Table 54 Residues in cucumbers from supervised trials in the USA involving two inter-row soil applications of flumioxazin (WG formulation)

CUCUMBER COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2005 Salisbury, MD (Genuine)	2	0.142 0.14 [a]	279 273	0.282	whole fruit	6	< 0.02, < 0.02	< 0.02	SBR-0121 MD08
USA, 2005 Charleston, SC (Poinsett 76)	2	0.141 0.173	265 289	0.313	whole fruit	15	< 0.02, < 0.02	< 0.02	SBR-0121 SC*02
USA, 2005 Holt, MI (Journey)	2	0.151 0.149	201 199	0.3	whole fruit	15	< 0.02, < 0.02	< 0.02	SBR-0121 MI05
USA, 2005 Citra, FL (Dasher II)	2	0.145 0.143	192 191	0.288	whole fruit	7	< 0.02, < 0.02	< 0.02	SBR-0121 FL19
USA, 2005 Arlington, WI (Zapata)	2	0.14 0.141	312 317	0.281	whole fruit	29	< 0.02, < 0.02	< 0.02	SBR-0121 WI07
USA, 2005 Clinton, NC (Dasher II)	2	0.14 0.141	204 205	0.281	whole fruit	11	< 0.02, < 0.02	< 0.02	SBR-0121 NC29
USA, 2005 Tifton, GA (Diva)	2	0.137 0.137 [a]	193 192	0.273	whole fruit	21	0.024, 0.027	0.03	SBR-0121 GA*07
USA, 2005 Wesalco, TX (Poinsett 76)	2	0.139 0.142	271 214	0.281	whole fruit	31	< 0.02, < 0.02	< 0.02	SBR-0121 TX*17

[a] 2nd application after the start of flowering

Melon (cantaloupe)

In eight trials on cantaloupes, two treatments of 0.14–0.15 kg ai flumioxazin/ha (WG formulations) were applied as inter-row shielded applications, the first application about 10–14 days before transplanting or 4–7 days before sowing and the second application about 21 days after transplanting or 28 days after the crop emergence (at or before flowering). Treatments were made using tractor-mounted or backpack sprayers with 1–2 shielded nozzle minibooms.

Duplicate samples of mature fruit (min 12 units) were subsampled in the field (2–5 cm longitudinal sections) to give sample sizes of at least 1.8 kg. Samples were frozen within 3 hours and analysed for flumioxazin within 4 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 72–108% and the validated LOQ was 0.02 mg/kg.

Table 55 Residues in melons from supervised trials in the USA involving two inter-row soil applications of flumioxazin (WG formulation)

MELON COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2003 Clinton, NC (Athena)	2	0.14 0.14	206 202	0.28	whole fruit	41	< 0.02, < 0.02	< 0.02	SBR-0112 NC13
USA, 2003 Five Points, CA (Gold Express)	2	0.14 0.14	202 219	0.28	whole fruit	47	< 0.02, < 0.02	< 0.02	SBR-0112 CA72
USA, 2003 Holt, MI (Athena)	2	0.14 0.15	190 195	0.29	whole fruit	69	< 0.02, < 0.02	< 0.02	SBR-0112 MI20
USA, 2003 Mesilla, NM (Top Mark SR)	2	0.14 0.14	231 206	0.289	whole fruit	53	< 0.02, < 0.02	< 0.02	SBR-0112 NM07
USA, 2003 Mesilla, NM (Top Mark SR)	2	0.14 0.14	230 229	0.28	whole fruit	51	< 0.02, < 0.02	< 0.02	SBR-0112 NM08
USA, 2003 Parlier, CA (Top Mark)	2	0.14 0.15	291 286	0.29	whole fruit	36	< 0.02, < 0.02	< 0.02	SBR-0112 CA73
USA, 2003 Wesalco, TX (Cruiser)	2	0.14 0.14	298 275	0.28	whole fruit	47	< 0.02, < 0.02	< 0.02	SBR-0112 TX*23
USA, 2003 Wesalco, TX USA, 2003 (Primo)	2	0.14 0.14	223 225	0.28	whole fruit	52	< 0.02, < 0.02	< 0.02	SBR-0112 TX22

Summer squash

In eight trials on summer squash, two treatments of 0.14–0.15 kg ai flumioxazin/ha (WG formulations) were applied as inter-row shielded applications, the first application about 10–14 days before planting or before crop emergence and the second application about 20–26 days after transplanting or 29–30 days after the crop emergence (at or before flowering or fruiting). Treatments were made using tractor-mounted or backpack sprayers with 1–4 shielded nozzle minibooms.

Duplicate samples of mature fruit (min 12 units, 1.8 kg). In three trials, samples were quartered or halved in the field. Samples were frozen within 25 minutes and analysed for

flumioxazin within 12 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 70–120% (except for one recovery at 130%) and the validated LOQ was 0.02 mg/kg.

Table 56 Residues in summer squash from supervised trials in the USA involving two inter-row soil applications of flumioxazin (WG formulation)

SUMMER SQUASH COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2004 Citra, FL (Gentry)	2	0.14 0.142	235 238	0.282	whole fruit	30	< 0.02, < 0.02	< 0.02	SBR-0120 FL12
USA, 2004 Davis, CA (Straight Neck Early Prolific)	2	0.145 0.141	292 318	0.286	whole fruit	14	< 0.02, < 0.02	< 0.02	SBR-0120 CA30
USA, 2004 Freeville, NY (Revune)	2	0.151 0.144	301 288	0.295	whole fruit	34	< 0.02, < 0.02	< 0.02	SBR-0120 NY04
USA, 2004 Holt, MI (Black Beauty)	2	0.144 0.149	193 200	0.294	whole fruit	16	< 0.02, < 0.02	< 0.02	SBR-0120 MI03
USA, 2004 Prosser, WA (Early Summer Crookneck)	2	0.139 0.141	273 277	0.28	whole fruit	25	< 0.02, < 0.02	< 0.02	SBR-0120 WA03
USA, 2004 Salisbury, MD (Seneca Prolific)	2	0.144 0.145	133 134	0.289	whole fruit	7	< 0.02, < 0.02	< 0.02	SBR-0120 MD03
USA, 2004 Tifton, GA (Crookneck Early Summer)	2	0.144 0.142 [a]	193 191	0.286	whole fruit	11	< 0.02, < 0.02	< 0.02	SBR-0120 GA*02
USA, 2004 Wesalco, TX (Golide)	2	0.143 0.143 [a]	257 239	0.286	whole fruit	12	< 0.02, < 0.02	< 0.02	SBR-0120 TX08

[a] 2nd application after the start of flowering

Fruiting vegetables other than cucurbits

Supervised trials on fruiting vegetables other than cucurbits were conducted in the USA in 2003.

Peppers (sweet, chili)

In nine trials on peppers (bell and non-bell/chilli), two treatments of 0.14–0.15 kg ai flumioxazin/ha (WG formulations) with added adjuvant were applied as inter-row shielded applications, the first application at transplanting or shortly after emergence and the second application from 15–21 days before harvest. Treatments were made using tractor-mounted or backpack sprayers with 1–2 shielded nozzle minibooms.

Duplicate samples of mature fruit (min 12 units, 1.8 kg) were frozen within 6 hours and analysed for flumioxazin within 27 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 86–121% (0.02 mg/kg spike level) and 59–111% (0.2 mg/kg spike level). The validated LOQ was 0.02 mg/kg.

Table 57 Residues in peppers from supervised trials in the USA involving two inter-row soil applications of flumioxazin (WG formulation)

PEPPERS COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
BELL PEPPER									
USA, 2003 Citra, FL (Camelot)	2	0.143 0.144	193 194	0.286	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0118 03-FL25
USA, 2003 Clinton, NC (Camelot)	2	0.14 0.136	194 188	0.276	whole fruit	19	< 0.02, < 0.02	< 0.02	SBR-0118 03-NC09
USA, 2003 Holtville, CA (Valiant)	2	0.143 0.146	108 110	0.29	whole fruit	20	< 0.02, < 0.02	< 0.02	SBR-0118 03-CA48
USA, 2003 Parlier, CA (Valiant)	2	0.145 0.143	149 147	0.287	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0118 03-CA49
USA, 2003 Tifton, GA (Capistrano)	2	0.144 0.145	192 194	0.289	whole fruit	15	< 0.02, < 0.02	< 0.02	SBR-0118 03-GA*10
USA, 2003 Wesalco, TX (Capistrano)	2	0.14 0.144	244 206	0.284	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0118 03-TX*16
NON-BELL PEPPER									
USA, 2003 Mesilla, NM (Big Jim Chile)	2	0.144 0.14	190 192	0.284	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0118 03-NM11
USA, 2003 Rocky Ford, CO (Joe Parker)	2	0.143 0.147	191 197	0.29	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0118 03-CO11
USA, 2003 Wesalco, TX (TAM Veracruz)	2	0.142 0.145	222 210	0.286	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0118 03-TX15

Tomato

In twelve trials on tomatoes, two treatments of 0.14–0.15 kg ai flumioxazin/ha (WG formulations) with added adjuvant were applied as inter-row shielded applications, the first application at transplanting or shortly after emergence and the second application from 15–21 days before harvest. Treatments were made using tractor-mounted or backpack sprayers with 1–2 shielded nozzle minibooms.

Duplicate samples of mature fruit (min 12 units, 1.8 kg) were frozen within 2.3 hours and analysed for flumioxazin within 7 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 70–120% (except for one recovery at 130%) and the validated LOQ was 0.02 mg/kg.

Table 58 Residues in tomatoes from supervised trials in the USA involving two inter-row soil applications of flumioxazin (WG formulation)

TOMATO COUNTRY, YEAR	APPLICATION	MATRIX	DAT	RESIDUES (MG/KG)	REFERENCE & COMMENTS

LOCATION (VARIETY)	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2003 Arlington, WI (Capri VF)	2	0.145 0.142	272 264	0.287	whole fruit	19	< 0.02, < 0.02	< 0.02	SBR-0117 03-WI06
USA, 2003 Charleston, SC (Sunleaper)	2	0.138 0.136	271 237	0.273	whole fruit	15	< 0.02, < 0.02	< 0.02	SBR-0117 03-SC*03
USA, 2003 Citra, FL (FLA47)	2	0.146 0.142	197 192	0.288	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0117 03-FL23
USA, 2003 Citra, FL (FLA47)	2	0.147 0.14	198 189	0.287	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0117 03-FL24 not independent
USA, 2003 Davis, CA (Hypeel 303)	2	0.149 0.141	299 236	0.29	whole fruit	19	< 0.02, < 0.02	< 0.02	SBR-0117 03-CA155
USA, 2003 Freeville, NY (Celebrity)	2	0.15 0.14	290 259	0.284	whole fruit	20	< 0.02, < 0.02	< 0.02	SBR-0117 03-NY04
USA, 2003 Glenn, CA (H-8892)	2	0.141 0.143	208 258	0.284	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0117 03-CA45
USA, 2003 Madera, CA (Rio Grande)	2	0.141 0.145	236 242	0.286	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0117 03-CA46
USA, 2003 Parlier, CA (Heinz 3155)	2	0.144 0.139	235 247	0.282	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0117 03-CA44
USA, 2003 Parlier, CA (Quality 21)	2	0.145 0.141	150 154	0.287	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0117 03-CA43
USA, 2003 Porterville, CA (Better Boy)	2	0.138 0.14	239 236	0.278	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0117 03-CA42 not independent
USA, 2003 Porterville, CA (UC82-L)	2	0.139 0.139	234 233	0.278	whole fruit		< 0.02, < 0.02	< 0.02	SBR-0117 03-CA41

Pulses

Supervised trials on pulses (beans, peas and soya beans) were conducted in North America between 1989 and 2009.

Beans (dry)

In twelve trials on beans, one foliar broadcast spray of 0.1–0.11 kg ai flumioxazin/ha (WG formulations) with added adjuvant was applied as a pre-harvest desiccant and harvest aid using tractor-mounted or back-pack sprayers with 4–11 nozzle booms.

Duplicate samples were harvested, allowed to dry in the field for up to 13 days before being shelled (manually or mechanically) to obtain minimum samples of 1 kg dry seeds. These samples were frozen within 5 hours and analysed for flumioxazin within 10 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 79–119% and the validated LOQ was 0.02 mg/kg.

Table 59 Residues in beans, dry from supervised trials in the USA involving one pre-harvest foliar application of flumioxazin (WG formulation)

BEANS, DRY COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2004 Fargo, ND (Navigator)	1	0.104	111	0.104	seeds	5 + 2	< 0.02, < 0.02	< 0.02	SBR-0114 ND11
USA, 2004 Fort Collins, CO (Bill Z)	1	0.106	190	0.106	seeds	5 + 1	0.02, 0.02	0.02	SBR-0114 CO13
USA, 2004 Fort Collins, CO (Ohello)	1	0.108	194	0.108	seeds	4 + 3	0.02, < 0.02	0.02	SBR-0114 CO12
USA, 2004 Freeville, NY (Cabarnet)	1	0.107	286	0.107	seeds	6 + 8	< 0.02, < 0.02	< 0.02	SBR-0114 NY18
USA, 2004 Fremont, OH (Midnight Black)	1	0.107	324	0.107	seeds	4 + 13	< 0.02, < 0.02	< 0.02	SBR-0114 OH*12
USA, 2004 Fremont, OH (Topaz)	1	0.106	323	0.106	seeds	4 + 13	< 0.02, < 0.02	< 0.02	SBR-0114 OH*13 not independent
USA, 2004 Holtville, CA (Apache)	1	0.106	162	0.106	seeds	5	< 0.02, < 0.02	< 0.02	SBR-0114 CA128
USA, 2004 Kimberly, ID (Othello)	1	0.102	183	0.102	seeds	5 + 10	0.02, < 0.02	0.02	SBR-0114 ID09
USA, 2004 Minot, ND (Maverick)	1	0.106	94	0.106	seeds	4 + 3	< 0.02, < 0.02	< 0.02	SBR-0114 ND09 not independant
USA, 2004 Minot, ND (Maverick)	1	0.103	93	0.103	seeds	4 + 3	0.02, < 0.02	0.02	SBR-0114 ND10
USA, 2004 Minot, ND (Maverick)	1	0.104	93	0.104	seeds	4	< 0.02, < 0.02	< 0.02	SBR-0114 ND14
USA, 2004 Scottsbluff, NE (Beryl)	1	0.106	205	0.106	seeds	6 + 6	0.04, 0.03	0.04	SBR-0114 NE03 not independent
USA, 2004 Scottsbluff, NE (Kelly Bean 99124)	1	0.103	203	0.103	seeds	6 + 6	0.04, 0.05	0.05	SBR-0114 NE04

DAT = Interval from last application to cutting + field drying interval (in days)

Peas (dry)

In thirteen trials on field peas, one foliar broadcast spray of 0.11 kg ai flumioxazin/ha (WG formulations) with added adjuvant was applied as a pre-harvest desiccant and harvest aid using tractor-mounted or back-pack sprayers with 4–8 nozzle booms.

Duplicate plant samples were collected using small plot combines or cut and harvested using a stationary combine to obtain minimum samples of 1 kg dry seeds. These samples were frozen within 6 hours and analysed for flumioxazin within 4.5 months of harvest using method

RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 96–112% and the validated LOQ was 0.02 mg/kg.

Table 60 Residues in peas, dry from supervised trials in North America involving one pre-harvest foliar application of flumioxazin (WG formulation)

PEAS, DRY COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
Canada, 2009 Blaine Lake, Saskatchewan (Golden)	1	0.106	199	0.106	seeds	5	< 0.02, < 0.02	< 0.02	SBR-0124 V-32901-H
Canada, 2009 Boissevain, Manitoba (Golden)	1	0.106	158	0.106	seeds	5	< 0.02, < 0.02	< 0.02	SBR-0124 V-32901-F
Canada, 2009 Carberry, Manitoba (Golden)	1	0.105	195	0.105	seeds	6	0.03, 0.01	0.02	SBR-0124 V-32901-A
Canada, 2009 Elgin, Manitoba (Golden)	1	0.109	162	0.109	seeds	6	< 0.02, < 0.02	< 0.02	SBR-0124 V-32901-I
Canada, 2009 Hepburn, Saskatchewan (Golden)	1	0.111	184	0.111	seeds	6	< 0.02, < 0.02	< 0.02	SBR-0124 V-32901-G
Canada, 2009 Justice, Manitoba (Golden)	1	0.108	200	0.108	seeds	6	0.03, 0.02	0.03	SBR-0124 V-32901-B
Canada, 2009 Waldheim, Saskatchewan (Admiral)	1	0.107	201	0.107	seeds	6	< 0.02, < 0.02	< 0.02	SBR-0124 V-32901-D
USA, 2009 Northwood, ND (Admiral)	1	0.107 +NIS	184	0.107	seeds	5	< 0.02, < 0.02	< 0.02	SBR-0125 V-32857-A
	1	0.108 +MSO	186	0.108	seeds	5	< 0.02, < 0.02	< 0.02	
USA, 2009 Norwich, ND (Golden)	1	0.109	140	0.109	seeds	4	< 0.02, < 0.02	< 0.02	SBR-0125 V-32857-C
	1	0.216	141	0.216	seeds	4	0.02, 0.02	0.02	
USA, 2009 Parkdale, OR (Bluebird)	1	0.112	188	0.112	seeds	5	0.04, 0.02	0.03	SBR-0125 V-32857-E
USA, 2009 Payette, ID (Austrian Winter Pea)	1	0.108	186	0.108	seeds	5+2	0.05, 0.07	0.06	SBR-0125 V-32857-F
	1	0.216	188	0.216	seeds	5	0.07, 0.09	0.08	

PEAS, DRY COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2009 Sharon, ND (Admiral)	1	0.108	186	0.108	seeds	1 3 5 7	0.02, 0.03 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	0.03 < 0.02 <u>≤ 0.02</u> < 0.02	SBR-0125 V-32857-B
USA, 2009 Velva, ND, (Golden)	1	0.109 +NIS	141	0.109	seeds	4	0.02, 0.02	0.02	SBR-0125 V-32857-D
	1	0.11 +MSO	141	0.11	seeds	4	0.02, 0.02	0.02	

DAT = Interval from last application to cutting + field drying interval (in days)

NIS = Non-ionic surfactant

MSO = Methylated seed oil surfactant

Soya beans

In supervised trials on <u>soya beans</u> conducted in the USA between 1989 and 1993, single broadcast soil applications of 0.1–0.11 kg ai flumioxazin/ha (WG, FL or WP formulations) were applied using back-pack or tractor-mounted boom sprayers, either as pre-plant treatments (with or without soil incorporation) or just after sowing, before crop emergence.

Duplicate samples of seed (min 1 kg) were frozen within 24 hours and stored for up to 9 months before analysis for flumioxazin using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 mg/kg ranged from 71–112% in seeds, with a validated LOQ of 0.02 mg/kg.

In the trials conducted in 1992–93, seeds were also analysed for the 1-OH-HPA metabolite, using method RM 30M (GC-MS), with an LOQ of 0.02 mg/kg and recovery rates of 71–100% in samples spiked with 0.02 mg/kg.

Table 61 Residues in soya bean seeds from supervised trials in the USA involving one broadcast pre-plant or pre-emergence soil application of flumioxazin

SOYA BEAN COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	TYPE	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1989 Dallas Center, IA (Asgrow 1937)	PE	0.101	94	0.101	seed	139	< 0.02, < 0.02	< 0.02	SBR-0003 T-7262
USA, 1989 Dallas Center, IA (Wells II)	PE	0.101	187	0.101	seed	132	< 0.02, < 0.02	< 0.02	SBR-0003 T-7370 no cultivation
USA, 1989 Geneseo, IL (Pioneer 9271)	PE	0.101	187	0.101	seed	133	< 0.02, < 0.02	< 0.02	SBR-0003 T-7374
USA, 1989 Greenville, MS (Forrest)	PE	0.101	187	0.101	seed	141	< 0.02, < 0.02	< 0.02	SBR-0003 T-7373
USA, 1989 Hollandale, MN (NK523-12)	PE	0.101	187	0.101	seed	123	< 0.02, < 0.02	< 0.02	SBR-0003 T-7260
USA, 1989 Lanoke, AR (Asgrow 5980)	PE	0.101	94	0.101	seed	131	< 0.02, < 0.02	< 0.02	SBR-0003 T-7263
USA, 1989 Leonard, MO (Williams 82)	PE	0.101	374	0.101	seed	128	< 0.02, < 0.02	< 0.02	SBR-0003 T-7368
USA, 1989 Metcalfe, MS (Forrest)	PE	0.101	187	0.101	seed	128	< 0.02, < 0.02	< 0.02	SBR-0003 T-7375
USA, 1989 New Holland, OH (Pioneer 9361)	PE	0.101	365	0.101	seed	128	< 0.02, < 0.02	< 0.02	SBR-0003 T-7369
USA, 1989 Noblesville IN (Pioneer 9361)	PE	0.101	206	0.101	seed	138	< 0.02, < 0.02	< 0.02	SBR-0003 T-7261
USA, 1989 Rosa, LA (Forrest)	PE	0.101	212	0.101	seed	149	< 0.02, < 0.02	< 0.02	SBR-0003 T-7372
USA, 1989 York, NE (Hack)	PE	0.101	187	0.101	seed	138	< 0.02, < 0.02	< 0.02	SBR-0003 T-7371
USA, 1990 Clarence, MO (Williams 82)	PE	0.101	187	0.101	seed	126	< 0.02, < 0.02	< 0.02	SBR-0003 T-7512
USA, 1990 Cloverport, TN (FFR 562)	PE	0.101	187	0.101	seed	121	< 0.02, < 0.02	< 0.02	SBR-0003 T-7501
USA, 1990 Dallas Center, IA (Asgrow 2187)	PE	0.101	187	0.101	seed	136	< 0.02, < 0.02	< 0.02	SBR-0003 T-7507
USA, 1990 Dallas Center, IA (Asgrow 2187)	PP	0.101	187	0.101	seed	131	< 0.02, < 0.02	< 0.02	SBR-0003 T-7509
USA, 1990 Elwood, IL (Pioneer 9202)	PP	0.101	196	0.101	seed	138	< 0.02, < 0.02	< 0.02	SBR-0003 T-7508

SOYA BEAN COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	TYPE	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1990 Geneseo, IL (Pioneer 9272)	PE	0.101	187	0.101	seed	111	< 0.02, < 0.02	< 0.02	SBR-0003 T-7502
USA, 1990 Greenville, MS (Forrest)	PE	0.101	187	0.101	seed	130	< 0.02, < 0.02	< 0.02	SBR-0003 T-7506
USA, 1990 Hollandale, MN (Agri Pro 1776)	PE	0.101	187	0.101	seed	133	< 0.02, < 0.02	< 0.02	SBR-0003 T-7511
USA, 1990 Hollendale, MN (Agri Pro1776)	PE	0.101	187	0.101	seed	133	< 0.02, < 0.02	< 0.02	SBR-0003 T-7500 no cultivation
USA, 1990 New Holland, OH (Pioneer 9391)	PE	0.101	243	0.101	seed	128	< 0.02, < 0.02	< 0.02	SBR-0003 T-7510
USA, 1990 Noblesville, IN (Pioneer 9361)	PE	0.101	253	0.101	seed	125	< 0.02, < 0.02	< 0.02	SBR-0003 T-7503
USA, 1990 Proctor, AR (DPL 105)	PE	0.101	187	0.101	seed	140	< 0.02, < 0.02	< 0.02	SBR-0003 T-7513
USA, 1992 Goldsboro, NC (Ransom)	PP	0.105	187	0.105	seed	154	< 0.02, < 0.02	< 0.02	SBR-0021 V-1039-A
USA, 1992 Greenville, MS (Pioneer 9641)	PE	0.105	187	0.105	seed	127	<< 0.02, < 0.02	< 0.02	SBR-0021 V-1039-H
USA, 1992 Leonard, MO (Pioneer 9443)	PP	0.102	187	0.102	seed	126	< 0.02, < 0.02	< 0.02	SBR-0021 V-1039-M
USA, 1992 Little Rock, AR (Hutcheson)	PP	0.105	187	0.105	seed	146	< 0.02, < 0.02	< 0.02	SBR-0021 V-1039-C
USA, 1992 New Holland, OH (GL 2910)	PE	0.105	150	0.105	seed	132	< 0.02, < 0.02	< 0.02	SBR-0021 V-1039-G
USA, 1992 Noblesville, IN (Pioneer 9361)	PP	0.105	234	0.105	seed	131	< 0.02, < 0.02	< 0.02	SBR-0021 V-1039-D
USA, 1992 Seymour, IL (Asgrow 2543)	PP	0.105	187	0.105	seed	130	< 0.02, < 0.02	< 0.02	SBR-0021 V-1039-B
USA, 1992 Seymour, IL (Asgrow 2543)	PE	0.105	187	0.105	seed	126	< 0.02, < 0.02	< 0.02	SBR-0021 V-1039-J
	PE	0.526	187	0.526	seed	126	< 0.02, < 0.02	< 0.02	
USA, 1992 Waukee, IA (Asgrow 2543)	PP	0.105	187	0.105	seed	129	< 0.02, < 0.02	< 0.02	SBR-0021 V-1039-F
USA, 1993 Jamesville, NC (Hutcheson)	PP	0.108	253	0.108	seed	160	< 0.02, < 0.02	< 0.02	SBR-0021 V-10719-A

SOYA BEAN COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	TYPE	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1993 Leonard, MO (Linford)	PP	0.107	271	0.107	seed	123	< 0.02, < 0.02	< 0.02	SBR-0021 V-10719-E
USA, 1993 Noblesville, IN (Pioneer 9361)	PP	0.11	206	0.11	seed	138	< 0.02, < 0.02	< 0.02	SBR-0021 V-10719-D
USA, 1993 Seymour, IL (Asgrow 2506)	PE	0.536	187	0.536	seed	112	< 0.02, < 0.02	< 0.02	SBR-0021 V-10719-K
USA, 1993 Theilman, MN (Pioneer 9061)	PP	0.107	187	0.107	seed	160	< 0.02, < 0.02	< 0.02	SBR-0021 V-10719-B
USA, 1993 Webster City, IA (L-1700)	PP	0.108	206	0.108	seed	112	< 0.02, < 0.02	< 0.02	SBR-0021 V-10719-F
USA, 1993 York, NE (Hack)	PP	0.107	187	0.107	seed	126	< 0.02, < 0.02	< 0.02	SBR-0021 V-10719-C

The 1992–1993 supervised trials also analysed for residues of the metabolite, 1-OH HPA in seeds. Residues in all samples were below the LOQ of 0.02 mg/kg (18 trials).

PE = pre-emergence application (within 5 days after sowing)

PP = pre-plant application

Root and tuber vegetables

Potato

In supervised trials on potatoes (14) conducted in the USA during 2001, single broadcast soil applications of 0.13–0.15 kg ai flumioxazin/ha (WG formulations) were applied using back-pack, wheeled or tractor-mounted sprayers with 2–12 nozzle booms after the last hilling operation, before potato emergence. In several trials, transitory phytotoxicity and stunting was observed.

Duplicate samples of at least 1.8 kg potatoes were wiped, brushed or rinsed to remove adhering soil, frozen within 2.5 hours and analysed for flumioxazin within 9 months of harvest using method RM 30A-2 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 77–118% and the validated LOQ was 0.02 mg/kg.

Table 62 Residues in potatoes from supervised trials in the USA involving one broadcast pre-emergent soil application of flumioxazin (WG formulations)

POTATO COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2001 Aberdeen, ID (Russet Burbank)	1	0.138	279	0.138	Tuber	118	< 0.02, < 0.02	< 0.02	SBR-0091 ID01
USA, 2001 Clinton, NC (Atlantic)	1	0.138	184	0.138	Tuber	62	< 0.02, < 0.02	< 0.02	SBR-0091 NC05
USA, 2001 E. Corinth, ME (Atlantic)	1	0.132	177	0.132	Tuber	105	< 0.02, < 0.02	< 0.02	SBR-0091 ME01

POTATO COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2001 Fort Collins, CO (Russet Norkotah)	1	0.139	183	0.139	Tuber	96	< 0.02, < 0.02	< 0.02	SBR-0091 CO02
USA, 2001 Fort Collins, CO (Russet Norkotah)	1	0.139	182	0.139	Tuber	91	< 0.02, < 0.02	< 0.02	SBR-0091 CO03 not independent
USA, 2001 Freemont, OH (Yukon Gold)	1	0.148	258	0.148	Tuber	92	< 0.02, < 0.02	< 0.02	SBR-0091 OH*03
USA, 2001 Freeville, NY (Atlantic)	1	0.127	254	0.127	Tuber	111	< 0.02, < 0.02	< 0.02	SBR-0091 NY03
USA, 2001 Gainesville, FL (Red La Soda)	1	0.123	247	0.123	Tuber	67	< 0.02, < 0.02	< 0.02	SBR-0091 FL09
USA, 2001 Holtville, CA (California White)	1	0.141	309	0.141	Tuber	104	< 0.02, < 0.02	< 0.02	SBR-0091 CA09
USA, 2001 Prosper, ND (Red La Soda)	1	0.14	156	0.14	Tuber	101	< 0.02, < 0.02	< 0.02	SBR-0091 ND04
USA, 2001 Prosper, ND (Russet Burbank)	1	0.147	164	0.147	Tuber	101	< 0.02, < 0.02	< 0.02	SBR-0091 ND05 not independent
USA, 2001 Prosser, WA (Russet Burbank)	1	0.147	252	0.147	Tuber	126	< 0.02, < 0.02	< 0.02	SBR-0091 WA05
USA, 2001 Prosser, WA (Russet Burbank)	1	0.141	268	0.141	Tuber	126	< 0.02, < 0.02	< 0.02	SBR-0091 WA06 not independent
USA, 2001 Prosser, WA (Russet Burbank)	1	0.141	149	0.141	Tuber	107	< 0.02, < 0.02	< 0.02	SBR-0091 WA*07

Stem and stalk vegetables

Supervised trials on stem and stalk vegetables (asparagus, Globe artichoke and celery) were conducted in North America between 2003 and 2007.

Artichoke, Globe

In three supervised trials on Globe artichokes, single broadcast soil applications of 0.21 kg ai flumioxazin/ha (WG formulations) were applied 1–4 days before transplanting using back-pack sprayers with hand-held 5-nozzle minibooms.

Duplicate samples of flower heads (12 units, min 2.7 kg) were frozen within 1 hour and analysed for flumioxazin within 7.5 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 70–115% and the validated LOQ was 0.02 mg/kg.

Table 63 Residues in Globe artichokes from supervised trials in the USA involving one broadcast pre-plant soil application of flumioxazin (WG formulations)

ARTICHOKE,	APPLICATION	MATRIX	DAT	RESIDUES (MG/KG)	REFERENCE &

GLOBE COUNTRY, YEAR LOCATION (VARIETY)	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	COMMENTS
GAP:USA		0.21	94–280	0.21			Before planting or cut-back		
USA, 2007 Castroville, CA (F1 1855)	1	0.214	280	0.214	Head	147	< 0.02, < 0.02	< 0.02	SBR-0128 CA37
USA, 2007 Watsonville, CA (F1 41)	1	0.21	367	0.21	Head	134	< 0.02, < 0.02	< 0.02	SBR-0128 CA38
USA, 2007 Castroville, CA (F1 1855)	1	0.214	468	0.214	Head	126	< 0.02, < 0.02	< 0.02	SBR-0128 CA39

Asparagus

In eight supervised trials on asparagus, single broadcast soil applications of 0.21–0.22 or 0.43–0.45 kg flumioxazin/ha (WG formulations) were applied using back-pack, ATV or tractor-mounted 3–6 nozzle booms about 2 weeks before spear emergence. Phytotoxicity was observed in several trials.

Duplicate samples of at least 1.3 kg spears were brushed (if necessary) to remove adhering soil, frozen within 4.5 hours and analysed for flumioxazin within 3.5 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 90–114% and the validated LOQ was 0.02 mg/kg.

Table 64 Residues in asparagus from supervised trials in the USA involving one broadcast soil application of flumioxazin (WG formulations)

ASPARAGUS COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
GAP:USA		0.21	140–280	0.21		14	Pre-emergent		
USA, 2004 Porterville, CA (UC157)	1	0.22	231	0.22	spears	15	< 0.02, < 0.02	< 0.02	SBR-0116 CA74
		0.43	229	0.43	spears	15	< 0.02, < 0.02	< 0.02	
USA, 2004 Stockton, CA (UC157)	1	0.22	198	0.22	spears	14	< 0.02, < 0.02	< 0.02	SBR-0116 CA75 min 0.5kg sample
		0.43	198	0.43	spears	14	< 0.02, < 0.02	< 0.02	
USA, 2004 Stockton, CA (UC157)	1	0.22	295	0.22	spears	14	< 0.02, < 0.02	< 0.02	SBR-0116 CA76
		0.44	924	0.44	spears	14	< 0.02, < 0.02	< 0.02	
USA, 2003 Holt, MI (Jersey Knight)	1	0.22	192	0.22	spears	14	< 0.02, < 0.02	< 0.02	SBR-0116 MI23
		0.43	191	0.43	spears	14	< 0.02, < 0.02	< 0.02	
USA, 2003 East Lansing, MI (Jersey Giant)	1	0.22	193	0.22	spears	14	< 0.02, < 0.02	< 0.02	SBR-0116 MI24
		0.44	196	0.44	spears	14	< 0.02, < 0.02	< 0.02	
USA, 2003 Bridgeton, NJ (New Jersey hybrids)	1	0.22	217	0.22	spears	15	< 0.02, < 0.02	< 0.02	SBR-0116 NJ17
		0.45	227	0.45	spears	15	< 0.02, < 0.02	< 0.02	
USA, 2003 Prosser, WA (Jersey Giant)	1	0.21	343	0.21	spears	15	< 0.02, < 0.02	< 0.02	SBR-0116 WA09
		0.43	343	0.43	spears	15	< 0.02, < 0.02	< 0.02	

ASPARAGUS COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2003 Moxee, WA (Mary Washington)	1	0.22	261	0.22	spears	20	< 0.02, < 0.02	< 0.02	SBR-0116 WA*10
		0.44	260	0.44	spears	20	< 0.02, < 0.02	< 0.02	

Celery

In eight supervised trials on celery, single broadcast soil applications of 0.1–0.11 or 0.2–0.22 kg ai flumioxazin/ha (WG formulations) were applied 0–2 days before transplanting using back-pack plot sprayers with 3–4 nozzle minibooms or tractor-mounted 9-nozzle boom sprayers. Phytotoxicity was reported in several of the high-rate plots.

Duplicate samples of 12 untrimmed bunches (12 units, min 1.8 kg) were brushed or rinsed if necessary to remove adhering soil, frozen within 5 hours and analysed for flumioxazin within 9 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 90–120% (except for one recovery at 150%) and the validated LOQ was 0.02 mg/kg.

Table 65 Residues in celery from supervised trials in the USA involving one broadcast pre-plant soil application of flumioxazin (WG formulations)

CELERY COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
GAP:USA		0.105	140–280	0.105			Before or 3–7 days after transplanting		
USA, 2004 Citra, FL (M-9)	1	0.105	278	0.105	Stalk	104	< 0.02, < 0.02	< 0.02	SBR-0122 FL10
	1	0.212	282	0.212	Stalk	104	< 0.02, < 0.02	< 0.02	
USA, 2004 Citra, FL (M-9)	1	0.107	285	0.107	Stalk	108	< 0.02, < 0.02	< 0.02	SBR-0122 FL11
	1	0.216	287	0.216	Stalk	108	< 0.02, < 0.02	< 0.02	
USA, 2004 Laingsburg, MI (Dutchess)	1	0.107	191	0.107	Stalk	73	< 0.02, < 0.02	< 0.02	SBR-0122 MI02 subsampled in the field
	1	0.221	196	0.221	Stalk	73	< 0.02, < 0.02	< 0.02	
USA, 2004 Salinas, CA (Dutchess)	1	0.112	367	0.112	Stalk	98	< 0.02, < 0.02	< 0.02	SBR-0122 CA*18 subsampled in the field
	1	0.214	358	0.214	Stalk	98	< 0.02, < 0.02	< 0.02	
USA, 2004 Paso Robles, CA (Conquistado)	1	0.107	283	0.107	Stalk	95	< 0.02, < 0.02	< 0.02	SBR-0122 CA19
	1	0.204	272	0.204	Stalk	95	< 0.02, < 0.02	< 0.02	
USA, 2004 Camarillo, CA (BSM2)	1	0.107	283	0.107	Stalk	127	< 0.02, < 0.02	< 0.02	SBR-0122 CA20
	1	0.211	282	0.211	Stalk	127	< 0.02, < 0.02	< 0.02	
USA, 2004 Irvine, CA (Conquistador 1703)	1	0.105	233	0.105	Stalk	112	< 0.02, < 0.02	< 0.02	SBR-0122 CA21
	1	0.21	235	0.21	Stalk	112	< 0.02, < 0.02	< 0.02	
USA, 2004 Salinas, CA (Challenger)	1	0.104	318	0.104	Stalk	90	< 0.02, < 0.02	< 0.02	SBR-0122 CA*22 subsampled in the field
	1	0.21	322	0.21	Stalk	90	< 0.02, < 0.02	< 0.02	

Cereal grains

Supervised trials on cereal grains (maize and wheat) were conducted in North America between 2005 and 2010.

Maize

In twenty-one supervised trials on maize, single broadcast soil applications of 0.1–0.11 or 0.2–0.22 kg ai flumioxazin/ha (WG formulations) with added surfactant were applied 6–14 days before sowing, using back-pack plot sprayers, wheeled or tractor-mounted boom sprayers (3–9 nozzles).

Duplicate samples of kernels (min 1 kg) were taken at maturity, frozen within 2 hours and analysed for flumioxazin within 14 months using method RM 30A-3 (GC-MS) in the 2005 trials and method NCL 293 (HPLC-MS/MS) in the 2006 trials. Recoveries from control kernel samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 85–122% in the two methods and the validated LOQ was 0.02 mg/kg.

Table 66 Residues in maize from supervised trials in North America involving one broadcast pre-plant soil application of flumioxazin (WG formulations)

MAIZE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
GAP:USA		0.105	140–280	0.105			14–30 days before sowing		
USA, 2005 New Holland, OH (Syngenta N73-F7)	1	0.107	191	0.107	grain	148	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-A
	1	0.211	188	0.211	grain	148	< 0.02, < 0.02	< 0.02	
USA, 2005 Carlyle, IL (FS 6455)	1	0.107	190	0.107	grain	171	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-B
	1	0.212	187	0.212	grain	171	< 0.02, < 0.02	< 0.02	
USA, 2005 Clarence, MO (Pioneer 35P12)	1	0.107	191	0.107	grain	154	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-C
USA, 2005 Greenville, MS (69-71 757 HXJINX)	1	0.104	185	0.104	grain	135	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-D
USA, 2006 North Rose, NY (Dairyland Stealth 8711)	1	0.108	191	0.108	grain	131	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-E
USA, 2006 Elko, SC (Pioneer 31R87)	1	0.105	180	0.105	grain	158	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-F
Canada, 2006 City of Hamilton, Ontario (Pioneer 38B84)	1	0.107	187	0.107	grain	166	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-G
	1	0.211	184	0.211	grain	166	< 0.02, < 0.02	< 0.02	
USA, 2006 Conklin, MI (N45-M2 Field Corn)	1	0.106	189	0.106	grain	138	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-H
USA, 2006 Carlyle, IL (DKC-65-16)	1	0.108	185	0.108	grain	168	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-I
USA, 2006 Bellmore, IN (Wyffels 5531)	1	0.104	185	0.104	grain	136	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-J
USA, 2006 York, NE (NK N70-F1)	1	0.106	184	0.106	grain	155	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-K
USA, 2006 Richland, IA (Pioneer 33P65)	1	0.105	189	0.105	grain	151	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-L
USA, 2006 Geneva, MN (Pioneer 38H66)	1	0.106	180	0.106	grain	156	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-M
USA, 2006 Fairmount, ND (Dekalb 35-02)	1	0.106	188	0.106	grain	145	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-N

MAIZE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2006 Campbell, MN (Pioneer 39H83)	1	0.106	188	0.106	grain	155	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-O
USA, 2006 Hudson, KS (Midwest Seed Genetics 8127RB)	1	0.106	188	0.106	grain	134	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-P
Canada, 2006 Portage la Prairie, Manitoba (Roundup Ready-Monsanto)	1	0.102	181	0.102	grain	154	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-Q
USA, 2006 Arkansaw, WI (Pioneer 38B85)	1	0.106	188	0.106	grain	137	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-R
Canada, 2006 St. Pie, Quebec (NK 3030 BT)	1	0.101	176	0.101	grain	156	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-S
USA, 2006 Dill City, OK (DKC48-53)	1	0.107	193	0.107	grain	130	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-T
USA, 2006 Clarence, MO (Pioneer 34B20)	1	0.107	187	0.107	grain	165	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-U

Wheat

In twenty supervised trials on wheat, single foliar broadcast sprays of 0.07–0.075 kg ai flumioxazin/ha (WG formulations) with added adjuvants were applied as pre-harvest desiccants and harvest aids using tractor-mounted or back-pack sprayers with 4–8 nozzle booms.

Duplicate samples were collected using small plot combines or cut and harvested using a stationary combine to obtain minimum samples of 1 kg dry seeds. Samples were frozen within 5 hours and analysed for flumioxazin within 17 months of harvest using method RM 30A-3 (GC-MS). Concurrent recoveries from control grain samples fortified with flumioxazin at levels of 0.02–0.5 mg/kg ranged from 70–122% and the validated LOQ was 0.02 mg/kg.

Table 67 Residues in wheat grain from supervised trials in the USA involving one pre-harvest foliar application of flumioxazin (WG formulations)

WHEAT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2009 Lexington, GA (USG 3592)	1	0.071 + NIS	193	0.071	grain	10	0.03, 0.04	0.04	SBR-0092 V-33037-A
	1	0.071 + MSO	192	0.071	grain	10	0.04, 0.06	0.05	
USA, 2009 Leland, MS (Gore)	1	0.071 + MSO	185	0.071	grain	3 7 10 13	0.04, 0.08 0.05, 0.05 0.11, 0.11 0.07, 0.11	0.06 0.05 0.11 0.09	SBR-0092 V-33037-B
USA, 2009	1	0.072 + MSO	199	0.072	grain	10	0.07, 0.08	0.08	SBR-0092

WHEAT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
Carlyle, IL (Branson)	1	0.145 + MSO	200	0.145	grain	10	0.22, 0.24	0.23	V-33037-C
USA, 2009 York, NE (Traverse Hard red Spring)	1	0.071 + NIS	184	0.071	grain	10	0.04, 0.05	0.05	SBR-0092 V-33037-D
	1	0.071 + MSO	186	0.071	grain	10	0.03, 0.04	0.04	
USA, 2009 Rockville, IN (Becks 164)	1	0.072 + NIS	148	0.072	grain	11	0.08, 0.13	0.11	SBR-0092 V-33037-E
	1	0.072 + MSO	148	0.072	grain	11	0.07, 0.09	0.08	
USA, 2009 Clarence, MO (Ernie)	1	0.074 + NIS	193	0.074	grain	10	0.11, 0.15	0.13	SBR-0092 V-33037-F
	1	0.07 + MSO	183	0.07	grain	10	0.09, 0.13	0.11	
USA, 2009 Bagley, IA (Briggs hrS)	1	0.071 + NIS	148	0.071	grain	10	0.18, 0.28	0.23	SBR-0092 V-33037-G
	1	0.072 + MSO	150	0.072	grain	10	0.13, 0.19	0.16	
USA, 2009 Ulvade, TX (Fannin)	1	0.07 + NIS	138	0.07	grain	9	0.11, 0.12	0.12	SBR-0092 V-33037-H
	1	0.072 + MSO	142	0.072	grain	9	0.08, 0.1	0.09	
USA, 2009 Grand Island, NE (Traverse Hard Red Spring)	1	0.072 + NIS	189	0.072	grain	10	0.05, 0.07	0.06	SBR-0092 V-33037-I not independent
	1	0.071+MSO	186	0.071	grain	10	0.06, 0.06	0.06	
USA, 2009 Velva, ND (Faller)	1	0.072 + MSO	141	0.072	grain	10	0.06, 0.08	0.07	SBR-0092 V-33037-J
USA, 2009 Grand Island, NE (Kelby Hard Red Spring)	1	0.071 + NIS	185	0.071	grain	10	0.08, 0.09	0.09	SBR-0092 V-33037-K
USA, 2009 Norwich, ND (Faller)	1	0.072 + MSO	172	0.072	grain	10	0.09, 0.11	0.1	SBR-0092 V-33037-L
	1	0.146 + MSO	143	0.146	grain	10	0.13, 0.14	0.14	
USA, 2009 Malta, MT (McNeal)	1	0.069 + NIS	181	0.069	grain	10	0.3, 0.31	0.31	SBR-0092 V-33037-AM
USA, 2009 Levelland, TX (TAM 105)	1	0.072 + NIS	189	0.072	grain	9	0.1, 0.16	0.13	SBR-0092 V-33037-N
USA, 2009 Wellington, TX (TAM 111)	1	0.072 + MSO	165	0.072	grain	9	0.03, 0.06	0.05	SBR-0092 V-33037-O
	1	0.142 + MSO	163	0.142	grain	9	0.03, 0.05	0.04	
USA, 2009 Larned, KS (Jagger)	1	0.074 + NIS	212	0.074	grain	10	0.07, 0.13	0.10	SBR-0092 V-33037-P
USA, 2009 Hinton, OK (Jagger)	1	0.07 + MSO	164	0.07	grain	4 7 10 13	0.07, 0.09 0.16, 0.16 0.06, 0.08 0.15, 0.2	0.08 0.16 0.07 <u>0.18</u>	SBR-0092 V-33037-Q
USA, 2009 Cordell, OK (Fuller)	1	0.072 + MSO	172	0.072	grain	11	0.07, 0.12	0.1	SBR-0092 V-33037-R
USA, 2009 Jerome, ID (AC Andrew)	1	0.071 + NIS	181	0.071	grain	10	0.04, 0.05	0.05	SBR-0092 V-33037-S
USA, 2009	1	0.071 + MSO	165	0.071	grain	10	0.05, 0.06	0.06	SBR-0092

WHEAT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
Hinton, OK (Deliver)	1	0.344 + MSO	152	0.344	grain	10	0.37, 0.35	0.36	V-33037-T not independent

NIS = Non-ionic surfactant

MSO = Methylated seed oil surfactant

Grasses for sugar production

Sugar cane

In supervised trials on sugar cane (nine) conducted in the USA during 1998, single broadcast applications of 0.4–0.42 kg ai flumioxazin/ha (WG formulations) with added crop oil were applied over the top of 2–2.5 m high canes using back-pack sprayers with elevated 6-nozzle booms or extended single-nozzle hand lances.

Duplicate samples of at least 12 canes with leaves attached (min 5 kg) were frozen within 10 hours and analysed for flumioxazin within 5 months of harvest using method RM 30A-1 (GC-MS). Samples were also analysed within 7 months of harvest for the 1-OH-HPA metabolite using method RM-30C (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.01–0.5 mg/kg ranged from 67–113% and in samples fortified with 0.02–0.2 mg/kg 1-OH-HPA, recoveries were 70–114%. The validated LOQs for both compounds were both 0.02 mg/kg.

Table 68 Residues in sugar cane from supervised trials in the USA involving one broadcast foliar application of flumioxazin (WG formulation)

SUGAR CANE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1998 Clewiston, FL (CP-70-1133)	1	0.424	160	0.424	cane	89	< 0.02, < 0.02	< 0.02	SBR-0022 V-11945-A
USA, 1998 Clewiston, FL (CP-72-2086)	1	0.416	157	0.416	cane	89	0.02, 0.03	0.03	SBR-0022 V-11945-B
USA, 1998 Canal Point, FL (CP80-1827)	1	0.409	154	0.409	cane	89	0.04, < 0.02	0.03	SBR-0022 V-11945-C
USA, 1998 Clewiston, FL (CL77-79786)	1	0.421	159	0.421	cane	89	< 0.02, < 0.02	< 0.02	SBR-0022 V-11945-D
USA, 1998 Washington, LA (La 384)	1	0.423	143	0.423	cane	91	0.07, 0.11	0.09	SBR-0022 V-11945-E
USA, 1998 Raymondville, TX (1210)	1	0.415	139	0.415	cane	90	0.02, 0.09	0.06	SBR-0022 V-11945-F
USA, 1998 LeBeau, LA (La 384)	1	0.408	143	0.408	cane	90	0.07, 0.07	0.07	SBR-0022 V-11945-G

SUGAR CANE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1998 Spreckelsville, HI (78-4153)	1	0.421	187	0.421	cane	90	< 0.02, < 0.02	< 0.02	SBR-0022 V-11945-H
USA, 1998 Washington, LA (CP-845)	1	0.417	146	0.417	cane	90	0.03, 0.06	0.05	SBR-0022 V-11945-J
	1	1.254	147	1.254	cane	90	0.33, 0.14	0.23	

Residues of 1-OH-HPA < 0.02 mg/kg in all samples

Tree nuts

Supervised trials on tree nuts (almonds and pecans) were conducted in the US during 1999 and 2003, respectively.

Almonds

In supervised trials on almonds (five), two inter-row/berm broadcast soil treatments of 0.42 kg ai flumioxazin/ha (WG formulations) with added crop oil were applied using tractor-mounted 4–8 nozzle boom sprayers. Treatments were applied about 60 days apart, with the last application about 60 days before harvest.

Duplicate samples of mature nuts (min 1 kg) shaken from the trees, shelled in the field, frozen within 3 hours and analysed for flumioxazin within 6 months of harvest using method RM 30A-1 (GC-MS). Samples of almond hulls were also analysed within 8.5 months of harvest for the 1-OH-HPA metabolite using method RM-30M (GC-MS).

Recoveries from control samples fortified with flumioxazin at levels of 0.01 and 0.05 mg/kg ranged from 89–114% in nutmeat and 71–96% in hulls. In hull samples fortified with 0.1 or 0.5 mg/kg 1-OH-HPA, recoveries were 81–98%.The validated LOQs were 0.01 mg/kg (flumioxazin) and 0.1 mg/kg for the 1-OH-HPA metabolite.

Table 69 Residues in almonds (nutmeat and hulls) from supervised trials in the USA involving two broadcast soil application of flumioxazin (WG formulation)

ALMOND COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
GAP:USA		0.42	140–280	0.84		60	Directed inter-row band applications, min 60 day RTI		
USA, 1999 Chico, CA (Carmel)	2	0.419 0.425	168 168	0.844	nutmeat hulls	60	< 0.01, < 0.01 0.01, 0.01	< 0.01 0.01	SBR-0024 V-20116-A
	2	0.838 0.847	168 168	1.685	nutmeat hulls	60	< 0.01, < 0.01 0.03, 0.03	< 0.01 0.03	
USA, 1999 Hughson, CA (Carmel)	2	0.425 0.424	234 234	0.849	nutmeat hulls	60	< 0.01, < 0.01 0.49, 0.62	< 0.01 0.55	SBR-0024 V-20116-B
USA, 1999 Kerman, CA (Carmel)	2	0.417 0.419	187 187	0.836	nutmeat hulls	60	< 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01	SBR-0024 V-20116-C

ALMOND COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1999 Madera, CA (Non-pareil)	2	0.424 0.418	187 187	0.842	nutmeat hulls	60	< 0.01, < 0.01 0.04, 0.04	< 0.01 0.04	SBR-0024 V-20116-D
USA, 1999 Terra Bella CA (Carmel)	2	0.421 0.419	187 224	0.84	nutmeat hulls	60	< 0.01, < 0.01 0.06, 0.07	< 0.01 0.06	SBR-0024 V-20116-E

Residues of 1-OH-HPA all < 0.05 mg/kg in almond hulls

Pecans

In supervised trials on pecans (five), two inter-row/berm broadcast soil treatments of 0.42–0.43 kg ai flumioxazin/ha (WG formulations) were applied using knapsack or wheeled sprayers with 3–4 nozzle booms. Treatments were applied about 60 days apart, with the last application about 60 days before harvest.

Duplicate samples of mature nuts (min 1.2 kg) were shaken from the trees, shelled within 2 days of harvest, with nutmeat samples frozen within 6.25 hours of shelling and analysed for flumioxazin within 3.3 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 77–99% and the validated LOQ was 0.02 mg/kg.

Table 70 Residues in pecans from supervised trials in the USA involving one broadcast soil application of flumioxazin (WG formulation)

PECAN COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2003 Roseboro, NC (Pawnee)	2	0.419 0.426	290 299	0.845	nutmeat	59	< 0.02, < 0.02	< 0.02	SBR-0062 NC19
USA, 2003 Roseboro, NC (Kiowah)	2	0.422 0.427	299 309	0.849	nutmeat	61	< 0.02, < 0.02	< 0.02	SBR-0062 NC20
USA, 2003 Neches, TX (Desirable)	2	0.425 0.42	206 206	0.845	nutmeat	42	< 0.02, < 0.02	< 0.02	SBR-0062 TX31
USA, 2003 Shreveport, LA (Cape Fear)	2	0.421 0.42	206 206	0.841	nutmeat	61	< 0.02, < 0.02	< 0.02	SBR-0062 TX32
USA, 2003 Mesilla, NM (Western Shleigh)	2	0.419 0.433	196 215	0.852	nutmeat	61	< 0.02, < 0.02	< 0.02	SBR-0062 NM10

Oilseeds

Supervised trials on oilseeds (oilseed rape, cotton seed, sunflower seed and peanuts) were conducted in the USA between 1992 and 2009.

Cotton seed

In supervised trials on cotton seed (13), two foliar broadcast sprays of 0.1–0.11 kg ai flumioxazin/ha (WG formulations) with added crop oil were applied using tractor-mounted boom sprayers. The first applications were made about 90 days before harvest using shielded nozzles to minimise spray contact with the plants and the second applications were made about 60 days before harvest as directed inter-row sprays at layby, with spray contacting only the lower 5–10 cm cotton stems.

Duplicate samples of cotton seed, either ginned in the field (min 1 kg) or unginned (min 20 kg), were frozen within 4 hours (undelinted seed) or within 24 hours (unginned cotton). The cotton seed samples were stored frozen for up to 30 days before being ginned to separate the undelinted seed and gin trash and refrozen. All samples were analysed for flumioxazin within 3 months using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.01 and 0.05 mg/kg ranged from 76–106% (cottonseed) and 70–102% in gin trash. The validated LOQs were 0.01 mg/kg for cotton seed and gin trash.

Samples of gin trash were also analysed within 8 months of harvest for the 1-OH-HPA metabolite using method RM-30M (GC-MS) with recoveries from control samples fortified with 1-OH-HPA at levels of 0.1 and 0.5 mg/kg ranging from 81–121% and the validated LOQ was 0.1 mg/kg.

Table 71 Residues in cotton seed and gin trash from supervised trials in the USA involving two inter-row soil applications of flumioxazin (WG formulation)

COTTONSEED COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1999 Brookshire, TX (DPL 50B)	2	0.107 0.109	218 219	0.216	seed	60	< 0.01, < 0.01	< 0.01	SBR-0026 V-20124-E
USA, 1999 Greenville, MS (ST474)	2	0.105 0.106	186 190	0.211	seed gin trash	59 59	< 0.01, < 0.01 0.19, 0.3	< 0.01 0.25	SBR-0026 V-20124-L
USA, 1999 Greenville, MS (Stoneville 474)	2	0.107 0.106	148 143	0.213	seed gin trash	61 61	< 0.01, < 0.01 0.03, 0.03	< 0.01 0.03	SBR-0026 V-20124-C not independent
USA, 1999 Jamesville, NC (Stoneville 474)	2	0.117 0.107	236 258	0.224	seed gin trash	62 62	< 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01	SBR-0026 V-20124-A
USA, 1999 Kerman, CA (Maxxa)	2	0.109 0.112	193 198	0.221	seed	62	< 0.01, < 0.01	< 0.01	SBR-0026 V-20124-K
USA, 1999 Levelland, TX (PM 2200 RR)	2	0.106 0.106	187 187	0.212	seed gin trash	60	< 0.01, < 0.01 0.18, 0.13	< 0.01 0.16	SBR-0026 V-20124-H
USA, 1999 Littlefield, TX (DP 2379)	2	0.107 0.107	188 188	0.214	seed gin trash	61 61	< 0.01, 0.01 0.48, 0.48	0.01 0.48	SBR-0026 V-20124-F
USA, 1999 Madera, CA (Maxxa)	2	0.107 0.104	216 210	0.211	seed	60	< 0.01, 0.01	0.01	SBR-0026 V-20124-J
USA, 1999 Maricopa, AZ (Delta Pine 50B)	2	0.107 0.106	188 187	0.213	seed	60	< 0.01, < 0.01	< 0.01	SBR-0026 V-20124-I

COTTONSEED COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1999 Newport, AR (Paymaster 1220RR)	2	0.107 0.107	143 140	0.214	seed	60	< 0.01, < 0.01	< 0.01	SBR-0026 V-20124-B
USA, 1999 Ulvade, TX (PM 2326)	2	0.107 0.107	188 187	0.214	seed gin trash	59 59	< 0.01, < 0.01 0.03, 0.05	< 0.01 0.04	SBR-0026 V-20124-N
	2	0.213 0.211	190 188	0.424	seed gin trash	59 59	< 0.01, < 0.01 0.06, 0.1	< 0.01 0.08	
USA, 1999 Washington, LA (DLP Nuc.33B)	2	0.106 0.107	203 146	0.213	seed	60	< 0.01, < 0.01	< 0.01	SBR-0026 V-20124-D
USA, 1999 Wolfforth, TX (HS 26)	2	0.106 0.107	187 188	0.213	seed gin trash	62	< 0.01, < 0.01 0.24, 0.23	< 0.01 0.24	SBR-0026 V-20124-G

Residues of 1-OH-HPA all < 0.1 mg/kg in gin trash (8 trials, including one at 2× rate)

Oilseed rape

In supervised trials on oilseed rape (eight), single foliar broadcast sprays of 0.1–0.11 kg ai flumioxazin/ha (WG formulations) were applied with added adjuvant as pre-harvest desiccants and harvest aids using tractor-mounted or back-pack sprayers with 4–8 nozzle booms.

Duplicate samples of seed (min 0.5 kg) were collected using small plot combines, frozen within 4 hours and analysed for flumioxazin within 14 months of harvest using method RM 30A-3 (GC-MS). Concurrent recoveries from control samples fortified with flumioxazin at levels of 0.02–1.0 mg/kg ranged from 74–120% and the validated LOQ was 0.02 mg/kg.

Table 72 Residues in rape seed from supervised trials in the USA involving one pre-harvest foliar application of flumioxazin (WG formulation)

OILSEED RAPE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2009 Stephens, GA (Sumner)	1	0.104 + MSO	198	0.104	seed	5	0.04, 0.04	0.04	SBR-0123 V-32833-A
		0.105 + NIS	201	0.105	seed	5	0.05, 0.05	0.05	
USA, 2009 Campbell, MN (Hyola 357 RR Mag)	1	0.109 + MSO	188	0.109	seed	1 3 5 8	0.15, 0.17 0.16, 0.16 0.15, 0.17 0.04, 0.04	0.16 0.16 0.16 0.04	SBR-0123 V-32833-B
USA, 2009 Norwich, ND (Invigor 5550)	1	0.11 + MSO	141	0.11	seed	4	0.05, 0.06	0.05	SBR-0123 V-32833-C
	1	0.218 + MSO	142	0.218	seed	4	0.16, 0.16	0.16	
USA, 2009 Carrington, ND (Pioneer 45H26)	1	0.108 + MSO	186	0.108	seed	5 + 16	0.02, 0.03	0.03	SBR-0123 V-32833-D
	1	0.108 + NIS	186	0.108	seed	5 + 16	0.04, 0.04	0.04	

OILSEED RAPE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2009 Scobey, MT (Xceed 8571)	1	0.109+ NIS	186	0.109	seed	4	0.03, 0.12	0.07	SBR-0123 V-32833-E
	1	0.11 + MSO	187	0.11	seed	4	0.3, 0.21	0.25	
USA, 2009 Payette, ID (Hyola 308)	1	0.109 + MSO	188	0.109	seed	5	0.04, 0.04	0.04	SBR-0123 V-32833-F
	1	0.214 + MSO	186	0.214	seed	5	0.09, 0.1	0.1	
USA, 2009 Minidoka, ID (46A76)	1	0.113 + MSO	160	0.113	seed	5 + 6	0.05, 0.09	0.07	SBR-0123 V-32833-G
USA, 2009 Ephrata, WA (71-45 RR)	1	0.109 + MSO	187	0.109	seed	5 + 9	0.05, 0.06	0.06	SBR-0123 V-32833-H
	1	0.541 + MSO	188	0.541	seed	5 + 9	0.6, 0.66	0.63	

DAT = Interval from last application to cutting + field drying interval (in days)

In trials V-32833-D, V-32833-G and V-32833-H, vines were cut and allowed to dry for up to 16 days before seeds were collected.

Peanuts

In fifteen supervised trials on peanuts, single broadcast soil applications of 0.1–0.11 kg ai flumioxazin/ha (WG formulations) were applied either as pre-plant broadcast sprays (with shallow soil incorporation) within 7 days before sowing or as pre-emergent broadcast sprays within 5 days after sowing, using tractor-mounted boom sprayers (8–13 nozzles).

Duplicate samples of whole peanuts were collected after 3–19 days of field drying, shelled in the field and samples of nutmeat (min 2.2 kg) and hulls (min 0.22 kg) were taken for analysis. All samples were kept in frozen storage up to 210 days before analysis for flumioxazin using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02–0.1 mg/kg ranged from 80–105% (nutmeat) and 70–101% (hulls), and the validated LOQs were 0.02 mg/kg.

Table 73 Residues in peanuts (nutmeat and hulls) from supervised trials in the USA involving one broadcast pre-plant or pre-emergent soil application of flumioxazin (WG formulations)

PEANUT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1992 Grangerburg, AL (Florunner)	1	0.109	187	0.109	Nutmeat Hull	140 + 8	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0018 V-1040-A PPSI
USA, 1992 Pattison, TX (Spanish)	1	0.105	187	0.105	Nutmeat Hull	110 + 10	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0018 V-1040-B PPSI
USA, 1992 Hawkinsville, GA	1	0.108	215	0.108	Nutmeat Hull	134 + 7	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0018 V-1040-C

PEANUT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
(Florunner)	1	0.539	215	0.539	Nutmeat Hull	134 + 7	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	PE
USA, 1992 Hobgood, NC (NC-7)	1	0.105	187	0.105	Nutmeat Hull	148 + 10	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0018 V-1040-E PE
USA, 1993 Columbia, AL (Florunner)	1	0.108	185	0.108	Nutmeat Hull	135 + 4	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0019 V-10716-A PPSI
USA, 1993 Melrose, FL (Florunner)	1	0.109	238	0.109	Nutmeat Hull	148 + 3	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0019 V-10716-B PPSI
USA, 1993 Goldsboro NC (NC-7)	1	0.11	193	0.11	Nutmeat Hull	127 + 6	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0019 V-10716-C PPSI
USA, 1993 Pattison, TX (Spanish)	1	0.111	271	0.111	Nutmeat Hull	97 + 5	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0019 V-10716-D PE
USA, 1993 Hawkinsville, GA (Florunner)	1	0.106	215	0.106	Nutmeat Hull	152 + 8	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0019 V-10716-F PE
USA, 1993 Pattison, TX (STARR Spanish)	1	0.549	268	0.549	Nutmeat Hulls	101	< 0.02, < 0.02 0.04, 0.04	< 0.02 0.04	SBR-0019 V-10716-I PE
USA, 1996 Levelland, TX (Valonica McRan)	1	0.108	187	0.108	Nutmeat	154 + 7	< 0.02, < 0.02	< 0.02	SBR-0020 V-11438-B PE
USA, 1996 Unadilla, GA (Georgia Runner)	1	0.107	196	0.107	Nutmeat	139 + 4	< 0.02, < 0.02	< 0.02	SBR-0020 V-11438-C PE
USA, 1996 Columbia, AL (Southern Runner)	1	0.107	242	0.107	Nutmeat	154 + 6	< 0.02, < 0.02	< 0.02	SBR-0020 V-11438-D PE
USA, 1996 Malone, FL (GK-7)	1	0.102	243	0.102	Nutmeat	138 + 19	< 0.02, < 0.02	< 0.02	SBR-0020 V-11438-E PE
USA, 1996 Dill City, OK (Spanco)	1	0.11	131	0.11	Nutmeat	131 + 5	< 0.02, < 0.02	< 0.02	SBR-0020 V-11438-F PE

DAT = Interval from last application to cutting + field drying interval (in days)

PPSI = pre-plant soil incorporation

PE = pre-emergent broadcast soil treatment

Sunflower seed

In supervised trials on sunflowers (eight), single foliar broadcast sprays of 0.11 kg ai flumioxazin/ha (WG formulations) were applied with added adjuvant as pre-harvest desiccants and harvest aids using tractor-mounted or back-pack sprayers with 4–8 nozzle booms.

Duplicate samples of seed (min 1 kg from 12 flower heads) were frozen within 3.5 hours and analysed for flumioxazin within 11 months of harvest using method RM 30A-3 (GC-MS).

Concurrent recoveries from control samples fortified with flumioxazin at levels of 0.02–3.0 mg/kg ranged from 82–102% and the validated LOQ was 0.02 mg/kg.

Table 74 Residues in sunflower seed from supervised trials in the USA involving one pre-harvest foliar application of flumioxazin (WG formulation)

SUNFLOWER SEED COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2009 Northwood, ND (Pioneer 63M80)	1	0.11 + NIS	189	0.11	seed	5	0.03, 0.05	0.04	SBR0126 V-32835-A
	1	0.109 + MSO	187	0.109	seed	5	0.03, 0.03	0.03	
USA, 2009 Campbell, MN (Jaguar)	1	0.109 + MSO	187	0.109	seed	1 3 5 7	0.05, 0.05 0.06, 0.07 0.03, 0.04 0.02, 0.03	0.05 0.06 <u>0.04</u> 0.03	SBR0126 V-32835-B
USA, 2009 Stafford, KS (Pioneer 63M91)	1	0.105 + MSO	200	0.105	seed	5	0.05, 0.05	0.05	SBR0126 V-32835-C
	1	0.216 + MSO	207	0.216	seed	5	0.14, 0.15	0.14	
USA, 2009 Norwich, ND (Mycogen 8N358CL)	1	0.108 + MSO	187	0.108	seed	5	0.09, 0.1	0.1	SBR0126 V-32835-D
	1	0.219 + MSO	191	0.219	seed	5	0.14, 0.2	0.17	
USA, 2009 Velva, ND (Mycogen 8N358CL)	1	0.109 + NIS	188	0.109	seed	5	0.13, 0.15	0.14	SBR0126 V-32835-E
	1	0.11 + MSO	189	0.11	seed	5	0.17, 0.2	0.18	
USA, 2009 Grand Island, NE (3080 DMR NS)	1	0.108 + NIS	187	0.108	seed	4 + 1	0.18, 0.18	0.18	SBR0126 V-32835-F
	1	0.108 + MSO	186	0.108	seed	4 + 1	0.06, 0.07	0.07	
USA, 2009 Malta, MT (Croplan Genetics)	1	0.108 + NIS	187	0.108	seed	5	0.11, 0.12	0.12	SBR0126 V-32835-G
USA, 2009 Hinton, OK (Mycogen 8N435DM)	1	0.111 + MSO	144	0.111	seed	5	0.23, 0.34	0.29	SBR0126 V-32835-H

DAT = Interval from last application to cutting + field drying interval (in days)

Herbs

Mints

In supervised trials on <u>mint</u> (six) conducted in the USA during 2001, two foliar broadcast sprays of 0.28 or 0.42 kg ai flumioxazin/ha (WG formulations) were applied to dormant mint plants (February-April). The intervals between treatments were not reported in the study report. Duplicate samples of mint tops (leaves and stems) were stored frozen for up to 9 months before analysis for flumioxazin using method RM 30A-2 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 72–113% and the validated LOQ was 0.02 mg/kg.

In two of the trials, mint oil was extracted on the same day of harvest and stored frozen for up to 8 months before dilution with acetone and analysis for flumioxazin using method RM 30A-2 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 91–111% and the validated LOQ was 0.02 mg/kg.

Table 75 Residues in mint leaves and oil from supervised trials in the USA involving two foliar applications of flumioxazin (WG formulation)

MINT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
GAP:USA		0.14	140–180	0.28		80	Foliar sprays to dormant plants		
USA, 2001 Roza Unit C-9, WA (Mint)	2	0.28		0.56	leaves oil	112	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0136 WA*01
	2	0.42		0.84	leaves oil	112	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	
USA, 2001 Paterson, WA (Peppermint)	2	0.28		0.56	leaves	80	< 0.02, < 0.02	< 0.02	SBR-0136 WA*02
	2	0.42		0.84	leaves	80	< 0.02, < 0.02	< 0.02	
USA, 2001 Paterson, WA (Spearmint)	2	0.28		0.56	leaves	80	0.02, 0.02	0.02	SBR-0136 WA*03
	2	0.42		0.84	leaves	80	0.03, 0.03	0.03	
USA, 2001 Portage, WI (Peppermint)	2	0.28		0.56	leaves oil	112	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0136 WI-01
	2	0.42		0.84	leaves oil	112	< 0.02, < 0.02	< 0.02	
USA, 2001 Portage, WI (Spearmint)	2	0.28		0.56	leaves	79	< 0.02, < 0.02	< 0.02	SBR-0136 WI-02
	2	0.42		0.84	leaves	79	< 0.02, 0.02	0.02	
USA, 2001 Portage, WI (Spearmint)	2	0.28		0.56	leaves	79	< 0.02, < 0.02	< 0.02	SBR-0136 WI-03 not independent
	2	0.42		0.84	leaves	79	< 0.02, < 0.02	< 0.02	

Legume animal feeds

Alfalfa forage and fodder

In supervised trials on alfalfa (six) conducted in the USA during 2003, two foliar broadcast sprays of 0.14–0.15 kg ai flumioxazin/ha (WG formulations) were applied 24–26 days before the first cutting (with added surfactant) and to the alfalfa regrowth 6–8 days after the first cutting using back pack or tractor-mounted boom sprayers (6–9 nozzles). Retreatment intervals ranged from 30–33 days. In further trials conducted in 2005, single foliar broadcast sprays of 0.14 kg ai flumioxazin/ha (with added surfactants) were applied to alfalfa regrowth 7–9 days after the first cutting using back pack or tractor-mounted boom sprayers (4–8 nozzles).

Duplicate samples of forage (min 1 kg) were taken 6–26 days after the second application and fodder (hay) samples (min 0.5 kg) were taken after a further 2–8 days drying in the field. Samples were frozen within 6 hours and analysed for flumioxazin within 15 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02–2.0 mg/kg (forage) and 0.02–7.0 mg/kg (fodder) ranged from 71–120% and the validated LOQ was 0.02 mg/kg.

Table 76 Residues in alfalfa forage from supervised trials in the USA involving one or two foliar applications of flumioxazin (WG formulation)

ALFALFA	APPLICATION	MATRI	DAT	RESIDUES (MG/KG)	REFEREN

FORAGE COUNTRY, YEAR LOCATION (VARIETY)	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON	X		FLUMIOXAZIN	MEAN	CE & COMMENTS
USA, 2003 Germansville, PA (WL-325)	2	0.14 0.144	234 241	0.284	forage	25 60 113	0.09, 0.12 < 0.02, < 0.02 < 0.02, < 0.02	0.11 < 0.02 < 0.02	SBR-0111 V-25814-A
	2	0.279 0.284	234 237	0.563	forage	25 60 113	0.36, 0.44 0.03, 0.04 < 0.02, < 0.02	0.4 0.04 < 0.02	
USA, 2003 Columbia, MO (Cody)	2	0.141 0.15	239 220	0.291	forage	6 15 24 35 65 107	2.2, 2.3 0.33, 0.37 0.08, 0.16 0.03, 0.06 0.02, 0.03 < 0.02, < 0.02	2.3 0.35 0.12 0.05 0.03 < 0.02	SBR-0111 V-25814-B
USA, 2003 York, NE (Haymark)	2	0.14 0.14	187 187	0.28	forage	25 50 97	0.12, 0.12 0.07, 0.1, 0.18, 0.19 < 0.02, < 0.02	0.12 0.14 < 0.02	SBR-0111 V-25814-C
USA, 2003 Britton, SD (Dekalb DK 122)	2	0.14 0.14	187 187	0.28	forage	25 55 90	0.03, 0.03 0.02, 0.02 < 0.02 (3), 0.06 (2), 0.09	0.03 0.02 0.04	SBR-0111 V-25814-D
USA, 2003 Clarence, MO (UNS Missouri Certified Seed)	2	0.14 0.139	187 186	0.279	forage	25 61 104	0.09, 0.11 < 0.02, < 0.02 < 0.02, < 0.02	0.1 < 0.02 < 0.02	SBR-0111 V-25814-E
USA, 2003 Eden, AZ (Mesa Circi)	2	0.14 0.137	190 186	0.277	forage	25 60 101	0.35, 0.43 0.02, 0.02 < 0.02, < 0.02	0.39 0.02 < 0.02	SBR-0111 V-25814-G
USA, 2005 Franklin, GA (Emerald)	1	0.141	208	0.141	forage	25 70 128	0.79, 0.8 < 0.02, < 0.02 < 0.02, < 0.02	0.8 < 0.02 < 0.02	SBR-0111 V-25814-H
	1	0.283	208	0.283	forage	25 70	1.1, 1.7 < 0.02, < 0.02	1.4 < 0.02	
USA, 2005 New Holland, OH (Rocket)	1	0.144	144	0.144	forage	24 62 87	0.02, 0.03 < 0.02, < 0.02 < 0.02, < 0.02	0.03 < 0.02 < 0.02	SBR-0111 V-25814-I
USA, 2005 Carlyle, IL (Buffalo)	1	0.138	148	0.138	forage	24 49 76	0.07, 0.13 < 0.02, < 0.02 < 0.02, < 0.02	0.1 < 0.02 < 0.02	SBR-0111 V-25814-J
	1	0.278	149	0.278	forage	24 49 76	< 0.02, 0.26 < 0.02, < 0.02 < 0.02, < 0.02	0.14 < 0.02 < 0.02	
USA, 2005 Velva, ND (Vernal)	1	0.139	139	0.139	forage	24 56 99	0.05, 0.06 < 0.02, < 0.02 < 0.02, < 0.02	0.06 < 0.02 < 0.02	SBR-0111 V-25814-K
USA, 2005 Live Oak, CA (Achiever)	1	0.136	137	0.136	forage	25 45 71	0.22, 0.24 0.03, 0.03 < 0.02, < 0.02	0.23 0.03 < 0.02	SBR-0111 V-25814-L
USA, 2005 Payette, ID (Unknown Pioneer variety)	1	0.141	236	0.141	forage	26 57 97	0.14, 0.21 < 0.02, 0.02 < 0.02, < 0.02	0.18 0.02 < 0.02	SBR-0111 V-25814-M

Table 77 Residues in alfalfa fodder (hay) from supervised trials in the USA involving one or two foliar applications of flumioxazin (WG formulation)

ALFALFA FODDER COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2003 Germansville, PA (WL-325)	2	0.14 0.144	234 241	0.284	fodder	25 + 4 60 + 7 113 + 7	0.36, 0.34 < 0.02, 0.02 < 0.02, < 0.02	0.35 0.02 < 0.02	SBR-0111 V-25814-A
		0.279 0.284	234 237	0.563	fodder	25 + 4 60 + 7 113 + 7	2.1, 2.2 0.06, 0.08 < 0.02, < 0.02	2.2 0.07 < 0.02	
USA, 2003 Columbia, MO (Cody)	2	0.141 0.15	239 220	0.291	fodder	6 + 2 15 + 2 24 + 2 35 + 2 65 + 4 107 + 5	5.4, 5.0 1.2, 1.4 0.27, 0.27 0.08, 0.12 0.04, 0.05 < 0.02, < 0.02	5.2 1.3 0.27 0.10 0.05 < 0.02	SBR-0111 V-25814-B
USA, 2003 York, NE (Haymark)	2	0.14 0.14	187 187	0.28	fodder	25 + 4 50 + 5 97 + 4	0.29, 0.17 0.04, 0.03, 0.05, 0.09 < 0.02, < 0.02	0.23 0.05 < 0.02	SBR-0111 V-25814-C
USA, 2003 Britton, SD (Dekalb DK 122)	2	0.14 0.14	187 187	0.28	fodder	25 + 3 55 + 4 90 + 4	0.07, 0.06 0.02, 0.03 0.10, 0.14, 0.13, 0.15	0.07 0.03 0.13	SBR-0111 V-25814-D
USA, 2003 Clarence, MO (UNS Missouri Certified Seed)	2	0.14 0.139	187 186	0.279	fodder	25 + 4 61 + 2 104 + 5	0.23, 0.18 0.02, 0.02 < 0.02, < 0.02	0.21 0.02 < 0.02	SBR-0111 V-25814-E
USA, 2003 Eden, AZ (Mesa Circi)	2	0.14 0.137	190 186	0.277	fodder	25 + 3 60 + 4 101 + 3	1.1, 1.3 0.02, 0.04 < 0.02, < 0.02	1.2 0.03 < 0.02	SBR-0111 V-25814-G
USA, 2005 Franklin, GA (Emerald)	1	0.141	208	0.141	fodder	25 + 7 70 + 4	1.4, 1.6 0.03, < 0.02	1.5 0.02	SBR-0111 V-25814-H
	1	0.283	208	0.283	fodder	25 + 7 70 + 4	5.5, 3.0 0.03, 0.03	4.3 0.03	
USA, 2005 New Holland, OH (Rocket)	1	0.144	144	0.144	fodder	24 + 3 62 + 1 87 + 3	0.11, 0.11 < 0.02, < 0.02 < 0.02, < 0.02	0.11 < 0.02 < 0.02	SBR-0111 V-25814-I
USA, 2005 Carlyle, IL (Buffalo)	1	0.138	148	0.138	fodder	24 + 3 49 + 9 76 + 3	0.23, 0.36 0.03, 0.04 < 0.02, < 0.02	0.3 0.04 < 0.02	SBR-0111 V-25814-J
	1	0.278	149	0.278	fodder	24 + 3 49 + 9 76 + 3	0.94, 0.51 0.07, 0.07 < 0.02, < 0.02	0.73 0.07 < 0.02	
USA, 2005 Velva, ND (Vernal)	1	0.139	139	0.139	fodder	24 + 1 56 + 4 99 + 2	0.22, 0.25 0.02, < 0.02 < 0.02, < 0.02	0.24 0.02 < 0.02	SBR-0111 V-25814-K
USA, 2005 Live Oak, CA (Achiever)	1	0.136	137	0.136	fodder	25 + 4 45 + 4 71 + 2	0.47, 0.45 0.07, 0.09 < 0.02, < 0.02	0.46 0.08 < 0.02	SBR-0111 V-25814-L
USA, 2005 Payette, ID (Unknown Pioneer variety)	1	0.141	236	0.141	fodder	26 + 5 57 + 3 97 + 8	0.88, 0.84 0.04, 0.05 < 0.02, < 0.02	0.86 0.05 < 0.02	SBR-0111 V-25814-M

DAT = Interval from last application to cutting + field drying interval (in days)

Peanut forage and fodder

In fifteen supervised trials on peanuts, single broadcast soil applications of 0.1–0.11 kg ai flumioxazin/ha (WG formulations) were applied either as pre-plant broadcast sprays (with shallow soil incorporation) within 7 days before sowing or as pre-emergent broadcast sprays within 5 days after sowing, using tractor-mounted boom sprayers (8–13 nozzles).

Duplicate samples of peanut vines were collected immediately after digging, and samples of hay (min 0.45 kg) were collected after 3–19 days of field drying and kept in frozen storage up to 210 days before analysis for flumioxazin using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02–0.1 mg/kg ranged from 76–113% (vines) and 63–86% (hay) and the validated LOQs were 0.02 mg/kg.

Table 78 Residues in peanut vines and hay from supervised trials in the USA involving one broadcast pre-plant or pre-emergent soil application of flumioxazin (WG formulations)

PEANUT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1992 Alabama (Florunner)	1	0.109	187	0.109	Vines Hay	132	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0018 V-1040-A
USA, 1992 Georgia (Florunner)	1	0.108	215	0.108	Vines Hay	134	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0018 V-1040-C
USA, 1992 North Carolina (NC-7)	1	0.105	187	0.105	Vines Hay	148	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0018 V-1040-E
USA, 1992 Texas (Spanish)	1	0.105	187	0.105	Vines Hay	110	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0018 V-1040-B
USA, 1993 Alabama (Florunner)	1	0.108	185	0.108	Vines Hay	135	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0019 V-10716-A
USA, 1993 Florida (Florunner)	1	0.109	238	0.109	Vines Hay	148	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0019 V-10716-B
USA, 1993 Georgia (Florunner)	1	0.106	215	0.106	Vines Hay	152 14 21 28 152	< 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	SBR-0019 V-10716-F
USA, 1993 North Carolina (NC-7)	1	0.11	193	0.11	Vines Hay	127 21 28	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02	SBR-0019 V-10716-C
USA, 1993 Texas (Spanish)	1	0.111	271	0.111	Vines Hay	97	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0019 V-10716-D

Soya bean forage and fodder

In supervised trials on soya beans conducted between 1989 and 1993, single broadcast soil application of 0.1–0.11 kg ai flumioxazin/ha (WG, FL or WP formulations) were applied using back-pack or

tractor-mounted boom sprayers, either as pre-plant treatments (with or without soil incorporation) or just after sowing, before crop emergence.

Duplicate samples of forage (min 0.9 kg) and hay (min 0.45 kg) were frozen within 24 hours and stored for up to 13 months (forage) and 11 months (hay) before analysis for flumioxazin using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 mg/kg ranged from 67–120% in forage and 73–130% in hay, with a validated LOQ of 0.02 mg/kg.

Table 79 Residues in soya bean forage and fodder from supervised trials in the USA involving one broadcast soil application of flumioxazin (WG formulations)

SOYA BEAN COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
GAP:USA		0.105	140–280	0.105			pre-plant or pre-emergent		
USA, 1989 Dallas Center, IA (Asgrow 1937)	1	0.101	94	0.101	forage hay	40 111	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7262
USA, 1989 Dallas Center, IA (Wells II)	1	0.101	187	0.101	forage hay	40 103	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7370 no cultivation
USA, 1989 Geneseo, IL (Pioneer 9271)	1	0.101	187	0.101	forage hay	40 103	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7374
USA, 1989 Greenville, MS (Forrest)	1	0.101	187	0.101	forage hay seed	40 113	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7373
USA, 1989 Hollandale, MN (NK523-12)	1	0.101	187	0.101	forage hay	67 95	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7260
USA, 1989 Lanoke, AR (Asgrow 5980)	1	0.101	94	0.101	forage hay	40 102	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7263
USA, 1989 Leonard, MO (Williams 82)	1	0.101	374	0.101	forage hay	40 100	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7368
USA, 1989 Metcalfe, MS (Forrest)	1	0.101	187	0.101	forage hay	40 100	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7375
USA, 1989 New Holland, OH (Pioneer 9361)	1	0.101	365	0.101	forage hay	40 100	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7369
USA, 1989 Noblesville IN (Pioneer 9361)	1	0.101	206	0.101	forage hay	40 110	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7261
USA, 1989 Rosa, LA (Forrest)	1	0.101	212	0.101	forage hay	64 149	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7372
USA, 1989 York, NE (Hack)	1	0.101	187	0.101	forage hay	40 90	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7371
USA, 1990 Clarence, MO (Williams 82)	1	0.101	187	0.101	forage hay	40 91	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7512
USA, 1990 Cloverport, TN (FFR 562)	1	0.101	187	0.101	forage hay	40 79	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7501

SOYA BEAN COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1990 Dallas Center, IA (Asgrow 2187)	1	0.101	187	0.101	whole plant whole plant whole plant whole plant whole plant whole plant hay	8 15 29 40 60 90 90	< 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 < 0.02 < 0.02 < 0.02 < 0.02	SBR-0003 T-7507 pre-emergence
USA, 1990 Dallas Center, IA (Asgrow 2187)	1	0.101	187	0.101	forage hay	40 99	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7509 pre-plant
USA, 1990 Elwood, IL (Pioneer 9202)	1	0.101	196	0.101	forage hay	40 107	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7508
USA, 1990 Geneseo, IL (Pioneer 9272)	1	0.101	187	0.101	forage hay	40 40	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7502
USA, 1990 Greenville, MS (Forrest)	1	0.101	187	0.101	whole plant whole plant whole plant whole plant whole plant whole plant hay	7 15 30 39 60 90 90	 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	0.07 0.06 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	SBR-0003 T-7506
USA, 1990 Hollandale, MN (Agri Pro 1776)	1	0.101	187	0.101	forage hay	40 102	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7511
USA, 1990 Hollendale, MN (Agri Pro1776)	1	0.101	187	0.101	forage hay	40 102	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7500 no cultivation
USA, 1990 New Holland, OH (Pioneer 9391)	1	0.101	243	0.101	forage hay	41 93	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7510
USA, 1990 Noblesville, IN (Pioneer 9361)	1	0.101	253	0.101	forage hay	40 72	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7503
USA, 1990 Proctor, AR (DPL 105)	1	0.101	187	0.101	forage hay	40 110	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7513
USA, 1992 Goldsboro, NC (Ransom)	1	0.105	187	0.105	forage forage forage hay seed	22 29 41 123	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-1039-A
USA, 1992 Greenville, MS (Pioneer 9641)	1	0.105	187	0.105	forage forage forage forage hay	13 20 28 39 99	< 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	SBR-0021 V-1039-H
USA, 1992 Leonard, MO (Pioneer 9443)	1	0.102	187	0.102	forage forage forage hay	21 28 40 91	 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	0.03 < 0.02 < 0.02 < 0.02	SBR-0021 V-1039-M
USA, 1992 Little Rock, AR (Hutcheson)	1	0.105	187	0.105	forage forage forage hay	21 28 40 110	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-1039-C

SOYA BEAN COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1992 New Holland, OH (GL 2910)	1	0.105	150	0.105	forage forage forage hay	22 29 42 106	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-1039-G
USA, 1992 Noblesville, IN (Pioneer 9361)	1	0.105	234	0.105	forage forage hay	28 40 97	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02	SBR-0021 V-1039-D
USA, 1992 Seymour, IL (Asgrow 2543)	1	0.105	187	0.105	forage forage forage hay	21 28 40 102	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-1039-B pre-plant
USA, 1992 Seymour, IL (Asgrow 2543)	1	0.105	187	0.105	forage forage forage hay	21 28 40 98	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-1039-J pre-emergence
USA, 1992 Waukee, IA (Asgrow 2543)	1	0.105	187	0.105	forage forage forage forage hay	14 21 28 39 98	< 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	SBR-0021 V-1039-F
USA, 1993 Greenville, MS (Asgrow 5979)	1	0.109	187	0.109	forage forage forage forage hay	14 21 28 41 98	< 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	SBR-0021 V-10719-H
USA, 1993 Jamesville, NC (Hutcheson)	1	0.108	253	0.108	forage forage forage hay	21 28 40 122	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-10719-A
USA, 1993 Leonard, MO (Linford)	1	0.107	271	0.107	forage forage forage hay	21 28 40 88	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-10719-E
USA, 1993 New Holland, OH (Madison GL 2910)	1	0.107	196	0.107	forage forage forage hay	22 31 40 88	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-10719-G
USA, 1993 Noblesville, IN (Pioneer 9361)	1	0.11	206	0.11	forage forage forage hay	21 27 40 108	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-10719-D
USA, 1993 Seymour, IL (Asgrow 2506)	1	0.107	187	0.107	forage forage forage forage hay	14 22 28 40 83	< 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	SBR-0021 V-10719-J
USA, 1993 Theilman, MN (Pioneer 9061)	1	0.107	187	0.107	forage forage hay	28 40 101	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02	SBR-0021 V-10719-B
USA, 1993 Webster City, IA (L-1700)	1	0.108	206	0.108	forage forage hay	28 41 80	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02	SBR-0021 V-10719-F
USA, 1993 York, NE (Hack)	1	0.107	187	0.107	forage forage forage hay	21 28 40 85	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-10719-C

The 1992–1993 supervised trials also analysed for residues of the metabolite, 1-OH HPA in seeds and the results also showed levels below the LOQ of 0.02 mg/kg.

Straw, forage, fodder of cereal grains

Maize forage and fodder

In twenty-one supervised trials on maize, single broadcast soil applications of 0.1–0.11 or 0.2–0.22 kg ai flumioxazin/ha (WG formulations) with added surfactant were applied up to 7 days before sowing, using back-pack plot sprayers, wheeled or tractor-mounted boom sprayers (3–9 nozzles).

Duplicate samples of forage (min 12 units) were taken at the late dough/early dent growth stage (about BBCH 86) and stover samples (min 12 units) were taken at grain harvest. Samples were all frozen within 2 hours and analysed for flumioxazin within 14 months using method RM 30A-3 (GC-MS) in the 2005 trials and method NCL 293 (HPLC-MS/MS) in the 2006 trials. Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 87–117% (forage) and 79–118% (stover) in the two methods and the validated LOQs were 0.02 mg/kg.

Table 80 Residues in maize forage and fodder from supervised trials in the USA involving one broadcast pre-plant soil application of flumioxazin (WG formulations)

MAIZE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2005 New Holland, OH (Syngenta N73-F7)	1	0.107	191	0.107	forage stover	103 148	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-A
	1	0.211	188	0.211	forage stover	103 148	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	
USA, 2005 Carlyle, IL (FS 6455)	1	0.107	190	0.107	forage stover	102 171	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-B
	1	0.212	187	0.212	forage stover	102 171	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	
USA, 2005 Clarence, MO (Pioneer 35P12)	1	0.107	191	0.107	forage stover	119 154	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-C
USA, 2005 Greenville, MS (69-71 757 HXJINX)	1	0.104	185	0.104	forage stover	118 135	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-D
USA, 2006 North Rose, NY (Dairyland Stealth 8711)	1	0.108	191	0.108	forage stover	93 131	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-E
USA, 2006 Elko, SC (Pioneer 31R87)	1	0.105	180	0.105	forage stover	106 158	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-F
Canada, 2006 City of Hamilton, Ontario (Pioneer 38B84)	1	0.107	187	0.107	forage stover	107 166	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-G
	1	0.211	184	0.211	forage stover	107 166	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	
USA, 2006 Conklin, MI (N45-M2 Field Corn)	1	0.106	189	0.106	forage stover	97 138	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-H

MAIZE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2006 Carlyle, IL (DKC-65-16)	1	0.108	185	0.108	forage stover	112 168	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-I
USA, 2006 Bellmore, IN (Wyffels 5531)	1	0.104	185	0.104	forage stover	106 136	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-J
USA, 2006 York, NE (NK N70-F1)	1	0.106	184	0.106	forage stover	117 155	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-K
USA, 2006 Richland, IA (Pioneer 33P65)	1	0.105	189	0.105	forage stover	109 151	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-L
USA, 2006 Geneva, MN (Pioneer 38H66)	1	0.106	180	0.106	forage stover	110 163	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-M
USA, 2006 Fairmount, ND (Dekalb 35-02)	1	0.106	188	0.106	forage stover	100 145	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-N
USA, 2006 Campbell, MN (Pioneer 39H83)	1	0.106	188	0.106	forage stover	100 155	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-O
USA, 2006 Hudson, KS (Midwest Seed Genetics 8127RB)	1	0.106	188	0.106	forage stover	104 134	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-P
Canada, 2006 Portage la Prairie, Manitoba (Roundup Ready-Monsanto)	1	0.102	181	0.102	forage stover	114 154	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-Q
USA, 2006 Arkansaw, WI (Pioneer 38B85)	1	0.106	188	0.106	forage stover	110 137	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-R
Canada, 2006 St. Pie, Quebec (NK 3030 BT)	1	0.101	176	0.101	forage stover	122 156	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-S
USA, 2006 Dill City, OK (DKC48-53)	1	0.107	193	0.107	forage stover	98 130	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-T
USA, 2006 Clarence, MO (Pioneer 34B20)	1	0.107	187	0.107	forage stover	105 165	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-U
USA, 2006 Clarence, MO (Pioneer 34B20)	1	0.536	187	0.536	forage stover	105 165	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-U (Processing)

Wheat forage, hay and straw

In three supervised trials on wheat, single pre-plant broadcast soil applications of 0.07 or 0.14 kg ai flumioxazin/ha (WG formulations) with added surfactant were applied 7 or 14 days before sowing respectively, using tractor-mounted boom sprayers (4–8 nozzles).

Duplicate samples of wheat forage (from plants about 13 cm tall) and hay (sampled at BBCH 61–85 and allowed to dry to 10–20% moisture content) were frozen within 2 hours and analysed for flumioxazin within 38 days of harvest using method RM 30A-3 (GC-MS). Concurrent recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 93–109% in forage, 89–120% in hay and the validated LOQ was 0.02 mg/kg.

Table 81 Residues in wheat forage and hay from supervised trials in the USA involving one broadcast pre-plant soil application of flumioxazin (WG formulations)

WHEAT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2010 Leland, MS (Gore)	1	0.07	182	0.07	forage hay	129 172 + 4 d dry	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0127 V-37119-A
	1	0.14	184	0.14	forage hay	129 172 + 4 d dry	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	
USA, 2010 Levelland, TX (TAM 112)	1	0.71	186	0.71	forage hay	85 247 + 4 d dry	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0127 V-37119-B
		0.144	188	0.144	forage hay	79 241 + 4 d dry	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	
USA, 2010 Larned, KS (Santa Fe Winter Wheat)	1	0.72	188	0.72	forage hay	71 262 + 5 d dry	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0127 V-37119-C
	1	0.143	186	0.143	forage hay	64 255 + 5 d dry	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	

In twenty supervised trials on wheat, single foliar broadcast sprays of 0.07–0.075 kg ai flumioxazin/ha (WG formulations) with added adjuvants were applied as pre-harvest desiccants (harvest aids) using tractor-mounted or back-pack sprayers with 4–8 nozzle booms.

Duplicate samples of straw were collected using small plot combines or cut and harvested using a stationary combine, frozen within 5 hours and analysed for flumioxazin within 17 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control straw samples fortified with flumioxazin at levels of 0.02–5.0 mg/kg ranged from 70–115% and the validated LOQ was 0.02 mg/kg.

Table 82 Residues in wheat straw from supervised trials in the USA involving one pre-harvest foliar application of flumioxazin (WG formulation)

WHEAT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	

WHEAT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2009 Lexington, GA (USG 3592)	1	0.071 + NIS	193	0.071	straw	10	1.88, 1.74	1.82	SBR-0092 V-33037-A
	1	0.071 + MSO	192	0.071	straw	10	3.46, 3.95	3.71	
USA, 2009 Leland, MS (Gore)	1	0.071 + MSO	185	0.071	straw	3 7 10 13	3.53, 2.85 1.18, 1.42 2.4, 2.69 1.14, 0.92	3.19 1.30 2.55 1.03	SBR-0092 V-33037-B
USA, 2009 Carlyle, IL (Branson)	1	0.072 + MSO	199	0.072	straw	10	0.86, 0.66	0.76	SBR-0092 V-33037-C
		0.145 + MSO	200	0.145	straw	10	2.11, 2.62	2.37	
USA, 2009 York, NE (Traverse Hard red Spring)	1	0.071 + NIS	184	0.071	straw	10	0.88, 0.99	0.94	SBR-0092 V-33037-D
	1	0.071 + MSO	186	0.071	straw	10	1.91, 1.75	1.83	
USA, 2009 Rockville, IN (Becks 164)	1	0.072 + NIS	148	0.072	straw	11	1.79, 1.13	1.46	SBR-0092 V-33037-E
	1	0.072 + MSO	148	0.072	straw	11	1.42, 1.1	1.26	
USA, 2009 Clarence, MO (Ernie)	1	0.074 + NIS 0.07 + MSO	193 183	0.074 0.07	straw	10 10	2.01, 1.67 2.5, 2.19	1.84 2.35	SBR-0092 V-33037-F
USA, 2009 Bagley, IA (Briggs hrS)	1	0.71 + NIS	148	0.071	straw	10	0.49, 1.2	0.85	SBR-0092 V-33037-G
	1	0.072 + MSO	150	0.072	straw	10	1.34, 1.83	1.59	
USA, 2009 Ulvade, TX (Fannin)	1	0.07 + NIS	138	0.07	straw	9	2.9, 3.53	3.22	SBR-0092 V-33037-H
	1	0.072 + MSO	142	0.072	straw	9	3.32, 3.48	3.40	
USA, 2009 Grand Island, NE (Traverse Hard Red Spring)	1	0.072 + NIS	189	0.072	straw	10	1.64, 1.49	1.57	SBR-0092 V-33037-I
	1	0.071 + MSO	186	0.071	straw	10	2.0, 1.48	1.74	
USA, 2009 Velva, ND (Faller)	1	0.072 + MSO	141	0.072	straw	10	3.1, 3.3	3.2	SBR-0092 V-33037-J
USA, 2009 Grand Island, NE (Kelby Hard Red Spring)	1	0.071 + NIS	185	0.071	straw	10	0.99, 1.13	1.06	SBR-0092 V-33037-K not independent
USA, 2009 Norwich, ND (Faller)	1	0.072 + MSO	172	0.072	straw	10	1.73, 1.36	1.55	SBR-0092 V-33037-L
	1	0.146 + MSO	143	0.146	straw	10	4.21, 2.48	3.35	
USA, 2009 Malta, MT (McNeal)	1	0.069 + NIS	181	0.069	straw	10	2.48, 3.63	3.19	SBR-0092 V-33037-M
USA, 2009 Levelland, TX (TAM 105)	1	0.072 + NIS	189	0.072	straw	9	1.39, 2.03	1.71	SBR-0092 V-33037-N
USA, 2009	1	0.072 + MSO	165	0.072	straw	9	1.64, 2.02	1.83	SBR-0092

WHEAT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
Wellington, TX (TAM 111)	1	0.142 + MSO	163	0.142	straw	9	3.33, 3.93	3.63	V-33037-O
USA, 2009 Larned, KS (Jagger)	1	0.074 + NIS	212	0.074	straw	10	0.21, 0.25	0.23	SBR-0092 V-33037-P
USA, 2009 Hinton, OK (Jagger)	1	0.07 + MSO	164	0.07	straw	4 7 10 13	2.09, 2.53 2.85, 2.35 1.93, 2.18 1.9, 1.91	2.31 2.60 2.06 1.91	SBR-0092 V-33037-Q
USA, 2009 Cordell, OK (Fuller)	1	0.072 + MSO	172	0.072	straw	11	1.2, 1.94	1.57	SBR-0092 V-33037-R
USA, 2009 Jerome, ID (AC Andrew)	1	0.071 + NIS	181	0.071	straw	10	1.33, 1.4	1.37	SBR-0092 V-33037-S
USA, 2009 Hinton, OK (Deliver)	1	0.071 + MSO	165	0.071	straw	10	1.49, 1.93	1.71	SBR-0092 V-33037-T not independent
	1	0.344 + MSO	152	0.344	straw	10	7.66, 9.29	8.48	

NIS = Non-ionic surfactant

MSO = Methylated seed oil surfactant

Fate of residues in storage and processing

The meeting received processing studies on apples, plums, grapes, olives, soya beans, potatoes, sugar cane, maize, wheat, sugar cane, oilseed rape, sunflower seed, peanuts and mint. In all cases, fresh commodity samples collected from supervised trials at exaggerated rates were processed simulating commercial practices.

Apple

In a supervised trial on apples conducted in the USA and reported by Stearns, 2004 [Ref: SBR-0031], two inter-row/berm soil treatments of 0.86 kg ai flumioxazin/ha (SC formulation) were applied using an ATV-mounted boom sprayer (six nozzles). Treatments were applied 60 days apart, with the last application 60 days before harvest.

Duplicate samples of 30 kg mature fruit were frozen within 1 hours and stored for 5 days before processing into apple wet pomace and juice, simulating commercial practices. Unwashed apples were ground using a hammer mill, then pressed in a hydraulic press to provide apple juice and wet pomace. The processed samples were stored frozen for up to 9 months before analysis for flumioxazin using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02–0.1 mg/kg ranged from 79–110% (apples) and 90–98% (wet pomace). Recoveries in juice spiked with 0.005–0.5 mg/kg were 95–103%. In juice the validated LOQ was 0.02 mg/kg.

Table 83 Residues in apples, pomace and juice from a supervised trial in the USA involving two directed inter-row soil applications of flumioxazin (SC formulation)

APPLE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	

APPLE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2002 Ephrata, WA (Rome)	2	0.86 0.86	200 201	1.723	whole fruit wet pomace juice	60	< 0.02, < 0.02 < 0.02, < 0.02 < 0.005, < 0.005	< 0.02 < 0.02 < 0.005	SBR-0031 V-24504-02-G

Plum

In a supervised trial on plums conducted in the USA and reported by Kowalsky, 2004 [Ref: SBR-0030], two inter-row/berm soil treatments of 0.86 kg ai flumioxazin/ha (SC formulation) were applied using a tractor-mounted boom sprayer (six nozzles). Treatments were applied 64 days apart, with the last application 60 days before harvest.

Duplicate samples of mature plums (33 kg) were processing into prunes on the day of harvest by removing stems and leaves, washing the fruits with a hose, and air dying in drying tunnels for about 19 at 86 °C and allowed to cool for 24 hours. Duplicate samples were analysed for flumioxazin within 9 months of processing using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 83–98% (plums) and 105–109% (prunes) and the validated LOQ was 0.02 mg/kg.

Table 84 Residues in plums and prunes from a supervised trial in the USA involving two directed inter-row soil applications of flumioxazin (SC formulation)

PLUM COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2002 Hughson, CA (French)	2	0.86 0.864	375 375	1.72	whole fruit prunes	60	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0030 V-24539-G

Grape

In a supervised trial on grapes conducted in the USA and reported by Schreier, 2000 [Ref: SBR-0025], two directed inter-row/berm soil treatments of 2.1 kg ai flumioxazin/ha (WG formulation) with added crop oil were applied using a back-pack sprayer with hand-held 4-nozzle boom. Treatments were applied 60 days apart with the last application 60 days before harvest. Duplicate samples of grapes (9 kg for juice processing and 56 kg for raisin processing) were processed into juice and raisins within 24 hours of sampling.

Juice was prepared by washing the grape bunches with water then hand feeding them into a crusher/stemmer machine. The grape pulp was separated from the stems and seeds and transferred to a hydraulic fruit press. The fresh juice collected from the press was filtered to remove coarse solids prior to freezing and storage for up to 1.6 months before analysis for flumioxazin using method RM 30A-1.

Grapes were processed into raisins by sun-drying on trays in the field for about a month before being screened to remove loose dirt, stems and debris and hand sorted to remove the cap stems and any additional unacceptable product. The raisins were batch washed for 10–15 seconds, re-hydrated to approximately 18% moisture, frozen and stored for up to 5 months before analysis for flumioxazin using method RM 30A-1.

Recoveries from control samples fortified with flumioxazin at levels of 0.01 and 0.05 mg/kg ranged from 82–123% (grapes), 95–115% (juice) and 96–106% (raisins). The validated LOQs were 0.01 mg/kg.

Table 85 Residues in fresh and processed grapes from a supervised trial in the USA involving two inter-row soil applications of flumioxazin (WG formulation)

GRAPES COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1999 Kerman, CA (Thompson seedless)	2	2.13 2.12	184 187	4.25	grapes washed grapes raisins juice	60	< 0.01, < 0.01 < 0.01, < 0.01 < 00.1, < 0.01 < 0.01, < 0.01	< 0.01 < 001 < 0.01 < 0.01	SBR-0025 V-20108-L

Olive

In a supervised trial on olives conducted in the USA and reported by Arsenovic and Leonard, 2011 [Ref: SBR-0130], two directed inter-row/berm soil treatments of 2.1 kg ai flumioxazin/ha (WG formulation) with added crop oil were applied using a back-pack sprayer with a hand-held 3-nozzle miniboom. Treatments were applied 62 days apart with the last application 56 days before harvest.

Duplicate samples (22 kg) of olives were refrigerated overnight and sent to the processing facility where the samples were cleaned of extraneous materials and then warmed in an oven for about 20 minutes at 24–29 °C. Warmed olives were ground in a food chopper to produce a paste, which was then placed in a mixer and transferred into a filter press. Pressure was applied to remove oil from the paste. The oil was filtered, collected and stored frozen for up to 17 months before analysis for flumioxazin using method RM 30A-03 (GC-MDS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 76–122% (olives) and 82–116% (oil) and the validated LOQ was 0.02 mg/kg.

Table 86 Residues in olives and oil from a supervised trial in the USA involving two inter-row soil applications of flumioxazin (WG formulations)

OLIVE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2008 Glenn, CA (Arbegnina 1-18 clone)	2	2.05 2.07	211 212	4.13	fruit without pits oil	56	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0130 CA91

Soya bean

In a supervised trial on soya beans conducted in the USA and reported by Pensyl, 1996 [Ref: SBR-0021], one broadcast soil applications of 0.536 kg ai flumioxazin/ha (WG formulation) was applied using a tractor-mounted boom sprayer (six nozzles), immediately after sowing.

Duplicate samples of seed (min 22 kg) were frozen within 24 hours and shipped overnight to the processing facility where the samples were dried, aspirated and screening before being mechanically cracked. Aspiration was used to separate the hull and kernel fractions and the kernels were heat-conditioned, flaked, expanded into collets, and solvent extracted to obtain the crude oil. The crude oil was degummed, refined, bleached, and deodorized.

Samples were stored for up to 13 months before analysis for flumioxazin using method RM 30A-3 (GC-MS) and also for the 1-OH-HPA metabolite, using method RM 30M (GC-MS). Recovery rates in samples spiked with 0.02 mg/kg flumioxazin ranged from 75–113% in seed and the processed commodities and were 71–100% in samples spiked with the 1-OH-HPA metabolite.

Table 87 Residues in soya bean seeds and processed commodities from a supervised trial in the USA involving one broadcast pre-emergence soil application of flumioxazin (WG formulation)

SOYA BEAN COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1993 Seymour, IL (Asgrow 2506)	1	0.536	187	0.536	seed hulls extracted meal crude oil crude lecithin refined oil soapstock	112	< 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 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-10719-K

The 1992–1993 studies also analysed for residues of the metabolite, 1-OH HPA. Residues in all samples were below the LOQ of 0.02 mg/kg.

Potato

In a supervised trial on potatoes conducted in the USA and reported by Arsenovic, 2003 [Ref: SBR-0091], one broadcast soil applications of 0.14 kg ai flumioxazin/ha (WG formulation) was applied using an ATV-mounted boom sprayer (five nozzles) after the last hilling operation, before potato emergence.

Duplicate samples of 22 kg potatoes were cool-stored for 2 days before processing into wet peel, chips, and flakes. Potato tubers were cleaned, washed, peeled with an abrasive peeler and sliced into chips using a food cutter. The slices of potato were rinsed in warm water to remove free starch, and then fried. The oil was drained and the chips salted, packed and stored. Potato flakes were prepared from cleaned potato tubers, which were washed, peeled using a steam peeler, inspected and trimmed. The potato peel was collected and pressed hydraulically in a fruit press. The pressed peel was then blended with trim waste and placed in a freezer. The peeled potatoes were cut into slabs and spray-washed with cold water to remove free starch. The slabs were then pre-cooked at 70–77 °C in a steam-jacketed kettle. The pre-cooked slabs were cooled to less than 32 °C. A small amount of the cooled slabs was removed and steam-cooked at atmospheric pressure at 94–100 °C for 40 minutes. The cooked potato slabs were then mashed and fed into a dryer to produce a thin sheet, which was initially broken into large flakes by hand. The flakes were then fed into a hammermill for uniform milling of the flakes.

Samples were stored frozen for up to 8 months before analysis for flumioxazin using method RM 30A-2 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 77–118% (tubers) and 98–111% in the processed commodities. The validated LOQ was 0.02 mg/kg.

Table 88 Residues in potatoes and processed commodities from a supervised trial in the USA involving one broadcast pre-emergent soil application of flumioxazin (WG formulation)

POTATO COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	

POTATO COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2001 Prosser, WA (Russet Burbank)	1	0.141	149	0.141	Tuber wet peel chips flakes	107	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0091 WA*07

Maize

In a supervised trial on maize conducted in the USA and reported by Kowalsky, 2007 [Ref: SBR-0078] one broadcast soil application of 0.536 kg ai flumioxazin/ha (WG formulation) with added surfactant was applied 7 days before sowing, using a tractor-mounted boom sprayer (six nozzles).

Duplicate samples of kernels (min 230 kg) were taken at maturity, frozen within 2 hours and stored for up to 4 months before processing (11 months for processing into refined oil) by dry milling (to obtain grits, meal, flour and refined oil) and by wet milling (to obtain starch and refined oil).

For the dry mill processing, samples were conditioned to 21% moisture content and tempered for 2.5 hours. The kernels were cracked in a mill and corn stock from the mill was dried in an oven at 54–71 °C. Dried corn stock was screened to separate germ, bran, grits, meal and flour. The germ material was heated to 71–79 °C, flaked and triple-extracted with hexane (at 50–60 °C). The spent flakes were exposed to ambient air to remove residual hexane. The resulting fractions were miscella (crude oil and hexane) and solvent extracted germ meal. The miscella was passed through a vacuum evaporator and heated to 73–90 °C to remove hexane from the crude oil which was then mixed with sodium hydroxide in a water bath and centrifuged, decanted, filtered to produce refined oil.

For the wet mill processing, samples of kernels were steeped in 49–54 °C water containing 0.1–0.2% sulphur dioxide for 22–48 hours and passed through a disc mill and centrifuged to remove most of the germ and hulls. After drying to 5–10% moisture content, the remaining germ and hull were separated by aspiration and screening. Corn stock (without germ and hull) was ground in a disc mill, passed over a 325 mesh screen. Material on top of the screen was discarded. Process water passing through the screen was separated into starch and gluten by centrifugation. Germ samples were conditioned to 12% moisture content, heated to 88–104 °C, flaked and pressed in an expeller to liberate part of the crude oil. The presscake was double-extracted with hexane (at 50–60 °C). The spent presscake were exposed to ambient air to remove residual hexane. The resulting fractions were miscella and solvent extracted presscake (germ cake). The miscella was passed through a vacuum evaporator and heated to 73–90 °C to remove hexane from the crude oil. The expelled and extracted crude oil samples were filtered, combined and mixed with sodium hydroxide in a water bath and centrifuged, decanted, filtered to produce refined oil.

The processed samples were analysed for flumioxazin within one month using the GC-MS methods RM 30A-3 and RM-30B for the dry milled samples. Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 85–100% (kernels) and 71–117% in the processed commodities. The validated LOQs were 0.02 mg/kg.

Table 89 Residues in maize and processed commodities from a supervised trial in the USA involving one broadcast pre-plant soil application of flumioxazin (WG formulation)

MAIZE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2006 Clarence, MO (Pioneer 34B20)	1	0.536	187	0.536	Grain Starch Refined oil Grits Flour Meal Refined oil	166	< 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 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	SBR-0078 V-28566-U

Wheat

In one supervised trial on wheat conducted in the USA and reported by Kowalsky, 2011 [Ref: SBR-0092], a single foliar broadcast spray of 0.344 kg ai flumioxazin/ha (WG formulation) with added adjuvant was applied as a pre-harvest desiccant/harvest aid using an ATV-mounted boom sprayer (eight nozzles).

Duplicate samples of wheat grain (400 kg) were collected using small plot combines, frozen within 2 days after harvest and stored for up to 3.5 months before processing into bran, flour, middlings, shorts, germ, and aspirated grain fractions.

Grain samples were aspirated to remove grain dust with the materials passing through a 2360 µm sieve being collected as the aspirated grain fraction. The cleaned grain samples were adjusted to 16% moisture content, milled and passed through a 34 mesh sieve to separate the bran from the germ fraction. This bran sample was further sieved through a number of 128 µm screens, with the material passing through the screen being collected as “shorts” and the retained material was collected as “bran”. The germ fraction (with endosperm) was passed through a reduction mill and again sifted to separate the germ from the endosperm. Cleaned grain samples (conditioned by 16.5% moisture content) were also milled to crack the grains and passed through sifter screens, with material passing through a 140 µm screen being collected as “break flour”. Material passing through an 800 µm screen was collected as “middlings” and the retained material was collected as “bran”. The “middlings” sample was subjected to further milling and sieving with material passing through a 160 µm screen being collected as “reduction flour” and the retained material collected as “shorts”. The “break flour” and “reduction flour” were mixed together to produce the flour samples.

Samples were analysed for flumioxazin within 14.5 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02–0.5 mg/kg ranged from 96–114% (grain) and 79–120% in the processed fractions. The validated LOQs were 0.02 mg/kg.

Table 90 Residues in wheat grain and processed commodities from a supervised trial in the USA involving one pre-harvest foliar application of flumioxazin (WG formulation)

WHEAT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2009 Hinton, OK (Deliver)	1	0.344+MSO	152	0.344	grain bran flour middling shorts germ asp grain fraction	10	0.37, 0.35 0.35, 0.33 0.05, 0.05 0.08, 0.08 0.11, 0.1 0.38, 0.36 117, 105	0.36 0.34 0.05 0.08 0.11 0.37 111	SBR-0092 V-33037-T

Middlings = The larger particles coming from the floury part (endosperm) of the grain during milling, possibly including small bits of bran

Shorts = A low-grade mill product containing principally germ and fine bran particles, used for animal feed

Sugar cane

In one supervised trial on sugar canes conducted in the USA and reported by Schreier, 1999 [Ref: SBR-0022], a single broadcast application of 1.25 kg ai flumioxazin/ha (WG formulation) with added crop oil was applied over the top of 2–2.5 m high canes using back-pack sprayers with an extended single-nozzle hand lance.

Duplicate samples of canes were frozen within 2 days of harvest and stored for up to 2 months before being processed into refined sugar and blackstrap molasses in a way that simulated commercial practices as closely as possible. Refined sugar was obtained by chopping the cane stalks, pressing out the juice, clarifying, and concentrating the juice to syrup. Syrup, water and seed sugar were vacuum-concentrated to massecuite, which was then centrifuged to produce raw sugar and 'final' or 'blackstrap' molasses. The raw sugar was dissolved in distilled water, adjusted to a pH to 7.2 with calcium hydroxide and heated. The resulting solution was filtered, decolorized with bone char, and filtered again before boiling under vacuum to crystallize out the sugar which was centrifuged and washed with a water spray to produce refined sugar.

Samples were stored frozen for up to 2 months before analysis for flumioxazin using method RM 30C (GC-MS) and for up to 2.7 months before analysis for the 1-OH-HPA metabolite using method RM-30M (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 67–113% in the canes and 97–110% in the processed commodities. In samples fortified with 0.02–0.2 mg/kg 1-OH-HPA, recoveries were 70–114% in canes and 78–114% in the processed commodities. The validated LOQs for both compounds were both 0.02 mg/kg.

Table 91 Residues in sugar cane and processed commodities from a supervised trial in the USA involving one broadcast foliar application of flumioxazin (WG formulation)

SUGAR CANE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	no	kg ai/ha	water (L/ha)	kg ai/ha/ season			flumioxazin	mean	
USA, 1998 Spreckelsville, HI (78-4153)	1	1.263	187	1.263	cane (field) cane (bulk) molasses sugar	90	0.08, 0.09 0.11 0.055 < 0.02	0.08 0.11 0.055 < 0.02	SBR-0022 V-11945-I

Residues of 1-OH-HPA < 0.02 mg/kg in sugar cane and sugar, 0.037 mg/kg in molasses

Oilseed rape

In a supervised trial on oilseed rape conducted in the USA and reported by Stearns, 2011 [Ref: SBR 0123], one foliar broadcast spray of 0.54 kg ai flumioxazin/ha (WG formulation) was applied with added adjuvant as a pre-harvest desiccant/harvest aid using a tractor-mounted boom sprayer (seven nozzles).

Duplicate samples of seed (min 22 kg) were collected 9 days after cutting using a small plot combine, frozen within 1 hour and stored for 3 months until processed into oil and meal using simulated commercial practice. After conditioning to a moisture content of 7–10%, samples of seed were cleaned by aspiration and screening, flaked and heated to 82–90 °C, then pressed in an expeller to remove a portion of the crude oil. The residual oil in the presscake was extracted twice with hexane (50–60 °C) to produce miscella and after evaporating the remaining solvent, the resulting presscake fraction was collected as rape seed meal.

Miscella was passed through a vacuum extractor (90–96 °C) to separate the crude oil and hexane. Crude oil recovered from the expeller and solvent extraction was combined, filtered and refined by adding 85% phosphoric acid and mixing with sodium hydroxide for 20 minutes at 40–44 °C then 10 minutes at 65–70 °C. Neutralized oil was centrifuged to extract the refined oil which was then decanted, filtered, bleached (at 249 °C with bleaching earth) and deodorised with citric acid.

Samples were stored for up to 5.5 months before analysis for flumioxazin using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02–1.0 mg/kg ranged from 92–101% (seed) and from 89–108% in the processed commodities. The validated LOQs were 0.02 mg/kg.

Table 92 Residues in oilseed rape from supervised trials in the USA involving one pre-harvest foliar application of flumioxazin (WG formulation)

OILSEED RAPE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	no	kg ai/ha	water (L/ha)	kg ai/ha/ season			flumioxazin	mean	
USA, 2009 Ephrata, WA (71-45 RR)	1	0.541 + MSO	188	0.541	seed (field) seed (bulk) oil meal	5 + 9 [a]	0.6, 0.66 0.5, 0.53 < 0.02, < 0.02 0.05, 0.06	0.63 0.51 < 0.02 0.06	SBR-0123 V-32833-H

[a] Vines were cut and allowed to dry for 9 days before seeds were collected.

Cotton seed

In one supervised trial on cotton seed in the USA, reported by Schreier, 2001 [Ref: SBR-0026], two foliar broadcast sprays of 0.1–0.11 kg ai flumioxazin/ha (WG formulation) with added crop oil were applied using tractor-mounted boom sprayers. The first application was made 89 days before harvest using shielded nozzles to minimise spray contact with the plants and the second application were made 59 days before harvest as a directed inter-row spray at layby, with spray contacting only the lower 5–10 cm cotton stems.

Duplicate samples of 22 kg unginned cotton seed were frozen for up to 3 weeks before processing into cotton seed hull, meal and oil. The seed cotton samples were tower-dried, extracted (to remove burrs, sticks, and other plant parts), ginned and delinted. A huller was used to obtain the fractions kernels and hulls. The kernels were flaked and the flakes washed with hexane, dissolved and oil recovered with a precision laboratory evaporator. The oil was then refined by adding sodium hydroxide while stirring at 20–24 °C and then allowing the oil to settle at a temperature 60–65 °C. The oil was then refrigerated and filtered to obtain the refined oil and soapstock fractions.

Samples stored frozen for up to 3 months before analysis for flumioxazin using method RM 30A-1 (GC-MS) or in the case of the oil, method RM 30B (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.01 and 0.05 mg/kg ranged from 76–106% (cottonseed) and 97–135% in the processed commodities. The validated LOQs were 0.01 mg/kg.

Table 93 Residues in cotton seed and processed commodities from a supervised trial in the USA involving two inter-row soil applications of flumioxazin (WG formulation)

COTTONSEED COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1999 Ulvade, TX (PM 2326)	1	0.433	188	0.433	Seed Meal Hulls Oil	59	< 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	SBR-0026 V-20124-N

Sunflower seed

In a supervised trial on sunflower conducted in the USA and reported by Stearns, 2011 [Ref: SBR-0126], one foliar broadcast spray of 0.54 kg ai flumioxazin/ha (WG formulation) was applied with added adjuvant as a pre-harvest desiccant/harvest aid using a back-pack sprayer with a 5-nozzle boom.

Duplicate samples of seed (20 kg) were frozen within 1 hour and stored for up to 3 months before being processed into sunflower oil and meal using a procedure similar to that described above for rape seed. (Stearns, 2011; SBR-0126). Processing was done simulating commercial practices as closely as possible. The procedure was very similar to that used in the processing of oilseed rape, involving conditioning, aspiration, flaking and crude oil extraction by pressing followed by hexane double-extraction of the remaining oil from the presscake, with the combined crude oil extracts being filtered, refined by heating with phosphoric acid and sodium hydroxide and the resulting neutral oil being centrifuged, decanted bleached and deodorized with citric acid.

Samples were stored frozen for up to 10 months before analysis for flumioxazin using method RM 30A-3 (GC-MS). Concurrent recoveries from control samples fortified with flumioxazin at levels of 0.02–3.0 mg/kg ranged from 90–101% (seed) and 70–110% in the processed commodities. The validated LOQs were 0.02 mg/kg.

Table 94 Residues in sunflower seed, oil and meal from supervised trials in the USA involving one pre-harvest foliar application of flumioxazin (WG formulation)

SUNFLOWER SEED COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2009 Hinton, OK (Mycogen 8N435DM)	1	0.545+ MSO	143	0.545	seed oil meal		2.31, 2.31 < 0.02, < 0.02 0.16, 0.15	2.31 < 0.02 0.15	SBR0126 V-32835-H

MSO = Methylated seed oil

Peanut

In two supervised trials on peanuts conducted in the USA and reported by Pensyl, 1994 [Ref: SBR-0018] and Pensyl, 1996 [Ref: SBR-0019], single broadcast soil applications of about 0.53 kg ai flumioxazin/ha (WG formulations) were applied as pre-emergent broadcast sprays within 5 days after sowing, using tractor-mounted boom sprayers (6–13 nozzles).

Duplicate samples of 22–27 kg whole peanuts were collected and processed within 7 days to produce presscake, crude oil, refined oil, soapstock, bleached oil, and deodorized oil.

Peanut samples were dried and then cleaned by aspiration and screening. A sheller was used to mechanically crack the hull surrounding the kernel (nutmeat). Aspiration was used to separate the hull and kernel fractions. The raw peanut kernels were heat-conditioned and pressed in an expeller to extract most of the crude oil. After pressing, the presscake was flaked and the remaining oil was extracted from the flake with hexane. The hexane in the solvent-extracted presscake was evaporated. The crude oil recovered from the expeller and solvent extraction was combined, refined, bleached and deodorized.

Samples were kept in frozen storage up to 2.6 months before analysis for flumioxazin using method RM 30A-3 (GC-MS) and method RM 30B (GC-MS) for peanut oil. Residues of 1-OH-HPA were determined in one of these studies using method RM 30M (GC-MS) for nutmeats, hulls and presscake and method RM 30P (GC-MS) for all oil samples. Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 80–99% (nutmeat) and 72–125% (other processed commodities), and in samples spiked with the 1-OH-HPA (0.02 mg/kg) recoveries were 84% in nutmeat and 63-119% in the processed commodities. The validated LOQs were 0.02 mg/kg.

Table 95 Residues in peanuts and processed commodities from supervised trials in the USA involving one broadcast pre-plant or pre-emergent soil application of flumioxazin (WG formulations)

PEANUT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1992 Hobgood, NC (NC-7)	1	0.524	187	0.524	Whole nuts	148	< 0.02, < 0.02	< 0.02	SBR-0018 V-1040-E PREM
					Hulls		< 0.02, < 0.02	< 0.02	
					Nutmeat		< 0.02, < 0.02	< 0.02	
					Presscake		< 0.02, < 0.02	< 0.02	
					Extracted presscake		< 0.02, < 0.02	< 0.02	
					Crude oil		< 0.02, < 0.02	< 0.02	
					Extracted crude oil		< 0.02, < 0.02	< 0.02	
					Refined oil		< 0.02, < 0.02	< 0.02	
					Soapstock		< 0.02, < 0.02	< 0.02	
					Bleached oil		< 0.02, < 0.02	< 0.02	
					Deodorized oil		< 0.02, < 0.02	< 0.02	
USA, 1993 Hawkinsville, GA (Florunner)	1	0.536	215	0.536	Whole peanuts	152	< 0.02, < 0.02	< 0.02	SBR-0019 V-10716-F PREM
					Hulls		< 0.02, < 0.02	< 0.02	
					Nutmeat		< 0.02, < 0.02	< 0.02	
					Presscake		< 0.02, < 0.02	< 0.02	
					Extracted presscake		< 0.02, < 0.02	< 0.02	
					Crude oil		< 0.02, < 0.02	< 0.02	
					Extracted crude oil		< 0.02, < 0.02	< 0.02	
					Refined oil		< 0.02, < 0.02	< 0.02	
					Soapstock		< 0.02, < 0.02	< 0.02	
					Bleached oil		< 0.02, < 0.02	< 0.02	
					Deodorized oil		< 0.02, < 0.02	< 0.02	

In Trial V-10716-F, residues of 0.02 mg/kg 1-OH-HPA reported in hulls and were < 0.02 mg/kg in all other commodities

Mints

In a supervised trial on mint conducted in the USA and reported by Schreier, 2003 [Ref: SBR-0136], two foliar broadcast sprays of 0.28 or 0.42 kg ai flumioxazin/ha (WG formulations) were applied to dormant mint plants (February-April).

Duplicate samples of mint tops (leaves and stems) were processed into oil on the day of harvest and samples were stored frozen for up to 8 months before dilution with acetone and analysis for flumioxazin using method RM 30A-2 (GC-MS). Recoveries from control samples of oil fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 91–111% and the validated LOQ was 0.02 mg/kg.

Table 96 Residues in mint and mint oil from supervised trials in the USA involving two foliar applications of flumioxazin (WG formulation)

MINT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2001 Portage, WI (Peppermint)	2	0.28		0.56	leaves	112	< 0.02, < 0.02	< 0.02	SBR-0136 WI-01
					oil		< 0.02, < 0.02	< 0.02	
	2	0.42		0.84	leaves	112	< 0.02, < 0.02	< 0.02	
					oil				

Summary of Processing Studies

Processing studies on apples, plums, grapes, olives, soya beans, potatoes, sugar cane, maize, wheat, sugar cane, oilseed rape, sunflower seed, peanuts and mint were conducted, simulating commercial practices. In all cases, except for wheat, there was no concentration of flumioxazin residues in processed commodities. Except for wheat, sugar cane, oilseed rape and sunflower seed, processing factors could not be estimated because residues in the fresh commodities were below the respective method LOQs. For wheat, residues do not concentrate in wheat bran, flour, middlings, shorts, and germ. However, residues of flumioxazin concentrate by 308× in aspirated grain fractions. For rape seed, sunflower, and sugar cane, there is no concentration of flumioxazin residues in the corresponding processed fractions.

Table 97 Summary of processing factors for flumioxazin

RAC	Matrix	Flumioxazin [a]	
		Calculated processing factors	PF median
Wheat grain (0.36 mg/kg)	bran	0.94	0.94
	flour	0.14	0.14
	middling	0.22	0.22
	shorts	0.31	0.31
	germ	1.03	1.03
	aspirated grain fraction	308	308
Sugar cane 0.11 mg/kg)	molasses	0.5	0.5
	sugar	< 0.18	< 0.18
Oilseed rape seed (0.63 mg/kg)	oil	< 0.04	< 0.04
	meal	0.12	0.12
Sunflower seed 2.31 mg/kg)	oil	< 0.009	< 0.009
	meal	0.065	0.065

[a] Each value represents a separate study where residues were above the LOQ in the RAC. The factor is the ratio of flumioxazin residues in the processed item divided by the residue of flumioxazin in the RAC.

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

In a lactating cow feeding study reported by Kowalsky, 2006 [Ref: SBR-0138], three groups of dairy cattle (three cows per group, 3–7 years old and weighing 560–675 kg) were dosed orally with capsules containing flumioxazin at levels equivalent to 2, 6.2 and 19.5 ppm in the diet for 28 consecutive days (0.7 mg/kg bw/day, 0.22 mg/kg bw/day and 0.73 mg/kg bw/day respectively).

Composite milk samples from the post-dose afternoon and next morning (pre-dose) milk collections were taken at intervals during the dosing period and stored frozen for less than 30 days before analysis. On day 29, less than 24 hours after the final dosing, the animals were sacrificed and liver, muscle, kidney and fat were sampled and stored frozen for less than 30 days before analysis.

Tissue and milk samples were analysed for flumioxazin, 3-OH-flumioxazin and 4-OH-flumioxazin by HPLC-MS/MS. Residues were extracted with acetone (milk) or acetonitrile and acidic acetonitrile:water (tissues), partitioned with dichloromethane:water and the organic phases containing the residues further partitioned with acetonitrile:hexane, concentrated and diluted in methanol:water for analysis. Mean recovery rates in samples spiked with 0.02 mg/kg and 0.1 mg/kg ranged from 77–98% (flumioxazin), 84–102% (3-OH-flumioxazin and 4-OH-flumioxazin) and the LOQ was 0.02 mg/kg.

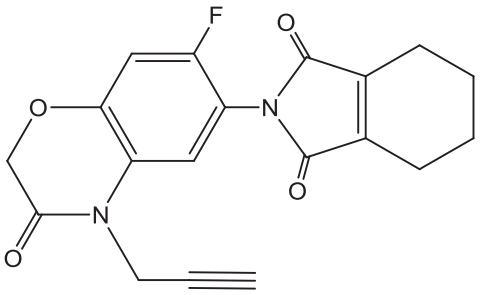
At the 19.5 ppm dose level in the feeding study, residues of flumioxazin were non-detectable (LOD of 0.01 mg/kg) in all samples of milk, skim milk, cream, liver, kidneys, muscle, and fat from all three cows. Samples from the lower dose group animals were not analysed.

APPRAISAL

Flumioxazin is a phenylthalimide protoporphyrogen oxidase inhibiting herbicide used for pre-emergent and post-emergent control of a range of broad-leaf weeds and suppression of some grass weed species in a range of fruit, vegetable and field crops.

It was scheduled by the Forty-sixth Session of the CCPR as a new compound for consideration by the 2015 JMPR. The manufacturer submitted studies on metabolism, analytical methods, supervised field trials, processing, freezer storage stability and environmental fate in soil.

Authorisations exist for the use of flumioxazin as pre-emergence or early post-emergence broadcast treatments, as directed inter-row band soil treatments and as a pre-harvest desiccant (harvest aid) treatment in North America, Europe, Latin America, Australia and some Asian countries.



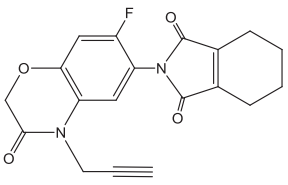
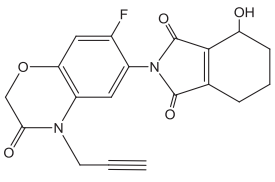
Flumioxazin

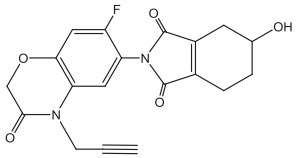
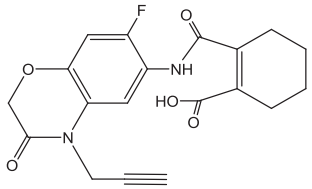
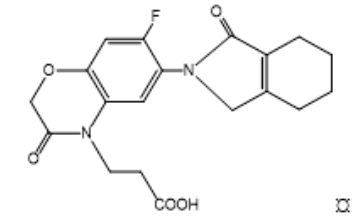
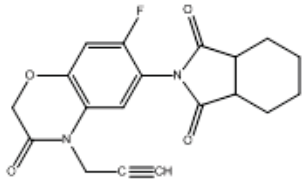
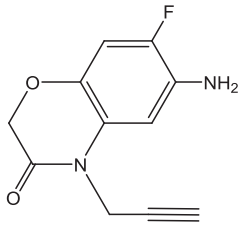
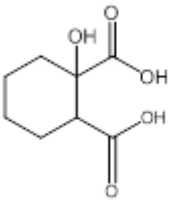
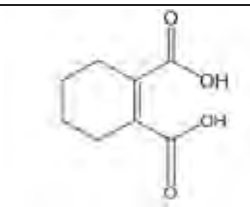
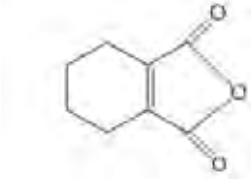
(MW 354.3)

Flumioxazin has a low vapour pressure and water solubility (approximately 0.8 mg/L) that is not pH dependent. It is soluble in medium polarity organic solvents (e.g. dichloromethane, acetone or ethyl acetate), but only slightly soluble in hexane. The octanol/water partition co-efficient (Log P_{OW} 2.55) is not pH dependent and indicates limited potential to bioaccumulation. Hydrolysis in aqueous media is pH-dependant, with half-lives ranging from 3–5 days at pH 5 to less than 25 minutes at pH 9 and the photolytic half-life is about 1 day.

The following abbreviations are used for the major metabolites discussed below:

Major flumioxazin metabolites identified in plant, animal and soil matrices.

Compound Name/Code	Structure		Matrices
Flumioxazin (S-53482) (V-53482)		*N*-(7-fluoro-3,4-dihydro-3-oxo-4-prop-2-ynyl-2*H*-1,4-benzoxazin-6-yl)cyclohex-1-ene-1,2-dicarboxamide	Plants Goat Hen Rat Soil Photolysis
3-OH-Flumioxazin		7-fluoro-6-(3-hydroxy-3,4,5,6-tetrahydrophthalimido)-4-(2-propynyl)-2H-1,4-benzoxazin -3(4H)-one	Goat Hen Rat

Compound Name/Code	Structure		Matrices
4-OH-Flumioxazin		7-fluoro-6-(4-hydroxy-3,4,5,6-tetrahydrophthalimido)-4-(2-propynyl)-2H-1,4-benzoxazin-3(4H)-one	Goat Hen Rat
482-HA		N-(7-fluoro-3,4-dihdyro-3-oxo-4-prop-2-ynyl-2H- 1,4-benzoxazin-6-yl)cyclohex-1-ene-1-carboxamide-2-carboxylic acid	Plants (rotational) Rat Soil Photolysis
482-CA		2-[7-fluoro-3-oxo-6-(3,4,5,6-tetrahydrophthalimido)-2H-1,4-benzoxazin-4-yl] propionic acid	Plants (rotational) Soil
SAT-482		6-(cis-1,2-cyclohexanedicarboximido)-7-fluoro-4-(2-propynyl)2H-1,4-benzoxazin-3(4H)-one	Goat Rat
APF		6-amino-7-fluoro-4-(2-propenyl)-2H-1,4-benzoxazin-3(4H)-one	Plants Rat Soil Photolysis
1-OH-HPA		1-hydroxy-*trans*-1,2-cyclohexanedicarboxylic acid	Plants Rat Photolysis
THPA		3,4,5,6-tetrahydrophthalic acid	Plants Goat Hen Rat Soil Photolysis
Δ^1-TPA		3,4,5,6-tetrahydrophthalic anhydride	Plants (rotational) Hen Soil Photolysis

Environmental fate

The Meeting received information on the environmental fate and behaviour of flumioxazin, including hydrolytic stability, photochemical degradation in soils and aerobic metabolism studies.

Hydrolysis

Radiolabelled flumioxazin (0.1 mg/L) incubated in the dark in sterile aqueous buffered solutions at pH 5, 7, and 9 for up to 30 days at 25 °C was rapidly hydrolysed, with calculated half-lives of about 3.4–5 days at pH 5, 19–26 hours at pH 7 and 14–23 minutes at pH 9. At pH 7, hydrolysis was biphasic, with longer half-lives of 11–14 days after the first 2–3 days.

The major degradation products after 30 days of incubation at pH 7 and pH 5 were APF (80–87% AR) and THPA (84–96% AR). At pH 9, the major degradate was 482-HA (96–99% AR).

Photochemical degradation in soil

In a photochemical degradation study in a sandy loam soil, unextracted residues in the phenyl-label study increased from an initial 3% AR to 43% AR by Day 6 and were significantly lower in the THP-label study, up to 9.3% AR on day 14. Volatiles did not exceed 0.5% of the applied radiocarbon for the irradiated samples or 0.2% for the dark controls.

Flumioxazin accounted for 97–99% AR in the day-0 samples, decreasing in the irradiated samples to 29% (Day 6—phenyl-label) and 82% AR (Day 7—THP-label) and to 37% AR in the THP-label samples on day 14. The only significant degradates identified at more than 10% AR were Δ^1-TPA and THPA.

Levels of Δ^1-TPA peaked at 22% AR on Day 9 in the irradiated samples, but were < 10% AR at all other sampling times. THPA reached a maximum of about 13% AR (9% AR in the dark control samples) at the end of the 14-day study period.

The calculated photolytic soil degradation half-lives were 3.2 days (phenyl-label study) and 8.4 days (THP-label study) and were 12–16 days in non-irradiated samples.

Aerobic soil metabolism

Under aerobic conditions, unextracted or mineralised residues increased from about 6% AR to 84% AR after 91 days in the THP-label study (55% AR released as carbon dioxide) and in the phenyl-label study, increased to a plateaux level of about 77–85% AR from day 60 (6–12% AR released as carbon dioxide). Extraction efficiencies ranged from 94–102% in the two studies.

Flumioxazin residues decreased from 93–98% AR to 60–64% AR after 7 days and 7.6–12% AR by about day 60 with calculated half-lives of 12–17.5 days. Calculated DT_{90} values (FOMC) were about 51 days (phenyl-label) and 95 days (THP-label). No identified or characterized degradates accounted for more than 8% AR.

The proposed degradation pathways include hydrolysis of the parent compound to 482-HA or oxidation to 482-CA, leading to THPA (in equilibrium with Δ^1-TPA). THPA appears to be an end product that is incorporated into soil organic components or oxidized to CO_2.

In summary, flumioxazin is rapidly hydrolysed in aqueous solutions, with the cleavage products APF and THPA being the predominant degradates at pH 7. In soil it is susceptible to photochemical degradation (average DT_{50} of about 5 days) and is not persistent in soil, with an average DT_{50} of about 15 days. Aqueous hydrolysis, photochemical degradation and aerobic soil metabolism are all likely to be a significant degradation pathways.

Plant metabolism

The Meeting received information on the metabolism of [^{14}C]flumioxazin, separately labelled in the phenyl and the tetrahydrophthaloyl (THP) rings, in soya bean and peanut (pre-emergent treatments),

grape and apple (inter-row soil treatments), sugar cane (directed soil/foliar treatments) and rotational crops.

Peanut

In a metabolism study on peanuts, [^{14}C]flumioxazin was applied either as a pre-emergent broadcast soil treatment 3 days after sowing at a rate equivalent to 0.11 kg ai/ha, or as a pre-plant treatment 32 days before sowing at 0.33 kg ai/ha. Samples of mature foliage and whole peanuts were harvested from the Pre-em plots 194 days after treatment (DAT) and from the Pre-plant plots 245 days after resowing (277 DAT).

Total radioactive residues (TRR) in all matrices from the pre-emergent treatment were below 0.02 mg eq/kg (phenyl-label) and less than 0.04 mg eq/kg (THP-label). In the pre-plant treatment, TRRs were *ca.* 3× higher except for the phenyl-label hulls and the THP-label vines. Radioactive residues were generally lowest in vines (up to 0.03 mg eq/kg) and highest in hulls (up to 0.17 mg eq/kg). Nutmeat from the pre-emergent treatment contained up to 0.03 mg eq/kg and from the pre-plant treatment were up to 0.09 mg eq/kg.

Solvent extraction and more aggressive acid, base and enzyme hydrolysis were able to extract 65–77% TRR in nutmeats and hulls and more than 90% TRR in vines.

Flumioxazin residues were < 1% TRR (< 0.001 mg/kg) in hulls and vines and not detected in nutmeat. The majority of the ^{14}C-residues were found in four chromatographic regions, each of which accounted for up to 0.005 mg eq/kg in hulls and up to 0.01 mg eq/kg in nutmeat and vines except in hulls from the pre-plant treatment, where one region contained up to 0.04 mg eq/kg, mostly multiple unknown components.

Soya bean

In metabolism studies on soya beans, [^{14}C]flumioxazin was applied to soil (sandy loam) three days after sowing at rates equivalent to 0.1 kg ai/ha or 0.2 kg ai/ha. Forage and root samples were taken 53 or 70 days after treatment and samples of plants (without pods), pods, seeds and roots were harvested at maturity, 100 or 138 days after treatment.

In the 0.1 kg ai/ha treatment plots, total radioactive residues in mature seeds were less than 0.25 mg eq/kg and were found at up to 0.06 mg eq/kg (phenyl-label) and 0.33 mg eq/kg (THP-label) in pods. In immature foliage, TRRs were up to 0.05 mg eq/kg (phenyl-label) and 0.07 mg eq/kg (THP-label). Hay from immature forage contained up to 0.19 mg eq/kg (phenyl-label) and up to 0.29 mg eq/kg (THP-label). TRRs in the samples from the 0.2 kg ai/ha treatment plots were generally about twice those in the equivalent samples from the 0.1 kg ai/ha treatment plots. The higher levels of radioactivity found in the THP-label samples suggested a preferential uptake of the THP-derived cleavage products from soil.

Sequential acetone:water and acetone:HCl extractions were able to extract 60–76% TRR in hay and forage and 25–66% TRR in seeds and more aggressive extraction techniques were able to extract most of the remaining radioactivity, with about 1–4% remaining in the post-extraction solids.

Flumioxazin made up < 1.8–6.1% TRR in 53 DAT forage (< 0.01 mg/kg) and hay (< 0.03 mg/kg) and were found at trace levels (< 2.3% TRR, < 0.004 mg/kg) only in seed from the 0.2 kg ai/ha treatment in the THP-label study. Metabolite 1-OH-HPA (free or partly cellulose conjugated) was the predominant residue, making up 15–25% of the TRR in immature forage, 26–32% TRR in hay and about 38–42% TRR (0.06–0.09 mg/kg) in seed.

Apples and grapes

In metabolism studies on apples and grapes, [^{14}C]flumioxazin was applied as sprays to bare soil (1.2 m × 1.2 m loamy sand plots) surrounding the trees or vines. The apple study involved two treatments equivalent to 0.47 kg ai/ha, applied 47 days before fruit thinning and 60 days later (about

60 days before fruit maturity) with about 30 cm of tree trunks receiving direct spray. In the grape study, one treatment equivalent to 0.6 kg ai/ha was applied about 90 days before harvest.

Total radioactive residues (TRR) were extremely low in all samples analysed, up to 0.003 mg eq/kg in apples, up to 0.005 mg eq/kg in grapes and up to 0.04 mg eq/kg in grape shoots.

In the grape study, 78–92% TRR could be solvent-extracted and HPLC analysis indicated the presence of a number of metabolites, the majority of which (58% TRR) were polar in nature. In both studies, further characterization or identification of the residues was not conducted.

Sugar cane

In a metabolism study on sugar cane, [^{14}C]flumioxazin was applied at a rate equivalent to 0.48 kg ai/ha as a directed soil/foliar spray to 1.5–2 m high sugar canes prior to stem elongation (at the 6–10 leaf stage) with up to 1 m of the plants receiving direct spray. Immature sugarcane forage (leaves and canes) were sampled about a month after the application and mature canes and leaves (3–3.6 m high) were also sampled at maturity, 90 days after treatment, when the canes were 5 cm in diameter.

Total radioactive residues were 0.001–0.004 mg eq/kg in mature cane, 0.23–0.89 mg/kg in immature forage and 0.5–1.0 mg/kg in mature leaves. More than 90% TRR was able to be extracted in acetonitrile and water.

Flumioxazin was the predominant residue in immature forage and mature leaves, accounting for 81–93% TRR (up to 0.83 mg/kg and 0.92 mg/kg repectively) and 68–75% TRR in canes, but at levels below 0.003 mg/kg.

Other minor components were all < 5% TRR in imature foliage and below 10% TRR or < 0.001 mg eq/kg in mature leaves. In the post-extraction solids (PES), radioactivity was distributed into all leaf constituents including the starch, cellulose, lignin, lipids and proteins, but did not exceed 0.03 mg eq/kg in any individual PES sub-fraction, with none of the individual TLC bands containing significant residue and none corresponded to any of the reference standards.

In summary, when applied to soil prior to crop emergence or as directed treatments to soil surrounding established perennial plants, flumioxazin does not translocate or accumulate in significant concentrations in plant matrices. In general, no parent residues were found in any of the plant matrices except in soya beans and peanut hulls. Low levels of flumioxazin were found in soya bean forage and soya bean hay and trace levels were present in soya bean seed and peanut hulls. The only significant metabolite was 1-OH-HPA (free or partly cellulose conjugated), which was present at 15–25% TRR in immature soya bean forage, and about 38–42% TRR (0.06–0.09 mg/kg) in soya bean seed.

Following directed foliar applications to sugar canes, flumioxazin is not translocated, with only traces of radioactivity found in canes. Flumioxazin accounted for more than 90% of the TRR in immature leaves (30 days after treatment), more than 81% TRR in mature leaves (90 days after treatment) and up to 75% TRR (up to 0.003 mg/kg) in canes.

Rotational crops

Two confined rotational crop studies using lettuce, carrots and wheat as rotational crops planted in bare sandy loam soil, were treated at rates equivalent to 0.105 kg ai/ha or 0.21 kg ai/ha. The rotational crops were planted 30 days after treatment in all plots and 120, 180 and 365 days after treatment in the higher treatment plots.

Radioactive residues were only detected in small amounts in all rotational crops at all plant-back intervals, with the highest radioactivity being 0.13 mg eq/kg in the straw from wheat planted 120 days after treatment with the THP-label. In the phenyl-label study, TRRs decreased in the longer plant-back intervals but in the THP-label study, TRRs increased in some

commodities at the 120-day and 180-day plant-back intervals, suggesting that THP-derived cleavage products in soil are either more readily assimilated by the plants or less tightly bound to soil than those from the phenyl label.

In the soil the majority of the radioactivity stayed at the upper 0–10 cm layer, with flumioxazin accounting for the majority of the extracted residue in most samples.

From 47–84% TRR was able to be solvent-extracted (including refluxing with acetonitrile:0.25N HCl) from wheat forage, straw and chaff, lettuce, carrot tops and roots, with 5–12% TRR being extracted from wheat grain.

Flumioxazin residues were present at less than 0.01 mg/kg in all matrices except wheat straw where levels of 0.03 mg/kg were found in the 120-day plant-back treatment. The only identified metabolites found above 10% TRR were 1-OH-HPA, THPA, and Δ^1-TPA each found at up to 15% TRR (but below 0.004 mg/kg eq) in wheat straw from the 120-day and 180-day PBI plots.

In summary, radioactive residues in rotational crops planted 30–365 days after bare soil treatments with [^{14}C]flumioxazin were low, less than 0.05 mg eq/kg in all matrices except wheat straw, where THP-labelled radioactivity was present at up to 0.13 mg eq/kg, 40% of which was flumioxazin.

The Meeting concluded that since the application rates in the rotational crop studies generally covered the range of GAP treatment rates for annual crops, residues are not expected in rotational crops following treatments according to the GAPs under consideration.

Animal metabolism

The Meeting received information on the metabolism of [^{14}C]flumioxazin, separately labelled in the phenyl and the tetrahydrophthaloyl (THP) rings, in rats, lactating goats and laying hens.

The metabolism of flumioxazin in rats was evaluated by the WHO Core Assessment Group of the 2015 JMPR. Excretion of radioactivity was rapid, with 69–87% being eliminated in urine and faeces within 24 hours with the remainder found mainly in excretory organs. Flumioxazin was extensively metabolized (29–35 metabolites detected and quantified), with 7–10 of these being identified. Flumioxazin accounted for 47–66% of the administered dose in the 100 mg/kg bw dose group and up to 2% in the 1 mg/kg bw dose group. Metabolites found at more than 5% of the applied dose were 3-OH-flumioxazin, 3-OH-flumioxazin-SA, 4-OH-flumioxazin and 4-OH-flumioxazin-SA.

Lactating goats were orally dosed with [^{14}C]flumioxazin at doses equivalent to 11.8 ppm (phenyl-label) and 7.2 ppm (THP-label) in the feed for 5 consecutive days and sacrificed 6 hours after the last dose.

The majority of the radioactivity (80–93% AR) was found in urine, faeces or the GI tract, with < 1% AR remaining in tissues and 0.22% AR in milk. Radioactivity was extremely low in fat (up to 0.008 mg/kg), low in muscle, up to 0.014 mg/kg (phenyl-label) and 0.028 mg/kg (THP-label), but higher in liver, up to 0.21 mg/kg (phenyl-label) and 0.33 mg/kg (THP-label). In kidney the radioactive residues were up to 0.18 mg/kg (phenyl-label) and 0.24 mg/kg (THP-label). The average total radioactivity concentration in milk plateaued around Day 3 at about 0.04 mg/kg (phenyl-label) and about 0.06 mg/kg in the THP-label study.

More than 80% TRR from milk, liver and kidney and 58–74% TRR from muscle was able to be solvent-extracted. TRR in fat were not investigated further.

The parent compound was extensively metabolized, with residues above 0.001 mg/kg found only in liver (up to 0.01 mg/kg and < 5% TRR).

The 4-OH-flumioxazin metabolite accounted for up to 14% TRR in kidney (up to 0.025 mg/kg) and muscle (up to 0.003 mg/kg). In liver, both the 4-OH-flumioxazin and 3-OH-flumioxazin residues did not exceed 0.025 mg/kg (about 9% TRR).

Metabolite 482-HA was the predominant component in milk (14% TRR) but absolute levels were below 0.005 mg/kg eq and it was also found in liver and kidney at close to 10% TRR, 0.02 mg/kg).

Metabolite B, tentatively identified as 3- or 4-OH-SAT-482, made up about 14% TRR (0.024 mg/kg) in kidney and 18% TRR in milk (0.005 mg/kg). In liver, metabolite F, tentatively identified as an isomer of 3- or 4-OH-SAT-482, made up about 11% TRR (0.03 mg/kg).

In muscle, metabolite C accounted for 20–23% TRR and 12% TRR in milk but absolute levels were all below 0.005 mg/kg.

Laying hens were orally dosed with [^{14}C]flumioxazin (phenyl-label or THP-label) at doses equivalent to 10 ppm in the feed for 14 consecutive days and sacrificed 4 hours after the last dose (in order to ensure sufficient radiolabel remained to allow further investigation).

Radioactivity in the excreta, GI tract contents and cage wash accounted for 83–94% AR, with liver, kidney, muscle, fat, skin and eggs contained relatively small amounts of radioactivity (totalling < 0.6–0.9% of the administered dose). Radioactivity in egg yolks accounted for 0.35–0.36% AR, with < 0.01% AR in the corresponding egg whites. Liver contained 0.08–0.27% AR (0.24 mg/kg eq and 1.14 mg/kg eq) in the phenyl-label study and the THP-label study respectively. In egg yolks, residues reached a plateau of 0.4–0.6 mg/kg eq by Day 10 or 11 in the two studies.

More than 87% TRR in eggs was extracted with methanol or ethanol, and acetonitrile was able to extract 37–67% TRR from muscle. In the phenyl-label liver and kidney samples, sequential extractions with acetonitrile and bicarbonate were able to extract more than 90% TRR and further enzyme extraction released an additional 10% TRR. In the THP-label liver and kidney samples, sequential acetonitrile and acetonitrile:water extractions were able to extract 80–87% TRR.

In solvent-extracted samples, the parent compound was the predominant residue in fat (49% TRR), skin + fat (12–25% TRR), muscle (10–14% TRR), a significant component in liver and kidney (7–9% TRR), made up about 4–9% TRR in egg yolk and was not detected in egg white. Absolute levels of flumioxazin were up to 0.13 mg/kg in skin + fat and fat, < 0.08 mg/kg in liver and kidney, < 0.04 mg/kg in egg yolk and about 0.02 mg/kg in muscle.

Metabolites present at more than 10% TRR or more than 0.01 mg/kg were 4-OH-flumioxazin, 3-OH-flumioxazin and 4-OH-flumioxazin-SA.

The 4-OH-flumioxazin accounted for 9–12% TRR in all tissues (< 0.03 mg/kg in muscle and fat, < 0.1 mg/kg in kidney and skin + fat and 0.12 mg/kg in liver) while the 3-OH-flumioxazin accounted for 8–12% TRR (0.015 mg/kg) in muscle. Metabolite 4-OH-flumioxazin-SA accounted for 32% TRR in egg yolk (0.14 mg/kg).

All other identified metabolites were found at < 8% TRR and the highest level of any single unidentified metabolite was measured in liver, at 12% TRR.

In summary, in the ruminant and poultry metabolism studies, flumioxazin is extensively metabolized with limited transfer into tissues, eggs or milk (less than 0.5% of the administered dose). Flumioxazin was not found at levels above 0.01 mg/kg in goat milk or tissues but was present in most poultry commodities, highest residues being found in fat (0.13 mg/kg, 49% TRR), with lower levels (up to 0.08 mg/kg) in other tissues and egg yolks.

Other metabolites present at more than 10% TRR in various commodities were 4-OH-flumioxazin, 4-OH-flumioxazin-SA, 3-OH-flumioxazin, metabolite B, tentatively identified as 3- or 4-OH-SAT-482 and metabolite F, tentatively identified as an isomer of metabolite B.

Analytical methods

Several analytical methods have been reported and validated for the analysis of flumioxazin in plant and animal commodities. The basic approach employs extraction with acetone/water or

hexane/acetonitrile, partitioning into dichloromethane and/or acetonitrile, Florisil or silica gel clean-up and analysis by GC-MS. For processed plant oils, the initial acetone extraction and dichloromethane partitioning steps are omitted and for animal commodities the dichloromethane partitioning step is also omitted. The LOQs for these methods is 0.02 mg/kg.

Two methods have also been validated for measuring residues of the 1-OH-HPA metabolite (free and conjugated) in some food and feed commodities. Residues are extracted using acid hydrolysis, partitioned into ethyl acetate and refluxed for 30 minutes with acetone, triisopropanolamine and dimethyl sulphate to convert the 1-OH-HPA to its dimethyl ester. After partitioning into hexane and Florisil column clean-up, residues are analysed by GC/MS. The LOQs for the method range from 0.02–0.1 mg/kg.

A more recent HPLC-MS/MS method was reported in the lactating cow feeding study for measuring residues of flumioxazin, 3-OH-flumioxazin and 4-OH-flumioxazin. Tissue samples are extracted in acetonitrile and acidic acetonitrile:water and milk samples are extracted with acetone. The extracts are then partitioned with dichloromethane/water and the organic phase further partitioned with acetonitrile/hexane. Analysis for flumioxazin, 3-OH-flumioxazin and 4-OH-flumioxazin was by HPLC-MS/MS (flumioxazin: m/z 355→299, 3-OH-flumioxazin: m/z 371→299/107 and 4-OH-flumioxazin: m/z 371 →299/107) with an LOQ of 0.02 mg/kg.

For plant and processed plant commodities, the DFG S19 (GC-MS) method was validated for the analysis of flumioxazin in cereals, potatoes and oily substrates (sunflower seeds). After extraction with aqueous acetone and partitioned into ethyl acetate/cyclohexane, extracts are cleaned-up by gel permeation chromatography and residues are determined by GC-MS. The LOQ is 0.02 mg/kg. Recovery rates ranged from 84–102% for all analytes in all matrices.

The Meeting concluded that suitable methods are available to measure flumioxazin in plant and animal commodities.

Stability of pesticide residues in stored analytical samples

Flumioxazin residues were stable in analytical samples stored frozen (–18 to –20 °C) for at least the storage intervals used in the supervised residue trials, with residues in the stored samples usually more than 80% of the spiked sample levels. In general, residue stability was shown for up to:

26–30 months	non-bell peppers, alfalfa (forage, hay)
12–18 months	maize (forage, grain, stover), olives, summer squash, olive oil, soya bean (forage, hay)
9–12 months	celery, cherries, cotton seed, soya bean seed, peanut (forage, hay, hulls, nutmeat), mint, potatoes (fresh and processed)
6–9 months	apple (juice, wet pomace), globe artichoke, asparagus, cabbage, cucumber, tomato, almond (nutmeat, hulls), mint oil, strawberry, grape (fresh, dried)
2–6 months	onions, cottonseed (meal, hulls gin trash), blueberries, melons, pecans, grape juice, sugarcane, molasses and refined sugar.

Definition of the residue

When flumioxazin is applied to soil prior to crop emergence or as directed treatments to soil surrounding established plants, flumioxazin does not translocate or accumulate in significant concentrations in plant matrices. In general, no parent or identifiable metabolites are found in the plant matrices except in soya beans, where low levels of flumioxazin (below 0.01 mg/kg) were found in forage and seeds, and up to 0.03 mg/kg in hay from immature forage.

The only significant metabolite in plant commodities following pre-emergence treatment is 1-OH-HPA (free or partly cellulose conjugated), present at 15–25% TRR in immature soya bean forage, and about 38–42% TRR (0.06–0.09 mg/kg) in soya bean seed. However, in supervised field trials on soya beans, residues of this metabolite were all below the LOQ (0.02 mg/kg) and the Meeting concluded that 1-OH-HPA need not be included in the residue definition for dietary intake estimation.

Following directed foliar applications, flumioxazin is not translocated, with the majority of the residue in sugar cane leaves about 1 month after treatment being the parent. The Meeting concluded that this would also be the case where flumioxazin was used as a pre-harvest treatment to scenescing plants.

In confined crop rotation studies, radioactive residues in rotational crops planted 30–365 days after bare soil treatments were low, generally less than 0.01 mg/kg eq in all matrices except wheat straw, where flumioxazin was found at up to 0.03 mg/kg in straw from wheat planted 120 days after treatment with 0.21 kg ai/ha (2× GAP).

Based on the above, the Meeting considered that a suitable residue definition for plant commodities would be flumioxazin (parent only), both for MRL-compliance and dietary intake estimation.

In animal commodities, metabolism studies in goats and poultry indicate that flumioxazin is almost completely excreted, with < 1% of the applied radioactivity remaining in milk, eggs and tissues after 6 hours. In animals dosed with about 7–10 ppm flumioxazin in the diet, residues of parent compound were below 0.01 mg/kg in goat milk and tissues, but were higher in poultry, being the predominant identified residue, found at up to 0.13 mg/kg (49% TRR) in poultry fat and up to 0.08 mg/kg in other tissues and egg yolks.

Identified metabolites found above 10% TRR and above 0.01 mg/kg in various matrices were 4-OH-flumioxazin and 3-OH-flumioxazin and 4-OH-flumioxazin-SA (only in egg yolk).

In the animal metabolism studies, metabolites 3-OH-flumioxazin and 4-OH-flumioxazin were present at up to 15% TRR in most tissues from animals sacrificed 6 hours after the last dose. However in the dairy cow feeding study, these metabolites were not found in milk or tissues from animals sacrificed 24 hours after dosing at about 2–3× the dose used in the goat metabolism study. The Meeting concluded that because of the short interval to sacrifice, the animal metabolism studies over-estimated the expected residues in cattle and noted that no detectable residues of parent or metabolites are expected in poultry. Since safety concerns with 3-OH-flumioxazin or 4-OH-flumioxazin are not anticipated, the Meeting agreed they need not be included in the residue definitions.

The Meeting noted that 4-OH-flumioxazin-SA was not a significant residue in any matrix except egg yolk and that the calculated dietary burden (0.57 ppm) was about 0.04% of the dose rate used in the metabolism study. The Meeting therefore considered that 4-OH-flumioxazin-SA need not be included in the residue definition for dietary intake estimation.

The Meeting noted that a multi-residue method exists to measure parent residues in plant commodities and that the analytical method used in the goat feeding study was able to measure both the parent compound and the 3-OH-flumioxazin and 4-OH-flumioxazin metabolites.

The Meeting agreed that for MRL-compliance and dietary intake estimation for plant and animal commodities the residue definitions should be flumioxazin.

The Meeting noted that the octanol/water partition coefficient (Log P_{ow}) for flumioxazin was 2.55, and while the information on the relative distribution of flumioxazin in fat/muscle and egg yolk/egg white was limited, the Meeting concluded that the residue was not fat soluble.

Proposed definition of the residue (for compliance with the MRL and estimation of dietary intake for plant and animal commodities): *flumioxazin.*

The residue is not fat-soluble.

Results of supervised residue trials on crops

The Meeting received supervised trial data for flumioxazin applied as pre-emergence or early post-emergence broadcast treatments on a range of vegetable and field crops, as directed inter-row band soil treatments on a number of fruit crops and as a pre-harvest desiccant (harvest aid) treatment on several pulse and cereal crops. These trials were conducted in North America.

Where residues have been reported in the studies as being not quantifiable, the values have been considered as < LOQ for the purposes of MRL setting

Perennial crops

The critical GAP for pome fruit, stone fruit, bush berries, grapes, olives, pomegranates and tree nuts in the USA is for soil treatments of up to 0.42 kg ai/ha as directed band sprays under the crop canopy, avoiding contact with trunks or vines, with a maximum seasonal rate of 0.82 kg ai/ha, a retreatment interval of at least 30 days and a PHI of 60 days (7 days for bush berries).

In more than 60 independent trials on these crops conducted in the USA and matching the USA GAP, flumioxazin residues in the fruit and nutmeat were all < 0.02 mg/kg.

The Meeting noted that when applied to soil, flumioxazin remained predominantly in the upper 10 cm layer and was not persistent or root-absorbed. In the grape and apple metabolism studies where the treatments reflected the above GAP, total radioactivity levels in the fruit were extremely low (< 0.005 mg eq/kg).

The Meeting therefore agreed to estimate maximum residue limits of 0.02(*) mg/kg for flumioxazin on pome fruit, stone fruit, bush berries, grapes, olives, pomegranate and tree nuts.

The Meeting also agreed that as no flumioxazin residues are to be expected in mature fruit at harvest, STMRs and HRs could be established at 0 mg/kg for these fruit and nut commodities.

Strawberry

The critical GAP for strawberries in the USA is for soil treatments of up to 0.105 kg ai/ha as a shielded inter-row band spray (avoiding contact with fruit or foliage) applied up to fruit set, with a maximum seasonal rate of 0.105 kg ai/ha.

Trials on strawberries conducted in the USA involved one directed inter-row soil application, 1–2 days before harvest, with a previous broadcast soil application to dormant strawberries in some of these trials.

The Meeting agreed that these trials did not match the USA GAP. No maximum residue level for strawberries was estimated.

Bulb vegetables

Results from supervised trials on bulb onions conducted in the USA were provided to the Meeting.

Onion, dry bulb

The critical GAP for bulb onions in the USA is for broadcast soil/foliar treatments of up to 0.07 kg ai/ha to onions between the 2-leaf and 6-leaf stage, with a maximum seasonal rate of 0.105 kg ai/ha.

In nine independent trials on bulb onions conducted in the USA where two broadcast applications of 0.1–0.115 kg ai/ha were applied at or about the 2-leaf stage and 29–78 days later (42–49 days before harvest), residues in the dry bulbs were all < 0.02 mg/kg.

The Meeting noted that since residues were all < LOQ in these supervised trials with application rates higher than specified in the USA GAP, the data could be used to estimate a maximum residue level.

The Meeting estimated an STMR of 0 mg/kg, and HR of 0 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on onion, bulb.

Garlic

The critical GAP for garlic in the USA is for one pre-emergent broadcast soil application of up to 0.21 kg ai/ha, no later than 3 days after planting. No trials matching this GAP were provided and no maximum residue level for garlic was estimated by the Meeting.

Cabbage, head

Results from supervised trials on head cabbages conducted in the USA were provided to the Meeting.

The critical GAP for head cabbages in the USA is for inter-row soil treatments of up to 0.14 kg ai/ha between raised plastic-mulched beds up to just before transplanting, with a maximum seasonal rate of 0.28 kg ai/ha.

In seven independent trials on head cabbages conducted in the USA where one broadcast soil application of 0.1–0.11 kg ai/ha was applied just before transplanting, residues in cabbage heads (with wrapper leaves) were all < 0.02 mg/kg.

Although the broadcast treatment method used in the supervised trials did not match the USA GAP for inter-row applications just before transplanting, the Meeting agreed that since the use directions specified treatment only to the row middles between raised plastic mulched beds that are at least 60 cm wide and since the broadcast treatment method represented the worst-case situation, the data set (all < LOQ) could be used to estimate a maximum residue level and that the STMR and HR could be established at 0 mg/kg as no flumioxin residues would be expected in mature cabbages at harvest.

The Meeting estimated an STMR of 0 mg/kg, an HR of 0 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on cabbages, head.

Fruiting vegetables, Cucurbits

Results from supervised trials on outdoor cucumbers, summer squash and melons (cantaloupes) conducted in the USA were provided to the Meeting.

The critical GAP for cucurbit vegetables in the USA is for inter-row soil treatments of up to 0.14 kg ai/ha between raised plastic-mulched beds up to 14 days before planting with an option to apply an additional inter-row soil treatment up to 21 days after transplanting/emergence but before the start of flowering, with a maximum seasonal rate of 0.28 kg ai/ha.

In six independent trials matching the GAP in the USA, residues of flumioxazin in cucumbers were all < 0.02 mg/kg.

In seven independent trials matching the GAP in the USA, residues of flumioxazin in summer squash were all < 0.02 mg/kg.

In eight independent trials matching the GAP in the USA, residues of flumioxazin in melons were all < 0.02 mg/kg.

Based on the combined results of the cucumber, summer squash and melon trials, with residues of < 0.02 (n=21), the Meeting agreed to consider establishing a group maximum residue level for fruiting vegetables, cucurbits.

The Meeting estimated an STMR of 0.02 mg/kg, an HR of 0.02 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on fruiting vegetables, cucurbits.

Fruiting vegetables, other than Cucurbits

Results from supervised trials on outdoor tomatoes, sweet peppers and chilli peppers conducted in the USA were provided to the Meeting.

The critical GAP for fruiting vegetables in the USA is for inter-row soil treatments of up to 0.14 kg ai/ha between raised plastic-mulched beds up to 14 days before planting with an

option to apply an additional inter-row soil treatment up to 21 days after transplanting/emergence but before the start of flowering, with a maximum seasonal rate of 0.28 kg ai/ha.

In seven independent trials matching the GAP in the USA but with the last application 15–21 days before harvest, when immature fruit were present, residues of flumioxazin in tomatoes were all < 0.02 mg/kg.

In nine independent trials on sweet peppers (6) and chilli peppers (3) matching the GAP in the USA but with the last application 15–21 days before harvest, when immature fruit were present, residues of flumioxazin in peppers were all < 0.02 mg/kg.

Although the timing of the last application in the supervised trials did not match the USA GAP for use up to the start of flowering, the Meeting agreed that the later applications (when fruitlets were present) represented a worst-case situation and that since residues were all < LOQ, the data set could be used to estimate a maximum residue level.

Based on the combined results of tomato, sweet pepper and chilli pepper trials, with residues of < 0.02 (16), the Meeting agreed to consider establishing a group maximum residue level for fruiting vegetables, other than cucurbits.

The Meeting estimated an STMR of 0.02 mg/kg, an HR of 0.02 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on fruiting vegetables, other than cucurbits (except sweetcorn and mushrooms).

Pulses

Results from supervised trials on dry beans, dry peas and soya beans conducted in North America were provided to the Meeting.

Beans (dry)

In the USA, the critical GAP for beans, dry is for a broadcast foliar application of up to 0.105 kg ai/ha as a harvest aid (desiccant) up to 5 days before harvest.

In 10 independent trials matching the GAP in the USA, residues of flumioxazin in dry bean seeds were < 0.02 (5), 0.02, (4), and 0.05 mg/kg.

The Meeting noted that the GAP in the USA for dry beans includes lupins, chickpeas and lentils, and agreed to extrapolate the data for dry beans to these commodities.

The Meeting estimated a maximum residue level of 0.07 mg/kg and an STMR of 0.02 mg/kg for flumioxazin on beans (dry), lupins (dry), chickpeas (dry) and lentils (dry).

Peas (dry)

The critical GAP for field peas in the USA is for a broadcast foliar application of up to 0.105 kg ai/ha as a harvest aid (desiccant) up to 5 days before harvest.

In 13 independent trials matching the GAP in the USA, residues of flumioxazin in dry pea seeds were < 0.02 (8), 0.02, 0.02, 0.03, 0.03 and 0.06 mg/kg. (Highest residue of duplicate samples = 0.07 mg/kg)

The Meeting estimated an STMR of 0.02 mg/kg and a maximum residue level of 0.07 mg/kg for flumioxazin on peas (dry).

Soya bean (dry)

The critical GAP for soya beans in the USA is for pre-plant or pre-emergent broadcast soil applications of 0.105 kg ai/ha (up to 3 days after sowing), with a maximum seasonal rate of 0.105 kg ai/ha.

In 39 independent trials matching the GAP in the USA, residues of flumioxazin in soya bean seeds were all < 0.02 mg/kg. In one processing study involving an exaggerated rate of 0.54 kg ai/ha, residues in soya bean were also < 0.02 mg/kg.

The Meeting estimated an STMR of 0 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on soya bean (dry).

Root and tuber vegetables

Results from supervised trials on potatoes conducted in the USA were provided to the Meeting.

Potato

The critical GAP for potatoes in the USA is for broadcast soil applications of up to 0.053 kg ai/ha, after planting (hilling) but before crop emergence, with a maximum seasonal rate of 0.053 kg ai/ha.

In 11 independent trials conducted in the USA, flumioxazin residues in tubers were all < 0.02 mg/kg following one pre-emergence application of 0.13–0.15 kg ai/ha.

The Meeting noted that since residues were all < LOQ in these supervised trials with application rates higher than specified in the USA GAP, the data could be used to estimate a maximum residue level and would support an STMR and HR of 0 mg/kg.

The Meeting estimated an STMR of 0 mg/kg, an HR of 0 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on potato.

Sweet potato

The Meeting noted that GAP also existed in the USA for the use of flumioxazin on sweet potato as a broadcast soil application of up to 0.105 kg ai/ha prior to transplanting and agreed that the results of the USA potato trials, matching this GAP could be used to estimate a maximum residue level for sweet potatoes.

The Meeting estimated an STMR of 0 mg/kg, an HR of 0 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on sweet potato.

Stem and stalk vegetables

Results from supervised trials on asparagus, globe artichoke and celery conducted in North America were provided to the Meeting.

Artichoke, Globe

The critical GAP for globe artichokes in the USA is for broadcast pre-plant or pre-emergence soil applications of up to 0.21 kg ai/ha, with a maximum seasonal rate of 0.21 kg ai/ha.

In three independent trials matching the pre-plant GAP in the USA, flumioxazin residues in artichoke heads were all < 0.02 mg/kg.

The Meeting estimated an STMR of 0.02 mg/kg, an HR of 0.02 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on artichoke, Globe.

Asparagus

The critical GAP for asparagus in the USA is for broadcast soil applications of up to 0.21 kg ai/ha not later than 14 days before spear emergence, with a maximum seasonal rate of 0.21 kg ai/ha.

In eight independent trials matching the GAP in the USA, flumioxazin residues in spears were all < 0.02 mg/kg.

The Meeting noted that in these trials, residues were all < LOQ in the 2× plots, and agreed that the data would support an STMR and HR of 0 mg/kg.

The Meeting estimated an STMR of 0 mg/kg, an HR of 0 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on asparagus.

Celery

The critical GAP for celery in the USA is for broadcast soil applications of up to 0.105 kg ai/ha, 3–7 days after transplanting), with a maximum seasonal rate of 0.105 kg ai/ha.

No trials matched this broadcast post-transplanting GAP in the USA and no maximum residue level for celery was estimated by the Meeting.

Cereal grains

Results from supervised trials on maize and wheat conducted in North America were provided to the Meeting.

Maize

The critical GAP for maize in the USA is for broadcast soil applications of up to 0.105 kg ai/ha applied from 30 to 14 days before sowing, with a maximum seasonal rate of 0.105 kg ai/ha.

In 21 independent trials matching the GAP in the USA, with pre-planting intervals of 6–14 days, flumioxazin residues in maize grain were all < 0.02 mg/kg.

The Meeting noted that in three of these trials and in the processing study involving exaggerated application rates, residues were also < LOQ, and agreed that the data would support an STMR of 0 mg/kg.

The Meeting estimated an STMR of 0 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on maize.

Wheat

The critical GAP for wheat in the USA is for a broadcast foliar application of up to 0.07 kg ai/ha as a harvest aid (desiccant) up to 10 days before harvest.

In 20 independent trials matching the GAP in the USA, residues of flumioxazin in wheat grain were 0.05 (4), 0.06, 0.06, 0.07, 0.08, 0.09, 0.1 (3), 0.11, 0.11, 0.12, 0.13, 0.13, 0.18, 0.23 and 0.31 mg/kg.

The Meeting estimated an STMR of 0.1 mg/kg and a maximum residue level of 0.4 mg/kg for flumioxazin on wheat.

Sugar cane

Results from supervised trials on sugar cane conducted in the USA were provided to the Meeting.

The critical GAP for sugar cane in the USA is for directed inter-row soil/stem band applications of up to 0.14 kg ai/ha after the canes are 60 cm in height or at layby (when canes are more than 76 cm in height), with a minimum 14-day retreatment interval, a maximum seasonal use of 0.42 kg ai/ha and a PHI of 90 days. The label also states that the spray solution must not contact foliage above 15 cm from the base of cane.

The Meeting noted that the supervised trials did not match the GAP in the USA, as they involved single foliar treatments applied over the top of the canes. No maximum residue level for sugar cane was estimated by the Meeting.

Oilseeds

Results from supervised trials on oilseed rape, cottonseed, sunflower seed and peanuts conducted in the USA were provided to the Meeting.

Cotton seed

The critical GAP for cotton seed in the USA is for directed inter-row band soil treatments of up to 0.07 kg ai/ha after cotton has reached 15 cm in height or at layby (when plants are more than 40 cm in height), with a maximum seasonal rate of 0.14 kg ai/ha, a retreatment interval of at least 30 days and a PHI of 60 days.

In 12 independent trials on cotton, involving higher application rates of 0.1–0.12 kg ai/ha but otherwise matching the GAP in the USA, residues in cotton seed were < 0.01(11) and 0.01 mg/kg. The Meeting agreed to use the proportionality approach to estimate a maximum residue level by scaling these results to the 0.07 kg ai/ha rate (scaling factors of 0.63-0.67). Proportionaly adjusted residues were all < 0.01 mg/kg (n=12).

The Meeting estimated an STMR of 0.01 mg/kg and a maximum residue level of 0.01 mg/kg for flumioxazin on cotton seed.

Linseed

The critical GAP for linseed (flax seed) is in the USA, involving a broadcast foliar application of up to 0.105 kg ai/ha as a harvest aid (desiccant) up to 5 days before harvest.

No trials on linseed were available and while there were trials provided on rape seed matching the GAP for linseed in the USA, the Meeting agreed not to extrapolate these data to linseed because of the different seed-head morphologies. No maximum residue level was estimated for linseed.

Peanuts

The critical GAP for peanuts in the USA is for broadcast soil applications of up to 0.105 kg ai/ha prior to sowing or pre-emergent (up to 2 days after sowing), with a maximum seasonal rate of 0.105 kg ai/ha.

In 13 independent trials on peanuts matching the GAP in the USA, flumioxazin residues in peanut nutmeat were all < 0.02 mg/kg. In one processing study involving an exaggerated rate of 0.54 kg ai/ha, residues in nutmeat were also < 0.02 mg/kg.

The Meeting estimated an STMR of 0 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on peanut.

Sunflower seed

The critical GAP for sunflower seed in the USA is for a broadcast foliar application of up to 0.105 kg ai/ha as a harvest aid (desiccant) up to 5 days before harvest.

In eight independent trials on sunflower seed matching the GAP in the USA, residues of flumioxazin in sunflower seed were 0.04, 0.04, 0.05, 0.1, 0.12, 0.18, 0.18 and 0.29 mg/kg.

The Meeting estimated an STMR of 0.11 mg/kg and a maximum residue level of 0.5 mg/kg for flumioxazin on sunflower seed.

Mints

Results from supervised trials on fresh mints conducted in the USA were provided to the Meeting.

The critical GAP for mints (spearmint, peppermint) in the USA is for broadcast applications of up to 0.14 kg ai/ha to dormant plants in autumn and spring, with a maximum seasonal rate of 0.28 kg ai/ha, a retreatment interval of at least 60 days and a PHI of 80 days.

In three independent trials on spearmint and peppermint, involving higher application rates of 0.28 kg ai/ha but otherwise matching the GAP in the USA, residues in fresh mint leaves were all < 0.02 mg/kg. In these trials, separate plots were also treated with 0.42 kg ai/ha (3× GAP), and residues in mint leaves from these plots ranged from < 0.02–0.03 mg/kg.

The Meeting agreed that the results from the 0.42 kg ai/ha application rate, when scaled down to the GAP application rate (scaling factor of 0.5) would support an STMR and HR of 0.01 mg/kg.

The Meeting estimated an STMR of 0.01 mg/kg, an HR of 0.01 mg/kg and a maximum residue level of 0.02 mg/kg for flumioxazin on mints.

Animal feeds

Results from supervised trials on alfalfa and on animal feed commodities from almonds (hulls), cotton (gin trash), peanuts (hulls, vines and hay), soya beans (forage and hay), maize (forage and stover) and wheat (forage, hay and straw) were provided to the Meeting.

For peanuts and soya beans, the US GAP includes a condition that treated crops must not be grazed or fed to livestock, and the Meeting did not evaluate the trial results for feed commodities from these crops.

Alfalfa forage and fodder

The critical GAP for alfalfa in the USA is for broadcast foliar applications of up to 0.14 kg ai/ha in winter (after the final cut) and in spring, after the first cut, before the crop reaches 15 cm in height, with a minimum retreatment interval of 60 days, a maximum seasonal rate of 0.28 kg ai/ha and a PHI of 25 days for harvest or grazing.

In six independent trials on alfalfa involving one broadcast application 24–26 days before the first cut and a second application to regrowth 6–8 days after the first cut, flumioxazin residues in forage were 0.04, 0.1, 0.11, 0.12, 0.14 and 0.39 mg/kg (fresh weight).

In six independent trials on alfalfa involving one broadcast application to regrowth 7–9 days after the first cut, flumioxazin residues in forage were 0.03, 0.06, 0.1, 0.18, 0.23 and 0.8 mg/kg (fresh weight).

The Meeting noted that the residue populations from the single and double treatments were not statistically different, suggesting that the residue contribution from first application (prior to the foliage being cut and removed) was not significant and agreed to use the data from the single post-cutting treatment to estimate median and highest residues for estimating livestock dietary burdens.

The Meeting estimated a median residue of 0.14 mg/kg (fresh weight) and a highest residue of 0.8 mg/kg (fresh weight) for alfalfa forage.

In alfalfa hay sampled from the same trials and same PHIs but allowed to dry in the field for 2-7 days, residues were: 0.11, 0.24, 0.3, 0.46, 0.86 and 1.5 mg/kg.

The Meeting estimated, a median residue of 0.38 mg/kg (fresh weight), a highest residue of 1.5 mg/kg (fresh weight) and after correcting for an average 89% dry matter, estimated a maximum residue level of 3.0 mg/kg (dry weight)for flumioxazin on alfalfa fodder.

Almond hulls

In five independent trials on almonds matching the inter-row soil band treatment GAP in the USA, residues in almond hulls were < 0.01, 0.01, 0.04, 0.06 and 0.55 mg/kg.

The Meeting noted that residues in perennial fruit and nuts are not expected following the use of flumioxazin as an inter-row soil band treatment, and that while in these trials, no residues were present in almond nutmeat, the levels reported in hulls were likely to have arisen from contamination at harvest when the nuts were shaken from the tree and picked up off the ground.

The Meeting estimated a median residue of 0.04 mg/kg for flumioxazin on almond hulls.

Cotton gin trash

In seven independent trials on cotton, involving higher application rates of 0.1–0.12 kg ai/ha but otherwise matching the GAP in the USA, residues in cotton gin trash were < 0.01, 0.03, 0.04, 0.16, 0.24, 0.25 and 0.48 mg/kg. When proportionally adjusted to the 0.07 kg ai/ha GAP application rate (scaling factor of 0.65), the scaled residues are < 0.01, 0.02, 0.03, 0.1, 0.15, 0.16 and 0.31 mg/kg.

The Meeting estimated a median residue of 0.1 mg/kg and a highest residue of 0.31 mg/kg for flumioxazin on cotton gin trash.

Maize forage and fodder

In 21 independent trials matching the pre-plant broadcast soil application GAP in the USA, flumioxazin residues in maize forage sampled at the late dough/early dent growth stage (BBCH 86) were all < 0.02 mg/kg.

The Meeting estimated a median residue of 0 mg/kg and a highest residue of 0 mg/kg for flumioxazin on maize forage.

In maize fodder (stover) sampled from the same 21 trials at grain maturity, flumioxazin residues were all < 0.02 mg/kg.

The Meeting estimated a maximum residue level of 0.02* mg/kg (dry weight), a median residue of 0 mg/kg and a highest residue of 0 mg/kg for flumioxazin on maize fodder.

Wheat forage and hay

The critical GAP for wheat grown for forage or hay in the USA is for a pre-plant or pre-emergence broadcast soil application of up to 0.07 kg ai/ha, with no grazing until the wheat is at least 13 cm high.

In three independent trials matching the pre-plant GAP in the USA, residues of flumioxin in forage were all < 0.02 mg/kg and residues in hay sampled at BBCH 61–85 were also < 0.02 mg/kg.

The Meeting estimated a maximum residue level of 0.02* mg/kg (dry weight), a median residue of 0 mg/kg (fresh weight) and a highest residue of 0 mg/kg (fresh weight) for wheat hay and a median residue of 0 mg/kg for wheat forage.

Wheat straw

The critical GAP for wheat in the USA is for a broadcast foliar application of up to 0.07 kg ai/ha as a harvest aid (desiccant) up to 10 days before harvest.

In 21 independent trials matching the pre-harvest desiccant GAP in the USA, residues of flumioxazin in wheat straw sampled at grain maturity (10 day PHI) were 0.23, 0.76, 1.1, 1.4, 1.5, 1.6, 1.6, 1.6, 1.6, 1.7, 1.7, 1.8, 1.8, 2.1, 2.4, 2.4, 2.6, 3.2, 3.2, 3.4 and 3.7 mg/kg.

The Meeting estimated a a median residue of 1.7 mg/kg (fresh weight), a highest residue of 3.7 mg/kg (fresh weight) and after correction for an average 88% dry matter content, estimated a maximum residue level of 7.0 mg/kg (dry weight), for wheat straw.

Fate of residues during processing

Hydrolysis in aqueous media is pH-dependant, with half-lives at 25 °C ranging from 3–5 days at pH 5 to less than 25 minutes at pH 9 in acetate buffer. After incubation at pH 7 for 2 hours, 482-HA was the only degradate observed (at about 5% TRR) and in the pH 5 buffer solution incubated for 8 hours, levels of 482-HA, THPA and Δ^1-TPA had each increased to about 5% TRR.

The fate of flumioxazin residues has been examined in a number of studies simulating household and commercial processing of apples, plums, grapes, olives, soya beans, potatoes, sugar cane, maize, wheat, oilseed rape, sunflower seed, peanuts and mint. Except for wheat,

sugar cane, oilseed rape and sunflower seed, processing factors could not be estimated because residues in the fresh commodities were below the respective method LOQs.

Estimated processing factors for sugar cane were 0.5 for molasses and < 0.18 for sugar and for oilseed rape, the calculated processing factors were 0.12 for meal and < 0.04 for oil.

Estimated processing factors and STMR-Ps for wheat and sunflower seed, where residues in the raw agricultural commodities (RACs) were above the respective method LOQs are summarized below.

Summary of selected processing factors and STMR-P values for flumioxazin

RAC	Matrix	Flumioxazin [a] Calculated processing factors	STMR-P (mg/kg)
Wheat (0.1 mg/kg)	bran	0.94	0.094
	flour	0.14	0.014
	middling	0.22	0.022
	shorts	0.31	0.031
	germ	1.03	0.103
	aspirated grain fraction	308	30.8
Sunflower seed (0.11 mg/kg)	oil	< 0.009	0.001
	meal	0.065	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 flumioxazin residues in the processed item divided by the residues in the RAC.

The Meeting noted that for wheat, residues do not concentrate in wheat bran, flour, middlings, shorts, and germ. However residues of flumioxazin concentrate by 308× in the aspirated grain fractions. For rape seed, sunflower, and sugar cane, there is no concentration of flumioxazin residues in the corresponding processed fractions.

Residues in animal commodities

Farm animal feeding studies

In a lactating cow feeding study three groups of dairy cattle (three cows per group) were dosed orally with flumioxazin at levels equivalent to 2, 6.2 and 19.5 ppm in the diet for 28 consecutive days (0.7 mg/kg bw/day, 0.22 mg/kg bw/day and 0.73 mg/kg bw/day respectively) and the animals were sacrificed 24 hours after the last dose. Analysis for flumioxazin, 3-OH-flumioxazin and 4-OH-flumioxazin was by HPLC-MS/MS with an LOQ of 0.02 mg/kg.

At the 19.5 ppm dose level, residues of flumioxazin, 3-OH-flumioxazin and 4-OH-flumioxazin were all non-detectable (LOD of 0.01 mg/kg) in all samples of milk, skim milk, cream, liver, kidneys, muscle, and fat. Samples from the lower dose group animals were not analysed.

No poultry feeding studies were provided. In the poultry metabolism study, where two groups of 10 hens were dosed with at levels equivalent to 10 ppm [^{14}C]flumioxazin (phenyl-label or THP-label) in the diet for 14 consecutive days (average of 0.68 mg/kg bw/day), THP-labelled flumioxazin residues were found at levels of up to 0.13 mg/kg in fat, 0.06–0.08 mg/kg in edible offal (liver and kidney), 0.04 mg/kg in egg yolk and up to 0.17 mg/kg in muscle.

Residues of the 4-OH-flumioxazin metabolite (THP-label) were up to 0.07 mg/kg in skin + fat, 0.03 mg/kg in fat, 0.12–0.08 mg/kg in edible offal (liver and kidney), 0.02 mg/kg in egg yolk and up to 0.18 mg/kg in muscle.

Residues of the 3-OH-flumioxazin metabolite (THP-label) were up to 0.04 mg/kg in skin + fat, 0.03 mg/kg in fat, 0.08–0.06 mg/kg in edible offal (liver and kidney), 0.016 mg/kg in egg yolk and up to 0.16 mg/kg in muscle.

Farm animal dietary burden

The Meeting estimated the dietary burden of flumioxazin in farm animals on the basis of the diets listed in Appendix IX of the 2009 edition of the JMPR Manual. Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are presented in Annex X and are summarized below:

Estimated maximum and mean dietary burdens of farm animals

	Animal dietary burden, flumioxazin, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	2.6	2.2 [c]	2.5	0.7	3.8 [a]	1.6	0.39	0.26
Dairy cattle	1.0	0.41	1.9	0.67	2.3 [b]	0.71 [d]	0.59	0.28
Poultry—broiler	0.23	0.23	0.15	0.15	0.15	0.15	0.14	0.049
Poultry—layer	0.23	0.23	0.57 [e, g]	0.34 [f, h]	0.14	0.14	0.11	0.11

[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 3.8 ppm dry weight of feed and for poultry, noting that in some countries, laying hens may also be consumed, suitable calculated maximum and mean dietary burdens are 0.57 ppm and 0.34 ppm dry weight of feed respectively.

Animal commodity maximum residue levels

The Meeting noted that in the cow feeding study, no detectable residues of flumioxazin or the 3-OH-flumioxazin or 4-OH-flumioxazin metabolites were found in milk or any tissues from the 19.5 ppm dose group animals.

As this dose rate is more than 5× the maximum dietary burdens of 3.82 ppm for beef and dairy cattle, the Meeting concluded that residues of flumioxazin, 3-OH-flumioxazin and 4-OH-flumioxazin are not expected in mammalian milk, meat, fat or edible offal.

The Meeting estimated maximum residue levels of 0.02* mg/kg for flumioxazin in meat (from mammals other than marine mammals), edible offal (mammalian), mammalian fat and for milks. Estimated STMRs and HRs for dietary intake estimation are 0 mg/kg for meat, 0 mg/kg for edible offal, 0 mg/kg for fat and 0 mg/kg for milk.

In the hen metabolism study, the highest residues of flumioxazin were up to 0.08 mg/kg in liver and kidney, 0.13 mg/kg in fat, 0.02 mg/kg in muscle and 0.04 mg/kg in egg yolk, equivalent to 0.014 mg/kg in eggs (35:65 yolk:white). As the 10 ppm dose rate in this study is 17.5× the maximum dietary burdens of 0.57 ppm for poultry broilers and layers, the Meeting concluded that the maximum residues of flumioxazin are not expected to exceed 0.005 mg/kg in poultry edible offal, 0.007 mg/kg in fat and would be lower in poultry meat and eggs (0.001 mg/kg or less).

The 10 ppm dose rate is also 29× the mean dietary burdens of 0.34 ppm for poultry broilers and layers, and the Meeting concluded that the mean residues of flumioxazin are not expected to exceed 0.003 mg/kg in poultry edible offal, 0.004 mg/kg in fat and less than 0.001 mg/kg in poultry meat and eggs.

The Meeting estimated maximum residue levels of 0.02* mg/kg for flumioxazin in poultry meat, poultry offal, poultry fat and eggs. Estimated HRs for dietary intake estimation are

0.007 mg/kg for poultry fat, 0.001 mg/kg for poultry meat, 0.005 mg/kg for poultry offal and 0.001 mg/kg for eggs and the STMRs are 0.003 mg/kg for poultry offal, 0.004 mg/kg in fat, 0.001 mg/kg for poultry meat and 0.001 mg/kg for eggs.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed in Annex 1 are suitable for establishing maximum residue limits and for IEDI assessment.

Definition of the residue (for MRL-compliance and estimation of dietary intake, plant and animal commodities): *flumioxazin.*

The residue is not fat soluble.

	Commodity	MRL	STMR or	HR or
CCN	Name	New	STMR-P	HR-P
AL 1021	Alfalfa forage (green)		0.14 (fw)	0.8 (fw)
AL 1020	Alfalfa fodder	3.0 (dw)	0.38 (fw)	1.5 (fw)
	Almond hulls		0.04	
VS 0620	Artichoke, Globe	0.02 *	0.02	0.02
VS 0621	Asparagus	0.02 *	0	0
	Aspirated wheat grain fraction (feed)		30.8	
VD 0071	Beans, dry	0.07	0.02	
FB 2006	Bush berries	0.02 *	0	0
VB 0041	Cabbages, Head	0.02 *	0	0
VD 0524	Chick-pea (dry)	0.07	0.02	
	Cotton gin trash		0.1	0.31
SO 0691	Cotton seed	0.01	0.01	
MO 0105	Edible offal (Mammalian)	0.02 *	0	0
PE 0112	Eggs	0.02 *	0.001	0.001
VC 0045	Fruiting vegetables, Cucurbits	0.02 *	0.02	0.02
VO 0050	Fruiting vegetables, other than Cucurbits (except sweetcorn and mushrooms)	0.02 *	0.02	0.02
FB 0269	Grapes	0.01 *	0	0
VD 0533	Lentil (dry)	0.07	0.02	
VD 0545	Lupin (dry)	0.07	0.02	
GC 0645	Maize	0.02 *	0	
AS 0645	Maize fodder	0.02 *	0	0
AF 0645	Maize forage		0	
MM 0100	Mammalian fats (except milk fats)	0.02 *	0	0
MM 0095	Meat (from mammals other than marine mammals)	0.02 *	0	0
ML 0106	Milks	0.02 *	0	

	Commodity	MRL	STMR or	HR or
CCN	Name	New	STMR-P	HR-P
HH 0738	Mints	0.02	0.01	0.01
FT 0305	Olives	0.02 *	0	0
VA 0385	Onion, Bulb	0.02 *	0	0
SO 0697	Peanut	0.02 *	0	
VD 0072	Peas, dry	0.07	0.02	
FP 0009	Pome fruit	0.02 *	0	0
FI 0355	Pomegranate	0.02 *	0	0
VR 0589	Potato	0.02 *	0	0
PF 0111	Poultry fat	0.02 *	0.004	0.007
PM 0110	Poultry meat	0.02 *	0.001	0.001
PO 0111	Poultry, Edible offal of	0.02 *	0.003	0.005
VD 0541	Soya bean (dry)	0.02 *	0	
FS 0012	Stone fruit	0.02 *	0	0
	Sunflower meal		0.007	
	Sunflower oil		0.001	
SO 0702	Sunflower seed	0.5	0.11	
VR 0508	Sweet potato	0.02 *	0	0
TN 0085	Tree nuts	0.02 *	0	
GC 0654	Wheat	0.4	0.1	
	Wheat hay	0.02 * (dw)	0 (fw)	0 (fw)
CF 0654	Wheat bran, Processed		0.094	
CF 1211	Wheat flour		0.014	
	Wheat forage		0	
CF 1210	Wheat germ		0.103	
	Wheat middling (stock feed)		0.022	
	Wheat shorts (stock feed)		0.031	
	Wheat straw	7.0 (dw)	1.7 (fw)	3.7 (fw)

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intake (IEDI) for flumioxazin 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 flumioxazin for the 17 GEMS/Food cluster diets, based on estimated STMRs were 0–1% of the maximum ADI of 0.02 mg/kg bw (Annex 3). The Meeting concluded that the long-term intake of residues of flumioxazin 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 flumioxazin 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.

For flumioxazin, the IESTI varied from 0–7% of the ARfD (0.03 mg/kg bw for women of child-bearing age) and the Meeting concluded that the short-term intake of residues of flumioxazin from uses considered by the Meeting is unlikely to present a public health concern.

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SBP-0031	Russell, S	1994	S-53482: Determination of the auto-flammability properties according to EC requirements. Hazleton Europe, UK. Report No. 333/13K-1014. Sumitomo Chemical Co., Ltd. Sumitomo Reference: SBP-0031. GLP, Unpublished. 22-Apr-94
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SBR-0026	Schreier, T	2001	Magnitude of the Residue of Flumioxazin on Cotton and Its Processed Products. Valent U.S.A. Corporation. Report No. 20124. Sumitomo Ref: SBR-0026. GLP; Unpublished. 30-Jan-01
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SBR-0116	Arsenovic, M	2006	Flumioxazin: Magnitude of the Residue on Asparagus. Rutgers, The State University of New Jersey. Report No. 08059. Sumitomo Ref: SBR-0116. GLP; Unpublished. 12-Jun-06
SBR-0117	Salzman, FP	2006	Flumioxazin: Magnitude of the Residue on Tomato. Rutgers, The State University of New Jersey. Report No. 08320. Sumitomo Ref: SBR-0117. GLP; Unpublished. 20-Jul-06
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SBR-0128	Arsenovic, M	2011	Flumioxazin: Magnitude of the Residue on Artichoke. Rutgers, The State University of New Jersey. Report No. 09815. Sumitomo Ref: SBR-0128. GLP; Unpublished. 30-Jun-11
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SBR-0131	Arsenovic, M & Leonard, R	2011	Flumioxazin: Magnitude of the Residue on Pomegranate. Rutgers, The State University of New Jersey. Report No. 08671. Sumitomo Ref: SBR-0131. GLP; Unpublished. 29-Mar-11
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FLUOPYRAM (243)

The first draft was prepared by Mr David Lunn, Plants, Food & Environment Directorate, Ministry for Primary Industries, Wellington, New Zealand

EXPLANATION

Fluopyram, a pyridylethylamide broad spectrum fungicide was first evaluated by the 2010 JMPR, where residue definitions were proposed, an ADI of 0–0.01 mg/kg bw and an ARfD of 0.5 mg/kg bw were established 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 JMPR in 2012 and in 2014, with a number of additional maximum residue levels being recommended.

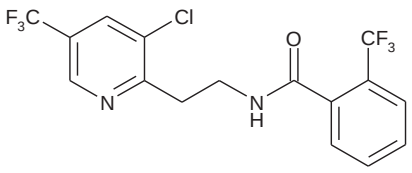
The 2010 JMPR also established residue definitions for fluopyram:

- 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 46th Session of the CCPR (2014) listed fluopyram for further evaluation by the 2015 JMPR for additional MRLs and the current Meeting received new GAP information and/or new supporting residue information from the manufacturer for tomatoes, eggplants, beans, peas, soya beans (dry), sunflower seeds and cotton seed.

The Meeting also considered relevant residue information provided to the JMPR in 2010 for tomatoes, beans and peas (fresh and dry) and sunflower seeds.

Fluopyram is N-{2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethyl}-2-(trifluoromethyl)benzamide. It is relatively insoluble in water (15 mg/L), stable to hydrolysis, of low volatility (1.2×10^{-6} Pa at 20 °C), has a log P_{OW} of 3.3 and is soluble (> 250 g/L) in methanol, dichloromethane, acetone, ethyl acetate and dimethyl sulfoxide.



Fluopyram (AE C656948)

The following abbreviations are used for the metabolites discussed below.

BZM	-benzamide	2-(trifluoromethyl)benzamide
E-olefine		N-{(E)-2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl}-2-trifluoromethyl) benzamide
Z-olefine		N-{(Z)-2-[3-chloro-5-(trifluoromethyl)pyridin-2-yl]ethenyl}-2-trifluoromethyl) benzamide

Methods of residue analysis

Analytical methods

The 2010 JMPR reviewed and summarized analytical method descriptions and validation data for fluopyram and major metabolites (BZM, 7-OH, PCA, PAA and the methyl-sulfoxide) in crop and animal commodities, and in soil. These included Methods 00984 and GM-001-P07-01, which were used to measure residues of fluopyram in the new supervised residue trials on tomatoes, beans and peas, soya beans, sunflower and cotton.

In Method 00984 and its minor variants, fluopyram residues were extracted by maceration with acetonitrile/water and residues were quantified by reversed-phase chromatography with tandem mass spectrometry (MS/MS) with electrospray ionisation. Method GM-001-P07-01, a modification Method 00984, used an isotopically labelled internal standard and included an additional C_{-18} solid phase extraction (SPE) clean-up step.

USE PATTERNS

Information on GAP in the USA and Canada, South Africa and a number of countries in Europe was provided to the Meeting for foliar applications or seed treatments to crops for which new or previously submitted data were available. This GAP information is summarized in Table 1.

Table 1 Registered uses of fluopyram, SC or FS formulations (including co-formulations with tebuconazole, trifloxystrobin and triadimenol)

Crop	Country	Application				Max/season		PHI (days)	Remarks:
		method	max kg ai/ha	kg ai/hL (max)	water L/ha	no	kg ai/ha		
Fruiting vegetables (except Cucurbits)									
Eggplant (indoor)	Greece	spray [d]	0.15	0.01	750–1500	3		3	14 day RTI
Peppers (indoor)	Greece	spray [d]	0.15	0.01	750–1500	3		3	14 day RTI
Tomato	Chile	spray [a]	0.25	0.025	1000	2		4	7 day RTI
Tomato (indoor)	Greece	spray [d]	0.15	0.01	750–1500	3		3	14 day RTI
Tomato	Morocco (2012)	spray [b]		0.0125		2		3	7 day RTI
Tomato	Ukraine	spray [a]	0.15	0.01		2		7	
Legume vegetables									
Beans	Netherlands	spray	0.25			2	0.5	7	7 day RTI
Beans	Belgium	spray	0.25		200–800	2	0.5	7	7 day RTI from flowering (BBCH 60–79)
Peas (without pods)	Netherlands	spray	0.25		200–800	2	0.5	7	7 day RTI
Peas (without pods)	Belgium	spray	0.25			2	0.5	7	7 day RTI from flowering (BBCH

Crop	Country	Application				Max/season		PHI (days)	Remarks:
		method	max kg ai/ha	kg ai/hL (max)	water L/ha	no	kg ai/ha		
									60–79)
Pulses									
Soya bean	USA	seed [e]		0.25 mg ai/seed		1	0.25	pre-plant	No grazing or feed use
Oilseeds									
Sunflower	Moldovia	spray [c]	0.125			2		50	BBCH 32–57
Sunflower	Ukraine	spray [c]	0.125			2		50	BBCH 32–57
Cotton seed	USA	seed [e]		0.35 mg ai/seed		1		pre-plant	
Cotton seed	USA	in-furrow spray	0.25		94 (gr)	1		pre-plant	

RTI = Re-treatment interval

[a] SC formulation containing 200 g ai/L fluopyram + 200 g ai/L tebuconazole

[b] SC formulation containing 250 g ai/L fluopyram + 250 g ai/L trifloxystrobin

[c] SE formulation containing 125 g ai/L fluopyram + 125 g ai/L prothioconazole

[d] SC formulation containing 250 g ai/L fluopyram + 250 g ai/L triadimenol

[e] FS seed treatment formulation containing 600 g ai/L fluopyram

Residues resulting from supervised trials

The Meeting reviewed supervised field trial information provided to the JMPR in 2010 and received new information on supervised field trials involving applications of fluopyram to the following crops.

Crop Group	Commodity	Region	Table No.
Fruiting vegetables, other than cucurbits	Tomato (protected)	Europe	2
	Tomato (outdoor)	Europe	3
Legume vegetables	Beans (protected)	Europe	4 [a]
	Beans (outdoor)	Europe	5 [a], 6
	Peas	Europe	7 [a], 8
Pulses	Soya bean (dry)	North America	9 [a], 10
Oilseeds	Sunflower seed	Europe	11
	Cottonseed	North America	12, 13
Legume animal feeds	Bean forage	Europe	14 [a], 15
	Pea vines and hay	Europe	16 [a], 17

[a] Data from trials evaluated by the 2010 JMPR

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.

When multiple applications were made to a crop, the application rates, spray concentrations and spray volumes 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 values 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.

Fruiting vegetables (except Cucurbits)

Tomatoes

Results from supervised trials from Europe on greenhouse and field tomatoes were provided to the Meeting to supplement the data provided to the 2010 JMPR.

In four greenhouse trials provided to the Meeting, three applications of fluopyram (SC formulations) were made at 13–14 day intervals to mature plants as foliar sprays using knapsack sprayers with hand lances (1–4 nozzles) to apply 0.15 kg ai/ha in 1000–1500 L water/ha. Plot sizes in these trials ranged from 15–46 m^2. In a further eight greenhouse trials, two applications of 0.15 kg ai/ha fluopyram in 1000 L water/ha were made at 7-day intervals using similar equipment and with plot sizes of 12–38 m^2.

In eight field trials provided to the Meeting, two applications of fluopyram (SC formulations) were made at 6–7 day intervals to mature plants as foliar sprays using knapsack or motorised sprayers with 1–2 nozzle hand lances or mini-booms (3–9 flat-fan nozzles) to apply 0.15 kg ai/ha in 500–1000 L water/ha. Plot sizes in these trials ranged from 23–80 m^2.

In all trials, unreplicated samples of at least 2 kg or 24–28 fruit (48–100 for cherry tomatoes) were taken from each plot, frozen within 24 hours of sampling and stored at or below –18 °C for up to 440 days before analysis for fluopyram and metabolites using LC/MS/MS Methods 00984 or 00984/M001 (LOQ 0.01 mg/kg). Mean recovery rates in samples spiked with 0.01–1.0 mg/kg fluopyram ranged from 87–109%.

Table 2 Residues in tomatoes from supervised greenhouse trials in Europe involving two or three foliar applications of fluopyam (SC formulation)

TOMATO Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Spain, 2010 Sanlucar de Barrameda (Matias)	3	0.15	0.01	1500	fruit	–0 0 1 3 7 10	0.02 0.05 0.03 <u>0.04</u> 0.03 0.02	10-2194 10-2194-01
Italy, 2010 Croce Camerina (Parsifal)	3	0.15	0.012	1300	fruit	–0 0 1 3 7 10	0.07 0.09 0.07 0.06 <u>0.13</u> 0.08	10-2194 10-2194-02
Germany, 2010 Leichlingen (Albis)	3	0.15	0.015	1000	fruit	–0 0 1 3 7 10	0.05 0.08 0.10 <u>0.08</u> 0.07 0.06	10-2194 10-2194-03
Netherlands, 2010 Honselersdyk (Doloress)	3	0.15	0.015	1000	fruit	–0 0 1 3 7 10	0.02 0.06 0.07 0.06 <u>0.07</u> 0.05	10-2194 10-2194-04
Germany, 2013 Leichlingen (Meceno)	2	0.15	0.015	1000	fruit	–0 0 1 3 7 10 14	0.017 0.049 0.019 0.045 0.034 0.026 0.023	13-2121 13-2121-01
Netherlands, 2013 Zwaagdijk (Super Sweet 100) Cherry tomato	1+ 1	0.14 0.15	0.015	950 980	fruit	–0 0 1 3 7 10 14	0.044 0.16 0.17 0.15 0.14 0.069 0.079	13-2121 13-2121-02
Belgium, 2013 Saint-Amand (Macarena Beef tomato)	2	0.15	0.015	1000	fruit	–0 0 1 3 7 10 14	0.062 0.064 0.15 0.12 0.075 0.098 0.08	13-2121 13-2121-03
France, 2013 Castelsarrasin (Kiveli F1 Hybrid)	1+ 1	0.14 0.15	0.015	950 980	fruit	–0 0 1 3 7 10 14	0.054 0.11 0.12 0.11 0.093 0.11 0.086	13-2121 13-2121-04

TOMATO Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Spain, 2013 Bigues i Riells (Plumcher) Cherry tomato	2	0.15 0.16	0.015	1000 1060	fruit	–0 0 1 3 7 10 14	0.23 0.1 0.24 0.23 0.21 0.22 0.21	13-2121 13-2121-05
Italy, 2013 Vittoria (RG) (Creativo) Cherry tomato	2	0.15	0.015	1000	fruit	–0 0 1 3 7 10 14	0.068 0.17 0.12 0.13 0.097 0.11 0.099	13-2121 13-2121-06
Greece, 2013 Katerini, Pieria (Corbus) Cherry tomato	1+ 1	0.14 0.15	0.015	950 1000	fruit	–0 0 1 3 7 10 14	0.032 0.063 0.052 0.04 0.029 0.034 0.041	13-2121 13-2121-07
Portugal, 2013 Silveira-Torres Vedras (Bigran)	2	0.15	0.015	1000	fruit	–0 0 1 3 7 10 14	0.11 0.16 0.18 0.15 0.19 0.21 0.19	13-2121 13-2121-08

Table 3 Fluopyram residues in tomatoes from supervised field trials in Europe involving two foliar applications of fluopyam (SC formulation)

TOMATO Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
France (S), 2013 St Etienne du gres (Leader)	2	0.15	0.025	600	fruit	–0 0 1 3 7 10 14	0.046 0.22 0.14 0.10 0.058 0.047 0.047	13-2120 13-2120-01
Spain, 2013 Los Palacios (Albatross)	2	0.15	0.019	800	fruit	–0 0 1 3 7 10 14	0.066 0.30 0.24 0.18 0.097 0.13 0.095	13-2120 13-2120-02
Italy, 2013 Bologna (Monti)	2	0.15	0.025	600	fruit	–0 0 1 3 7 10 14	0.034 0.21 0.13 0.18 0.067 0.07 0.053	13-2120 13-2120-03
Portugal, 2013 Almeirim (H-1015)	2	0.15	0.03	500	fruit	–0 0 1 3 7 10 14	0.018 0.075 0.046 0.03 0.020 0.014 0.016	13-2120 13-2120-04
Greece, 2013 Aronas, Katerini (Evia)	2	0.15	0.015	1000	fruit	–0 0 1 3 7 10 14	< 0.01 0.014 0.014 0.017 < 0.01 < 0.01 < 0.01	13-2120 13-2120-05
RA-13-1210, 13-2120-06 France (S), 2013 Boé (Leader)	2	0.15	0.019	800	fruit	–0 0 1 3 7 10 14	0.037 0.16 0.10 0.14 0.096 0.087 0.082	13-2120 13-2120-06
Spain, 2013 Alginet (Maplica)	2	0.15	0.019	800	fruit	–0 0 1 3 7 10 14	0.070 0.14 0.18 0.10 0.17 0.12 0.13	13-2120 13-2120-07
Italy, 2013 Ostellato (5408 f1)	2	0.15	0.03	500	fruit	–0 0 1 3 7 10 14	0.066 0.24 0.11 0.15 0.11 0.13 0.098	13-2120 13-2120-08

Legume vegetables

Common beans

Results from supervised trials from Europe on protected and outdoor common beans were provided to the Meeting to supplement the data provided to the 2010 JMPR.

In the 2007–2008 trials evaluated by the 2010 JMPR, two applications of fluopyram (SC 500 formulations) were made 7–8 days apart as foliar sprays using knapsack or wheel barrow sprayers with 1–4 flat-fan, solid or hollow-cone nozzles or hand-held-booms (3–12 flat-fan nozzles), applying 0.3 kg ai/ha in 600-1500 L water/ha to the protected crops and 0.25 kg ai/ha in 300–1000 L water/ha to the outdoor crops. Plot sizes in these trials ranged from 8–108 m^2.

In the more recent trials, two foliar applications of 0.2 kg ai/ha fluopyram (SC formulations) were made 7–9 days apart using knapsack sprayers with 1–3 flat-fan, solid or hollow-cone nozzles or plot boom sprayers (5–12 nozzles) to apply 300–500 L spray mix/ha to the protected crops and 0.25 kg ai/ha in 300–1000 L water/ha to the outdoor crops. Plot sizes in these trials ranged from 18–125 m^2.

Unreplicated samples of 1–3 kg of pods (including seeds) and in the outdoor trials at least 12–18 kidney bean plants (min 1 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 456 days before analysis for fluopyram using LC/MS/MS Methods 00984 or 00984/M001. The reported LOQs were 0.01 mg/kg for each analyte. Mean fluopyram recovery rates ranged from 87–98% in fresh pods spiked with 0.01–4.0 mg/kg, 85–100% in vines spiked with 0.01–10 mg/kg and 101% in seeds (fresh) spiked with 0.01–0.1 mg/kg.

Table 4 Fluopyram residues in protected common beans from supervised trials in Europe, evaluated by the 2010 JMPR [Ref: JMPR 2010 E, Table 143, pp 1565–66]

BEANS Country, year Location (variety)	Application				matrix	DALA	Residues (mg/kg)	Reference & Comments
	no	kg ai/ha	kg ai/hl	water (L/ha)			Fluopyram	
France, 2006 Rognonas (Nadal)	2	0.3	0.03	1000	pods	–0 0 3 7 10 14	0.24 1.2 0.63 <u>0.43</u> 0.33 0.24	RA-2596/06 0379-06
France, 2006 Noves (Donna)	2	0.3	0.03	1000	pods	–0 0 3 7 10 14	0.43 1.2 0.78 <u>0.69</u> 0.49 0.36	RA-2596/06 0752-06
Spain, 2006 Almerimar (Donna)	2	0.3	0.02	1500	pods	–0 0 3 7 10 14	0.14 0.83 0.63 <u>0.2</u> 0.16 0.09	RA-2596/06 0753-06
Spain, 2006 St M del Aguila (Festival)	2	0.3	0.02	1500	pods	–0 0 3 7 10 14	0.27 0.99 0.68 <u>0.22</u> 0.15 0.09	RA-2596/06 0754-06

BEANS Country, year Location (variety)	Application				matrix	DALA	Residues (mg/kg)	Reference & Comments
	no	kg ai/ha	kg ai/hl	water (L/ha)			Fluopyram	
Germany, 2006 Langenfeld (Markant)	2	0.3	0.05	600	pods	–0 0 3 7 10 14	0.23 0.39 0.28 <u>0.16</u> 0.09 0.1	RA-2596/06 0755-06
Germany, 2006 Meckenbeuren (Eva)	2	0.3	0.02	1500	pods	–0 0 3 7 10 13	0.21 0.55 0.49 0.12 <u>0.16</u> 0.08	RA-2596/06 0756-06
Netherlands, 2006 Andijk (Overvloed)	2	0.3	0.03	1000	pods	–0 0 3 7 10 14	0.16 0.33 0.11 0.06 <u>0.07</u> 0.03	RA-2596/06 0757-06
Belgium, 2006 Villers-Perwin (Grappes de Malines)	1+ 1	0.3 0.327	0.03 0.03	1000 1090	pods	–0 0 3 7 10 14	0.1 0.27 0.22 0.14 <u>0.15</u> 0.08	RA-2596/06 0759-06
Spain, 2007 Puebla de Vicar (Donna)	2	0.3	0.02	1500	pods	–0 0 3 7 10 14	0.2 0.73 0.59 <u>0.22</u> 0.09 0.06	RA-2607/07 0248-07

Table 5 Residues in outdoor common beans from supervised trials in Europe, evaluated by the 2010 JMPR [Ref: JMPR 2010 E, Table 144, pp 1567–71]

BEANS Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Germany, 2006 Lampertheim (Albani)	2	0.25	0.05	500	pods	–0 0 3 7 10 14	0.09 0.57 0.53 <u>0.24</u> 0.18 0.15	RA-2594/06 0377-06
Germany, 2006 Langenfeld-Reusrath (Classic)	2	0.25	0.0835	300	pods	–0 0 3 7 10 14	0.05 0.2 0.1 <u>0.07</u> 0.06 0.06	RA-2594/06 0654-06
Netherlands, 2006 Zwaagdijk-Oost (Unknown)	2	0.25 0.23	0.05	500 460	pods	–0 0 3 7 10 14	0.05 0.22 0.21 <u>0.2</u> 0.19 0.13	RA-2594/06 0655-06
Belgium, 2006 Villers-Perwin (Polder)	2	0.25	0.0385	650	pods	–0 0 3 7 10 14	0.14 0.47 0.41 <u>0.21</u> 0.18 0.12	RA-2594/06 0656-06
Belgium, 2007 Villers-Perwin (Cadillac)	2	0.25	0.025	1000	pods	–0 0 3 7 10 14	0.06 0.34 0.3 <u>0.12</u> 0.09 0.08	RA-2511/07 0014-07
Germany, 2007 Langenfeld-Reusrath (Classic)	2	0.25	0.0415	600	pods	–0 0 3 7 10 14	0.09 0.39 0.23 <u>0.18</u> 0.14 0.13	RA-2511/07 0546-07
France, 2007 Fresnoy les Roye (Lugos)	2	0.25	0.05	500	pods	–0 0 3 7 10 14	0.13 0.45 0.39 <u>0.26</u> 0.18 0.13	RA-2511/07 0547-07
Netherlands, 2007 Biddinghuizen (Cadillac)	2	0.25	0.05	500	pods	–0 0 3 7 10 14	0.07 0.27 0.19 <u>0.19</u> 0.17 0.15	RA-2511/07 0548-07
Germany, 2007 Swisttal-Heimerzheim (Sonesta)	2	0.25	0.0415	600	pods	–0 0 3 6 10 13	0.08 0.41 0.35 <u>0.17</u> 0.1 0.08	RA-2511/07 0549-07

BEANS Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Spain, 2006 Alginet (Cleo)	2	0.25	0.0415	600	pods	–0 0 3 7 10 14	0.03 0.4 0.17 <u>0.24</u> 0.13 0.14	RA-2595/06 0378-06
Italy, 2006 Pradelle di Nogarole Rocca (Jamaica)	2	0.25	0.05	500	pods	–0 0 3 7 10 14	0.08 0.6 0.15 <u>0.1</u> 0.08 0.06	RA-2595/06 0620-06
Spain, 2006 Malgrat de Mar (Nasao)	2	0.25	0.05 0.0415	500 600	pods	–0 0 2 7 10 14	0.03 0.42 0.1 <u>0.05</u> 0.05 0.03	RA-2595/06 0657-06
Italy, 2006 Ladispoli (Bronco)	2	0.25	0.0315	800	pods	–0 0 3 7 10 14	0.06 0.33 0.18 <u>0.11</u> 0.08 0.1	RA-2595/06 0658-06
France, 2007 Chazay d'azergues (Contender)	2	0.25	0.025	1000	pods	–0 0 3 7 10 14	0.07 0.45 0.25 <u>0.11</u> 0.1 0.08	RA-2512/07 0035-07
Italy, 2007 Ladispoli (Bronco)	2	0.25	0.0315	800	pods	–0 0 3 7 10 14	0.02 0.47 0.05 <u>0.03</u> 0.02 0.02	RA-2512/07 0550-07
Spain, 2007 Alginet (Cleo)	2	0.25	0.025	1000	pods	–0 0 3 7 10 14	0.12 0.17 0.26 <u>0.25</u> 0.19 0.16	RA-2512/07 0551-07
Portugal, 2007 Ribafria Peniche (Tradicional)	2	0.25	0.025	1000	pods	–0 0 3 7 10 14	0.3 0.52 0.45 <u>0.43</u> 0.27 0.32	RA-2512/07 0552-07

Table 6 Fluopyram residues in common beans from supervised outdoor trials in Europe involving two foliar applications of fluopyam (SC formulation)

BEANS Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	

BEANS Country, Year Location (Variety)	Application				Matrix	DAL A	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Germany, 2012 Werl-Mawicke (Primel bean)	2	0.2	0.067	300	pods	7 10 14 21	0.078 0.058 0.021 < 0.01	12-2030 12-2030-01
France (N), 2012 Fondettes (Contender)	2	0.2	0.04	500	pods	–0 0 7 10 14 21	0.068 0.51 0.14 0.13 0.11 0.052	12-2030 12-2030-02
France (N), 2008 Picardie (Flavert) SC500 formulation A	2	0.25	0.05	500	pods	–0 0 3 7 10 14	0.07 0.41 0.48 0.17 0.14 0.16	08-2034 08-2034-01
France (N), 2008 (Flavert) SC500 formulation B	2	0.25	0.05	500	pods	–0 0 3 7 10 14	0.06 0.48 0.45 0.17 0.12 0.14	08-2034 08-2034-01
Italy, 2008 Lazio (Bronco) Kidney bean SC500 formulation A	2	0.25	0.031	800	pods	–0 0 3 7 10 14	0.04 0.39 0.24 0.11 0.09 0.06	08-2096 08-2096-01
Italy, 2008 Lazio (Bronco) Kidney bean SC500 formulation B	2	0.25	0.031	800	pods	–0 0 3 7 10 14	0.05 0.42 0.32 0.15 0.13 0.08	08-2096 08-2096-01
France (N), 2010 Criquebeuf sur Seine (Flagrano)	2	0.2	0.067	300	pods seeds (green)	–0 0 7 14 21 28	0.07 0.41 0.05 0.02 < 0.01 < 0.01	10-2128 10-2128-01
France (N), 2010 Damery (Flagrano)	2	0.2	0.067	300	pods seeds (green)	–0 0 7 14 21 28	< 0.01 0.19 0.04 0.02 < 0.01 < 0.01	10-2128 10-2128-02
Germany, 2010 Heimerzheim (Orinoko)	2	0.2	0.067	300	pods	15 21 28	< 0.01 < 0.01 < 0.01	10-2125 10-2125-01
Belgium, 2010 Villers-Perwin (Beaufort)	2	0.2	0.05	400	pods	7 14 21 28	< 0.01 < 0.01 < 0.01 < 0.01	10-2125 10-2125-02

BEANS Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Spain, 2010 Alginet (Cleo)	2	0.2	0.04	500	pods	–0 0 7 14 22 28	< 0.01 0.02 <u>0.01</u> 0.01 0.01 0.01	10-2125 10-2125-03
Italy, 2010 Ladispoli (Orinoko)	2	0.2	0.04	500	pods	–0 0 7 14 21 28	< 0.01 0.05 <u>< 0.01</u> < 0.01 < 0.01 < 0.01	10-2125 10-2125-04
France (S), 2010 Toulouse (Argus)	2	0.2	0.04	500	pods	–0 0 7 14 21 28	< 0.01 0.03 < 0.01 <u>0.04</u> 0.02 0.03	10-2125 10-2125-05
Portugal, 2010 Ribafria (Bolinhas)	2	0.212	0.04	530	pods	–0 0 7 14 21 28	0.16 0.26 <u>0.08</u> 0.05 0.02 0.02	10-2125 10-2125-06
France (S), 2011 Toulouse-Croix daurade (Argus French bean)	2	0.2	0.04	500	pods	–0 0 7 14 21 28	0.077 0.62 <u>0.088</u> 0.054 0.045 0.038	11-2001 11-2001-01
Spain, 2011 Alginet (Cleo dwarf bean)	2	0.2	0.04	500	pods	–0 0 7 14 21 30	0.12 0.87 <u>0.32</u> 0.084 0.067 0.041	11-2001 11-2001-02
Italy, 2011 Andria (Blue lake)	2	0.2	0.04	500	pods	–0 0 7 14 21 28	0.056 4.3 <u>0.17</u> 0.072 0.015 < 0.01	11-2001 11-2001-03
Portugal, 2011 Atowia da Enreia (Bolinhas)	2	0.2	0.04	500	pods	–0 0 7 14 21 28	0.072 0.33 <u>0.1</u> 0.05 0.037 0.039	11-2001 11-2001-04

Peas

Results from supervised trials from Europe conducted in 2012 on <u>field peas</u> were provided to the Meeting to supplement the data provided to the 2010 JMPR.

In the trials evaluated by the 2010 JMPR, two applications of fluopyram (SC formulation) were made to peas 7–8 days apart as foliar sprays using knapsack or wheel barrow

sprayers with hand-held spray booms (3–12 flat-fan or hollow cone nozzles), applying 0.25 kg ai/ha in 300–600 L water/ha. Plot sizes in these trials ranged from 24–101 m^2.

Unreplicated samples of at 0.5–4 kg succulent seeds, pods and/or whole plants (including pods and seeds but without roots) were taken from each plot, frozen within 24 hours of sampling and stored at –18 °C or below for up to 329 days before analysis for fluopyram and its metabolites using LC/MS/MS Method 00984. The reported LOQs were 0.01 mg/kg for each analyte and average fluopyram recovery rates were 100–101% in plants and pods spiked with 0.01–10 mg/kg and 88–99% in succulent seeds spiked with 0.01–0.5 mg/kg.

In the 2012 trials, two applications of fluopyram (SC formulations) were made to peas 7–9 days apart as foliar sprays using knapsack or plot sprayers with hand-held single-nozzle lances or spray booms (3–12 flat-fan or hollow cone nozzles), applying 0.2–0.25 kg ai/ha in 300–500 L water/ha. Plot sizes in these trials ranged from 45–160 m^2.

Unreplicated samples of at least 1 kg of fresh pods and vines (without pods and roots) and at least 0.5 kg of dry seeds and straw were taken from each plot, frozen within 24 hours of sampling and stored at –18 °C or below for up to 467 days before analysis for fluopyram using LC/MS/MS Method 00984 or 00984/M003. The reported LOQs were 0.01 mg/kg for each analyte and average fluopyram recovery rates ranged from 91–100% in fresh pods spiked with 0.01–2.0 mg/kg, 93–101% in vines spiked with 0.01–20 mg/kg, 87–100% in seeds (fresh and dry) spiked with 0.01–1.0 mg/kg and 89–98% in straw spiked with 0.01–20 mg/kg.

Table 7 Residues in fresh peas (with and without pods) from supervised trials in Europe evaluated by the 2010 JMPR. [Ref: JMPR 2010 E, Table 147, pp 1573–75]

PEAS Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Germany, 2006 Machern (Harnaß)	2	0.25	0.079 0.0745	317 336	pods seeds (green)	3 7 10 14	0.2 <u>0.03</u> 0.02 0.03	RA-2597/06 0380-06
United Kingdom, 2006 Needham (Hawk)	2	0.25	0.0835	300	pods seeds (green)	3 7 10 14	0.57 <u>0.05</u> 0.03 0.04	RA-2597/06 0722-06
Germany, 2006 Meckenbeuren (Rondo)	2	0.25	0.0835	300	pods seeds (green)	3 7 10 13	0.4 <u>0.01</u> < 0.01 < 0.01	RA-2597/06 0723-06
Netherlands, 2007 Kopstukken (unknown)	2	0.25	0.0415	600	seeds (green)	7 10 14	<u>0.05</u> 0.04 0.03	RA-2513/07 0036-07
Germany, 2007 Burscheid (Wunder von Kelvedon)	2	0.25	0.0835	300	seeds (green)	7 10 14	<u>0.02</u> 0.02 0.02	RA-2513/07 0553-07
France, 2007 Goyencourt (Arabelle)	2	0.25	0.0835	300	seeds (green)	7 10 14	0.03 0.03 <u>0.05</u>	RA-2513/07 0554-07
Belgium, 2007 Landenne-Sur-Meuse (Tristar)	2	0.25	0.0625	400	seeds (green)	7 10 14	<u>0.02</u> 0.02 0.02	RA-2513/07 0555-07

PEAS Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Germany, 2007 Swisttal-Heimerzheim (Spring)	2	0.25	0.0835	300	seeds (green)	7 10 13	0.02 0.01 < 0.01	RA-2513/07 0556-07
Spain, 2006 Brenes (Rondo)	2	0.25	0.0835	300	pods seeds (green)	3 7 9 14	0.94 0.1 0.05 0.03	RA-2598/06 0381-06
Italy, 2006 Migliarino (Agami)	2	0.25	0.0625	400	pods seeds (green)	3 7 9 14	0.06 < 0.01 < 0.01 < 0.01	RA-2598/06 0724-06
France, 2007 Chazay d'Azergues (Douce de provence)	2	0.25	0.0625	400	seeds (green)	7 10 14	0.03 0.03 0.02	RA-2514/07 0037-07
Spain, 2007 Brenes (Rondo)	2	0.25	0.0625	400	seeds (green)	7 10 14	0.01 0.02 0.01	RA-2514/07 0557-07

Table 8 Residues in peas (with and without pods) from supervised field trials in Europe involving two applications of fluopyram (SC formulations)

PEAS Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Spain, 2012 Salobrena Granada (Utrillo)	2	0.2	0.05	400	pods seeds (green) seeds (dry)	–0 0 6 6 9 14 22	0.56 1.2 0.53 0.085 0.032 0.029 0.083	12-2155 12-2155-01
Spain, 2012 Malaga (Utrillo)	2	0.2	0.05	400	pods seeds (green) seeds (dry)	–0 0 7 7 10 14 20 34	0.13 0.49 0.33 0.014 0.014 0.017 0.014 0.043	12-2155 12-2155-02
Italy, 2012 Papiana Marsciano (Gran Rugoso Tondo)	2	0.2	0.04	500	pods seeds (green) seeds (dry)	–0 0 7 7 10 14 21 28	0.41 0.66 0.61 0.055 0.045 0.045 0.13 0.062	12-2155 12-2155-03

PEAS Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Southern France, 2012 Lapalud (Isard)	2	0.2	0.05	400	pods seeds (green) seeds (dry)	–0 0 6 6 10 14 21 40	0.019 0.35 0.25 <u>0.02</u> < 0.01 < 0.01 0.013 0.017	12-2032 12-2032-01
Spain, 2012 Dos Hermanas (Cartouche)	1+ 1	0.2 0.19	0.067 0.067	300 282	pods seeds (green) seeds (dry)	–0 7 7 10 14 21	0.29 0.21 0.064 0.053 <u>0.092</u> 0.097	12-2032 12-2032-02
Italy, 2012 Ladispoli (RM) (Attika)	2	0.2	0.067	300	pods seeds (green) seeds (dry)	–0 0 7 7 10 14 21	0.029 0.18 0.052 < 0.01 < 0.01 <u>0.012</u> 0.024	12-2032 12-2032-03
Greece, 2012 Nea Messimvria (Li Violetta)	2	0.2	0.05	400	pods seeds (green) seeds (dry)	–0 0 7 7 10 14 21 33	0.031 0.48 0.015 <u>0.027</u> 0.017 0.017 0.029 0.029	12-2032 12-2032-04
Spain, 2012 Alginet (Lincoln)	2	0.2	0.04	500	pods seeds (green) seeds (dry)	–0 0 7 7 9 13 21	0.19 0.58 0.5 <u>0.057</u> 0.046 0.055 0.2	12-2032 12-2032-05
Germany, 2012 Burscheid (Respect)	2	0.2	0.067	300	pods seeds (green) seeds (dry)	–0 0 7 7 10 14 21 39	0.095 0.44 0.051 <u>< 0.01</u> < 0.01 < 0.01 < 0.01 0.01	12-2031 12-2031-01

PEAS Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
France (N), 2012 Chaussy (Genial)	2	0.2	0.067	300	pods seeds (green) seeds (dry)	–0 0 7 7 10 14 21 35	0.028 0.39 0.11 0.011 <u>0.012</u> < 0.01 0.012 0.02	12-2031 12-2031-02
Germany, 2012 Beucha-Wolfshain (Rocket)	2	0.2	0.067	300	pods seeds (green) seeds (dry)	–0 0 7 7 10 14 21 43	0.16 0.49 0.04 0.021 <u>0.024</u> 0.015 0.016 0.034	12-2031 12-2031-03
Belgium, 2012 Villers-Perwin (Ravenna)	2	0.2	0.05	400	pods seeds (green) seeds (dry)	–0 0 7 7 10 14 21 37	0.053 0.22 0.03 <u>< 0.01</u> < 0.01 < 0.01 < 0.01 0.019	12-2031 12-2031-04
United Kingdom, 2012 Cambridge (Tommy)	2	0.2	0.067	300	pods seeds (green) seeds (dry)	–0 0 6 7 10 13 20	0.017 0.4 0.42 (c=0.011) <u>0.028</u> 0.022 0.024 0.056	12-2031 12-2031-05
Germany, 2012 Langförden (Alvesta)	2	0.2	0.067	300	pods seeds (green) seeds (dry)	–0 0 7 7 10 13 22 32	0.033 0.16 0.021 <u>< 0.01</u> < 0.01 < 0.01 < 0.01 0.01	12-2031 12-2031-06
Germany, 2011 Burscheid (Mascara)	2	0.2	0.067	300	pods seeds (dry)	–0 0 7 14 21 28	0.037 0.74 0.1 0.017 0.016 0.05	11-2000 11-2000-02

PEAS Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
France (N), 2011 Ambleville (Athos)	2	0.2	0.067	300	pods seeds (dry)	–0 0 0 7 14 21 28	0.094 0.33 4mm rain 0.18 0.15 0.017 0.017 0.015	11-2000 11-2000-01 4mm rain within 1 hour after 2nd application
Spain, 2012 Salobrena (Utrillo)	2	0.25	0.063	400	pods seeds (green) seeds (dry)	–0 0 6 9 14 22	0.46 12 0.9 <u>0.035</u> 0.027 0.084	12-2159 12-2159-01
Italy, 2012 Zibido San Giacomo (Utrillo)	2	0.25	0.083	300	pods seeds (green) seeds (dry)	–0 0 7 7 10 14 21 28	0.43 0.82 0.3 <u>0.053</u> 0.045 0.037 0.11 0.098	12-2159 12-2159-02
Spain, 2013 Alginet (Lincoln)	2	0.25	0.05	500	pods seeds (green) seeds (dry)	–0 0 7 7 9 13 21	0.21 0.64 0.59 <u>0.063</u> 0.05 0.043 0.14	12-2048 12-2048-01
Spain, 2013 Dos Hermanas (Cartouche)	2	0.25	0.05	500	pods seeds (green) seeds (dry)	–0 7 7 10 14 21	0.4 0.26 0.066 0.076 <u>0.12</u> 0.13	12-2048 12-2048-01

Pulses

Soya bean (dry)

Results from supervised trials from the USA on <u>soya beans</u> were provided to the Meeting. In these trials, fluopyram (SC formulation) was applied either as seed treatment to soya bean seeds, or as a seed treatment followed by two foliar applications to the plants. In the plots involving seed treatments, the seeds were slurry-treated with either 0.15 or 0.25 mg ai/seed and the targeted seeding rate was about 544,000 seeds/ha (equivalent to 0.082 kg ai/ha or 0.136 kg ai/ha respectively). Actual seeding rates ranged from 257,000–642,000 seeds/ha. Plot sizes in these trials ranged from 46–370 m^2.

In the plots that also included foliar fluopyram treatments, one application of 0.11–0.12 kg ai/ha was made about 21 days before harvest with a second treatment of 0.25–0.26 kg ai/ha applied 5–8 days later, using CO_2 plot or knapsack sprayers with hand-held spray booms or tractor-mounted boom sprayers to apply 90–190 L spray mix/ha.

Duplicate samples of at least 1 kg seeds were taken from each plot, frozen within 4 hours of sampling, held in frozen storage for up to 585 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg and with an average fluopyram recovery rate of 93% in dry soya bean seeds spiked with 0.01–0.4 mg/kg.

Table 9 Fluopyram residues in in soya beans (dry) from supervised trials in the USA involving seed treatment applications of fluopyram (SC formulations)

SOYA BEAN Country, Year Location (Variety)	Application			Matrix	DALA	Fluopyram residues (mg/kg)		Reference
	no.	kg ai/ha	mg ai/seed				mean	
Seed treatment—0.15 mg ai fluopyram/seed								
USA, 2012 Athens, GA (DP 4546 RR)	1	0.082	0.15	seeds, dry	145	0.01, < 0.01	0.01	RAGMY006 GM001-12HA
USA, 2012 Suffolk, VA (DP 4546 RR)	1	0.083	0.15	seeds, dry	148	< 0.01, < 0.01	< 0.01	RAGMY006 GM002-12HA
USA, 2012 Fisk, MO (Pioneer 97B52)	1	0.082	0.15	seeds, dry	131	< 0.01, < 0.01	< 0.01	RAGMY006 GM003-12HA
USA, 2012 Proctor, AR (Asgrow STB 4404)	1	0.081	0.15	seeds, dry	130	< 0.01, < 0.01	< 0.01	RAGMY006 GM004-12HA
USA, 2012 Cheneyville, LA (AG4403RR)	1	0.081	0.15	seeds, dry	120 127 130 132 138	0.031, < 0.01 < 0.01, < 0.01 0.018, 0.017 0.026, 0.024 0.021, 0.021	< 0.02 < 0.01 0.018 0.025 0.021	RAGMY006 GM005-12DA
USA, 2012 Stewardson, IL (DP 5634 RR)	1	0.082	0.15	seeds, dry	145 148 152 155 159	0.019, 0.022 0.029, 0.023 0.021, 0.028 0.029, 0.026 0.032, 0.018	0.021 0.026 0.025 0.028 0.025	RAGMY006 GM006-12DA
USA, 2012 Marysville, OH (Garst 2834RR)	1	0.038	0.15	seeds, dry	110	< 0.01, < 0.01	< 0.01	RAGMY006 GM007-12HA
USA, 2012 Northwood, ND (Agripro 3212 RR/N)	1	0.054	0.15	seeds, dry	136	< 0.01, < 0.01	< 0.01	RAGMY006 GM008-12HA
USA, 2012 Seymour, IL (NKs28 G1)	1	0.082	0.15	seeds, dry	130	0.013, 0.011	0.012	RAGMY006 GM009-12HA
USA, 2012 Gardner, KS (S2783-4)	1	0.079	0.15	seeds, dry	136	< 0.01, < 0.01	< 0.01	RAGMY006 GM010-12HA
USA, 2012 Clarence, MO (RG 200)	1	0.082	0.15	seeds, dry	136	< 0.01, < 0.01	< 0.01	RAGMY006 GM011-12HA
USA, 2012 Sheridan, IN (Sucrosco 935-01RNX	1	0.061	0.15	seeds, dry	143	0.018, 0.019	0.019	RAGMY006 GM012-12HA

SOYA BEAN Country, Year Location (Variety)	Application			Matrix	DALA	Fluopyram residues (mg/kg)		Reference
	no.	kg ai/ha	mg ai/seed				mean	
USA, 2012 Campbell, MN (NSQ49-Q9)	1	0.097	0.15	seeds, dry	121	< 0.01, < 0.01	< 0.01	RAGMY006 GM013-12HA
USA, 2012 Richland, IA (NK S49-Q9)	1	0.082	0.15	seeds, dry	143	< 0.01, 0.01	0.01	RAGMY006 GM014-12HA
USA, 2012 Gardner, ND (DP 4546 RR)	1	0.085	0.15	seeds, dry	118	< 0.01, < 0.01	< 0.01	RAGMY006 GM015-12HB
USA, 2012 Geneva, MN (Hutchinson)	1	0.081	0.15	seeds, dry	140	< 0.01, < 0.01	< 0.01	RAGMY006 GM016-12HA
USA, 2006 Springfield, NE (RT3253)	1	0.084	0.15	seeds, dry	140	0.012, 0.012	0.012	RAGMP039 GM017-12HA
USA, 2012 Verona, WI (Pioneer 91M90)	1	0.08	0.15	seeds, dry	116	0.02, 0.02	0.02	RAGMY006 GM018-12HA
USA, 2012 Stafford, KS (Pioneer 93B82)	1	0.081	0.15	seeds, dry	134	0.022, 0.032	0.027	RAGMY006 GM019-12HA
USA, 2012 Delavan, WI (SC 9384RR)	1	0.082	0.15	seeds, dry	136	< 0.01, < 0.01	< 0.01	RAGMY006 GM020-12HA
USA, 2012 Conklin, MI (91M91)	1	0.08	0.15	seeds, dry	146	< 0.01, < 0.01	< 0.01	RAGMY006 GM021-12HA
Seed treatment—0.25 mg ai fluopyram/seed								
USA, 2012 Athens, GA (DP 4546 RR)	1	0.136	0.25	seeds, dry	145	0.028, 0.023	0.026	RAGMY006 GM001-12HA
USA, 2012 Suffolk, VA (DP 4546 RR)	1	0.138	0.25	seeds, dry	148	< 0.01, < 0.01	< 0.01	RAGMY006 GM002-12HA
USA, 2012 Fisk, MO (Pioneer 97B52)	1	0.136	0.25	seeds, dry	131	< 0.01, < 0.01	< 0.01	RAGMY006 GM003-12HA
USA, 2012 Proctor, AR (Asgrow STB 4404)	1	0.136	0.25	seeds, dry	130	< 0.01, < 0.01	< 0.01	RAGMY006 GM004-12HA
USA, 2012 Cheneyville, LA (AG4403RR)	1	0.136	0.25	seeds, dry	120 127 130 132 138	0.031, < 0.01 < 0.01, < 0.01 0.018, 0.017 0.026, 0.024 0.021, 0.021	< 0.021 < 0.01 0.018 <u>0.025</u> 0.021	RAGMY006 GM005-12DA
USA, 2012 Stewardson, IL (DP 5634 RR)	1	0.136	0.25	seeds, dry	145 148 152 155 159	0.019, 0.022 0.029, 0.023 0.021, 0.028 0.029, 0.026 0.032, 0.018	0.021 0.026 0.025 <u>0.028</u> 0.025	RAGMY006 GM006-12DA
USA, 2012 Marysville, OH (Garst 2834RR)	1	0.065	0.25	seeds, dry	110	< 0.01, < 0.01	< 0.01	RAGMY006 GM007-12HA

SOYA BEAN Country, Year Location (Variety)	Application			Matrix	DALA	Fluopyram residues (mg/kg)		Reference
	no.	kg ai/ha	mg ai/seed				mean	
USA, 2012 Northwood, ND (Agripro 3212 RR/N)	1	0.090	0.25	seeds, dry	136	< 0.01, < 0.01	< 0.01	RAGMY006 GM008-12HA
USA, 2012 Seymour, IL (NKs28 G1)	1	0.136	0.25	seeds, dry	130	0.013, 0.011	0.012	RAGMY006 GM009-12HA
USA, 2012 Gardner, KS (S2783-4)	1	0.131	0.25	seeds, dry	136	< 0.01, < 0.01	< 0.01	RAGMY006 GM010-12HA
USA, 2012 Clarence, MO (RG 200)	1	0.136	0.25	seeds, dry	136	< 0.01, < 0.01	< 0.01	RAGMY006 GM011-12HA
USA, 2012 Sheridan, IN (Sucrosco 935-01RNX	1	0.102	0.25	seeds, dry	143	0.018, 0.019	0.019	RAGMY006 GM012-12HA
USA, 2012 Campbell, MN (NSQ49-Q9)	1	0.161	0.25	seeds, dry	121	< 0.01, < 0.01	< 0.01	RAGMY006 GM013-12HA
USA, 2012 Richland, IA (NK S49-Q9)	1	0.136	0.25	seeds, dry	143	< 0.01, 0.01	0.01	RAGMY006 GM014-12HA
USA, 2012 Gardner, ND (DP 4546 RR)	1	0.142	0.25	seeds, dry	118	< 0.01, < 0.01	< 0.01	RAGMY006 GM015-12HB
USA, 2012 Geneva, MN (Hutchinson)	1	0.134	0.25	seeds, dry	140	< 0.01, < 0.01	< 0.01	RAGMY006 GM016-12HA
USA, 2006 Springfield, NE (RT3253)	1	0.140	0.25	seeds, dry	140	0.012, 0.012	0.012	RAGMP039 GM017-12HA
USA, 2012 Verona, WI (Pioneer 91M90)	1	0.133	0.25	seeds, dry	116	0.02, 0.02	0.02	RAGMY006 GM018-12HA
USA, 2012 Stafford, KS (Pioneer 93B82)	1	0.136	0.25	seeds, dry	134	0.022, 0.032	0.027	RAGMY006 GM019-12HA
USA, 2012 Delavan, WI (SC 9384RR)	1	0.136	0.25	seeds, dry	136	< 0.01, < 0.01	< 0.01	RAGMY006 GM020-12HA
USA, 2012 Conklin, MI (91M91)	1	0.132	0.25	seeds, dry	146	< 0.01, < 0.01	< 0.01	RAGMY006 GM021-12HA

Table 10 Fluopyram residues in in soya beans (dry) from supervised trials in the USA involving seed treatment plus two foliar applications of fluopyram (SC formulations)

SOYA BEAN Country, Year Location (Variety)	Application			Matrix	DALA	Fluopyram residues (mg/kg)		Reference
	no.	kg ai/ha	mg ai/seed (water/ha)				mean	
Seed treatment—0.25 mg ai fluopyram/seed								

SOYA BEAN Country, Year Location (Variety)	Application			Matrix	DALA	Fluopyram residues (mg/kg)		Reference
	no.	kg ai/ha	mg ai/seed (water/ha)				mean	
USA, 2012 Athens, GA (DP 4546 RR)	1+ 1 1	0.136 0.115 0.252	0.25 (156) (162)	seeds, dry	14	0.02, 0.024	0.022	RAGMY006 GM001-12HA
USA, 2012 Suffolk, VA (DP 4546 RR)	1+ 1 1	0.138 0.116 0.256	0.25 (113) (111)	seeds, dry	24	< 0.01, < 0.01	< 0.01	RAGMY006 GM002-12HA
USA, 2012 Fisk, MO (Pioneer 97B52)	1+ 1 1	0.136 0.115 0.25	0.25 (187) (187)	seeds, dry	14	< 0.01, < 0.01	< 0.01	RAGMY006 GM003-12HA
USA, 2012 Proctor, AR (Asgrow STB 4404)	1+ 1 1	0.136 0.114 0.251	0.25 (146) (146)	seeds, dry	13	0.06, 0.077	0.069	RAGMY006 GM004-12HA
USA, 2012 Cheneyville, LA (AG4403RR)	1+ 1 1	0.136 0.118 0.252	0.25 (167) (164)	seeds, dry	3 10 13 15 21	0.072, 0.087 0.25, 0.11 0.107, 0.189 0.153, 0.098 0.092, 0.085	0.08 0.18 0.148 0.126 0.089	RAGMY006 GM005-12DA
USA, 2012 Stewardson, IL (DP 5634 RR)	1+ 1 1	0.136 0.119 0.253	0.25 (139) (133)	seeds, dry	3 10 14 17 21	0.021, 0.018 0.049, 0.018 0.018, 0.017 0.019, 0.018 0.013, 0.019	0.02 0.034 0.018 0.019 0.016	RAGMY006 GM006-12DA
USA, 2012 Marysville, OH (Garst 2834RR)	1+ 1 1	0.064 0.115 0.255	0.25 (165) (164)	seeds, dry	14	< 0.01, < 0.01	< 0.01	RAGMY006 GM007-12HA
	1+ 1	0.254 0.255	(164) (164)	seeds, dry	14	< 0.01, < 0.01	< 0.01	
USA, 2012 Northwood, ND (Agripro 3212 RR/N)	1+ 1 1	0.09 0.116 0.25	0.25 (142) (140)	seeds, dry	14	< 0.01, < 0.01	< 0.01	RAGMY006 GM008-12HA
	1+ 1	0.251 0.256	(140) (143)	seeds, dry	14	0.01, < 0.01	0.01	
USA, 2012 Seymour, IL (NKs28 G1)	1+ 1 1	0.136 0.113 0.254	0.25 (94) (94)	seeds, dry	13	0.032 0.025	0.029	RAGMY006 GM009-12HA
	1+ 1	0.249 0.245	(93) (92)	seeds, dry	13	< 0.01, < 0.01	< 0.01	
USA, 2012 Gardner, KS (S2783-4)	1+ 1 1	0.131 0.114 0.253	0.25 (142) (145)	seeds, dry	12	0.015, 0.015	0.015	RAGMY006 GM010-12HA
USA, 2012 Clarence, MO (RG 200)	1+ 1 1	0.136 0.112 0.261	0.25 (175) (184)	seeds, dry	12	0.015, 0.014	0.015	RAGMY006 GM011-12HA
USA, 2012 Sheridan, IN (Sucrosco 935-01RNX	1+ 1 1	0.102 0.114 0.251	0.25 (179) (181)	seeds, dry	14	0.011, < 0.01	0.01	RAGMY006 GM012-12HA

SOYA BEAN Country, Year Location (Variety)	Application			Matrix	DALA	Fluopyram residues (mg/kg)		Reference
	no.	kg ai/ha	mg ai/seed (water/ha)				mean	
USA, 2012 Campbell, MN (NSQ49-Q9)	1+ 1 1	0.161 0.114 0.251	0.25 (187) (187)	seeds, dry	13	0.083, 0.076	0.08	RAGMY006 GM013-12HA
USA, 2012 Richland, IA (NK S49-Q9)	1+ 1 1	0.136 0.115 0.249	0.25 (163) (171)	seeds, dry	14	< 0.01, < 0.01	< 0.01	RAGMY006 GM014-12HA
USA, 2012 Gardner, ND (DP 4546 RR)	1+ 1 1	0.142 0.116 0.256	0.25 (142) (144)	seeds, dry	13	0.049, 0.057	0.053	RAGMY006 GM015-12HB
USA, 2012 Geneva, MN (Hutchinson)	1+ 1 1	0.134 0.116 0.248	0.25 (172) (183)	seeds, dry	14	0.022, 0.03	0.026	RAGMY006 GM016-12HA
USA, 2006 Springfield, NE (RT3253)	1+ 1 1	0.14 0.115 0.252	0.25 (131) (131)	seeds, dry	12	0.024, 0.032	0.028	RAGMP039 GM017-12HA
USA, 2012 Verona, WI (Pioneer 91M90)	1+ 1 1	0.133 0.116 0.254	0.25 (174) (171)	seeds, dry	14	0.122, 0.132	0.127	RAGMY006 GM018-12HA
USA, 2012 Stafford, KS (Pioneer 93B82)	1+ 1 1	0.136 0.114 0.25	0.25 (173) (172)	seeds, dry	14	0.242, 0.179	0.211	RAGMY006 GM019-12HA
USA, 2012 Delavan, WI (SC 9384RR)	1+ 1 1	0.136 0.114 0.25	0.25 (173) (172)	seeds, dry	14	< 0.01, 0.013	0.012	RAGMY006 GM020-12HA
USA, 2012 Conklin, MI (91M91)	1+ 1 1	0.132 0.114 0.25	0.25 (148) (149)	seeds, dry	14	< 0.01, < 0.01	< 0.01	RAGMY006 GM021-12HA

Oilseeds

Sunflower seed

Results from supervised trials from Europe on sunflowers were provided to the Meeting. In these trials, two applications of 0.117–0.13 kg ai/ha (SC formulations) were made to sunflower plants, 13–15 days apart as foliar sprays using knapsack or CO_2 plot sprayers with hand-held or wheeled spray booms (1–12 nozzles) to apply 275–400 L spray mix/ha. Applications were made up to the seed development or early ripening stages (BBCH 67–85)Plot sizes in these trials ranged from 36–740 m^2.

Unreplicated samples (min 1 kg seed) were taken from each plot, frozen within 24 hours of sampling, held in frozen storage for up to 358 days before analysis of whole seeds. In a number of trials, seeds (min 9 kg samples) were also conditioned to < 8% moisture content, cleaned, crushed (between 1 mm rubber rollers), shelled and dry-fractioned to separate the kernels (the commodity in trade), prior to analysis. The analytical methods used in these trials for measuring fluopyram residues were LC/MS/MS Method 00948/M001 or 00948/M003, with a reported LOQ of 0.01 mg/kg and with average fluopyram recovery rates of 92–100% in seeds, kernels and seed fractions spiked with 0.01–0.1 mg/kg.

Table 11 Fluopyram residues in sunflower seed (dried) from supervised trials in Europe, involving two foliar applications (SE formulations)

SUNFLOWER SEED Study, Trial Country, Year (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Germany, 2010 Burscheid (Rigasol)	2	0.125	0.042	300	seed	28	< 0.01	10-2238 10-2238-01 BBCH 71 & 73
Belgium, 2010 Frasnes-Lez-Gosselies (LG 54.50 HO)	2	0.125	0.0455	275	seed	27	< 0.01	10-2238 10-2238-02 BBCH 69 & 85
Greece, 2010 Gallikos, Kilkis (Sanay MP)	2	0.125	0.031	400	seed	28	0.04	10-2238 10-2238-03 BBCH 71 & 79
Spain, 2010 Fuentes de Andalucia (Transol)	2	0.125	0.042	300	seed	27+ 6 [a]	0.04	10-2247 10-2247-01 BBCH 71 & 79
Germany, 2011 Burscheid (Rigasol)	2	0.13	0.042	300	seed kernel seed fraction	21 24 28 31 35 28 28	0.019 0.011 0.011 < 0.01 < 0.01 < 0.01 < 0.01	11-2002 11-2002-01 BBCH 61 & 71
Belgium, 2011 Villers-Perwin (P64HE01)	2	0.13	0.045	275	seed kernel seed fraction	21 23 28 30 35 28 28	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	11-2002 11-2002-02 BBCH 65 & 69
Greece, 2011 Kissa/Kozani (Sanay)	2	0.13	0.031	400	seed kernel seed fraction	21 24 28 31 35 28 28	0.011 0.019 0.032 < 0.01 < 0.01 < 0.01 < 0.01	11-2002 11-2002-03 BBCH 83 & 85
France (S), 2011 Gargas (Tekny)	2	0.13	0.042	300	seed kernel seed fraction	21 23 28 30 35 28 28	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	11-2002 11-2002-04 BBCH 71 & 81
France (N), 2008 Mesnil Milon (Vellox Early variety)	2	0.125	0.042	300	seed kernel seed fraction	21 24 28 31 35 28 28	0.011 < 0.01 < 0.01, 0.015 0.011 0.01 < 0.01 < 0.01	12-2008 12-2008-01 BBCH 71 & 83

SUNFLOWER SEED Study, Trial Country, Year (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Belgium, 2008 Marbais (P64HE01)	2	0.125	0.042	300	seed kernel seed fraction	21 25 28 31 35 28 28	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	12-2008 12-2008-02 BBCH 76 & 83
France, 2009 Tarascon (CSF 10902)	2	0.125	0.042	300	seed kernel seed fraction	21 24 28 31 35 28 28	0.019 0.074 0.073, 0.065 < 0.01 0.016 < 0.01 < 0.01	12-2009 12-2009-01 BBCH 79 & 83
Spain, 2009 Dos Hermanas (PR64H37)	2	0.125	0.042	300	seed kernel seed fraction	21 24 28 31 35 28 28	0.021 0.023 0.019, 0.022 0.028 0.02 < 0.01 < 0.01	12-2009 12-2009-02 BBCH 72 & 83
Italy, 2009 Furbara Cerveteri (RM) (Starsol)	2	0.125	0.031	400	seed kernel seed fraction	21 24 28 31 35 28 28	0.023 0.029 0.016, 0.018 0.018 0.016 < 0.01 < 0.01	12-2009 12-2009-03 BBCH 73 & 79
Portugal, 2009 Aramanha-Várzea (PR64H47)	2	0.125	0.042	300	seed kernel seed fraction	21 24 28 31 35 28 28	0.051 0.041 0.031 0.092 0.04 < 0.01 < 0.01	12-2009 12-2009-04 BBCH 65 & 73
Greece, 2009 Kissa, Kozani (PR64LE20)	2	0.125	0.031	400	seed kernel seed fraction	21 24 28 31 35 28 28	< 0.01 0.015 0.016, 0.021 0.016 0.025 < 0.01 < 0.01	12-2009 12-2009-05 BBCH 63 & 67
Italy, 2009 Bologna (PR64H41)	1+ 1	0.117 0.125	0.031 0.031	374 400	seed kernel seed fraction	21 24 28 31 35 28 28	0.020 < 0.01 0.013 0.014 0.01 < 0.01 < 0.01	12-2009 12-2009-06 BBCH 69 & 79

[a] Seeds stored for 6 days at 18–31 °C before sampling

Cotton seed

Results from supervised trials from the USA on cotton were provided to the Meeting. In these trials fluopyram was applied either as a pre-plant seed treatment, as a seed treatment in combination with an in-furrow soil treatment at planting or as a combination of a seed treatment, in-furrow soil treatment and a foliar spray applied about 30 days before harvest.

For the plots receiving treated seed, cotton seeds were slurry-treated with 0.5 mg ai/seed and the targeted seeding rate was about 148,000 seeds/ha (equivalent to 0.074 kg ai/ha). Actual seeding rates ranged from 144,495–148,650 seeds/ha. Residues in cotton seed and gin byproducts from the seed treatment plots and from plots involving the combination of seed treatment and in-furrow soil treatments are summarized in the following tables.

Plots were harvested by mechanical picker, mechanical stripper or manually, with duplicate samples of at least 30 kg (undelinted seed plus gin trash) taken from the mechanically harvested plots and at least 1 kg (seed cotton) from the manually harvested plots. Samples were frozen within 24 hours of sampling, ginned and held in frozen storage for up to 148 days before analysis for fluopyram using LC/MS/MS Method GM-001-P07-01, with a reported LOQ of 0.01 mg/kg and with average fluopyram recovery rates of 98% in undelinted seed spiked with 0.01–1.0 mg/kg and 97% in gin by-products spiked with 0.01–18 mg/kg.

Table 12 Fluopyram residues in cotton seed and gin byproducts from supervised trials in the USA involving fluopyram seed treatment applications (0.5 mg ai/seed (FS formulation)

COTTON SEED Country, Year Location (Variety)	Application			Matrix	DALA	Fluopyram residues (mg/kg)		Reference
	no.	kg ai/ha	mg ai/seed				mean	
USA, 2012 Chula, GA (FM 1740)	1	0.074	0.5	seed	136	< 0.01, < 0.01	< 0.01	RAGML206-01 GM022-12HA
				gin by-products		0.469, 0.239	0.354	
USA, 2012 Parma, MO (ST4145 LLB2)	1	0.073	0.5	seed	181	< 0.01, < 0.01	< 0.01	RAGML206-01 GM023-12HA
				gin by-products		0.03, 0.017	0.024	
USA, 2012 Proctor, AR (ST4145)	1	0.074	0.5	seed	153	< 0.01, < 0.01	< 0.01	RAGML206-01 GM024-12HA
				gin by-products		0.016, 0.013	0.015	
USA, 2012 Greenville, MS, (ST 5458 (B2RF))	1	0.074	0.5	seed	144	< 0.01, < 0.01	< 0.01	RAGML206-01 GM025-12HA
USA, 2012 Claude, TX, (ST 4145)	1	0.074	0.5	seed	194	< 0.01, < 0.01	< 0.01	RAGML206-01 GM027-12HA
					200	< 0.01, < 0.01	< 0.01	
					206	< 0.01, < 0.01	< 0.01	
					213	< 0.01, < 0.01	< 0.01	
					219	< 0.01, < 0.01	< 0.01	
				gin by-products	194	< 0.01, < 0.01	< 0.01	
					200	< 0.01, < 0.01	< 0.01	
					206	< 0.01, < 0.01	< 0.01	
					213	< 0.01, < 0.01	< 0.01	
					219	< 0.01, < 0.01	< 0.01	
USA, 2012 Levelland, TX (ST5458 (B2RF))	1	0.074	0.5	seed	158	< 0.01, < 0.01	< 0.01	RAGML206-01 GM028-12HA
				gin by-products		< 0.01, < 0.01	< 0.01	
USA, 2012 Hinton, OK (FM1740 B2RF)	1	0.074	0.5	seed	142	< 0.01, < 0.01	< 0.01	RAGML206-01 GM029-12HA
USA, 2012 Wall, TX (FM1740 B2RF)	1	0.072	0.5	seed	151	< 0.01, < 0.01	< 0.01	RAGML206-01 GM030-12HA

COTTON SEED Country, Year Location (Variety)	Application			Matrix	DALA	Fluopyram residues (mg/kg)		Reference
	no.	kg ai/ha	mg ai/seed				mean	
USA, 2012 Sanger, CA (Acala)	1	0.072	0.5	seed	170	< 0.01, < 0.01	< 0.01	RAGML206-01 GM031-12HA
USA, 2012 Madera, CA (Acala)	1	0.074	0.5	seed	170	< 0.01, < 0.01	< 0.01	RAGML206-01 GM033-12HA
USA, 2012 East Bernard, TX (ST 5458 (B2RF))	1	0.073	0.5	seed gin by-products	143	< 0.01, < 0.01 0.021, 0.02	< 0.01 0.02	RAGML206-01 GM073-12HA

Table 13 Fluopyram residues in cotton seed and gin byproducts from supervised trials in the USA involving fluopyram seed treatments (FS formulation) in combination with in-furrow soil applications (SC formulations)

COTTON SEED Country, Year Location (Variety)	Application			Matrix	DALA	Fluopyram residues (mg/kg)		Reference
	no.	kg ai/ha	mg ai/seed				mean	
USA, 2012 Chula, GA (FM 1740)	1+ 1	0.074 0.261	0.5	seed gin by-products	136	< 0.01, < 0.01 0.064, 0.05	< 0.01 0.057	RAGML206-01 GM022-12HA
USA, 2012 Parma, MO (ST4145 LLB2)	1+ 1	0.073 0.252	0.5	seed gin by-products	181	< 0.01, < 0.01 0.035, 0.027	< 0.01 0.031	RAGML206-01 GM023-12HA
USA, 2012 Proctor, AR (ST4145)	1+ 1	0.073 0.252	0.5	seed gin by-products	153	< 0.01, < 0.01 0.018, 0.021	< 0.01 0.02	RAGML206-01 GM024-12HA
USA, 2012 Greenville, MS, (ST 5458 (B2RF))	1+ 1	0.074 0.25	0.5	seed	144	< 0.01, < 0.01	< 0.01	RAGML206-01 GM025-12HA
USA, 2012 Claude, TX, (ST 4145)	1+ 1	0.074 0.257	0.5	seed gin by-products	194 200 206 213 219 194 200 206 213 219	< 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	RAGML206-01 GM027-12HA
USA, 2012 Levelland, TX (ST5458 (B2RF))	1+ 1	0.074 0.25	0.5	seed gin by-products	158	< 0.01, < 0.01 0.017, 0.015	< 0.01 0.016	RAGML206-01 GM028-12HA
USA, 2012 Hinton, OK (FM1740 B2RF)	1+ 1	0.074 0.252	0.5	seed	142	< 0.01, < 0.01	< 0.01	RAGML206-01 GM029-12HA
USA, 2012 Wall, TX (FM1740 B2RF)	1+ 1	0.072 0.248	0.5	seed	151	< 0.01, < 0.01	< 0.01	RAGML206-01 GM030-12HA
USA, 2012 Sanger, CA (Acala)	1+ 1	0.072 0.248	0.5	seed	170	< 0.01, < 0.01	< 0.01	RAGML206-01 GM031-12HA
USA, 2012 Madera, CA (Acala)	1+ 1	0.074 0.255	0.5	seed	170	< 0.01, < 0.01	< 0.01	RAGML206-01 GM033-12HA

COTTON SEED Country, Year Location (Variety)	Application			Matrix	DALA	Fluopyram residues (mg/kg)		Reference
	no.	kg ai/ha	mg ai/seed				mean	
USA, 2012 East Bernard, TX (ST 5458 (B2RF))	1+ 1	0.073 0.25	0.5	seed gin by-products	143	< 0.01, < 0.01 0.031, 0.03	< 0.01 0.03	RAGML206-01 GM073-12HA

Primary feed commodities

Legume animal feeds

Bean fodder and forage

In the European outdoor field trials on beans evaluated by the Meeting, two applications of fluopyram (SC 500 formulations) were made 7–8 days apart as foliar sprays using knapsack or wheel barrow sprayers with 1–4 flat-fan, solid or hollow-cone nozzles or hand-held-booms (3–12 flat-fan nozzles), applying 0.25 kg ai/ha in 300–1000 L water/ha to plots ranging from 8–108 m^2.

Unreplicated samples of 1–3 kg of pods (including seeds) and at least 12–18 bean plants (min 1 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 456 days before analysis for fluopyram using LC/MS/MS Methods 00984 or 00984/M001. The reported LOQs were 0.01 mg/kg for each analyte. Mean fluopyram recovery rates ranged from 87–98% in fresh pods spiked with 0.01–4.0 mg/kg, 85–100% in vines spiked with 0.01–10 mg/kg and 101% in seeds (fresh) spiked with 0.01–0.1 mg/kg.

Table 14 Residues in bean forage from supervised trials in Europe, evaluated by the 2010 JMPR [Ref: JMPR 2010 E, Table 144, pp 1567–71]

BEAN FORAGE Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Germany, 2006 Lampertheim (Albani)	2	0.25	0.05	500	vines	–0 0 3 7 10 14	0.24 3.2 4.1 <u>0.71</u> 0.4 0.26	RA-2594/06 0377-06
Germany, 2006 Langenfeld-Reusrath (Classic)	2	0.25	0.0835	300	vines	–0 0 3 7 10 14	0.25 7.8 0.42 <u>0.24</u> 0.17 0.08	RA-2594/06 0654-06
Netherlands, 2006 Zwaagdijk-Oost (Unknown)	2	0.25 0.23	0.05	500 460	vines	–0 0 3 7 10 14	0.33 5.7 2.6 <u>0.88</u> 0.55 0.37	RA-2594/06 0655-06
Belgium, 2006 Villers-Perwin (Polder)	2	0.25	0.0385	650	vines	–0 0 3 7 10 14	0.98 14 8 <u>1.3</u> 0.99 0.99	RA-2594/06 0656-06

BEAN FORAGE Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Belgium, 2007 Villers-Perwin (Cadillac)	2	0.25	0.025	1000	vines	–0 0 3 7 10 14	0.37 7 4.2 <u>0.42</u> 0.33 0.19	RA-2511/07 0014-07
Germany, 2007 Langenfeld-Reusrath (Classic)	2	0.25	0.0415	600	vines	–0 0 3 7 10 14	0.14 3 1.4 <u>0.57</u> 0.37 0.2	RA-2511/07 0546-07
France, 2007 Fresnoy les Roye (Lugos)	2	0.25	0.05	500	vines	–0 0 3 7 10 14	0.37 6.2 3.4 <u>0.68</u> 0.34 0.17	RA-2511/07 0547-07
Netherlands, 2007 Biddinghuizen (Cadillac)	2	0.25	0.05	500	vines	–0 0 3 7 10 14	0.56 5.4 1.8 <u>0.72</u> 0.65 0.46	RA-2511/07 0548-07
Germany, 2007 Swisttal-Heimerzheim (Sonesta)	2	0.25	0.0415	600	vines	–0 0 3 6 10 13	0.2 5.9 5.4 <u>0.31</u> 0.18 0.09	RA-2511/07 0549-07
Spain, 2006 Alginet (Cleo)	2	0.25	0.0415	600	vines	–0 0 3 7 10 14	0.25 3.0 2.9 <u>1.6</u> 1.3 1.2	RA-2595/06 0378-06
Italy, 2006 Pradelle di Nogarole Rocca (Jamaica)	2	0.25	0.05	500	vines	–0 0 3 7 10 14	0.22 7.7 0.42 <u>0.34</u> 0.26 0.2	RA-2595/06 0620-06
Spain, 2006 Malgrat de Mar (Nasao)	2	0.25	0.05 0.0415	500 600	vines	–0 0 2 7 10 14	0.18 3.9 0.81 <u>0.25</u> 0.19 0.13	RA-2595/06 0657-06
Italy, 2006 Ladispoli (Bronco)	2	0.25	0.0315	800	vines	–0 0 3 7 10 14	0.51 5.8 4.1 0.84 <u>0.86</u> 0.39	RA-2595/06 0658-06

BEAN FORAGE Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
France, 2007 Chazay d'azergues (Contender)	2	0.25	0.025	1000	vines	–0 0 3 7 10 14	0.52 8.2 5.7 0.61 0.69 <u>0.8</u>	RA-2512/07 0035-07
Italy, 2007 Ladispoli (Bronco)	2	0.25	0.0315	800	vines	–0 0 3 7 10 14	0.28 8.9 0.58 <u>0.26</u> 0.19 0.1	RA-2512/07 0550-07
Spain, 2007 Alginet (Cleo)	2	0.25	0.025	1000	vines	–0 0 3 7 10 14	2.6 5.9 3.7 <u>4.3</u> 2.4 1.2	RA-2512/07 0551-07
Portugal, 2007 Ribafria Peniche (Tradicional)	2	0.25	0.025	1000	vines	–0 0 3 7 10 14	1.7 7.8 3.4 2.2 <u>2.8</u> 2.0	RA-2512/07 0552-07

Table 15 Fluopyram residues in bean forage from supervised outdoor trials in Europe involving two foliar applications of fluopyam (SC formulations)

BEAN FORAGE Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Germany, 2012 Werl-Mawicke (Primel bean)	2	0.2	0.067	300	vines	–0 0	1.7 10	12-2030 12-2030-01
France (N), 2008 Picardie (Flavert) SC500 formulation A	2	0.25	0.05	500	vines	–0 0 3 7 10 14	0.19 3.52 3.57 <u>0.55</u> 0.33 0.30	08-2034 08-2034-01
France (N), 2008 (Flavert) SC500 formulation B	2	0.25	0.05	500	vines	–0 0 3 7 10 14	0.13 3.75 3.78 <u>0.58</u> 0.32 0.27	08-2034 08-2034-01
Italy, 2008 Lazio (Bronco) Kidney bean SC500 formulation A	2	0.25	0.031	800	vines	–0 0 3 7 10 14	0.65 7.7 4.6 <u>0.82</u> 0.67 0.41	08-2096 08-2096-01

BEAN FORAGE Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Italy, 2008 Lazio (Bronco) Kidney bean SC500 formulation B	2	0.25	0.031	800	vines	–0 0 3 7 10 14	0.59 6.5 5.4 <u>0.85</u> 0.73 0.36	08-2096 08-2096-01
Germany, 2010 Heimerzheim (Orinoko)	2	0.2	0.067	300	vines	–0 0 8	0.63 5.5 <u>0.38</u>	10-2125 10-2125-01
Belgium, 2010 Villers-Perwin (Beaufort)	2	0.2	0.05	400	vines	–0 0	0.07 0.64	10-2125 10-2125-02

Pea vines and hay

In the European trials on <u>peas</u> evaluated by the Meeting, two applications of fluopyram (SC formulation) were made to peas 7–9 days apart as foliar sprays using knapsack or wheel barrow sprayers with hand-held spray booms (1–12 flat-fan or hollow cone nozzles), applying 0.2–0.25 kg ai/ha in 300–600 L water/ha. Plot sizes in these trials ranged from 24–160 m^2.

Unreplicated samples of at 0.5–4 kg whole plants (including pods and seeds but without roots), at least 1 kg of fresh pods and vines (without pods and roots) and at least 0.5 kg of dry seeds and straw were taken from each plot, frozen within 24 hours of sampling and stored at –18 °C or below for up to 467 days before analysis for fluopyram and metabolites using LC/MS/MS Method 00984 or 00984/M003. The reported LOQs were 0.01 mg/kg for each analyte and average fluopyram recovery rates ranged from 100–101% in plants spiked with 0.01–10 mg/kg, 93–101% in vines spiked with 0.01–20 mg/kg, 87–100% in seeds (fresh and dry) spiked with 0.01–1.0 mg/kg and 89–98% in straw spiked with 0.01–20 mg/kg.

Table 16 Residues in fresh pea vines from supervised trials in Europe evaluated by the 2010 JMPR [Ref: JMPR 2010 E, Table 147, pp 1573–75]

PEA VINES Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Germany, 2006 Machern (Harnaß)	2	0.25	0.079 0.0745	317 336	vines	–0 0 3 7 10 14	1.2 3.4 3.4 <u>3.5</u> 1.8 2.1	RA-2597/06 0380-06
United Kingdom, 2006 Needham (Hawk)	2	0.25	0.0835	300	vines	–0 0 3 7 10 14	0.91 3.9 4.1 <u>3.3</u> 2.7 1.7	RA-2597/06 0722-06
Germany, 2006 Meckenbeuren (Rondo)	2	0.25	0.0835	300	vines	–0 0 3 7 10 13	0.05 4.2 3.8 <u>0.46</u> 0.37 0.24	RA-2597/06 0723-06

PEA VINES Country, Year Location (Variety)	Application				Matrix	DAL A	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Netherlands, 2007 Kopstukken (unknown)	2	0.25	0.0415	600	vines	–0 0 3 7 10 14	0.29 5.7 4.9 <u>1.9</u> 1.8 0.66	RA-2513/07 0036-07
Germany, 2007 Burscheid (Wunder von Kelvedon)	2	0.25	0.0835	300	vines	–0 0 3 7 10 14	2.1 6.6 1.6 <u>1.1</u> 0.7 0.51	RA-2513/07 0553-07
France, 2007 Goyencourt (Arabelle)	2	0.25	0.0835	300	vines	–0 0 3 7 10 14	0.97 7.6 3.8 1.3 1.3 <u>2.3</u>	RA-2513/07 0554-07
Belgium, 2007 Landenne-Sur-Meuse (Tristar)	2	0.25	0.0625	400	vines	–0 0 3 7 10 14	0.31 2.5 2.4 <u>0.81</u> 0.7 0.45	RA-2513/07 0555-07
Germany, 2007 Swisttal- Heimerzheim (Spring)	2	0.25	0.0835	300	vines	–0 0 3 7 10 13	0.58 6.7 7.1 <u>4.3</u> 2.5 1.1	RA-2513/07 0556-07
Spain, 2006 Brenes (Rondo)	2	0.25	0.0835	300	vines	–0 0 3 7 9 14	4.5 8.8 8 <u>6.6</u> 4.6 5.7	RA-2598/06 0381-06
Italy, 2006 Migliarino (Agami)	2	0.25	0.0625	400	vines	–0 0 3 7 9 14	0.21 5.8 0.24 <u>0.2</u> 0.15 0.12	RA-2598/06 0724-06
France, 2007 Chazay d'Azergues (Douce de provence)	2	0.25	0.0625	400	vines	–0 0 3 7 10 14	0.75 7.9 6 <u>0.93</u> 0.51 0.35	RA-2514/07 0037-07
Spain, 2007 Brenes (Rondo)	2	0.25	0.0625	400	vines	–0 0 3 7 10 14	0.3 3.4 2.2 <u>0.45</u> 0.39 0.24	RA-2514/07 0557-07

Table 17 Residues in pea vines and hay from supervised field trials in Europe involving two applications of fluopyram (SC formulations)

PEA VINES/HAY Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Spain, 2012 Salobrena Granada (Utrillo)	2	0.2	0.05	400	vines straw	–0 0 6 14 22	0.46 0.93 <u>0.5</u> 0.4 <u>6.3</u>	12-2155 12-2155-01
Spain, 2012 Malaga (Utrillo)	2	0.2	0.05	400	vines straw 	–0 0 7 14 20 34	0.1 0.35 <u>0.14</u> 0.14 2.0 <u>3.4</u>	12-2155 12-2155-02
Italy, 2012 Papiana Marsciano (Gran Rugoso Tondo)	2	0.2	0.04	500	vines straw 	–0 0 7 14 21 28	0.51 0.79 <u>0.92</u> 0.89 <u>19</u> 15	12-2155 12-2155-03
Southern France, 2012 Lapalud (Isard)	2	0.2	0.05	400	vines straw	–0 0 6 14 21 40	0.11 3.9 <u>3.2</u> 0.45 0.43 <u>0.33</u>	12-2032 12-2032-01
Spain, 2012 Dos Hermanas (Cartouche)	1+ 1	0.2 0.19	0.067 0.067	300 282	vines straw	–0 0 7 14 21	6.2 13 3.9 <u>4.9</u> <u>3.6</u>	12-2032 12-2032-02
Italy, 2012 Ladispoli (RM) (Attika)	2	0.2	0.067	300	vines straw	–0 0 7 14 21	0.28 3.8 1.2 <u>2.1</u> <u>3.6</u>	12-2032 12-2032-03
Greece, 2012 Nea Messimvria (Li Violetta)	2	0.2	0.05	400	vines straw	–0 0 7 14 21 33	0.24 3.8 <u>1.7</u> 0.71 0.51 <u>0.82</u>	12-2032 12-2032-04
Spain, 2012 Alginet (Lincoln)	2	0.2	0.04	500	vines straw	–0 0 7 14 21	2.8 6.3 4.5 <u>8.4</u> <u>0.64</u>	12-2032 12-2032-05

PEA VINES/HAY Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Germany, 2012 Burscheid (Respect)	2	0.2	0.067	300	vines straw	–0 0 7 14 21 39	0.8 3.3 <u>0.46</u> 0.43 0.36 <u>0.44</u>	12-2031 12-2031-01
France (N), 2012 Chaussy (Genial)	2	0.2	0.067	300	vines straw	–0 0 7 14 21 35	0.13 3.0 <u>0.54</u> 0.49 0.29 <u>0.44</u>	12-2031 12-2031-02
Germany, 2012 Beucha-Wolfshain (Rocket)	2	0.2	0.067	300	vines straw 	–0 0 7 14 21 43	0.4 2.4 <u>0.28</u> 0.13 0.095 <u>0.15</u>	12-2031 12-2031-03
Belgium, 2012 Villers-Perwin (Ravenna)	2	0.2	0.05	400	vines straw	–0 0 7 14 21 37	1.6 3.4 <u>0.4</u> 0.23 0.21 <u>0.8</u>	12-2031 12-2031-04
United Kingdom, 2012 Cambridge (Tommy)	2	0.2	0.067	300	vines straw	–0 0 6 13 20	0.19 6.1 <u>8.0</u> 4.2 <u>4.8</u> (c=0.01)	12-2031 12-2031-05
Germany, 2012 Langförden (Alvesta)	2	0.2	0.067	300	vines straw	–0 0 7 13 22 32	0.7 2.9 0.56 <u>0.63</u> 0.6 <u>0.93</u> (c=0.029)	12-2031 12-2031-06
Spain, 2012 Salobrena (Utrillo)	2	0.25	0.063	400	vines straw	–0 0 6 14 22	4.9 12 6.2 <u>9.2</u> <u>11</u> (c=0.013)	12-2159 12-2159-01
Italy, 2012 Zibido San Giacomo (Utrillo)	2	0.25	0.083	300	vines straw	–0 0 7 14 21 28	7.6 13 <u>4.0</u> 2.9 3.9 <u>7.2</u>	12-2159 12-2159-02

PEA VINES/HAY Country, Year Location (Variety)	Application				Matrix	DALA	Residues (mg/kg)	Reference
	no.	kg ai/ha	kg ai/hL	water (L/ha)			Fluopyram	
Spain, 2013 Alginet (Lincoln)	2	0.25	0.05	500	vines straw	–0 0 7 13 21	3.1 11 9.5 <u>9.6</u> <u>18</u>	12-2048 12-2048-01
Spain, 2013 Dos Hermanas (Cartouche)	2	0.25	0.05	500	vines straw	–0 0 7 14 21	6.9 15 5.3 <u>7.7</u> <u>4.9</u>	12-2048 12-2048-01

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 JMPR in 2012 and 2014 JMPRs 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.

Results of supervised residue trials on crops

The Meeting received new supervised trial data for foliar applications of fluopyram (SC formulations, generally in combinations with other fungicides) on tomatoes, beans, peas, and sunflower and for seed treatments or in-furrow soil treatments on soya bean and cotton. The Meeting also noted that data for some of these crops had been provided to the 2010 JMPR.

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. Frozen sample storage times in the new trials were within the storage intervals considered acceptable by the 2010 JMPR and the analytical methods used in these trials were the same as those evaluated by JMPR in 2010.

Fruiting vegetables (except Cucurbits)

Tomato

The Meeting was advised that new GAP exists in Greece for fluopyram on protected tomatoes, involving up to three foliar applications of 0.15 kg ai/ha with a 3-day PHI.

In four independent protected tomato trials matching this GAP in Greece, residues were 0.04, 0.07, 0.08 and 0.13 mg/kg.

New GAP was also provided for tomatoes in Ukraine, up to two foliar applications of 0.15 kg ai/ha with a 7 day PHI.

In eight independent trials on field tomatoes conducted in Europe and matching this GAP in Ukraine, fluopyram residues were: < 0.01, 0.02, 0.06, 0.07, 0.1, 0.13, 0.13 and 0.17 mg/kg.

The Meeting noted that the 2010 JMPR had recommended a fluopyram maximum residue level of 0.4 mg/kg based on trials on protected tomatoes where residues had been proportionally adjusted to the GAP in Morocco.

The Meeting agreed that the 2010 JMPR recommendations accommodated the new GAPs for tomatoes in Greece and Ukraine.

Peppers and eggplant

The Meeting noted that the new GAP in Greece for fluopyram on protected tomatoes (3× 0.15 kg ai/ha, 3-day PHI) also applied to protected peppers and eggplants and that the 2012 JMPR had recommended a maximum residue level of 0.5 mg/kg for peppers based on the GAP in Turkey.

No trials matching the GAP in Greece on peppers and eggplants were available. The Meeting noted that the previous trials on protected peppers provided to the 2010 and 2012 JMPRs all involved only two applications and application rates of either 0.06 kg ai/ha or 0.3 kg ai/ha and agreed that the proportionality approach could not be used to support revised recommendations for peppers and/or extrapolation to eggplants.

Legume vegetables

Beans (except broad bean and soya bean)

The critical GAP for beans in Netherlands and Belgium is for up to two foliar applications of 0.25 kg ai/ha, with a 7-day PHI and results from supervised trials from Europe on protected and outdoor beans were provided to the Meeting to supplement the data provided to the 2010 JMPR.

In nine independent trials on protected beans matching this critical GAP, fluopyram residues were: 0.07, 0.15, 0.16, 0.16, 0.2, 0.22, 0.22, 0.43 and 0.69 mg/kg in beans with pods.

In 32 independent trials on outdoor beans conducted in Europe and matching this critical GAP, fluopyram residues were: **< 0.01, < 0.01, 0.01,** 0.03, **0.04,** 0.04, **0.05**, 0.05, 0.07, **0.08, 0.08**, **0.09, 0.1**, 0.1, 0.11, 0.11, 0.12, **0.14**, **0.15**, **0.17**, **0.17**, 0.17, 0.18, 0.19, 0.2, 0.21, 0.24, 0.24, 0.25, 0.26, **0.32** and 0.43 mg/kg in beans with pods. (Results in bold are from the new trials).

The Meeting noted that the data sets for protected and outdoor bean were statistically different and agreed to use the data from the trials on protected beans to estimate a maximum residue level of 1 mg/kg, an STMR of 0.2 mg/kg and an HR of 0.69 mg/kg for fluopyram on beans (except broad bean and soya bean).

Peas, shelled

The critical GAP for peas (without pods) in Netherlands and Belgium is for up to two foliar applications of 0.25 kg ai/ha, with a 7-day PHI and results from supervised trials from Europe on outdoor peas were provided to the Meeting to supplement the data provided to the 2010 JMPR.

In 30 independent trials conducted in Europe and matching this critical GAP, fluopyram residues were: **< 0.01, < 0.01, < 0.01**, < 0.01, **0.01, 0.01**, 0.01, **0.02, 0.02, 0.02**, 0.02, 0.02, 0.02, 0.02, **0.03, 0.03**, 0.03, 0.03, **0.04**, 0.05, 0.05, 0.05, **0.05**, **0.06, 0.06**, **0.06**, **0.09, 0.09**, 0.1 and **0.12** mg/kg in peas without pods. (Results in bold are from the new trials).

The Meeting estimated a maximum residue level of 0.2 mg/kg, an STMR of 0.03 mg/kg and an HR of 0.12 mg/kg for fluopyram on peas, shelled.

Beans, shelled

The Meeting noted that the GAP for beans in Netherlands and Belgium (up to two foliar applications of 0.25 kg ai/ha, with a 7-day PHI) was for beans with and without pods, and since this GAP for beans was the same as for peas, the Meeting agreed to extrapolate the data from peas, shelled to beans, shelled.

The Meeting estimated a maximum residue level of 0.2 mg/kg, an STMR of 0.03 mg/kg and an HR of 0.12 mg/kg for fluopyram on beans, shelled.

Soya bean (dry)

Results from supervised trials from the USA on soya beans were provided to the Meeting. In 21 independent trials matching the GAP in the USA for use as a seed treatment (0.25 mg ai/seed) fluopyram residues were < 0.01 (12), 0.01, 0.01, 0.01, 0.02, 0.02, 0.03, 0.03 and 0.03 mg/kg in dry soya beans.

The Meeting noted that the metabolism studies did not cover the use of fluopyram as a seed treatment. However the Meeting noted the 2010 JMPR conclusions that fluopyram is slowly degraded in soil and when present, is the major residue in 30-day PBI rotational crops and agreed that the established residue definitions would also cover the use of fluopyram as a seed treatment.

The Meeting estimated a maximum residue level of 0.05 mg/kg and an STMR of 0.01 mg/kg for fluopyram on soya bean (dry).

Oilseeds

Sunflower seed

Results from supervised trials from Europe on sunflowers were provided to the Meeting.

The critical GAP in Ukraine and Moldovia is for up to two foliar sprays of 0.125 kg ai/ha, applied before the start of flowering (BBCH 57) and with a minimum PHI of 50 days.

The Meeting received results from 12 independent trials conducted in Europe, where two foliar sprays of 0.125–0.13 kg fluopyram/ha were applied up to the seed development or early ripening stages (BBCH 67–85). As these trials did not match the critical GAP, the Meeting did not recommend a maximum residue level for fluopyram on sunflower seed.

Cotton seed

Results from supervised trials from the USA on cotton were provided to the Meeting. These trials included separate plots where fluopyram was applied as a pre-plant seed treatment, or as as a combination of a seed treatment and an in-furrow soil treatment at planting.

In the USA, GAP exists for the use of fluopyram as a pre-plant seed treatment (0.35 mg ai/seed) and also as an in-furrow soil treatment of 0.25 kg ai/ha.

In the plots from 11 independent trials where the seed was treated with 0.5 mg/kg/seed (1.4× GAP), fluopyram residues in cotton seed were all < 0.01 mg/kg (n=11) and in the plots treated with a seed treatment (0.5 mg ai/seed—1.4× GAP) followed by an in-furrow soil treatment matching the US GAP (0.25 kg ai/ha) residues in cotton seed were also < 0.01 mg/kg.

The Meeting noted that the US GAP did not exclude the use of both a seed treatment and an in-furrow treatment at planting, and since residues following the seed treatment + in-furrow soil treatment were all < 0.01 mg/kg (n=11), the Meeting estimated a maximum residue level of 0.01 mg/kg and an STMR of 0.01 mg/kg for fluopyram on cotton seed.

Animal feeds

Bean forage

In 22 of the European trials on outdoor beans evaluated by the Meeting, residues of fluopyram in fresh bean forage from trials matching the GAP in Belgium and Netherlands (two applications of 0.25 kg ai/ha, PHI 7 days) were: 0.24, 0.25, 0.26, 0.31, 0.34, **0.38**, 0.42, **0.55**, 0.57, **0.58**, 0.68, 0.71, 0.72, 0.8, **0.82**, **0.85**, 0.86, 0.88, 1.3, 1.6, 2.8 and 4.3 mg/kg (Results in bold are from the new trials).

The Meeting estimated a median residue of 0.7 mg/kg (fresh weight) and a highest residue of 4.3 mg/kg (fresh weight) for fluopyram on bean forage.

Cotton gin by-products

In the trials from the USA on cotton, residues of fluopyram were measured in gin by-products from six plots that were treated with a combination of a seed treatment (at 1.4× GAP) and an in-furrow soil treatment (at GAP). Residues in these trials were: < 0.01, 0.02, 0.02, 0.03, 0.03 and 0.06 mg/kg.

The Meeting noted that although the in-furrow soil treatment rates in these trials matched the USA GAP, the seed treatment rates were 1.4× higher than GAP and agreed it was not possible to apply the proportionality approach for this combined treatment regime to derive median and highest residues for calculating the livestock dietary burden.

Pea vines and hay

In 30 of the European trials on outdoor peas evaluated by the Meeting, residues of fluopyram in fresh pea vines from trials matching the GAP in Belgium and Netherlands (two applications of 0.25 kg ai/ha, PHI 7 days) were: **0.14**, 0.2, **0.28**, **0.4**, 0.45, **0.46**, 0.46, **0.5**, **0.54**, **0.63**, 0.81, **0.92**, 0.93, 1.1, **1.7**, 1.9, **2.1**, 2.3, **3.2**, 3.3, 3.5, **4.0**, 4.3, **4.9**, 6.6, **7.7**, **8.0**, **8.4**, **9.2** and **9.6** mg/kg (Results in bold are from the new trials).

The Meeting estimated a median residue of 1.8 mg/kg (fresh weight) and a highest residue of 9.6 mg/kg (fresh weight) for fluopyram on pea vines (green).

Residues of fluopyram in pea hay/straw from the new European trials matching the GAP in Belgium and Netherlands and sampled 20–43 days after the last application were: 0.15, 0.33, 0.44, 0.44, 0.64, 0.8, 0.82, 0.93, 3.4, 3.6, 3.6, 4.8, 4.9, 6.3, 7.2, 11, 18 and 19 mg/kg (n=18).

The Meeting estimated a median residue of 3.5 mg/kg (fresh weight), a highest residue of 19 mg/kg (fresh weight) and after correction for an average 88% dry matter content, estimated a maximum residue level of 40 mg/kg for fluopyram on pea hay.

Animal commodity maximum residue levels

Farm animal feeding studies

The 2010 JMPR reviewed feeding studies with fluopyram on lactating dairy cows and laying hens and the conclusions from these residue transfer studies were used to estimate residue levels of fluopyram and its metabolites in milk, eggs and livestock tissues, based on the above dietary burdens.

Farm animal dietary burden

The Meeting estimated the dietary burden of fluopyram in farm animals on the basis of the diets listed in Annex 6 of the 2009 JMPR Report (OECD Feedstuffs Derived from Field Crops) and using the estimated residues in livestock feed commodities evaluated by the Meeting and by previous JMPRs.

	Animal dietary burden, fluopyram, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0.14	0.13	16	2.4	32 a	7.6 c	0.04	0.04
Dairy cattle	4.5	1.3	21	2.7	25 b	7 d	0.07	0.07
Poultry—broiler	0.041	0.041	0.21	0.12	0.021	0.021	–	–
Poultry—layer	0.041	0.041	5.8 e, g	0.92 f, h	0.021	0.021	–	–

[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

Animal commodity maximum residue levels

The calculations used to estimate total residues for use in estimating maximum residue levels, STMRs and HRs are shown below. For maximum residue level estimation, the total residues are the sum of fluopyram plus BZM (expressed as fluopyram equivalents) and for dietary intake estimation (STMRs and HRs) the total residues are the sum of fluopyram, BZM and total olefins (expressed as fluopyram equivalents).

Cattle

For beef and dairy cattle, the highest maximum dietary burdens were 32 ppm and 25 ppm (dairy) and the mean dietary burdens were 7.6 ppm and 7 ppm (dairy).

	Feed level for milk (ppm)	Total residues in milk (mg/kg)	Feed level for tissues (ppm)	Total residues (mg/kg)			
				Muscle	Liver	Kidney	Fat
MRL beef or dairy cattle (fluopyram + BZM)							
Feeding study [a]	14.4 44	0.25 0.64	14.4 44	0.045 0.83	2.88 6.	0.39 0.93	0.4 0.78
Dietary burden/residue estimate	25	0.38	32	0.52	4.7	0.71	0.63
High residue beef or dairy cattle (fluopyram + BZM + Total olefins)							
Feeding study [a]			14.4 44	0.47 0.86	2.9 6.1	0.41 0.97	0.52 1.2
Dietary burden/residue estimate			32	0.7	4.8	0.74	0.86
STMR beef or dairy cattle ((fluopyram + BZM + Total olefins)							
Feeding study [b]	1.5 14.4	0.02 0.27	1.5 14.4	0.02 0.32	0.35 2	0.03 0.31	0.04 0.31
Dietary burden/residue estimate	7	0.12	7.6	0.16	1.1	0.16	0.17

[a] For estimating highest residues for tissues and mean residues for milk

[b] For estimating mean residues for tissues and for milk

Total residues of fluopyram and BZM (expressed as fluopyram equivalents) calculated in cattle milk and tissues for use in estimating maximum residue levels are: 0.63 mg/kg (fat), 0.52 mg/kg (muscle), 4.7 mg/kg (liver) and 0.71 mg/kg (kidney) and the mean residue for milk is 0.38 mg/kg.

The Meeting estimated maximum residue levels of 0.7 mg/kg for fluopyram in meat (from mammals other than marine mammals), 0.7 mg/kg for mammalian fats (except milk fats), 0.8 mg/kg for edible offal (mammalian) except liver, 5 mg/kg for liver of cattle, goats, pigs and sheep and 0.5 mg/kg for milks and agreed to withdraw the previous recommendations.

Estimated HRs for dietary intake estimation for fluopyram (and including residues of BZM and total olefins) are 0.86 mg/kg for mammalian fat, 0.7 mg/kg for mammalian muscle, 4.8 mg/kg for liver and 0.74 mg/kg for kidney and other edible offal.

Estimated STMRs for dietary intake estimation for fluopyram (and including residues of BZM and total olefins) are 0.17 mg/kg for mammalian fat, 0.16 mg/kg for mammalian muscle, 1.1 mg/kg for liver of cattle, goats, pigs and sheep, 0.16 mg/kg for kidney and other edible offal of cattle, goats, pigs and sheep and 0.12 mg/kg for milks

Poultry

The dietary burdens for poultry broilers are 0.21 ppm (maximum) and 0.12 ppm (mean) but the Meeting decided to estimate residue levels in poultry tissues using the higher maximum and mean dietary burdens in poultry layers (5.8 ppm and 0.92 ppm respectively) as they may also be consumed. Since the dose-response curves in the poultry feeding study showed a linear relationship (R^2 values of 0.97–0.99) and as the maximum dietary burden estimates were not more than 120% of the highest dose, the Meeting agreed to estimate maximum total residues by extrapolation from the results of the poultry feeding study.

	Feed level for eggs (ppm)	Total residues in eggs (mg/kg)	Feed level for tissues (ppm)	Total residues (mg/kg)		
				Muscle	Liver	Skin with Fat
MRL broiler or laying hen (fluopyram + BZM)						
Feeding study [a]	4.8	0.72	4.8	0.33	1.6	0.64
Dietary burden/residue estimate	5.8	0.87	5.8	0.39	1.9	0.75
High residue broiler or laying hen (fluopyram + BZM + Total olefins)						
Feeding study [a]	4.8	0.74	4.8	0.39	1.64	0.72
Dietary burden/residue estimate	5.8	0.8	5.8	0.46	1.9	0.85
STMR broiler or laying hen (fluopyram + BZM + Total olefins)						
Feeding study [b]	0.49 1.6	0.08 0.22	0.49 1.6	0.03 0.09	0.16 0.43	0.06 0.12
Dietary burden/residue estimate	0.92	0.13	0.92	0.058	0.26	0.086

[a] For estimating highest residues for tissues and mean residues for eggs

[b] For estimating mean residues for tissues and for eggs

Combined residues of fluopyram and BZM (expressed as fluopyram equivalents) expected in poultry eggs and tissues for use in estimating maximum residue levels are: 0.75 mg/kg (fat), 0.39 mg/kg (muscle), 1.9 mg/kg (liver) and 0.87 mg/kg (eggs).

The Meeting estimated maximum residue levels of 0.5 mg/kg for fluopyram in poultry meat, 1 mg/kg for poultry fat, 2.0 mg/kg for poultry edible offal and 1.0 mg/kg for eggs.

Estimated HRs for dietary intake estimation for fluopyram (and including residues of BZM and total olefins) are 0.85 mg/kg for poultry fat, 0.46 mg/kg for poultry muscle, 1.9 mg/kg for poultry edible offal and 0.8 mg/kg for eggs.

Estimated STMRs for dietary intake estimation for fluopyram (and including residues of BZM and total olefins) are 0.086 mg/kg for poultry fat, 0.058 mg/kg for poultry muscle, 0.26 mg/kg for poultry edible offal and 0.13 mg/kg for eggs.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed in Annex 1 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.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
VP 0061	Beans, except broad bean and soya bean	1		0.2	0.69
VP 0062	Beans, shelled	0.2		0.03	0.12
SO 0691	Cotton seed	0.01		0.01	
PE 0112	Eggs	1	0.3	0.13	
MO 0098	Kidney of cattle, goats, pigs and sheep	0.8	0.5	0.16	0.74
MO 0099	Liver of cattle, goats, pigs and sheep	5	3	1	4.8
MM 0095	Meat (from mammals other than marine mammals)	0.8	0.5	0.16	0.7
ML 0106	Milks	0.6	0.3	0.12	
VP 0064	Peas, Shelled	0.2		0.03	0.12
AL 0072	Pea hay or pea fodder (dry)	40 (dw)[a]		3.5 (fw)	19 (fw)
PO 0111	Poultry, Edible offal of	2	0.7	0.27	1.9
PM 0110	Poultry meat	0.5	0.2	0.058	0.46
VD 0541	Soya bean (dry)	0.05		0.01	
AL 0528	Pea vines (green)			1.8 (fw)	9.6 (fw)
AL 1030	Bean Forage (green)			0.7 (fw)	4.3 (fw)

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 to the 2015 Report.

The International Estimated Daily Intakes of fluopyram for the 17 GEMS/Food regional diets, based on estimated STMRs were 4–30% of the maximum ADI of 0.01 mg/kg bw (Annex 3). 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 2015 Report).

For fluopyram the IESTI varied from 0–10% 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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Code	Author	Year	Title, Institute, Report reference
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M-473248-01-1	Noss, G & Guerleyen, N	2013	Determination of the residues of AE C656948 in/on field pea after spray application of fluopyram SC 500 in Spain and Italy. Bayer CropScience. Report includes Trial Nos: 12-2159-01, 12-2159-02. Date: 2013-12-11. GLP/GEP: yes, unpublished. 12-2159

Code	Author	Year	Title, Institute, Report reference
M-489607-01-1	Glaubitz, J	2014	Determination of the residues of fluopyram and trifloxystrobin in/on tomato after spray application of AE C656948 & CGA279202 SC 500 in the field in southern France, Spain, Italy, Portugal and Greece. Bayer CropScience. Report includes Trial Nos: 13-2120-01, 13-2120-02, 13-2120-03, 13-2120-04, 13-2120-05, 13-2120-06, 13-2120-07, 13-2120-08. Date: 2014-06-12. GLP/GEP: yes, unpublished. 13-2120
M-487392-01-1	Glaubitz, J & Diehl, P	2014	Determination of the residues of AE C656948 and trifloxystrobin in/on tomato and cherry tomato after spray application of AE C656948 & CGA279202 SC 500 in the greenhouse in Germany, the Netherlands, Belgium, southern France, Spain, Italy, Greece and Portugal. Bayer CropScience. Report includes Trial Nos: 13-2121-01, 13-2121-02, 13-2121-03, 13-2121-04, 13-2121-05, 13-2121-06, 13-2121-07, 13-2121-08. Date: 2014-05-27. GLP/GEP: yes, unpublished. 13-2121
M-456704-02-1	Dallstream, K & Fain, J	2013	Fluopyram 500 SC and fluopyram 400 SC—Magnitude of the residue in cotton (Amended). Bayer CropScience LP, RTP, NC, USA. EPA MRID No: 49242803. Date: 2013-06-18. Amended: 2014-05-12. GLP/GEP: yes, unpublished. RAGML206-01
M-307802-01-1	Beedle, E & Schumacher, B	2008	AE C656948 500 SC—Magnitude of the residue in/on soya bean. Bayer CropScience LP, Stilwell, KS, USA. EPA MRID No: 47567016. Date: 2008-09-24. GLP/GEP: yes, unpublished. RAGMP039
M-454914-01-1	Lenz, C & Netzband, D	2013	Fluopyram 500 SC—Magnitude of the residue in soya beans. Bayer CropScience LP, Stilwell, KS, USA. EPA MRID No: 49006006. Date: 2013-05-30. GLP/GEP: yes, unpublished. RAGMY006

FLUTRIAFOL (248)

First draft prepared by Dr D.J. MacLachlan, Department of Agriculture and Water Resources, Canberra, Australia

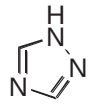
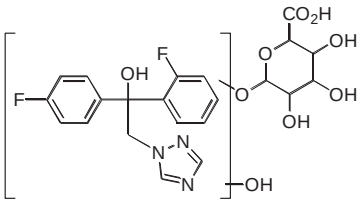
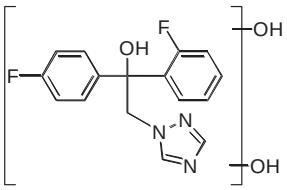
EXPLANATION

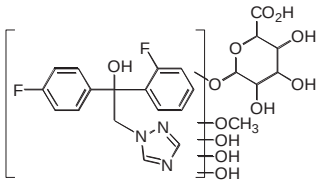
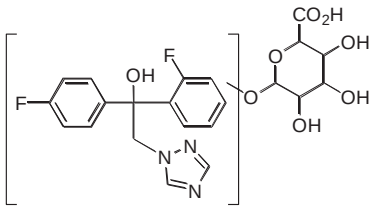
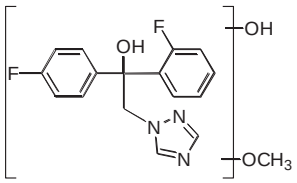
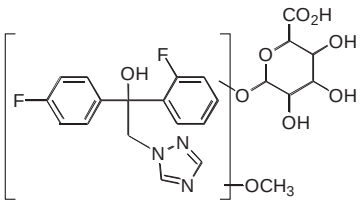
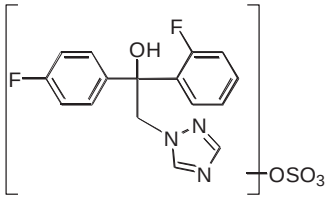
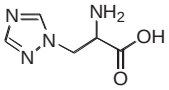
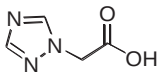
Flutriafol is a triazole fungicide used in many crops for control of a broad spectrum of leaf and ear cereal diseases, particularly embryo borne diseases e.g., bunts and smuts. The Meeting received information on identity, animal and plant metabolism, environmental fate in soil, rotational crops, analytical methods, storage stability, use patterns, supervised trials, farm animal feeding studies and fates of residues in processing. It was first evaluated for residues and toxicology by the 2011 JMPR. The ADI of flutriafol was 0–0.01 mg/kg bw and the ARfD was 0.05 mg/kg bw. The compound was listed by the 46th Session of CCPR for the JMPR to consider additional MRLs. The residue definition for compliance with MRL and for estimation of dietary intake (for animal and plant commodities) is flutriafol.

For the current evaluation the Meeting received new metabolism studies in lactating goats, storage stability data for animal commodities, residue trials on apples, pears, peaches/nectarines, plums, cherries, strawberries, Brassica vegetables (cabbages and broccoli), cucurbits (cucumbers, summer squash and muskmelons), tomatoes, peppers, leafy vegetables (lettuce, spinach, celery and mustard greens), sugar beets, maize, rice, sorghum, almonds, pecans, cotton, and rape, as well as a lactating cow feeding study (residue transfer study).

The chemical structures of the major degradation compounds from the metabolism of flutriafol are provided below.

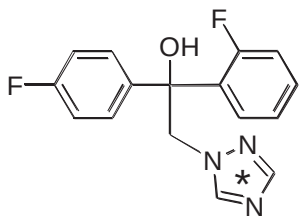
List of metabolites in this evaluation:

Code	Compound	Structure	
M1 T	1,2,4-triazole		
M3	hydroxyl flutriafol glucuronide		
M3e	dihydroxy flutriafol		

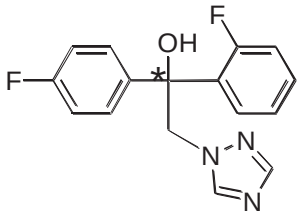
Code	Compound	Structure	
M3e-f1	trihydroxymethoxy flutriafol glucuronide		
M4	flutriafol glucuronide		
M5	hydroxymethoxy flutriafol		
M7	methoxy flutriafol glucuronide		
M10	flutriafol sulfate		
TA	1,2,4-triazole analine		
TAA	1,2,4-triazole acetic acid		

METABOLISM

La Mar (2012 2470) studied the metabolism of flutriafol in lactating goats.



Triazole-label



Carbinol-label

Two lactating goats (crossbreeds, 2–4 years old, 35 and 41 kg bw) were administered either [triazole-3(5)-^{14}C]-flutriafol or [carbinol-^{14}C]-flutriafol by capsule once daily in the morning for five consecutive days at a rate equivalent to 12.0 ppm in the feed (triazole) or 12.2 ppm (carbinol). Animals were fed 1.5 kg goat chow and 1 kg alfalfa hay daily. Milk production during the study averaged 0.54 L/day and 0.65 L/day respectively for the two goats. Excreta were collected once a day (in the morning, before dose administration). Milk was collected twice daily (morning and evening). The goats were sacrificed approximately 20–22 h after the last dose was administered and the following tissues were collected at necropsy—liver, kidney, muscle (loin and flank), fat (subcutaneous, omental and renal), bile, blood and gastrointestinal tract with contents. Analytical work was completed within 30 days after sacrifice.

The majority of the administered dose was recovered in the faeces (60–69%) with 31.5–40.6% excreted in urine and 0.05–0.07% in milk (Table 1). The amount of administered radioactivity found in tissues was 0.35–0.45% while the gastrointestinal tract and contents contained 2.5–7.1% giving a total recovery of administered radioactivity of 103–110%. TRR in edible tissues were generally low (0.002–0.01 mg equiv/kg) with the exception of liver (0.264–0.305 mg equiv/kg) and kidney (0.035–0.061 mg equiv/kg).

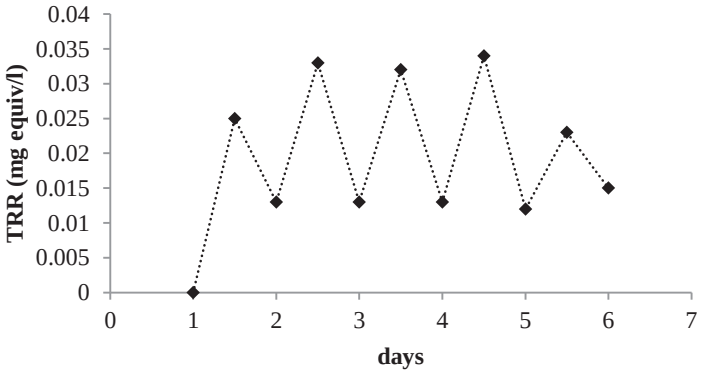
Table 1 Distribution of TRR following dosing of [^{14}C]flutriafol at 12 ppm for 5 days

	Triazole-label		Carbinol-label	
	%AD	mg equiv/kg	%AD	mg equiv/kg
Tissues				
Liver	0.34	0.305	0.27	0.264
Kidney	0.01	0.061	< 0.01	0.035
Omental fat	< 0.01	0.004	< 0.01	0.002
Subcutaneous fat	< 0.01	0.005	< 0.01	0.003
Renal fat	< 0.01	0.004	< 0.01	0.002
Flank muscle	< 0.01	0.01	< 0.01	0.004
Loin muscle	0.01	0.01	< 0.01	0.004
Blood	–	0.022	–	0.009
Excreta/secretions				
Faeces	60.0		69.0	
GIT and contents	7.12		2.5	
Urine	40.6		31.5	
Whole milk	0.05	–	0.06	–
Bile	0.04	1.33	0.02	0.687
Cage wash	0.01		0.2	
Total	110.2		103.4	

Residues in milk appeared to reach plateau levels by Day 3 of dosing, with significant differences in ^{14}C levels between milk collected in the morning (low levels) compared to evening

milk (higher levels), suggesting flutriafol residues are rapidly eliminated following dosing (Figure 1).

A



B

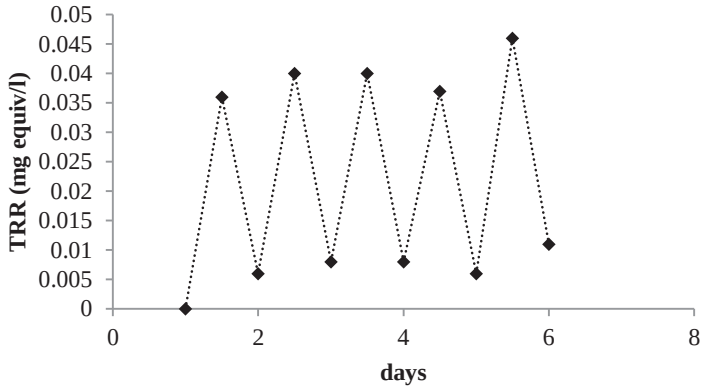


Figure 1 TRR in milk for goats dosed at the equivalent of 12 ppm in the feed with flutriafol (A) triazole label, (B) carbinol label

Acetonitrile and water extraction (2× CH_3CN/H_2O, 1× CH_3CN) of liver, kidney and in the case of the triazole-label also composite muscle, resulted in extraction efficiencies of 25.5–27.5% (liver), 67.7–79.7% (kidney) and 90% (muscle) (Table 2). The CH_3CN/H_2O extracts were concentrated, acidified (0.1% formic acid) and then partitioned with ethyl acetate to give aqueous/acetonitrile (aqueous) and ethyl acetate (organic) phases. Muscle from the carbinol-label and fat (both labels) were not subject to further analysis as the TRR levels were insignificant (< 0.01 mg eq/kg).

Radioactivity in PES of liver and kidney was characterized further. Samples of PES were treated with 1 M HCl in CH_3CN/H_2O (1:1) followed by 1 M KOH in H_2O. Sub-samples of liver PES were also treated with and without pepsin in 0.1 M HCl/glycine buffer pH 2.2 at 37 ºC overnight, followed by treatment with and without pancreatin and bile extract in 0.1 M sodium bicarbonate overnight at 37 ºC. Any remaining radioactivity was solubilised by treatment with 24% KOH.

Milk samples (whole milk) with the highest residue present (typically Day 4, pm) were separated into milk fat and skim milk for extraction. Protein was precipitated from skim milk by adding acetone and chilling in an ice bath. The protein pellet was then extracted with acetone/H_2O (1:1) followed by acetone. Skim milk and protein pellet extracts were combined, concentrated, acidified (0.1% formic acid) and then partitioned with ethyl acetate. Milk fat was extracted with acetone/hexane 1:4 (2×) and acetone (1×). Solids were separated by centrifugation and fat extracts were then concentrated to remove acetone, and partitioned with acetonitrile.

For the TZ label, extraction of liver with CH_3CN/H_2O released M1 (2.9% TRR), M2 (1.5% TRR), M3 (2.6% TRR), M3e (1.8% TRR), M5 (4.7% TRR) and flutriafol (1.5% TRR). The total identified residues in the liver accounted for 13.5% of TRR. A number of unidentified compounds (10% TRR) were observed that were individually present at $\leq$ 2.9% TRR ($\leq$ 0.008 mg equiv/kg). Hydrolysis of the liver PES under mild acid and alkaline conditions released all of the remaining ^{14}C residues which were able to be resolved into more than six peaks by chromatography. Subsequent treatment of the hydrolysis extracts with enzymes to release conjugates did not result in additional compounds being identified; largest individual component 9.8% TRR.

In kidneys the main ^{14}C residue components were 1,2,4-triazole (M1, 10% TRR), M2 (10% TRR), hydroxyl flutriafol glucuronide (M3, 30% TRR) and dihydroxy flutriafol (M3e, 3.4% TRR). No other single metabolite comprised more than 10% of TRR (0.006 mg equiv/kg).

Residues in skim milk were extracted with acetonitrile and water. Main components identified were 1,2,4-triazole (M1, 26.5% TRR), M2 (2.9% TRR), hydroxyl flutriafol

glucuronide (M3, 23.5% TRR) and dihydroxy flutriafol (M3e, 17.6% TRR). No other single metabolite comprised more than 8.8% of TRR (0.003 mg equiv/kg).

Residues in milk fat were extracted with acetone/hexane. Main components identified were 1,2,4-triazole (M1, 13.8% TRR), dihydroxyl flutriafol (M3e, 37.9% TRR) and flutriafol (3.4% TRR). No other single metabolite comprised more than 6.9% of TRR (0.002 mg equiv/kg).

Table 2 Characterisation and identification of ^{14}C residues in tissues and milk of a goat dosed at 12 ppm with triazole-label

Matrix	Liver	Kidney	Skim Milk	Milk Fat	Flank muscle [c]
TRR (ppm)	0.274	0.059	0.034	0.029	0.01
			%TRR		
Solvent extracts [a]	25.5	79.7	97.1	86.2	90.0
Aqueous soluble [b]	12.4	66.1	70.6	79.3 (CH_3CN)	70.0
M1	2.9	10.2	26.5	13.8	40.0
M2	1.5	10.2	2.9		10.0
M3 [d]	2.6	30.5	23.5		10.0
M3e				37.9	
Flutriafol				3.4	
Unknowns	4 (2)	15.3 (2)	11.7 (2)	13.8 (2)	10 (1)
Organic soluble [b]	13.1	13.6	26.5	6.9 (hexane)	20.0
M3e	1.8	3.4	17.6		
M5	4.7				
Flutriafol	1.5		< 2.9		
Unknowns	4.4 (2)	10.2 (4)	< 2.9 (1)		
PES	74.4	20.4	2.9	13.8	10.0
Released by 1 N HCl	3.6	1.7			
Released by 1 N KOH	15.7	5.1			
Overall					
Extracted [d]	100[D]	83.5[D]	97.1	86.2	90.0
identified	13.5	44.1	< 70.5	55.1	50.0
characterized	86.0	42.5	< 17.5	20.7	40.0
Unextracted [d]	0.0	13.6	2.9	13.8	10.0

[a] Solvent systems: CH_3CN/H_2O for liver, kidney, skim milk and muscle; acetone/hexane for fat and milk fat

[b] Represents free residues from partition of initial extracts with ethyl acetate. (Aqueous is CH_3CN phase and organic is hexane phase for milk fat)

[c] Extraction and analysis data represent composite of flank and loin muscle

[d] M3 is combination of M3 (major component), M4 and M7. Levels were too low to accurately quantify

M1 = 1,2,4-triazole, M3= hydroxyl flutriafol glucuronide, M4 = flutriafol glucuronide, M7 = methoxy flutriafol glucuronide, M3e = di-hydroxy flutriafol, M5= hydroxy methoxy flutriafol

For the carbinol-label, liver contained M2 (1.7% TRR), hydroxyl flutriafol glucuronide (M3, 4.3% TRR), dihydroxy flutriafol (M3e, 0.9% TRR), hydroxy methoxy flutriafol (M5 11.1% TRR) and flutriafol (0.9% TRR). The total identified residues in the liver accounted for 17.2% of TRR. A number of unidentified compounds (6.9% TRR) were observed that were individually present at $\leq$ 3% TRR ($\leq$ 0.007 mg equiv/kg). Hydrolysis of the liver PES under mild acid and alkaline conditions released all of the remaining ^{14}C residues which was able to be resolved into multiple peaks by chromatography. Subsequent treatment of the hydrolysis extracts with enzymes to release conjugates did not result in additional compounds being identified; largest individual component 9.0% TRR.

In kidneys the main ^{14}C residue components were M2 (9.7% TRR), hydroxyl flutriafol glucuronide (M3, 22.6% TRR) and dihydroxy flutriafol (M3e, 6.5% TRR). No other single metabolite comprised more than 6.5% of TRR (0.002 mg equiv/kg).

Residues in skim milk were extracted with acetonitrile and water. Main components identified were M2 (10.8% TRR, hydroxyl flutriafol glucuronide (M3, 27%TRR) and dihydroxy flutriafol (M3e, 29.7%TRR). No other single metabolite comprised more than 11% of TRR (0.004 mg equiv/kg).

Residues in milk fat were extracted with acetone/hexane. Main components identified were dihydroxy flutriafol (M3e, 42.3%TRR) and flutriafol (3.8% TRR). No other single metabolite comprised more than 11.5% of TRR (0.003 mg equiv/kg).

Table 3 Characterisation and identification of ^{14}C residues in tissues and milk of a goat dosed at 12 ppm with carbinol-label

Matrix	Liver	Kidney	Skim milk	Milk fat	Flank muscle [c]
TRR (mg equiv/kg)	0.234	0.031	0.037	0.026	0.004*
			%TRR		
Solvent extracts [a]	27.8	67.7	54.1	76.9	
Aqueous soluble [b]	9.4	54.8	54.1	76.9 (CH_3CN)	
M2	1.7	9.7	10.8		
M3 [d]	4.3	22.6	27.0		
M3e				42.3	
M10				3.8	
Flutriafol				3.8	
Unknowns	2.2 (2)	12.9 (3)	13.5 (3)	15.3 (2)	
Organic soluble [b]	18.4	12.9	40.5	< 3.8% (hexane)	
M3e	0.9	6.5	29.7		
M5	11.1		2.7		
Flutriafol	0.9		< 2.7		
Unknowns	4.7 (2)	<3.2 (1)	8.1 (2)		
PES	72.2	32.3	5.4	23.1	
Released by 1 N HCl	4.3	3.2			
Released by 1 N KOH	16.2	9.7			
Overall					
Extracted [d]	100.0	80.6	94.6	76.9	
identified	17.2	32.3	62.1	49.9	
characterized	80.8	38.7	32.4	15.3	
Unextracted [d]	0.0	19.4	5.4	23.1	

[a] Solvent systems: CH_3CN/H_2O for liver, kidney, skim milk and muscle; acetone/hexane for fat and milk fat

[b] Represents free residues from partition of initial extracts with ethyl acetate. (Aqueous is CH_3CN phase and organic is hexane phase for milk fat)

[c] Extraction and analysis data represent composite of flank and loin muscle

[d] M3 is combination of M3 (major component), M4 and M7. Levels were too low to accurately quantify

M1 = 1,2,4-triazole, M2 = possible amino acid conjugate, M3 = hydroxyl flutriafol glucuronide, M3e = di-hydroxy flutriafol, M4 = flutriafol glucuronide, M5 = hydroxy methoxy flutriafol, M7 = methoxy flutriafol glucuronide, M10 = flutriafol sulfate

*Residues too low for further characterisation / identification

In an additional study on the metabolism of flutriafol in lactating goats La Mar (2012 2438) used a higher dose rate to allow for better identification of metabolites. Two lactating goats (crossbreeds, 2–4 yrs old, 38 and 58 kg bw) were administered either [triazole-3(5)-^{14}C]-flutriafol or [carbinol-^{14}C]-flutriafol once daily for five consecutive days at a rate equivalent to 30 ppm (triazole) or 30.7 ppm (carbinol) in the feed. Animals consumed 1.8 and 1.3 kg feed/d respectively for the 30 and 31 ppm dose goats. Milk production was 1.6 L/d and 1.5 L/d respectively for the two goats. Excreta were collected once a day (in the morning, before dose administration). Milk was collected twice daily (morning and evening). The goats were sacrificed approximately 20–22 h after the last dose was administered and the following tissues were collected at necropsy—liver, kidney, muscle (loin and flank), fat (subcutaneous, omental and renal), bile, blood and gastrointestinal tract with contents. Analytical work was completed within 30 days after sacrifice.

The majority of the administered dose was recovered in the faeces (35–55%) with 30–54% excreted in urine and 0.09–0.1% in milk. The amount of administered radioactivity found in tissues was 0.27–0.29% while the gastrointestinal tract and contents contained 2.1–6.8% giving a total recovery of administered radioactivity of 88–96%. TRR in edible tissues were generally low

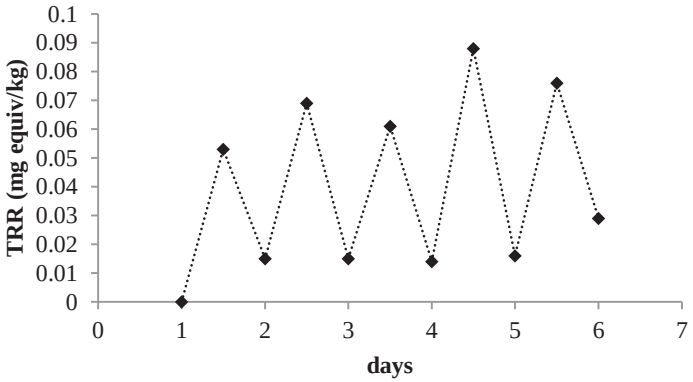
(0.008–0.024 mg equiv/kg) with the exception of liver (0.68–0.70 mg equiv/kg) and kidney (0.11–0.31 mg equiv/kg).

Table 4 Distribution of TRR following dosing of [^{14}C]flutriafol at 30 ppm for 5 days

	Triazole-label		Carbinol-label	
	%AD	mg equiv/kg	%AD	mg equiv/kg
Tissues				
Liver	0.22	0.698	0.22	0.676
Kidney	0.01	0.107	0.02	0.309
Omental fat	< 0.01	0.008	< 0.01	0.018
Subcutaneous fat	< 0.01	0.011	< 0.01	0.018
Renal fat	< 0.01	0.009	< 0.01	0.014
Flank muscle	< 0.01	0.02	< 0.01	0.024
Loin muscle	0.01	0.02	0.01	0.017
Blood	–	0.047	–	0.044
Excreta/secretions				
Faeces	55.32		34.67	
GI tract and contents	2.15		6.84	
Urine	30.03		53.77	
Whole milk	0.1	–	0.09	–
Bile	0.03	4.684	0.05	13.541
Cage wash	0.04		0	
Total	87.91		95.63	

Residues in milk appeared to reach plateau levels by Day 3 of dosing with significant differences in ^{14}C levels between milk collected in the morning (low levels), compared to evening milk (higher levels), suggesting flutriafol residues are rapidly eliminated following dosing (Figure 2).

A



B

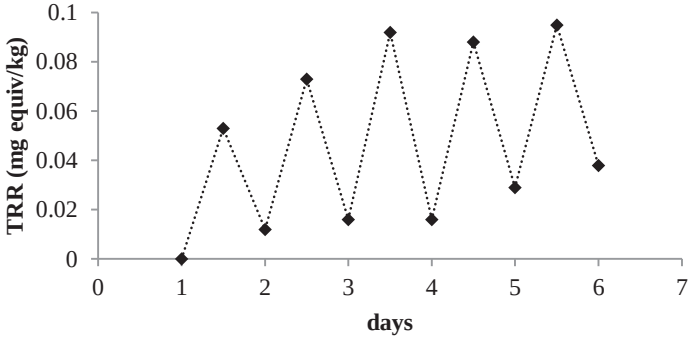


Figure 2 TRR in milk for goats dosed at the equivalent of 30 ppm in the feed with flutriafol (A) triazole label, (B) carbinol label

Acetonitrile and water extraction of liver, kidney, muscle, fat, skim milk and milk fat resulted in extraction efficiencies of 28.7–38.7% (liver), 66.7–86.5% (kidney), > 82% (muscle), > 72% fat, 98% (skim milk) and 82–87% (milk fat) (Tables 5 and 6).

For the TZ label, extraction of liver with CH_3CN/H_2O released 1,2,4-triazole (M1, 2.5% TRR), M2 (1.3% TRR), hydroxy flutriafol glucuronide (M3, 1.8% TRR), dihydroxy flutriafol (M3e, 0.7% TRR), flutriafol glucuronide (M4, 1.6% TRR), hydroxy methoxy flutriafol (M5, 6.9% TRR) and flutriafol (1.0% TRR). The total identified residues in the liver accounted for 16.9% of TRR. A number of unidentified compounds (7.9% TRR) were observed that were individually present at ≤ 2.5% TRR (≤ 0.015 mg equiv/kg). Hydrolysis of the liver PES under mild acid and alkaline conditions released all of the remaining ^{14}C residues which was able to be resolved into more than eight peaks by chromatography. Subsequent treatment of the hydrolysis extracts with enzymes to release conjugates did not result in additional compounds being identified.

In kidneys the main ^{14}C residue components were 1,2,4-triazole (M1, 8.9% TRR), M2 (1.3% TRR), hydroxy flutriafol glucuronide (M3, 9.8% TRR) and dihydroxy flutriafol (M3e, 3.3% TRR), hydroxy methoxy flutriafol (M5, 1.6% TRR), methoxy flutriafol glucuronide (M7, 5.7% TRR) and M8 (4.1% TRR). No other single metabolite comprised more than 4.9% of TRR (0.006 mg equiv/kg).

Muscle and fat contained low levels of ^{14}C. Major metabolites identified were 1,2,4-triazole (M1, 21–42% TRR), M2 (< 5–5.3% TRR), hydroxy flutriafol glucuronide (M3, 5.3–10% TRR). No other single metabolite comprised more than 0.003 mg equiv/kg.

Main components identified in skim milk were 1,2,4-triazole (M1, 14.9% TRR), M2 (3.2% TRR), hydroxy flutriafol glucuronide (M3, 23.4% TRR) and dihydroxy flutriafol (M3e, 35.1% TRR). No other single metabolite comprised more than 0.004 mg equiv/kg.

In milk fat components identified were 1,2,4-triazole (M1, 10.6% TRR), M2 (2.1% TRR), dihydroxy flutriafol (M3e, 43.6% TRR) and M8 (10.6% TRR). No other single metabolite comprised more than 0.005 mg equiv/kg.

Table 5 Characterisation and identification of ^{14}C residues in tissues and milk of a goat dosed with 30 ppm triazole label

Matrix	Liver	Kidney	Skim Milk	Milk Fat	Flank Muscle	Loin Muscle	Omental Fat	Subcut. Fat	Renal Fat
TRR (ppm)	0.607	0.123	0.094	0.094	0.02	0.019	0.014	0.011	0.008
			%TRR						
Solvent extracts [a]	28.7	66.7	97.9	87.2	90.0	89.5	92.9	72.7	75.0
Aqueous soluble [b]	14.3	57.7	54.3	87.2 (CH_3CN)	65.0	63.2	92.9 (CH_3CN)	72.7 (CH_3CN)	75.0 (CH_3CN)
M1	2.5	8.9	14.9	10.6	40.0	42.1	21.4	27.3	25.0
M2	1.3	4.1	3.2	2.1	< 5.0	5.3			
M3	1.8	9.8	23.4	43.6	10.0	5.3		9.1	
M4	1.6	13.0							
M5				1.1					
M7	1.6	5.7	2.1						
M8	0.8	4.1	3.2	10.6					
Flutriafol				3.2			7.1	9.1	
Unknowns	3.6 (4)	7.3 (2)	5.3 (2)	12.8 (3)		< 10.3 (2)	21.4 (2)		< 50 (2)
Organic soluble [b]	14.3	8.9	43.6	< 1.1 [c]	25.0	26.3	< 7.1 [c]	< 9.1 [c]	< 12.5 [c]
M3e	0.7	3.3	35.1						
M5	6.9	1.6	1.1						
Flutriafol	1.0		< 1.1						
Unknowns	4.3 (4)	3.2 (3)	6.5 (3)						
PES	71.3	33.3	2.1	12.8	10.0	10.5	7.1	27.3	25
1 N HCl	2.3	1.6							
1 N KOH	16.0	21.1							
Overall									
extracted	99.9	89.3	97.9	87.2	90	89.3	92.9	81.8	75
identified	16.9	46.4	80.9	69.1	50.0	47.4	28.5	45.5	25
characterized	78.1	37.3	15.0	11.7	35.0	36.9	21.4	18.2	50
unextracted	0.0	10.6	2.1	12.8	10.0	10.5	7.4	27.3	25

[a] Solvent systems: CH_3CN/H_2O for liver, kidney, skim milk and muscle; acetone/hexane for fat and milk fat

[b] Represents free residues from partition of initial extracts with ethyl acetate. (Aqueous is CH_3CN phase and organic is hexane phase for fat matrices)

[c] up to five components each < 0.007 mg equiv/kg and < 14% TRR in tissue with the exception of renal fat = 0.03 mg equiv/kg and 38% TRR

M1 = 1,2,4-triazole, M2 = possible amino acid conjugate, M3 = hydroxyl flutriafol glucuronide, M3e = di-hydroxy flutriafol, M4 = flutriafol glucuronide, M5 = hydroxy methoxy flutriafol, M7 = methoxy flutriafol glucuronide, M10 = flutriafol sulfate

For the carbinol-label the metabolites identified were M2 (2.5% TRR), hydroxyl flutriafol glucuronide (M3, 2.2% TRR), dihydroxy flutriafol (M3e, 1.1% TRR), flutriafol glucuronide (M4, 4.3% TRR), hydroxy methoxy flutriafol (M5, 7.3% TRR), methoxy flutriafol glucuronide (M7, 3.3% TRR), M8 (2.2% TRR) and flutriafol (2.5% TRR). The total identified residues in the liver accounted for 22.9% of TRR. A number of unidentified compounds (7.9% TRR) were observed that were individually present at $\leq$ 3.6% TRR ($\leq$ 0.023 mg equiv/kg). As with the earlier study and the triazole-label, hydrolysis of the liver PES under mild acid and alkaline conditions released all of the remaining ^{14}C residues. In the case of the carbinol label the released

^{14}C was able to be resolved into more than seven peaks by chromatography. Subsequent treatment of the hydrolysis extracts with enzymes to release conjugates did not result in additional compounds being identified.

In kidneys, the main ^{14}C residue components were M2 (8.6% TRR), hydroxyl flutriafol glucuronide (M3, 12.8% TRR) and dihydroxy flutriafol (M3e, 1.6% TRR), flutriafol glucuronide (M4, 24% TRR), hydroxy methoxy flutriafol (M5, 1.0% TRR), methoxy flutriafol glucuronide (M7, 10.5% TRR), M8 (5.3% TRR) and flutriafol (0.7% TRR). No other single metabolite comprised more than 4.3% of TRR (0.013 mg equiv/kg).

Muscle and fat contained low levels of ^{14}C. Major components identified in muscle were hydroxyl flutriafol glucuronide (M3, 4.3–5.9% TRR) and flutriafol glucuronide (M4, 5.9–17.4% TRR). No other single metabolite comprised more than 0.004 mg equiv/kg. In fat, the major component identified was flutriafol (21–59% TRR).

Main components identified in skim milk were M2 (4.7% TRR), hydroxyl flutriafol glucuronide (M3, 17.6% TRR), dihydroxy flutriafol (M3e, 27.1% TRR), methoxy flutriafol glucuronide (M7, 3.5% TRR), M8 (5.9% TRR) and flutriafol sulfate (M10, 8.2% TRR). Flutriafol was present at 1.2% TRR. No other single metabolite comprised more than 0.005 mg equiv/kg.

In milk fat components identified were M2 (4.3% TRR), hydroxyl flutriafol glucuronide (M3, 30.5% TRR), hydroxy methoxy flutriafol (M5, 2.1% TRR), M8 (7.8% TRR), flutriafol sulfate (M10, 17% TRR) and flutriafol (4.3% TRR). No other single metabolite comprised more than 0.01 mg equiv/kg.

Table 6 Characterisation and identification of ^{14}C residues in tissues and milk of a goat dosed with 30 ppm carbinol label

Matrix	Liver	Kidney	Skim Milk	Milk Fat	Flank Muscle	Loin Muscle	Omental fat	Subcut. fat	Renal Fat
TRR (mg equiv/kg)	0.631	0.304	0.085	0.141	0.023	0.017	0.017	0.017	0.014
				%TRR					
Solvent extracts [a]	38.7	86.5	97.6	82.3	87.0	82.4	82.4	88.2	78.6
Aqueous soluble [b]	21.4	80.3	54.1	82.3 CH_3CN	52.2	47.1	76.5 CH_3CN	88.2 CH_3CN	78.6 CH_3CN
M2	2.5	8.6	4.7	4.3					
M3	2.2	12.8	17.6	30.5	4.3	5.9			
M4	4.3	25.0			17.4	5.9			
M5				2.1					
M7	3.3	10.5	3.5			5.9			
M8	2.2	5.3	5.9	7.8					
M10			8.2	17.0					
Flutriafol				4.3			23.5	58.8	21.4
Unknowns	3.4 (4)	8.6 (3)	7.1 (3)	10.6 (2)	21.7 (2)	29.4 (2)	47 (3)	17.7 (2)	50 (3)
Organic soluble [b]	17.3	6.3	43.5	< 0.7 (h)	34.8	35.3	5.9	< 5.9	< 7.1
M3e	1.1	1.6	27.1						
M5	7.3	1.0	1.2						
Flutriafol	2.5	0.7	1.2						
Unknowns	5.2 (3)	1.0 (1)	11.8 (3)						
PES	47.4	6.3	2.4	17.7	13.0	17.6	17.6	11.8	21.4
1 N HCl	2.4	2.3							
1 N KOH	11.6	4.9							
Overall									
extracted	100.0	93.8	97.6	82.3	87.0	82.4	82.4	88.2	78.6
identified	22.9	56.9	64.7	61.7	21.7	17.7	23.5	58.8	21.4
characterized	71.7	25.4	23.9	25.5	56.5	64.7	52.9	17.7	50.0
unextracted	0.0	6.3	2.4	17.7	13.0	17.6	17.6	11.8	21.4

[a] Solvent systems: CH_3CN/H_2O for liver, kidney, skim milk and muscle; acetone/hexane for fat and milk fat

[b] Represents free residues from partition of initial extracts with ethyl acetate. (Aqueous is CH_3CN phase and organic is hexane phase for fat matrices)

M1 = 1,2,4-triazole, M2 = possible amino acid conjugate, M3 = hydroxyl flutriafol glucuronide, M3e = di-hydroxy flutriafol, M4 = flutriafol glucuronide, M5 = hydroxy methoxy flutriafol, M7 = methoxy flutriafol glucuronide, M10 = flutriafol sulfate

Residues in goat milk and edible tissues resulted from extensive metabolism of flutriafol. In the major metabolic pathway, one of the phenyl rings is oxidised and then conjugated with glucuronic acid to form flutriafol glucuronide (M4), or is further oxidised to form dihydroxy flutriafol (M3e), of which there are a number of possible isomers. M3e is then further transformed via methylation to hydroxyl methyl flutriafol (M5) which can in turn be conjugated with glucuronic acid to form methoxy flutriafol glucuronide (M7). M3e was also conjugated with glucuronic acid to form hydroxyl flutriafol glucuronide (M3). A minor pathway is the cleavage of flutriafol at the 1-nitrogen of the triazole ring to give free triazole. One unique carbinol metabolite designated as M10 was identified as flutriafol sulfate.

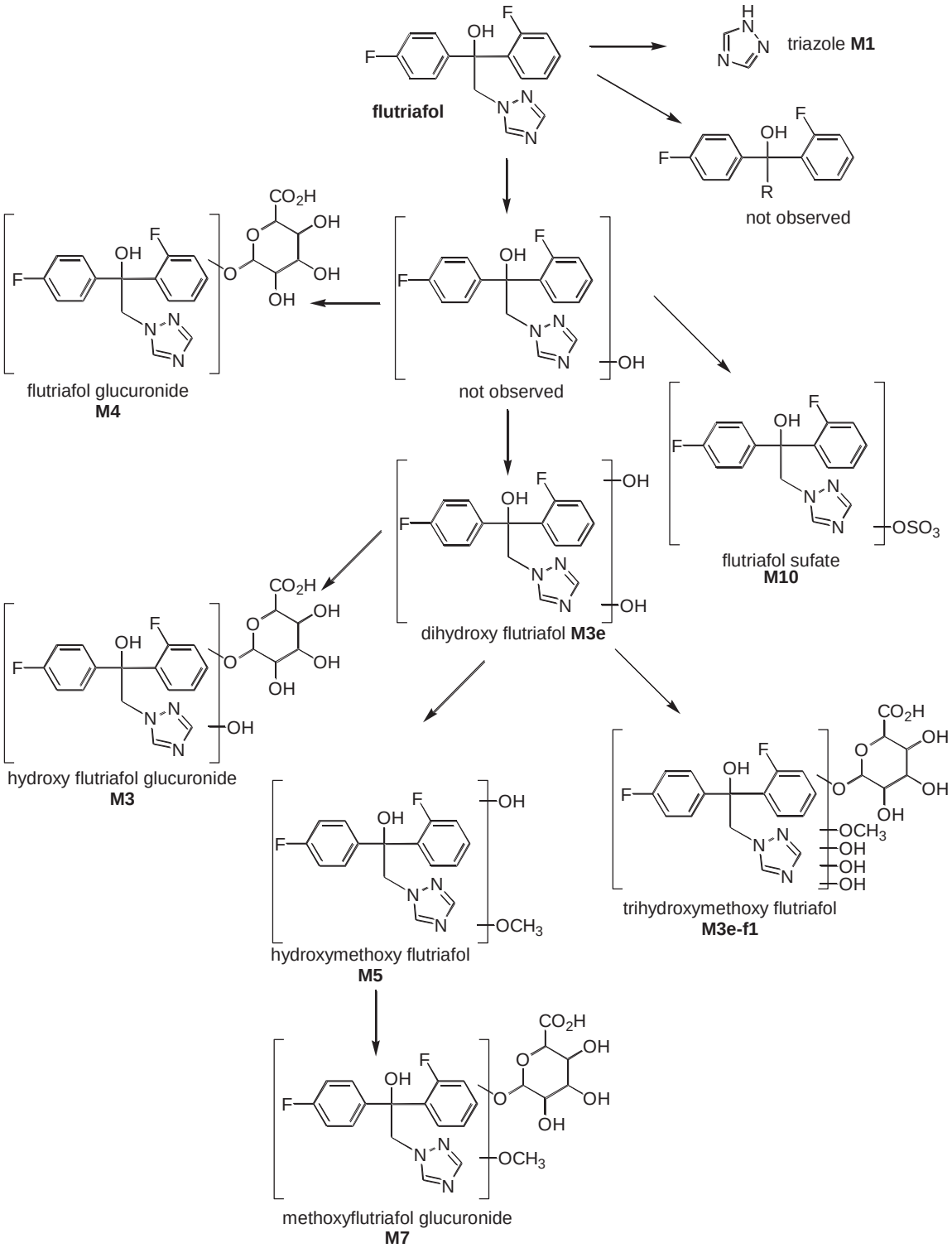


Figure 3 Possible metabolic pathway for flutriafol in goats

RESIDUE ANALYSIS

Analytical method

Stability of pesticide residues in stored analytical samples

The 2011 JMPR evaluated data on the storage stability of flutriafol residues in plant commodities that included apples, grapes, cabbages, sugar beet roots, pea seeds, soybeans, barley grains, wheat and oilseed rape, processed commodities (apple juice, soybean meal and refined oil) and animal commodities (milk, eggs, muscle and fat).

The 2011 JMPR also received information on the freezer storage stability of triazole metabolites in apple (fruit and juice), milk, eggs, muscle and fat.

Storage stability results indicate that flutriafol residues were stable for at least 4 months in animal commodities, for at least 5 months in soybean seeds, for at least 12 months in apples, barley grains and coffee beans, for at least 23 months in grapes, for at least 24 months in cabbages and oilseed rape, and for at least 25 months in wheat (grains and straw), pea seeds, and sugar beet roots. The results also indicate that triazole metabolite residues were stable for at least 4 months in apple fruits and juice, and for at least 5 months in animal commodities.

Mason (2012 2649) studies the freezer storage stability of residues in bovine matrices. The deep freeze storage stability of flutriafol and triazole metabolites 1,2,4-triazole (T), triazole alanine (TA) and triazole acetic acid (TAA) in muscle, fat, liver and kidney was conducted by fortifying separate control samples of homogeneous matrix with flutriafol, T, TA and TAA at levels of 0.1 mg/kg. These samples were placed in freezer storage and analysed after 0, 1, 3, 6, 9 and 12 months frozen storage. All samples were analysed in duplicate. Unfortified control samples were analysed at the same time alongside duplicate freshly fortified samples of control matrix at 0.1 mg/kg.

Residues of flutriafol, and T, TA and TAA in ruminant tissues (muscle, fat, liver and kidney) remain stable for at least 12 months for flutriafol, TA and TAA and at least 6 months for T when samples are stored under deep frozen conditions.

Table 7 Recovery of flutriafol and metabolite residues on frozen storage of animal commodity samples separately fortified with flutriafol, T, TA or TAA

Analyte	Storage time (days)	Amount recovered from stored sample (mg/kg)	Mean procedural recovery (%)
Muscle			
Flutriafol	0	0.077, 0.072	75
	182	0.100, 0.096	79
	275	0.122, 0.104	102
	372	0.118, 0.108	97
T	0	0.093, 0.094	94
	183	0.096, 0.090	90, 97
	322	0.086, 0.091	90
	366	0.078, 0.076	80
TA	0	0.109, 0.106	108
	183	0.108, 0.109	101
	322	0.098, 0.094	88
	366	0.114, 0.101	98
TAA	0	0.104, 0.100	102
	183	0.097, 0.091	103
	322	0.096, 0.092	95
	366	0.108, 0.108	109
Fat			
Flutriafol	0	0.080, 0.078	79
	183	0.069, 0.074	71
	279	0.070, 0.082	86

Analyte	Storage time (days)	Amount recovered from stored sample (mg/kg)	Mean procedural recovery (%)
	370	0.095, 0.106	86
T	0	0.088, 0.087	88
	189	0.066, 0.066	92
	321	0.081, 0.083	94
	367	0.056, 0.065	90
TA	0	0.110, 0.110	110
	189	0.101, 0.104	101
	321	0.106, 0.080	107
	367	0.100, 0.097	105
TAA	0	0.099, 0.099	99
	189	0.094, 0.090	110
	321	0.108, 0.097	105
	367	0.097, 0.089	111
Liver			
Flutriafol	0	0.104, 0.104	104
	32	0.063, 0.067	74
	152	0.093, 0.103	99
	185	0.100, 0.095	76
	276	0.115, 0.114	89
	369	0.126, 0.119	108
T	0	0.089, 0.09	90
	35	0.075, 0.075	77
	117	0.087, 0.089	90
	186	0.087, 0.086	94
	313	0.081, 0.079	92
	370	0.082, 0.071	90
TA	0	0.102, 0.102	102
	35	0.103, 0.097	107
	117	0.103, 0.105	92
	186	0.107, 0.109	99
	313	0.096, 0.093	89
	370	0.108, 0.116	103
TAA	0	0.083, 0.082	83
	35	0.109, 0.109	110
	117	0.110, 0.110	110
	186	0.092, 0.087	101
	313	0.104, 0.107	108
	370	0.113, 0.117	109
Kidney			
Flutriafol	0	0.096, 0.094	95
	37	0.085, 0.080	91
	92	0.092, 0.093	99
	184	0.112, 0.120	110
	365	0.107, 0.109	95
T	0	0.092, 0.095	94
	30	0.095, 0.098	101
	91	0.087, 0.082	90
	198	0.093, 0.093	106
	365	0.061, 0.061	75
TA	0	0.105, 0.107	106
	30	0.099, 0.102	106
	91	0.102, 0.100	102
	198	0.078, 0.080	86
	365	0.092, 0.087	101
TAA	0	0.107, 0.107	107
	30	0.100, 0.100	103
	91	0.110, 0.112	104
	198	0.111, 0.109	110
	365	0.107, 0.099	96

Analytical method flutriafol: muscle, liver, kidney, fat—Method No. ICIA AM00306
Analytical method T, TA, TAA—Meth-160 rev 2.

USE PATTERN

Table 8 Registered uses of flutriafol on crops relevant to this submission

Crop	Country	GS	Rate (g ai/ha)	Water (L/ha)	N	Interval (days)	PHI (days)
Almond walnut	USA		128 Max single 128 Max/year 511	> 93.5 grd/air	4	7	14
Apple	Belarus		25–37.5	1000–1200	4	10–14	40
Apple	Italy		20–30 (or 2–3 g ai/hL)		2	10–14	21
Apple	Kazakhastan		25–37.5		2		20
Brassica (Cole) leafy vegetables	USA		91–128 Max single 128 Max/year 511	> 93.5 grd > 46.8 air	4	7	7
Celery and Chinese celery	USA		91–128 Max single 128 Max/year 511	> 93.5 grd > 46.8 air	4	7	7
Corn (field, pop, seed)	USA	apply no later than R4 (early dough stage	128 Max single 128 Max/year 256	> 93.5 grd > 18.7 air	2	7	7, except forage 0 days
Cotton	USA		Max one 146–290 (soil appl. at planting) + 64–128 (foliar appl.) max total soil + foliar 547	56–93 92–187	1 2	n/a 7	 30
Cucurbit vegetables (except muskmelon)	USA	–	91–128 Max single 128 Max/year 511	> 93.5 grd > 46.8 air	4	7	0
Fruiting vegetables group 8–10	USA	Onset of fruit up to harvest	128 Max single 128 Max/year 511	> 93.5 grd > 46.8 air	4	7	0
Leafy vegetables (except Brassica vegetables)	USA		91–128 Max single 128 Max/year 511	> 93.5 grd > 46.8 air	4	7	7
Muskmelons	USA	–	91–128 Max single 128 Max/year 511	> 93.5 grd > 46.8 air	4	7	0
Pecan and other tree nuts	USA		64–128 Max single 128 Max/year 511	> 93.5 grd/air	4	7	14
Pome fruit	USA	–	73–119 Max single 119 Max/year 475	> 93.5 grd > 46.8 air	4	7–10	14
Rapeseed	Belarus	End of flowering/ beginning of pod	125		1		30

Crop	Country	GS	Rate (g ai/ha)	Water (L/ha)	N	Interval (days)	PHI (days)
		formation					
Rapeseed	Kazakhastan		125	200	1		30
Rapeseed	Russia	n/a	125	200–300	1–2	10–14	30
Rice	Italy	onset of the 1st symptoms of disease, repeating on appearance panicle	125–187.5		2		28
Rice	Kazakhastan		187.5–250	200 L/ha	1		30
Rice	Russia		250	50–100 L/ha	1		27
Sorghum	USA	–	64–128 Max single 128 Max/year 256	> 93.5 grd > 46.8 air	4	7	30 stover forage grain
Stone fruit (except cherry)	USA	–	128 Max single 128 Max/year 511	> 93.5 grd > 46.8 air	4	7	7
Stone fruit (inc cherry)	USA	–	128 Max single 128 Max/year 511	> 93.5 grd > 46.8 air	4	7	7
Strawberry	USA	Onset of fruit up to harvest	91–128 Max single 128 Max/year 511	> 93.5 grd > 46.8 air	4	7	0
Sugar beet	Belarus		62.5–125	300	1		30
Sugar beet	Russia		62.5	300	1–2		30
Sugar beet	USA	–	91–128 Max single 128 Max/year 256	> 93.5 grd > 46.8 air	2	14	21
Tomato	USA	Onset of fruit up to harvest	64–128 Max single 128 Max/year 511	> 93.5 grd > 46.8 air	4	7	0

Stone Fruit: Apricot, Nectarine, Peach, Plum, Cherries (Sweet and Tart), Chickshaw plum, Damson plum, Japanese plum, Plumcot, Prune

Muskmelons: True Cantaloupe, Cantaloupe, Casaba, Crenshaw Melon, Golden Pershaw Melon, Honeydew Melon, Honey Balls, Mango Melon, Persian Melon, Pineapple Melon, Santa Claus Melon, and Snake Melon

Cucurbits: Chayote (Fruit), Chinese Waxgourd, Citron Melon, Cucumber, Gherkin, Gourd Edible (Lagenaria spp.) (Includes Hyotan, Cucuzza, Hechima, Chinese Okra), Momordica spp. (Includes Balsam Apple, Balsam Pear, Bittermelon, Chinese Cucumber), Pumpkin, Squash (Summer), Squash (Winter—Includes Butternut Squash, Calabaza, Hubbard Squash, Acorn Squash, Spaghetti Squash), Watermelon

Brassica (Cole) Leafy Vegetables: Broccoli, Broccoli (Chinese and Raab), Brussels Sprouts, Cabbage, Cabbage (Chinese, Bok Choy, Chinese Mustard/Gai Choy), Cauliflower, Cavalo Broccolo, Collards, Kale, Kohlrabi, Mizuna, Mustard Greens, Mustard Spinach, Rape Greens. Including all cultivars and/or hybrids of these crops.

Leafy Vegetables (except Brassica): Amaranth, Arugula, Cardoon, Celery, Celery (Chinese), Celtuce, Chervil, Chrysanthemum (Edible and Garland), Corn Salad, Cress (Garden and Upland), Dandelion, Dock, Endive, Fennel (Florence), Lettuce (Head and Leaf), Orach, Parsley, Purslane (Garden and Winter), Radicchio, Rhubarb, Spinach, Spinach (New Zealand and Vine), Swiss Chard. Including cultivars and/or hybrids of these crops.

Pecans and other tree nuts: African Tree Nut, Brazil Nut, Burr Oak, Butternut, Cajou, Cashew, Castanha-Do-Maranhao, Coconut, Coquito Nut, Dika nut, Guiana Chestnut, Hazelnut, Heartnut, Hickory Nut, Japanese Horse-Chestnut, Macadamia Nut, Monogongo Nut, Monkey-Pot, Pachira Nut, Pecan, Sapucaia Nut

Fruiting Vegetables (group 8-10): African Eggplant, Bell Pepper, Eggplant, Martynia, Non-Bell Pepper, Okra, Pea Eggplant, Pepino, Roselle, Scarlet Eggplant. Including cultivars, varieties and/or hybrids of these crops.

Crop Rotation: Fields treated with an application rate of greater than 252 g ai/ha/season may be planted to crops that have tolerances established for residues of flutriafol including: field corn, popcorn, cucurbits, fruiting vegetables, grapes, peanuts, pome fruits, soybeans, stone fruits, strawberries, sugar beets, tree nuts, triticale, or wheat immediately after last application.

Fields treated with application rates less than or equal to 252 g ai/ha/season may be planted to the crops listed above, and may also be planted to cotton or sweet corn 180 days after the last application. Rotation to any other crop is prohibited.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on supervised residue trials of foliar treatments of flutriafol for apples, pears, peaches/nectarines, plums, cherries, strawberries, Brassica vegetables (cabbage and broccoli), cucurbits (cucumbers, summer squash and muskmelons), tomatoes, peppers, leafy vegetables (lettuce, spinach, celery and mustard greens), sugar beets, maize, rice, sorghum, almonds, pecans, cotton, and rape.

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 9 Summary of sprayers, plot sizes and field sample sizes in the supervised trials

Location	Year	Crop	Sprayer	Plot size	Sample size	SAI (days)
Europe	2004	Sugar beet	Boom sprayer, knapsack sprayer	60–120 m^2	Plants ≥ 0.6 kg Leaves ≥ 0.5 kg Roots ≥ 1.0 kg Leaves with tops ≥ 1.0 kg	< 80
Europe	2005	Tomato	CO_2 sprayer	14–33 m^2	≥ 2.0 kg	< 52
Europe	2005	Rape	Boom sprayer	60–90 m^2	Shoots no roots ≥ 1.1 kg Pods ≥ 0.6 kg Shoots no pods ≥ 1.0 kg Seeds ≥ 0.5 kg	< 30
Europe	2005	Sugar beet	Boom sprayer	30–90 m^2	Leaves with tops ≥ 1.0 kg Roots ≥ 1.0 kg	< 80
Europe	2006	Rape	Boom sprayer	30–60 m^2	Seeds ≥ 0.5 kg	< 20
Spain	2006	Sugar beet	Boom sprayer	30 m^2	Leaves with tops ≥ 2.8 kg Roots ≥ 4.8 kg	< 20
France	2007	Rape	Boom sprayer	120 m^2	Seeds ≥ 0.5 kg	< 38
Spain	2005	Rice	Boom sprayer	25–50 m^2	Seeds ≥ 1.0 kg	< 130
USA	2009	Cherry sweet	Tractor-mounted Airblast Sprayer	6–16 trees	Fruit ≥ 1.1 kg	79 F 84 T
USA	2009	Cherry tart	Tractor-mounted Airblast Sprayer	6–16 trees	Fruit ≥ 1.1 kg	64–107 F 58–127 T
USA	2009	Peach	Tractor-mounted Airblast Sprayer	6–8 trees	Fruit ≥ 2.0 kg	45–135 F 40–114 T
USA	2009	Plum	Tractor-mounted Airblast Sprayer	6–8 trees	Fruit ≥ 2.0 kg	9–154 F 13–149 T
USA	2009	Pear	Tractor-mounted Airblast Sprayer	6–7 trees	Fruit ≥ 2.3 kg	24–188 F 23–192 T
USA	2009	Maize	CO_2 backpack sprayer, Tractor mounted side-mount sprayer	56–1110 m^2	Forage ≥ 1.6 kg Grain ≥ 1.0 kg Stover ≥ 0.4 kg	Forage 64–211 F 67–211 T Grain 84–186 F 72–201 T
USA	2009	Sugar beet		46–372 m^2	Leaves with tops ≥ 1.0 kg Roots 12 roots	183 F 194 T
USA	2010	Strawberry	CO_2 backpack sprayer, Hand-held boom sprayer	31–186 m^2	Fruit ≥ 0.6 kg	12–90 F 31–88 T

Location	Year	Crop	Sprayer	Plot size	Sample size	SAI (days)
USA	2010	Apple	Tractor-mounted Airblast Sprayer	6–8 trees	Fruit ≥ 3.0 kg	33–60 F 64–89 T
USA	2010	Tree nuts (Almond, Pecan)	Tractor-mounted Airblast Sprayer	6–8 trees	≥ 1.2 kg	Pecan 162 Almond 230 Hulls 92
Spain	2006	Peach	Boom + knapsack sprayer	3–4 trees	≥ 2.0 kg	< 139
USA	2011	Cucurbits	CO_2 backpack + tractor mounted sprayers	48–180 m^2	≥ 1.5 kg (melon: each fruit quartered opposing 2 quarters selected 24 quarters)	16–104 F 16–176 T
USA	2011	Tomato	CO_2 backpack + boom + tractor mounted sprayers	48–180 m^2	≥ 2.0 kg	18–134
USA	2011	Pepper	CO_2 backpack + boom + tractor mounted sprayers	45–140 m^2	≥ 2.0 kg	18–134
Spain	2004	Strawberry	Backpack + knapsack sprayer	16.5–44 m^2 macrotunnels	≥ 1.0 kg	212
USA	2012	Brassica vegetables	CO_2 backpack + tractor mounted sprayers	45–167 m^2	≥ 1.0 kg (cabbage: Heads were quartered and one quarter of 12 heads collected for each sample OR **Heads were halved and one half of 12 heads collected for each sample	7–195 F 24–178 T
USA	2011	Leafy vegetables	CO_2 backpack + tractor mounted sprayers	43–206 m^2	≥ 1.0 kg	18–184 F 11–212 T
USA	2012	Sorghum	CO_2 backpack + tractor mounted sprayers	93–1490 m^2	≥ 1.0 kg	27–196 F 56–189 T
USA	2012	Cotton	CO_2 backpack + tractor mounted sprayers	93–696 m^2	≥ 1.0 kg	15–110 F 21–141 T

Residues of the triazoles, TA and TAA were frequently observed in both untreated control and samples from treated plots, however, the source of the residues is unknown. That residues were detected in untreated controls suggests a natural origin. Triazole-related compounds are also common metabolites of a number of fungicides which contain the 1,2,4-triazole moiety.

Table 10 Residues of flutriafol in apples following application of an SC formulation in the USA (Carringer 2011 2159) (duplicate samples)

Location,		g		g	GS		Residue (mg/kg)			
year, variety	No	ai/ha	L/ha	ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
Cambridge, ON, Canada	6 (14 14 14	120 120	889 898	13	71–73 75	14	0.02 0.02	< 0.01 < 0.01	< 0.01 0.01	< 0.01 < 0.01
2010 McIntosh	13 14)	120 120 122 119	879 879 889 926		76–77 77–78 79 81–85	Mean	0.02	< 0.01	< 0.01	< 0.01
St George,	6 (14	119	739	16	74–76	14	0.02 0.01	< 0.01	0.04	< 0.01

Location,		g		g	GS		Residue (mg/kg)			
year, variety	No	ai/ha	L/ha	ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
ON, Canada 2010	14 14 14 13)	117 120	730 730		77 78			< 0.01	0.03 c0.04	< 0.01
Northern spy		119 119 119	702 730 720		79 81 81–85	Mean	0.02	< 0.01	0.04	< 0.01
Conklin, MI, USA 2010 Ida	6 (14 14 14	120 120	804 776	15	75 76	14	0.07 0.05	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
Red	14 14)	120 120 121 120	795 776 795 776		77 78 79 85	Mean	0.06	< 0.01	002	< 0.01
Marengo, IL, USA 2010 Gala	6 (14 15 13 14 14)	122 119 122	758 730 730	16	75 76 77	14	0.10 0.12	< 0.01 < 0.01	0.07 0.08 c0.05	0.01 0.01
		121 119 122	748 758 758		80 82 85	Mean	0.11	< 0.01	0.08	0.01

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2

Table 11 Flutriafol and triazole metabolites residues on apple fruits from supervised trials in USA reported by the 2011 JMPR (Willard, 2007 1471)

Country, year	Application				DALA		Residue (mg/kg)	
(variety) APPLE	Form	kg ai/ha	water, L/ha	no.		Flutriafol	TA	TAA
USA/CA, 2006	SC	0.12	798–936	6	14	0.07, 0.05	0.02, 0.02	< 0.01, < 0.01
(Granny smith)					Mean	0.06		
USA/ ID, 2006	SC	0.12	759–931	6	15	0.07, 0.09	< 0.01, < 0.01	< 0.01, < 0.01
(Macintosh)					Mean	0.08		
USA/IL, 2006	SC	0.12	795–840	6	14	0.06, 0.06	0.02, 0.02	< 0.01, < 0.01
(Golden Supreme)					Mean	0.06		
USA/MI, 2006	SC	0.12	801–843	6	14	0.09, 0.09	0.04, 0.04 c0.06	< 0.01, < 0.01
(Golden Delicious)					Mean	0.09		
USA/MI, 2006	SC	0.12	807–827	6	0	0.07, 0.07	0.06, 0.06	< 0.01, < 0.01
(Ida Red)					Mean	0.07		
					7	0.05 0.04	0.07 0.06	< 0.01, < 0.01
					Mean	0.05		
					13	0.05 0.04	0.05 0.05 c0.03	< 0.01, < 0.01
					Mean	0.05		
					21	0.04 0.04	0.07 0.07	< 0.01, < 0.01
					Mean	0.04		
					27	0.05 0.04	0.06 0.05	< 0.01, < 0.01
					Mean	0.05		
	SC	0.12	804–838	5	0	0.06, 0.06	0.08, 0.05	< 0.01, < 0.01
					Mean	0.06		
					7	0.04 0.04	0.07 0.08	< 0.01, < 0.01
					Mean	0.04		
					13	0.04 0.04	0.07 0.07	< 0.01, < 0.01
					Mean	0.04		
					21	0.04 0.04	0.08 0.09	< 0.01, < 0.01
					Mean	0.04		
					27	0.03 0.03	0.07 0.07	< 0.01, < 0.01
					Mean	0.03		
USA/NY, 2006	SC	0.12	924–981	6	15	0.05, 0.03	0.02, 0.01 c0.03	< 0.01, < 0.01
(Cortland)					Mean	0.04		
USA/NY, 2006	SC	0.12	939–953	6	14	0.05, 0.07	0.03, 0.02 c0.01	< 0.01, < 0.01
(Ida Red)					Mean	0.06		
		0.12-	933–942	6	14	0.10, 0.12	0.03, 0.03	< 0.01, < 0.01
		0.24			Mean	0.11		

Country, year	Application				DALA		Residue (mg/kg)	
(variety) APPLE	Form	kg ai/ha	water, L/ha	no.		Flutriafol	TA	TAA
USA/OR, 2006	SC	0.12	830–849	6	14	0.09, 0.12	0.03, 0.02 c0.03	< 0.01, < 0.01
(Pacific Gala)					Mean	0.10		
USA/OR, 2006	SC	0.12	815–840	6	14	0.05, 0.05	0.03, 0.03	< 0.01, < 0.01
(Jonagold)					Mean	0.05		
USA/PA, 2006	SC	0.12	895–903	6	14	0.11, 0.14	0.02, 0.02 c0.03	< 0.01, < 0.01
(Royal Gala)					Mean	0.12		
USA/PA, 2006	SC	0.12	789–808	6	0	0.14, 0.19	0.05, 0.05	0.01, 0.02
(Loe Rome)					Mean	0.17		
					7	0.09 0.08	0.05 0.05	0.01 0.01
					Mean	0.09		
					14	0.05 0.06	0.05 0.05	0.01 0.01
					Mean	0.05		
					21	0.07 0.09	0.06 0.06 c0.05	0.01 0.01
					Mean	0.08		
					28	0.06 0.05	0.05 0.05	0.01 0.01
					Mean	0.06		
	SC	0.12	800–815	5	0	0.14, 0.17	0.03, 0.04	0.01, 0.01
					Mean	0.16		
					7	0.05 0.05	0.04 0.04	< 0.01, < 0.01
					Mean	0.05		
					14	0.05 0.06	0.04 0.04	< 0.01, < 0.01
					Mean	0.06		
					21	0.07 0.07	0.04 0.04	< 0.01, < 0.01
					Mean	0.07		
					28	0.08 0.05	0.03 0.03	< 0.01, < 0.01
					Mean	0.07		
USA/UT, 2006	SC	0.12	748–804	6	14	0.03, 0.03	< 0.01, < 0.01	< 0.01, < 0.01
(Empire)					Mean	0.03		
USA/VA, 2006	SC	0.12	706–748	6	13	0.06, 0.04	0.03, 0.02 c0.06	< 0.01, < 0.01
(Rome)					Mean	0.05		
USA/VA, 2006	SC	0.12	805–817	6	13	0.12, 0.09	0.03, 0.02 c0.03	< 0.01, < 0.01
(York)					Mean	0.10		
USA/WA, 2006	SC	0.12	861–879	6	0	0.09 0.10	< 0.01, < 0.01	< 0.01, < 0.01
(Braeburn)					Mean	0.10		
					7	0.10 0.12	< 0.01, < 0.01	< 0.01, < 0.01
					Mean	0.11		
					14	0.09 0.12	0.01, 0.01	< 0.01, < 0.01
					Mean	0.11		
					21	0.13 0.13	< 0.01, 0.01	< 0.01, < 0.01
					Mean	0.13		
					27	0.07 0.11	0.01, < 0.01	< 0.01, < 0.01
					Mean	0.09		
	SC	0.12	864–871	5	0	0.16 0.13	0.02 0.02	< 0.01, < 0.01
					Mean	0.14		
					7	0.15 0.13	0.02 0.02	< 0.01, < 0.01
					Mean	0.14		
					14	0.14 0.11	0.02 0.02	< 0.01, < 0.01
					Mean	0.13		
					21	0.15 0.16	0.02 0.02	< 0.01, < 0.01
					Mean	0.16		
					27	0.09 0.16	0.02 0.02	< 0.01, < 0.01
					Mean	0.13		
USA/WA, 2006	SC	0.12	861–872	6	14	0.13, 0.11	0.04 0.03 c0.02	< 0.01 < 0.01
(Red Delicious)					Mean	0.12		
		0.12-	859–877	6	14	0.17, 0.21	0.04 0.04	< 0.01 < 0.01
		0.24			Mean	0.19		

Table 12 Residues of flutriafol in pears following application of an SC formulation in the USA (Carringer 2010 1809) (duplicate samples)

Location,		g		g	GS		Residue (mg/kg)			
year, variety	No	ai/ha	L/ha	ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
Alton, NY,	6 (14	122	1141	11	71	0	0.02 0.03	< 0.01	< 0.01	< 0.01
2009 Clapp's	14 14	118	1094		72			< 0.01	< 0.01	< 0.01
Favorite	14 14)	119	1113		74	Mean	0.02	< 0.01	< 0.01	< 0.01
		120	1122		75	14	0.03 0.04	< 0.01	< 0.01	< 0.01
		120	1122		76			< 0.01	< 0.01	< 0.01
		120	1122		81					
						Mean	0.04	< 0.01	< 0.01	< 0.01
Poplar, CA,	6 (14	120	561	21	76	0	0.15 0.11	< 0.01	0.01	< 0.01
2009 Olympic	14 14	121	589		77			< 0.01	< 0.01	< 0.01
	14 14)	122	571		78	Mean	0.13	< 0.01	< 0.01	< 0.01
		121	571		79	14	0.09 0.26	< 0.01	< 0.01	< 0.01
		121	561		79			< 0.01	0.01	< 0.01
		121	561		85					
						Mean	0.18	< 0.01	< 0.01	< 0.01
Lindsay, CA,	6 (14	119	2170	5.5	74	0	0.07 0.08	< 0.01	0.02	< 0.01
2009 Olympic	14 14	121	2170		75			< 0.01	0.03	< 0.01
	14 14)	119	2142		76	Mean	0.08	< 0.01	0.02	< 0.01
		122	2170		77	0	0.14 0.09	< 0.01	0.02	< 0.01
		120	2151		78			< 0.01	0.06	< 0.01
		120	2198		87	Mean	0.12	< 0.01	0.04	< 0.01
						7	0.10 0.09	< 0.01	0.03	< 0.01
								< 0.01	0.02	< 0.01
						Mean	0.10	< 0.01	0.02	< 0.01
						14	0.13 0.07	< 0.01	< 0.01	< 0.01
								< 0.01	< 0.01	< 0.01
						Mean	0.10	< 0.01	< 0.01	< 0.01
						21	0.18 0.21	< 0.01	< 0.01	< 0.01
								< 0.01	0.01	< 0.01
						Mean	0.20	< 0.01	< 0.01	< 0.01
						29	0.17 0.25	< 0.01	0.01	< 0.01
								< 0.01	0.01	< 0.01
						Mean	0.21	< 0.01	0.01	< 0.01
Ephrata, WA,	6 (14	120	571	21	74	0	0.28 0.29	< 0.01	< 0.01	< 0.01
2009 Concord	14 14	119	561		75			< 0.01	< 0.01	< 0.01
	14 14)	120	571		76	Mean	0.28	< 0.01	< 0.01	< 0.01
		120	571		78	14	0.22 0.25	< 0.01	< 0.01	< 0.01
		120	571		81			< 0.01	< 0.01	< 0.01
		119	561		85	Mean	0.24	< 0.01	< 0.01	< 0.01
Payette, ID,	6 (13	119	1384	8.6	74	0	0.12 0.13	< 0.01	0.05	< 0.01
2009 Bartlett	15 13	120	1403		75			< 0.01	0.05	< 0.01
	16 13)	120	1403		76	Mean	0.12	< 0.01	0.05	< 0.01
		119	1384		77	0	0.24 0.20	< 0.01	0.04	< 0.01
		122	1431		78			< 0.01	0.05	< 0.01
		123	1440		79	Mean	0.22	< 0.01	0.04	< 0.01
						7	0.14 0.17	< 0.01	0.04	< 0.01
								< 0.01	0.04	< 0.01
						Mean	0.16	< 0.01	0.04	< 0.01
						14	0.14 0.12	< 0.01	0.06	< 0.01
								< 0.01	0.05	< 0.01
									c0.05	
						Mean	0.13	< 0.01	0.06	< 0.01
						21	0.13 0.10	< 0.01	0.04	< 0.01
								< 0.01	0.04	< 0.01
						Mean	0.12	< 0.01	0.04	< 0.01
						28	0.08 0.08	< 0.01	0.04	< 0.01
								< 0.01	0.03	< 0.01
						Mean	0.08	< 0.01	0.04	< 0.01
Buhl, ID,	6 (16	120	599	20	72	0	0.08 0.09	< 0.01	< 0.01	< 0.01
2009 Bartlett	13 13	120	543		73			< 0.01	< 0.01	< 0.01

Location,		g		g	GS		Residue (mg/kg)			
year, variety	No	ai/ha	L/ha	ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
	14 14)	120	589		74	Mean	0.08	< 0.01	< 0.01	< 0.01
		121 121	580 552		76 78	14	0.08 0.10	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
		119	617		83	Mean	0.09	< 0.01	< 0.01	< 0.01

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2

Table 13 Residues of flutriafol in sweet cherry following application of an SC formulation in the USA (Carringer 2010 1805) (duplicate samples, fruit without pit)

Location,				g	GS		Residue (mg/kg)			
year, variety	No	g ai/ha	L/ha	ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
Conklin, MI, USA, 2009 Napoleon (sweet)	4 (7 7 7)	128 127 128 129	1777 1777 1805 1833	7	75 78 81 83-85	7	0.31 0.32	< 0.01 < 0.01	0.35 0.32 c0.26	0.03 0.03 c0.02
						Mean	0.32	< 0.01	0.34	0.03
Mears, MI, USA, 2009 Golds (sweet)	4 (7 7 7)	128 128 128 129	580 580 580 599	22	75 78 81 85	7	0.26 0.25	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
						Mean	0.26	< 0.01	< 0.01	< 0.01
Plainview, CA, USA, 2009 Tulare (sweet)	4 (7 7 7)	128 128 128 128	1843 1861 1805 1833	7	72 76 78 89	7	0.29 0.21	< 0.01 < 0.01	0.92 0.83 c0.60	0.03 0.03 c0.02
						Mean	0.25	< 0.01	0.88	0.03
Poplar, CA, USA, 2009 Brooks (sweet)	4 (7 7 7)	128 127 128 127	571 617 608 599	22	71 75 79 87	7	0.14 0.19	< 0.01 < 0.01	0.11 0.13 c0.14	< 0.01 < 0.01
						Mean	0.16	< 0.01	0.12	< 0.01
Marsing, ID, USA, 2009 Sweet heart (sweet)	4 (7 7 7)	127 126 126 130	1945 2020 1927 1917	7	78 81 83 86	7	0.66 0.52	< 0.01 < 0.01	< 0.01 < 0.01 c0.12	< 0.01 < 0.01 c0.01
						Mean	0.59	< 0.01	< 0.01	< 0.01
Ephrata, WA, USA, 2009 Bing (sweet)	4 (6 7 7)	129 130 130 130	561 561 561 571	23	75 78 85 87	7	0.40 0.40	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
						Mean	0.40	< 0.01	< 0.01	< 0.01
Weiser, ID, USA, 2009	4 (7 7 7)	128	1422	9	75	0	0.41 0.57	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
						Mean	0.49	< 0.01	< 0.01	< 0.01
Benton (sweet)		131	1431		77	1	0.51 0.45	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
						Mean	0.48	< 0.01	< 0.01	< 0.01
		131	1431		83	3	0.45 0.52	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
						Mean	0.48	< 0.01	< 0.01	< 0.01
		131	1431		85	7	0.46 0.45	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
						Mean	0.46	< 0.01	< 0.01	< 0.01
						14	0.39 0.49	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
						Mean	0.44	< 0.01	< 0.01	< 0.01
						19	0.36 0.38	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
						Mean	0.37	< 0.01	< 0.01	< 0.01
Dallas, OR,	4 (7	128	589	22	75	7	0.35 0.31	< 0.01	< 0.01	< 0.01

Location,				g	GS		Residue (mg/kg)			
year, variety	No	g ai/ha	L/ha	ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
USA, 2009	7 7)	128	589		78			< 0.01	< 0.01	< 0.01
Lambert		128	608		81					
(sweet)		129	608		85					
						Mean	0.33	< 0.01	< 0.01	< 0.01

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2 LOQ 0.01 mg/kg for flutriafol T and TAA and 0.08 mg/kg for TA, however this was based on lowest fortification level and background found in the untreated sample used for spiking. Subsequent work with tart cherries shows an LOQ of 0.01 mg/kg id more appropriate.

Table 14 Residues of flutriafol in tart cherry following application of an SC formulation in the USA (Carringer 2010 1806) (duplicate samples, fruit without pit)

Location,		g			GS		Residue (mg/kg)			
year, variety	No	ai/ha	L/ha	g ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
Alton, NY,	4 (7 7	128	1122	11	75	7	0.45 0.31	< 0.01	0.08	< 0.01
USA, 2009	7)	129	1132		77			< 0.01	0.07	< 0.01
Montmorency		128	1122		79				c0.13	
		130	1141		85					
						Mean	0.38	< 0.01	0.08	< 0.01
Conklin, MI,	4 (7 7	128	580	22	75	0	0.35 0.33	< 0.01	0.12	< 0.01
USA, 2009	7)	128	589		78			< 0.01	0.11	< 0.01
Montmorency		128	589		81				c0.04	
		128	589		85–87	Mean	0.34	< 0.01	0.12	< 0.01
						1	0.35 0.35	< 0.01	0.12	0.01
								< 0.01	0.12	< 0.01
						Mean	0.35	< 0.01	0.12	< 0.01
						3	0.36 0.31	< 0.01	0.12	< 0.01
								< 0.01	0.12	< 0.01
						Mean	0.34	< 0.01	0.12	< 0.01
						7	0.29 0.30	< 0.01	0.11	< 0.01
								< 0.01	0.11	< 0.01
						Mean	0.30	< 0.01	0.11	< 0.01
						14	0.23 0.24	< 0.01	0.11	< 0.01
								< 0.01	0.15	0.01
						Mean	0.24	< 0.01	0.13	< 0.01
						21	0.17 0.20	< 0.01	0.22	0.02
								< 0.01	0.10	0.01
						Mean	0.18	< 0.01	0.16	0.02
Fremont, MI,	4 (6 7	128	1665	8	75	7	0.43 0.35	< 0.01	0.45	0.02
USA, 2009	7)	128	1646		78			< 0.01	0.46	0.03
Montmorency		128	1665		81				c0.29	c0.02
		128	1655		85	Mean	0.39	< 0.01	0.46	0.02
Casnovia,	4 (7 7	129	645	20	75	7	0.33 0.35	< 0.01	0.12	< 0.01
MI, USA,	7)	128	655		78			< 0.01	0.15	0.01
2009		128	655		81				c0.13	
Montmorency		127	664		85	Mean	0.34	< 0.01	0.14	< 0.01
Sturgeon	4 (7 7	128	2750	5	77	7	0.30 0.29	< 0.01	0.04	< 0.01
Bay, WI,	7)	128	2965		81			< 0.01	0.04	< 0.01
USA, 2009		128	3049		84				c0.02	
Montmorency		128	2750		86	Mean	0.30	< 0.01	0.04	< 0.01
Marengo, IL,	4 (7 7	128	636	23	80	7	0.25 0.23	< 0.01	0.12	0.01
USA, 2009	7)	128	673		82			< 0.01	0.12	0.01
Northstar		129	645		85				c0.48	c0.05
		130	599		87	Mean	0.24	< 0.01	0.12	0.01
Perry UT,	4 (8 6	127	2011	6	75	7	0.42 0.41	< 0.01	< 0.01	< 0.01
USA, 2009	7)	128	2048		79			< 0.01	< 0.01	< 0.01
Montmorency		126	2002		81–85	Mean	0.42	< 0.01	< 0.01	< 0.01
		128	1917		85					
Royal City,	4 (7 7	131	571	22	78	7	0.49 0.45	< 0.01	0.01	< 0.01
WA, USA,	7)	129	561		79			< 0.01	0.01	< 0.01
2009		129	561		81	Mean	0.47	< 0.01	0.01	< 0.01

Location,		g			GS		Residue (mg/kg)			
year, variety	No	ai/ha	L/ha	g ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
Montmorency		130	561		85					

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2

Table 15 Residues of flutriafol in peach following application of an SC formulation in Spain (López Benet 2005 2186) (whole fruit basis)

Location, year, variety PEACH	No	g ai/ha	L/ha	g ai/hL	GS (BBCH)	DALA	Crop part	Flutriafol (mg/kg)	% flesh
Bugarra, Valencia,	3 (10	31	998	3.125	77	0	Fruit	0.06	90.7
Spain, 2005 San	11)	32	1004	3.125	78	3		0.06	91
Lorenzo		31	998	3.125	80	7		0.04	92.3
						10		0.06	91.3
						14		0.03	91.4
Jumilla, Murcia,	3 (9	32	1002	3.125	78	0	Fruit	0.11	92.1
Spain, 2005	11)	31	1000	3.125	80	3		0.09	94.0
Kandros		31	1002	3.125	87	7		0.08	92.8
						10		0.05	95.0
						14		0.03	93.2
Sun Late	3 (9	31	1005	3.125	78	0	Fruit	0.11	92.4
	11)	32	1008	3.125	80	3		0.06	95.5
		32	1009	3.125	87	7		0.07	94.7
						10		0.04	93.4
						14		0.03	93.5
Jalance, Valencia,	3 (10	31	1006	3.125	74	0	Fruit	0.07	95.4
Spain, 2005	11)	33	1036	3.125	77	3		0.06	90.3
Cofrentes		31	976	3.125	81	7		0.05	92.2
						10		0.03	93.4
						14		0.04	92.6
Jumilla, Murcia,	3 (10	34	1068	3.13	77	0	Fruit	0.06	93.7
Spain, 2006 Amiga	10)	36	1146	3.13	78	7	Fruit	0.03	94.4
		34	1094	3.13	80		Juice	0.05	94.2
							Marmalade	0.02	94.9
Blanca, Murcia,	3 (11	30	958	3.13	77	0	Fruit	0.04	92.5
Spain, 2006 Elegant	10)	32	1021	3.13	78	7	Fruit	0.05	91.5
Lady		31	1000	3.13	80		Juice	0.04	93.2
							Marmalade	0.05	92.6
Summer Lady	3 (10	32	1030	3.13	77	0	Fruit	0.09	91.4
	10)	30	958	3.13	78	7		0.05	91.9
		31	993	3.13	80				
Jalance, Valencia,	3 (11	31	975	3.13	77	0	Fruit	0.12	93.0
Spain, 2006 Andru	10)	30	978	3.13	81	7		0.08	94.3
		30	961	3.13	85				

Analytical method flutriafol: LARP SOP E050/1

Table 16 Residues of flutriafol in peaches following application of an SC formulation in the USA (Carringer 2010 1807) (duplicate samples, fruit without stone)

Location,		g			GS		Residue (mg/kg)			
year, variety	No	ai/ha	L/ha	g ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
Alton, NY,	4 (8 7	128	1122	11	75	7	0.17 0.21	< 0.01	0.45	0.02
USA, 2009	6)	128	1122		76			< 0.01	0.36	0.02
Red Haven		128	1122		77				c0.24	c0.01
		128	1122		79	Mean	0.19	< 0.01	0.40	0.02
Montezuma,	4 (7 7	128	599	21	77	7	0.16 0.17	< 0.01	0.33	0.03
GA, USA,	7)	127	608		79			< 0.01	0.31	0.02
2009		128	599		81				c0.26	c0.02
Summer Gold		129	589		85	Mean	0.16	< 0.01	0.32	0.02
Chula, GA,	4 (7 7	128	982	13	76	7	0.26 0.21	< 0.01	0.15	0.01
USA, 2009	7)	128	963		77			< 0.01	0.18	0.02
Hawthorne [a]		128	982		81				c0.09	
		127	982		85	Mean	0.24	< 0.01	0.16	0.02
Chula, GA,	4 (7 8	127	664	19	74	0	0.37 0.37	< 0.01	0.17	0.01
USA, 2009	7)	127	664		74			< 0.01	0.16	0.01
June Gold [b]		127	673		75	Mean	0.37	< 0.01	0.16	0.01
		127	673		77	1	0.31 0.26	< 0.01	0.16	0.01
								< 0.01	0.14	0.01
						Mean	0.28	< 0.01	0.15	0.01
						3	0.24 0.20	< 0.01	0.14	0.01
								< 0.01	0.15	0.01
						Mean	0.22	< 0.01	0.14	0.01
						7	0.13 0.16	< 0.01	0.14	0.01
								< 0.01	0.13	0.01
									c0.13	c0.01
						Mean	0.14	< 0.01	0.14	0.01
						14	0.08 0.08	< 0.01	0.09	< 0.01
								< 0.01	0.12	< 0.01
						Mean	0.08	< 0.01	0.10	< 0.01
						21	0.07 0.06	< 0.01	0.13	< 0.01
								< 0.01	0.13	< 0.01
						Mean	0.06	< 0.01	0.13	< 0.01
Pikeville, NC,	4 (6 7	128	1178	11	75	6	0.40 0.42	< 0.01	0.05	< 0.01
USA, 2009	6)	129	1160		75			< 0.01	0.06	< 0.01
New		129	1178		78				c0.04	
Haven		130	1207		81	Mean	0.41	< 0.01	0.06	< 0.01
Deville, LA,	4 (7 8	131	673	19	77	6	0.24 0.23	< 0.01	0.02	< 0.01
USA,	8)	129	673		81			< 0.01	0.02	< 0.01
2009 Regal		127	673		81	Mean	0.24	< 0.01	0.02	< 0.01
		127	655		85					
Conklin, MI,	4 (7 7	127	2020	6	76	7	0.13 0.11	< 0.01	0.16	< 0.01
USA, 2009	7)	128	2011		77			< 0.01	0.16	< 0.01
		128	1973		78				c0.15	
Bellaire		128	1936		79-81	Mean	0.12	< 0.01	0.16	< 0.01
Blanco, TX,	4 (7 7	128	486	26	78	7	0.13 0.13	< 0.01	< 0.01	< 0.01
USA,	7)	129	580		81			< 0.01	< 0.01	< 0.01
2009 Dixieland		130	599		81	Mean	0.13	< 0.01	< 0.01	< 0.01
		129	514		85					
Fresno, CA,	4 (7 7	130	1880	7	81	7	0.20 0.16	< 0.01	0.01	< 0.01
USA, 2009	7)	131	1889		81			< 0.01	0.02	< 0.01
Kaweah		130	1880		85	Mean	0.18	< 0.01	0.02	< 0.01
		130	1889		87					
Kingsburg,	4 (7 7	124	627	20	77	7	0.12 0.18	< 0.01	0.05	< 0.01
CA, USA,	7)	128	645		78			< 0.01	0.04	< 0.01
2009		129	655		79				c0.06	
Fayette		131	636		81	Mean	0.15	< 0.01	0.04	< 0.01
Dinuba, CA,	4 (7 7	127	1814	7	78	7	0.05 0.05	< 0.01	0.01	< 0.01
USA, 2009	7)	128	1833		79			< 0.01	0.01	< 0.01
		128	1852		81				c0.02	
Duchess		129	1861		87	Mean	0.05	< 0.01	0.01	< 0.01

Location,		g			GS		Residue (mg/kg)			
year, variety	No	ai/ha	L/ha	g ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
Portville, CA,	4 (7 6	128	673	19	81	7	0.16 0.20	< 0.01	< 0.01	< 0.01
USA,	8)	129	673		85			< 0.01	< 0.01	< 0.01
2009 Alberta		129	683		85	Mean	0.18	< 0.01	< 0.01	< 0.01
		128	664		87					

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2

[a] Last application 15/09/2009

[b] Last application 12/05/2009

Table 17 Residues of flutriafol in plum following application of an SC formulation in the USA (Carringer 2010 1808) (duplicate samples, fruit without stone)

Location,					GS		Residue (mg/kg)			
year, variety	No	g ai/ha	L/ha	g ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
Conklin, MI,	4 (7 7 7)	129	2002	6	77	7	0.20 0.25	< 0.01	0.34	< 0.01
USA, 2009		128	2002		78			< 0.01	0.31	< 0.01
Stanley		128	2011		79				c0.67	c0.02
		128	2039		85	Mean	0.22	< 0.01	0.32	< 0.01
Fresno, CA,	4 (7 7 7)	129	561	23	81	7	0.02 0.02	< 0.01	0.05	< 0.01
USA, 2009		129	561		81			< 0.01	0.05	< 0.01
Flavor Rich		130	561		85				c0.04	
		130	561		87	Mean	0.02	< 0.01	0.05	< 0.01
Dinuba, CA,	4 (7 7 7)	127	1777	7	81	0	0.05 0.05	< 0.01	0.04	< 0.01
USA, 2009		127	1861		81			< 0.01	0.04	< 0.01
Fryer's		128	1861		85				c0.04	
		128	1814		87	Mean	0.05	< 0.01	0.04	< 0.01
						1	0.03 0.04	< 0.01	0.04	< 0.01
								< 0.01	0.03	< 0.01
						Mean	0.04	< 0.01	0.04	< 0.01
						3	0.04 0.05	< 0.01	0.04	< 0.01
								< 0.01	0.04	< 0.01
						Mean	0.04	< 0.01	0.04	< 0.01
						7	0.03 0.02	< 0.01	0.04	< 0.01
								< 0.01	0.05	< 0.01
						Mean	0.02	< 0.01	0.04	< 0.01
						14	0.03 0.04	< 0.01	0.06	< 0.01
								< 0.01	0.05	< 0.01
						Mean	0.04	< 0.01	0.06	< 0.01
						21	0.03 0.03	< 0.01	0.08	< 0.01
								< 0.01	0.08	< 0.01
						Mean	0.03	< 0.01	0.08	< 0.01
Poplar, CA,	4 (7 7 7)	127	683	19	81	7	0.10 0.11	< 0.01	0.04	< 0.01
USA, 2009		128	617		81			< 0.01	0.05	< 0.01
French prunes		128	683		85	Mean	0.10	< 0.01	0.04	< 0.01
		129	692		87					
Plainview, CA,	4 (7 7 7)	129	1637	8	81	7	0.09 0.09	< 0.01	0.05	< 0.01
USA, 2009		129	1655		85			< 0.01	0.05	< 0.01
prunes		129	1655		85				c0.04	
(French plum)		128	1637		85	Mean	0.09	< 0.01	0.05	< 0.01
Hughson, CA,	4 (7 7 7)	127	608	21	81	7	0.12 0.12	< 0.01	0.05	< 0.01
USA, 2009		127	608		81			< 0.01	0.05	< 0.01
French plum		128	608		81				c0.02	
		127	608		85	Mean	0.12	< 0.01	0.05	< 0.01
Ephrata, WA,	4 (7 7 7)	128	1871	7	77	7	0.03 0.03	< 0.01	< 0.01	< 0.01
USA, 2009		128	1880		79			< 0.01	< 0.01	< 0.01
Italian		128	1871		81	Mean	0.03	< 0.01	< 0.01	< 0.01
		129	1880		85					
Monmouth, OR,	4 (7 7 7)	130	599	22	79	7	0.07 0.06	< 0.01	0.13	< 0.01
USA, 2009		130	599		81			< 0.01	0.12	< 0.01
Moyer		129	599		81				c0.02	

Location,					GS		Residue (mg/kg)			
year, variety	No	g ai/ha	L/ha	g ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
		128	589		85	Mean	0.06	< 0.01	0.12	< 0.01

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2

Table 18 Residues of flutriafol in strawberries (macro- and micro-tunnels) following application of an SC formulation in Spain (López Benet 2005 2582 Partington 2006 2583)

Location, year, variety	No	g ai/ha	L/ha	g ai/hL	GS (BBCH)	DALA	Sample	Flutriafol (mg/kg)
Villanueva de los	3 (10	210	1136	18.5	85	0	Fruit	0.44
Castillejos, Huelva,	10)	170	909	18.7	87	3		0.27
Spain, 2004 Ventana		170	909	18.7	87	5		0.33
						7		0.22
						10		0.05
Finca La Nina,	3 (11	232	1236	18.8	85	0	Fruit	0.14
Almonte, Huelva,	10)	170	909	18.7	87	3		0.07
Spain, 2004 Camarosa		168	897	18.7	87	5		0.09
						7		0.05
						10		0.04
Finca El Lote,	3 (11	250	1327	18.8	85	0	Fruit	0.23
Almonte, Huelva,	10)	175	939	18.6	87	3		0.15
Spain, 2004 Camarosa		170	909	18.7	87	5		0.17
						7		0.09
						10		0.06
Finca Amanto,	3 (11	238	1255	18.9	85	0	Fruit	0.49
Almonte, Huelva,	10)	172	915	18.9	87	3		0.22
Spain, 2004 Camarosa		165	885	18.6	87	5		0.25
						7		0.14
						10		0.13
Almonte, Spain, 2005	3 (10	191	1018	18.75	61	0	Fruit	0.31
Camarosa	10)	189	1009	18.75	87	1	Fruit	0.37
		199	1059	18.75	88	3	Fruit	0.24 0.32
Bonares, Spain, 2005	3 (10	195	1041	18.75	61	0	Fruit	0.29
Camarosa	10)	191	1018	18.75	87	1	Fruit	0.23
		194	1036	18.75	88	3	Fruit	0.18 0.23
Huelva, Spain, 2005	3 (10	197	1050	18.75	61	0	Fruit	0.18
Ventana [a]	10)	178	950	18.75	87	1	Fruit	0.16
		194	1032	18.75	88	3	Fruit	0.15 0.13
Ventana [a]	3 (10	194	1034	18.75	61	0	Fruit	0.37
	10)	192	1023	18.75	87	1	Fruit	0.33
		195	1041	18.75	88	3	Fruit	0.24 0.31

Analytical method flutriafol: LARP SOP E033/1

[a] Similar location, same date for last application

Table 19 Residues of flutriafol in strawberries following application of an SC formulation in the USA and Canada (Carringer 2011 2158) (duplicate samples, applications include non-ionic surfactant)

Location, year,					GS		Residue (mg/kg)			
variety	No	g ai/ha	L/ha	g ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
East Williamson,	4 (4 7 7)	129 128	281 281	46	73 74	0	0.19 0.09	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
NY, USA, 2010 Idea		129 126	281 281		75 87	Mean	0.14	< 0.01	< 0.01	< 0.01
Seven Springs, NC, USA, 2010	4 (7 8 6)	129 123	430 412	30	86 86	0	0.19 0.30	< 0.01 < 0.01	0.01 0.01	< 0.01 < 0.01
Camino Real		131 126	421 402		87 88	Mean	0.24	< 0.01	0.01	< 0.01
Lawtly, FL,	4 (7	128	262	49	71–73	0	0.42 0.31	< 0.01	0.07	< 0.01

Location, year,					GS		Residue (mg/kg)			
variety	No	g ai/ha	L/ha	g ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
USA, 2010	7 8)	128	253		81			< 0.01	0.07	< 0.01
Camarosa		127 130	262 262		85 87	Mean	0.36	< 0.01	0.07	< 0.01
Richland, IA, USA, 2010	4 (8 6 7)	130 123	262 243	50	65 81	0	0.41 0.42	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
Extra sweet		126 127	253 243		81 87	Mean	0.42	< 0.01	0.02	< 0.01
Brantford ON, CAN, 2010	4 (7 8 7)	131 131	355 355	37	59–65 61–71	0	0.58 0.52	< 0.01 < 0.01	0.01 0.01	< 0.01 < 0.01
Sapphire		136 127	365 337		67–73 81–87	Mean	0.55	< 0.01	0.01	< 0.01
Brampton, ON, CAN, 2010	5 (7 7	137 130	365 346	38	59–65 65–67	0 (after 4th)	0.58 0.73	< 0.01 < 0.01	0.01 0.01	< 0.01 < 0.01
Mira	8 8)	128 136	346 365		65–73 67–73	Mean	0.66	< 0.01	0.01	< 0.01
		135	355	38	85–87	0 (after 5th)	0.43 0.47	< 0.01 < 0.01	0.01 0.01	< 0.01 < 0.01
						Mean	0.45	< 0.01	0.01	< 0.01
Salinas, CA, USA, 2010	4 (6 8	126 121	449 430	28	71–81 83	0	0.73 0.53	< 0.01 < 0.01	0.08 0.07	< 0.01 < 0.01
Albion	7)	129 132	468 486		73–85 89	Mean	0.63	< 0.01	0.08	< 0.01
Porterville, CA, USA, 2010	4 (6 8 7)	129 127	327 327	39	71–83 73–83	0	0.31 0.29	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
Diamante [a]		129 128	327 327		71–83 85–87	Mean	0.30	< 0.01	0.02	< 0.01
Porterville, CA, USA, 2010	4 (7 7 6)	127 127	290 290	44	73–81 73–81	0	0.67 0.78	< 0.01 < 0.01	0.07 0.06	< 0.01 < 0.01
Diamante [b]		128	327		73–85	Mean	0.72	< 0.01	0.06	< 0.01
		128	327		85–87	1	0.63 0.47	< 0.01 < 0.01	0.09 0.06	< 0.01 < 0.01
						Mean	0.55	< 0.01	0.08	< 0.01
						3	0.69 0.52	NA	NA	NA
						Mean	0.60			
						5	0.42 0.54	< 0.01 < 0.01	0.09 0.08	< 0.01 < 0.01
						Mean	0.48	< 0.01	0.08	< 0.01
						7	0.13 0.15	< 0.01 < 0.01	0.03 0.02	< 0.01 < 0.01
						Mean	0.14	< 0.01	0.02	< 0.01
						10	0.08 0.08	< 0.01 < 0.01	0.02 0.03	< 0.01 < 0.01
						Mean	0.08	< 0.01	0.02	< 0.01
Elmira, OR, USA, 2010	4 (7 7 6)	129 127	290 281	20	73–85 73–85	0	0.44 0.45	< 0.01 < 0.01	0.01 0.01	< 0.01 < 0.01
Benton		131 127	299 281		73–85 87	Mean	0.44	< 0.01	0.01	< 0.01

Induce 0.25% v/v, Induce 0.14–0.28% v/v, Induce 0.25% v/v, Activator 90 0.25% v/v, Agral 90 0.5% v/v, Agral 90 0.5% /v, Pro 90 0.25% v/v, Pro 90 0.5% v/v, Pro 90 0.25% v/v, Dyne-Amic 0.25% v/v.

NA=not analysed

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2

[a] Last application 16/06/2010

[b] Last application 02/06/2010, different location to other Porterville trial [a]

Table 20 Residues of flutriafol in cabbage and broccoli following application of an SC formulation in the USA (Carringer 2013 2697) (duplicate samples, applications include non-ionic surfactant)

Location, year,				GS			Residue (mg/kg)			
variety	No	g ai/ha	L/ha	(BBCH)	DALA	Sample	Flutriafol	T	TA	TAA

Location, year,				GS			Residue (mg/kg)			
variety	No	g ai/ha	L/ha	(BBCH)	DALA	Sample	Flutriafol	T	TA	TAA
CABBAGE										
Alton, NY, USA, 2012 Blue lagoon	4 (7 7	128 127 127	281 281 281	18 41 42	0	Heads	2.64 2.68	< 0.01 < 0.01	0.12 0.13 c0.08	< 0.01 0.01
	7)	128	281	46	Mean		2.66	< 0.01	0.12	< 0.01
					3	Heads	0.62 0.79	< 0.01 < 0.01	0.14 0.12	< 0.01 < 0.01
					Mean		0.70	< 0.01	0.13	< 0.01
					7	Heads	0.46 0.43	< 0.01 < 0.01	0.12 0.13	< 0.01 < 0.01
					Mean		0.44	< 0.01	0.12	< 0.01
					10	Heads	0.33 0.33	< 0.01 < 0.01	0.08 0.11	< 0.01 < 0.01
					Mean		0.33	< 0.01	0.10	< 0.01
					14	Heads	0.30 0.27	< 0.01 < 0.01	0.10 0.12	< 0.01 < 0.01
					Mean		0.28	< 0.01	0.11	< 0.01
Seven Springs, NC, USA, 2011 Bravo	4 (7 7	129 129 131	290 299 299	41 41 42	7	Heads	0.80 0.68	< 0.01 < 0.01	0.04 0.04 c0.02	< 0.01 < 0.01
	7)	127	290	44	Mean		0.74	< 0.01	0.04	< 0.01
Oviedo, FL USA, 2011 Cheers	4 (6 6	128 127 128	281 281 281	42 44 46	8	Heads	0.22 0.18	< 0.01 < 0.01	0.05 0.05 c0.02	< 0.01 < 0.01
	7)	128	281	48	Mean		0.20	< 0.01	0.05	< 0.01
Conklin, MI, USA, 2012 Megaton	4 (7 7	129 129 128	48 49 47	41–42 42–43 43–44	7	Heads	0.13 0.08	< 0.01 < 0.01	0.07 0.07 c0.02	< 0.01 < 0.01
	7)	128	47	46–47	Mean		0.10	< 0.01	0.07	< 0.01
Uvalde, TX, USA, 2011	4 (7	128 127	187 178	46 47	7	Heads	0.07 0.08	< 0.01 < 0.01	0.01 0.01	< 0.01 < 0.01
Pennant	7	131	168	48	Mean		0.08	< 0.01	0.01	< 0.01
	7)	128	206	49						
Porterville, CA, USA, 2011	4 (7	127 130	45 50	45 47	7	Heads	0.13 0.05	< 0.01 < 0.01	0.03 0.04	< 0.01 < 0.01
Supreme	7	128	48	48	Mean		0.09	< 0.01	0.04	< 0.01
Vantage	7)	129	49	49						
BROCCOLI										
Uvalde, TX, USA, 2011	4 (7	128 128	47 47	41 43	6	Heads	0.18 0.10	< 0.01 < 0.01	0.04 0.03	< 0.01 < 0.01
Green Magic	7 7)	128 128	47 47	43 48	Mean		0.14	< 0.01	0.04	< 0.01
Porterville, CA, USA, 2012	4 (7	128 128	365 365	42 45	0	Heads	0.24 0.24	< 0.01 < 0.01	0.04 0.04	< 0.01 < 0.01
Heritage [a]	7	128	365	45	Mean		0.24	< 0.01	0.04	< 0.01
	7)	129	365	49	3	Heads	0.11 0.07	< 0.01 < 0.01	0.04 0.04	< 0.01 < 0.01
					Mean		0.09	< 0.01	0.04	< 0.01
					7	Heads	0.07 0.08	< 0.01 < 0.01	0.04 0.05	< 0.01 < 0.01
					Mean		0.08	< 0.01	0.04	< 0.01
					10	Heads	0.12 0.08	< 0.01 < 0.01	0.04 0.04	< 0.01 < 0.01
					Mean		0.10	< 0.01	0.04	< 0.01
					14	Heads	0.07 0.07	< 0.01 < 0.01	0.04 0.04	< 0.01 < 0.01
					Mean		0.07	< 0.01	0.04	< 0.01
King City, CA, USA, 2011 Legacy	4 (7 7	128 131 130	299 309 309	46 47 47	7	Heads	0.20 0.17	< 0.01 < 0.01	0.02 0.02 c0.01	< 0.01 < 0.01
	6)	128	299	49	Mean		0.18	< 0.01	0.02	< 0.01
Porterville, CA,	4	129	48	47	7	Heads	0.21 0.27	< 0.01	0.10	< 0.01

Location, year,				GS			Residue (mg/kg)			
variety	No	g ai/ha	L/ha	(BBCH)	DALA	Sample	Flutriafol	T	TA	TAA
USA, 2011	(6	129	47	47				< 0.01	0.09	< 0.01
Heritage [b]	7	129	49	47					c0.02	
	7)	129	48	49	Mean		0.24	< 0.01	0.10	< 0.01
Santa Maria,	4	128	281	41	7	Heads	0.36 0.34	< 0.01	0.02	< 0.01
CA, USA, 2011	(8	128	281	43				< 0.01	0.02	< 0.01
Heritage	7	130	281	43	Mean		0.35	< 0.01	0.02	< 0.01
	6)	128	281	46						
Hilsboro, OR,	4	162	187	18–19	7	Heads	0.05 0.08	< 0.01	0.51	< 0.01
USA, 2011 Bay	(8	123	187	21				< 0.01	0.52	< 0.01
Meadows	7	127	187	42–43					c0.20	
	7)	127	187	42	Mean		0.06	< 0.01	0.52	< 0.01

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2

Induce 0.5% v/v, Induce 0.29-0.41% v/v, Triangle D-W Surfactant 0.25% v/v, R11 0.06% v/v, Induce 0.25% v/v, Pro 90 0.25% v/v, Induce 0.25% v/v, Pro 90 0.5% v/v, Pro 90 0.5% v/v, Pro 90 0.5% v/v, DyneAmic 0.38% v/v, Induce 0.13% v/v

[a] Last application 29/05/2012

[b] Last application 29/11/2011, different location to other Porterville trial [a]

Table 21 Residues of flutriafol in cucumber application of an SC formulation in the USA (Carringer 2012 2439) (duplicate samples, applications include non-ionic surfactant)

Location, year,		g		g	GS		Residue (mg/kg)			
variety	N	ai/ha	L/ha	ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
Seven Springs,	4 (7	129	150	82	14	0	0.05 0.07	< 0.01	0.10	< 0.01
NC, USA, 2011	7 7)	131	159		51			< 0.01	0.12	< 0.01
Lancer 152		129	159		61				c0.03	
		128	159		71	Mean	0.06	< 0.01	0.11	< 0.01
						3	0.05 0.07	< 0.01	0.15	< 0.01
								< 0.01	0.15	< 0.01
						Mean	0.06	< 0.01	0.15	< 0.01
						7	0.02 0.04	< 0.01	0.14	< 0.01
								< 0.01	0.14	< 0.01
						Mean	0.03	< 0.01	0.14	< 0.01
						10	0.03 0.02	< 0.01	0.15	< 0.01
								< 0.01	0.18	< 0.01
						Mean	0.02	< 0.01	0.16	< 0.01
						14	0.02 0.02	< 0.01	0.32	< 0.01
								< 0.01	0.24	< 0.01
						Mean	0.02	< 0.01	0.28	< 0.01
Chula, GA,	4 (7	128	46	278	54	0	0.02 0.03	< 0.01	0.06	< 0.01
USA, 2011	7 7)	127	47		68			< 0.01	0.06	< 0.01
Thunder		129	46		75				c0.02	
		127	46		78	Mean	0.02	< 0.01	0.06	< 0.01
Newberry, FL,	4 (7	128	225	57	54	0	0.04 0.04	< 0.01	0.05	< 0.01
USA, 2011	7 7)	124	253		67			< 0.01	0.05	< 0.01
Thunder		131	234		72				c0.01	
		126	234		77	Mean	0.04	< 0.01	0.05	< 0.01
Conklin, MI,	4 (7	129	215	60	63	0	0.03 0.04	< 0.01	0.09	< 0.01
USA, 2011	7 7)	127	215		69			< 0.01	0.09	< 0.01
Impact		128	206		70	Mean	0.04	< 0.01	0.09	< 0.01
		128	206		73					
Delavan, WI,	4 (7	129	196	66	82	0	0.02 0.01	< 0.01	0.02	< 0.01
USA, 2011	7 7)	128	206		83			< 0.01	0.02	< 0.01
Marketmore 76		129	196		84	Mean	0.02	< 0.01	0.02	< 0.01
		130	206		89					
Richland, IA,	4 (7	129	150	86	65	0	0.04 0.03	< 0.01	0.05	< 0.01
USA, 2011	6 7)	129	150		67			< 0.01	0.04	< 0.01
Straight Eight		128	150		75				c0.03	
		129	140		88	Mean	0.04	< 0.01	0.04	< 0.01
Branchton, ON,	4 (7	114	43	265	71	0	0.06 0.05	< 0.01	0.06	< 0.01

Location, year, variety	N	g ai/ha	L/ha	g ai/hL	GS (BBCH)	DALA	Residue (mg/kg) Flutriafol	T	TA	TAA
CAN, 2011 Talladega	7 7)	117 129	41 49		85 87-89			< 0.01	0.06 c0.03	< 0.01
		126	47		89	Mean	0.06	< 0.01	0.06	< 0.01
Uvalde, TX, USA, 2011	4 (7 7 7)	130 129	187 253	51	71 75	0	0.05 0.04	< 0.01 < 0.01	0.03 0.03	< 0.01 < 0.01
Stonewall		127 132	243 234		77 79	Mean	0.04	< 0.01	0.03	< 0.01
Hillsboro, OR, USA, 2011 Raider F1	4 (7 7 7)	127 131 129	234 243 234	54	51-71 61-83 61-83	0	0.03 0.03	< 0.01 < 0.01	0.05 0.05 c0.07	< 0.01 < 0.01
		129	234		61-85	Mean	0.03	< 0.01	0.05	< 0.01

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2

Induce 0.4–0.5% v/v, Induce 0.25% v/v, Induce 0.25% v/v, R-11 0.06% v/v, Preference 0.5% v/v, Preference 0.25% v/v, Agral 90 0.25% v/v, Induce 0.25–0.26% v/v, Induce 0.5% v/v

Table 22 Residues of flutriafol in summer squash application of an SC formulation in the USA (Carringer 2012 2439) (duplicate samples, applications include non-ionic surfactant)

Location, year, variety	N	g ai/ha	L/ha	g ai/hL	GS (BBCH)	DALA	Residue (mg/kg) Flutriafol	T	TA	TAA
Alton, NY, USA, 2011Superpik F1	4 (7 7 7)	127 129 128 129	281 290 281 290	45 44 46 44	63 65 71 75	0	0.05 0.05	< 0.01 < 0.01	0.04 0.06	< 0.01 < 0.01
						Mean	0.05	< 0.01	0.05	< 0.01
Chula, GA, USA, 2011 Dixie	4 (7 7 7)	127 129 130 131	234 234 243 243	54 55 53 53	15 62 81 89	0	0.04 0.05	< 0.01 < 0.01	0.08 0.07 c0.04	< 0.01 < 0.01
						Mean	0.04	< 0.01	0.08	< 001
Newberry, FL, USA, 2011 Dixie	4 (7 7 7)	128 128 124 127	234 234 225 234	55 55 55 54	16 61 71 89	0	0.05 0.05	< 0.01 < 0.01	0.07 0.11	< 0.01 < 0.01
						Mean	0.05	< 0.01	0.09	< 0.01
Conklin, MI, CAN, 2011 Black Beauty	4 (7 7 7)	129 128 128 128	225 215 215 206	57 60 60 62	12 63 70 71	0	0.04 0.03	< 0.01 < 0.01	0.06 0.06	< 0.01 < 0.01
						Mean	0.04	< 0.01	0.06	< 0.01
Richland, IA, USA, 2011 Black Beauty	4 (8 7 7)	128 131 129 129	159 168 206 206	81 78 63 63	51 54 73 86	0	0.06 0.06	< 0.01 < 0.01	< 0.03 0.03	< 0.01 < 0.01
						Mean	0.06	< 001	< 0.03	< 0.01
Branchton, ON, CAN, 2011 Senator	4 (7 7 7)	126 130 130 123	49 49 48 45	257 265 265 273	69–72 84–89 85–89 70–89	0	0.06 0.07	< 0.01 < 0.01	0.04 0.05	< 0.01 < 0.01
						Mean	0.06	< 0.01	0.04	< 0.01
Porterville, CA, USA, 2011	4 (6 8 7)	127	49	259	62	0	0.05 0.05	< 0.01 < 0.01	0.03 < 0.03	< 0.01 < 0.01
						Mean	0.05	< 0.01	< 0.03	< 0.01
Black Beauty		129	49	263	65	3	0.05 0.06	< 0.01 < 0.01	0.05 0.04	< 0.01 < 0.01
						Mean	0.06	< 0.01	0.04	< 0.01
		126	48	263	72	7	0.03 0.03	< 0.01 < 0.01	0.04 0.05	< 0.01 < 0.01
						Mean	0.03	< 0.01	0.04	< 001
		128	49	261	74	10	0.03 0.03	< 0.01	0.04	< 0.01

Location, year,		g		g	GS		Residue (mg/kg)			
variety	N	ai/ha	L/ha	ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
								< 0.01	0.04	< 0.01
						Mean	0.03	< 001	0.04	< 0.01
						14	0.03 0.03	< 0.01 < 0.01	0.04 0.04	< 0.01 < 0.01
						Mean	003	< 001	0.04	< 0.01
Hillsboro, OR, USA, 2011 Zucchini RSQ5119	4 (7 7 7)	128 131 128 128	234 243 234 234	55 54 55 55	51–71 61–83 61–83 61–85	0	0.04 0.04	< 0.01 < 0.01	0.03 < 0.03	< 0.01 < 0.01
						Mean	0.04	< 0.01	< 0.03	< 0.01

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2

Induce 0.5% v/v, Induce 0.25% v/v, Induce 0.25% v/v, R-11 0.06% v/v, Preference 0.25–0.26% v/v, Agral 90 0.24–0.25% v/v, Pro 90 0.25% v/v, Induce 0.5% v/v

Table 23 Residues of flutriafol in muskmelon application of an SC formulation in the USA (Carringer 2012 2439) (duplicate samples, applications include non-ionic surfactant)

Location, year,		g		g	GS			Residue (mg/kg)			
variety	N	ai/ha	L/ha	ai/hL	(BBCH)	DALA	Sample	Flutriafol	T	TA	TAA
Chula, GA, USA, 2011	4 (7 6 6)	127 131	234 159	54 82	73 76	0	Fruit	0.06 0.08	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
Athena		129	150	86	83	Mean		0.07	< 0.01	0.02	< 0.01
		128	150	86	89	0	Pulp	0.01 < 0.01	< 0.01 < 0.01	0.03 0.03	< 0.01 < 0.01
						Mean		< 0.01	< 0.01	0.03	< 0.01
						0	Peel	0.17 0.13	< 0.01 < 0.01	0.02 0.01	< 0.01 < 0.01
						Mean		0.15	< 0.01	0.02	< 0.01
Conklin, MI, USA, 2011	4 (7 7 7)	128 128	206 206	62 62	70 70	0	Fruit	0.04 0.05	< 0.01 < 0.01	0.07 0.07	< 0.01 < 0.01
Minerva		127	215	59	70	Mean		0.04	< 0.01	0.07	< 0.01
		127	206	62	87–89	0	Pulp	0.01 0.02	< 0.01 < 0.01	0.06 0.06	< 0.01 < 0.01
						Mean		0.02	< 0.01	0.06	< 0.01
						0	Peel	0.12 0.13	< 0.01 < 0.01	0.06 0.07	< 0.01 < 0.01
						Mean		0.12	< 0.01	0.06	< 0.01
Richland, IA, USA, 2011	4 (7 7 7)	129 129	159 196	81 66	71 74	0	Fruit	0.10 0.10	< 0.01 < 0.01	0.03 0.03	< 0.01 < 0.01
Delicious 51		129 131	196 196	66 67	82 89	Mean		0.10	< 0.01	0.03	< 0.01
Branchton, ON, CAN, 2011	4 (7 7 7)	129 118	46 43	280 274	79–82 71–81	0	Fruit	0.13 0.11	< 0.01 < 0.01	0.06 0.05	< 0.01 < 0.01
Primo		141 124	52 44	271 282	86–88 89	Mean		0.12	< 0.01	0.06	< 0.01
Uvalde, TX, USA, 2011	4 (7 7 7)	129 130	253 253	51 51	69 71	0	Fruit	0.09 0.12	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Rocket F1		127	225	56	72	Mean		0.10	< 0.01	< 0.01	< 0.01
		129	225	56	82	0	Pulp	< 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	Peel	0.15 0.22	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
						Mean		0.18	< 0.01	< 0.01	< 0.01
Porterville, CA, USA, 2011	4 (7 7 7)	129 128	262 262	49 49	71 79	0	Fruit	0.01 0.01	< 0.01 < 0.01	0.01 0.01	< 0.01 < 0.01
Green Flesh		129	262	49	82	Mean		0.01	< 0.01	0.01	< 0.01
		128	262	49	88	3	Fruit	0.01 0.02	< 0.01 < 0.01	0.01 0.01	< 0.01 < 0.01

Location, year, variety	N	g ai/ha	L/ha	g ai/hL	GS (BBCH)	DALA	Sample	Residue (mg/kg) Flutriafol	T	TA	TAA
						Mean		0.02	< 0.01	0.01	< 0.01
						7	Fruit	< 0.01 < 0.01	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
						Mean		< 0.01	< 0.01	0.02	< 0.01
						10	Fruit	< 0.01 < 0.01	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
						Mean		< 0.01	< 0.01	0.02	< 0.01
						14	Fruit	< 0.01 0.03[AB] [0.03 0.03 0.02]	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
						Mean		0.02	< 0.01	0.02	< 0.01
Visalia, CA, USA, 2011	4 (7 7 7)	128 129	51 51	251 253	86 87	0	Fruit	0.08 0.09	< 0.01 < 0.01	0.05 0.05	< 0.01 < 0.01
Hale's Best Jumbo		128 131	51 53	251 247	88 89	Mean		0.08	< 0.01	0.05	< 0.01
Porterville; CA, USA, 2011	4 (7 7 7)	127 128	262 262	48 49	86 87	0	Fruit	0.10 0.15	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
Hale's Best		128	262	49	88	Mean		0.12	< 0.01	0.02	< 0.01
Jumbo		128	262	49	89	0	Pulp	0.02 0.02	< 0.01 < 0.01	0.02 0.01	< 0.01 < 0.01
						Mean		0.02	< 0.01	0.02	< 0.01
						0	Peel	0.23 0.20	< 0.01 < 0.01	0.02 0.01	< 0.01 < 0.01
						Mean		0.22	< 0.01	0.02	< 0.01

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2

Induce 0.25% v/v, R-11 0.06% v/v, Preference 0.25% v/v, Agral 90 0.25% v/v, Induce 0.25-0.26% v/v, Pro 90 0.25% v/v, Pro 90 0.25% v/v, Pro 90 0.25% v/v, Induce 0.5% v/v, Induce 0.25% v/v

[a] Mean of triplicate analysis, individual results in brackets

[b] Last application 19/08/2011

[c] Last application 21/09/2011, same location as other Porterville trial [b] but considered independent as one month between crops and different varieties involved

Table 24 Residues of flutriafol in greenhouse tomato from trials in Spain using an SC formulation (Gimeno 2004a 1263; Gimeno 2004b 1267;López Benet 2004 1262, López Benet 2004 1266)

Location, year, variety	No	g ai/ha	L/ha	g ai/hL	GS (BBCH)	DALA	Sample	Flutriafol (mg/kg)
Picasent, Valencia,	3	179	1017	18.75	83	0	Fruit	0.07
Spain, 2003 Bou	(10	179	1017	18.75	85	3		0.11
	10)	174	989	18.75	87	7		0.15
						14		0.16
						21		0.09
Meliana, Valencia,	3	176	1000	18.75	83	0	Fruit	0.16
Spain, 2003 Gardel	(10	176	1000	18.75	85	3		0.23
	10)	175	1000	18.75	87	7		0.24
						14		0.18
						21		0.18
El Ejido, Almeria,	3	178	1014	18.75	82	0	Fruit	0.16
Spain, 2003 Brillante	(10	178	1014	18.75	84	3		0.14
	10)	175	993	18.75	87	7		0.06
						14		0.1
						21		0.1
El Ejido, Almeria,	3	180	1029	18.75	82	0	Fruit	0.24
Spain, 2003 Zinal	(10	176	1000	18.75	84	3		0.15
	10)	180	1029	18.75	87	7		0.15

Location, year, variety	No	g ai/ha	L/ha	g ai/hL	GS (BBCH)	DALA	Sample	Flutriafol (mg/kg)
						14		0.14
						21		0.09
Picasent, Valencia,	3	188	1004	18.75	87	0	Fruit	0.15
Spain, 2004	(10	187	996	18.75	88	3	Fruit	0.19
Marmande Raf	10)	190	1019	18.75	89	3	Preserved	0.05
						3	Juice	0.07
						7	Fruit	0.17
						7	Preserved	0.06
						7	Juice	0.06
Meliana, Valencia,	3	185	989	18.75	87	0	Fruit	0.12
Spain, 2004 Gardel	(10	183	976	18.75	88	3	Fruit	0.09
	10)	184	979	18.75	89	3	Preserved	0.05
						3	Juice	0.05
						7	Fruit	0.13
						7	Preserved	0.05
						7	Juice	0.04
Almeria, Spain, 2004	3	183	975	18.75	81	0	Fruit	0.18
Durintia	(10	188	1000	18.75	83	3	Fruit	0.14
	11)	184	980	18.75	85	3	Preserved	0.08
						3	Juice	0.08
						7	Fruit	0.15
						7	Preserved	0.06
						7	Juice	0.07
Almeria, Spain, 2004	3[a]	225	1200	18.75	81	0	Fruit	0.15
Tirade		228	1220	18.75	82	3	Fruit	0.16
		224	1200	18.75	82	3	Preserved	0.11
						3	Juice	0.12
						7	Fruit	0.15
						7	Preserved	0.13
						7	Juice	0.1

Table 25 Residues of flutriafol in tomato following application of an SC formulation in the USA (Carringer 2012 2440) (duplicate samples, applications include non-ionic surfactant)

Location, year,		g		g	GS		Residue (mg/kg)			
variety	No	ai/ha	L/ha	ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
Germansville, PA,	4	131	48	273	81	0	0.08 0.06	< 0.01	< 0.01	< 0.01
USA, 2011	(7	132	48		83			< 0.01	< 0.01	< 0.01
Mountain Spring	7	135	49		85	Mean	0.07	< 0.01	< 0.01	< 0.01
	7)	132	49		87					
Seven Springs,	4	131	159	82	61	0	0.10 0.13	< 0.01	0.06	< 0.01
NC, USA, 2011	(7	129	159		71			< 0.01	0.06	< 0.01
Homestead	7	127	159		72				c0.02	
	7)	129	159		82	Mean	0.12	< 0.01	0.06	< 0.01
Greenville, FL,	4	128	234	55	71	0	0.17 0.13	< 0.01	0.01	< 0.01
USA, 2011 Amelia	(7	127	225		74			< 0.01	0.02	< 0.01
	7	128	225		79				c0.03	
	7)	127	225		79	Mean	0.15	< 0.01	0.02	< 0.01
Greenville, FL,	4	128	243	53	73	0	0.12 0.12	< 0.01	0.02	< 0.01
USA, 2011 6-02	(7	128	253		75			< 0.01	0.02	< 0.01
	7	128	253		77				c0.01	
	7)	128	262		81	Mean	0.12	< 0.01	0.02	< 0.01
Richland, IA,	4	129	140	92	72	0	0.07 0.04	< 0.01	< 0.01	< 0.01
USA, 2011	(7	128	206		75			< 0.01	0.01	< 0.01
Rutgers	7	130	140		81				c0.01	
	7)	131	140		87	Mean	0.06	< 0.01	< 0.01	< 0.01
Carlyle, IL, USA,	4	127	243	52	71	0	0.06 0.06	< 0.01	0.04	< 0.01
2011 La Roma	(7	128	253		76			< 0.01	0.05	< 0.01
	7	129	253		79				c0.03	
	7)	129	262		81	Mean	0.06	< 0.01	0.04	< 0.01

Location, year,		g		g	GS		Residue (mg/kg)			
variety	No	ai/ha	L/ha	ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
Wyoming, IL,	4	127	178	71	78–79	0	0.07 0.05	< 0.01	0.02	< 0.01
USA, 2011 Better	(7	129	187		81			< 0.01	0.02	< 0.01
Boy	7	127	187		82–83				c0.01	
	7)	130	187		85	Mean	0.06	< 0.01	0.02	< 0.01
Delavan, WI,	4	129	225	57	74	0	0.12 0.08	< 0.01	< 0.01	< 0.01
USA, 2011 Sweet	(7	129	206		79			< 0.01	< 0.01	< 0.01
Treat (cherry)	7	128	196		83	Mean	0.10	< 0.01	< 0.01	< 0.01
	7)	129	196		89					
Sparta, MI, USA,	4	128	206	62	71	0	0.05 0.05	< 0.01	< 0.01	< 0.01
2011 Sunoma	(7	128	206		80			< 0.01	< 0.01	< 0.01
(Red Roma)	7	128	206		81–82	Mean	0.05	< 0.01	< 0.01	< 0.01
	7)	127	206		83					
Conklin, MI, USA,	4	128	215	60	71	0	0.04 0.05	< 0.01	< 0.01	< 0.01
2011 Big	(7	127	206		80			< 0.01	< 0.01	< 0.01
Beef	7	128	215		81–82	Mean	0.04	< 0.01	< 0.01	< 0.01
	7)	127	215		82–83					
Branchton, ON,	4	122	46	265	69	0	0.06 0.07	< 0.01	0.03	< 0.01
CAN, 2011	(7	132	47		69			< 0.01	0.05	< 0.01
Biltmore	7	131	47		79–81				c0.05	
	7)	123	46		73–79	Mean	0.06	< 0.01	0.04	< 0.01
Burford, ON,	4	128	290	44	79–80	0	0.32 0.34	< 0.01	0.02	< 0.01
CAN, 2011 Sweet	(7	122	281		81–82			< 0.01	0.02	< 0.01
Million	7	121	290		85–86				c0.01	
(cherry)	7)	119	290		87	Mean	0.33	< 0.01	0.02	< 0.01
Porterville, CA,	4	130	299	43	87	0	0.14 0.15	< 0.01	< 0.01	< 0.01
USA, 2011 Roma	(7	130	299		88			< 0.01	< 0.01	< 0.01
VF [a]	8	131	290		89	Mean	0.14	< 0.01	< 0.01	< 0.01
	6)	129	299		89					
	4	637	299	213	87	0	0.63 0.47	< 0.01	< 0.01	< 0.01
	(7	642	290		88			< 0.01	< 0.01	< 0.01
	8	641	290		89	Mean	0.55	< 0.01	< 0.01	< 0.01
	6)	644	299		89					
Champion [a]	4	129	262	49	83	0	0.09 0.12	< 0.01	< 0.01	< 0.01
	(7	128	262		85			< 0.01	< 0.01	< 0.01
(Fresh Market)	7	128	262		87	Mean	0.10	< 0.01	< 0.01	< 0.01
	7)	128	262		88	3	0.08 0.13	< 0.01	< 0.01	< 0.01
								< 0.01	< 0.01	< 0.01
						Mean	0.10	< 0.01	< 0.01	< 0.01
						7	0.08 0.09	< 0.01	< 0.01	< 0.01
								< 0.01	< 0.01	< 0.01
						Mean	0.08	< 0.01	< 0.01	< 0.01
						14	0.05 0.05	< 0.01	< 0.01	< 0.01
								< 0.01	0.02	< 0.01
						Mean	0.05	< 0.01	< 0.02	< 0.01
						21	0.08 0.09	< 0.01	0.01	< 0.01
								< 0.01	0.01	< 0.01
						Mean	0.08	< 0.01	0.01	< 0.01
Visalia, CA, USA,	4	127	51	249	86	0	0.09 0.16	< 0.01	0.01	< 0.01
2011 AB2	(7	128	51		87			< 0.01	0.01	< 0.01
(Roma Processing)	7	127	51		88	Mean	0.12	< 0.01	0.01	< 0.01
	7)	129	51		89					
King City, CA,	4	128	281	46	85	0	0.07 0.10	< 0.01	< 0.01	< 0.01
USA, 2011	(7	129	290		86			< 0.01	< 0.01	< 0.01
Champion (Fresh	7	129	290		88	Mean	0.08	< 0.01	< 0.01	< 0.01
Market)	7)	129	281		89					
Porterville, CA,	4	128	309	41	79	0	0.17 0.18	< 0.01	< 0.01	< 0.01
USA, 2011 AB2	(7	128	309		86			< 0.01	< 0.01	< 0.01
(Roma Processing)	7	128	309		87	Mean	0.18	< 0.01	< 0.01	< 0.01
[b]	7)	129	309		89					
Corning, CA,	4	132	187	71	81	0	0.38 0.43	< 0.01	0.02	< 0.01
USA, 2011 Sun	(7	132	187		83			< 0.01	0.02	< 0.01
6366	7	132	187		87	Mean	0.40	< 0.01	0.02	< 0.01

Location, year,		g		g	GS		Residue (mg/kg)			
variety	No	ai/ha	L/ha	ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
	7)	131	187		89					
Paso Robles, CA,	4	130	384	34	84	0	0.42 0.42	< 0.01	< 0.01	< 0.01
USA, 2011	(6	128	374		85			< 0.01	< 0.01	< 0.01
Washington cherry	7	129	374		87	Mean	0.42	< 0.01	< 0.01	< 0.01
	7)	128	374		88					

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2

Induce 0.125% v/v, Induce 0.3–0.48% v/v, Induce 0.25% v/v, Induce 0.25% v/v, Preference 0.25% v/v, NIS 0.25% v/v, Aquagene 90 0.05% v/v, preference 0.5% v/v, R-11 0.065% v/v, R-11 0.064% v/v, Agral 90 0.25% v/v, Agral 90 0.25% v/v, Pro 90 0.5% v/v, Pro 90 0.5% v/v, Pro 90 0.25% v/v, Pro 90 0.25% v/v, Pro 90 0.25% v/v, Pro 90 0.25% v/v, R-11 0.096% v/v, Dyne-Amic 0.5% v/v.

[a] Last application 12/09/2011 for Roma VF and 14/09/2011 for Champion

[b] Last application 08/08/2011, also different location to other Porterville trial [a]

Table 26 Residues of flutriafol in pepper following application of an SC formulation in the USA (Carringer 2012 2440) (duplicate samples, applications include non-ionic surfactant)

Location, year,		g		g	GS		Residue (mg/kg)			
variety	No	ai/ha	L/ha	ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
Seven Springs,	4	130	159		53	0	0.16 0.14	< 0.01	0.07	< 0.01
NC, USA, 2011	(7	129	159		71			< 0.01	0.07	< 0.01
California	7	131	168		81				c0.02	
Wonder (Bell)	7)	129	159		89	Mean	0.15	< 0.01	0.07	< 0.01
Greenville, FL,	4	128	196		71	0	0.09 0.10	< 0.01	0.03	< 0.01
USA, 2011	(7	127	187		73			< 0.01	0.03	< 0.01
Aristotle (Bell)	7	128	196		75	Mean	0.10	< 0.01	0.03	< 0.01
	7)	126	196		77					
Delavan, WI,	4	129	225		74	0	0.03 0.03	< 0.01	0.02	< 0.01
USA, 2011	(7	129	206		79			< 0.01	0.02	< 0.01
California	7	128	196		83				c0.01	
Wonder (Bell)	7)	128	196		89	Mean	0.03	< 0.01	0.02	< 0.01
Conklin, MI, USA,	4	127	206		71	0	0.07 0.06	< 0.01	0.03	< 0.01
2011 Aristotle	(7	127	206		72			< 0.01	0.03	< 0.01
(Bell)	7	127	206		73				c0.01	
	7)	128	206		74	Mean	0.06	< 0.01	0.03	< 0.01
Sparta, MI, USA,	4	128	206		71	0	0.08 0.08	< 0.01	< 0.01,	< 0.01
2011 Sopron	(7	128	206		72			< 0.01	< 0.01	< 0.01
(non-bell, large	7	128	206		73	Mean	0.08	< 0.01	< 0.01	< 0.01
banana)	7)	128	206		74–75					
Burford OR	4	127	47		69–73	0	0.05 0.07	< 0.01	0.03	< 0.01
Canada, 2011	(7	123	45		79–85			< 0.01	0.03	< 0.01
Aristotle (Bell) [a]	7	124	47		82–84				c0.01	
	7)	123	46		83–84	Mean	0.06	< 0.01	0.03	< 0.01
Burford OR	4	133	299		65–71	0	0.08 0.15	< 0.01	0.07	< 0.01
Canada, 2011	(7	135	318		73–82			< 0.01	0.06	< 0.01
Crimson hot	7	129	299		81–87				c0.02	
(chilli) [b]	7)	132	309		85–87	Mean	0.12	< 0.01	0.06	< 0.01
Uvalde TX, USA,	4	128	159		Mature	0	0.14 0.14	< 0.01	< 0.01,	< 0.01
2011 Tauras	(7	131	150		82			< 0.01	< 0.01	< 0.01
(Bell)	7	129	150		83	Mean	0.14	< 0.01	< 0.01	< 0.01
	7)	131	140		85	2	0.14 0.10	< 0.01	< 0.01,	< 0.01
								< 0.01	< 0.01	< 0.01
						Mean	0.12	< 0.01	< 0.01	< 0.01
						7	0.08 0.09	< 0.01	0.01	< 0.01
								< 0.01	< 0.01	< 0.01
						Mean	0.08	< 0.01	< 0.01	< 0.01
						14	0.04 0.05	< 0.01	0.02	< 0.01
								< 0.01	0.02	< 0.01
						Mean	0.04	< 0.01	0.02	< 0.01

Location, year, variety	No	g ai/ha	L/ha	g ai/hL	GS (BBCH)	DALA	Residue (mg/kg) Flutriafol	T	TA	TAA
						21	0.04 0.05	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
						Mean	0.04	< 0.01	0.02	< 0.01
Levelland TX, USA, 2011	4 (7	129 129	187 187		Start frt Fruiting	0	0.31 0.31	< 0.01 < 0.01	0.03 0.03	< 0.01 < 0.01
Jalapeno M (chilli)	7 7)	128 130	187 187		Most mat	Mean	0.31	< 0.01	0.03	< 0.01
Porterville, CA, USA, 2011 P33R	4 (7	129 133	49 50		48 48	0	0.18 0.14	< 0.01 < 0.01	0.01 0.01	< 0.01 < 0.01
(Bell) [c]	7 7)	129 129	48 49		49 49	Mean	0.16	< 0.01	0.01	< 0.01
King City, USA, 2011 P33R	4 (7	128 128	299 290		48 48	0	0.11 0.11	< 0.01 < 0.01	0.01 0.01	< 0.01 < 0.01
(Bell) [e]	7 7)	128 129	290 299		48 49	Mean	0.11	< 0.01	0.01	< 0.01
Porterville, CA, USA, 2011	4 (7	131 128	290 290		47 48	0	0.22 0.19	< 0.01 < 0.01	0.02 0.03	< 0.01 < 0.01
Fresno (chilli) [d]	7 7)	130 133	299 318		48 49	Mean	0.20	< 0.01	0.02	< 0.01
King City, USA, 2011 Serrano	4 (7	131 128	299 299		47 49	0	0.26 0.26	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
(chilli) [f]	7 7)	127 128	290 299		49 49	Mean	0.26	< 0.01	0.02	< 0.01

Analytical method flutriafol: RAM 219/04
Analytical method T, TA, TAA: Meth-160, revision 2
Induce 0.3–0.48% v/v, Induce 0.25% v/v, Preference 0.5% v/v, R-11 0.063% v/v, R-11 0.063% v/v, Agral 90 0.25% v/v, Agral 90 0.25% v/v, Induce 0.25% v/v, R-11 0.23% v/v, Pro 90 0.25% v/v, Pro 90 0.5% v/v, Pro 90 0.25% v/v, Pro 90 0.5% v/v.
[a] Last application 02/09/2011
[b] Last application 26/08/2011, same location but different varieties with significantly different residues potential
[c] Last application 11/08/2011
[d] Last application 10/08/2011, different location and different varieties with significantly different residues potential
[e] Last application 09/09/2011
[f] Last application 30/09/2011, location close but different varieties with significantly different residues potential and different application times

Table 27 Residues of flutriafol in lettuce (head and leaf) following application of an SC formulation in the USA (Carringer 2013 2698) (duplicate samples, applications include non-ionic surfactant)

Location, year, variety	No	g ai/ha	L/ha	GS (BBCH)	DALA	Crop part	Flutriafol	T	TA	TAA
HEAD LETTUCE										
Germansville, PA, USA, 2012	4 (6	131 132	48 49	Vegetative Early	7	Heads	0.05 0.05	< 0.01 < 0.01	0.01 0.01	< 0.01 < 0.01
Ithaca (head)	6	130	48	head	Mean		0.05	< 0.01	0.01	< 0.01
	7)	136	50	formation Heads 5–						
				10 cm dia Heads 15– 20 cm dia						
Oviedo, FL, USA, 2011 Great	4 (7	127 127	281 281	41 42	7	Heads	0.15 0.14	0.04, 0.03	< 0.01 < 0.01	< 0.01 < 0.01
Lakes (head)	7 7)	128 127	281 281	45 48	Mean		0.14	0.04	< 0.01	< 0.01
Porterville, CA, USA, 2011	4 (7	128 129	309 318	41 43	0	Heads	0 82 1.17	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Vandenberg	7	128	309	46	Mean		1.00	< 0.01	< 0.01	< 0.01
(head) [a]	7)	128	309	47	2	Heads	0.12 0.20	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean		0.16	< 0.01	< 0.01	< 0.01
					7	Heads	0.28 0.17	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01

Location, year, variety	No	g ai/ha	L/ha	GS (BBCH)	DALA	Crop part	Flutriafol	T	TA	TAA
					Mean		0.22	< 0.01	< 0.01	< 0.01
					10	Heads	0.19 0.30	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean		0.20	< 0.01	< 0.01	< 0.01
					14	Heads	0.07 0.06	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean		0.06	< 0.01	< 0.01	< 0.01
King City, CA, USA, 2011	4 (8	128 128	281 281	44 45	7	Heads	0.46 0.46	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Venus (head)	7	128	281	47	Mean		0.46	< 0.01	< 0.01	< 0.01
	7)	127	281	48						
Porterville, CA, USA, 2011	4 (7	126 126	49 50	44 45	7	Heads	0.08 0.08	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Vandenberg (head) [b]	7 7)	130 128	50 48	47 48	Mean		0.08	< 0.01	< 0.01	< 0.01
Arroyo Grande, CA, USA, 2012	4 (7	130 129	384 371	19 24	7	Heads	0.66 0.67	< 0.01 < 0.01	0.02 0.03	< 0.01 < 0.01
Vandenberg	6	128	374	47	Mean		0.66	< 0.01	< 0.02	< 0.01
(head)	7)	129	374	48						
Visalia, CA, USA, 2012	4 (7	129 129	318 309	45 46	7	Heads	0.47 0.57	< 0.01 < 0.01	0.01 < 0.01	< 0.01 < 0.01
Regency (head)	7 7)	128 128	309 309	47 48	Mean		0.52	< 0.01	< 0.01	< 0.01
Greenfield, CA, USA, 2012 Delta	4 (6	129 128	299 309	46 46	7	Heads	0.03 0.05	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
John (head)	7 7)	129 129	309 309	46 49	Mean		0.04	< 0.01	< 0.01	< 0.01
LEAF LETTUCE										
Germansville, PA, USA, 2011 Red Sails (leaf)	4 (6 7	135 127 129	50 46 47	15 7.6–10 cm diameter	7	Leaves	0.39 0.33	< 0.01 < 0.01	< 0.01 < 0.01 c0.04	< 0.01 < 0.01
	7)	129	47	10–15 cm diameter 15–20 cm diameter	Mean		0.36	< 0.01	< 0.01	< 0.01
Oviedo, FL, USA, 2011 Butter	4 (7	128 126	281 281	43 43	7	Leaves	0.34 0.27	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
Crunch (leaf)	7 7)	124 128	271 281	47 49	Mean		0.30	< 0.01	0.02	< 0.01
Porterville, CA, USA, 2011 Butter	4 (7	128 130	281 281	16 42	0	Leaves	3.71 4.06	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Crunch (leaf) [c]	6	130	281	44	Mean		3.88	< 0.01	< 0.01	< 0.01
	7)	129	281	49	3	Leaves	1.58 1.53	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean		1.56	< 0.01	< 0.01	< 0.01
					7	Leaves	1.47 1.43	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean		1.45	< 0.01	< 0.01	< 0.01
					9	Leaves	1.22 1.41	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean		1.32	< 0.01	< 0.01	< 0.01
					14	Leaves	0.55 0.59	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean		0.57	< 0.01	< 0.01	< 0.01
Butter	4 (7	124 128	271 281	16 42	7	Leaves	0.63 0.68	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Crunch (leaf) [d]	7 7)	128 132	281 290	44 45	Mean		0.66	< 0.01	< 0.01	< 0.01
Visalia, CA, USA, 2012	4 (7	128 128	318 318	44 45	7	Leaves	2.95 2.33	< 0.01 < 0.01	0.01 0.01	< 0.01 < 0.01
Greenstar (leaf)	7 7)	128 129	318 318	47 48	Mean		2.64	< 0.01	0.01	< 0.01

Location, year, variety	No	g ai/ha	L/ha	GS (BBCH)	DALA	Crop part	Flutriafol	T	TA	TAA
San Ardo, CA, USA, 2012	4 (7	129 130	309 309	45 45	7	Leaves	0.24 0.39	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Salvius (leaf)	7 7)	132 129	327 318	45 49	Mean		0.32	< 0.01	< 0.01	< 0.01
COS LETTUCE										
King City, CA, USA, 2011	4 (6	123 129	47 48	45 46	7	Leaves	0.26 0.30	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Romaine (leaf) [e]	7 6)	126 131	47 49	49 49	Mean		0.28	< 0.01	< 0.01	< 0.01
King City, CA, USA, 2012	4 (7	129 128	281 281	19 19	8	Leaves	0.21 0.19	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Paragon (Romaine) (leaf) [f]	7 7)	130 128	290 281	41 47	Mean		0.20	< 0.01	< 0.01	< 0.01

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2

Induce 0.25–0.33% v/v, D-W 0.25% v/v, Pro 90 0.25% v/v, Pro 90 0.25% v/v, Pro 90 0.25% v/v, Kinetic 0.064% v/v, Pro 90 0.25% v/v, Pro 90 0.25% v/v, Induce 0.125% v/v, Triangle D-W 0.25% v/v, Pro 90 0.5% v/v, Pro 90 0.5% v/v, Pro 90 0.25% v/v, FC Spreader Sticker 0.045% v/v, Pro 90 0.25% v/v, Pro 90 0.5 % v/v.

[a] Last application 01/11/2011

[b] Last application 10/11/2011, related location, same varieties as other Porterville trial[A]

[c] Last application 01/11/2011

[d] Last application 03/11/2011, related location, same varieties as other Porterville trial[C]

[e] Last application 16/11/2011

[f] Last application 06/04/2011, same location but application dates significantly different

Table 28 Residues of flutriafol in celery following application of an SC formulation in the USA (Carringer 2013 2698) (duplicate samples, applications include non-ionic surfactant)

Location, year,		g		GS			Residue (mg/kg)			
Variety	No	ai/ha	L/ha	(BBCH)	DALA	Sample	Flutriafol	T	TA	TAA
Oviedo, FL, USA, 2011	4 (7 7 7)	128 129	281 281	37 38	7	Plant	0.87 0.97	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
Tango		126 128	281 281	40 48	Mean		0.92	< 0.01	0.02	< 0.01
Sparta, MI, USA, 2012	4 (7 6 8)	129 128	46 47	45 46	7	Plant	0.74 0.72	0.06 0.06	< 0.01 < 0.01	< 0.01 < 0.01
Greenbay	6 8)	128	46	47	Mean		0.73	0.06	< 0.01	< 0.01
		128	46	48		SPCF	0.56 0.51	0.04 0.05	< 0.01 < 0.01	< 0.01 < 0.01
					Mean		0.54	0.04	< 0.01	< 0.01
King City, CA, USA, 2011	4 (7 7 6)	128 133	299 318	4747	0	Plant	0.99 0.81	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
SSCI		129	309	48	Mean		0.90	< 0.01	< 0.01	< 0.01
		127	299	49	2	Plant	0.54 0.46	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean		0.50	< 0.01	< 0.01	< 0.01
					7	Plant	0.41 0.47	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean		0.44	< 0.01	< 0.01	< 0.01
					10	Plant	0.32 0.42	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean		0.37	< 0.01	< 0.01	< 0.01
					14	Plant	0.43 0.38	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean		0.40	< 0.01	< 0.01	< 0.01
Porterville, CA, USA, 2011	4 (8 7 7)	130 128	47 47	45 46	7	Plant	1.40 1.41	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Command		133 131	133 131	48 49	Mean		1.40	< 0.01	< 0.01	< 0.01

Location, year,		g		GS			Residue (mg/kg)			
Variety	No	ai/ha	L/ha	(BBCH)	DALA	Sample	Flutriafol	T	TA	TAA
Porterville, CA, USA, 2012	4 (7 7 6)	129 128	365 365	44 46	7	Plant	0.96 1.20	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
Mission		129	365	46	Mean		1.08	< 0.01	0.02	< 0.01
		127	365	48		SPCF	1.4 1.3	< 0.01 < 0.01	0.02 0.01	< 0.01 < 001
					Mean		1.35	< 0.01	0.02	< 0.01
Guadalupe, CA, USA, 2011 Conquistador	4 (6 7 6)	128 129 129	271 262 271	45 46 47	8	Plant	0.79 0.76	0.04, 0.04	0.06 0.05 c0.03	< 0.01 < 0.01
		128	271	48	Mean		0.78	0.04	0.06	< 0.01
						SPCF	0.64 0.50	0.04 0.02	0.05 0.05	< 0.01 < 0.01
					Mean		0.57	0.03	0.05	< 0.01
Oviedo, FL, USA, 2012	4 (7 7 7)	127 130	281 290	45 45-49	7	Plant	0.48 0.49	< 0.01 < 0.01	0.03 0.03	< 0.01 < 0.01
Tango		127 129	281 281	47 49	Mean		0.48	< 0.01	0.03	< 0.01
King City, CA, USA, 2012	4 (8 7 7)	130 130	309 309	46 46	7	Plant	0.32 0.36	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Conquistador		129 130	309 309	46 48	Mean		0.34	< 0.01	< 0.01	< 0.01

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2

Triangle D-W 0.25% v/v, R-11 0.07% v/v, Pro 90 0.5% v/v, Pro 90 0.25% v/v, FC Spreader Sticker 0.065% v/v, Triangle D-W 0.25% v/v, Pro 90 0.5% v/v

Table 29 Residues of flutriafol in spinach following application of an SC formulation in the USA (Carringer 2013 2698) (duplicate samples, applications include non-ionic surfactant)

Location, year,		g		GS		Residue (mg/kg)			
variety	No	ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
Alton NY, USA, 2011 Space	4 (7 7 7)	127 127 127	281 281 281	15 17 17	7	2.3 1.9	< 0.01 < 0.01	0.03 0.03 c0.07	< 0.01 < 0.01
		127	281	18	Mean	2.1	< 0.01	0.03	< 0.01
Chula GA USA 2011 Vancouver	4 (7 6 8)	128 128	47 47	12 14	7	1.25 1.4	< 0.01 < 0.01	0.03 0.03	< 0.01 < 0.01
		128 128	47 47	17 37	Mean	1.32	< 0.01	0.03	< 0.01
Uvalde TX USA, 2011 DMC 66-07	4 (7 7 6)	126 128	168 168	45 45	7	0.96 0.93	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
		129 128	206 196	46 46	Mean	0.94	< 0.01	< 0.01	< 0.01
Jerome ID, USA, 2011 Unipack 151	4 (8 7 7)	129 131	215 206	15 19	6	1.6 1.5	< 0.01 < 0.01	0.01 0.01	< 0.01 < 0.01
		128 129	206 206	35 45	Mean	1.55	< 0.01	< 0.01	< 0.01
Porterville, CA, USA, 2011 Shasta	4 (7 7 6)	128 132	365 365	10 11	7	0.59 0.51	< 0.01 < 0.01	0.04 0.04	< 0.01 < 0.01
		132 130	365 365	14 17	Mean	0.55	< 0.01	0.04	< 0.01
Arroyo Grande CA, USA, 2011 Falcon	4 (6 7 6)	128 127	196 196	45 45	7	5.2 4.9	< 0.01 < 0.01	0.03 0.02	< 0.01 < 0.01
		128 128	196 196	46 47	Mean	5.05	< 0.01	0.02	< 0.01
Blackville SC USA 2012	4 (8 6 7)	129 128	140 140	12 13	7	1.7 1.85	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
		129 128	140 140	15 17	Mean	1.78	< 0.01	0.02	< 0.01
Raymondville TX USA 2012	4 (6 7 7)	132 132	196 196	17–18 19	0	8.0 7.8	< 0.01 < 0.01	0.01 0.01	< 0.01 < 0.01

Location, year,		g		GS		Residue (mg/kg)			
variety	No	ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
		132	196	38	Mean	7.9	< 0.01	0.01	< 0.01
		131	196	47–49	3	6.1 6.3	< 0.01 < 0.01	0.02 0.01	< 0.01 < 0.01
					Mean	6.2	< 0.01	0.02	< 0.01
					6	5.4 5.5	< 0.01 < 0.01	0.01 0.02	< 0.01 < 0.01
					Mean	5.45	< 0.01	0.02	< 0.01
					10	3.4 3.1	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
					Mean	3.25	< 0.01	0.02	< 0.01
					13	2.3 3.0	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
					Mean	2.65	< 0.01	0.02	< 0.01

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2

Induce 0.25% v/v, Induce 0.25% v/v, Induce 0.25% v/v, Induce 0.5% v/v, Induce 0.5% v/v, Pro 90 0.5% v/v, First Choice 0.03% v/v, Scanner 0.25–0.26% v/v, R11 0.25% v/v

Table 30 Residues of flutriafol in mustard greens following application of an SC formulation in the USA (Carringer 2013 2697) (duplicate samples, applications include non-ionic surfactant)

Location, year,				GS		Residue (mg/kg)			
variety	No	g ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
Seven Springs, NC, USA, 2011 Southern	4 (7 7	128 127 131	290 290 299	35 39 42	7	2.37 1.88	< 0.01 < 0.01	0.05 0.05 c0.02	< 0.01 < 0.01
Curly Giant	7)	131	299	45	Mean	2.12	< 0.01	0.05	< 0.01
Proctor AR USA, 2011	4 (7	128 128	150 150	2–4 lf 3–4 lf	7	2.53 3.03	< 0.01 < 0.01	0.01 0.02	< 0.01 < 0.01
Florida Broadleaf	7 7)	128 128	150 150	4–6 lf 4–6 lf	Mean	2.78	< 0.01	0.02	< 0.01
Conklin, MI, USA, 2012 Green Wave	4 (7 7	130 129 129	50 49 49	12–16 13–17 16–20	7	2.0 2.24	< 0.01 < 0.01	0.06 0.06 c0.02	< 0.01 < 0.01
	7)	128	48	46–48	Mean	2.12	< 0.01	0.06	< 0.01
Uvalde, TX, USA, 2011	4 (7	126 129	150 140	45 46	7	2.24 2.06	< 0.01 < 0.01	0.03 0.03	< 0.01 < 0.01
India Mustard	7 7)	128 128	159 159	47 48	Mean	2.15	< 0.01	0.03	< 0.01
Porterville, CA, USA, 2011	4 (6	124 132	46 49	13 14	0	3.4 3.41	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Florida	8	122	45	17	Mean	3.40	< 0.01	< 0.01	< 0.01
Broadleaf	7)	124	46	49	3	1.97 1.84	< 0.01 < 0.01	0.01 0.01	< 0.01 < 0.01
					Mean	1.90	< 0.01	0.01	< 0.01
					7	1.59 0.80	< 0.01 < 0.01	0.01 0.01	< 0.01 < 0.01
					Mean	1.20	< 0.01	0.01	< 0.01
					10	0.66 0.84	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
					Mean	0.75	< 0.01	0.02	< 0.01
					14	0.55 0.45	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
					Mean	0.50	< 0.01	0.02	< 0.01
Elko SC, USA 2011 Florida	4 (7	128 128	140 140	13 17	7	3.53 3.32	< 0.01 < 0.01	0.04 0.05	< 0.01 < 0.01
	7 7)	129 127	140 140	18 19	Mean	3.42	< 0.01	0.04	< 0.01
Oveido FL USA 2011 Florida	4 (7 7	128 130	281 290	19 43	7	1.45 1.53	< 0.01 < 0.01	0.18 0.13	< 0.01 < 0.01

Location, year,				GS		Residue (mg/kg)			
variety	No	g ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
		126	281	46				c0.01	
Broadleaf	7)	128	281	48	Mean	1.49	< 0.01	0.16	< 0.01
Visalia CA USA 2011 Florida	4 (7 7	128 129 128	309 318 309	19 33 35	7	1.92 2.12	< 0.01 < 0.01	0.04 0.04 c0.02	< 0.01 < 0.01
Broadleaf	7)	128	318	47	Mean	2.02	< 0.01	0.04	< 0.01

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2

Induce 0.3–0.4% v/v, DyneAmic 0.5% v/v, R11 0.06% v/v, Induce 0.25% v/v, Pro 90 0.5–1% v/v, Scanner 0.24–0.25% v/v, Triangle D-W 0.25% v/v, Pro 90 0.25% v/

Table 31 Residues of flutriafol in sugar beet (roots) in Europe following application of an SC formulation (Pollmann 2005a 1235; 2005b 1236; 2006a 1368; 2006b 1335; 2007b 1381)

Location, year, variety SUGAR BEET	No	g ai/ha	L/ha	GS (BBCH)	DALA	Flutriafol (mg/kg)
Northern Europe (1235)						
Scherwiller, Alsace, Northern	2	120	290	39	15	0.01
France 2004 Guepard	(21)	135	327	39	22	< 0.01
					29	< 0.01
					41	< 0.01
Dollern, Niedersachsen,	2	131	263	45	14	< 0.01
Germany 2004 Famosa	(22) [a]	126	253	43–44	22	< 0.01
					27	0.01
					41	< 0.01
Haderslev, Jutland, Denmark	2	125	303	39	15	< 0.01
2004 Verity	(21) [b]	111	269	46	21	< 0.01
					28	< 0.01
					42	< 0.01
Holme, Peterborough, UK 2004	2	121	293	45	15	0.02
Cinderella	(21) [c]	120	292	47	20	0.01
					29	< 0.01
					41	< 0.01
Dudenbuttel, Lower Saxony,	2	126	300	43	22	< 0.01
Germany 2005 Ricardo	(21) [d]	131	311	44–46	28	< 0.01
Haderslav, Sonderjylland,	2	133	316	43–44	20	< 0.01
Denmark 2005 Verity	(21) [e]	138	329	46	28	< 0.01
Scherwiller, Alsace, Northern	2	123	292	39	21	0.02
France 2005 Canyon	(20) [f]	138	328	39	27	0.03
Bishop's Tachbrook,	2	127	302	47	21	0.03
Warwickshire, UK 2005	(21) [g]	130	310	48	29	0.02
Cinderella						
Southern Europe (1236, 1335)						
Castelnuovo della Daunia,	3	132	320	35–37	7	< 0.01
Puglia, Italy, 2004 Monatonno	(21	131	317	36–38	15	< 0.01
	22) [h]	127	308	45–47	22	< 0.01
					29	< 0.01
Poggio Renatico, Emilia	3	127	410	37	6	< 0.01
Romagna, Italy, 2004 Gea	(21	125	402	39–41	13	< 0.01
	21)	124	400	44	20	< 0.01
					29	< 0.01
Pozoarmargo, Cuenca, Spain,	3	127	408	39	7	< 0.01
2004 Vincent	(21	127	410	39	15	0.02
	20)	124	401	39	22	0.01
					30	< 0.01
Tobarra, Albacete, Spain, 2004	3	128	412	39	7	0.01
Brigitta	(21	132	427	39	14	< 0.01
	21)	126	405	39	21	< 0.01
					29	< 0.01

Location, year, variety SUGAR BEET	No	g ai/ha	L/ha	GS (BBCH)	DALA	Flutriafol (mg/kg)
Tobarra, Albacete, Spain, 2005	3	122	390	39	20	0.02
Heracles	(22	125	401	39	27	0.02
	20)	117	373	42		
Poggio Renatico, Emilia	3	125	397	45	22	0.01, < 0.01 (< 0.01)
Romagna, Italy, 2005 Opera	(21	124	393	47	28	0.02, 0.01 (0.02)
	21) [i]	127	403	47		
Ponte Pietra, Cesena, Emilia	3	128	407	42	22	0.02
Romagna, Italy, 2005 Gea	(20	123	390	44	28	< 0.01
	20) [j]	124	393	46		
Arevalo, Avila, Spain, 2006	3	131	312	39	22	0.04
Brigitta	(20	138	328	39	29	0.03
	21)	126	299	39		

[a] 6 mm rainfall within 24 h of 1st application

[b] 2 mm and 3 mm rain within 24 h 1st and 2nd spray

[c] 10.2 mm after 2nd spray

[d] 7 mm after 2nd spray

[e] 3 and 9 mm rain within 24 h 1st and 2nd spray

[f] 3 and 3 mm rain within 24 h 1st and 2nd spray

[g] 5 mm rainfall within 24 h of 1st application

[h] 0.4 mm rain within 24 h 1st spray

[i] 3.6 mm rain within 24 h 2nd spray

[j] 0.6 mm rain within 24 h 3rd spray

Table 32 Residues of flutriafol in sugar beet (roots) in the USA following application of an SC formulation (Jones 2009 1812) (duplicate samples)

Location, year, variety	No	g ai/ha	L/ha	GS (BBCH)	DALA	Residue (mg/kg) Flutriafol	T	TA	TAA
Porterville, CA,	3	129	306	81	14	0.05 0.05	< 0.01 < 0.01	0.02 0.01	
USA, 2009 Pheonix	(14 14)	127 124	307 292	81–83 87	Mean	0.05	< 0.01	0.02	< 0.01
Fresno, CA,	3	125	325	48	14	0.02 0.02	< 0.01 < 0.01	0.04 0.03 c0.02	
USA, 2009 HH142	(14 14)	128 128	329 329	48 49	Mean	0.02	< 0.01	0.04	< 0.01
American Falls,	3	123	279	49	14	0.01 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
ID, USA, 2009 Hillshog 9026	(14 15)	129 123	295 318	49 49	Mean	0.02	< 0.01	< 0.01	< 0.01
Jerome, ID,	3	128	345	49	14	0.01 0.02	< 0.01 < 0.01	0.01 0.01	
USA, 2009 BTSCT01RR07	(14 14)	128 124	332 339	49 49	Mean	0.02	< 0.01	0.01	< 0.01
Geneva, MN,	3	129	288	Vegetative	14	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	
USA, 2009 Beta 130R	(15 13)	128 129	280 289	Vegetative Vegetative	Mean	< 0.01	< 0.01	< 0.01	< 0.01
Campbell, MN, USA, 2009 4012RR	3 (13 14)	128 128 129	328 328 330	33 35 49	0	< 0.01 0.01	< 0.01 < 0.01	< 0.01 < 0.01	
					Mean	< 0.01	< 0.01	< 0.01	< 0.01
					7	0.01 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
					Mean	0.02	< 0.01	< 0.01	< 0.01
					14	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	
					Mean	< 0.01	< 0.01	< 0.01	< 0.01
					21	0.01	< 0.01	< 0.01	

Location, year, variety	No	g ai/ha	L/ha	GS (BBCH)	DALA	Residue (mg/kg) Flutriafol	T	TA	TAA
						< 0.01	< 0.01	< 0.01	
					Mean	< 0.01	< 0.01	< 0.01	< 0.01
					28	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	
					Mean	< 0.01	< 0.01	< 0.01	< 0.01
Paynesville, MN, USA, 2009 Crystal RR202	3 (13 14)	130 131 130	283 285 281	45 45 47	14	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	
					Mean	< 0.01	< 0.01	< 0.01	< 0.01
Pavillion, WY, USA, 2009 Beta 36RR11	3 (14 14)	128 130 130	304 302 318	49 49 49	14	0.04 0.06	< 0.01 < 0.01	< 0.01 < 0.01	
					Mean	0.05	< 0.01	< 0.01	< 0.01
Northwood, ND, USA, 2009 Beta 1305R	3 (15 13)	127 129 127	325 329 324	39 39 39	14	< 0.01 0.01	< 0.01 < 0.01	< 0.01 < 0.01	
					Mean	< 0.01	< 0.01	< 0.01	< 0.01
Velva, ND, USA, 2009 R308	3 (14 14)	130 131 127	284 286 284	37 39 39	14	0.02 0.02	< 0.01 < 0.01	< 0.01 < 0.01	
					Mean	0.02	< 0.01	< 0.01	< 0.01
York, NE, USA, 2009 Beta 734IR	3 (14 14)	129 130 129	329 329 325	42d before harvest 39 49	14	0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	
					Mean	< 0.01	< 0.01	< 0.01	< 0.01
Levelland, TX, USA, 2009 Phoenix	3 (14 15)	130 124 127	324 322 325	Roots starting to enlarge roots enlarging maturing roots	14	0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	
					Mean	< 0.01	< 0.01	< 0.01	< 0.01

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2

Table 33 Residues of flutriafol in maize (grain) following application of an SC formulation in the USA (Carringer 2010 1810) (duplicate samples) A non-ionic surfactant was added to the tank mix at all sites except for decline trials where plots were sprayed with and without surfactant.

Location, year, variety	No	g ai/ha	L/ha	g ai/hL	GS (BBCH)	DALA	Residue (mg/kg) Flutriafol	T	TA	TAA
Germansville, PA, USA,	2 (6)	129 132	140 140	77 79	87 89	6	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
2009 Hybrid 2D324 Mycogen Seed						Mean	< 0.01	< 001	< 0.01	< 0.01
Seven Springs, NC,	2 (7)	129 131	131 131	82 84	86 89	6	< 0.01 < 0.01	< 0.01 < 0.01	0.05 0.06	< 0.01 < 0.01
USA, 2009 N77-P5						Mean	< 0.01	< 0.01	0.06	< 0.01
Wyoming, IL, USA, 2009	2 (7)	129	112	96	89	0	< 0.01 < 0.01	< 0.01 < 0.01	0.06 0.07	< 0.01 < 0.01
						Mean	< 0.01	< 0.01	0.06	< 0.01
DKC 61–69		128	112	95	89	1	< 0.01 < 0.01	< 0.01 < 0.01	0.08 0.06	< 0.01 < 0.01
						Mean	< 0.01	< 0.01	0.07	< 0.01
						7	< 0.01 < 0.01	< 0.01 < 0.01	0.07 0.07	< 0.01 < 0.01
						Mean	< 0.01	< 0.01	0.07	< 0.01
						15	< 0.01 < 0.01	< 0.01 < 0.01	0.08 0.07	< 0.01 < 0.01
						Mean	< 0.01	< 0.01	0.08	< 0.01
						21	< 0.01	< 0.01	0.06	< 0.01

Location, year,		g		g	GS		Residue (mg/kg)			
variety	No	ai/ha	L/ha	ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
							< 0.01	< 0.01	0.06	< 0.01
						Mean	< 0.01	< 0.01	0.06	< 0.01
No surfactant	2 (7)	128	112	96	89	0	< 0.01	< 0.01	0.07	< 0.01
							< 0.01	< 0.01	0.07	< 0.01
						Mean	< 0.01	< 0.01	0.07	< 0.01
		128	112	95	89	1	< 0.01	< 0.01	0.06	< 0.01
							< 0.01	< 0.01	0.06	< 0.01
						Mean	< 0.01	< 0.01	0.06	< 0.01
						7	< 0.01	< 0.01	0.06	< 0.01
							< 0.01	< 0.01	0.07	< 0.01
						Mean	< 0.01	< 0.01	0.06	< 0.01
						15	< 0.01	< 0.01	0.08	< 0.01
							< 0.01	< 0.01	0.10	< 0.01
						Mean	< 0.01	< 0.01	0.09	< 0.01
						21	< 0.01	< 0.01	0.08	< 0.01
							< 0.01	< 0.01	0.09	< 0.01
						Mean	< 0.01	< 0.01	0.08	< 0.01
Carlyle, IL,	2 (8)	127	122	87	87	7	< 0.01	< 0.01	0.08	< 0.01
USA, 2009		128	140	76	89		< 0.01	< 0.01	0.08	< 0.01
8G23						Mean	< 0.01	< 0.01	0.08	< 0.01
Grantfork, IL,	2 (7)	130	122	89	89	7	< 0.01	< 0.01	0.03	< 0.01
USA, 2009		130	112	97	89		< 0.01	< 0.01	< 0.01	< 0.01
AgriGolg						Mean	< 0.01	< 0.01	< 0.02	< 0.01
AG457										
Conklin, MI,	2 (8)	128	122	88	87	6	< 0.01	< 0.01	< 0.01	< 0.01
USA, 2009		128	122	88	88		< 0.01	< 0.01	< 0.01	< 0.01
A1005113						Mean	< 0.01	< 0.01	< 0.01	< 0.01
Richland, IA,	2 (7)	129	140	77	89	0	< 0.01	< 0.01	0.05	< 0.01
USA, 2009							< 0.01	< 0.01	0.04	< 0.01
						Mean	< 0.01	< 0.01	0.04	< 0.01
Pioneer 34R67		129	140	77	89	1	< 0.01	< 0.01	0.05	< 0.01
							< 0.01	< 0.01	0.04	< 0.01
						Mean	< 0.01	< 0.01	0.04	< 0.01
						7	< 0.01	< 0.01	0.06	< 0.01
							< 0.01	< 0.01	0.06	< 0.01
						Mean	< 0.01	< 0.01	0.06	< 0.01
						13	< 0.01	< 0.01	0.05	< 0.01
							< 0.01	< 0.01	0.05	< 0.01
						Mean	< 0.01	< 0.01	0.05	< 0.01
						20	< 0.01	< 0.01	0.04	< 0.01
							< 0.01	< 0.01	0.04	< 0.01
						Mean	< 0.01	< 0.01	0.04	< 0.01
No surfactant	2 (7)	128	140	77	89	0	< 0.01	< 0.01	0.06	< 0.01
							< 0.01	< 0.01	0.05	< 0.01
						Mean	< 0.01	< 0.01	0.06	< 0.01
		129	140	77	89	1	< 0.01	< 0.01	0.05	< 0.01
							< 0.01	< 0.01	0.05	< 0.01
						Mean	< 0.01	< 0.01	0.05	< 0.01
						7	< 0.01	< 0.01	0.06	< 0.01
							< 0.01	< 0.01	0.06	< 0.01
						Mean	< 0.01	< 0.01	0.06	< 0.01
						13	< 0.01	< 0.01	0.05	< 0.01
							< 0.01	< 0.01	0.06	< 0.01
						Mean	< 0.01	< 0.01	0.06	< 0.01
						20	< 0.01	< 0.01	0.07	< 0.01
							< 0.01	< 0.01	0.06	< 0.01
						Mean	< 0.01	< 0.01	0.06	< 0.01
Douds, IA, USA,	2 (7)	126	140	75	87	7	< 0.01	< 0.01	< 0.01	< 0.01
2009		127	131	81	87–89		< 0.01	< 0.01	< 0.01	< 0.01
Garst 84N57						Mean	< 0.01	< 0.01	< 0.01	< 0.01
Batavia, IA,	2 (7)	129	140	77	87	7	< 0.01	< 0.01	0.08	< 0.01
USA, 2009		126	131	80	87–89		< 0.01	< 0.01	0.08	< 0.01

Location, year,		g		g	GS		Residue (mg/kg)			
variety	No	ai/ha	L/ha	ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
Garst 82K79						Mean	< 0.01	< 0.01	0.08	< 0.01
LaPlata, MO, USA, 2009	2 (7)	130 128	140 140	77 76	87 89	6	< 0.01 < 0.01	< 0.01 < 0.01	0.03 0.04	< 0.01 < 0.01
LG 2614 VT						Mean	< 0.01	< 0.01	0.04	< 0.01
Jefferson, IA, USA, 2009	2 (7)	129 127	112 103	96 103	87 87	7	< 0.01 < 0.01	< 0.01 < 0.01	0.08 0.04	< 0.01 < 0.01
33H27						Mean	< 0.01	< 0.01	0.06	< 0.01
Bagley, IA, USA, 2009	2 (7)	126 127	103 103	102 103	87 87	7	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
33M16						Mean	< 0.01	< 0.01	< 0.01	< 0.01
Bristol, IN, USA, 2009	2 (7)	128 128	122 122	88 88	87 88	8	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
34F97						Mean	< 0.01	< 0.01	< 0.01	< 0.01
York, NE, USA, 2009	2 (8)	129 124	140 140	77 74	87 87	6	< 0.01 < 0.01	< 0.01 < 0.01	0.08 0.11	< 0.01 < 0.01
7B15RRY GCBP						Mean	< 0.01	< 0.01	0.10	< 0.01
Osceola, NE, USA, 2009	2 (7)	129 129	140 140	77 77	87 87	7	< 0.01 < 0.01	< 0.01 < 0.01	0.05 0.05	< 0.01 < 0.01
7B15RRY GCBP						Mean	< 0.01	< 0.01	0.05	< 0.01
Geneva, NE, USA, 2009	2 (7)	128 128	140 140	76 76	87 87	6	< 0.01 < 0.01	< 0.01 < 0.01	0.04 0.04	< 0.01 < 0.01
7B15RRY GCBP						Mean	< 0.01	< 0.01	0.04	< 0.01
Geneva, MN, USA, 2009	2 (6)	129 129	140 140	77 77	87 87	8	< 0.01 < 0.01	< 0.01 < 0.01	0.04 0.04	< 0.01 < 0.01
Pioneer 38P43						Mean	< 0.01	< 0.01	0.04	< 0.01
Paynesville, MN, USA,	2 (7)	129 130	131 131	82 83	87 89	7	< 0.01 < 0.01	< 0.01 < 0.01	0.08 0.06	< 0.01 < 0.01
2009 Dekalb DKC35						Mean	< 0.01	< 0.01	0.07	< 0.01
Fitchburg, WI, USA,	2 (6)	128 128	131 131	81 81	87 89	9	< 0.01 < 0.01	< 0.01 < 0.01	0.03 0.05	< 0.01 < 0.01
2009 Pioneer 37Y14						Mean	< 0.01	< 0.01	0.04	< 0.01
Hinton, OK, USA, 2009	2 (7)	129 129	131 131	82 82	87 87	7	< 0.01 < 0.01	< 0.01 < 0.01	0.11 0.09	< 0.01 < 0.01
DKC 52-59						Mean	< 0.01	< 0.01	0.10	< 0.01

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1 X-77 @ 0.25% v/v; 2 Induce @ 0.34% v/v; 3 Aquagene 90 @ 0.05% v/v; 4 Surfac 820 @ 0.25% v/v; 5 NIS @ 0.25% v/v; 6 R-11 @ 0.064% v/v; 7 Silwet L-77 @ 0.25% v/v; 8 X-77 @ 0.25% v/v; 9 X-77 @ 0.25% v/v; 10 X-77 @ 0.25% v/v; 11 Hel-Fire 90 @ 0.25% v/v; 12 Hel-Fire 90 @ 0.25% v/v; 13 R11 @ 0.064% v/v; 14 Cornbelt Premier 90 @ 0.25% v/v; 15 Cornbelt Premier 90 @ 0.063% v/v; 16 Cornbelt Premier 90 @ 0.25% v/v; 17 Dyne Amic NIS @ 0.375% v/v; 18 Preference @ 0.25% v/v; 19 Preference @ 0.25% v/v; 20 Baron @ 0.076% v/v

Moisture content %: 27.7, 20.8, 34.2 (0 d), 33.7 (1 d), 30.9 (7 d), 25.7 (15 d), 22.8 (21 d), 29.5, 19.4, 33.3, 28.6 (0 d), 29.6 (1 d), 26.7 (7 d), 23.0 (13 d), 21.4 (20 d), 32.6, 37.0, 24.4, 22.8, 26.0, 35.8, 28.1, 31.8, 28.5, 33.8, 14.4, 27.0, 15.2

Table 34 Residues of flutriafol in paddy rice following application of an SC formulation in southern Europe (Gimeno 2006 1629-2, López Benet 2006 1629-1, Gimeno Martos 2007 1630)

Location, year, variety	No	g ai/ha	L/ha	g ai/hL	GS (BBCH)	DALA	Sample	Flutriafol (mg/kg)
Amposta, Tarragona,	2	189	404	47	BBCH 83	0	Paddy rice	3.4
Spain, 2005 Fonsa	(14)	188	400	47	BBCH 89		Husked rice	0.25
	2	183	392	47	BBCH 77	7	Paddy rice	2.47
	(14)	182	388	47	BBCH 87			
	2	186	396	47	BBCH 65	14	Paddy rice	1.25
	(14)	188	400	47	BBCH 83		Husked rice	0.35
	2	182	388	47	BBCH 58	21	Paddy rice	1.68
	(14)	186	396	47	BBCH 77		Husked rice	0.47
	2	195	416	47	BBCH 51	28	Paddy rice	0.74
	(14)	182	388	47	BBCH 65			
Sueca, Valencia,	2	191	408	47	BBCH 83	0	Paddy rice	2.89
Spain, 2005, Masso	(14)	193	412	47	BBCH 87–89		Husked rice	0.23
	2	191	400	48	BBCH 79	7	Paddy rice	1.4
	(14)	193	400	48	BBCH 85			
	2	193	412	47	BBCH 77	14	Paddy rice	1.79
	(14)	187	400	47	BBCH 83		Husked rice	0.42
	2	186	396	47	BBCH 57	21	Paddy rice	1.28
	(14)	187	400	47	BBCH 79		Husked rice	0.36
	2	191	428	45	BBCH 49	28	Paddy rice	1.06
	(14)	193	388	50	BBCH 77			
Perello, Valencia,	2	187	400	47	BBCH 85	0	Paddy rice	3.23
Spain, 2005 Fonsa	(14)	189	404	47	BBCH 89		Husked rice	0.36
	2	187	400	47	BBCH 85	7	Paddy rice	1.93
	(14)	189	400	47	BBCH 87			
	2	204	436	47	BBCH 83	14	Paddy rice	1.85
	(14)	187	400	47	BBCH 85		Husked rice	0.46
	2	182	388	47	BBCH 71	21	Paddy rice	1.92
	(14)	186	396	47	BBCH 85		Husked rice	0.42
	2	187	372	50	BBCH 57	28	Paddy rice	1.51
	(14)	189	396	48	BBCH 83			
Valencia, Valencia,	2	189	404	47	BBCH 83	0	Paddy rice	4.07
Spain, 2005	(14)	188	400	47	BBCH 89		Husked rice	0.15
Montsianell	2	187	380	49	BBCH 77	7	Paddy rice	3.07
	(14)	189	406	47	BBCH 85			
	2	182	388	47	BBCH 77	14	Paddy rice	2.02
	(14)	187	400	47	BBCH 83		Husked rice	0.28
	2	186	396	47	BBCH 59	21	Paddy rice	1.75
	(14)	182	388	47	BBCH 77		Husked rice	0.29
	2	187	386	47	BBCH 55	28	Paddy rice	1.32
	(14)	189	400	47	BBCH 77			
Mareny de	2	187	400	47	BBCH 80	0	Paddy rice	3.19
Barraquetes,	(14)	186	396	47	BBCH 89		Husked rice	0.16
Valencia, Spain, 2006						0	Polished rice	0.08
Montsianell	2	187	400	47	BBCH 69	14	Paddy rice	1.57
	(14)	183	390	47	BBCH 89	14	Husked rice	0.37
						14	Polished rice	0.26
Sueca, Valencia,	2	187.5	400	47	BBCH 81	0	Paddy rice	1.73
Spain, 2006 J. Sendra	(14)	183	390	47	BBCH 89	0	Husked rice	0.07
						0	Polished rice	0.03
	2	187	398	47	BBCH 75	14	Paddy rice	0.9
	(14)	186	396	47	BBCH 81	14	Husked rice	0.19
						14	Polished rice	0.17
Amposta, Tarragona,	2	189	404	47	BBCH 80	0	Paddy rice	2.62
Spain, 2006 Fonsa	(14)	187.5	400	47	BBCH 89	0	Husked rice	0.33
						0	Polished rice	0.21
	2	185	394	47	BBCH 69	14	Paddy rice	1.74
	(14)	190	406	47	BBCH 80	14	Husked rice	0.37

Location, year, variety	No	g ai/ha	L/ha	g ai/hL	GS (BBCH)	DALA	Sample	Flutriafol (mg/kg)
						14	Polished rice	0.32
Sueca, Valencia,	2	190	406	47	BBCH 85	0	Paddy rice	2.76
Spain, 2006 Fonsa	(14)	183	390	47	BBCH 89	0	Husked rice	0.28
						0	Polished rice	0.14
	2	187.5	400	47	BBCH 76	14	Paddy rice	1.23
	(14)	187.5	400	47	BBCH 85	14	Husked rice	0.38
						14	Polished rice	0.33

Table 35 Residues of flutriafol in sorghum grain following application of an SC formulation in the USA (Carringer 2013 2699) (duplicate samples, applications include non-ionic surfactant)

				GS		Residue (mg/kg)			
Location, year, variety	No	g ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
Seven Springs, NC, USA, 2012 DKS54-00	2 (7)	131 127	168 131	60 69	30	0.03 0.03	< 0.01 < 0.01	0.38 0.37 c0.04	0.03 0.03
					Mean	0.03	< 0.01	0.38	0.03
Proctor, AR, USA, 2012 GX12564	2 (7)	128 129	140 140	Mature grain	30	0.40 0.35	< 0.01 < 0.01	0.02 0.03 c0.01	0.02 0.02 c0.01
				Mature grain	Mean	0.38	< 0.01	0.02	0.02
Richland, IA, USA, 2012 Pioneer 84G62	2 (7)	127 129	178 178	85 87	30	0.24 0.27	< 0.01 < 0.01	0.06 0.05 c0.05	< 0.01 < 0.01
					Mean	0.26	< 0.01	0.06	< 0.01
Kirksville, MO, USA, 2012 Pioneer 84G62	2 (7)	128 129	159 159	81–85 85	30	0.20 0.19	< 0.01 < 0.01	0.08 0.09 c0.07	< 0.01 < 0.01
					Mean	0.20	< 0.01	0.08	< 0.01
Stafford, KS, USA, 2012 84G62	2 (7)	128 127	168 168	85 85	29	0.26 0.31	< 0.01 < 0.01	0.04 0.03 c0.03	0.01 0.01 c0.01
					Mean	0.28	< 0.01	0.04	0.01
York, NE, USA, 2012 85G01	2 (7)	127 128	178 178	85 85	31	0.33 0.35	< 0.01 < 0.01	0.07 0.06 c0.07	0.04 0.03 c0.03
					Mean	0.34	< 0.01	0.06	0.04
Uvalde, TX USA, 2012 Pioneer 83G19	2 (7)	126 128	150 159	73 87	30	0.77 0.72	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.74	< 0.01	< 0.01	< 0.01
Hinton, OK, USA, 2012 DKS29-28	2 (7)	127 126	159 168	85 85	30	0.15 0.16	< 0.01 < 0.01	0.07 0.07 c0.05	0.04 0.03 c0.02
					Mean	0.16	< 0.01	0.07	0.04
Grand Island, NE, USA, 2012 85G01	2 (7)	128 128	187 178	85 85	30	0.41 0.38	< 0.01 < 0.01	0.08 0.08 c0.13	0.03 0.03 c0.06
					Mean	0.40	< 0.01	0.08	0.03
Larned, KS, USA, 2012 84G62	2 (7)	129 128	168 168	85 87	23	0.24 0.24	< 0.01 < 0.01	0.06 0.07	0.01 0.01
					Mean	0.24	< 0.01	0.06	0.01
					29	0.25 0.22	< 0.01 < 0.01	0.05 0.05 c0.03	0.01 0.01
					Mean	0.24	< 0.01	0.05	0.01
					36	0.24 0.22	< 0.01 < 0.01	0.05 0.06	< 0.01 < 0.01
					Mean	0.23	< 0.01	0.06	< 0.01
					43	0.23 0.17	< 0.01 < 0.01	0.05 0.06	< 0.01 0.01

				GS		Residue (mg/kg)			
Location, year, variety	No	g ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
					Mean	0.20	< 0.01	0.06	< 0.01
					50	0.22 0.22	< 0.01 < 0.01	0.06 0.06	< 0.01 < 0.01
					Mean	0.22	< 0.01	0.06	< 0.01
Wall, TX, USA, 2012 DKS44-20	2 (7)	127 129	140 140	85 87	29	0.17 0.16	< 0.01 < 0.01	< 0.01 0.01	< 0.01 < 0.01
					Mean	0.16	< 0.01	< 0.01	< 0.01
Levelland, TX, USA, 2012 165310	2 (7)	128 127	178 178	85 85–87	30	0.81 0.66	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.74	< 0.01	< 0.01	< 0.01

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Induce 0.28–0.3% v/v, Dyne-Amic 0.5% v/v, Preference 0.5% v/v, Preference 0.5% v/v, Spreader 90 0.25% v/v, Cornbelt Premier 90 0.03% v/v, Induce 0.2% v/v, Baron 0.25% vv, Cornbelt Premier 0.03% v/v, Spreader 90 0.25% v/v, Induce 0.5% v/v, R-11 0.22% v/v

Table 36 Residues of flutriafol in tree nuts (nutmeat) following application of an SC formulation in the USA (Rice 2011 2161) (duplicate samples)

Location,		g		g	GS		Residue (mg/kg)			
year, variety	No	ai/ha	L/ha	ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
Pecan										
Chula, GA, USA, 2010 Pecan Sumner	6 (7 7 7 7 7)	128 128 128 128 128 128	1370 1505 1524 1440 1425 1340	9.3 8.5 8.4 8.9 9.0 9.6	Nut fill Nut fill Nut fill Nut fill Shuck split Shuck split (falling)	14	< 0.01 < 0.01	< 0.01 < 0.01	0.52 0.42 c0.24	0.04 0.04 c0.01
						Mean	< 0.01	< 0.01	0.47	0.04
Pecan Sumner Steward	6 (7 7 7 7 7	129 130 128 130 129 129	571 632 632 612 603 565	23 21 20 21 21 23	Nut fill Nut fill Nut fill Nut fill Shuck split Shuck split (falling)	14	< 0.01 < 0.01	< 0.01 < 0.01	0.41 0.40 c0.31	0.05 0.05 c0.01
						Mean	< 0.01	< 0.01	0.40	0.05
Bertrand, MO, USA, 2010 Pecan Pawnee	6 (8 7 6 7 7)	125 127 128 127 127 127	1590 1590 1590 1590 1590 1590	7.9 8 8 8 8 8	89 89 89 89 89 89	14	< 0.01 < 0.01	< 0.01 < 0.01	0.02 0.02 c0.02	< 0.01 < 0.01
						Mean	< 0.01	< 0.01	0.02	< 0.01
D'Haris, TX, USA, 2010 Pecan Cheyenne	6 (6 8 7 7 7)	129 125 128 128 127 127	1549 1545 1521 1545 1524 1559	8.3 8.1 8.4 8.3 8.3 8.1	85 85 85 85 87 87	14	0.01 0.01	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
						Mean	0.01	< 0.01	0.02	< 0.01
Anton, TX, USA, 2010 Pecan Western Schley	6 (7 7 6 8 8)	132 127 125 125 131 128	560 560 560 560 560 560	24 23 22 22 23 23	green shuck green shuck green shuck shuck split shuck split shuck split	11	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01

Location,		g		g	GS		Residue (mg/kg)			
year, variety	No	ai/ha	L/ha	ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
						Mean	< 0.01	< 0.01	< 0.01	< 0.01
Almond										
Dinuba, CA, USA, 2010 Almond Sonora	6 (8 8 8 8 8 8)	128 129 128 129 128 128	731 750 781 788 791 883	17 17 16 16 16 14	75 75 78 78 81 81	14	0.08 0.05	< 0.01 < 0.01	< 0.2 < 0.2 c0.2	< 0.01 < 0.01
						Mean	0.06	< 0.01	< 0.2	< 0.01
Strathmore, CA, USA, 2010 Almond Fritz	6 (6 7 7 7 7 7)	128 128 129 128 128 128	2759 2751 2768 2761 2753 2773	4.6 4.6 4.7 4.6 4.6 4.6	79 79 79 80 80 88	14	0.01 0.01	0.02 0.02 c0.11	0.91 0.92 c2.68	0.01 < 0.01 c0.03
						Mean	0.01	0.02	0.92	< 0.01
Wasco, CA, USA, 2010	6 (8 6 7 7 7 7)	128 128 128 128 128 128	809 788 791 786 785 827	16 16 16 16 16 15	79 79 79 79 79 85	14	0.07 0.06	< 0.01 < 0.01	0.56 0.55 c0.29	< 0.01 < 0.01
						Mean	0.06	< 0.01	0.56	< 0.01
Buttonwillow, CA, USA, 2010 Almond Monterey's	6 (7 7 7 7 7 7)	128 127 133 128 128 128	3301 3321 3313 3304 3327 3223	3.9 3.8 4 3.9 3.8 4	78 79 79 83 85 87	14	< 0.01 < 0.01	< 0.01 < 0.01	0.61 0.63 c0.49	< 0.01 < 0.01
						Mean	< 0.01	< 0.01	0.62	< 0.01
Terra Bella, CA, USA, 2010 Almond Non Pareil	6 (9 7 9 8 8 8)	127 128 127 129 129 128	661 605 627 661 661 661	19 21 20 19 19 19	75 72 78 79 79 81	1	0.40 0.42	< 0.01 < 0.01	0.67 0.61	< 0.01, < 0.01
						Mean	0.41	< 0.01	0.64	< 0.01
						7	0.27 0.26	< 0.01 < 0.01	0.57 0.59	< 0.01 < 0.01
						Mean	0.27	< 0.01	058	< 0.01
						14	0.32 0.27	< 0.01 < 0.01	0.63 0.78 c2.08	< 0.01 < 0.01
						Mean	0.30	< 0.01	0.71	< 0.01
						21	0.38 0.45	0.01 < 0.01	1.02 0.78	< 0.01 < 0.01
						Mean	0.42	< 0.01	0.90	< 0.01
						28	0.26 0.23	< 0.01 < 0.01	0.61 0.75	< 0.01 < 0.01
						Mean	0.24	< 0.01	0.68	< 0.01

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Table 37 Residues of flutriafol in cotton (undelinted seed) following application of an SC formulation in the USA (Carringer 2013 2700) (duplicate samples, applications include non-ionic surfactant) one soil pre-emergence application and two post-emergence foliar applications

Location,				GS		Residue (mg/kg)			
year, variety	No	g ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
Elko, SC, USA, 2012	3 (131 6)	294 PP 129 PO	42 187	0 80	30	0.05 0.06	< 0.01 < 0.01	0.94 0.42	0.02 0.01

Location,				GS		Residue (mg/kg)			
year, variety	No	g ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
DP 0912		128PO	178	81				c0.04	
B2RF					Mean	0.06	< 0.01	0.68	0.02
Proctor, AR, USA, 2012 DP	3 (1207)	290 PP 128 PO 128 PO	44 92 187	0 82 84	30	0.13 0.15	< 0.01 < 0.01	0.17 0.14 c0.03	< 0.01 < 0.01
0912 B2RF					Mean	0.14	< 0.01	0.16	< 0.01
Fisk, MO, USA, 2012 PHY 375	3 (1207)	294 PP 128 PO 128 PO	47 187 187	0 80 81	29	< 0.01 < 0.01	< 0.01 < 0.01	0.44 0.41 c0.19	0.01 0.01
					Mean	< 0.01	< 0.01	0.42	0.01
Cheneyville, LA, USA, 2012 DP	3 (1197)	304 PP 135 PO 129 PO	47 168 178	0 82–83 84–85	30	0.08 0.10	< 0.01 < 0.01	0.14 0.16 c0.04	< 0.01 < 0.01
0912 B2RF					Mean	0.09	< 0.01	0.15	< 0.01
Uvalde, TX, USA,	3 (1127)	288 PP 127 PO	30 178	0 82	30	0.02 0.03	< 0.01 < 0.01	0.11 0.11	< 0.01 < 0.01
2012 DP 0912 B2RF		126 PO	159	86	Mean	0.02	< 0.01	0.11	< 0.01
Wall, TX, USA, 2012	3 (1057)	295 PP 124 PO	41 168	0 82	30	0.32 0.19	< 0.01 < 0.01	0.07 0.09	< 0.01 < 0.01
DP 0912 B2RF		127 PO	168	83	Mean	0.26	< 0.01	0.08	< 0.01
Edmonson, TX, USA, 2012 DP	3 (1317)	294 PP 128 PO 128 PO	41 140 150	0 81–82 82–83	30	0.08 0.08	< 0.01 < 0.01	0.05 0.05 c0.04	< 0.01 < 0.01
0912 B2RF					Mean	0.08	< 0.01	0.05	< 0.01
Hinton, OK, USA,	3 (1128)	291 PP 128 PO	41 112	0 80	22	0.06 0.05	< 0.01 < 0.01	0.75 0.97	0.03 0.03
2012		128 PO	140	87	Mean	0.06	< 0.01	0.86	0.03
DP 0912 B2RF					29	0.06 0.06	< 0.01 < 0.01	0.83 0.73 c0.05	0.03 0.02
					Mean	0.06	< 0.01	0.78	0.02
					36	0.07 0.07	< 0.01 < 0.01	0.93 0.91	0.03 0.04
					Mean	0.07	< 0.01	0.92	0.04
					44	0.08 0.06	< 0.01 < 0.01	0.71 0.81	0.02 0.03
					Mean	0.07	< 0.01	0.76	0.02
					51	0.06 0.03	< 0.01 < 0.01	0.85 0.51	0.03 0.02
					Mean	0.04	< 0.01	0.68	0.02
Levelland, TX, USA,	3 (1237)	299 PP 130 PO	38 178	0 80	30	0.04 0.04	< 0.01 < 0.01	0.09 0.09	< 0.01 < 0.01
2012 DP 0912 B2RF		129 PO	178	81	Mean	0.04	< 0.01	0.09	< 0.01
Porterville, CA, USA,	3 (1466)	291 PP 128 PO	45 140	0 84	30	0.13 0.08	< 0.01 < 0.01	0.23 0.24	< 0.01 < 0.01
2012 Untreated Upland [a]		128 PO	140	84	Mean	0.10	< 0.01	0.24	< 0.01
Porterville, CA, USA,	3 (1426)	299 PP 128 PO	46 140	0 84	30	0.32 0.21	< 0.01 < 0.01	0.21 0.18	< 0.01 < 0.01
2012 Untreated Upland [b]		128 PO	140	84	Mean	0.26	< 0.01	0.20	< 0.01
Visalia, CA, USA, 2012 Untreated	3 (1366)	295 PP 128 PO 128 PO	46 140 140	0 84 84	30	0.17 0.15	< 0.01 < 0.01	0.21 0.21 c0.08	0.01 0.01
Upland					Mean	0.16	< 0.01	0.21	0.01

1st spray at planting as a band spray (T-band) followed by two foliar sprays closer to harvest

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Scanner 0.25% v/v, Dyne-Amic 0.5% v/v, Induce 0.25% v/v, 80-20 Surfactant 0.25% v/v, Activator 90 0.25% v/v, Activator 90 0.25% v/v, Induce 0.5% v/v, Preference 1% v/v, Baron 0.06% v/v, R-11 0.22% v/v, Pro 90 0.5% v/v, Pro 90 0.5% v/v, Pro 90 0.5% v/v

Undelinted seed % moisture: 9.2, 14.6, 12.0, 11.6, 8.4, 9.8, 8.2, 9.6 (23 d), 7.8 (37 d), 8.9 (44 d), 9.4 (51 d), 7.9, 8.8, 8.8, 10.6

[a] Last application 10/10/2012

[b] Last application 10/10/2012, related location, same variety as other Porterville trial [a]

Table 38 Residues of flutriafol in rape seed in Europe following application of an SC formulation (Pollmann 2006a 1298; 2006b 1334; 2007a 1542)

Location, year, variety	No	g ai/ha	L/ha	GS (BBCH)	DALA	Sample	Flutriafol (mg/kg)
Northern Europe							
Bietigheim, Baden-	2	124	293	62	13	pods	0.62
Wurttemberg,	(26) [a]	131	311	80	20	pods	0.61
Germany, 2005					26	seed	0.13
Lisanne							
Padborg,	2	138	329	62	13	pods	0.08
Sonderjylland,	(49)	127	302	80	20	pods	0.11
Denmark, 2005					54	seed	0.03
Trabant							
Meistratzheim,	2	129	255	62	13	pods	0.2
Alsace, Northern	(28) [b]	125	247	80	21	pods	0.26
France, 2005					35	seed	0.07
Hability							
Charndon, Bicester,	2	131	313	62	13	pods	1.61
Oxfordshire, UK,	(55) [c]	129	307	80	20	pods	1.04
2005 Labrador					34	seed	0.31 (0.31 0.30)
Padborg, Sonderjylland, Denmark, 2006 Excalibur	2 (43) [d]	135 126	320 300	62 80	28	seed	0.04
Burweg, Niedersachsen, Germany, 2006 Titan	2 (39) [e]	137 137	327 327	62 80	32	seed	0.08
Wiesloch-Baiertal, Baden Wurrtemberg, Germany, 2006 Titan	2 (38)	136 121	323 287	62 80	28	seed	0.15
Drusenheim, Alsace, Northern France, 2007	2 (30) [f]	127 126	201 200	62 80	17	seed	0.08
Southern Europe							
Lavaur, Midi-	2	133	420	62	13	pods	0.42
Pyrénées, Southern	(42) [g]	134	424	80	21	pods	0.48
France, 2005 Corail					34	seed	0.15
+ Cocktail							
St. Paul Trois	2	132	345	62	15	pods	0.23 (0.24 0.22)
Chateaux, Rhone-	(41) [h]	117	305	80	22	pods	0.45 (0.45 0.44)
Alpes, Southern					29	seed	0.03
France, 2005 Navajo							
11420 Plaigne, Languedoc-Roussillon, Southern France, 2006	2 (50)	130 131	412 415	62 80	27	seed	0.05
Lavaur, Midi-Pyrenees, Southern France, 2006 Exagone	2 (50) [i]	134 126	425 400	62 80	24	seed	0.13

[a] 8 and 0.3 mm rain within 24 h 1st and 2nd sprays

[b] 6–7 mm rain within 24 h of the 2nd spray

[c] 2.6 mm rain within 24 h of the 2nd spray

[d] 1 mm rain within 24 h of the 2nd spray

[e] 1 mm rain within 24 h of the 2nd spray

[f] 10 mm rain within 24 h of the 2nd spray

[g] 14.4 and 0.2 mm rain within 24 h 1st and 2nd sprays

[h] 8.6 mm rain within 24 h of the 2nd spray

[i] 0.2 mm rain within 24 h of the 2nd spray

Animal feeds

Table 39 Residues of flutriafol in sugar beet (tops) following application of an SC formulation in the European Union (Pollmann 2006 1298)

Location, year, variety SUGAR BEET	No	g ai/ha	L/ha	GS (BBCH)	DALA	Sample	Flutriafol (mg/kg)
Scherwiller, Alsace, Northern	2	120	290	39	0	plant	0.45
France 2004 Guepard	(21) [a]	135	327	39	15	leaves	0.24
					22	leaves	0.28
					29	leaves	0.22
					41	leaves	0.13
Dollern, Niedersachsen,	2	131	263	45	0	plant	0.72
Germany 2004 Famosa	(22) [b]	126	253	43–44	14	leaves	0.45
					22	leaves	0.38
					27	leaves	0.14
					41	leaves	0.11
Haderslev, Jutland, Denmark	2	125	303	39	0	plant	1.08
2004 Verity	(21) [c]	111	269	46	15	leaves	0.5
					21	leaves	0.27
					28	leaves	0.18
					42	leaves	0.11
Holme, Peterborough, UK 2004	2	121	293	45	0	plant	1.02
Cinderella	(21) [d]	120	292	47	15	leaves	0.49
					20	leaves	0.32
					29	leaves	0.18
					41	leaves	0.14
Dudenbuttel, Lower Saxony,	2	126	300	43	22	leaves	0.14
Germany 2005 Ricardo	(21) [e]	131	311	44–46	28	leaves	0.1
Haderslav, Sonderjylland,	2	133	316	43–44	20	leaves	0.15
Denmark 2005 Verity	(21) [f]	138	329	46	28	leaves	0.14
Scherwiller, Alsace, Northern	2	123	292	39	21	leaves	0.64
France 2005 Canyon	(20) [g]	138	328	39	27	leaves	0.75
Bishop's Tachbrook,	2	127	302	47	21	leaves	0.33
Warwickshire, UK 2005	(21)	130	310	48	29	leaves	0.22
Cinderella							

[a] 6 mm rainfall within 24 h of 1st application

[b] 2 mm and 3 mm rain within 24 h 1st and 2nd spray

[c] 10.2 mm after 2nd spray

[d] 7 mm after 2nd spray

[e] 3 and 9 mm rain within 24 h 1st and 2nd spray

[f] 3 and 3 mm rain within 24 h 1st and 2nd spray

[g] 5 mm rainfall within 24 h of 1st application

Table 40 Residues of flutriafol in sugar beet (tops) following application of an SC formulation in Spain (Pollmann 2007 1381)

Location, year, variety SUGAR BEET	No	g ai/ha	L/ha	GS (BBCH)	DALA	Sample	Flutriafol (mg/kg)
Castelnuovo della Daunia,	3	132	320	35–37	0	plant	0.13
Puglia, Italy, 2004 Monatonno	(21	131	317	36–38	7	leaves	0.21
	22) [a]	127	308	45–47	15	leaves	0.22
					22	leaves	0.05
					29	leaves	0.01
Poggio Renatico, Emilia	3	127	410	37	0	plant	2.35
Romagna, Italy, 2004 Gea	(21	125	402	39–41	6	leaves	1.47
	21)	124	400	44	13	leaves	1.23
					20	leaves	0.36
					29	leaves	0.3
Pozoarmargo, Cuenca, Spain,	3	127	408	39	0	plant	0.51
2004 Vincent	(21	127	410	39	7	leaves	0.3

Location, year, variety SUGAR BEET	No	g ai/ha	L/ha	GS (BBCH)	DALA	Sample	Flutriafol (mg/kg)
	20)	124	401	39	15	leaves	0.28
					22	leaves	0.22
					30	leaves	0.29
Tobarra, Albacete, Spain, 2004	3	128	412	39	0	plant	0.54
Brigitta	(21	132	427	39	7	leaves	0.5
	21)	126	405	39	14	leaves	0.19
					21	leaves	0.14
					29	leaves	0.46
Tobarra, Albacete, Spain, 2005	3	122	390	39	20	leaves	0.26, 0.31
Heracles	(22	125	401	39	27	leaves	0.33, 0.34
	20)	117	373	42			
Poggio Renatico, Emilia	3	125	397	45	22	leaves	0.15, 0.14
Romagna, Italy, 2005 Opera	(21	124	393	47	28	leaves	0.05, 0.04
	21) [b]	127	403	47			
Ponte Pietra, Cesena, Emilia	3	128	407	42	22	leaves	0.84
Romagna, Italy, 2005 Gea	(20	123	390	44	28	leaves	0.74
	20) [c]	124	393	46			
Arevalo, Avila, Spain, 2006	3	131	312	39	22	leaves	0.33
Brigitta	(20	138	328	39	29	leaves	0.18
	21)	126	299	39			

[a] 0.4 mm rain with 24 h 1st spray

[b] 3.6 mm rain with 24 h 2nd spray

[c] 0.6 mm rain with 24 h 3rd spray

Table 41 Residues of flutriafol in sugar beet (tops) in the USA following application of an SC formulation (Jones 2009 1812) (duplicate samples)

Location, year,		g		GS		Residue (mg/kg)			
variety	No	ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
Porterville, CA, USA, 2009	3	129	306	81	14	1.44 1.20	< 0.01 < 0.01	0.03 0.04	< 0.01 < 0.01
Pheonix	(14 14)	127 124	307 292	81–83 87	Mean	1.32	< 0.01	0.04	< 0.01
Fresno, CA, USA, 2009 HH142	3 (14 14)	125 128 128	325 329 329	48 48 49	14	0.83 0.96	< 0.01 < 0.01	0.03 0.04 c0.01	< 0.01 < 0.01
					Mean	0.9	< 0.01	0.04	< 0.01
American Falls, ID, USA, 2009	3	123	279	49	14	0.08 0.06	< 0.01 < 0.01	< 0.01 0.01	< 0.01 < 0.01
Hillshog 9026	(14 15)	129 123	295 318	49 49	Mean	0.07	< 0.01	< 0.01	< 0.01
Jerome, ID, USA, 2009	3	128	345	49	14	0.27 0.25	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
BTSCT01RR07	(14 14)	128 124	332 339	49 49	Mean	0.26	< 0.01	< 0.01	< 0.01
Geneva, MN, USA, 2009 Beta	3	129	288	Vegetative	14	0.65 0.61	< 0.01 < 0.01	0.01 0.01	< 0.01 < 0.01
130R	(15 13)	128 129	280 289	Vegetative Vegetative	Mean	0.63	< 0.01	0.01	< 0.01
Campbell, MN, USA, 2009	3 (13 14)	128 128	328 328	33 35	0	3.75 3.11	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
4012RR		129	330	49	Mean	3.43	< 0.01	< 0.01	< 0.01
					7	0.67 0.63	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.65	< 0.01	< 0.01	< 0.01
					14	0.40 0.45	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.43	< 0.01	< 0.01	< 0.01
					21	0.21 0.28	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01

Location, year,		g		GS		Residue (mg/kg)			
variety	No	ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
					Mean	0.25	< 0.01	< 0.01	< 0.01
					28	0.23 0.23	< 0.01 < 0.01	0.01 0.01	< 0.01 < 0.01
					Mean	0.23	< 0.01	0.01	< 0.01
Paynesville, MN, USA,	3 (13 14)	130 131	283 285	45 45	14	0.02 0.04	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
2009 Crystal RR202		130	281	47	Mean	0.03	< 0.01	< 0.01	< 0.01
Pavillion, WY, USA, 2009	3 (14 14)	128 130	304 302	49 49	14	1.72 1.83	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Beta 36RR11		130	318	49	Mean	1.78	< 0.01	< 0.01	< 0.01
Northwood, ND, USA, 2009	3	127	325	39	14	0.16 0.11	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Beta 1305R	(15 13)	129 127	329 324	39 39	Mean	0.14	< 0.01	< 0.01	< 0.01
Velva, ND,	3	130	284	37	14	1.22 1.11	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
USA, 2009 R308	(14 14)	131 127	286 284	39 39	Mean	1.17	< 0.01	< 0.01	< 0.01
York, NE, USA, 2009 Beta	3 (14 14)	129	329	42 d before harvest	14	0.84 0.72	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
734IR		130 129	329 325	39 49	Mean	0.78	< 0.01	< 0.01	< 0.01
Levelland, TX, USA, 2009 Phoenix	3 (14 15)	130	324	Roots starting to enlarge	14	0.50 0.64	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
		124 127	322 325	roots enlarging maturing roots	Mean	0.57	< 0.01	< 0.01	< 0.01

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2

Table 42 Residues of flutriafol in almond hulls following application of an SC formulation in the USA (Rice 2011 2161) (duplicate samples)

Location,		g		g	GS		Residue (mg/kg)			
year, variety	No	ai/ha	L/ha	ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
Dinuba, CA, USA, 2010 Almond	6 (8 8 8 8 8 8)	128 129 128	731 750 781	17 17 16	75 75 78	14	2.17, 1.78	< 0.01 < 0.01	0.02 0.02 c0.02	< 0.01 < 0.01
Sonora		129 128 128	788 791 883	16 16 14	78 81 81	Mean	1.98	< 0.01	0.02	< 0.01
Strathmore, CA, USA, 2010	6 (6 7 7 7 7 7)	128 128 129	2759 2751 2768	4.6 4.6 4.7	79 79 79	14	6.90, 6.47	< 0.01, < 0.01	0.11 0.10 c0.16	0.02, 0.02 c0.04
Almond Fritz		128 128 128	2761 2753 2773	4.6 4.6 4.6	80 80 88	Mean	6.54	< 0.01	0.10	0.02
Wasco, CA, USA, 2010	6 (8 6 7 7 7 7)	128 128 128	809 788 791	16 16 16	79 79 79	14	1.77, 1.84	ND, ND	0.02 0.02 c0.02	< 0.01, < 0.01
		128 128 128	786 785 827	16 16 15	79 79 85	Mean	1.80	< 0.01	0.02	< 0.01
Buttonwillow, CA, USA, 2010	6 (7 7 7 7 7 7)	128 127 133	3301 3321 3313	3.9 3.8 4	78 79 79	14	4.28, 3.67	< 0.01, < 0.01	0.06 0.05 c0.03	0.02 0.02 c0.02
Almond Monterey's		128 128	3304 3327	3.9 3.8	83 85	Mean	3.98	< 0.01	0.06	0.02

Location,		g		g	GS		Residue (mg/kg)			
year, variety	No	ai/ha	L/ha	ai/hL	(BBCH)	DALA	Flutriafol	T	TA	TAA
		128	3223	4	87					
Terra Bella, CA, USA,	6 (9 7 9 8 8)	127 128	661 605	19 21	75 72	1	2.68, 2.52	ND, < 0.01	0.04 0.06	< 0.01, < 0.01
2010		127	627	20	78	Mean	2.60	< 0.01	0.05	< 0.01
Almond Non Pareil		129 129	661 661	19 19	79 79	7	0.99, 1.19	< 0.01 < 0.01	0.03 0.06	< 0.01 < 0.01
		128	661	20	81	Mean	1.09	< 0.01	0.04	< 0.01
						14	0.93, 1.21	< 0.01 < 0.01	0.04 0.05 c0.11	< 0.01 < 0.01 c0.02
						Mean	1.07	< 0.01	0.04	< 0.01
						21	1.12, 1.39	< 0.01 < 0.01	0.05 0.05	< 0.01 < 0.01
						Mean	1.26	< 0.01	0.05	< 0.01
						28	0.81, 0.70	< 0.01 < 0.01	0.03 0.04	< 0.01 < 0.01
						Mean	0.76	< 0.01	0.04	< 0.01

Table 43 Residues of flutriafol in maize forage following application of an SC formulation in the USA (Carringer 2010 1810) (duplicate samples). A non-ionic surfactant was added to the tank mix at all sites except for decline trials where plots were sprayed with and without surfactant.

Location,		g		GS		Residue (mg/kg)			
year, variety	No	ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
Germansville, PA, USA, 2009 Hybrid	2 (6)	131 130	140 140	79 85	0	2.30 2.57	< 0.01 < 0.01	0.01 0.01 c0.01	< 0.01 < 0.01
2D324 Mycogen Seed					Mean	2.44	< 0.01	0.01	< 0.01
Seven Springs, NC, USA, 2009	2 (7)	128 126	131 131	83 85	0	2.08 2.30	< 0.01 < 0.01	0.02 0.02 c0.03	< 0.01 < 0.01
N77-P5					Mean	2.19	< 0.01	0.02	< 0.01
Wyoming, IL, USA, 2009	2 (7)	129 129	112 112	75–83 83–85	0	1.37 1.22	< 0.01 < 0.01	0.01 < 0.01 c0.01	< 0.01 < 0.01
DKC 61–69					Mean	1.30	< 0.01	< 0.01	< 0.01
					1	0.987 0.160	< 0.01 < 0.01	0.01 < 0.01	< 0.01 < 0.01
					Mean	0.57	< 0.01	< 0.01	< 0.01
					7	1.26 1.11	< 0.01 < 0.01	0.02 0.01	< 0.01 < 0.01
					Mean	1.18	< 0.01	0.02	< 0.01
					14	0.87 1.11	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
					Mean	0.99	< 0.01	0.02	< 0.01
					21	0.74 0.87	< 0.01 < 0.01	0.01 0.02	< 0.01 < 0.01
					Mean	0.80	< 0.01	0.02	< 0.01
No surfactant		128 129	112 112	75–83 83–85	0	2.00 0.94	< 0.01 < 0.01	0.01 0.02	< 0.01 < 0.01
					Mean	1.47	< 0.01	0.02	< 0.01
					1	1.58 0.98	< 0.01 < 0.01	0.01 0.02	< 0.01 < 0.01
					Mean	1.28	< 0.01	0.02	< 0.01
					7	1.35 1.17	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
					Mean	1.26	< 0.01	0.02	< 0.01
					14	0.76 1.01	< 0.01 < 0.01	0.02 0.06	< 0.01 < 0.01
					Mean	0.88	< 0.01	0.04	< 0.01

Location,		g		GS		Residue (mg/kg)			
year, variety	No	ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
					21	0.64 0.50	< 0.01 < 0.01	0.03 0.03	< 0.01 < 0.01
					Mean	0.57	< 0.01	0.03	< 0.01
Carlyle, IL, USA, 2009 8G23	2 (7)	130 133	112 131	85 85	0	0.53 0.53	< 0.01 < 0.01	0.02 0.02 c0.02	< 0.01 < 0.01
					Mean	0.53	< 0.01	0.02	< 0.01
Grantfork, IL, USA, 2009	2 (7)	130 128	122 103	85 85	0	1.85 1.93	< 0.01 < 0.01	< 0.01 0.01 c0.02	< 0.01 < 0.01
AgriGolg AG457					Mean	1.89	< 0.01	< 0.01	< 0.01
Conklin, MI, USA, 2009 A1005113	2 (7)	128 128	122 122	85 85–86	0	1.01 1.27	< 0.01 < 0.01	0.02 0.02 c0.02	< 0.01 < 0.01
					Mean	1.14	< 0.01	0.02	< 0.01
Richland, IA, USA, 2009	2 (8)	129 129	140 140	79 87	0	1.83 1.47	< 0.01 < 0.01	0.02 0.03	< 0.01 < 0.01
Pioneer					Mean	1.65	< 0.01	0.02	< 0.01
34R67					1	1.26 1.20	< 0.01 < 0.01	0.03 0.02	< 0.01 < 0.01
					Mean	1.23	< 0.01	0.02	< 0.01
					7	0.31 0.30	< 0.01 < 0.01	0.03 0.02	< 0.01 < 0.01
					Mean	0.30	< 0.01	0.02	< 0.01
					13	0.32 0.34	< 0.01 < 0.01	0.03 0.02	< 0.01 < 0.01
					Mean	0.33	< 0.01	0.02	< 0.01
					20	0.32 0.34	< 0.01 < 0.01	0.03 0.02	< 0.01 < 0.01
					Mean	0.33	< 0.01	0.02	< 0.01
No surfactant	2 (8)	129 129	140 140	79 87	0	1.05 0.99	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
					Mean	1.02	< 0.01	0.02	< 0.01
					1	0.68 0.74	< 0.01 < 0.01	0.02 0.03	< 0.01 < 0.01
					Mean	0.71	< 0.01	0.02	< 0.01
					7	0.13 0.13	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
					Mean	0.13	< 0.01	0.02	< 0.01
					13	0.19 0.21	< 0.01 < 0.01	0.02 0.03	< 0.01 < 0.01
					Mean	0.20	< 0.01	0.02	< 0.01
					20	0.19 0.18	< 0.01 < 0.01	0.04 0.03	< 0.01 < 0.01
					Mean	0.19	< 0.01	0.04	< 0.01
Douds, IA, USA, 2009	2 (6)	131 128	150 140	75–78 85	0	1.48 1.42	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Garst 84N57					Mean	1.45	< 0.01	< 0.01	< 0.01
Batavia, IA, USA, 2009 Garst 82K79	2 (6)	132 130	150 140	75–78 85	0	1.56 1.17	< 0.01 < 0.01	0.03 0.03 c0.05	< 0.01 < 0.01
					Mean	1.36	< 0.01	0.03	< 0.01
LaPlata, MO, USA, 2009 LG 2614 VT	2 (6)	127 129	140 140	75–80 83–85	0	0.74 1.08	< 0.01 < 0.01	< 0.01 0.01 c0.01	< 0.01 < 0.01
					Mean	0.91	< 0.01	< 0.01	< 0.01
Jefferson, IA, USA, 2009 33H27	2 (7)	131 130	131 122	85 85	0	3.47 1.84	< 0.01 < 0.01	0.02 0.01 c0.02	< 0.01 < 0.01
					Mean	2.66	< 0.01	0.02	< 0.01
Bagley, IA,	2 (7)	131	140	85	0	1.50 1.76	< 0.01	0.01	< 0.01

Location,		g		GS		Residue (mg/kg)			
year, variety	No	ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
USA, 2009 33M16		130	103	85			< 0.01	0.01 c0.02	< 0.01
					Mean	1.63	< 0.01	0.01	< 0.01
Bristol, IN, USA, 2009	2 (7)	128 128	122 122	83–85 86	0	1.50 1.56	< 0.01 < 0.01	0.02 0.01	< 0.01 < 0.01
34F97					Mean	1.53	< 0.01	0.02	< 0.01
York, NE, USA, 2009 7B15RRY	2 (8)	129 129	140 140	83 85	0	2.20 1.50	< 0.01 < 0.01	0.02 0.02 c0.02	< 0.01 < 0.01
GCBP					Mean	1.85	< 0.01	0.02	< 0.01
Osceola, NE, USA, 2009 7B15RRY	2 (7)	128 129	140 140	83 85	0	1.8 1.74	< 0.01 < 0.01	0.05 0.04 c0.02	< 0.01 < 0.01
GCBP					Mean	1.77	< 0.01	0.04	< 0.01
Geneva, NE, USA, 2009 7B15RRY	2 (8)	129 129	140 140	83 85	0	1.07 1.10	< 0.01 < 0.01	0.02 0.02 c0.02	< 0.01 < 0.01
GCBP					Mean	1.08	< 0.01	0.02	< 0.01
Geneva, MN, USA, 2009 Pioneer	2 (7)	127 128	140 140	R4 86	0	1.41 1.90	< 0.01 < 0.01	0.01 0.01 c0.01	< 0.01 < 0.01
38P43					Mean	1.66	< 0.01	0.01	< 0.01
Paynesville, MN, USA, 2009 Dekalb	2 (7)	129 129	131 131	85 85	0	1.99 1.51	< 0.01 < 0.01	< 0.01 < 0.01 c0.02	< 0.01 < 0.01
DKC35					Mean	1.75	< 0.01	< 0.01	< 0.01
Fitchburg, WI, USA, 2009 Pioneer	2 (7)	127 127	131 131	83 85–86	0	2.71 2.77	< 0.01 < 0.01	0.01 0.01 c0.01	< 0.01 < 0.01
37Y14					Mean	2.74	< 0.01	0.01	< 0.01
Hinton, OK, USA, 2009 DKC 52–59	2 (7)	128 128	131 131	85 85	0	0.77 0.71	< 0.01 < 0.01	0.04 0.04 c0.05	< 0.01 < 0.01
					Mean	0.74	< 0.01	0.04	< 0.01

1 X-77 @ 0.25% v/v; 2 Induce @ 0.34% v/v; 3 Aquagene 90 @ 0.05% v/v; 4 Surfac 820 @ 0.25% v/v; 5 NIS @ 0.25% v/v; 6 R-11 @ 0.064% v/v; 7 Silwet L-77 @ 0.25% v/v; 8 X-77 @ 0.25% v/v; 9 X-77 @ 0.25% v/v; 10 X-77 @ 0.25% v/v; 11 Hel-Fire 90 @ 0.25% v/v; 12 Hel-Fire 90 @ 0.25% v/v; 13 R11 @ 0.064% v/v; 14 Cornbelt Premier 90 @ 0.25% v/v; 15 Cornbelt Premier 90 @ 0.063% v/v; 16 Cornbelt Premier 90 @ 0.25% v/v; 17 Dyne Amic NIS @ 0.375% v/v; 18 Preference @ 0.25% v/v; 19 Preference @ 0.25% v/v; 20 Baron @ 0.076% v/v

Moisture content %: 70.6, 68.2, 69.9 (0 d), 69.8 (1 d), 67.2 (7 d), 57.7 (14 d), 56.3 (21 d), 71.5, 70.4, 72.7, 70.6 (0 d), 66.5 (1 d), 69.0 (7 d), 68.0 (13 d), 67.1 (20 d), 69.8, 70.0, 71.3, 68.6, 71.2, 72.3, 67.7, 65.3, 65.9, 71.3, 54.2, 62.4, 61.4

Table 44 Residues of flutriafol in maize stover following application of an SC formulation in the USA (Carringer 2010 1810) (duplicate samples). A non-ionic surfactant was added to the tank mix at all sites except for decline trials where plots were sprayed with and without surfactant.

Location,		g		GS		Residue (mg/kg)			
year, variety	No	ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
Germansville, PA, USA,	2 (6)	129 132	140 140	87 89	6	2.67 3.31	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
2009 Hybrid 2D324 Mycogen Seed					Mean	2.99	< 0.01	< 0.01	< 0.01
Seven Springs, NC, USA, 2009	2 (7)	129 131	131 131	86 89	6	2.25 1.89	< 0.01 < 0.01	< 0.01 0.02 c0.03	< 0.01 < 0.01
N77-P5					Mean	2.07	< 0.01	< 0.02	< 0.01
Wyoming, IL, USA,	2 (7)	129 128	112 112	89 89	0	1.23 0.92	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
2009					Mean	1.08	< 0.01	< 0.01	< 0.01
DKC 61-69					1	1.04 1.76	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	1.40	< 0.01	< 0.01	< 0.01
					7	0.62 0.93	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.78	< 0.01	< 0.01	< 0.01
					15	0.84 0.71	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.78	< 0.01	< 0.01	< 0.01
					21	0.90 0.84	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.87	< 0.01	< 0.01	< 0.01
No surfactant	2 (7)	128 128	112 112	89 89	0	1.09 1.07	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	1.08	< 0.01	< 0.01	< 0.01
					1	1.48 1.40	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	1.44	< 0.01	< 0.01	< 0.01
					7	0.96 0.74	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.85	< 0.01	< 0.01	< 0.01
					15	0.74 0.72	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.73	< 0.01	< 0.01	< 0.01
					21	0.77 0.58	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.68	< 0.01	< 0.01	< 0.01
Carlyle, IL, USA, 2009	2 (8)	127 128	122 140	87 89	7	1.63 2.24	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
8G23					Mean	1.94	< 0.01	< 0.01	< 0.01
Grantfork, IL, USA,	2 (7)	130 130	122 112	89 89	7	0.87 0.90	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
2009 AgriGolg AG457					Mean	0.88	< 0.01	< 0.01	< 0.01
Conklin, MI, USA, 2009	2 (8)	128 128	122 122	87 88	6	1.06 1.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
A1005113					Mean	1.04	< 0.01	< 0.01	< 0.01
Richland, IA, USA, 2009	2 (7)	129 129	140	89 89	0	3.30 2.77	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Pioneer					Mean	3.04	< 0.01	< 0.01	< 0.01
34R67					1	0.77 0.89	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.83	< 0.01	< 0.01	< 0.01
					7	0.95 1.06	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	1.00	< 0.01	< 0.01	< 0.01

Location,		g		GS		Residue (mg/kg)			
year, variety	No	ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
					13	0.69 0.71	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.70	< 0.01	< 0.01	< 0.01
					20	0.78 1.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.90	< 0.01	< 0.01	< 0.01
No surfactant	2 (7)	128 129	140 140	89 89	0	2.46 2.36	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	2.41	< 0.01	< 0.01	< 0.01
					1	0.81 0.78	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.80	< 0.01	< 0.01	< 0.01
					7	0.56 0.64	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.59	< 0.01	< 0.01	< 0.01
					13	0.49 0.72	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.60	< 0.01	< 0.01	< 0.01
					20	0.62 0.60	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.61	< 0.01	< 0.01	< 0.01
Douds, IA, USA, 2009	2 (7)	126 127	140 131	87 87–89	7	1.34 1.54	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Garst 84N57					Mean	1.44	< 0.01	< 0.01	< 0.01
Batavia, IA, USA, 2009	2 (7)	129 126	140 131	87 87–89	7	2.73 2.54	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Garst 82K79					Mean	2.64	< 0.01	< 0.01	< 0.01
LaPlata, MO, USA, 2009	2 (7)	130 128	140 140	87 89	6	1.48 1.45	< 0.01 < 0.01	0.01 < 0.01	< 0.01 < 0.01
LG 2614 VT					Mean	1.46	< 0.01	< 0.01	< 0.01
Jefferson, IA, USA,	2 (7)	129 127	112 103	87 87	7	6.12 4.77	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
2009 33H27					Mean	5.44	< 0.01	< 0.01	< 0.01
Bagley, IA, USA, 2009	2 (7)	126 127	103 103	87 87	7	2.82 2.15	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
33M16					Mean	2.48	< 0.01	< 0.01	< 0.01
Bristol, IN, USA, 2009	2 (7)	128 128	122 122	87 88	8	0.87 0.56	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
34F97					Mean	0.72	< 0.01	< 0.01	< 0.01
York, NE, USA, 2009	2 (8)	129 124	140 140	87 87	6	2.82 3.27	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
7B15RRY GCBP					Mean	3.04	< 0.01	< 0.01	< 0.01
Osceola, NE, USA, 2009	2 (7)	129 129	140 140	87 87	7	3.71 4.25	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
7B15RRY GCBP					Mean	3.98	< 0.01	< 0.01	< 0.01
Geneva, NE, USA, 2009	2 (7)	128 128	140 140	87 87	6	3.25 2.73	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
7B15RRY GCBP					Mean	2.99	< 0.01	< 0.01	< 0.01
Geneva, MN, USA, 2009	2 (6)	129 129	140 140	87 87	8	2.33 2.43	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Pioneer 38P43					Mean	2.38	< 0.01	< 0.01	< 0.01
Paynesville, MN, USA,	2 (7)	129 130	131 131	87 89	7	0.02 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
2009 Dekalb DKC35					Mean	< 0.02	< 0.01	< 0.01	< 0.01
Fitchburg, WI, USA,	2 (6)	128 128	131 131	87 89	9	1.23 1.40	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
2009 Pioneer 37Y14					Mean	1.32	< 0.01	< 0.01	< 0.01

Location,		g		GS		Residue (mg/kg)			
year, variety	No	ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
Hinton, OK, USA, 2009	2 (7)	129 129	131 131	87 87	7	2.65 1.89	< 0.01 < 0.01	0.03, 0.03 c0.02	< 0.01 < 0.01
DKC 52–59					Mean	2.27	< 0.01	0.03	< 0.01

1 X-77 @ 0.25% v/v; 2 Induce @ 0.34% v/v; 3 Aquagene 90 @ 0.05% v/v; 4 Surfac 820 @ 0.25% v/v; 5 NIS @ 0.25% v/v; 6 R-11 @ 0.064% v/v; 7 Silwet L-77 @ 0.25% v/v; 8 X-77 @ 0.25% v/v; 9 X-77 @ 0.25% v/v; 10 X-77 @ 0.25% v/v; 11 Hel-Fire 90 @ 0.25% v/v; 12 Hel-Fire 90 @ 0.25% v/v; 13 R11 @ 0.064% v/v; 14 Cornbelt Premier 90 @ 0.25% v/v; 15 Cornbelt Premier 90 @ 0.063% v/v; 16 Cornbelt Premier 90 @ 0.25% v/v; 17 Dyne Amic NIS @ 0.375% v/v; 18 Preference @ 0.25% v/v; 19 Preference @ 0.25% v/v; 20 Baron @ 0.076% v/v

Moisture contents %: 57.2, 57.2, 63.2 (0 d), 67.8 (1 d), 57.8 (7 d), 61.2 (15 d), 55.1 (21 d), 61.4, 45.8, 69.6, 63.4 (0 d), 72.3 (1 d), 66.7 (7 d), 61.6 (13 d), 52.1 (20 d), 63.9, 67.7, 60.8, 33.0, 65.6, 62.2, 56.1, 61.9, 61.7, 64.6, 39.2, 65.2, 55.0.

Plots were established for the collection of the forage samples and the applications timed such that the forage samples were collected nominally at soft dough to hard dough stage (BBCH 85–87) 30 days (± 1) after the last application (30-day PHI).

Table 45 Residues of flutriafol in sorghum forage following application of an SC formulation in the USA (Carringer 2013 2699) (duplicate samples, applications include non-ionic surfactant, separate plots to those used for grain and stover)

				GS		Residue (mg/kg)			
Location, year, variety	No	g ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
Seven Springs, NC, USA, 2012 DKS54-00	2 (7)	129 129	178 168	37 39	30	0.21 0.17	< 0.01 < 0.01	0.10 0.08	0.04 0.03
					Mean	0.19	< 0.01	0.09	0.04
Proctor, AR, USA, 2012 GX12564	2 (7)	128 129	150 150	Pre-heading Pre-heading	30	0.36 0.21	< 0.01 < 0.01	0.03 0.03 c0.01	0.01 0.01
					Mean	0.28	< 0.01	0.03	0.01
Richland, IA, USA, 2012 Pioneer 84G62	2 (7)	128 131	178 178	39 51	30	0.07 0.10	< 0.01 < 0.01	0.04 0.04 c0.03	< 0.01 < 0.01
					Mean	0.08	< 0.01	0.04	< 0.01
Kirksville, MO, USA, 2012 Pioneer 84G62	2 (7)	123 126	159 159	39 51	30	0.26 0.22	< 0.01 < 0.01	0.03 0.03 c0.02	< 0.01 < 0.01
					Mean	0.24	< 0.01	0.03	< 0.01
Stafford, KS, USA, 2012 84G62	2 (7)	124 130	159 168	47 53	29	0.23 0.28	< 0.01 < 0.01	0.05 0.04	< 0.01 < 0.01
					Mean	0.26	< 0.01	0.04	< 0.01
York, NE, USA, 2012 85G01	2 (7)	127 128	178 187	65 71	31	0.20 0.21	< 0.01 < 0.01	0.05 0.06 c0.03	0.02 0.03 c0.01
					Mean	0.20	< 001	0.06	0.02
Uvalde, TX USA, 2012 Pioneer 83G19	2 (7)	128 128	140 150	16 18	30	0.47 0.61	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.54	< 0.01	< 001	< 0.01
Hinton, OK, USA, 2012 DKS29-28	2 (7)	128 128	168 178	68 69	30	0.82 1.18	< 0.01 < 0.01	0.06 0.06 c0.02	0.02 0.03
					Mean	1.0	< 0.01	0.06	0.02
Grand Island, NE, USA, 2012 85G01	2 (7)	128 128	178 178	75 85	30	0.61 0.67	< 0.01 < 0.01	0.02 0.02 c0.03	0.02 0.02 c0.02
					Mean	0.64	< 0.01	0.02	0.02
Larned, KS, USA, 2012 84G62	2 (7)	131 132	178 178	59 69	22	0.61 0.65	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
					Mean	0.63	< 0.01	0.02	< 0.01
					29	0.57 0.48	< 0.01 < 0.01	0.03 0.02 c0.01	< 0.01 < 0.01

				GS		Residue (mg/kg)			
Location, year, variety	No	g ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
					Mean	0.52	< 0.01	0.02	< 0.01
					37	0.27 0.28	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
					Mean	0.28	< 0.01	0.02	< 0.01
					44	0.21 0.24	< 0.01 < 0.01	0.02 0.03	< 0.01 < 0.01
					Mean	0.22	< 0.01	0.02	< 0.01
					50	0.23 0.23	< 0.01 < 0.01	0.04 0.03	< 0.01 < 0.01
					Mean	0.23	< 0.01	0.04	< 0.01
Wall, TX, USA, 2012 DKS44-20	2 (7)	128 129	131 140	38 43	29	0.77 0.66	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
					Mean	0.72	< 0.01	0.02	< 0.01
Levelland, TX, USA, 2012 165310	2 (7)	129 130	178 178	55 51–59	30	0.79 0.78	< 0.01 < 0.01	0.02 0.02	< 0.01 < 0.01
					Mean	0.78	< 0.01	0.02	< 0.01

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2

Induce 0.28-0.3% v/v, Dyne-Amic 0.5% v/v, Preference 0.5% v/v, Preference 0.5% v/v, Spreader 90 0.25% v/v, Cornbelt Premier 90 0.03% v/v, Induce 0.2% v/v, Baron 0.25% vv, Cornbelt Premier 0.03% v/v, Spreader 90 0.25% v/v, Induce 0.5% v/v, R-11 0.22% v/v

Table 46 Residues of flutriafol in sorghum stover following application of an SC formulation in the USA (Carringer 2013 2699) (duplicate samples, applications include non-ionic surfactant)

				GS		Residue (mg/kg)			
Location, year, variety	No	g ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
Seven Springs, NC, USA, 2012 DKS54-00	2 (7)	129 129	178 168	37 39	30	0.44 0.41	< 0.01 < 0.01	0.01 < 0.01	0.02 0.02
					Mean	0.42	< 0.01	< 0.01	0.02
Proctor, AR, USA, 2012 GX12564	2 (7)	128 129	150 150	Pre-heading Pre-heading	30	0.44 0.46	< 0.01 < 0.01	0.02 0.01 c0.02	< 0.01 < 0.01
					Mean	0.45	< 0.01	0.02	< 0.01
Richland, IA, USA, 2012 Pioneer 84G62	2 (7)	128 131	178 178	39 51	30	1.35 0.93	< 0.01 < 0.01	0.01 0.01 c0.02	< 0.01 < 0.01
					Mean	1.14	< 0.01	0.01	< 0.01
Kirksville, MO, USA, 2012 Pioneer 84G62	2 (7)	123 126	159 159	39 51	30	0.86 0.89	< 0.01 < 0.01	< 0.01 0.01 c0.02	< 0.01 < 0.01
					Mean	0.88	< 0.01	< 0.01	< 0.01
Stafford, KS, USA, 2012 84G62	2 (7)	124 130	159 168	47 53	29	0.80 0.80	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.80	< 0.01	< 0.01	< 0.01
York, NE, USA, 2012 85G01	2 (7)	127 128	178 187	65 71	31	0.67 0.70	< 0.01 < 0.01	0.02 0.04 c0.01	< 0.01 0.01
					Mean	0.68	< 0.01	0.03	< 0.01
Uvalde, TX USA, 2012 Pioneer 83G19	2 (7)	128 128	140 150	16 18	30	1.70 1.21	< 0.01 < 0.01	0.02 0.01	< 0.01 < 0.01
					Mean	1.46	< 0.01	0.02	< 0.01
Hinton, OK, USA, 2012 DKS29-28	2 (7)	128 128	168 178	68 69	30	0.92 0.92	< 0.01 < 0.01	0.06 0.06 c0.01	0.02 0.02
					Mean	0.92	< 0.01	0.06	0.02
Grand Island, NE, USA, 2012 85G01	2 (7)	128 128	178 178	75 85	30	0.55 0.50	< 0.01 < 0.01	0.01 0.01 c0.01	< 0.01 < 0.01
					Mean	0.52	< 0.01	0.01	< 0.01

				GS		Residue (mg/kg)			
Location, year, variety	No	g ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
Larned, KS, USA, 2012 84G62	2 (7)	131 132	178 178	59 69	23	0.29 0.28	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.28	< 0.01	< 0.01	< 0.01
					29	0.33 0.26	< 0.01 < 0.01	0.01 < 0.01	< 0.01 < 0.01
					Mean	0.30	< 0.01	< 0.01	< 0.01
					36	0.27 0.23	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.25	< 0.01	< 0.01	< 0.01
					43	0.22 0.25	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	0.24	< 0.01	< 0.01	< 0.01
					50	0.25 0.27	< 0.01 < 0.01	< 0.01 0.01	< 0.01 < 0.01
					Mean	0.26	< 0.01	< 0.01	< 0.01
Wall, TX, USA, 2012 DKS44-20	2 (7)	128 129	131 140	38 43	29	5.05 [5.78 4.86 4.52] 3.74 [4.30 3.28 3.65]	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
					Mean	4.40	< 0.01	< 0.01	< 0.01
Levelland, TX, USA, 2012 165310	2 (7)	129 130	178 178	55 51–59	30	1.72 1.33	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 0.01
					Mean	1.52	< 0.01	< 0.01	< 0.01

Analytical method flutriafol: RAM 219/04

Analytical method T, TA, TAA: Meth-160, revision 2

Induce 0.28-0.3% v/v, Dyne-Amic 0.5% v/v, Preference 0.5% v/v, Preference 0.5% v/v, Spreader 90 0.25% v/v, Cornbelt Premier 90 0.03% v/v, Induce 0.2% v/v, Baron 0.25% vv, Cornbelt Premier 0.03% v/v, Spreader 90 0.25% v/v, Induce 0.5% v/v, R-11 0.22% v/v

Table 47 Residues of flutriafol in rape plants in Europe following application of an SC formulation (Pollmann 2006a 1298; 2006b 1334; 2007a 1542)

Location, year, variety	No	g ai/ha	L/ha	GS (BBCH)	DALA	Sample	Flutriafol residues (mg/kg)
Northern Europe							
Bietigheim, Baden-	2	124	293	62	0	shoots	2.2
Wurttemberg,	(26)	131	311	80	7	shoots	0.39
Germany, 2005	[a]				13	plant	0.22
Lisanne					20	plant	0.12
Padborg,	2	138	329	62	0	shoots	2.4
Sonderjylland,	(49)	127	302	80	6	shoots	0.28
Denmark, 2005					13	plant	0.26
Trabant					20	plant	0.17
Meistratzheim,	2	129	255	62	0	shoots	1.88
Alsace, Northern	(28)	125	247	80	7	shoots	0.24
France, 2005	[b]				13	plant	0.19
Hability					21	plant	0.07
Charndon, Bicester,	2	131	313	62	0	shoots	3.18
Oxfordshire, UK,	(55)	129	307	80	7	shoots	1.75
2005 Labrador	[c]				13	plant	0.62
					20	plant	0.41
Southern Europe							
Lavaur, Midi-	2	133	420	62	0	shoots	2.22
Pyrénées, Southern	(42)	134	424	80	6	shoots	0.59
France, 2005 Corail	[d]				13	plant	0.42
+ Cocktail					21	plant	0.23
St. Paul Trois	2	132	345	62	0	shoots	2.19
Chateaux, Rhone-	(41)	117	305	80	6	shoots	0.22
Alpes, Southern	[e]				15	plant	0.1
France, 2005 Navajo					22	plant	0.06

[a] 8 and 0.3 mm rain within 24 h 1st and 2nd sprays
[b] 6-7 mm rain within 24 h of the 2nd spray
[c] 2.6 mm rain within 24 h of the 2nd spray
[d] 14.4 and 0.2 mm rain within 24 h 1st and 2nd sprays
[e] 8.6 mm rain within 24 h of the 2nd spray

Table 48 Residues of flutriafol in cotton gin by-products (trash) following application of an SC formulation in the USA (Carringer 2013 2700) (duplicate samples, applications include non-ionic surfactant)

Location,				GS		Residue (mg/kg)			
year, variety	No	g ai/ha	L/ha	(BBCH)	DALA	Flutriafol	T	TA	TAA
Wall, TX, USA, 2012	3 (105	295 124	41 168	0 82	30	2.25 2.28	< 0.01 < 0.01	< 0.01 < 0.01	0.02 0.02
DP 0912 B2RF	7)	127	168	83	Mean	2.26	< 0.01	< 0.01	0.02
Hinton, OK, USA,	3 (112	291 128	41 112	0 80	23	0.88 0.94	< 0.01 < 0.01	0.02 0.03	0.16 0.15
2012	8)	128	140	87	Mean	0.91	< 0.01	0.02	0.16
DP 0912 B2RF					30	0.93 0.82	< 0.01 < 0.01	0.03 0.02	0.22 0.18 c0.01
					Mean	0.88	< 0.01	0.02	0.20
					37	1.19 1.05	< 0.01 < 0.01	0.01 0.02	0.18 0.22
					Mean	1.12	< 0.01	0.02	0.20
					44	1.02 0.85	< 0.01 < 0.01	0.03 0.03	0.16 0.16
					Mean	0.94	< 0.01	0.03	0.16
					51	0.82 0.97	< 0.01 < 0.01	0.02 0.03	0.12 0.14
					Mean	0.90	< 0.01	0.02	0.13
Levelland, TX, USA,	3 (123	299 130	38 178	0 80	30	1.74 1.80	< 0.01 < 0.01	0.01 0.01	0.02 0.03
2012 DP 0912 B2RF	7)	129	178	81	Mean	1.77	< 0.01	0.01	0.02

1st spray at planting as a band spray (T-band) followed by two foliar sprays closer to harvest
Gin by-products %moisture: 10.4, 18.0 (23 d), 18.0 (30 d), 9.6 (37 d), 13.6 (44 d), 13.4 (51 d), 10.4

FATE OF RESIDUES IN STORAGE AND POCESSING

In processing

The hydrolytic behaviour of [^{14}C]flutriafol was studied under conditions at high temperatures in sterile aqueous buffers at pH 4, 5 and 6 for periods of up to 60 minutes in order to simulate common processing practices (pasteurisation, baking/brewing/boiling, and sterilisation) (Hiler 2012 2441). The concentration of flutriafol was approximately 1 mg/L.

Table 49 Conditions for simulated processing trials (Hiler 2012 2441)

Simulated process	pH	Nominal temperature	Test period
Pasteurisation	4 ± 0.1	90 ± 5 °C	20 minutes
Baking/Brewing/Boiling	5 ± 0.1	100 ± 5 °C	60 minutes
Sterilisation	6 ± 0.1	120 ± 5 °C	20 minutes

Recoveries of ^{14}C ranged from 98.6 to 108.1% of that applied. Flutriafol was not degraded under any of the sets of conditions tested. Therefore it is concluded that flutriafol should remain stable in /on processed commodities during common processing practices.

Table 50 Stability of flutriafol during simulations of typical processing conditions (Hiler 2012 2441)

	Flutriafol % of Applied Dose		
	pH 4 Buffer Test System (90 °C ± 5 °C)	pH 5 Buffer Test	pH 6 Buffer Test System

Sample		System (100 °C ± 5 °C)	(120 °C)
Time 0 Rep A	99.1	98.6	99.1
Time 0 Rep B	99.9	98.7	99.2
Time 20 min Rep A	100.7	101	108.1
Time 20 min Rep B	100.4	100.4	105.9

Peach

Two processing trials were conducted on peaches and nectarines in Spain (Martos 2011 2187.2 FLU amdt-1). Three foliar air blast applications were made using an SC formulation of flutriafol at a rate of 30 g ai/ha with a 7 day interval. Mature peaches and nectarines were sampled at a PHI of 7 days and were transported at ambient temperature to the processing facility where they were processed into juice and jam within 24 hours.

The fresh fruit was washed with water sprayed from a constant gas pressure sprayer (approx. 0.75 L water per kg fruit). Thereafter the fruit sample was divided into two portions and a minimum of 10 kg was used for processing into juice and 2 kg was used for processing into jam. Stones were removed and the separated pulp and stones weighed before discarding the stones.

Processing to Juice

Fruit pulp was then passed through a liquidiser to obtain the juice. Extracted fruit pulp (flesh) and raw juice were both weighed before discarding the extracted fruit pulp (waste). The pH of the juice was checked to be in the region of pH 3.5 before filtration and bottling.

Processing to jam

The fruit flesh was then cut into small pieces and heated until boiling. The heat was then reduced and the fruit allowed to simmer for approximately 15 minutes to provide raw fruit purée. Sugar was added at a ratio of 1:1 to the purée and the jam heated for 45 minutes until the Brix reached 65–68 °. The pH of the jam was checked to be in the region of pH 3.5 before being filled into glass bottles. The bottles were then tightly sealed and sterilized for 10 minutes (boiling water method).

Samples were stored frozen until analysed using a validated analytical method for residues of flutriafol. The LOQ of the method is 0.01 mg/kg for flutriafol.

Results show no significant difference of residues in processed products compared to the raw agricultural commodity with residues ranging from 0.03 to 0.05 mg/kg in fruit, 0.05 to 0.04 mg/kg in juice and 0.05 to 0.02 mg/kg in jam. The worst case PF was approximately 1.7 for juice and 1.0 for jam.

Table 51 Residues of flutriafol in peach juice and jam following processing of fruit (Martos 2011 2187.2 FLU amdt-1)

Location	N	g ai/ha	g ai/hL	BBCH	Matrix	Residue (mg/kg)	PF
Jumilla, Murcia,	3 (10 10)	34	3.13	77	Fruit	0.03	–
Spain, 2006 Amiga		36	3.13	78	Juice	0.05	1.7
		34	3.13	80	Jam	0.02	0.7
Blanca, Murcia,	3 (11 10)	30	3.13	77	Fruit	0.05	–
Spain, 2006 Elegant		32	3.13	78	Juice	0.04	0.8
Lady		31	3.13	80	Jam	0.05	1.0

Plums

One processing trial has been conducted on plums in the USA in 2009 (Carringer 2010 1808). Four foliar air blast applications were made using flutriafol formulated as a 125 g/L SC. All applications

were made at a rate of 640 g ai/ha. Applications were made with a 7 day interval with the final application being made 7 days before harvest. Mature plums were transported overnight at ambient temperature to the processing facility where they were processed into prunes.

Fruit (18 kg) were inspected, sorted and culls removed. The fresh plums were washed for 5 minutes using a ratio of 2 kg of cold water to each 1 kg of fruit. The washed fruit were placed on drying trays and air-dried at 68–79 °C. The fruit was removed when average moisture contents of 19.3 to 20.0% were achieved which is lower than the target of approximately 21 to 32%. The prunes were allowed to cool for approximately 20 minutes. The cooled prunes were packaged, labelled, and placed in frozen storage for the required prune sample fraction. The LOQ of the method is 0.01 mg/kg for flutriafol, T, TA and TAA in plums but the LOQ was raised to 0.05 mg/kg for TA in prunes due to the presence of endogenous material.

Fresh plums and prunes were analysed for residues of flutriafol and the three triazole metabolites using a validated analytical method. Results show an increase in residues of flutriafol in prunes from 0.64 mg/kg to 1.4 mg/kg. No residues of T or TAA were observed in fresh plums or prunes. Residues of TA were 0.07 mg/kg in plums and 0.10 mg/kg in prunes. It is therefore concluded that flutriafol and TA do concentrate in processed commodities. The PF was approximately 2.2 for flutriafol.

Table 52 Residues of flutriafol in dried prunes following processing of plums (Carringer 2010 1808) (means of duplicate samples)

						Residue (mg/kg)		
Location	N	g ai/ha	g ai/hL	BBCH	Sample	Flutriafol	TA	PF
Poplar, CA, USA, 2009 French	4 (7 7 7)	633 638 643 644	93	81 81 85 87	Fruit	0.64	0.07	-
prunes					Prune	1.4	0.10	2.2

PF = for flutriafol residues only

Grapes

Two trials have been conducted in Germany and Southern France, one trial in white grapes and one in red grapes in each country (Block 2013 2650). Each trial consists of three plots—one untreated and two treated plots. Four applications of an SC formulation of flutriafol were made to grape vines at an exaggerated rate of 450 g ai/ha. The interval between applications and the interval between last application and harvest was 14 days.

At the processing facility a total of eight processing trials were conducted, one for each treated plot. Two of these trials were balance trials, one balance trial in red wine and one in white wine. In the balance trials red grapes were processed into stems, must, alcohol fermented wine (AF wine), wet and dry pomace, malolactic fermented wine (MF wine), lees, sediments and red wine. The white grapes were processed into must, wet and dry pomace, must deposit, AF wine, sediments and white wine. In trials for magnitude of residues, samples were only taken in fresh grapes, must, dry pomace and wine.

For red wine, fresh grapes were crushed and stemmed. Potassium metabisulphite and dry yeast was added to must to initiate the fermentation. During this process sugar was added to enhance the alcohol content. The fermented must was then separated in a liquid (free-run wine) and solid part. The solid part was pressed to produce pressed wine and wet pomace. Pomace was dried at 60 °C to produce dry pomace. Free-run and pressed wine was combined (AF wine) before further processing. Lactic bacteria (*Leuconostoc oenos*) was added to AF wine in air-free conditions. Potassium metabisulphite was added and the clarification process started. The intermediate wine was racked to produce MF wine and lees. Further potassium metabisulphite plus gelatine was added to the MF wine. Clarification proceeds while the wine was stored at

10 °C. Solid matter was removed before filtration of the red wine. Finally potassium metabisulphite was added to the wine before bottling.

For white wine, fresh grapes were pressed directly into must and wet pomace. Dry pomace was produced as for red wine production. Pectolic enzymes and potassium metabisulphite were added to the must before racking. Then dry yeast was added to initiate the fermentation. During this process sugar was added to enhance the alcohol content. Potassium metabisulphite was added and the clarification process started. Then the fermented must was racked to produce AF wine and lees. Further clarification, removal of solid matter, filtration and bottling was performed as for red wine.

Both samples of fresh grapes and processed samples were stored and shipped at frozen conditions before analysis. All samples were analysed for the content of flutriafol and the three metabolites 1,2,4-triazole, triazole alanine and triazole acetic acid using two separate validated analytical methods. The LOQ and LOD are 0.01 mg/kg and 0.003 mg/kg respectively for both flutriafol and the metabolites.

For flutriafol in the mass balance processing results for red wine gave an increase in flutriafol mass to 300% of that originally present in the starting grapes. The results were recalculated assuming the original mass present is the sum of the mass of must and stems. Following the adjustment the mass balance for red and white wine are in general agreement. Most flutriafol is retained in the must (48–97%) and wet pomace (25–95%). The AF wine contained 32–35% of the flutriafol mass. Lees taken after fermentation contained 5–8% of the initial flutriafol amount. Wine at bottling contained 31–37% of the initial mass of flutriafol.

Table 53 Red wine balance—mass balance

Sample	Weight	Corrected weight	Residue flutriafol (mg/kg)	Mass flutriafol (mg)	%mass (grapes 38.56)	%mass (stems + must 118.51)
Grapes prior to processing	56.7	56.7	0.68	38.6	100	
Stems, after crushing and stemming	2.1	2.2	1.8	4.0	10	3
Must, after crushing and stemming	53.5	54.5	2.1	114.6	97	97
AF wine, after pressing	38.7	40.1	0.94	37.7	98	32
Wet pomace, after pressing	9.4	9.8	3	29.4	76	25
Dry pomace, after drying	1.7	3.3	10.2	33.2	86	28
MLF wine, after malolactic fermentation	29.5	37.6	0.92	34.6	90	29
Lees, after malolactic fermentation	1.7	2.2	2.8	6.0	16	5
Sediments, after clarification	0.53	1.3	1.0	1.3	3	1
Red wine, at bottling	14.9	35.9	1.0	37.0	96	31

Table 54 White wine balance—mass balance

Sample	Weight	Corrected weight	Residue flutriafol (mg/kg)	Mass flutriafol (mg)	%mass (grapes)
grape, prior processing	55.0	55.0	1.2	68.2	100
Must, after pressing	32.9	33.9	0.97	32.9	48
Wet pomace, after pressing	20.5	21.1	3.1	65.0	95
Dry pomace, after drying	1.2	4.98	6.7	33.6	49
Must deposit, after racking	3.0	3.2	1.2	3.9	6
AF wine, after alcoholic fermentation	24.4	26.8	0.90	24.1	35
Lees, after alcoholic fermentation	2.6	2.9	1.8	5.3	8
Sediment, after clarification	0.96	1.7	1.0	1.7	3
White wine, at bottling	14.2	24.6	1.0	25.5	37

No residues or very low levels of residues were seen for the metabolites in both fresh grapes and processed fractions. Therefore no PF is calculated for the metabolites. Flutriafol residues levels were higher and increased slightly in must and white wine. The PF is 1.8 for red

must, 1.6 for white must and 1.7 for white wine. No significant change in residue levels in red wine (PF of 1.1). A significant increase in flutriafol residues in dry pomace was observed with PFs of 10.7 and 6.5 for dry pomace from red and white wine production respectively.

Table 55 Transfer of residues of flutriafol in grape processed commodities (Block 2013 2650)

	kg ai/hL	kg ai/ha	PHI	GS BBCH	Portion analysed	Residue (mg/kg)	PF
Nieder-kirchen,	0.075	0.403	14	85	whole grape, prior processing	0.68	
Rheinland-Pfalz,	0.075	0.47			stems, after crushing and stemming	1.84	
Germany 2012	0.0749	0.436			must, after crushing and stemming	2.10	3.09
Spätbur-gunder	0.075	0.425			AF wine, after pressing	0.94	
(red grapes)					wet pomace, after pressing	3	4.4
					dry pomace, after drying	10.22	15.0
					MLF wine, after malolactic fermentation	0.92	
					lees, after malolactic fermentation	2.76	
					sediments, after clarification	1.01	
					red wine, at bottling	1.03	1.51
	0.0751	0.408	14	85	whole grape, prior processing	0.6	
	0.075	0.456			must, after crushing & stemming	1.67	2.42
	0.0751	0.453			dry pomace, after drying	12.25	17.75
	0.075	0.415			red wine, at bottling	1.09	1.58
Saint-Jean-d'Ardières,	0.0901	0.464	14	85	whole grape, prior processing	0.46	
Rhône, France 2012	0.09	0.487			must, after crushing and stemming	0.39	0.85
Gamay	0.0901	0.464			dry pomace, after drying	1.82	3.96
(red grapes)	0.0898	0.406			red wine, at bottling	0.26	0.57
	0.09	0.442	14	85	whole grape, prior processing	0.56	
	0.09	0.488			must, after crushing and stemming	0.54	0.98
	0.09	0.458			dry pomace, after drying	3.31	6.02
	0.09	0.45			red wine, at bottling	0.3	0.55
Nieder-kirchen,	0.075	0.44	14	85	whole grape, prior processing	1.24	
Rheinland-Pfalz,	0.075	0.426			must, after pressing	0.97	0.78
Germany 2012	0.075	0.422			wet pomace, after pressing	3.08	
Riesling (white	0.075	0.409			dry pomace, after drying	6.74	5.44
grapes)					must deposit, after racking	1.2	
					AF wine, after alcoholic fermentation	0.9	
					lees, after alcoholic fermentation	1.85	
					sediments, after clarification	1.02	
					white wine, at bottling	1.04	0.84
	0.0751	0.437	14	85	whole grape, prior processing	0.0751	
	0.075	0.441			must, after pressing	0.075	0.73
	0.0749	0.463			dry pomace, after drying	0.0749	6.71
	0.0749	0.433			white wine, at bottling	0.0749	0.79
Redessan, Gard,	0.0691	0.439	14	85	whole grape, prior processing	0.7	
France 2012	0.0692	0.505			must, after pressing	1.15	1.64
Roussanne Blanc	0.0692	0.462			dry pomace, after drying	3.04	4.34
(white grapes)	0.0693	0.488			white wine, at bottling	1.22	1.74
	0.0693	0.419	14	85	whole grape, prior processing	0.34	
	0.0692	0.465			must, after pressing	1.13	3.32
	0.0692	0.463			dry pomace, after drying	3.27	9.62
	0.0692	0.476			white wine, at bottling	1.14	3.35

Analytical method flutriafol: AGR/MOA/FLUTRI-1

Analytical method T, TA, TAA: AGR/MOA/TRZ-1

Strawberry

Four processing trials were conducted on protected strawberries in Spain in 2004 (Clark 2005 2583). Three applications of flutriafol were made, formulated as a 125 g/L SC using a hydraulic knapsack sprayer. All applications were made at a nominal rate of 18.75 g ai/hL using a nominal water volume

of 1000 L/ha. Applications were made with a 10 day interval with the final application being made 3 days before commercial harvest.

Mature fresh strawberries were harvested from the field and transported at cool temperature to the processing facility where they were processed into strawberry jam using processes considered typical of commercial practice.

Whole strawberries were washed with an automatic fruit washer (500–750 mL water per kg fruit) and strained. Strawberries (1.4–1.7 kg) were sorted and crushed and the Brix degree measured. White sugar was added to the crushed strawberries and then the sample was reduced in a double jacketed saucepan in order to reach 62 °Brix. The pH was adjusted with citric acid to approximately pH 3.5 and bottled. Packaged samples were then sterilised at 115 °C for 10 minutes.

Untreated and treated samples of fresh fruit prior to processing and processed jam were stored frozen and shipped under frozen conditions to the analytical laboratory for analysis. Samples were analysed using a validated analytical method. The LOQ of the method is 0.01 mg/kg.

Fresh strawberries and jam were both analysed for residues of flutriafol using a validated analytical method. Results show a decrease in residues in jam. The mean PF was 0.875 (range 0.75 to 0.96).

Table 56 Residues of flutriafol in strawberry jam following household processing of berries (Clark 2005 2583)

Location	n	g ai/ha	g ai/hL	BBCH	DALA	Sample	Residue (mg/kg)	PF
Almonte, Spain, 2005	3	191	18.75	61	3	Fruit	0.32	
Camarosa		189	18.75	87		Jam	0.24	0.75
		199	18.75	88				
Huelva, Spain, 2005	3	197	18.75	61	3	Fruit	0.13	
Ventana		178	18.75	87		Jam	0.12	0.92
		194	18.75	88				
Bonares, Spain, 2005	3	195	18.75	61	3	Fruit	0.23	
Camarosa		191	18.75	87		Jam	0.22	0.96
		194	18.75	88				
Huelva, Spain, 2005	3	194	18.75	61	3	Fruit	0.31[b]	
Ventana		192	18.75	87		Jam	0.27	0.87
		195	18.75	88				

Cabbage

Three processing trials were conducted on cabbage in the USA in 2011 (Carringer 2013 2697). Four applications of an SC flutriafol formulation were made at a nominal rate of 128 g ai/ha. Applications were made with a 7 day interval with the final application being made 7 days before harvest.

The cabbage heads for the Sample Prepared for Consumption (SPFC) samples were visually examined and any damaged or wilted leaves, as well as the wrapper leaves, removed. Each cabbage head was then rinsed under cold running tap water for approximately 15–20 seconds. The heads were turned top side down and allowed to drain for at least two minutes.

The control, RAC and SPFC samples were placed in frozen storage within 2.5 hours after collection from the field and maintained frozen during transportation to the analytical laboratory. Samples were analysed using validated analytical methods. The LOQ of the methods is 0.01 mg/kg for all analytes.

RAC samples and SPFC samples were all analysed for residues of flutriafol and triazole metabolites using a validated analytical method. Results show a decrease in residues of flutriafol in the samples prepared for consumption with PFs ranging from 0.05 to 0.14.

Table 57 Residues of flutriafol in cabbage following household processing of plants(Carringer 2013 2697) (means of duplicate samples)

Location	N	g ai/ha	g ai/hL	DALA	Sample	Residue (mg/kg)		PF
						flutriafol	TA	
Seven Springs, NC, USA, 2011 Bravo	4	129 129 131 127	41 41 42 44	7	RAC	0.74	0.04	
				7	SPFC	0.04	0.06	0.05
Uvalde, TX, USA, 2011 Pennant	4	128 127 131 128	46 47 48 49	7	RAC	0.07	0.01	
				7	SPFC	0.01	0.01	0.14
Porterville, CA, USA, 2011 Supreme Vantage	4	127 130 128 129	45 47 48 49	7	RAC	0.09	0.04	
				7	SPFC	< 0.01	0.05	< 0.11

PF = for flutriafol residues only

SPFC = samples prepared for consumption

Tomato

One processing study has been conducted on tomatoes in the USA in 2011 (Carringer 2012 2440). Four applications of flutriafol (SC formulation) were made at five times the nominal rate of 128 g ai/ha with a 7 day interval and the final application being made 0 days before commercial harvest. Mature tomato fruit were transported cool (approximately 4 °C) to the processing facility where they were processed into tomato purée and tomato paste.

For juice, tomatoes were soaked in aqueous NaOH (ca. 0.1 N) at 52–60 °C for 3 minutes and rinsed with warm (68–74 °C) water before being crushed, rapidly heated to 79–85 °C, held for 30 seconds and separated into pomace and juice. The wet pomace was pressed to recover additional juice which was combined.

For purée, an aliquot of 9 kg juice was evaporated under vacuum and when the required Brix was achieved, 1% salt and distilled water were added to adjust the Brix range to 12–13 °. The puree was then heated to 82–88 °C and sealed into cans before being placed into a boiling bath for 15 minutes at 96–100 °C. Cans were then cooled and stored frozen prior to analysis.

For paste, a 9 kg aliquot of juice was evaporated under vacuum until the desired Brix range was achieved, 0.5% salt and distilled water were added to adjust the Brix range to 24–33 °. The paste was then heated 82–88 °C and sealed into cans before being placed into a boiling bath for 15 minutes at 96–100 °C. Cans were then cooled and stored frozen prior to analysis.

The LOQ of the method is 0.01 mg/kg except for TA in purée (0.02 mg/kg) and paste (0.03 mg/kg).

Fresh tomatoes, purée and paste were analysed for residues of flutriafol and triazole metabolites T, TA and TAA using a validated analytical method. Results showed an increase in flutriafol residues in puree with a PF of 1.2 and an increase in residues in paste with a PF of 3.6. No residues of T, TA or TAA were present above LOQ in any control or treated samples analysed.

Table 58 Residues of flutriafol in tomato processed fractions following processing of fruit (Carringer 2012 2440)

Location	n	g ai/ha	g ai/hL	DALA	Sample	Residue (mg/kg)	PF
Porterville, CA, USA, 2011 Roma VF	5			0	RAC	0.55	

Location	n	g ai/ha	g ai/hL	DALA	Sample	Residue (mg/kg)	PF
99 kg batch					Purée	0.64	1.2
					Paste	1.98	3.6

Head lettuce

Three processing trial have been conducted on head lettuce in the USA in 2011 (Carringer 2013 2698). Four applications of flutriafol were made, formulated as a 125 g/L SC using a backpack or tractor-mounted boom sprayer. All applications were made at a nominal rate of 128 g ai/ha. Applications were made with a 7 day interval with the final application being made 7 days before harvest. Mature head lettuce (RAC) and samples prepared for consumption (SPFC) were transported frozen to the analytical facility for analysis.

The head lettuce for the SPFC samples were visually examined and any damaged or wilted leaves, as well as wrapper leaves, removed. Each head was rinsed under cold running tap water for 15 to 20 seconds and allowed to drain top side down for at least two minutes.

The control, RAC and SPFC samples were placed in frozen storage within 3.17 hours after collection from the field and maintained frozen during transportation to the analytical laboratory. Samples were analysed using validated analytical methods. The LOQ of the methods is 0.01 mg/kg for all analytes.

RAC samples and SPFC samples were all analysed for residues of flutriafol and triazole metabolites using a validated analytical method. PFs for flutriafol range from 0.03 to 0.4 (mean of 0.21). Flutriafol does not concentrate in processed commodities.

Table 59 Residues of flutriafol in head lettuce following household processing of plants(Carringer 2013 2698) (means of duplicate samples)

Location	DALA	Sample	Residue (mg/kg)		PF
			Flutriafol	TA	
Germansville, PA, USA, 2011 Ithaca	7	RAC/ Heads	0.05	0.01	-
		SPFC/ Heads	0.02	0.01	0.4
King City, CA, USA, 2011 Venus	7	RAC/ Heads	0.05	< 0.01	-
		SPFC/ Heads	< 0.01	< 0.01	0.2
Arroyo Grande, CA, USA, 2011 Vandenberg	7	RAC/ Heads	0.67	0.03	-
		SPFC/ Heads	0.02	0.01	0.03

PF = for flutriafol residues only

SPFC = samples prepared for consumption

Sugar beet

In a processing study conducted on sugar beet in the USA (Jones 2009 1812) three applications of flutriafol (SC formulation) were made at a nominal rate of 640 g ai/ha with a 14 day interval and the final application 14 days before harvest. Mature sugar beet roots were transported at ambient temperature to the processing facility where they were processed into refined sugar, molasses and dry pulp samples.

Sugar beets (45.4 kg batch) were cleaned prior to processing by washing with a brush and water thereby removing excess soil, loose leaves and other debris. Cleaned beets were then sliced in a Hobart food cutter and the slices (cossettes) were first exposed to 88.5–93 °C water for 30–45 seconds (only) and then diffused in five kettles in a 69–74.5 °C water bath for a minimum of 9 minutes. After diffusion the raw juice was screened with a US#100 standard sieve to remove small pieces of beet from the juice.

Diffused cossettes were then dewatered with a FMC pulper/finisher. Beet pulp was produced by drying the dewatered material in a Steelman Industries oven at 55–72 °C for final moisture of 15% or less. Juice from dewatering was screened with the 100 mesh sieve and combined with juice from diffusion. The resulting fraction from this step is dried beet pulp.

During the first phosphatisation step, raw juice was mixed and the temperature increased to 81–86 °C. 20% calcium oxide solution and if required 3 M phosphoric acid was added until a pH of around 10.5 was achieved resulting in a precipitate. The sample was centrifuged to separate the precipitate from the juice.

During the second phosphatisation step, the juice was mixed and the temperature increased to 81–86 °C and pH reduced using 3 M phosphoric acid to around 9.1–9.3. The juice was then centrifuged and vacuum filtered to separate precipitate from the clear juice (thin juice). The juice was light yellow to light brown in colour. The thin juice was mixed and heated to 81–86 °C and pH reduced to 8.8–9.0 with sodium bisulphite.

The juice was evaporated under vacuum until the juice was 50–60% solids (thick juice) during which time the temperature was maintained below 86 °C). After evaporation the thick juice was filtered through cotton.

Evaporation continued under vacuum until the juice was 70–80% solids (syrup). Commercially available white cane sugar was added to the juice (seeding) after which crystallisation began.

The solution was allowed to cool after which the sugar and molasses were separated by centrifuging in a Western States basket centrifuge with filter basket. Steam was added to remove residual molasses from crystallised sugar. After removing the molasses the refined white sugar could be dried if necessary in a Steelman Industry oven at 55–72 °C to achieve a final moisture content of 10%. Samples did not require drying. The resulting fraction from this step is refined sugar and molasses.

Untreated and treated samples of sugar beet, refined sugar, molasses and beet pulp were stored frozen and shipped under frozen conditions to the analytical laboratory for analysis. Samples were analysed using a validated analytical method. The LOQ of the method is 0.01 mg/kg.

Sugar beet roots, refined sugar, molasses and dry pulp samples were all analysed for residues of flutriafol and triazole metabolites using a validated analytical method. Residues were < 0.01 mg/kg in the RAC and the processed commodities with the exception of TA being observed in both untreated and treated molasses samples at 0.02 mg/kg. It is therefore concluded that flutriafol does not concentrate in refined sugar, molasses or dry pulp.

Celery

Three processing trial have been conducted on celery in the USA in 2011 (Carringer 2013 2698). Four applications of flutriafol SC formulation were made at a nominal rate of 128 g ai/ha.

The celery heads for the SPFC samples were prepared by removing the inedible portion of the stalk (i.e. the woody part at the base of the stalk) to separate the stems. The leaves were not removed unless discoloured or damaged. The stems were then rinsed under cold running tap water for approximately 15–20 seconds and allowed to drain for at least 2 minutes.

The control, RAC and SPFC samples were placed in frozen storage within 3.17 hours after collection from the field and maintained frozen during transportation to the analytical laboratory. Samples were analysed using validated analytical methods. The LOQ of the methods is 0.01 mg/kg for all analytes.

Mature celery (RAC) and samples prepared for consumption (SPFC) were transported frozen to the analytical facility for analysis.

RAC samples and SPFC samples were all analysed for residues of flutriafol and triazole metabolites using a validated analytical method. PFs for flutriafol ranging from 0.73 to 1.24 (mean of 0.9) indicates that flutriafol does not concentrate significantly in celery processed commodities.

Table 60 Residues of flutriafol in celery following household processing of plants(Carringer 2013 2698) (means of duplicate samples)

		g		GS	DALA	Crop	Residue (mg/kg)			
Location, year, variety	No	ai/ha	L/ha	(BBCH)		part	Flutriafol	T	TA	PF
Sparta, MI, USA, 2012 Greenbay	4 (7 6 8)	129 128 128 128	46 47 46 46	45 46 47 48	7	Plant	0.73	0.06	< 0.01	
						SPCF	0.53	0.04	< 0.01	0.73
Porterville, CA, USA, 2012 Mission	4 (7 7 6)	129 128 129 127	365 365 365 365	44 46 46 48	7	Plant	1.08	< 0.01	0.02	
						SPCF	1.34	< 0.01	0.02	1.2
Guadalupe, CA, USA, 2011 Conquistador	4 (6 7 6)	128 129 129 128	271 262 271 271	45 46 47 48	8	Plant	0.77	0.04	0.06 c0.03	
						SPCF	0.57	0.03	0.05	0.74

PF = for flutriafol residues only

SPFC = samples prepared for consumption

Maize

Processing trials were conducted on field corn in the USA (Carringer 2010 1810). Two applications of flutriafol, formulated as a SC, were made at 128 g ai/ha and samples of mature field corn grains were used for generation of aspirated grains fractions (AGF). Additionally at one trial, applications were made at an exaggerated rate of 640 g ai/ha/application and samples from this site were processed into grits, meal, flour, starch and refined oil (wet and dry milled). At all sites applications were made with a 7 day interval with the final application being made 7 days before harvest. Mature corn grain were transported frozen to the processing facility and stored frozen until processing. Field corn grains samples were dried at 43–57 °C until the moisture content was 9–15%.

Generation of aspirated grain fractions (AGF)

To generate AGF, dried field corn grain samples were placed in a dust generation room containing a holding bin, two bucket conveyors and a screw conveyor. As the samples were moved in the system, aspiration was used to remove light impurities (grain dust). The grain dust was sieved for classification before being recombined for analysis.

Refined oil, dry milling process.

In preparation for processing field corn grain into refined oil utilising the dry milling process, samples of dried field corn grains were cleaned by aspiration and screening. Light impurities were removed by aspiration after which samples were screened to separate large and small foreign particles (screenings) from the field corn. The dried and cleaned samples were then moisture conditioned to 21% and fed into a mill to crack the kernels. Cornstock from the mill was dried in an oven for 30 minutes at 54–71 °C and screened with a 3.2 mm screen to separate bran, germ and large grits from grits, meal and flour.

Material below 3.2 mm was separated into grits, meal and flour using a sieve fitted with two screens of different sizes. Material greater than 3.2 mm was by means of screening, aspiration and milling (if necessary) separated into grits, meal, flour and germ.

Germ material was heated to 72–80 °C and flaked in a flaking roll. The flakes were then placed in batch extractors and submerged in 49–60 °C hexane. The crude oil/hexane mixture was drained and the extraction process repeated twice more with fresh hexane. After extraction the

spent flakes were air dried to produce solvent extracted germ meal. The crude oil/hexane was passed through an evaporator to separate the crude oil from the hexane and then crude oil was heated to remove residual hexane before being filtered and refined. Crude oil and sodium hydroxide were mixed for 15 minutes at high RPM at approximately 20 °C and then for 12 minutes at low RPM at approximately 63–68 °C. The neutralised oil was centrifuged and the refined oil decanted and filtered.

Refined oil, wet milling process

A sample of dried and cleaned corn was steeped in 49–54 °C water containing 0.1–0.2% sulphur dioxide for 22–48 hours. The whole corn was then passed through a disc mill and the majority of the germ and hull was removed using a water centrifuge. Germ and hull were dried and separated using aspiration and screening.

Cornstock (without germ and hull) ground in the disc mill was passed over a 50µm screen where only bran was retained. The process water passing through the screen was separated into starch and gluten by centrifugation. Starch was dried in a dehydrator oven at 54–71 °C until moisture content was less than 15.0%.

The dried germ samples were moisture conditioned to 12%, heated to 88–104 °C in a mixer, flaked in a flaking roll and pressed in an expeller to liberate part of the crude oil (expelled crude oil). Residual crude oil was extracted from the presscake utilising the batch extractors submerged in hexane at 49–54 °C. The extraction procedure was repeated twice more with fresh hexane. The crude oil/hexane was passed through an evaporator to separate the crude oil from the hexane and then crude oil was heated to remove residual hexane before being filtered and refined. Crude oil and sodium hydroxide were mixed for 15 minutes at high RPM at approximately 20 °C and then for 12 minutes at low RPM at approximately 63–68 °C. The neutralised oil was centrifuged and the refined oil decanted and filtered.

Untreated and treated samples of from the processes were stored frozen and shipped under frozen conditions to the analytical laboratory for analysis. Samples were analysed using validated analytical methods. The LOQ of the methods is 0.01 mg/kg for flutriafol and its metabolites T and TAA. For TA the LOQ was 0.01 mg/kg in all matrices except grits (0.15 mg/kg), field corn grains (0.03 mg/kg), meal 0.034 mg/kg, flour (0.034 mg/kg) and AGF (0.1 mg/kg), where endogenous residues of TA resulted in LOQs higher than the target LOQ of 0.01 mg/kg.

Corn grains, AGF, grits, meal, flour, starch and refined oils were all analysed for residues of flutriafol and the triazole metabolites T, TA and TAA. Results show an increase in residues in meal, flour and oil (wet and dry milled), AGF. PFs range from > 4 for AGF, 3 for meal flour and oil and < 1 for grits and starch.

Table 61 Residues of flutriafol in maize processed fractions following processing of grain (Carringer 2010 1810)

Location, year,		kg		Crop	Residue (mg/kg)			
variety	No	ai/ha	DALA	part	Flutriafol	TA	TAA	PF
Carlyle Illinois USA		1.28	7	Grain	< 0.01	< 0.01	0.07	
2009 8G23				Grits	< 0.01	< 0.01	< 0.01	
				Meal	< 0.01	< 0.01	0.05	
232 kg batch				Flour	< 0.01	< 0.01	0.07	
milling				Refined oil (dry milling)	0.01	< 0.01	< 0.01	
				Starch	< 0.01	< 0.01	< 0.01	
				Refined oil (wet milling)	0.01	< 0.01	< 0.01	
299 kg batch				Grain	< 0.01	0.07	< 0.01	
306 kg batch				AGF	0.04	< 0.1	< 0.01	

%moisture: pre-processing 30%, AGF 9.8%, grits 16.6%, meal 18.0%, flour 17.6%, starch 7.0%

PF = flutriafol only

Rice

Four processing trial have been conducted on rice in Spain in 2006 (Gimeno 2007 1630). Two applications of flutriafol were made, formulated as a 125 g/L SC formulation using sprayer equipment typical of broadcast application. Applications were made at nominally 187.5 g ai/ha/application with a 14 day interval with the final application being made 14 days before harvest. Mature paddy rice were used for generation of husked (brown) rice and polished (white) rice.

At harvest plants were cut down and left to dry in a threshing floor, grains were then separated from straw and paddy rice samples obtained. The paddy rice was further dried and was then passed through a machine which removed the husks to obtain husked rice. The husked rice was fed into a mill where a set of huller reels removed the germ, outer bran and the waxy cuticle producing polished rice.

All samples were frozen immediately after processing and transported to the analytical facility. Samples were analysed for residues of flutriafol using a validated analytical method. See earlier table.

Sorghum

One processing trial has been conducted on grain sorghum in the USA in 2012 (Carringer 2013 2699). Two applications of flutriafol were made, formulated as a 125 g/L SC using sprayer equipment typical of broadcast application. Applications were made at the maximum use rate of nominally 128 g ai/ha/application with a 7 day interval with the final application being made 30 days before harvest. Mature grain sorghum grains were used for generation of aspirated grains fractions (AGF). Mature grain sorghum grain were transported frozen to the processing facility.

To generate AGF, dried field corn grain samples were placed in a dust generation room containing a holding bin, two bucket conveyors and a screw conveyor. As the samples were moved in the system, aspiration was used to remove light impurities (grain dust). The grain dust was sieved for classification before being recombined for analysis.

Untreated and treated samples from the processes were stored frozen and shipped under frozen conditions to the analytical laboratory for analysis. Samples were analysed using validated analytical methods. The LOQ of the methods is 0.01 mg/kg for flutriafol and its metabolites T, TA and TAA.

Residues are higher in AGF compared to grain with a processing factor of 8. The triazole metabolites T, TA and TAA are not concentrated in during processing into AGF.

Table 62 Residues of flutriafol in sorghum processed commodities following cleaning of grain harvested from a treated crop (Carringer 2013 2699) (duplicate samples)

Location, year, variety	No	g ai/ha	Run	DALA	Crop part	Flutriafol	TA	TAA	PF
York, NE, USA, 2012 85G01	2 (7)		1	31	Grain	0.39	0.06	0.04	
308 kg batch 1					AGF	2.78	0.03	0.04	7.1
310 kg batch 2			2		Grain	0.38	0.06	0.04	
					AGF	3.38	0.03	0.04	8.9

PF = flutriafol only

Cotton

One processing trial has been conducted on cotton in the USA in 2012 (Carringer 2013 2700).

The plot received one T-band application of flutriafol 125 g/L SC formulation at 290 g ai/ha/application at planting applied using a commercial tractor mounted T-band sprayer.

The T-band application was followed by two foliar applications at 5× rate (640 g ai/ha/application) 37 and 30 days before harvest applied using a CO_2 backpack sprayer. Seed cotton was ginned on the same day as harvest resulting in undelinted seeds with approximately 11–15% remaining lint. Undelinted cotton seeds were transported frozen to the processing facility and processed into meal, hulls and refined oil.

Delinting (Mechanical)

The undelinted cottonseed samples (41 kg) were saw delinted in a delinter to remove most remaining lint producing delinted cottonseed with approximately 3% lint remaining on the seed.

Hulling and separation

Delinted cottonseed was mechanically cracked in a roller mill. Kernel and hull material was separated with a careen cleaner.

Kernel material moisture was determined and then adjusted to 13.5% by placing the kernel material in a rotating mixer and adding water.

Oil and meal production

Kernel material was heated in a steam heated mixer to 79.4–90.6 °C and held for 30 minutes. After heating, kernel material was flaked in a flaking roll. Flaked kernel material was then fed into an expander. As the material moved through the expander, steam was injected directly on the product. Maximum exiting temperature range of the material was 93.3–121.1 °C. Collets were ground, dried in an oven at 65.6–82.2 °C for 30–40 minutes.

Ground collets were placed in batch extractors and submerged in 49–60 °C hexane. After 30 minutes the hexane/crude oil mixture was drained and extraction repeated three more times with fresh hexane.

After extraction the solvent extracted meal was toasted in a steam jacketed paddle mixer with steam injected directly on the material until the temperature of the meal reached 101.7–104.4 °C. Steam injection was stopped and the meal heated to 104.4–115.6 °C and held for 45–60 minutes. After toasting, the meal was cooled to room temperature.

The crude oil/hexane was passed through an evaporator to separate the crude oil from the hexane and then crude oil was heated to remove residual hexane before being filtered and refined.

Alkali refining, bleaching and deodorisation

Crude oil and sodium hydroxide was mixed for 15 minutes at high RPM at approximately 20 °C and then for 13 minutes at low RPM at approximately 63–68 °C. The neutralised oil was centrifuged and the refined oil decanted and filtered.

The refined oil was bleached by heating it to 40–50 °C and adding an activated bleaching earth. The mixture was placed under vacuum, heated to 85–100 °C and held there for 10–15 minutes. Heating was stopped and the oil was allowed to cool. During the cooling phase vacuum was broken, filter aid added and vacuum resumed. When the mixture reached approximately 60 °C vacuum was broken and the bleached oil filtered.

The blanched oil was then deodorised by steam bathing for approximately 30 minutes under vacuum at 220–230 °C. During the following cooling period 0.5% citric acid solution was added.

Untreated and treated samples from the processes were stored frozen and shipped under frozen conditions to the analytical laboratory for analysis. Samples were analysed using validated analytical methods. The LOQ of the methods is 0.01 mg/kg for flutriafol and its metabolites T and TAA. For TA the LOQ was 0.01 mg/kg in all matrices except for TA in undelinted

cottonseed and cottonseed meal, where the LOQs were 0.03 and 0.04 mg/kg respectively due to endogenous residues in available control samples.

Undelinted cotton seeds, meal, hulls and refined oil were all analysed for residues of flutriafol using a validated analytical method. Residues of flutriafol in undelinted cotton seeds were present at 0.12 mg/kg. Residues were all lower in the processed commodities ranging from < 0.01 mg/kg in refined oil to 0.04 mg/kg in hulls. Results indicates, that flutriafol does not concentrate during processing into refined cottonseed oil.

Table 63 Residues of flutriafol in cotton processed products (meal, hulls, oil) on processing seed from a treated crop (Carringer 2013 2700) (duplicate samples)

Location	N	g ai/ha	DALA	Sample	Residue (mg/kg) Flutriafol	TA	PF
Uvalde TX, USA, 2012 DP 0912 B2RF 40.9 kg batch	3		30	Undelinted Seed	0.12 0.12	0.13 0.10	
				Meal	0.01 0.01	0.14 0.19	0.08
				Hulls	0.04 0.03	0.04 0.07	0.33
				Refined oil	< 0.01 < 0.01	< 0.01 < 0.01	0.08

Meal 9.4% moisture

Hulls 9.4% moisture.

PF = flutriafol only

Livestock feeding

A livestock feeding study has been conducted in Holstein dairy cows to determine the magnitude of residues of flutriafol and three triazole metabolites 1,2,4-triazole (T), triazole alanine (TA) and triazole acetic acid (TAA) in milk, muscle, liver, kidney and fat (Rice 2012 2479). Three groups of three Holstein cows (3–7 years old, 450–690 kg bw) cows (three additional cows used for depuration phase) plus two concurrent control cows were dosed at 0, 5, 16 and 50 ppm (equivalent to 0, 0.15, 0.45 and 1.59 mg/kg bw of flutriafol) once daily for 28 consecutive days. Average feed consumption for the 5, 16 and 50 ppm groups were 18.5, 17.7 and 17.9 kg/day. Average milk production was 25.6, 22.0 and 21.3 L/d respectively for the 5, 16 and 50 ppm dose groups. Milk was collected twice daily and samples at 0, 3, 7, 10, 14, 17, 21, 24, 26 and 28 days were pooled and mixed before analysis. All cows were sacrificed within 24 hours after final dosing and samples of muscle (composite of round and loin), liver, kidneys, fat (renal, omental and subcutaneous fat deposits) were collected for analysis. Residues of flutriafol and triazole metabolites were analysed using validated analytical methods with an LOQ of 0.01 mg/kg for each analyte/matrix combination.

Highest average residues of flutriafol were found in liver and ranged from 0.33 mg/kg for the 5 ppm group, 0.59 mg/kg for the 16 ppm group and 1.83 mg/kg for the 50 ppm group. No residues were observed in liver samples taken from the depuration phase at 31, 35 and 42 days. For remaining matrices, highest average flutriafol residues ranged from < 0.01 mg/kg in milk, 0.01 mg/kg (50 ppm group) in cream at day 21, < 0.01 mg/kg in skimmed milk, 0.096 mg/kg (50 ppm group), 0.01 mg/kg (16 ppm group) in kidney, 0.04 mg/kg (50 ppm group) in muscle and 0.07–0.195 mg/kg (50 ppm group), 0.01 mg/kg (16 ppm group) in fat. All other residues of flutriafol from all dose groups were < 0.01 mg/kg. No residues were observed above LOQ in tissue or milk samples taken from the depuration phase at 31, 35 and 42 days.

Highest average residues of triazole metabolite residues were found in liver and ranged from < 0.01–0.02 mg/kg for 1,2,4-triazole, 0.03 to 0.157 mg/kg for triazole alanine and < 0.01 mg/kg for triazole acetic acid. Only triazole alanine residues were found during the depuration phase and ranged from 0.093 to 0.135 mg/kg. For remaining matrices, highest average residues ranged from 0.020 mg/kg 1,2,4-triazole in milk (50 ppm group), 0.015 mg/kg 1,2,4-triazole (50 ppm group) in cream at day 14/21, 0.021 mg/kg 1,2,4-triazole in skimmed milk (50 ppm group), 0.029 mg/kg 1,2,4-triazole and 0.058 mg/kg triazole alanine (50 ppm group) in kidney, 0.020 mg/kg 1,2,4-triazole and 0.086 mg/kg triazole alanine (50 ppm group) in muscle

and 0.02 mg/kg triazole alanine (50 ppm group) in fat. No average residues of triazole acetic acid were observed in tissue or milk samples. Only triazole alanine was observed above LOQ in tissues during the depuration phase

Table 64 Recovery data

Tissue matrix	Analyte	Fortification range	Recovery (%)		n
		(mg/kg)	Range	Mean	
Milk	Flutriafol	0.01–0.1	68–115	92	26
	T	0.01–0.1	70–103	90	32
	TA	0.01–0.1	86–119	101	30
	TAA	0.01–0.1	70–119	106	30
Cream	Flutriafol	0.01–0.1	72–95	81	8
	T	0.01–0.1	89–104	96	10
	TA	0.01–0.1	89–105	98	8
	TAA	0.01–0.1	73–124	105	8
Skim milk	Flutriafol	0.01–0.1	74–93	84	6
	T	0.01–0.1	86–101	93	12
	TA	0.01–0.1	91–109	100	8
	TAA	0.01–0.1	78–120	100	8
Liver	Flutriafol	0.01–2.0	99–120	110	6
	T	0.01–0.1	70–98	85	6
	TA	0.01–0.3	95–105	99	6
	TAA	0.01–0.1	96–114	106	6
Kidney	Flutriafol	0.01–0.3	91–120	98	8
	T	0.01–0.1	91–109	97	8
	TA	0.01–0.1	87–113	99	8
	TAA	0.01–0.1	95–118	108	8
Muscle	Flutriafol	0.01–0.1	83–120	99	6
(Round)	T	0.01–0.1	76–119	92	8
	TA	0.01–0.3	94–104	97	6
	TAA	0.01–0.1	97–118	106	6
Muscle	Flutriafol	0.01–0.3	75–116	98	6
(Loin)	T	0.01–0.1	75–102	90	8
	TA	0.01–0.3	84–98	92	8
	TAA	0.01–0.1	75–108	95	8
Fat	Flutriafol	0.01–3.0	66–120	95	6
(Omental)	T	0.01–0.1	71–107	91	8
	TA	0.01–0.1	93–99	96	6
	TAA	0.01–0.1	98–108	103	6
Fat	Flutriafol	0.01–3.0	72–89	80	6
(Renal)	T	0.01–0.1	86–100	94	6
	TA	0.01–0.1	93–107	99	6
	TAA	0.01–0.1	87–117	104	6
Fat	Flutriafol	0.01–3.0	76–103	87	6
(Subcutaneous)	T	0.01–0.1	83–108	96	6
	TA	0.01–0.1	96–116	108	6
	TAA	0.01–0.1	89–111	103	6

Table 65 Residues of flutriafol and triazine metabolites in milk

	Flutriafol		1,2,4 Triazole		Triazole Alanine	
	Range	Average	Range	Average	Range	Average
5 ppm						
–1	n/a	n/a	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
3	n/a	n/a	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
7	n/a	n/a	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
10	n/a	n/a	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
14	n/a	n/a	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
17	n/a	n/a	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
21	n/a	n/a	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
24	n/a	n/a	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01

	Flutriafol		1,2,4 Triazole		Triazole Alanine	
	Range	Average	Range	Average	Range	Average
26	n/a	n/a	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
28	n/a	n/a	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
16 ppm						
–1	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
3	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
7	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
10	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
14	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
17	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
21	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
24	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
26	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
28	< 0.01–< 0.01	< 0.01	< 0.01–0.01	< 0.01	< 0.01–< 0.01	< 0.01
50 ppm						
–1	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
3	< 0.01–< 0.01	< 0.01	0.02–0.02	0.02	< 0.01–< 0.01	< 0.01
7	< 0.01–< 0.01	< 0.01	0.01–0.03	0.02	< 0.01–< 0.01	< 0.01
10	< 0.01–< 0.01	< 0.01	0.01–0.03	0.02	< 0.01–< 0.01	< 0.01
14	< 0.01–< 0.01	< 0.01	0.01–0.02	0.02	< 0.01–< 0.01	< 0.01
17	< 0.01–< 0.01	< 0.01	0.01–0.02	0.01	< 0.01–< 0.01	< 0.01
21	< 0.01–< 0.01	< 0.01	0.01–0.02	0.02	< 0.01–< 0.01	< 0.01
24	< 0.01–< 0.01	< 0.01	0.01–0.02	0.02	< 0.01–< 0.01	< 0.01
26	< 0.01–< 0.01	< 0.01	< 0.01–0.02	0.02	< 0.01–< 0.01	< 0.01
28	< 0.01–< 0.01	< 0.01	0.01–0.03	0.02	< 0.01–< 0.01	< 0.01
28dep	< 0.01–< 0.01	< 0.01	< 0.01–0.02	0.01	< 0.01–< 0.01	< 0.01
31dep	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
35dep	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
42dep	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01

n/a = Sample not analysed

Table 66 Partitioning of residues of flutriafol and triazine metabolites between cream and skim milk

	Flutriafol		1,2,4 Triazole		Triazole Alanine	
	Range	Average	Range	Average	Range	Average
5 ppm						
14 (Cream)	n/a	n/a	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
21 (Cream)	n/a	n/a	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
14 (Skim)	n/a	n/a	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
21 (Skim)	n/a	n/a	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
16 ppm						
14 (Cream)	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
21 (Cream)	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
14 (Skim)	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
21 (Skim)	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
50 ppm						
14 (Cream)	< 0.01–0.0155	< 0.01	0.0110–0.0206	0.0146	< 0.01–< 0.01	< 0.01
21 (Cream)	< 0.01–0.0144	0.0106	0.0107–0.0198	0.0146	< 0.01–< 0.01	< 0.01
14 (Skim)	< 0.01–< 0.01	< 0.01	0.0154–0.0245	0.0211	< 0.01–< 0.01	< 0.01
21 (Skim)	< 0.01–< 0.01	< 0.01	0.0156–0.0267	0.0216	< 0.01–< 0.01	< 0.01

Table 67 Residues of flutriafol and triazine metabolites in tissues

	Flutriafol		1,2,4 Triazole		Triazole Alanine	
	Range	Average	Range	Average	Range	Average
5 ppm						
Liver	0.27–0.44	0.33	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
Kidney	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	0.01–0.02	0.01
Round	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	0.01–0.02	0.02
Loin	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–0.01	< 0.01

	Flutriafol		1,2,4 Triazole		Triazole Alanine	
	Range	Average	Range	Average	Range	Average
Omental	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
Renal	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
Subcutaneous	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
16 ppm						
Liver	0.23–0.77	0.59	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
Kidney	< 0.01–0.02	0.01	< 0.01–0.02	< 0.01	0.01–0.03	0.02
Round	< 0.01–< 0.01	< 0.01	< 0.01–0.01	< 0.01	0.01–0.03	0.02
Loin	< 0.01–< 0.01	< 0.01	< 0.01–0.01	< 0.01	< 0.01–0.02	0.01
Omental	< 0.01–0.02	0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
Renal	< 0.01–0.02	0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
Subcutaneous	< 0.01–0.02	0.01	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01
50 ppm						
Liver	1.64–1.95	1.83	0.01–0.02	0.02	0.13–0.19	0.16
Kidney	0.04–0.15	0.10	0.02–0.03	0.03	0.05–0.07	0.06
Round	0.02–0.06	0.04	0.01–0.03	0.02	0.08–0.10	0.09
Loin	0.02–0.07	0.04	0.01–0.03	0.02	0.04–0.06	0.05
Omental	0.08–0.34	0.19	< 0.01–0.01	< 0.01	< 0.01–0.01	< 0.01
Renal	0.07–0.32	0.18	< 0.01–< 0.01	< 0.01	< 0.01–0.01	< 0.01
Subcutaneous	0.04–0.11	0.07	< 0.01–0.02	< 0.01	0.01–0.03	0.02
Depuration						
31–42 Liver	< 0.01–< 0.01	< 0.01	0.02–0.02	0.02	0.09–0.14	0.11
31–42 Kidney	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	0.04–0.05	0.04
31–42 Round	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	0.04–0.05	0.05
31–42 Loin	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	0.03–0.04	0.03
31–42 Omental	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–0.01	< 0.01
31–42 Renal	< 0.01–< 0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–0.01	< 0.01
31–42 Subcutaneous	< 0.01–0.01	< 0.01	< 0.01–< 0.01	< 0.01	< 0.01–0.02	0.01

Note: residues of triazole analine were detected in muscle (loin and round) samples from control animals: The levels detected were < 0.01–0.01, mean < 0.01 mg/kg in round and 0.08–0.09 mg/kg, mean 0.09 mg/kg in loin muscle. The large difference between loin and round residues as well as the fact that no residues of TAA were detected in corresponding control liver, kidney or fat samples suggesting this detection is due to a mislabelling of the sample or cross-contamination during processing for analysis.

APPRAISAL

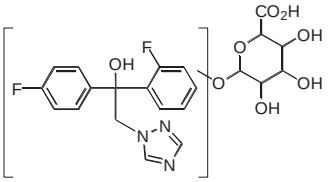
Flutriafol is a triazole fungicide used in many crops for control of a broad spectrum of leaf and ear cereal diseases, particularly embryo borne diseases e.g., bunts and smuts. It was first evaluated for residues and toxicology by the 2011 JMPR. The ADI of flutriafol was 0–0.01 mg/kg bw and the ARfD was 0.05 mg/kg bw. The compound was listed by the Forty-sixth Session of CCPR for the JMPR to consider additional MRLs. The residue definition for compliance with MRL and for estimation of dietary intake (for animal and plant commodities) is flutriafol.

For the current evaluation the Meeting received new metabolism studies in lactating goats, storage stability data for animal commodities, residue trials on apples, pears, peaches/nectarines, plums, cherries, strawberries, Brassica vegetables (cabbage and broccoli), cucurbits (cucumbers, summer squash and muskmelons), tomatoes, peppers, leafy vegetables (lettuce, spinach, celery and mustard greens), sugar beet, maize, rice, sorghum, almonds, pecans, cotton, and rape, as well as a lactating cow feeding study (residue transfer study).

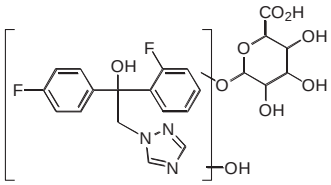
Metabolites referred to in the appraisal were addressed by their common names

1,2,4-triazole (M1, T)

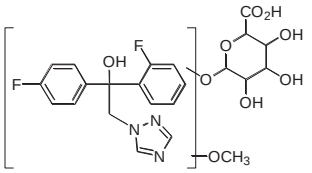
flutriafol glucuronide (M4)



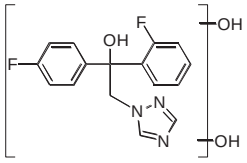
hydroxy flutriafol glucuronide (M3)



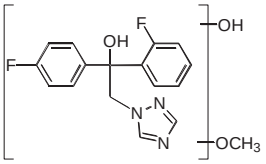
methoxy flutriafol glucuronide (M7)



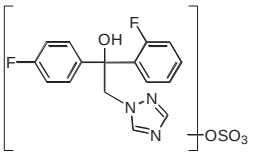
dihydroxy flutriafol (M3e)



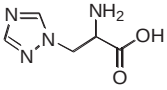
hydroxymethoxy flutriafol (M5)



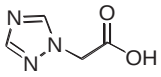
flutriafol sulfate (M10)



1,2,4-triazole analine (TA)



1,2,4-triazole acetic acid (TAA)



Animal metabolism

Metabolism of flutriafol in cattle involves hydroxylation of flutriafol to hydroxy flutriafol and a range of polar water soluble metabolites that are present at low levels, presumably additionally hydroxylated flutriafol compounds and their conjugates. The current Meeting received two additional studies on the metabolism of flutriafol in ruminants involving dosing lactating goats with triazole- or carbinol-labelled flutriafol at the equivalent of 12 or 30 ppm in the feed.

The majority of the ^{14}C residues were recovered in the excreta (urine 30–54% AD, faeces 35–55% AD). For tissues of goats dosed at 30 ppm, ^{14}C residues were highest in liver, (0.68–0.70 mg equiv/kg), followed by the kidney (0.11–0.31 mg equiv/kg) with only low levels detected in fat (0.011–0.018 mg equiv/kg) and muscle (0.02 mg equiv/kg). Residues in milk appeared to reach plateau levels by day three of dosing with significant differences in ^{14}C levels between milk collected in the morning (low levels) compared to evening milk (higher levels) suggesting flutriafol residues are rapidly eliminated following dosing. TRR in milk reached a maximum of 0.095 mg equiv/kg.

Acetonitrile and water extraction of liver, kidney, muscle, fat, skim milk and milk fat resulted in extraction efficiencies of 28.7–38.7% (liver), 66.7–86.5% (kidney) and > 82% (muscle), > 72% fat, 98% (skim milk) and 82–87% (milk fat).

Flutriafol was extensively metabolized and accounted for ≤ 2.5% TRR in liver, ≤ 0.7% TRR in kidney, ≤ 4.3% TRR in milk fat, not detected in muscle and ≤ 0.01 mg/kg in fat. Significant metabolites and the highest % TRR in tissues are 1,2,4-triazole (M1: 15% skim milk, 11% milk fat, 42% muscle, 27% fat), hydroxy flutriafol glucuronide (M3: 13% kidney, 23%

skim milk, 44% milk fat, 10% muscle), di-hydroxy flutriafol (M3e: 35% skim milk), flutriafol glucuronide (M4: 25% kidney, 17% muscle) and methoxy flutriafol glucuronide (M7: 10% kidney).

The Meeting noted that in the lactating cow evaluated by the 2011 JMPR, animals were dosed orally twice daily at the equivalent of 2 ppm in the diet for seven days and sacrificed at 4 hours after the last dose. In the current studies, goats were dosed once daily at 12 or 30 ppm with sacrifice occurring 20–22 hours after the last dose. The difference in sacrifice times and the higher dose rates have allowed for increased identification of residue components. The major residues in kidney, in both the lactating cow and goat studies, is flutriafol glucuronide (M4) (reported as M1B in the lactating cow study) at 22% TRR in cows and 13–15% TRR in goats at the highest dose. With the longer interval between the last dose and sacrifice, flutriafol is no longer found as the major component of the residue in liver (cow 27% TRR; goat 1.0–2.5% TRR) and no metabolite was individually present at > 10% TRR in liver in the goat studies. The levels of radioactivity in milk from the cow study were too low to allow for adequate characterisation and identification of components. In the goat study, considering the levels found in skim milk and in milk fat, three components are likely to be present at more than 10% TRR in whole milk: hydroxy flutriafol glucuronide (M3), di-hydroxy flutriafol (M3e) and flutriafol sulphate (M10).

The major metabolic pathway involves oxidation of one of the phenyl rings followed by conjugation with glucuronic acid to form flutriafol glucuronide (M4). Further oxidation results in formation of dihydroxy flutriafol (M3e), of which there are a number of possible isomers. M3e is then further transformed via methylation to hydroxyl methyl flutriafol (M5) which can, in turn, be conjugated with glucuronic acid to form methoxy flutriafol glucuronide (M7). M3e was also conjugated with glucuronic acid to form hydroxy flutriafol glucuronide (M3). The lactating goat study extends the knowledge of flutriafol metabolism and is consistent with earlier studies in lactating cow as well as laboratory animals.

The new goat metabolism studies have identified potential marker residues that could be included in the residue definitions for compliance and dietary intake risk assessment. However, the Meeting noted at the current livestock dietary burdens, residues in animal commodities of these components are expected to be at the limit of quantification or below. The Meeting agreed that the residue definitions for animal commodities did not need to be revised although this may change in the future if there are significant increases in the estimated livestock dietary burdens.

Stability of pesticide residues in stored analytical samples

The 2011 JMPR concluded that when stored, frozen flutriafol residues were stable for at least 5 months in soya bean seed, for at least 12 months in apple, barley grains and coffee beans, for at least 23 months in grapes, for at least 24 months in cabbage and oilseed rape, and for at least 25 months in wheat (grains and straw), pea seed, sugar beet root. Triazole metabolite residues were stable for at least 4 months in apple fruits and juice, and for at least 5 months in animal commodities.

The 2015 Meeting received information on the stability of flutriafol and triazole metabolites T, TA and TAA in samples of animal commodities stored frozen. Residues of flutriafol, TA and TAA in ruminant tissues (muscle, fat, liver and kidney) remain stable for at least 12 months, residues of T remains stable for at least 12 months in muscle and liver, and for a maximum 6.6 months in kidney and 10.7 months in fat when samples are stored under deep frozen conditions.

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

Results of supervised residue trials on crops

Pome fruit

Field trials involving apples and pears conducted in the USA were made available to the Meeting. The cGAP for pome fruit in the USA is four applications at 119 g ai/ha (7–10 day interval between sprays, PHI 14 days). None of the trials on apples and pears submitted matched cGAP. However, the number of sprays in the trials was six and available decline data suggest the additional two sprays do not significantly contribute to the final residues and trials conducted at the maximum application rate but with six sprays were considered to approximate cGAP.

Apples

Residues in trials evaluated by the 2015 JMPR approximating *c*GAP were (n=4): 0.02, 0.02, 0.06 and 0.11 mg/kg.

The 2011 JMPR reported residues from sixteen trials on apples that also approximated *c*GAP (n=16): 0.03, 0.04, 0.05 (3), 0.06 (3), 0.08 (2), 0.09, 0.10 (2), 0.12 (2) and 0.16 mg/kg.

Pears

Residues in trials on pears approximating *c*GAP were: 0.04, 0.09, 0.13, 0.18, 0.21 and 0.24 mg/kg.

The GAP in the USA is for the group Pome fruit. The median residues in apples and pears differed by less than a factor of five and the Meeting decided to recommend a group maximum residue level. In deciding which data set to use for the recommendation, as a Mann Whitney U-test indicated that the residue populations were not different it was decided to combine the data sets.

The combined apple and pear dataset is: 0.02 (2), 0.03, 0.04 (2), 0.05 (3), 0.06 (4), 0.08 (2), 0.09 (2), 0.10 (2), 0.11, 0.12 (2), 0.13, 0.16, 0.18, 0.21 and 0.24 mg/kg

The Meeting estimated a maximum residue level of 0.4 mg/kg for pome fruit together with an STMR of 0.08 mg/kg and an HR 0.26 mg/kg (highest individual analytical result from duplicate samples) and agreed to replace the previous recommendation of 0.3 mg/kg.

Stone fruit

Field trials involving applications to cherries, peaches and plums were made available from the USA.

The cGAP for stone fruit in the USA is four applications at 128 g ai/ha (maximum application per year 511 g ai/ha, 7day interval between sprays, PHI 7 days).

Residues in cherries (sweet and tart) from trials matching GAP were: 0.16, 0.24, 0.25, 0.26, 0.30, 0.30, 0.32, 0.33, 0.34, 0.38, 0.39, 0.40, 0.42, 0.46, 0.47 and 0.59 mg/kg.

Residues in peaches from trials matching cGAP were: 0.05, 0.12, 0.13, 0.14, 0.15, 0.16, 0.18, 0.18, 0.19, 0.24, 0.24 and 0.41 mg/kg

Residues in plums from trials matching cGAP were: 0.02, 0.03, 0.04, 0.06, 0.09, 0.10, 0.12 and 0.22 mg/kg.

The Meeting noted the use in the USA is for the group stone fruit and that a group MRL recommendation might be possible. Although the median residues differed by less than a factor of five, the Meeting decided to recommend maximum residue levels for all the sub-groups of stone fruit as there were sufficient trials available for each sub-group.

The Meeting estimated a maximum residue level of 0.8 mg/kg for the sub-group cherries together with an STMR of 0.335 mg/kg and an HR 0.66 (highest individual analytical result from duplicate samples) mg/kg.

The Meeting estimated a maximum residue level of 0.6 mg/kg for sub-group peaches together with an STMR of 0.17 mg/kg and an HR 0.42 (highest individual analytical result from duplicate samples) mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg for sub-group plums together with an STMR of 0.075 mg/kg and an HR 0.25 (highest individual analytical result from duplicate samples) mg/kg.

Strawberries

Trials were available from Spain and the USA. The cGAP for strawberries in the USA is four applications at 128 g ai/ha (maximum application per year 511 g ai/ha, 7 day interval between sprays, PHI 0 days).

Residues in strawberries from trials matching cGAP were (n=10): 0.14, 0.24, 0.30, 0.36, 0.42, 0.44, 0.45, 0.55, 0.63 and 0.72 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg for strawberries together with an STMR of 0.43 mg/kg and an HR 0.78 (highest individual analytical result from duplicate samples) mg/kg.

Brassica vegetables

Residue trials were available from the USA. The cGAP for Brassica (Cole) leafy vegetables in the USA is four applications 128 g ai/ha (maximum application per year 511 g ai/ha, 7 day interval between sprays, PHI 7 days). Residues in trials matching cGAP were cabbage (n=6) 0.08, 0.09, 0.10, 0.20, 0.44, 0.74 mg/kg and broccoli (n=5) 0.06, 0.08, 0.14, 0.18, 0.35 mg/kg.

The GAP in the USA is for the group Brassica vegetables. The median residues in cabbage and broccoli differed by less than a factor of five and the Meeting decided to recommend a group maximum residue level. In deciding which data set to use for the recommendation, as a Mann Whitney U-test indicated that the residue populations were not different it was decided to combine the data sets.

The combined data set is (n=11): 0.06, 0.08, 0.08, 0.09, 0.10, 0.14, 0.18, 0.20, 0.35, 0.44 and 0.74 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg for Brassica (Cole or cabbage) vegetables together with an STMR of 0.14 mg/kg and an HR 0.80 mg/kg (highest individual analytical result from duplicate samples).

Fruiting vegetables, cucurbits

Residue trials were available from the USA. The Meeting noted that there are GAPs in the USA that cover the whole group fruiting vegetables, cucurbits and that the cGAP is the same for all crops that are members of the group. It was agreed to consider the trials on melons and other cucurbits together. The cGAP for the muskmelons and cucurbit vegetables (except muskmelons) in the USA is four applications at 128 g ai/ha (maximum application per year 511 g ai/ha, 7 day interval between sprays, PHI 0 days).

Residues matching cGAP were muskmelons, whole fruit (n=8), 0.02, 0.04, 0.07, 0.08, 0.10, 0.10, 0.12 and 0.12 mg/kg (whole fruit); muskmelons, flesh (n=4), < 0.01, < 0.01, 0.02 and 0.02 mg/kg; cucumbers, (n=8), 0.02, 0.02, 0.03, 0.04, 0.04, 0.04, 0.06 and 0.06 mg/kg; summer squash, (n=7), 0.04, 0.04, 0.04, 0.05, 0.05, 0.06 and 0.06 mg/kg.

The GAP in the USA covers the whole group cucurbit vegetables. The median residues in cucumbers, muskmelons and summer squash datasets differed by less than a factor of five and the Meeting decided to recommend a group maximum residue level. In deciding which data set to use for the recommendation, as a Kruskal-Wallis H-test indicated that the residue populations were different it was decided to use the muskmelon dataset which has the highest residues.

The Meeting estimated a maximum residue level of 0.3 mg/kg for fruiting vegetables, cucurbits, together with an HR 0.13 mg/kg (highest individual analytical result from duplicate samples from muskmelons) and an STMR of 0.09 mg/kg.

Tomatoes

Flutriafol is approved in the USA for use on tomatoes. The cGAP for tomatoes in the USA is four applications at 128 g ai/ha (maximum application per year 511 g ai/ha, 7 day interval between sprays, PHI 0 days). Residues from trials matching cGAP were (n=18): 0.04, 0.05, 0.06, 0.06, 0.06, 0.06, 0.07, 0.08, 0.10, 0.12, 0.12, 0.12, 0.15, 0.18, 0.33, 0.40, 0.42 and 0.55 mg/kg.

The Meeting estimated a maximum residue level of 0.8 mg/kg for tomatoes together with an STMR of 0.11 mg/kg and an HR 0.63 (highest individual analytical result from duplicate samples) mg/kg.

Peppers

Residue trials were available from the USA. The cGAP for fruiting vegetables (USA group 8–10) which includes peppers in the USA is four applications at 128 g ai/ha (maximum application per year 511 g ai/ha, 7 day interval between sprays, PHI 0 days).

Residues in trials matching USA GAP were peppers, sweet (n=9), 0.03, 0.06, 0.06, 0.08, 0.10, 0.11, 0.14, 0.15 and 0.16 mg/kg, and chilli, (n=4), 0.12, 0.20, 0.26 and 0.31 mg/kg.

Residues in peppers and chilli, from trials submitted to the 2015 JMPR are covered by maximum residue levels recommended by the 2011 JMPR of 1 mg/kg for peppers, sweet however, the Meeting noted the commodity description from the 2011 JMPR should have been VO 0051 Peppers (subgroup including Peppers, Chilli and Peppers, Sweet) and not VO 0445 Peppers, Sweet (including pimento or pimiento). To resolve this Meeting recommends a maximum residue level of 1 mg/kg, STMR of 0.28 mg/kg and an HR of 0.41 mg/kg for peppers (VO 0051) to replace the previous recommendation of 1 mg/kg for peppers, sweet (VO 0445).

Leafy vegetables

Residue trials were available from the USA. The *c*GAP for leafy vegetables (except Brassica leafy vegetables) in the USA is four applications at 128 g ai/ha (maximum application per year 511 g ai/ha, 7 day interval between sprays, PHI 7 days). Brassica (Cole) leafy vegetables in the USA have the same *c*GAP as for other leafy vegetables and as mustard greens are considered leafy vegetables under Codex, the Meeting agreed to evaluate all leafy vegetables together.

Residues in trials matching *c*GAP were, head lettuce, (n=7), 0.04, 0.05, 0.14, 0.22, 0.46, 0.52 and 0.66 mg/kg; leaf lettuce, (n=5), 0.30, 0.32, 0.36, 1.45 and 2.64 mg/kg; Cos lettuce (Romaine), (n=2), 0.20 and 0.28 mg/kg; spinach, (n=8), 0.55, 0.94, 1.32, 1.55, 1.78, 2.1, 5.05 and 5.45 mg/kg; and mustard greens, (n=8), 1.20, 1.49, 2.02, 2.12, 2.12, 2.15, 2.78 and 3.42 mg/kg.

GAP in the USA is for leafy vegetables and a group maximum residue level recommendation may be possible. However, as the median residue levels in the datasets differed by more than 5×, residues in the individual commodities cannot be considered similar and the Meeting decided to recommend levels for the individual leafy vegetables for which data are available.

The Meeting estimated a maximum residue level of 1.5 mg/kg for head lettuce together with an STMR of 0.22 mg/kg and an HR 0.67 mg/kg (highest individual analytical result from duplicate samples).

The Meeting estimated a maximum residue level of 5 mg/kg for leaf lettuce together with an STMR of 0.36 mg/kg and an HR 2.95 mg/kg (highest individual analytical result from duplicate samples).

The Meeting agreed there were insufficient residue trials to estimate a maximum residue level for Cos lettuce.

The Meeting estimated a maximum residue level of 10 mg/kg for spinach together with an STMR of 1.665 mg/kg and an HR 5.5 mg/kg (highest individual analytical result from duplicate samples).

The Meeting estimated a maximum residue level of 7 mg/kg for mustard greens together with an STMR of 2.12 mg/kg and an HR 3.53 mg/kg (highest individual analytical result from duplicate samples).

The IESTI represented greater than 100% of the ARfD of 0.05 mg/kg bw in the case of leaf lettuce (110% children), mustard greens (350% children; 140% general population) and spinach (460% total or 160% raw spinach only, children; 130% general population). No alternative GAP was available.

Sugar beet

Residue trials were available from the countries of the EU and also the USA.

The *c*GAP for sugar beet in the USA is two applications at 128 g ai/ha (maximum application per year 256 g ai/ha, 14 day interval between sprays, PHI 21 days).

No trials matched cGAP as the number of sprays differed and there is insufficient data to conclude the additional spray does not significantly contribute to the terminal residue (three sprays in trials versus two sprays cGAP, PHI 14 day trials versus 21 days cGAP).

GAP in Russia is for two applications at 62.5 g ai/ha with a 30 day PHI. Residues in trials from northern Europe at approximately double the application rate were (n=8), < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, 0.01, 0.02 and 0.03 mg/kg. The Meeting decided to apply proportionality to the residue data.

Trial application rate (2nd spray) g ai/ha	Scaling factor = 62.5/trial application rate	Trial residue (mg/kg)	Scaled residue =scaling factor × trial residue (mg/kg)
135	0.463	< 0.01	< 0.01
111	0.563	< 0.01	< 0.01
120	0.521	< 0.01	< 0.01
131	0.477	< 0.01	< 0.01
138	0.453	< 0.01	< 0.01
126	0.496	0.01	0.0050
130	0.481	0.02	0.0096
138	0.453	0.03	0.0136

Based on the residues from Europe scaled to cGAP for Russia, the Meeting estimated an STMR of 0.01 mg/kg, an HR of 0.0136 mg/kg and a maximum residue level of 0.02 mg/kg for sugar beet.

Celery

Celery is classified as a leafy vegetable in the USA but as a stalk and stem vegetable in Codex. Residues in celery (whole plant) conducted according to cGAP in the USA (4× 128 g ai/ha, PHI 7 days) were (n=7), 0.44, 0.48, 0.73, 0.78, 0.92, 1.08 and 1.40 mg/kg.

The Meeting estimated a maximum residue level of 3 mg/kg for celery together with an STMR of 0.78 mg/kg and an HR 1.41 mg/kg (highest individual analytical result from duplicate samples).

Cereal grains

Maize

Residue trials were available from the USA. The cGAP for maize (field corn, popcorn and seed corn) in the USA is two applications at 128 g ai/ha (maximum application per year 256 g ai/ha, 7 day interval between sprays, PHI 7 days). Residues in trials matching cGAP were: < 0.01 (20) mg/kg. At

one site two applications were also made at an exaggerated rate of 640 g ai/ha with harvest of grain 7 days later. Residues in grain were < 0.01 mg/kg.

The Meeting estimated an STMR of 0 mg/kg and a maximum residue level of 0.01 (*) mg/kg for maize.

Rice

The Meeting received field trials performed in Italy on rice. The cGAP for Italy is for 2× 187.5 g ai/ha with a PHI of 28 days. In trials approximating critical GAP in the Italy total residues in rice grain (with husk) were (n=4), Paddy rice, 0.74, 1.06, 1.32 and 1.51 mg/kg.

The number of trials is insufficient to make a maximum residue level recommendation for rice.

Sorghum

Residue trials were available from the USA. The cGAP for sorghum in the USA is two applications at 128 g ai/ha (maximum application per year 256 g ai/ha, 7 day interval between sprays, PHI 30 days). Residues in trials matching *c*GAP were (n=12), 0.03, 0.16, 0.16, 0.20, 0.24, 0.26, 0.28, 0.34, 0.38, 0.40, 0.74 and 0.74 mg/kg.

The Meeting estimated an STMR of 0.27 mg/kg and a maximum residue level of 1.5 mg/kg for sorghum.

Tree nuts

Residue trials were available from the USA. The cGAP for almonds and walnuts as well as for pecans and other tree nuts in the USA is four applications at 128 g ai/ha (maximum application per year 511 g ai/ha, 7 day interval between sprays, PHI 14 days). No trials matched *c*GAP as the number of sprays differed and there is insufficient data to conclude the additional spray does not significantly contribute to the terminal residue.

Cotton seed

Residue trials were available from the USA. The cGAP for cotton in the USA is a pre-plant soil application at up to 290 g ai/ha followed by foliar applications at 128 g ai/ha (maximum application per year 547 g ai/ha, 7 day interval between sprays, PHI 30 days). Residues in trials matching cGAP were (n=11), < 0.01, 0.02, 0.04, 0.06, 0.07, 0.08, 0.09, 0.14, 0.16, 0.26 and 0.26 mg/kg.

The Meeting estimated an STMR of 0.08 mg/kg and a maximum residue level of 0.5 mg/kg for cotton seed.

Rape seed

Residue trials were available from the USA and member states of the European Union. The cGAP for rape in Russia is application at 125 g ai/ha (maximum two applications/year, interval 10–14 days, PHI 30 days). In trials conducted in member countries of the European Union approximating critical GAP in Russia, residues in rape seed were (n=8), mg/kg, Northern Europe, 0.04, 0.07, 0.13, 0.15 and 0.31 mg/kg, and Southern Europe, 0.03, 0.05 and 0.15 mg/kg.

The Meeting estimated an STMR of 0.1 mg/kg and a maximum residue level of 0.5 mg/kg for rape seed.

Animal feeds

Straw, forage and fodder of cereal grains and grasses

Maize forage and fodder

Residue trials were available from the USA. The cGAP for maize (field corn, popcorn and seed corn) in the USA is two applications at 128 g ai/ha (maximum application per year 256 g ai/ha, 7 day interval between sprays, PHI 7 days, 0 days for forage). Residues in forage from trials matching cGAP were (n=20), 0.53, 0.74, 0.91, 1.08, 1.14, 1.36, 1.45, 1.47, 1.53, 1.63, 1.65, 1.66, 1.75, 1.77, 1.85, 1.89, 2.19, 2.44, 2.66 and 2.74 mg/kg (as received basis). When corrected for measured moisture contents (33–70%) residues were , 1.86, 1.92, 3.17, 3.17, 3.82, 4.18, 4.53, 4.80, 4.88, 5.10, 5.52, 5.61, 5.66, 5.73, 5.78, 6.39, 6.89, 7.29, 8.30 and 8.47 mg/kg.

The Meeting estimated median residue of 5.31 mg/kg and a highest residue of 8.47 mg/kg for maize forage (dry weight basis).

Residues in maize fodder (stover) from trials matching cGAP were (n=20), < 0.02, 0.72, 0.88, 1.00, 1.04, 1.32, 1.40, 1.44, 1.46, 1.94, 2.07, 2.27, 2.38, 2.48, 2.64, 2.99, 2.99, 3.04, 3.98 and 5.44 mg/kg (as received basis). When corrected for measured moisture contents (54–73%) residues were 0.03, 1.62, 1.90, 3.00, 3.42, 3.72, 3.79, 3.99, 4.35, 4.84, 5.03, 5.04, 6.72, 6.92, 6.99, 7.21, 7.81, 8.12, 8.17 and 10.45 mg/kg.

The Meeting estimated median residue of 4.93 mg/kg, a highest residue of 10.45 mg/kg and a maximum residue level of 20 mg/kg for maize fodder (dry weight basis).

Sorghum

Residue trials were available from the USA. The *c*GAP for sorghum in the USA is two applications at 128 g ai/ha (maximum application per year 256 g ai/ha, 7 day interval between sprays, PHI 30 days for grain, forage and stover).

Sorghum forage (n=12), 0.08, 0.19, 0.20, 0.24, 0.26, 0.28, 0.52, 0.54, 0.64, 0.72, 0.78 and 1.0 mg/kg (fresh weight). Median and highest residues in sorghum forage are 0.40 and 1.0 mg/kg (fresh weight basis) or 1.1 and 2.85 mg/kg (dry weight basis) as forage contains 35% dry matter.

Sorghum fodder (n=12), 0.30, 0.42, 0.45, 0.52, 0.68, 0.80, 0.88, 0.92, 1.14, 1.46, 1.52 and 4.40 mg/kg (fresh weight). The Meeting estimated median and highest residues of 0.84 mg/kg and 4.4 mg/kg (fresh weight basis) or 0.95 and 5 mg/kg when expressed on a dry weight basis and assuming fodder contains 88% dry matter. The Meeting estimated a maximum residue level of 7 mg/kg for sorghum fodder (dry weight basis).

Miscellaneous fodder and forage crops

Sugar beet tops

The Meeting received trials performed in countries of the EU and also the USA.

The *c*GAP for sugar beet in the USA is two applications at 128 g ai/ha (maximum application per year 256 g ai/ha, 14 day interval between sprays, PHI 21 days). No trials matched GAP as the number of sprays differed and there is insufficient data to conclude the additional spray does not significantly contribute to the terminal residue (three sprays in trials vs two sprays cGAP).

GAP in Russia is for two applications at 62.5 g ai/ha with a 30 day PHI. Residues in trials from northern Europe at approximately double the application rate were (n=8), 0.1, 0.14, 0.14, 0.18, 0.18, 0.22, 0.22 and 0.75 mg/kg (on an as received basis). The Meeting decided to apply proportionality to the residue data.

Trial application rate (2nd spray) g ai/ha	Scaling factor = 62.5/trial application rate	Trial residue (mg/kg)	Scaled residue =scaling factor × trial residue (mg/kg)

131	0.477	0.10	0.048
128	0.488	0.14	0.068
126	0.496	0.14	0.069
120	0.520	0.18	0.094
111	0.563	0.18	0.101
135	0.463	0.22	0.102
130	0.481	0.22	0.106
138	0.453	0.75	0.340

Based on the residues from Europe scaled to cGAP for Russia, the Meeting estimated a median residue of 0.098 mg/kg and a highest residue of 0.340 mg/kg (on an as received basis). Sugar beet tops contain approximately 23% DM. The Meeting estimated a median residue of 0.424 mg/kg, a highest residue of 1.477 mg/kg and a maximum residue level of 3 mg/kg for sugar beet tops (on a dry weight basis).

Rape seed forage

Residue trials were available from the USA and member states of the European Union. The GAP for rape in Russia is application at 125 g ai/ha (maximum two applications/year, interval 10–14 days, PHI 30 days). The late application precludes the use of plant material as forage.

Cotton gin by-products

Residue trials were available from the USA. The cGAP for cotton in the USA is a pre-plant soil application at up to 290 g ai/ha followed by foliar applications at 128 g ai/ha (maximum application per year 547 g ai/ha, 7 day interval between sprays, PHI 30 days). Three trial matched cGAP with residues 1.12, 1.77 and 2.26 mg/kg (fresh weight basis). Three residue trials is insufficient to estimate a maximum residue level for cotton gin by-products.

Almond hulls

Residue trials were available from the USA. The cGAP for almonds, walnuts, pecans and other tree nuts in the USA is four applications at 128 g ai/ha (maximum application per year 511 g ai/ha, 7 day interval between sprays, PHI 14 days). No trials matched cGAP as the number of sprays differed and there is insufficient data to conclude the additional spray does not significantly contribute to the terminal residue (six sprays in trials versus four sprays for cGAP).

Fate of residues during processing

The Meeting received information on the nature of residues under simulated processing conditions on the fate of incurred residues of flutriafol during the processing of peaches, plums, grapes, strawberries, cabbages, tomatoes, lettuce, celery, sorghum, rice, and cotton seed. Flutriafol residues are stable under simulated processing conditions (pasteurization, baking/brewing/boiling and sterilisation).

Summary of selected processing factors for flutriafol

Raw commodity	Processed commodity	Individual PF	Best estimate PF	$STMR_{RAC}$ (mg/kg)	$STMR_{RAC}$ × PF (mg/kg)	HR_{RAC} (mg/kg)	HR_{RAC} × PF (mg/kg)
Apple	Juice [a]	0.50 0.45	0.48	0.08	0.038		
	Wet pomace [a]	1.9 1.9	1.9		0.152		
	Dry pomace [a]	10 8.5	9.3		0.744		
Peach	Juice	1.7 0.8	1.25	0.17	0.2125		
	Jam	0.7 1.0	0.85		0.1445		
Plum	Dried fruit	2.2	2.2	0.075	0.165	0.22	0.484
Grapes	Wet pomace	2.5 4.4	3.45	0.21	0.7245		
	Dry pomace	4.0 4.3 5.4 6.0 6.7 9.6 15, 17.8	8.6		1.806		
	Red wine	0.55 0.57 1.5 1.6	1.055		0.22155		
	White wine	0.79 0.84 1.7 3.4	1.68		0.3528		

Raw commodity	Processed commodity	Individual PF	Best estimate PF	$STMR_{RAC}$ (mg/kg)	$STMR_{RAC}$ × PF (mg/kg)	HR_{RAC} (mg/kg)	HR_{RAC} × PF (mg/kg)
Strawberry	Jam	0.75 0.87 0.92 0.96	0.875	0.43	0.3685		
Tomato	Purée	1.2	1.2	0.11	0.132		
	Paste	2.6	2.6		0.286		
Sorghum	Aspirated grain fraction	7.1 8.9	8.0	0.27	2.16		
Cottonseed	Hulls	0.33	0.33	0.08	0.0264		
	Meal	0.08	0.08		0.0064		
	Oil	0.08	0.08		0.0064		

[a] Values from 2011 JMPR

Residues concentrated in prunes (dried plums). Based on the estimated maximum residue level for plums of 0.4 mg/kg, the Meeting recommended a maximum residue level for prunes of 0.9 mg/kg (MRL × PF = 0.4 × 2.2 = 0.88 mg/kg rounded to 0.9 mg/kg).

Residues in animal commodities

Farm animal feeding studies

The Meeting received information on the residue levels arising in tissues and milk when dairy cows were fed a diet containing flutriafol at dietary levels of 5, 16 and 50 ppm for 28 consecutive days. Residues in whole milk were < 0.01 mg/kg. In cream, residues were < 0.01 mg/kg except for Day 21 where a residue of 0.01 mg/kg was detected. The highest residues (mean in brackets) in liver, kidney, fat and muscle from the 50 ppm dose group were 1.95 (1.83), 0.15 (0.10), 0.34 (0.19) and 0.07 (0.04) mg/kg 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 and poultry feed items include maize, peanut, soya bean and wheat commodities.

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	1.8	1.07	20.7 [a]	9.76 [c]	76	32	0.161	0.161
Dairy cattle	19.0	8.3	19.1 [b]	8.7 [d]	49.8	21.2	4.3	2.8
Poultry Broiler	0.26	0.26	0.24	0.24	0.24	0.24	0.23	0.23
Poultry Layer	0.26	0.26	7.9 [e]	3.45 [f]	0.24	0.24	0.20	0.20

[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.

The maximum dietary burden for cattle exceeds the maximum dosing level used in the feeding studies. It was noted that the dietary burdens are driven by the residues in wheat forage from trials that matched GAP in the USA (selected with a 0 day PHI) and that it may be possible to further refine the dietary burdens. In Australia, flutriafol is approved for use on wheat but the anticipated residues in forage are much lower as GAP requires a 49 day interval between last application and grazing and on other cereals with a 70 day interval for grazing. At these intervals

residues in forage and fodder are less than 3 mg/kg and the cattle dietary burdens for Australia listed in the table are overestimates. The Meeting decided to recalculate the cattle dietary burdens for Australia discounting cereal forages.

Additional refinement is also possible for the EU livestock burdens as in the EU uses on cereals are understood as "on cereal for grain production" and therefore, only residues in grains and straw are considered for the animal burden calculation and to utilise the cattle dietary burdens for the EU in estimating residues in cattle commodities (http://www.efsa.europa.eu/sites/default/files/event/140619-m.pdf). The maximum dietary burdens on refinement are 10.5 and 4.2 ppm for the maximum and mean burdens for beef and dairy cows in the Australian region. The refined poultry dietary burdens are 1.35 and 0.75 ppm for the maximum and mean burdens for laying hens in the EU region.

Animal commodity maximum residue levels

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

Flutriafol feeding study	Feed level	Residues	Feed level	Residues (mg/kg) in			
	(ppm) for milk residues	(mg/kg) in milk	(ppm) for tissue residues	Muscle	Liver	Kidney	Fat
MRL and HR beef or dairy cattle							
Feeding study [a]	16	< 0.01	16	< 0.01	0.77	0.02	0.02
Dietary burden and high residue	10.5	< 0.0066	10.5	0.0066	0.505	0.013	0.013
STMR beef or dairy cattle							
Feeding study [b]	16	< 0.01	5	< 0.01	0.33	< 0.01	< 0.01
Dietary burden and median residue	4.2	< 0.0026	4.2	< 0.008	0.277	< 0.008	< 0.008

[a] Highest residues for tissues and mean residues for milk

[b] Mean residues for tissues and mean residues for milk

The Meeting estimated a maximum residue levels of 0.01 (*) mg/kg for milk, 0.02 mg/kg for mammalian meat [in the fat], 0.02 for mammalian fats (except milk fats) and 1 mg/kg for mammalian edible offal.

The refined maximum dietary burden for broiler and layer poultry is lower than that estimated by the 2011 JMPR at 1.35 ppm and is now lower than the highest dose level in the feeding study of 5.0 ppm. The Meeting utilised the refined estimates of poultry dietary burdens and estimated maximum residue levels of 0.01 (*) mg/kg for poultry meat, 0.02 mg/kg for poultry fats, 0.03 mg/kg for poultry edible offal and 0.01 (*) mg/kg for eggs.

Flutriafol feeding study	Feed level	Residues	Feed level	Residues (mg/kg) in			
	(ppm) for egg residues	(mg/kg) in eggs	(ppm) for tissue residues	Muscle	Liver		Fat
MRL and HR chickens							
Feeding study [a]	5	0.03	5	< 0.01	0.10		0.07
Dietary burden and high residue	1.35	0.0081	1.35	< 0.0027	0.027		0.0189
STMR chickens							
Feeding study [b]	5	0.03	5	< 0.01	0.07		0.06
Dietary burden and residue estimate	0.75	0.0045	0.75	0.0015	0.0105		0.009

[a] Highest residues for tissues and mean residues for eggs

[b] Mean residues for tissues and mean residues for eggs

RECOMMENDATIONS FURTHER WORK OR INFORMATION

On the basis of the data obtained from supervised residue trials the Meeting concluded that the residue levels listed in Annex 1 are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

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

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

The residue is fat soluble.

Table of recommendations

Commodity		Recommended MRL (mg/kg)		STMR or STMR-P (mg/kg)	HR, HR-P, highest residue (mg/kg)
CCN	Name	New	Previous		
VB 0040	Brassica (cole or cabbage) vegetables, Head cabbages, Flowerhead brassicas	1.5		0.14	0.80
VS 0624	Celery	3		0.78	1.41
FS 0013	Cherries	0.8		0.335	0.66
SO 0691	Cotton seed	0.5		0.08	
MO 0105	Edible offal (mammalian)	1		0.277 liver 0.008 kidney	0.505 liver 0.013 kidney
PE 0112	Eggs	0.01 (*)		0.0045	0.0081
VC 0045	Fruiting vegetables, Cucurbits	0.3		0.09	0.13
VL 0482	Lettuce, Head	1.5		0.22	0.67
VL 0483	Lettuce, Leaf	5[a]		0.36	2.95
GC 0645	Maize	0.01 (*)		0	
AS 0645	Maize fodder (dry)	20		4.93 dw	10.45 dw
MF 0100	Mammalian fats (except milk fats)	0.02		0.008	0.013
MM 0095	Meat (from mammals other than marine mammals)	0.02 (fat)		0.008 fat 0.008 muscle	0.013 fat 0.007 muscle
ML 0106	Milks	0.01 (*)		0.0026	0.0066
VL 0485	Mustard greens	7[a]		2.12	3.53
FS 2001	Peaches (including nectarine and apricots)	0.6		0.17	0.42
VO 0051	Peppers (Subgroup including Peppers, Chili and Peppers, Sweet)	1		0.28	0.41
VO 0445	Peppers, Sweet (including pimento or pimiento)	W	1		
FS 0014	Plums (including prunes)	0.4		0.075	0.25
FP 0009	Pome fruit	0.4	0.3	0.08	0.26
PF 0111	Poultry fats	0.02		0.009	0.0189
PM 0110	Poultry meat	0.01 (*)		0.0015	0.0027
PO 0111	Poultry, Edible offal of	0.03		0.0105	0.027
DF 0014	Prunes	0.9		0.165	0.484
SO 0495	Rape seed	0.5		0.1	
GC 0651	Sorghum	1.5		0.27	
AS 0651	Sorghum straw and fodder, dry	7		0.95 dw	5 dw
VL 0502	Spinach	10[a]		1.665	5.5
FB 0275	Strawberry	1.5		0.43	0.78
VR 0596	Sugar beet	0.02		0.01	0.0136
AV 0596	Sugar beet leaves or tops	3 dw		0.424 dw	1.477 dw
VO 0448	Tomatoes	0.8		0.11	0.63

dw = dry weight basis

[a] On the basis of information provided to the JMPR, the Meeting concluded that the short-term intake of residues of flutriafol from consumption of leaf lettuce, mustard greens and spinach may present a public health concern.

Table of additional STMR/median and HR/highest residue values for use in dietary intake and livestock dietary burden estimation.

Commodity		Recommended MRL (mg/kg)		STMR or STMR-P (mg/kg)	HR, HR-P, highest residue (mg/kg)
CCN	Name	New	Previous		
OR 0691	Cotton seed oil, edible			0.0064	
	Cotton seed hulls			0.0264	
	Cotton seed meal			0.0064	
AB 0269	Grape pomace, dry			1.806	

Commodity		Recommended MRL (mg/kg)		STMR or STMR-P (mg/kg)	HR, HR-P, highest residue (mg/kg)
CCN	Name	New	Previous		
	Red wine			0.22155	
	White wine			0.3528	
AF 0645	Maize forage			5.31 dw	8.47 dw
	Peach juice			0.2125	
	Peach jam			0.1445	
AB 0226	Apple pomace, dry			0.744	
AF 0651	Sorghum forage (green)			1.1 dw	2.85 dw
	Sorghum aspirated grain fractions			2.16	
	Strawberry jam			0.3685	
	Tomato purée			0.132	
	Tomato paste			0.286	

dw = dry weight basis

DIETARY RISK ASSESSMENT

Long-term intake

The 2011 JMPR established an Acceptable Daily Intake (ADI) of 0–0.01 mg/kg bw for flutriafol.

The evaluation of flutriafol 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 results are shown in Annex 3.

The IEDIs in the seventeen Cluster Diets, based on the estimated STMRs were 3–10% of the maximum ADI (0.01 mg/kg bw). The Meeting concluded that the long-term intake of residues of flutriafol from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The 2011 JMPR established an Acute Reference Dose (ARfD) of 0.05 mg/kg bw for flutriafol. The International Estimated Short-term Intake (IESTI) for flutriafol was calculated for raw and processed commodities for which maximum residue levels, HR and STMR values were estimated. The results are shown in Annex 4 to the 2015 Report.

The IESTI represented greater than 100% of the ARfD of 0.05 mg/kg bw in the case of leaf lettuce (360% children; 120% general population), mustard greens (350% children; 140% general population) and spinach (490% children; 150% general population). No alternative GAP was available. On the basis of information provided to the JMPR, the Meeting concluded that the short-term intake of residues of flutriafol from consumption of leaf lettuce, mustard greens and spinach may present a public health concern.

Estimates of intake for the other commodities considered by the 2015 JMPR were within 0–90% of the ARfD. The Meeting concluded that the short-term intake of flutriafol for these other commodities considered is unlikely to present a public health concern when flutriafol is used in ways that considered by the Meeting.

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2699	Carringer, S.J.	2013	Magnitude and Decline of Flutriafol and Metabolite Residues in/on Grain Sorghum Raw Agricultural Commodities following two foliar Applications of Flutriafol 125 g/L SC – 2012, The Carringers, Inc. / Morse Laboratories, LLC , USA, Study No.: TCI-12-344 ; 68551, Report No.: TCI-12-344 ; 68551, Unpublished report, CHA Doc. No.: 2699 FLU
2700	Carringer, S.J.	2013	Magnitude and Decline of Flutriafol and Metabolite Residues in/on Cotton Raw Agricultural and Processed Commodities Following One In-furrow Application and Two Foliar Applications of Flutriafol 125 g/L SC – 2012, The Carringers, Inc. / Morse Laboratories, LLC , USA, Study No.: TCI-12-343; 68550, Report No.: TCI-12-343; 68550, Unpublished report, CHA Doc. No.: 2700 FLU

FLUXAPYROXAD (256)

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EXPLANATION

Fluxapyroxad is a fungicide belonging to the carboxamide group of chemicals. It acts through inhibition of the enzyme succinate dehydrogenase, which is also known as complex II, in the mitochondrial electron transport chain. It is used as a foliar and seed treatment fungicide for control of a range of fungal diseases in cereals, fruit and vegetables.

Fluxapyroxad was evaluated by JMPR for the first time in 2012, when an ADI of 0–0.02 mg/kg bw/day and an ARfD of 0.3 mg/kg bw were established. A residue definition of *fluxapyroxad* was recommended for plant and animal commodities, for compliance with MRLs. For estimation of dietary intake in plant commodities, a definition of *sum of fluxapyroxad, 3-(difluoromethyl)-N-(3',4',5'-trifluoro-1,1'-biphenyl-2-yl)-1H-pyrazole-4-carboxamide (M700F008), and 3-(difluoromethyl)-1-(β-D-glucopyranosyl)-N-(3',4',5'-trifluoro-1,1'-biphenyl-2-yl)-1H-pyrazole-4-carboxamide (M700F048), expressed as fluxapyroxad,* was recommended. For estimation of dietary intake in animal commodities, a definition of *sum of fluxapyroxad and 3-(difluoromethyl)-N-(3',4',5'-trifluoro-1,1'-biphenyl-2-yl)-1H-pyrazole-4-carboxamide (M700F008), expressed as fluxapyroxad,* was recommended. The residue is fat soluble.

At the 46th Session of the CCPR (2014), fluxapyroxad was scheduled for evaluation of additional use patterns by the 2015 JMPR.

The Meeting received residue data for citrus fruits, cherries, grapes, strawberries, caneberries, blueberries, mangoes, bananas, papaya, bulb vegetables, Brassica vegetables, cucurbits, leafy vegetables, root and tuber vegetables, celery, rice, sugar cane, almonds, pecans, and cotton (foliar application). Processing data for oranges, grapes, sugar cane and cotton were received. Product labels and information on MRLs established by national regulatory authorities were also provided.

Analytical methods

No new analytical methods were submitted to the Meeting. Residues of fluxapyroxad and its metabolites were determined using LC-MS/MS method number L0137/01 for all trials submitted to the Meeting. This method was reviewed by the 2012 Meeting. Appropriate concurrent recovery data was provided for all trials.

Stability of pesticide residues in stored analytical samples

Plant matrices

No new storage stability studies were submitted to the current Meeting. The 2012 Meeting evaluated the stability of residues of fluxapyroxad and the metabolites M700F002, M700F008, and M700F048 in a range of plant matrices. In the residue trials submitted to the Meeting, samples were analysed within 24 months of collection, within the period for which stability was verified by the studies submitted to the 2012 Meeting.

USE PATTERNS

Fluxapyroxad is a fungicide. It is registered for foliar and seed treatment use in a wide variety of fruits, vegetables, nuts, oilseeds, and cereals against a wide variety of diseases.

Table 1 Registered uses of fluxapyroxad on crops relevant to this submission

Crop	Country	Application						
		Formulation	Type	Rate, g ai/ha	Conc. (g ai/hL)	Spray volume (L/ha)	No. (RTI, days)	PHI, days
Citrus fruit								
Citrus	Brazil	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	–	0.84–2.5	2000	3 (7)	14
Grapefruit	Mexico	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	50–67	–	460–560	2 (20)	14
	Argentina	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	–	3.3	2000–5000	3	7
Lemon	Mexico	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	50–67	–	460–560	2 (20)	14
	Argentina	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	–	3.3	2000–5000	3	7
Lime	Mexico	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	50–67	–	460–560	2 (20)	14
Mandarin	Mexico	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	50–67	–	460–560	2 (20)	14
	Argentina	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	–	3.3	2000–5000	3	7
Orange	Mexico	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	50–67	–	460–560	2 (20)	14
Stone fruit								
Stone fruit	Canada	EC 62.5 g/L	Foliar	100	–		3 (7)	0
		SC 300 g/L	Foliar	100	–		3 (7)	0
	USA	EC 62.5 g/L	Foliar	123	–		3 (7)	0
		SC 300 g/L	Foliar	123	–		3 (7)	0
		SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	73–123	–		3 (7)	0
Berries and other small fruits								
Bushberries	USA	EC 62.5 g/L	Foliar	75–200	–		3 (7)	0
	USA	SC 300 g/L	Foliar	75–200	–		3(7)	0
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	73–107	–		3 (7)	0
	USA	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	73–200	–		3 (7)	0
Caneberries	USA	EC 62.5 g/L	Foliar	75–200	–		3 (7)	0
	USA	SC 300 g/L	Foliar	75–200	–		3 (7)	0
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	73–107	–		3 (7)	0
	USA	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	73–200	–		3 (7)	0
Low growing berries	USA	EC 62.5 g/L	Foliar	75–200	–		3 (7)	0
	USA	SC 300 g/L	Foliar	100–200	–		3 (7)	0

Crop	Country	Application						
		Formulation	Type	Rate, g ai/ha	Conc. (g ai/hL)	Spray volume (L/ha)	No. (RTI, days)	PHI, days
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	73–107	–		3 (7)	0
	USA	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	146–200	–		3 (7)	0
Small climbing vine fruit	USA	EC 62.5 g/L	Foliar	75–200	–		3 (7)	14
	USA	SC 300 g/L	Foliar	100–200	–		3 (7)	14
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	73–107	–		3(7)	14
	USA	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	146–200	–		3 (7)	14
Grapes	USA	EC 62.5 g/L	Foliar	46–100	–		6 (10)	14
	USA	EC 62.5 g/L	Foliar	100–200	–		3 (10)	14
	USA	SC 300 g/L	Foliar	44–99	–		6 (10)	14
	USA	SC 300 g/L	Foliar	99–199	–		3 (10)	14
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	49–84	–		3 (10)	14
	USA	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	73–100	–		6 (10)	14
	USA	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	100–200	–		3 (10)	14
	Chile	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	75	–	800–1500	2 (14), do not apply after flowering	–
Strawberries	USA	EC 62.5 g/L	Foliar	75–200	–		3 (7)	0
	USA	SC 300 g/L	Foliar	75–199	–		3 (7)	0
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	73–107	–		3 (7)	0
	USA	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	73–200	–		3 (7)	0
	Mexico	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	75–125	–	400–500	3 (7)	1
	Mexico	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	50–84	–	400–500	3 (7)	1
Assorted tropical and subtropical Fruits—inedible peel								
Banana	Belize	SC 300 g/L	Foliar (ground or aerial)	90–150 + 7–9 L/ha agricultural oil			4 (8)	0
	Colombia	SC 300 g/L	Foliar (ground or aerial)	150 + 7–9 L/ha agricultural oil		18–23 (aerial), 50–60 (ground)	3 (12)	0
	Costa Rica	SC 300 g/L	Foliar (ground or	90–150 + 7–9 L/ha			4 (8)	0

Crop	Country	Application						
		Formulation	Type	Rate, g ai/ha	Conc. (g ai/hL)	Spray volume (L/ha)	No. (RTI, days)	PHI, days
			aerial)	agricultural oil				
	Dominican Republic	SC 300 g/L	Foliar (ground or aerial)	90–150 + 7–9 L/ha agricultural oil			4 (8)	0
	Ecuador	SC 300 g/L	Foliar (ground or aerial)	150		18–23		1
	El Salvador	SC 300 g/L	Foliar (ground or aerial)	90–150 + 7–9 L/ha agricultural oil			4 (8)	0
	Guatemala	SC 300 g/L	Foliar (ground or aerial)	90–150 + 7–9 L/ha agricultural oil			4 (8)	0
	Honduras	SC 300 g/L	Foliar (ground or aerial)	90–150 + 7–9 L/ha agricultural oil			4 (8)	0
	Panama	SC 300 g/L	Foliar (ground or aerial)	90–150 + 7–9 L/ha agricultural oil			4 (8)	0
Mango	Brazil	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar		4.2–6.7	500–1000	4 (7)	7
Papaya	Mexico	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	75–100		400	2 (14)	7
Bulb vegetables								
Bulb vegetables	USA	SC 62.5 g/L	Foliar	75–200			3 (7)	7
	USA	SC 300 g/L	Foliar	75–200			3 (7)	7
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	73–90			3 (7)	7
	USA	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	73–200			3 (7)	7
Garlic	USA	FS 333 g/L	Seed treatment	20–40 g ai/100 kg seed			1	–
	USA	FS 333 g/L	Seed treatment	125–250 g ai/100 kg seed			1	–
	USA	FS 250 g/L (pyraclostrobin 250 g/L)	Seed treatment	33–40 g ai/100 kg seed			1	–
Leek	USA	FS 333 g/L	Seed treatment	20–40 g ai/100 kg seed			1	–
	USA	FS 333 g/L	Seed treatment	125–250 g ai/100 kg seed			1	–
	USA	FS 250 g/L (pyraclostrobin 250 g/L)	Seed treatment	33–40 g ai/100 kg seed			1	–
Onions (all)	USA	FS 333 g/L	Seed treatment	20–40 g ai/100 kg seed			1	–
	USA	FS 333 g/L	Seed treatment	125–250 g ai/100 kg seed			1	–
	USA	FS 250 g/L	Seed	33–			1	–

Crop	Country	Application						
		Formulation	Type	Rate, g ai/ha	Conc. (g ai/hL)	Spray volume (L/ha)	No. (RTI, days)	PHI, days
		(pyraclostrobin 250 g/L)	treatment	40 g ai/100 kg seed				
Onion	Brazil	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58		200–1000	4 (7)	7
	Dominican Republic	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58			3 (7)	7
	El Salvador	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	50–58			3 (7)	7
	Guatemala	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58			3 (7)	7
Shallots	USA	FS 333 g/L	Seed treatment	20–40 g ai/100 kg seed			1	–
	USA	FS 333 g/L	Seed treatment	125–250 g ai/100 kg seed			1	–
	USA	FS 250 g/L (pyraclostrobin 250 g/L)	Seed treatment	33–40 g ai/100 kg seed			1	–
Brassica vegetables								
Brassica vegetables	USA	EC 62.5 g/L	Foliar	75–100			3 (7)	3
	USA	SC 300 g/L	Foliar	75–100			3 (7)	3
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	73–100			3 (7)	3
	USA	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	73–100			3 (7)	3
	USA	FS 333 g/L	Seed treatment	20–40 g ai/100 kg seed			1	–
	USA	FS 250 g/L (pyraclostrobin 250 g/L)	Seed treatment	33–40 g ai/100 kg seed			1	–
Fruiting vegetables, Cucurbits								
Cucurbits	USA	EC 62.5 g/L	Foliar	75–100			3 (7)	0
	USA	SC 300 g/L	Foliar	75–100			3 (7)	0
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	73–100			3 (7)	0
	USA	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	73–100			3 (7)	0
	USA	FS 333 g/L	Seed treatment	20–40 g ai/100 kg seed			1	–
	USA		Seed treatment	30 g ai/100 kg seed			1	–
Cucumbers	Brazil	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58		400–1000	4 (7)	7
	Mexico	SC 250 g/L (pyraclostrobin	Foliar	62.5–100			4 (4)	1

Crop	Country	Application						
		Formulation	Type	Rate, g ai/ha	Conc. (g ai/hL)	Spray volume (L/ha)	No. (RTI, days)	PHI, days
		250 g/L)						
Melons	Brazil	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58		400–1000	4 (7)	7
	Mexico	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	62.5–100			4 (4)	1
	Dominican Republic	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58			3 (7)	7
	Guatemala	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58			3 (7)	7
	Honduras	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58			3 (7)	7
	Trinidad and Tobago	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58			3 (7)	7
Pumpkins	Mexico	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	62.5–100			4 (4)	1
Watermelons	Mexico	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	62.5–100			4 (4)	1
	Dominican Republic	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58			3 (7)	7
	Guatemala	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58			3 (7)	7
	Honduras	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58			3 (7)	7
Zucchini	Mexico	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	62.5–100			4 (4)	1
Leafy vegetables								
Brassica leafy vegetables	USA	EC 62.5 g/L	Foliar	75–100			3 (7)	3
	USA	SC 300 g/L	Foliar	75–100			3 (7)	3
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	75–100			3 (7)	3
	USA	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	75–100			3 (7)	3
	USA	FS 333 g/L	Seed treatment	20–40 g ai/100 kg seed			1	–
	USA	FS 250 g/L (pyraclostrobin 250 g/L)	Seed treatment	33–40 g ai/100 kg seed			1	–
Leafy vegetables (except Brassica leafy vegetables)	USA	EC 62.5 g/L	Foliar	75–200			3 (7)	1

Crop	Country	Application						
		Formulation	Type	Rate, g ai/ha	Conc. (g ai/hL)	Spray volume (L/ha)	No. (RTI, days)	PHI, days
	USA	SC 300 g/L	Foliar	75–200			3 (7)	1
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	73–112			3 (7)	1
	USA	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	73–200			3 (7)	1
	USA	FS 333 g/L	Seed treatment	20–40 g ai/100 kg seed			1	–
	USA	FS 333 g/L	Seed treatment	100–200 g ai/100 kg seed			1	–
	USA	FS 250 g/L (pyraclostrobin 250 g/L)	Seed treatment	30 g ai/100 kg seed			1	–
Root and tuber vegetables								
Potatoes	Brazil	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	33–58		400–500	4 (7)	3
Potatoes	Canada	EC 62.5 g/L	Foliar	50–100			3 (7)	7
	Canada	SC 300 g/L	Foliar	50–100			3 (7)	7
Potatoes	Canada	EC 62.5 g/L	In-furrow	100			1	–
	Canada	SC 300 g/L	In-furrow	100			1	–
	USA	EC 62.5 g/L	In-furrow	100			1	–
	USA	SC 300 g/L	In-furrow	100			1	–
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	In-furrow	100			1	–
	USA	SC 250 g/L (pyraclostrobin 250 g/L)	In-furrow	100			1	–
Potatoes	Mexico	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	50–150		400–500	2 (7)	7
	Mexico	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	33–50		400–500	2 (7)	7
	Mexico	SC 250 g/L (pyraclostrobin 250 g/L)	In-furrow	425–500		600–700	1	–
	Mexico	SC 167 g/L (pyraclostrobin 333 g/L)	In-furrow	250–330		600–700	1	–
Potatoes	Dominican Republic	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58			3 (7)	7
Potatoes	Guatemala	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58			3 (7)	7
Potatoes	Honduras	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58			3 (7)	7
Potatoes	Trinidad and Tobago	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58			3 (7)	7
Carrots	Brazil	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58		400–700	4 (7)	7

Crop	Country	Application						
		Formulation	Type	Rate, g ai/ha	Conc. (g ai/hL)	Spray volume (L/ha)	No. (RTI, days)	PHI, days
	Dominican Republic	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58			3 (7)	7
	Guatemala	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58			3 (7)	7
Chinese artichokes	Canada	EC 62.5 g/L	Foliar	50–100			3 (7)	7
	Canada	SC 300 g/L	Foliar	50–100			3 (7)	7
Jerusalem artichokes	Canada	EC 62.5 g/L	Foliar	50–100			3 (7)	7
	Canada	SC 300 g/L	Foliar	50–100			3 (7)	7
Chufa	Canada	EC 62.5 g/L	Foliar	50–100			3 (7)	7
	Canada	SC 300 g/L	Foliar	50–100			3 (7)	7
Sweet potatoes	Canada	EC 62.5 g/L	Foliar	50–100			3 (7)	7
	Canada	SC 300 g/L	Foliar	50–100			3 (7)	7
True yams	Canada	EC 62.5 g/L	Foliar	50–100			3 (7)	7
	Canada	SC 300 g/L	Foliar	50–100			3 (7)	7
Sugar beets	Canada	EC 62.5 g/L	Foliar	100			3 (7)	7
	Canada	SC 300 g/L	Foliar	100			3 (7)	7
Sugar beets	Canada	EC 62.5 g/L	In-furrow	100			1	–
	Canada	SC 300 g/L	In-furrow	100			1	–
Root and tuber vegetables (except sugar beets)	USA	EC 62.5 g/L	Foliar	75–100			3 (7)	7
	USA	SC 300 g/L	Foliar	75–100			3 (7)	7
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	73–100			3 (7)	7
	USA	FS 333 g/L	Seed treatment	20–40 g ai/100 kg seed			1	–
Sugar beets	USA	EC 62.5 g/L	Foliar	50–100			3 (7)	7
	USA	SC 300 g/L	Foliar	50–100			3 (7)	7
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	73–100			3 (7), or 4 at the lower rate	7
Ginger	USA	EC 62.5 g/L	Foliar	50–100			3(7)	7
		SC 300 g/L	Foliar	50–100			3 (7)	7
		SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	50–100			3 (7)	7
		SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	55–100			3 (7)	7
Turmeric	USA	EC 62.5 g/L	Foliar	50–100			3 (7)	7
		SC 300 g/L	Foliar	50–100			3 (7)	7
		SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	50–100			3 (7)	7
		SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	55–100			3 (7)	7
Stalk and stem vegetables								
Celery	USA	EC 62.5 g/L	Foliar	75–200			3 (7)	1

Crop	Country	Application						
		Formulation	Type	Rate, g ai/ha	Conc. (g ai/hL)	Spray volume (L/ha)	No. (RTI, days)	PHI, days
	USA	SC 300 g/L	Foliar	75–200			3 (7)	1
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	73–112			3 (7)	1
	USA	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	73–200			3 (7)	1
	USA	FS 333 g/L	Seed treatment	20–40 g ai/100 kg seed			1	–
	USA	FS 333 g/L	Seed treatment	100–200 g ai/100 kg seed			1	–
	USA	FS 250 g/L (pyraclostrobin 250 g/L)	Seed treatment	30 g ai/100 kg seed			1	–
Cereal grains								
Rice	USA	EC 62.5 g/L	Foliar	75–150			2 (7)	28
	USA	SC 300 g/L	Foliar	100–150			2 (7)	28
	USA	FS 333 g/L	Seed treatment	25–50 g ai/100 kg seed			1	–
	Cuba	EC 62.5 g/L (epoxiconazole 62.5 g/L)	Foliar	47–78			2 (25)	35
	Dominican Republic	EC 62.5 g/L (epoxiconazole 62.5 g/L)	Foliar	62.5–75			2 (25)	35
	El Salvador	EC 62.5 g/L (epoxiconazole 62.5 g/L)	Foliar	62.5–75			2 (25)	35
	Guatemala	EC 62.5 g/L (epoxiconazole 62.5 g/L)	Foliar	62.5–75			2 (25)	35
	Honduras	EC 62.5 g/L (epoxiconazole 62.5 g/L)	Foliar	62.5–75			2 (25)	35
Sorghum	USA	EC 62.5 g/L	Foliar	75–100			2	21
	USA	SC 300 g/L	Foliar	75–100			2	21
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	50–100			1	21
	USA	FS 333 g/L	Seed treatment	10–20 g ai/100 kg seed			1	–
	USA	FS 327 g/L	Seed treatment	10–20 g ai/100 kg seed			1	–
	Mexico	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	50–100		550–650	2 (14)	10
	Mexico	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	50–67		200–300	2 (14)	10
	Brazil	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58		150–200	2 (14)	30
Grasses for sugar or syrup production								

Crop	Country	Application						
		Formulation	Type	Rate, g ai/ha	Conc. (g ai/hL)	Spray volume (L/ha)	No. (RTI, days)	PHI, days
Sugar cane	USA	EC 62.5 g/L	Foliar	75–125			2 (14)	14
	USA	SC 300 g/L	Foliar	125			2 (14)	14
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	50–110			2 (14)	14
	Brazil	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	50–67		150–200	5 (21)	30
Tree nuts								
Tree nuts	USA	EC 62.5 g/L	Foliar	75–125			3 (7)	14
	USA	SC 300 g/L	Foliar	75–125			3 (7)	14
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	67			3 (7)	14
	USA	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	91–119			3 (7)	14
Oilseeds								
Cotton	USA	FS 333 g/L	Seed treatment	10–20 g ai/100 kg seed			1	–
	USA	FS 327 g/L	Seed treatment	10–20 g ai/100 kg seed			1	–
	USA	FS 250 g/L (pyraclostrobin 250 g/L)	Seed treatment	20 g ai/100 kg seed			1	–
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	50–100			2 (7)	21
	USA	SC 250 g/L (pyraclostrobin 250 g/L)	Foliar	73–100			2 (7)	21
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	In-furrow/soil directed banded spray	0.16–1 g ai/100 row metres			1	–
	USA	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	50–100			3 (7)	30
	USA	SC 300 g/L	Foliar	50–100			3 (7)	30
	USA	EC 62.5 g/L	Foliar	50–100			3 (7)	30
	Brazil	SC 167 g/L (pyraclostrobin 333 g/L)	Foliar	42–58		150–200	4 (12)	14

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received supervised trials for use of fluxapyroxad on citrus fruit (oranges, lemons and limes), cherries, berries and small fruits (grapes, blueberries, blackberries, raspberries and strawberries), tropical fruit, inedible peel (banana, papaya and mango), bulb vegetables (onion, bulb and green onion), Brassica vegetables (cabbage and broccoli), fruiting vegetables, cucurbits (cucumber, summer squash, melon (cantaloupe), and watermelon), leafy vegetables (head lettuce, leafy lettuce, spinach and mustard greens), root and tuber vegetables (carrots, radish and potato), celery, rice, sugar cane, tree nuts (almonds and pecans), and cotton.

Residue data for stone fruit, potatoes, sugar beet, and sorghum evaluated by the 2012 Meeting are also tabulated below. The data tables have been taken unaltered from the 2012 evaluation. These data were evaluated against registered uses for these crops submitted to the current Meeting.

In all trials, residues were determined using method L0137/01. The method LOQ was 0.01 mg/kg for each analyte as measured, or 0.01, 0.02, 0.01 and 0.01 mg/kg as parent equivalents for parent, M700F002, M700F008, and M700F048 respectively. For replicate samples from the same plot, the mean value was used for maximum residue level estimation, with the individual results being given in brackets. All residues below the LOQ are reported as < the appropriate LOQ value, as parent equivalents. For multiple trials from the same location in the same year, results from the trial yielding the highest residue were used for estimation of maximum residue levels and dietary intake assessment.

For dietary intake assessment, the residues are expressed as the sum of fluxapyroxad, M700F008, and M700F048, expressed as fluxapyroxad (total residues). Residues of the metabolites are reported as parent equivalents.

Group	Commodity	Countries	Table
FC Citrus fruits	Orange	Brazil, Argentina	2, 3
	Lemon	Argentina	4
	Lime	Brazil	5
FS Stone fruits	Cherry	USA, Canada	6
	Peach	USA, Canada	7
	Plum	USA, Canada	8
FB Berries and other small fruits	Blueberries	USA	9
	Caneberries (blackberries, raspberries)	USA	10
	Grapes	USA	11
	Strawberries	USA	12
FI Assorted tropical and sub-tropical fruits—inedible peel	Banana	Brazil, Colombia, Costa Rica, Ecuador	13, 14
	Mango	Brazil	15
	Papaya	Brazil	16
VA Bulb vegetables	Onion, bulb	USA	17
	Onion, green	USA	18
VB Brassica vegetables	Broccoli	USA	19
	Cabbage	USA	20
VC Fruiting vegetables, Cucurbits	Melons	USA, Brazil	21, 22
	Cucumber	USA	23
	Squash, summer	USA	24
	Watermelon	Brazil	25
VL Leafy vegetables	Lettuce, Head	USA	26

Group	Commodity	Countries	Table
	Lettuce, Leaf	USA	27
	Mustard greens	USA	28
	Radish leaves	USA	29
	Spinach	USA	30
VR Root and tuber vegetables	Carrot	USA	31, 32
	Potato	Germany, UK, the Netherlands, Belgium, France, Greece, Italy, Spain, USA, Canada	33, 34, 35
	Radish	USA	36
	Sugar beet	USA, Canada	37
VS Stalk and stem vegetables	Celery	USA	38
GC Cereal grains	Rice	USA	39
	Sorghum	USA	40
GS Grasses for sugar or syrup production	Sugar cane	USA	41
TN Tree nuts	Almonds	USA	42
	Pecans	USA	43
SO Oilseed	Cotton	USA	44
Animal feeds	Rice straw	USA	45
	Sorghum forage and stover	USA	46
	Almond hulls	USA	47
	Cotton gin by-products	USA	48

Citrus fruits

Residue trials in oranges, lemons and limes were conducted in Brazil and Argentina (Dantas *et al.*, 2012 and Guimaraes, 2014-a). Three foliar applications of an SC formulation containing 167 g/L fluxapyroxad and 333 g/L pyraclostrobin were made at each site using an airblast sprayer.

Table 2 Residues of fluxapyroxad and metabolites in oranges (whole fruit)

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
San Antonio de Posse, Sao Paolo, Brazil, 2010 (Pera Coroa)	3 (28, 28)	50, 50, 50	2000, 2000, 2000	0	0.15	< 0.02	< 0.01	< 0.01	0.15
				7	0.14	< 0.02	< 0.01	< 0.01	0.14
				14	0.14	< 0.02	< 0.01	< 0.01	0.14

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
				21	0.16	< 0.02	< 0.01	< 0.01	0.16
				28	0.15	< 0.02	< 0.01	< 0.01	0.15
San Antonio de Posse, Sao Paolo, Brazil, 2010 (Natal)	3 (28, 28)	50, 50, 50	2000, 2000, 2000	0	0.09	< 0.02	< 0.01	< 0.01	0.09
				7	0.12	< 0.02	< 0.01	< 0.01	0.12
				14	0.17	< 0.02	< 0.01	< 0.01	0.17
				21	0.11	< 0.02	< 0.01	< 0.01	0.11
				28	0.10	< 0.02	< 0.01	< 0.01	0.10
Jaboticabal, Sao Paolo, Brazil, 2010 (Pera)	3 (28, 23)	50, 50, 50	2000, 2000, 2000	14	0.14	< 0.02	< 0.01	< 0.01	0.14
Londrina, Parana, Brazil, 2010 (Pera Rio)	3 (28, 28)	50, 50, 50	2000, 2000, 2000	14	0.06	< 0.02	< 0.01	< 0.01	0.06
San Antonio de Posse, Sao Paolo, Brazil, 2013 (Pera Coroa)	3 (29, 27)	50, 50, 50	2000, 2000, 2000	0	0.17	< 0.02	< 0.01	< 0.01	0.17
				7	0.16	< 0.02	< 0.01	< 0.01	0.16
				14	0.12	< 0.02	< 0.01	< 0.01	0.12
				21	0.14	< 0.02	< 0.01	< 0.01	0.14
				28	0.10	< 0.02	< 0.01	< 0.01	0.10
Aguai, Sao Paolo, Brazil, 2013 (Pera Murcha)	3 (28, 28)	50, 50, 50	2000, 2000, 2000	0	0.06	< 0.02	< 0.01	< 0.01	0.06
				7	0.07	< 0.02	< 0.01	< 0.01	0.07
				14	0.04	< 0.02	< 0.01	< 0.01	0.04
				21	0.04	< 0.02	< 0.01	< 0.01	0.04
				28	0.02	< 0.02	< 0.01	< 0.01	0.02
Mogi Mirim, Sao Paolo, Brazil, 2013 (Pera Coroa)	3 (28, 28)	50, 50, 50	2000, 2000, 2000	0	0.07	< 0.02	< 0.01	< 0.01	0.07
				7	0.06	< 0.02	< 0.01	< 0.01	0.06
				14	0.03	< 0.02	< 0.01	< 0.01	0.03
				21	0.05	< 0.02	< 0.01	< 0.01	0.05

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
				28	0.05	< 0.02	< 0.01	< 0.01	0.05
Londrina, Parana, Brazil, 2013 (Pera Rio)	3 (28, 28)	50, 50, 50	2000, 2000, 2000	0	0.06	< 0.02	< 0.01	< 0.01	0.06
				7	0.03	< 0.02	< 0.01	< 0.01	0.03
				14	0.01	< 0.02	< 0.01	< 0.01	0.01
				21	0.02	< 0.02	< 0.01	< 0.01	0.02
				28	0.03	< 0.02	< 0.01	< 0.01	0.03

Method LODs were for 0.002, 0.005, 0.002, and 0.001 mg/kg for fluxapyroxad, M700F002, M700F008 and M700F048 respectively, while the LOQs were 0.01, 0.025, 0.01, and 0.005 mg/kg (all values in parent equivalents)

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Table 3 Residues of fluxapyroxad and metabolites in orange whole fruit, peel and pulp [b]

Location, Year (variety)	Application				Fraction	Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA		Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Concordia, Entre Rios, Argentina, 2013 (Valencia)	3 (28, 28)	50, 50, 50	2000, 2000, 2000	14	Whole fruit	0.06	< 0.02	< 0.01	< 0.01	0.06
				14	Peel	0.31	< 0.02	< 0.01	< 0.01	0.31
				14	Pulp	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Federacion, Entre Rios, Argentina, 2013 (Valencia)	3 (28, 28)	50, 50, 50	2000, 2000, 2000	14	Whole fruit	0.16	< 0.02	< 0.01	< 0.01	0.16
				14	Peel	0.17	< 0.02	< 0.01	< 0.01	0.17
				14	Pulp	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Jaguapita, Sao Paolo, Brazil, 2013 (Parana)	3 (28, 28)	50, 50, 50	2000, 2000, 2000	14	Whole fruit	0.05	< 0.02	< 0.01	< 0.01	0.05
				14	Peel	0.35	< 0.02	< 0.01	< 0.01	0.35
				14	Pulp	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Cambe, Parana, Brazil, 2013	3 (28, 28)	50, 50, 50	2000, 2000, 2000	14	Whole fruit	0.07	< 0.02	< 0.01	< 0.01	0.07

Location, Year (variety)	Application				Fraction	Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA		Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
(Valencia)										
				14	Peel	0.11	< 0.02	< 0.01	< 0.01	0.11
				14	Pulp	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

[b] Residues in whole fruit were determined by analyses of separate sub-samples, not by summing the residues in peel and pulp after adjusting for the mass fraction of each portion. Hence, residues in whole fruit may not correspond with the values expected based on typical proportions of peel and pulp in oranges.

Table 4 Residues of fluxapyroxad and metabolites in lemon whole fruit, peel and pulp [b]

Location, Year (variety)	Application				Fraction	Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA		Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Concordia, Entre Rios, Argentina, 2013 (Eureka)	3 (28, 28)	50, 50, 50	2000, 2000, 2000	14	Whole fruit	0.13	< 0.02	< 0.01	< 0.01	0.13
				14	Peel	0.20	< 0.02	< 0.01	< 0.01	0.20
				14	Pulp	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Federacion, Entre Rios, Argentina, 2013 (Eureka)	3 (28, 28)	50, 50, 50	2000, 2000, 2000	14	Whole fruit	0.09	< 0.02	< 0.01	< 0.01	0.09
				14	Peel	0.32	< 0.02	< 0.01	< 0.01	0.32
				14	Pulp	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

[b] Residues in whole fruit were determined by analyses of separate sub-samples, not by summing the residues in peel and pulp after adjusting for the mass fraction of each portion. Hence, residues in whole fruit may not correspond with the values expected based on typical proportions of peel and pulp in lemons.

Table 5 Residues of fluxapyroxad and metabolites in limes (whole fruit)

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Estrela do Sul, Minas Gerais, Brazil, 2013 (Tahitian)	3 (29, 27)	50, 50, 50	2000, 2000, 2000	0	0.05	< 0.02	< 0.01	< 0.01	0.05
				7	0.06	< 0.02	< 0.01	< 0.01	0.06
				14	0.04	< 0.02	< 0.01	< 0.01	0.04
				21	0.02	< 0.02	< 0.01	< 0.01	0.02
				28	0.02	< 0.02	< 0.01	< 0.01	0.02
Jaitaizinho, Parana, Brazil, 2013 (Tahitian)	3 (28, 28)	50, 50, 50	2000, 2000, 2000	0	0.10	< 0.02	< 0.01	< 0.01	0.10
				7	0.06	< 0.02	< 0.01	< 0.01	0.06
				14	0.06	< 0.02	< 0.01	< 0.01	0.06
				21	0.05	< 0.02	< 0.01	< 0.01	0.05
				28	0.03	< 0.02	< 0.01	< 0.01	0.03

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Stone fruits

Residue data from trials in cherries, peaches and plums considered by the 2012 Meeting are tabulated below.

Table 6 Residues from the foliar application of fluxapyroxad to cherries in the USA and Canada (Jordan 2010, 2009/7003328 and Schreier 2012, 2011/7004953)

Study No. Trial No. Country Year (Variety)	Application				Matrix	PHI days	Residues (mg/kg)				
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
GAP, USA	3		121-123			0					
2009/7003328 RCN R080182 USA (Allegan, Michigan) 2008 (Tart-Montmorency)	3	6 6	121 128 129 378	679 716 712	Fruit	0	1.05	< LOD	0.21	0.05	1.31
						1	1.10	< LOD	0.24	0.04	1.38
						7	0.32	< LOD	0.25	0.07	0.63
						14	0.09	< LOD	0.18	0.07	0.33
	3	6 6	119 128 129 376	1455 1540 1532	Fruit	0	0.86	< LOD	0.25	0.05	1.16
						1	0.78	< LOD	0.25	0.06	1.08
						7	0.32	< LOD	0.23	0.09	0.62
						14	0.12	< LOD	0.16	0.10	0.36
2009/7003328 RCN R080183 Canada (Niagara,	3	8 6	127 125 126 378	610 599 608	Fruit	0	0.43	< LOD	0.17	< 0.01	0.61
						1	(0.58, 0.52) 0.55	< LOD	0.16	< 0.01	0.72
						7	(0.31, 0.48)	< LOD	0.19	0.01	0.61

Study No. Trial No. Country Year (Variety)	Application				Matrix	PHI days	Residues (mg/kg)				
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Ontario) 2008 (Tart—Montmorency)							0.40				
						14	0.14	< LOD	0.26	< 0.01	0.41
	3	8 6	124 126 124 374	1194 1207 1190	Fruit	0	(0.05, 0.05) 0.05	< LOD	(0.21, 0.15, 0.14) 0.17	(0.04, 0.03, 0.03) 0.03	0.25
						1	0.20	< LOD	0.30	0.05	0.55
						7	0.02	< LOD	0.11	0.06	0.17
						14	0.06	< LOD	0.14	0.10	0.28
2009/7003328 RCN R080184 USA (Ottawa, Michigan) 2008 (Sweet—Sams)	3	6 7	125 125 125 375	723 719 708	Fruit	0	0.53	< LOD	0.17	< 0.01	0.71
						1	0.51	< LOD	0.17	< 0.01	0.69
						7	0.18	< LOD	0.23	< 0.01	0.42
						14	0.59	< LOD	0.18	< 0.01	0.78
	3	7 7	123 125 124 372	1751 1742 1697	Fruit	0	0.34	< LOD	0.19	< 0.01	0.54
						1	0.36	< LOD	0.17	< 0.01	0.54
						7	0.12	< LOD	0.19	< 0.01	0.32
						14	0.02	< LOD	0.16	< 0.01	0.19
2009/7003328 RCN R080185 USA (Tulare, California) 2008 (Sweet—Tulare)	3	7 7	123 123 124 370	769 789 796	Fruit	0	0.82	< 0.01	0.30	< 0.01	1.13
						1	0.37	< LOD	0.24	< 0.01	0.62
						7	0.12	< LOD	0.30	< 0.01	0.43
						14	0.07	< LOD	0.28	< 0.01	0.36
	3	7 7	124 125 124 373	1957 1887 1961	Fruit	0	0.39	< LOD	0.22	< 0.01	0.62
						1	0.41	< 0.01	0.23	< 0.01	0.65
						7	0.16	< 0.01	0.29	< 0.01	0.46
						14	0.14	< 0.01	0.29	< 0.01	0.44
2009/7003328 RCN R080186 USA (Grant, Washington) 2008 (Tart—Montmorency)	3	7 7	125 125 125 375	702 703 701	Fruit	0	0.49	< LOD	0.16	0.08	0.72
						1	0.38	< 0.01	0.17	0.07	0.61
						7	0.19	< LOD	0.23	0.08	0.49
						13	0.10	< LOD	0.16	0.11	0.35
	3	7 7	123 123 123 369	1869 1871 1872	Fruit	0	0.56	< LOD	0.13	0.05	0.73
						1	0.49	< LOD	0.15	0.05	0.69
						7	0.33	< LOD	0.19	0.08	0.59
						13	0.30	< LOD	0.15	0.10	0.53
2009/7003328 RCN R080187 USA (Wasco, Oregon) 2008 (Sweet—Lapin)	3	8 6	126 127 125 378	492 640 501	Fruit	0	0.19	< LOD	0.16	< 0.01	0.36
						1	0.19	< LOD	0.18	< LOD	0.38
						7	0.08	< LOD	0.21	< 0.01	0.30
						10	0.06	< LOD	0.26	< 0.01	0.33
						14	0.04	< LOD	0.13	< 0.01	0.18
	3	8 6	128 121 126 375	1554 1595 1623	Fruit	0	0.31	< LOD	0.18	< 0.01	0.50
						1	0.20	< LOD	0.19	< 0.01	0.40
						7	0.18	< LOD	0.22	< 0.01	0.41
						10	0.11	< LOD	0.22	< 0.01	0.34
						14	0.05	< LOD	0.11	< 0.01	0.16
2011/7004953 R110214 USA (Fennville, Michigan)	3	7 7	124 124 124 372	711 686 711	Fruit	0	(0.26, 0.25) 0.26	(< LOQ, < LOQ) < LOQ	(0.10, 0.074) 0.087	(0.028, 0.023) 0.026	0.37
						1	(0.29, 0.20) 0.25	(< LOQ, < LOQ)	(0.098, 0.085)	(0.030, 0.026) 0.028	0.37

Study No. Trial No. Country Year (Variety)	Application				Matrix	PHI days	Residues (mg/kg)				
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
2011 (Tart—Montmorency)								< LOQ	0.092)		
						7	(0.15, 0.18) 0.17	(< LOQ, < LOQ) < LOQ	(0.13, 0.17) 0.15	(0.048, 0.052) 0.050	0.37
2011/7004953 R110229 USA (Hotchkiss, Colorado) 2011 (Tart—Montmorency)	3	7 6	126 123 124 373	699 683 692	Fruit	0	(1.93, 1.80) 1.87	(< LOQ, < LOQ) < LOQ	(0.42, 0.43) 0.43	(0.022, 0.021) 0.022	2.32
						1	(1.03, 1.44) 1.24	(< LOQ, < LOQ) < LOQ	(0.34, 0.38) 0.36	(0.024, 0.027) 0.026	1.63
						7	(0.82, 0.75) 0.79	(< LOQ, < LOQ) < LOQ	(0.52, 0.64) 0.58	(0.045, 0.046) 0.046	1.42

[a] All analytes are reported in terms of themselves, except for the 2011 trials where residues are expressed as parent equivalents. Total residues ((Fluxapyroxad + M700F008 + M700F048) are expressed as parent equivalents.

LOQ is 0.01 mg/kg for each of parent fluxapyroxad and metabolites M700F008, M700F002 and M700F048

LOD is 0.002 mg/kg for each of parent fluxapyroxad and metabolites M700F008, M700F002 and M700F048

Table 7 Residues from the foliar application of fluxapyroxad to peaches in the USA and Canada (Jordan 2010, 2009/7003328)

Study No. Trial No. Country Year (Variety)	Application				Matrix	PHI days	Residues (mg/kg)				
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
GAP, USA	3		121-123			0					
2009/7003328 RCN R080188 USA (Wayne, New York) 2008 (Glohaven)	3	8 6	125 125 124 374	747 747 746	Fruit	0	0.37	< LOD	0.01	< LOD	0.38
						1	0.29	< 0.01	0.02	< LOD	0.31
						7	0.07	< LOD	0.01	< 0.01	0.08
						14	0.05	< LOD	0.01	< LOD	0.06
	3	8 6	124 125 126 375	1116 1119 1129	Fruit	0	0.43	< LOD	0.01	< LOD	0.44
						1	0.43	< LOD	0.02	< LOD	0.45
						7	0.10	< LOD	0.02	< LOD	0.12
						14	0.08	< LOD	0.03	< LOD	0.11
2009/7003328 RCN R080189 USA (Tift, Georgia) 2008 (Hawthorne)	3	7 7	124 124 124 372	511 504 488	Fruit	0	0.55	< LOD	0.02	0.01	0.58
						1	0.43	< LOD	0.03	0.01	0.47
						7	0.31	< LOD	0.04	0.03	0.37
						14	0.29	< LOD	0.03	0.04	0.35
	3	7 7	126 125 126 377	1228 1189 1197	Fruit	0	0.42	< LOD	0.02	< 0.01	0.44
						1	0.37	< LOD	0.02	< 0.01	0.39
						7	0.29	< 0.01	0.10	0.02	0.40
						14	0.30	< LOD	0.05	0.04	0.38
2009/7003328 RCN R080190 USA (Brooks, Georgia) 2008 (Mid white 9A54-13)	3	7 7	126 126 124 376	522 523 521	Fruit	0	0.55	< LOD	0.06	< LOD	0.61
						1	0.29	< LOD	0.04	< LOD	0.33
						7	0.22	< LOD	0.08	< 0.01	0.30
						14	(0.12, 0.10) 0.11	< LOD	0.09	< 0.01	0.20
	3	7	125	1251	Fruit	0	(0.19, 0.17)	< LOD	0.04	< LOD	0.22

Study No. Trial No. Country Year (Variety)	Application				Matrix	PHI days	Residues (mg/kg)				
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
		7	124 124 373	1257 1265			0.18				
						1	(0.50, 0.44) 0.47	< LOD	0.06	< LOD	0.53
						7	0.57	< LOD	0.05	< LOD	0.62
						14	0.12	< LOD	0.05	< 0.01	0.17
2009/7003328 RCN R080192 USA (Lenawee, Michigan) 2008 (Redhaven)	3	7 7	125 123 126 374	930 919 912	Fruit	0	0.39	< LOD	0.02	< 0.01	0.41
						1	0.45	< LOD	0.03	< 0.01	0.48
						7	(0.14, 0.14, 0.16) 0.15	< LOD	0.03	< 0.01	0.18
						14	0.16, 0.16 (0.16)	< LOD	0.03	< 0.01	0.19
	3	7 7	124 123 128 375	2005 1993 1975	Fruit	0	0.33	< LOD	0.02	< 0.01	0.35
						1	0.26	< LOD	0.02	< LOD	0.28
						7	0.15	< LOD	0.03	< 0.01	0.18
						14	0.12	< LOD	0.03	< 0.01	0.15
2009/7003328 RCN R080193 Canada (Niagara, Ontario) 2008 (Red Star)	3	7 7	129 129 120 378	627 621 578	Fruit	0	0.10	< LOD	< 0.01	< 0.01	0.10
						1	0.19	< 0.01	< 0.01	< LOD	0.19
						6	0.08	< LOD	0.01	< LOD	0.09
						13	0.07	< 0.01	0.02	< 0.01	0.09
	3	7 7	124 125 119 368	1206 1213 1165	Fruit	0	0.26	< 0.01	0.03	< 0.01	0.29
						1	0.28	< LOD	0.02	< 0.01	0.30
						6	0.26	< 0.01	0.03	< 0.01	0.29
						13	0.19	< LOD	0.04	< 0.01	0.23
2009/7003328 RCN R080194 USA (Ottawa, Michigan) 2008 (Bellaire)	3	7 7	124 125 124 373	738 726 711	Fruit	0	0.29	< LOD	0.01	< LOD	0.30
						1	0.28	< LOD	0.01	< 0.01	0.29
						7	0.21	< LOD	0.02	< 0.01	0.23
						14	0.19	< LOD	0.02	< 0.01	0.21
	3	7 7	124 125 124 373	1787 1765 1740	Fruit	0	0.34	< LOD	< 0.01	< 0.01	0.34
						1	0.28	< LOD	0.01	< 0.01	0.29
						7	0.15	< LOD	0.01	< 0.01	0.16
						14	0.17	< LOD	0.02	< 0.01	0.19
2009/7003328 RCN R080195 USA (Marion, Illinois) 2008 (Cresthaven)	3	7 7	126 129 133 388	505 548 555	Fruit	0	0.17	< 0.01	< 0.01	< LOD	0.17
						1	0.24	< LOD	< 0.01	< LOD	0.24
						7	0.08	< 0.01	< 0.01	< LOD	0.08
						14	0.08	< LOD	< 0.01	< LOD	0.08
	3	7 7	126 125 128 379	1857 1961 1971	Fruit	0	0.32	< 0.01	0.01	< LOD	0.33
						1	0.21	< 0.01	0.01	< LOD	0.22
						7	0.15	< LOD	0.01	< LOD	0.16
						14	0.08	< 0.01	0.02	< LOD	0.10
2009/7003328 RCN R080196 USA (Pontotoc, Oklahoma) 2008 (Contender)	3	6 7	119 126 124 369	826 815 870	Fruit	0	0.44	< LOD	0.04	< LOD	0.48
						1	0.50	< LOD	0.04	< LOD	0.54
						7	0.33	< LOD	0.05	< LOD	0.38
						14	0.25	< LOD	0.06	< 0.01	0.31
	3	6 7	118 124 123	1393 1368 1414	Fruit	0	0.58	< LOD	0.08	< LOD	0.66
						1	0.42	< LOD	0.04	< LOD	0.46
						7	0.33	< LOD	0.04	< LOD	0.37

Study No. Trial No. Country Year (Variety)	Application				Matrix	PHI days	Residues (mg/kg)				
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
			365			14	0.26	< LOD	0.06	< 0.01	0.32
2009/7003328 RCN R080197 USA (Kings, California) 2008 (Klamt Cling)	3	6 7	140 141 140 421	894 900 884	Fruit	0	0.59	< LOD	0.02	< LOD	0.61
						1	0.22	< LOD	0.02	< LOD	0.24
						7	0.13	< LOD	0.02	< LOD	0.15
						10	0.26	< LOD	0.02	< LOD	0.28
						14	0.08	< LOD	0.02	< LOD	0.10
	3	6 7	141 141 140 422	1837 1837 1836	Fruit	0	0.63	< LOD	0.03	< LOD	0.66
						1	0.39	< LOD	0.03	< LOD	0.42
						7	0.23	< LOD	0.03	< LOD	0.26
						10	0.13	< LOD	0.03	< LOD	0.16
						14	0.14	< LOD	0.04	< LOD	0.18
2009/7003328 RCN R080198 USA (Stanislaus, California) 2008 (Summerset)	3	7 7	124 123 125 372	617 612 620	Fruit	0	0.30	< LOD	0.01	< LOD	0.31
						1	0.24	< LOD	0.01	< LOD	0.25
						7	(0.20, 0.20) 0.20	< LOD	0.02	< LOD	0.22
						14	0.14	< 0.01	0.02	< 0.01	0.16
	3	7 7	125 124 125 374	1574 1487 1498	Fruit	0	0.24	< LOD	0.01	< LOD	0.25
						1	0.33	< LOD	0.02	< LOD	0.35
						7	0.18	< LOD	0.01	< 0.01	0.19
						14	0.14	< LOD	0.02	< LOD	0.16
2009/7003328 RCN R080199 USA (Madera, California) 2008 (Angelus)	3	7 7	125 125 125 375	704 706 703	Fruit	0	0.30	< LOD	0.01	< 0.01	0.31
						1	0.18	< LOD	0.01	< 0.01	0.19
						7	0.13	< LOD	0.02	< 0.01	0.15
						10	(0.08, 0.08, 0.09) 0.08	< LOD	0.01	0.01	0.10
						14	0.09	< LOD	0.03	< 0.01	0.12
	3	7 7	126 126 125 377	1884 1880 1871	Fruit	0	0.26	< LOD	0.01	< 0.01	0.27
						1	0.24	< LOD	0.01	< 0.01	0.25
						7	0.24	< LOD	0.05	< 0.01	0.29
						10	0.13	< LOD	0.02	< 0.01	0.15
						14	0.12	< LOD	0.02	< 0.01	0.14
2009/7003328 RCN R080200 USA (Grant, Washington) 2008 (Snow King)	3	7 7	125 125 125 375	842 843 840	Fruit	0	0.46	< LOD	0.03	< 0.01	0.49
						1	0.55	< LOD	0.05	< 0.01	0.60
						7	0.29	< LOD	0.03	< 0.01	0.32
						14	0.19	< LOD	0.05	< 0.01	0.24
	3	7 7	124 125 124 373	1870 1890 1880	Fruit	0	0.57	< LOD	0.03	< 0.01	0.60
						1	0.59	< LOD	0.04	< 0.01	0.63
						7	0.34	< LOD	0.05	< 0.01	0.39
						14	0.25	< LOD	0.06	0.01	0.32

[a] All analytes are reported in terms of themselves. Total residues ((Fluxapyroxad + M700F008 + M700F048) are expressed as parent equivalents.

LOQ is 0.01 mg/kg for each of parent fluxapyroxad and metabolites M700F008, M700F002 and M700F048

LOD is 0.002 mg/kg for each of parent fluxapyroxad and metabolites M700F008, M700F002 and M700F048

Table 8 Residues from the foliar application of fluxapyroxad to plums in the USA and Canada (Jordan 2010, 2009/7003328)

Study No. Trial No. Country Year (Variety)	Application				Matrix	PHI days	Residues (mg/kg)				
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
GAP, USA	3		121–123			0					
2009/7003328 RCN R080201 USA (Wayne, New York) 2008 (Stanley)	3	7 6	124 124 125 373	558 558 561	Fruit	0	0.95	< LOD	< LOD	< LOD	0.95
						1	0.32	< LOD	< LOD	< LOD	0.32
						7	0.46	< LOD	< LOD	< LOD	0.46
						14	0.43	< LOD	< LOD	< LOD	0.43
	3	7 6	129 126 126 381	1119 1125 1124	Fruit	0	0.79	< LOD	< LOD	< LOD	0.79
						1	0.29	< LOD	< LOD	< LOD	0.29
						7	0.40	< LOD	< LOD	< LOD	0.40
						14	0.09	< LOD	< LOD	< LOD	0.09
2009/7003328 RCN R080202 USA (Allegan, Michigan) 2008 (Early Golden)	3	6 6	121 128 131 380	681 720 720	Fruit	0	0.49	< LOD	< LOD	< LOD	0.49
						1	0.46	< LOD	< LOD	< LOD	0.46
						7	0.30	< LOD	< 0.01	< LOD	0.30
						14	0.17	< LOD	< LOD	< LOD	0.17
	3	6 6	120 129 129 378	1469 1543 1541	Fruit	0	0.42	< LOD	< LOD	< LOD	0.42
						1	0.34	< LOD	< LOD	< LOD	0.34
						7	0.26	< LOD	< LOD	< LOD	0.26
						14	0.20	< LOD	< LOD	< LOD	0.20
2009/7003328 RCN R080203 Canada (Niagara, Ontario) 2008 (Vanette)	3	7 7	123 121 120 364	592 579 577	Fruit	0	0.20	< LOD	< LOD	< LOD	0.20
						1	0.17	< LOD	< LOD	< LOD	0.17
						7	0.11	< LOD	< LOD	< LOD	0.11
						14	0.09	< LOD	< LOD	< LOD	0.09
	3	7 7	122 121 122 365	1182 1177 1179	Fruit	0	0.24	< LOD	< LOD	< LOD	0.24
						1	0.24	< LOD	< LOD	< LOD	0.24
						7	0.14	< LOD	< LOD	< LOD	0.14
						14	0.10	< LOD	0.01	< LOD	0.11
2009/7003328 RCN R080204 USA (Ottawa, Michigan) 2008 (Stanley)	3	7 7	123 123 124 370	717 718 707	Fruit	0	0.64	< LOD	< LOD	< LOD	0.64
						1	0.62	< LOD	< LOD	< LOD	0.62
						7	0.59	< LOD	< LOD	< LOD	0.59
						14	0.49	< LOD	< LOD	< LOD	0.49
	3	7 7	124 124 125 373	1741 1749 1724	Fruit	0	0.44	< LOD	< LOD	< LOD	0.44
						1	0.42	< LOD	< LOD	< LOD	0.42
						7	0.49	< LOD	0.02	< LOD	0.51
						14	0.37	< LOD	< 0.01	< LOD	0.37
2009/7003328 RCN R080205 USA (Tulare, California) 2008 (Prunes)	3	7 7	138 140 140 418	748 755 756	Fruit	0	0.37	< LOD	< LOD	< LOD	0.37
						1	0.38	< LOD	< LOD	< LOD	0.38
						7	0.29	< LOD	< 0.01	< LOD	0.29
						10	0.26	< LOD	< LOD	< LOD	0.26
						14	0.26	< LOD	< LOD	< LOD	0.26
	3	7 7	140 140 140 420	1540 1534 1535	Fruit	0	0.32	< LOD	< LOD	< LOD	0.32
						1	0.38	< LOD	< LOD	< LOD	0.38
						7	0.32	< LOD	< LOD	< LOD	0.32
						10	0.24	< LOD	< LOD	< LOD	0.24
						14	0.28	< LOD	< LOD	< LOD	0.28

Study No. Trial No. Country Year (Variety)	Application				Matrix	PHI days	Residues (mg/kg)				
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
2009/7003328 RCN R080206 USA (Stanislaus, California) 2008 (French Plum)	3	7 7	124 123 124 371	534 533 534	Fruit	0	0.48	< LOD	< 0.01	< LOD	0.48
						1	0.47	< LOD	< 0.01	< LOD	0.47
						7	0.53	< LOD	< LOD	< LOD	0.53
						14	0.51	< LOD	< LOD	< LOD	0.51
	3	7 7	124 124 124 372	1488 1524 1525	Fruit	0	0.49	< LOD	< LOD	< LOD	0.49
						1	0.56	< LOD	< 0.01	< LOD	0.56
						7	0.47	< LOD	< LOD	< LOD	0.47
						14	0.54	< LOD	< LOD	< LOD	0.54
2009/7003328 RCN R080207 USA (Fresno, California) 2008 (Flavor Rich)	3	7 7	124 124 125 373	701 701 705	Fruit	0	0.20	< LOD	< 0.01	< LOD	0.20
						1	0.18	< LOD	< LOD	< LOD	0.18
						7	0.23	< LOD	< LOD	< LOD	0.23
						14	0.09	< LOD	< LOD	< LOD	0.09
	3	7 7	125 126 126 377	1870 1883 1885	Fruit	0	0.18	< LOD	< LOD	< LOD	0.18
						1	0.17	< LOD	< LOD	< LOD	0.17
						7	0.17	< LOD	< LOD	< LOD	0.17
						14	0.08	< LOD	< LOD	< LOD	0.08
2009/7003328 RCN R080208 USA (Madera, California) 2008 (Fortune)	3	7 7	126 128 125 379	476 473 463	Fruit	0	0.24	< LOD	< 0.01	< LOD	0.24
						1	0.27	< LOD	< LOD	< LOD	0.27
						7	0.16	< LOD	< LOD	< LOD	0.16
						14	(0.12, 0.12) 0.12	< LOD	< 0.01	(< 0.01, < 0.01) < 0.01	0.12
	3	7 7	122 125 123 370	1851 1898 1866	Fruit	0	0.14	< LOD	< LOD	< LOD	0.14
						1	0.13	< LOD	< LOD	< LOD	0.13
						7	0.13	< LOD	< LOD	< LOD	0.13
						14	0.12	< LOD	< LOD	< LOD	0.12
2009/7003328 RCN R080209 USA (Grant, Washington) 2008 (Pluot)	3	7 7	125 124 123 372	843 836 831	Fruit	0	0.30	< LOD	< 0.01	< LOD	0.30
						1	0.37	< LOD	0.02	< LOD	0.39
						7	0.15	< LOD	< 0.01	< LOD	0.15
						14	0.20	< LOD	< 0.01	< 0.01	0.20
	3	7 7	124 123 125 372	1872 1858 1885	Fruit	0	0.27	< LOD	< 0.01	< LOD	0.27
						1	0.15	< LOD	< 0.01	< LOD	0.15
						7	0.17	< LOD	< 0.01	< LOD	0.17
						14	0.13	< LOD	< 0.01	< LOD	0.13
2009/7003328 RCN R080210 USA (Polk, Oregon) 2008 (Moyer)	3	7 7	124 126 127 377	752 770 776	Fruit	0	0.30	< LOD	< 0.01	< LOD	0.30
						1	0.39	< LOD	< LOD	< LOD	0.39
						7	0.37	< LOD	< LOD	< LOD	0.37
						14	0.27	< LOD	< 0.01	< LOD	0.27
	3	7 7	124 128 129 381	1508 1555 1527	Fruit	0	0.31	< LOD	< LOD	< LOD	0.31
						1	0.55	< LOD	< LOD	< LOD	0.55
						7	0.48	< LOD	< 0.01	< LOD	0.48
						14	0.29	< LOD	< 0.01	< LOD	0.29

[a] All analytes are reported in terms of themselves. Total residues ((Fluxapyroxad + M700F008 + M700F048) are expressed as parent equivalents.

LOQ is 0.01 mg/kg for each of parent fluxapyroxad and metabolites M700F008, M700F002 and M700F048

LOD is 0.002 mg/kg for each of parent fluxapyroxad and metabolites M700F008, M700F002 and M700F048

Berries and other small fruits

Blueberries

Residue trials in blueberries (highbush type) were conducted in the USA (Korpalski, 2012-b). Three foliar applications of a 62.5 g/L EC formulation were made at each site using hand-held equipment. A spray adjuvant (non-ionic surfactant or crop oil concentrate) was included with all applications. Duplicate fruit samples were collected on the day of the last application, with additional samples being collected at intervals up to 7 days after the last application at one site in order to generate decline data.

Table 9 Residues of fluxapyroxad and metabolites in blueberries

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
New Tripoli, PA, USA, 2011 (Bluecrop)	3 (7, 7)	200, 200, 200	960, 930, 970	0	1.7 (1.7, 1.7)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.7 (1.7, 1.7)
Oglethorpe, GA, USA, 2011 (Climax)	3 (7, 7)	200, 200, 200	960, 970, 950	0	2.4 (2.2, 2.6)	< 0.02 (< 0.02, < 0.02)	0.02 (0.01, 0.02)	< 0.01 (< 0.01, < 0.01)	2.4 (2.2, 2.6)
Oglethorpe, GA, USA, 2011 (Woodward)	3 (7, 7)	200, 200, 200	970, 960, 950	0	1.6 (1.7, 1.5)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.6 (1.7, 1.5)
				1	1.7 (1.8, 1.6)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.7 (1.8, 1.6)
				3	1.2 (1.0, 1.3)	< 0.02 (< 0.02, < 0.02)	0.01 (< 0.01, 0.01)	< 0.01 (< 0.01, < 0.01)	1.2 (1.0, 1.4)
				5	0.90 (0.80, 1.0)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.90 (0.80, 1.0)
				7	0.61 (0.59, 0.63)	< 0.02 (< 0.02, < 0.02)	0.01 (0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.62 (0.60, 0.63)
White Heath, IL, USA, 2011 (Duke)	3 (7, 7)	200, 200, 210	970, 960, 980	0	3.8 (3.9, 3.6)	< 0.02 (< 0.02, < 0.02)	0.01 (0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	3.8 (3.9, 3.6)
Fremont, MI, USA, 2011 (Bluecrop)	3 (7, 7)	200, 200, 200	960, 960, 960	0	1.3 (1.2, 1.4)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.3 (1.2, 1.4)
Hillsboro, OR, USA, 2011 (Bluecrop)	3 (7, 7)	200, 200, 200	970, 950, 960	0	2.4 (2.5, 2.3)	< 0.02 (< 0.02, < 0.02)	0.02 (0.02, 0.02)	< 0.01 (< 0.01, < 0.01)	2.4 (2.5, 2.3)

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Caneberries

Residue trials in raspberries and blackberries were conducted in the USA (Korpalski, 2012-b). Three foliar applications of a 62.5 g/L EC formulation were made at each site using hand-held equipment. A spray adjuvant (crop oil concentrate or non-ionic surfactant) was included with all applications. Duplicate treated fruit samples were collected on the day of the last application, with additional

samples being collected at intervals up to 7 days after the last application at one site in order to generate decline data.

Table 10 Residues of fluxapyroxad and metabolites in blackberries and raspberries

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
BLACKBERRIES									
Hillsboro, OR, USA, 2011 (Marion)	3 (7, 7)	200, 200, 200	950, 950, 970	0	1.4 (1.2, 1.5)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.001 (< 0.001, < 0.001)	1.4 (1.2, 1.5)
RASPBERRIES									
Oglethorpe, GA, USA, 2011 (Nova)	3 (7, 7)	200, 200, 200	940, 960, 950	0	1.1 (1.3, 0.86)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.1 (1.3, 0.86)
Oglethorpe, GA, USA, 2011 (Willamette)	3 (7, 7)	200, 210, 200	960, 990, 960	0	2.0 (2.1, 1.9)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	2.0 (2.1, 1.9)
				1	1.6 (1.4, 1.8)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.6 (1.4, 1.8)
				3	1.1 (1.1, 1.1)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.1 (1.1, 1.1)
				5	1.1 (1.0, 1.1)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.1 (1.0, 1.1)
				7	0.66 (0.59, 0.73)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.66 (0.59, 0.73)

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Grapes

Residue trials in grapes were conducted in the USA (Belcher and Riley, 2012-a). Three applications of a 300 g/L SC formulation of fluxapyroxad were made at target rates of 200 g ai/ha using an airblast or backpack sprayer. An adjuvant (non-ionic surfactant, crop oil concentrate or organosiloxane) was included in all tank mixes. Duplicate treated fruit samples were collected at intervals from 0–21 days after the last application.

Table 11 Residues of fluxapyroxad and metabolites in grape berries

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Lehigh, PA, USA, 2011 (Corot Noir)	3 (10, 10)	200, 200, 200	670, 660, 650	0	0.27 (0.29, 0.24)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.27 (0.29, 0.24)
				1	0.25 (0.21, 0.28)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.25 (0.21, 0.28)

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
				7	0.18 (0.18, 0.17)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.18 (0.18, 0.17)
				14	0.13 (0.11, 0.14)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.13 (0.11, 0.14)
Yates, NY, USA, 2011 (DeChaunac)	3 (10, 11)	200, 200, 200	940, 940, 940	0	0.87 (0.89, 0.84)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.87 (0.89, 0.84)
				1	0.66 (0.69, 0.62)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.66 (0.69, 0.62)
				7	0.75 (0.80, 0.70)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.75 (0.80, 0.70)
				14	0.60 (0.41, 0.78)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.60 (0.41, 0.78)
				21	0.71 (0.81, 0.61)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.71 (0.81, 0.61)
Fresno, CA, USA, 2011 (Thompson)	3 (10, 10)	200, 210, 200	470, 480, 460	0	0.20 (0.22, 0.18)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.20 (0.22, 0.18)
				1	0.25 (0.24, 0.26)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.25 (0.24, 0.26)
				7	0.19 (0.19, 0.19)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.19 (0.19, 0.19)
				14	0.27 (0.20, 0.34)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.27 (0.20, 0.34)
				21	0.26 (0.24, 0.28)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.26 (0.24, 0.28)
Fresno, CA, USA, 2011 (Cabernet)	3 (10, 10)	200, 200, 200	1850, 1870, 1860	0	1.5 (1.7, 1.2)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.5 (1.7, 1.2)
				1	1.5 (1.5, 1.5)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.5 (1.5, 1.5)
				7	1.5 (1.7, 1.3)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.5 (1.7, 1.3)
				14	1.4 (1.3, 1.4)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.4 (1.3, 1.4)
Fresno, CA, USA, 2011 (Flame Seedless)	3 (10, 10)	200, 200, 200	1860, 1860, 1870	0	0.82 (0.82, 0.81)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.82 (0.82, 0.81)
				1	0.85 (0.90, 0.80)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.85 (0.90, 0.80)
				7	0.62 (0.64, 0.60)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.62 (0.64, 0.60)

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
				14	0.76 (0.73, 0.78)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.76 (0.73, 0.88)
Madera, CA, USA, 2011 (Ruby Red)	3 (10, 10)	210, 200, 200	480, 470, 470	0	0.21 (0.20, 0.22)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.21 (0.20, 0.22)
				1	0.16 (0.18, 0.14)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.16 (0.18, 0.14)
				7	0.13 (0.12, 0.13)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.13 (0.12, 0.13)
				14	0.11 (0.13, 0.09)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.11 (0.13, 0.09)
San Luis Obispo, CA, USA, 2011 (Marsanne)	3 (11, 10)	200, 210, 200	430, 450, 450	0	0.23 (0.27, 0.18)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.23 (0.27, 0.18)
				1	0.20 (0.19, 0.21)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.20 (0.19, 0.21)
				7	0.17 (0.15, 0.18)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.17 (0.15, 0.18)
				14	0.13 (0.18, 0.08)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.13 (0.18, 0.08)
San Luis Obispo, CA, USA, 2011 (Cabernet Sauvignon)	3 (14, 13)	200, 200, 200	1490, 1440, 1490	0	0.65 (0.66, 0.64)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.65 (0.66, 0.64)
				1	0.71 (0.75, 0.66)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.71 (0.75, 0.66)
				7	0.39 (0.30, 0.48)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.39 (0.30, 0.48)
				14	0.23 (0.34, 0.11)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.23 (0.34, 0.11)
Tulare, CA, USA, 2011 (Crimson)	3 (10, 10)	200, 200, 200	650, 650, 660	0	0.59 (0.63, 0.54)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.59 (0.63, 0.54)
				1	0.53 (0.57, 0.48)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.53 (0.57, 0.48)
				7	0.45 (0.50, 0.39)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.45 (0.50, 0.39)
				14	0.51 (0.43, 0.59)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.51 (0.43, 0.59)
Tulare, CA, USA, 2011	3 (10, 10)	200, 200, 200	2320, 2320,	0	0.45 (0.46, 0.43)	< 0.02 (< 0.02,	< 0.01 (< 0.01,	< 0.01 (< 0.01,	0.45, (0.46,

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
(Globe)			2300			< 0.02)	< 0.01)	< 0.01)	0.43)
				1	0.43 (0.48, 0.38)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.43 (0.48, 0.38)
				7	0.43 (0.42, 0.43)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.43 (0.42, 0.43)
				14	0.27 (0.28, 0.26)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.27 (0.28, 0.26)
Grant, WA, USA, 2011 (White Riesling)	3 (10, 10)	210, 210, 210	1870, 1870, 1860	0	0.57 (0.59, 0.54)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.57 (0.59, 0.54)
				1	0.47 (0.50, 0.44)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.47 (0.50, 0.44)
				7	0.48 (0.56, 0.39)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.48 (0.56, 0.39)
				14	0.43 (0.43, 0.42)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.43 (0.43, 0.42)
Washington, OR, USA, 2011 (Red Flame)	3 (7, 7)	200, 200, 200	230, 240, 240	0	0.85 (0.79, 0.91)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.85 (0.79, 0.91)
				1	0.86 (0.92, 0.79)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.86 (0.92, 0.79)
				7	0.90 (0.71, 1.1)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.90 (0.71, 1.1)
				14	0.62 (0.63, 0.61)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.62 (0.63, 0.61)

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Strawberries

Residue trials in strawberries were conducted in the USA (Korpalski, 2012-a, and Lange and Korpalski, 2013).

Three foliar applications of a 62.5 g/L EC formulation were made at each site using hand-held equipment. A spray adjuvant (non-ionic surfactant or crop oil concentrate) was included with all applications. Duplicate treated fruit samples were collected on the day of the last application, with additional samples being collected at intervals up to 7 days after the last application at one site in order to generate decline data.

Table 12 Residues of fluxapyroxad and metabolites in strawberries

Location, Year (variety)	Application				Residues, mg/kg parent equivalents			

	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
New Tripoli, PA, USA, 2011 (Earliglow)	3 (7, 7)	200, 200, 210	190, 190, 200	0	0.21 (0.23, 0.19)	< 0.01 (< 0.02, < 0.01)	0.01 (< 0.01, 0.01)	< 0.01 (< 0.01, 0.01)	0.22 (0.23, 0.21)
Winter Garden, FL, USA, 2011 (Camarosa)	3 (7, 7)	200, 200, 200	190, 190, 190	0	2.3 (2.2, 2.5)	< 0.02 (< 0.02, < 0.02)	0.02 (0.01, 0.02)	< 0.01 (< 0.01, < 0.01)	2.4 (2.2, 2.5)
Sparta, MI, USA, 2011 (Jewel)	3 (7, 7)	200, 200, 200	190, 190, 190	0	0.26 (0.28, 0.24)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.26 (0.28, 0.24)
Guadalupe, CA, USA, 2011 (Albion)	3 (7, 7)	210, 210, 210	200, 200, 190	0	0.76 (0.80, 0.72)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.76 (0.80, 0.72
Fresno, CA, USA, 2011 (Albion)	3 (7, 7)	200, 200, 200	190, 190, 190	0	0.87 (0.89, 0.84)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.87 (0.89, 0.84)
				1	0.84 (0.80, 0.87)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.84 (0.80, 0.87)
				3	0.81 (0.80, 0.81)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.81 (0.80, 0.80)
				5	0.64 (0.63, 0.65)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.64 (0.63, 0.65)
				7	0.48 (0.34, 0.61)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.48 (0.34, 0.61)
Hillsboro, OR, USA, 2011 (Fern)	3 (7, 7)	200, 200, 200	190, 190, 190	0	0.97 (1.0, 0.90)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.97 (1.0, 0.90)
Sorrento, FL, USA, 2012 (Radiance)	3 (7, 7)	220, 200, 200	200, 190, 190	0	0.76 (0.67, 0.85)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.76 (0.67, 0.85)
				1	0.62 (0.64, 0.59)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.62 (0.64, 0.59)
Sanger, CA, USA, 2012 (Albion)	3 (7, 7)	200, 200, 200	190, 190, 180	0	0.94 (0.87, 1.0)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.94 (0.87, 1.0)
				1	1.0 (0.91, 1.1)	< 0.02 (< 0.02, < 0.02)	0.01 (< 0.01, 0.01)	< 0.01 (< 0.01, < 0.01)	1.0 (0.91, 1.1)

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Tropical fruit—inedible peel

Banana

A total of 12 trials was conducted in bananas in Brazil (Guimaraes, 2013-a), Costa Rica, Ecuador and Colombia (Guimaraes, 2013-b). Four applications of a 300 g/L SC formulation were made at a target rate of 150 g ai/ha using a pressurised backpack sprayer. A mineral oil and an emulsifier were included in the spray tank for each application. Prior to application, half the fruits in each plot were covered with plastic bags. Bananas (both bagged and unbagged) were sampled at 0, 1, 5 and 10 days after the last application for the decline trials, and at day 0 only for the single point trials. In the single point trials, separate analyses of peel and pulp were conducted.

Table 13 Residues of fluxapyroxad and metabolites in banana (Brazilian trials, Guimaraes, 2013-a)

Location, Year (variety)	Application				Sample	Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ ha	Spray volume (L/ha)	DALA		Parent	M700 F002	M700 F008	M700 F048	Total [a]
Sao Francisco, Sao Paolo, Brazil, 2013 (Maçã)	4 (12, 13, 11)	150, 150, 150, 150	25, 25, 25, 25	0	Unbagged fruit	0.22 (0.22, 0.22, 0.21)	< 0.02	< 0.01	< 0.01	0.22 (0.22, 0.22, 0.21)
				1	Unbagged fruit	0.36 (0.42, 0.31, 0.36)	< 0.02	< 0.01	< 0.01	0.36 (0.42, 0.31, 0.36)
				5	Unbagged fruit	0.30 (0.25, 0.32, 0.32)	< 0.02	< 0.01	< 0.01	0.30 (0.25, 0.32, 0.32)
				10	Unbagged fruit	0.21 (0.22, 0.20, 0.21)	< 0.02	< 0.01	< 0.01	0.21 (0.22, 0.20, 0.21)
				0	Bagged fruit	0.12	< 0.02	< 0.01	< 0.01	0.12
				1	Bagged fruit	0.04	< 0.02	< 0.01	< 0.01	0.04
				5	Bagged fruit	0.03	< 0.02	< 0.01	< 0.01	0.03
				10	Bagged fruit	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Palmeira d'Oeste, Sao Paolo, Brazil, 2013 (Maçã)	4 (12, 13, 11)	150, 150, 150, 150	25, 25, 25, 25	0	Unbagged fruit	0.77 (0.87, 0.69, 0.74)	< 0.02	< 0.01	< 0.01	0.77 (0.87, 0.69, 0.74)
				1	Unbagged fruit	0.56 (0.58, 0.52, 0.59)	< 0.02	< 0.01	< 0.01	0.56 (0.58, 0.52, 0.59)
				5	Unbagged fruit	0.63 (0.71, 0.57, 0.61)	< 0.02	< 0.01	< 0.01	0.63 (0.71, 0.57, 0.61)
				10	Unbagged fruit	0.46 (0.54, 0.43, 0.40)	< 0.02	< 0.01	< 0.01	0.46 (0.54, 0.43, 0.40)
				0	Bagged fruit	0.13	< 0.02	< 0.01	< 0.01	0.13
				1	Bagged fruit	0.06	< 0.02	< 0.01	< 0.01	0.06
				5	Bagged fruit	0.04	< 0.02	< 0.01	< 0.01	0.04
				10	Bagged fruit	0.03	< 0.02	< 0.01	< 0.01	0.03
Londrina, Parana, Brazil, 2013 (Grande	4 (12, 12, 12)	150, 150, 150, 150	25, 25, 25, 25	0	Unbagged fruit	0.04	< 0.02	< 0.01	< 0.01	0.04

Location, Year (variety)	Application				Sample	Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA		Parent	M700 F002	M700 F008	M700 F048	Total [a]
Naine)										
				1	Unbagged fruit	0.06	< 0.02	< 0.01	< 0.01	0.06
				5	Unbagged fruit	0.07	< 0.02	< 0.01	< 0.01	0.07
				10	Unbagged fruit	0.02	< 0.02	< 0.01	< 0.01	0.02
				0	Bagged fruit	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				1	Bagged fruit	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				5	Bagged fruit	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				10	Bagged fruit	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Ibipora, Parana, Brazil, 2013 (Grande Naine)	4 (12, 12, 12)	150, 150, 150, 150	25, 25, 25, 25	0	Unbagged fruit	0.14	< 0.02	< 0.01	< 0.01	0.14
				1	Unbagged fruit	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				5	Unbagged fruit	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				10	Unbagged fruit	0.01	< 0.02	< 0.01	< 0.01	0.01
				0	Bagged fruit	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				1	Bagged fruit	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				5	Bagged fruit	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				10	Bagged fruit	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01

Residues were largely undetected in the untreated control samples, with a few detections at levels < LOQ

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Table 14 Residues of fluxapyroxad and metabolites in bananas (Costa Rica, Ecuador and Colombia, Guimaraes, 2013-b)

Location, Year (variety)	Application				Sample	Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA		Parent	M700 F002	M700 F008	M700 F048	Total [a]
Unbagged fruit										
Cariari, Pococi, Limón, Costa Rica, 2013 (Cavendish)	4 (12, 12, 12)	150, 150, 160, 160	24, 25, 27, 27	0	Whole fruit	0.07	< 0.02	< 0.01	< 0.01	0.07
				1	Whole fruit	0.07	< 0.02	< 0.01	< 0.01	0.07

Location, Year (variety)	Application				Sample	Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA		Parent	M700 F002	M700 F008	M700 F048	Total [a]
				5	Whole fruit	0.08	< 0.02	< 0.01	< 0.01	0.08
				10	Whole fruit	0.05	< 0.02	< 0.01	< 0.01	0.05
Carrandi, Matina, Limón, Costa Rica, 2013 (Cavendish)	4 (12, 12, 12)	160, 150, 140, 150	27, 25, 24, 25	0	Whole fruit	0.10	< 0.02	< 0.01	< 0.01	0.10
				0	Peel	0.85	< 0.02	< 0.01	< 0.01	0.85
				0	Pulp	0.06	< 0.02	< 0.01	< 0.01	0.06
Bataan, Matina, Limón, Costa Rica, 2013 (Cavendish)	4 (12, 12, 12)	160, 160, 150, 150	27, 26, 25, 26	0	Whole fruit	0.06	< 0.02	< 0.01	< 0.01	0.06
				0	Peel	0.10	< 0.02	< 0.01	< 0.01	0.10
				0	Pulp	0.03	< 0.02	< 0.01	< 0.01	0.03
Triunfo, Guayas, Ecuador, 2013 (Williams)	4	150, 150, 150, 150	25, 25, 25, 25	0	Whole fruit	1.6	< 0.02	< 0.01	< 0.01	1.6
				0	Peel	1.0	< 0.02	< 0.01	< 0.01	1.0
				0	Pulp	0.09	< 0.02	< 0.01	< 0.01	0.09
Triunfo, Guayas, Ecuador, 2013 (Williams)	4	150, 150, 150, 150	25, 25, 25, 25	0	Whole fruit	0.17	< 0.02	< 0.01	< 0.01	0.17
				0	Peel	0.22	< 0.02	< 0.01	< 0.01	0.22
				0	Pulp	0.01	< 0.02	< 0.01	< 0.01	0.01
Setor Rancho Grande, Canar, Ecuador (Williams)	4	150, 150, 150, 150	25, 25, 25, 25	0	Whole fruit	0.16	< 0.02	< 0.01	< 0.01	0.16
				0	Peel	0.24	< 0.02	< 0.01	< 0.01	0.24
				0	Pulp	0.03	< 0.02	< 0.01	< 0.01	0.03
Zona Bananera Rio Frio, Zona Bananera Sector Centro, Colombia, 2013 (Gran Enano)	4	150, 150, 150, 150	25, 25, 25, 25	0	Whole fruit	0.66	< 0.02	< 0.01	< 0.01	0.66
				0	Peel	1.1 c0.01	< 0.02	< 0.01	< 0.01	1.1
				0	Pulp	0.10	< 0.02	< 0.01	< 0.01	0.10
S.A. Macondo, Zona Bananera, Sector Sur, Colombia, 2013 (Williams)	4	150, 150, 150, 150	25, 25, 25, 25	0	Whole fruit	0.15	< 0.02	< 0.01	< 0.01	0.15

Location, Year (variety)	Application				Sample	Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA		Parent	M700 F002	M700 F008	M700 F048	Total [a]
				0	Peel	0.34	< 0.02	< 0.01	< 0.01	0.34
				0	Pulp	0.05	< 0.02	< 0.01	< 0.01	0.05
Bagged fruit										
Cariari, Pococi, Limón, Costa Rica, 2013 (Cavendish)	4	150, 150, 150, 150	25, 25, 25, 25	0	Whole fruit	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				1	Whole fruit	0.01	< 0.02	< 0.01	< 0.01	0.01
				5	Whole fruit	0.02	< 0.02	< 0.01	< 0.01	0.02
				10	Whole fruit	0.01	< 0.02	< 0.01	< 0.01	0.01
Carrandi, Matina, Limón, Costa Rica, 2013 (Cavendish)	4	150, 150, 150, 150	25, 25, 25, 25	0	Whole fruit	0.02	< 0.02	< 0.01	< 0.01	0.02
				0	Peel	0.03	< 0.02	< 0.01	< 0.01	0.03
				0	Pulp	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Bataan, Matina, Limón, Costa Rica, 2013 (Cavendish)	4	150, 150, 150, 150	25, 25, 25, 25	0	Whole fruit	0.02	< 0.02	< 0.01	< 0.01	0.02
				0	Peel	0.02	< 0.02	< 0.01	< 0.01	0.02
				0	Pulp	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Triunfo, Guayas, Ecuador, 2013 (Williams)	4	150, 150, 150, 150	25, 25, 25, 25	0	Whole fruit	0.03	< 0.02	< 0.01	< 0.01	0.03
				0	Peel	0.12	< 0.02	< 0.01	< 0.01	0.12
				0	Pulp	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Triunfo, Guayas, Ecuador, 2013 (Williams)	4	150, 150, 150, 150	25, 25, 25, 25	0	Whole fruit	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				0	Peel	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				0	Pulp	< 0.002	< 0.02	< 0.01	< 0.01	< 0.002
Setor Rancho Grande, Canar, Ecuador (Williams)	4	150, 150, 150, 150	25, 25, 25, 25	0	Whole fruit	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				0	Peel	0.04	< 0.02	< 0.01	< 0.01	0.04
				0	Pulp	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Zona Bananera Rio Frio, Zona Bananera Sector Centro, Colombia, 2013 (Gran	4	150, 150, 150, 150	25, 25, 25, 25	0	Whole fruit	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01

Location, Year (variety)	Application				Sample	Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA		Parent	M700 F002	M700 F008	M700 F048	Total [a]
Enano)										
				0	Peel	0.02	< 0.02	< 0.01	< 0.01	0.02
				0	Pulp	< 0.002	< 0.02	< 0.01	< 0.01	< 0.002
S.A. Macondo, Zona Bananera, Sector Sur, Colombia, 2013 (Williams)	4	150, 150, 150, 150	25, 25, 25, 25	0	Whole fruit	0.02	< 0.02	< 0.01	< 0.01	0.02
				0	Peel	0.05	< 0.02	< 0.01	< 0.01	0.05
				0	Pulp	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01

Residues were largely undetected in the untreated control samples, with a few detections at levels < LOQ and a single detection at the LOQ (noted above)

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Mango

Four trials in mangoes were conducted in Brazil (Dantas and Cardoso, 2012). Four applications of an SC formulation containing 333 g/L pyraclostrobin + 167 g/L fluxapyroxad were made a target rate of 0.4 L/ha (0.133 kg ai/ha pyraclostrobin + 0.067 kg ai/ha fluxapyroxad) and a target interval of 7 days. Fruit was sampled 7 days after the last application, with additional samples being collected at intervals from 0–14 days at two sites to generate decline data.

Table 15 Residues of fluxapyroxad and metabolites in mangoes

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
San Antonio de Posse, Sao Paolo, Brazil, 2010 (Palmer)	4 (8, 6, 8)	67, 67, 67, 67	1000, 1000, 1000, 1000	0	0.13	< 0.02	< 0.01	< 0.01	0.13
				3	0.14	< 0.02	< 0.01	< 0.01	0.14
				7	0.14	< 0.02	< 0.01	< 0.01	0.14
				10	0.07	< 0.02	< 0.01	< 0.01	0.07
				14	0.08	< 0.02	< 0.01	< 0.01	0.08
Anapolis, Goiana, Brazil, 2010 (Tommy)	4 (10, 4, 7)	67, 67, 67, 67	1000, 1000, 1000, 1000	0	0.33	< 0.02	< 0.01	< 0.01	0.33
				3	0.31	< 0.02	< 0.01	< 0.01	0.31
				7	0.39	< 0.02	< 0.01	< 0.01	0.39
				10	0.21	< 0.02	< 0.01	< 0.01	0.21
				14	0.23	< 0.02	< 0.01	< 0.01	0.23

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Conchal, Sao Paolo, Brazil, 2010 (Palmer)	4 (7, 7, 7)	67, 67, 67, 67	1000, 1000, 1000, 1000	7	0.21	< 0.02	< 0.01	< 0.01	0.21
Jaboticabal, Sao Paolo, Brazil, 2010 (Tommy)	4 (7, 7, 7)	67, 67, 67, 67	1000, 1000, 1000, 1000	7	0.16	< 0.02	< 0.01	< 0.01	0.16

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad.

Papaya

Four trials in papaya were conducted in Brazil (Jones, 2011). Four applications of an SC formulation containing 333 g/L pyraclostrobin and 167 g/L fluxapyroxad were made at a target rate of 50 g ai/ha fluxapyroxad (and 100 g ai/ha pyraclostrobin) at target intervals of 7 days using backpack sprayers. Spray adjuvants were not used in any of the applications. Fruit samples were collected at 7 days after the last application, with additional samples being collected at 0 and 14 days after the last application at the decline trial sites.

Table 16 Residues of fluxapyroxad and metabolites in papaya

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Linhares, Espirito Santo, Brazil, 2011 (Golden)	4 (7, 7, 7)	50, 50, 50, 50,	1000, 1000, 1000, 1000	0	0.24	< 0.02	< 0.01	< 0.01	0.24
				7	0.23	< 0.02	< 0.01	< 0.01	0.23
				14	0.19	< 0.02	0.01	< 0.01	0.20
Sooretama, Espirito Santo, Brazil, 2011 (Golden)	4 (8, 6, 7)	50, 50, 50, 50	1000, 1000, 1000, 1000	0	0.37	< 0.02	< 0.01	< 0.01	0.37
				7	0.24	< 0.02	< 0.01	< 0.01	0.24
				14	0.23	< 0.02	< 0.01	< 0.01	0.23
Pinheiros, Espirito Santo, Brazil, 2011 (THB)	4 (8, 6, 7)	50, 50, 50, 50	1000, 1000, 1000, 1000	7	0.15	< 0.02	< 0.01	< 0.01	0.15
Bela Vista do Paraiso, Parana, Brazil, 2011 (Formosa)	4 (7, 7, 7)	50, 50, 50, 50	1000, 1000, 1000, 1000	7	0.02	< 0.02	< 0.01	< 0.01	0.02

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Bulb vegetables

Bulb onion

A series of trials in dry bulb onions was conducted in the USA (Csinos, 2012-a). Three foliar broadcast applications of a 62.5 g/L EC formulation were made at a target rate of 200 g ai/ha and a target interval of 7 days using pressurised backpack sprayers. Duplicate treated samples were collected at 7 days after the last application, with additional samples being collected at intervals from 0 to 14 days at one site to generate decline data.

Table 17 Residues of fluxapyroxad and its metabolites in bulb onions

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Germansville, PA. USA, 2011 (Stuttgarter)	3 (7, 6)	210, 210, 210	310, 310, 310	7	0.16 (0.19, 0.13)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.16 (0.19, 0.13)
Lebanon, OK, USA, 2011 (Walla Walla/Sweet Red/Sweet Jumbo/Red Candy Apple)	3 (7, 7)	210, 210, 210	320, 330, 320	0	0.20 (0.18, 0.21)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.20 (0.18, 0.21)
				3	0.16 (0.17, 0.15)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.16 (0.17, 0.15)
				7	0.23 (0.21, 0.25)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.23 (0.21, 0.25)
				10	0.08 (0.09, 0.06) c0.01	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.08 (0.09, 0.06)
				14	0.14 (0.13, 0.14)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.14 (0.13, 0.14)
Claude, TX, USA, 2011 (not specified)	3 (8, 7)	200, 210, 280	340, 340, 380	7	0.03 (0.03, 0.03)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.03 (0.03, 0.03)
Guadalupe, CA, USA, 2011 (Renegade)	3 (7, 7)	200, 200, 200	280, 280, 280	7	0.16 (0.16, 0.16)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.16 (0.16, 0.16)
Guadalupe, CA, USA, 2011 (Candy)	3 (7, 7)	200, 200, 200	280, 280, 280	7	0.23 (0.23, 0.22)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.23 (0.23, 0.22)
Malin, OR, USA, 2011 (Gilroy 550)	3 (7, 7)	200, 200, 210	280, 280, 290	7	0.27 (0.28, 0.26)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.27 (0.28, 0.26)

Residues were mostly undetectable in the untreated control samples, with a few detections below the LOQ, and a single detection of parent compound at the LOQ (noted above)

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Green onion

A series of trials in green onions was conducted in the USA (Csinos, 2012-a). Three foliar broadcast applications of a 62.5 g/L EC formulation were made at a target rate of 200 g ai/ha and a target interval of 7 days using pressurised backpack sprayers. Duplicate treated samples were collected at 7 days after the last application, with additional samples being collected at intervals from 0 to 14 days at one site to generate decline data.

Table 18 Residues of fluxapyroxad and its metabolites in green onions

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Pilot Point, TX, USA, 2011 (Walla Walla/Sweet Red/Sweet Jumbo/Red Candy Apple)	3 (7, 7)	210, 200, 210	320, 310, 330	7	0.24 (0.24, 0.23)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.24 (0.24, 0.23)
Yuba City, CA, USA, 2011 (White Bunching)	3 (6, 7)	200, 200, 200	280, 280, 280	7	0.56 (0.38, 0.73)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.56 (0.38, 0.73)
Yuba City, CA, USA, 2011 (White Bunching)	3 (7, 7)	200, 200, 200	280, 280, 280	0	0.33 (0.33, 0.33)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.33 (0.33, 0.33)
				3	0.33 (0.31, 0.34)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.33 (0.31, 0.34)
				7	0.29 (0.29, 0.29)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.29 (0.29, 0.29)
				10	0.25 (0.21, 0.28)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.25 (0.21, 0.28)
				14	0.36 (0.34, 0.37)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.36 (0.34, 0.37)

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Brassica vegetables

Broccoli

A series of trials in broccoli was conducted in the USA during 2011 and 2012 (Schreier, 2013-a). Three foliar broadcast applications of either a 62.5 g/L EC or a 300 g/L SC formulation of fluxapyroxad were made at target rates of 100 or 200 g ai/ha and an interval of 7 days. Duplicate broccoli head samples were collected at 0 and 3 days after the last application, with additional decline samples being collected from one site.

Table 19 Residues of fluxapyroxad and its metabolites in broccoli heads

Location, Year (variety)	Application					Residues, mg/kg parent equivalents				
	Form.	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Lebanon, OK, USA, 2011 (Premium Crop)	62.5 EC	3 (7, 7)	100, 100, 98	320, 310, 300	0	1.5 (1.1, 1.9)	< 0.02 (< 0.02, < 0.02)	0.04 (0.03, 0.05)	0.12 (0.04, 0.19)	1.7 (1.2, 2.1)
					1	1.9 (1.7, 2.1)	< 0.02 (< 0.02, < 0.02)	0.09 (0.09, 0.08)	0.15 (0.15, 0.14)	2.1 (1.9, 2.4)
					3	1.2 (1.5, 0.99)	< 0.02 (< 0.02, < 0.02)	0.09 (0.09, 0.08)	0.15 (0.16, 0.14)	1.5 (1.7, 1.2)
					5	0.98 (0.86, 1.1)	< 0.02 (< 0.02, < 0.02)	0.06 (0.06, 0.06)	0.16 (0.14, 0.18)	1.2 (1.1, 1.3)
					7	0.86 (0.85, 0.86)	< 0.02 (< 0.02, < 0.02)	0.05 (0.05, 0.05)	0.13 (0.17, 0.09)	1.0 (1.1, 1.0)
Lompoc, CA, USA, 2011 (Concord)	62.5 EC	3 (7, 7)	200, 200, 210	280, 280, 280	0	0.49 (0.53, 0.45)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.49 (0.53, 0.45)
					3	0.28 (0.28, 0.27)	< 0.02 (< 0.02, < 0.02)	0.01 (0.01, 0.01)	0.01 (< 0.01, 0.01)	0.29 (0.29, 0.29)
Lompoc, CA, USA, 2011 (Heritage)	62.5 EC	3 (7, 7)	200, 200, 210	280, 290, 280	0	0.46 (0.53, 0.39)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.46 (0.53, 0.39)
					3	0.57 (0.44, 0.70)	< 0.02 (< 0.02, < 0.02)	0.03 (0.02, 0.03)	0.01 (0.01, 0.01)	0.61 (0.47, 0.74)
Grants Pass, OR, USA, 2011 (Green Goliath)	62.5 EC	3 (7, 7)	100, 110, 100	280, 290, 280	0	0.45 (0.38, 0.52)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.45 (0.38, 0.52)
					3	0.32 (0.37, 0.27)	< 0.02 (< 0.02, < 0.02)	0.02 (0.02, 0.01)	< 0.01 (< 0.01, < 0.01)	0.34 (0.39, 0.28)
Guadalupe, CA, USA, 2012 (Heritage)	300 SC	3 (7, 7)	100, 100, 100	280, 280, 270	0	0.23 (0.22, 0.23)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.23 (0.22, 0.23)
					3	0.09 (0.12, 0.05)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.09 (0.12, 0.05)
Guadalupe, CA, USA, 2012 (Heritage)	300 SC	3 (7, 7)	100, 100, 100	290, 280, 290	0	0.09 (0.10, 0.08)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.09 (0.10, 0.08)
					3	0.35 (0.28, 0.42)	< 0.02 (< 0.02, < 0.02)	0.01 (< 0.01, 0.01)	< 0.01 (< 0.01, < 0.01)	0.36 (0.280, 0.43)

Location, Year (variety)	Application					Residues, mg/kg parent equivalents				
	For m.	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Santa Maria, CA, USA, 2012 (Patriot)	300 SC	3 (7, 7)	100, 100, 110	280, 280, 270	0	0.37 (0.47, 0.27)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.37 (0.47, 0.27)
					3	0.17 (0.12, 0.21)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.17 (0.12, 0.21)
Santa Maria, CA, USA, 2012 (Heritage)	300 SC	3 (7, 7)	100, 100, 100	280, 280, 280	0	0.49 (0.50, 0.48)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.49 (0.50, 0.48)
					3	0.10 (0.11, 0.09)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.10 (0.11, 0.09)

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Cabbage

A series of trials in cabbage was conducted in the USA during 2011 and 2012 (Schreier, 2013-a). Three foliar broadcast applications of either a 62.5 g/L EC (2011 trials) or a 300 g/L SC (2012 trials) formulation of fluxapyroxad were made at target rates of 100 or 200 g ai/ha and an interval of 7 days. Duplicate samples of cabbage heads (with and without wrapper leaves) were collected at 0 and 3 days after the last application, with additional decline samples being collected from one site.

Table 20 Residues of fluxapyroxad and its metabolites in cabbage

Location, Year (variety)	Application				Sample	Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA		Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Germansville, PA, USA, 2011 (Blue Lagoon)	3 (7, 7)	100, 100, 100	310, 310, 300	0	Heads w. wrapper leaves	0.21 (0.20, 0.21)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.21 (0.22, 0.21)
				3	Heads w. wrapper leaves	0.14 (0.14, 0.13)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.14 (0.14, 0.13)
				0	Heads w/o wrapper leaves	0.04 (0.03, 0.04)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.04 (0.03, 0.04)
				3	Heads w/o wrapper leaves	0.04 (0.04, 0.04)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.04 (0.04, 0.04)
Sycamore, GA, USA, 2011 (Bravo)	3 (7, 7)	100, 100, 100	290, 280, 280	0	Heads w. wrapper leaves	0.14 (0.15, 0.13)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.14 (0.15, 0.13)
				1	Heads	0.18 (0.16,	< 0.02	< 0.01	< 0.01	0.18

Location, Year (variety)	Application				Sample	Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA		Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
					w. wrapper leaves	0.19)	(< 0.02, < 0.02)	(< 0.01, < 0.01)	(< 0.01, < 0.01)	(0.16, 0.19)
				3	Heads w. wrapper leaves	0.11 (0.12, 0.10)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.11 (0.12, 0.10)
				5	Heads w. wrapper leaves	0.13 (0.13, 0.13)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	0.01 (0.01, 0.01)	0.14 (0.14, 0.14)
				7	Heads w. wrapper leaves	0.12 (0.12, 0.12)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	0.01 (0.01, < 0.01)	0.13 (0.13, 0.12)
				0	Heads w/o wrapper leaves	< 0.01 (< 0.01, < 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
				1	Heads w/o wrapper leaves	0.04 (0.05, 0.03)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.04 (0.05, 0.03)
				3	Heads w/o wrapper leaves	0.01 (0.01, 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.01 (0.01, 0.01)
				5	Heads w/o wrapper leaves	0.01 (0.01, < 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.01 (0.01, < 0.01)
				7	Heads w/o wrapper leaves	0.01 (0.01, 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.01 (0.01, 0.01)
Belle Glade, FL, USA, 2011 (Bravo)	3 (6, 7)	100, 100, 100	280, 280, 290	0	Heads w. wrapper leaves	0.15 (0.13, 0.17)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.15 (0.13, 0.17)
				3	Heads w. wrapper leaves	0.07 (0.09, 0.05)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.07 (0.09, 0.05)
				0	Heads w/o wrapper leaves	0.02 (0.03, 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.02 (0.03, 0.01)
				3	Heads w/o wrapper leaves	< 0.01 (< 0.01, < 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
Deerfield, MI, USA, 2011 (Bravo)	3 (6, 7)	100, 100, 100	280, 280, 280	0	Heads w. wrapper leaves	0.39 (0.34, 0.43)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.39 (0.34, 0.43)
				3	Heads w. wrapper leaves	0.11 (0.12, 0.09)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.11 (0.12, 0.09)
				0	Heads	0.04 (0.04,	< 0.02	< 0.01	< 0.01	0.04

Location, Year (variety)	Application				Sample	Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA		Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
					w/o wrapper leaves	0.04)	(< 0.02, < 0.02)	(< 0.01, < 0.01)	(< 0.01, < 0.01)	(0.04, 0.04)
				3	Heads w/o wrapper leaves	0.05 (0.04, 0.05)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.05 (0.04, 0.05)
Lebanon, OK, USA, 2011 (Copenhagen Market)	3 (7, 7)	100, 100, 100	310, 320, 310	0	Heads w. wrapper leaves	1.5 (1.9, 1.1)	< 0.02 (< 0.02, < 0.02)	0.02 (0.02, 0.02)	0.02 (0.02, 0.02)	1.5 (1.9, 1.2)
				3	Heads w. wrapper leaves	1.2 (1.2, 1.2)	< 0.02 (< 0.02, < 0.02)	0.02 (0.02, 0.02)	0.02 (0.02, 0.02)	1.3 (1.3, 1.3)
				0	Heads w/o wrapper leaves	0.20 (0.18, 0.22)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.20 (0.18, 0.22)
				3	Heads w/o wrapper leaves	0.07 (0.07, 0.07)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.07 (0.07, 0.07)
Guadalupe, CA, USA, 2011 (Pennet)	3 (7, 7)	200, 200, 200	290, 280, 280	0	Heads w. wrapper leaves	0.16 (0.13, 0.18)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.16 (0.13, 0.18)
				3	Heads w. wrapper leaves	0.07 (0.07, 0.07)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.07 (0.07, 0.07)
				0	Heads w/o wrapper leaves	0.03 (0.02, 0.03)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.03 (0.02, 0.03)
				3	Heads w/o wrapper leaves	0.01 (0.01, 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.01 (0.01, 0.01)
Guadalupe, CA, USA, 2012 (Red Jewel)	3 (7, 7)	100, 100, 100	280, 280, 270	0	Heads w. wrapper leaves	0.39 (0.35, 0.43)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.39 (0.35, 0.43)
				3	Heads w. wrapper leaves	0.22 (0.28, 0.16)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.22 (0.28, 0.16)
				0	Heads w/o wrapper leaves	0.03 (0.03, 0.03)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.03 (0.03, 0.03)
				3	Heads w/o wrapper leaves	0.04 (0.04, 0.04)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.04 (0.04, 0.04)

Residues were mostly undetectable in the untreated control samples, with the exception of two detections of parent compound below the LOQ

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Fruiting vegetables, Cucurbits

Melons, except watermelon

A series of trials in melons (cantaloupe) was conducted in the USA (Csinos, 2012-b). Three foliar broadcast applications of a 62.5 g/L EC formulation of fluxapyroxad were made using pressurised backpack handheld sprayers at a target rate of 200 g ai/ha and a target interval of 7 days. Duplicated treated samples were collected on the day of the last application, with additional samples being collected at intervals up to 7 days at one site to generate decline data.

Table 21 Residues of fluxapyroxad and its metabolites in cantaloupe (US trials)

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Chula, GA, USA, 2011 (Minerva)	3 (7, 7)	200, 200, 200	280, 280, 280	0	0.08 (0.08, 0.08)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.08 (0.08, 0.08)
Deerfield, MI, USA, 2011 (Edisto)	3 (6, 7)	200, 200, 200	290, 290, 290	0	0.05 (0.05, 0.04)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.05 (0.05, 0.04)
Madill, OK, USA, 2011 (Halona F1)	3 (6, 7)	200, 200, 200	310, 310, 310	0	0.24 (0.25, 0.23)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.24 (0.25, 0.23)
Guadalupe, CA, USA, 2011 (Primo)	3 (7, 7)	200, 200, 200	250, 250, 240	0	0.21 (0.18, 0.24)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.21 (0.18, 0.24)
Yuba City, CA, USA, 2011 (Honey Rock)	3 (7, 7)	200, 200, 200	280, 280, 280	0	0.05 (0.10, < 0.002)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.05 (0.10, < 0.002)
Yuba City, CA, USA, 2011 (Honey Rock)	3 (7, 7)	200, 200, 210	280, 280, 290	0	0.03 (0.03, 0.03)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.03 (0.03, 0.03)
				1	0.03 (0.03, 0.03)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.03 (0.03, 0.03)
				3	0.03 (0.03, 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.03 (0.03, 0.02)
				6	0.03 (0.02, 0.03)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.03 (0.02, 0.03)
				8	0.03 (0.04, 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.03 (0.04, 0.02)

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

A second series of trials was conducted in melons in Brazil (Guimaraes, 2010-a). Four foliar applications of an SC formulation (167 g/L fluxapyroxad and 333 g/L pyraclostrobin) were made at a target rate of 0.058 kg ai/ha fluxapyroxad + 0.117 kg ai/ha pyraclostrobin and a target interval of 7 days. Three trials were run as single point trials with sampling at 7 days after the last

application, and the other two were run to a reverse decline design, generating residues data for intervals of 0–28 days after the last application.

Table 22 Residues of fluxapyroxad and metabolites in melon (Brazilian trials)

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Senador Canedo, Goias, Brazil, 2010 (Gaucho)	4 (7, 7, 7)	58, 58, 58, 58	400, 400, 400, 400	0	0.03	< 0.02	< 0.01	< 0.01	0.03
				7	0.02	< 0.02	< 0.01	< 0.01	0.02
				14	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				21	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				28	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Ibipora, Parana, Brazil, 2010 (Louis)	4 (7, 7, 7)	58, 58, 58, 58	400, 400, 400, 400	0	0.09	< 0.02	< 0.01	< 0.01	0.09
				7	0.02	< 0.02	< 0.01	< 0.01	0.02
				14	0.03	< 0.01	< 0.01	< 0.01	0.03
				21	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				28	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Santo Antonio de Posse, Sao Paolo, Brazil, 2010 (Sunrise)	4 (7, 7, 7)	58, 58, 58, 58	400, 400, 400, 400	7	0.04	< 0.02	< 0.01	< 0.01	0.04
Mossoro, Rio Grande do Norte, Brazil, 2010 (Goldex)	4 (7, 7, 7)	58, 58, 58, 58	400, 400, 400, 400	7	0.04	< 0.02	< 0.01	< 0.01	0.04
Assai, Parana, Brazil, 2010 (Louis)	4 (7, 7, 7)	58, 58, 58, 58	400, 400, 400, 400	7	0.05	< 0.02	< 0.01	< 0.01	0.05

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Cucumber

A series of trials in cucumbers was conducted in the USA (Csinos, 2012-b). Three foliar broadcast applications of a 62.5 g/L EC formulation of fluxapyroxad were made using pressurised backpack handheld sprayers at a target rate of 200 g ai/ha and a target interval of 7 days. Duplicate treated samples were collected on the day of the last application, with additional samples being collected at intervals up to 7 days at one site to generate decline data.

Table 23 Residues of fluxapyroxad and its metabolites in cucumber

Location, Year (variety)	Application				Residues, mg/kg parent equivalents			

	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Sycamore, GA, USA, 2011 (Straight Eight)	3 (7, 7)	200, 200, 200	280, 280, 280	0	0.17 (0.20, 0.13)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.17 (0.20, 0.13)
				1	0.09 (0.10, 0.08)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.09 (0.10, 0.08)
				3	0.09 (0.09, 0.09)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.09 (0.09, 0.09)
				5	0.07 (0.07, 0.07)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.07 (0.07, 0.07)
				7	0.07 (0.09, 0.05)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.07 (0.09, 0.05)
Sycamore, GA, USA, 2011 (Impact)	3 (7, 7)	200, 200, 200	290, 280, 280	0	0.08 (0.10, 0.06)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.08 (0.10, 0.06)
Gainesville, FL, USA, 2011 (Impact)	3 (7, 7)	200, 200, 200	280, 280, 280	0	0.03 (0.02, 0.03)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.03 (0.02, 0.03)
Deerfield, MI, USA, 2011 (Alibi F1)	3 (7, 6)	200, 200, 200	280, 290, 290	0	0.16 (0.12, 0.19)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.16 (0.12, 0.19)
Deerfield, MI, USA, 2011 (Northern Pickling)	3 (7, 6)	200, 200, 200	280, 290, 290	0	0.17 (0.18, 0.16)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.17 (0.18, 0.16)
Madill, OK, USA, 2011 (Alibi F1)	3 (6, 7)	210, 210, 210	310, 310, 320	0	0.24 (0.25, 0.22)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.24 (0.25, 0.22)

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Squash, summer

A series of trials in summer squash was conducted in the USA (Csinos, 2012-b). Three foliar broadcast applications of a 62.5 g/L EC formulation of fluxapyroxad were made using pressurised backpack handheld sprayers at a target rate of 200 g ai/ha and a target interval of 7 days. Duplicate treated samples were collected on the day of the last application, with additional samples being collected at intervals up to 7 days at one site to generate decline data.

Table 24 Residues of fluxapyroxad and its metabolites in summer squash

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Germansville, PA, USA, 2011 (Super Pik)	3 (8, 6)	210, 210, 210	310, 310, 300	0	0.14 (0.11, 0.16)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.14 (0.11, 0.16)
Sycamore, GA, USA, 2011 (Gold Star)	3 (7, 7)	200, 200, 200	280, 290, 280	0	0.11 (0.13, 0.09)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.11 (0.13, 0.09)
				1	0.09 (0.08, 0.09)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.09 (0.08, 0.09)
				3	0.07 (0.08, 0.06)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.07 (0.08, 0.06)
				5	0.07 (0.06, 0.07)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.07 (0.06, 0.07)
				7	0.03 (0.03, 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.03 (0.03, 0.02)
Gainesville, FL, USA, 2011 (Gold Star)	3 (7, 7)	200, 200, 200	280, 280, 280	0	0.05 (0.05, 0.05)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.05 (0.05, 0.05)
Deerfield, MI, USA, 2011 (Gold Star)	3 (7, 6)	200, 200, 200	280, 290, 290	0	0.07 (0.05, 0.08)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.07 (0.05, 0.08)
Yuba City, CA, USA, 2011 (Yellow Summer Crookneck)	3 (7, 7)	220, 220, 220	280, 280, 280	0	0.10 (0.07, 0.12)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.10 (0.07, 0.12)

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Watermelon

Trials in watermelon were conducted in Brazil (Guimaraes, 2010-b). Four applications of an SC formulation containing 167 g/L fluxapyroxad and 333 g/L pyraclostrobin were made at a target rate of 0.058 kg ai/ha fluxapyroxad + 0.117 kg ai/ha pyraclostrobin and an interval of days. Two single point residue trials, with scheduled sampling at 7 days after the last application were conducted along with two reverse decline design trials, giving decline data from 0 to 10 days after the last application.

Table 25 Residues of fluxapyroxad and metabolites in watermelon (Brazilian trials)

Location, Year (variety)	Application				Residues, mg/kg parent equivalents					
	No. (RTI, days)	Rate, g ai/ ha	Spray volume (L/ha)	DALA	Sample	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]

Location, Year (variety)	Application				Residues, mg/kg parent equivalents					
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Sample	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Jaboticabal, Sao Paolo, Brazil, 2011 (Top Gun)	4 (7, 7, 7)	58, 58, 58, 58	400, 400, 400, 400	0	Peel	0.02	< 0.02	< 0.01	< 0.01	0.02
				0	Pulp	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				0	Whole fruit	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				7	Peel	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				7	Pulp	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				7	Whole fruit	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				10	Peel	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				10	Pulp	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
				10	Whole fruit	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
San Antonio de Posse, Sao Paolo, Brazil, 2010 (Rapid Fire)	4 (6-8)	58, 58, 58, 58	400, 400, 400, 400	0	Whole fruit	0.10	< 0.02	< 0.01	< 0.01	0.10
				7	Whole fruit	0.06	< 0.02	< 0.01	< 0.01	0.06
				10	Whole fruit	0.07	< 0.02	< 0.01	< 0.01	0.07
Ponta Grossa, Parana, Brazil, 2010 (Kodama)	4 (7, 7, 7)	58, 58, 58, 58	400, 400, 400, 400	7	Whole fruit	0.05	< 0.02	< 0.01	< 0.01	0.05
Senador Canedo, Goias, Brazil, 2010 (H. Elisa)	4 (6, 8, 7)	58, 58, 58, 58	400, 400, 400, 400	7	Whole fruit	0.06	< 0.02	< 0.01	< 0.01	0.06

Residues were mostly undetectable in the untreated control samples, except for one detection of parent compound at < LOQ

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Leafy vegetables

Lettuce, head

A series of trials in head lettuce was conducted in the USA (Schreier, 2013-b). Three foliar broadcast applications of a 62.5 g/L EC or a 300 g/L SC formulation were made at a target rate of 200 g ai/ha

and a target interval of 7 days using pressurised backpack sprayers. Duplicate treated samples were collected 0 and 1 day after the last application, with additional decline data samples being collected at one site.

Table 26 Residues of fluxapyroxad and its metabolites in head lettuce (heads with wrapper leaves)

Location, Year (variety)	Application					Residues, mg/kg parent equivalents			
	Formulation	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700 F008	M700 F048	Total [a]
Sycamore, GA, USA, 2011 (Iceberg)	300 SC	3 (7, 7)	200, 200, 200	280, 290, 280	0	0.45 (0.46, 0.43)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.45 (0.46, 0.43)
					1	0.51 (0.56, 0.45)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.51 (0.56, 0.45)
Belle Glade, FL, USA, 2011 (Iceberg)	300 SC	3 (6, 7)	200, 200, 200	290, 280, 280	0	0.33 (0.38, 0.28)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.33 (0.38, 0.28)
					1	0.14 (0.10, 0.18)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.14 (0.10, 0.18)
Guadalupe, CA, USA, 2011 (Escalade)	62.5 EC	3 (7, 7)	200, 200, 200	280, 280, 290	0	1.7 (1.9, 1.5)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.7 (1.9, 1.5)
					1	1.1 (0.74, 1.5)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.1 (0.74, 1.5)
Guadalupe, CA, USA, 2011 (Osoflaco)	62.5 EC	3 (7, 7)	200, 200, 200	280, 280, 290	0	3.5 (3.4, 3.6)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	3.5 (3.4, 3.6)
					1	1.9 (2.0, 1.9)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.9 (2.0, 1.9)
Lompoc, CA, USA, 2011 (Vision)	62.5 EC	3 (7, 7)	200, 200, 200	280, 280, 280	0	0.79 (0.75, 0.82)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.79 (0.75, 0.82)
					1	0.47 (0.38, 0.55)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.47 (0.38, 0.55)
Orcutt, CA, USA, 2011 (Quest)	62.5 EC	3 (7, 7)	200, 200, 200	280, 280, 280	0	2.6 (2.6, 2.7)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	2.7 (2.6, 2.7)
					1	2.0 (1.9, 2.0)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	2.0 (1.9, 2.0)
					3	0.54 (0.48, 0.60)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.54 (0.48, 0.60)
					5	0.66 (0.46, 0.86)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.66 (0.46, 0.86)
					7	0.28 (0.15, 0.40)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.28 (0.15, 0.40)

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Lettuce, leaf

A series of trials in leafy lettuce was conducted in the USA (Schreier, 2013-b). Three foliar broadcast applications of a 300 g/L SC formulation were made using pressurised backpack sprayers at a target rate of 200 g ai/ha and a target interval of 7 days. Duplicate treated samples were collected at 0 and 1 day after the last application with additional decline data samples being collected at a single site.

Table 27 Residues of fluxapyroxad and its metabolites in leafy lettuce

Location, Year (variety)	Application					Residues, mg/kg parent equivalents			
	Formulation	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F008	M700F048	Total [a]
Sycamore, GA, USA, 2011 (Romaine)	300 SC	3 (7, 7)	200, 200, 200	280, 280, 280	0	9.4 (9.2, 9.5)	0.06 (0.05, 0.07)	< 0.01 (< 0.01, < 0.01)	9.4 (9.3, 9.6)
					1	6.2 (6.5, 5.9)	0.04 (0.05, 0.03)	< 0.01 (,0.001, < 0.01)	6.2 (6.5, 5.9)
Belle Glade, FL, USA, 2011 (Romaine)	300 SC	3 (6, 7)	200, 200, 200	290, 280, 280	0	4.0 (3.8, 4.1)	0.11 (0.10, 0.12)	< 0.01 (< 0.01, < 0.01)	4.1 (3.9, 4.3)
					1	3.3 (4.2, 2.4)	0.10 (0.11, 0.08)	< 0.01 (< 0.01, < 0.01)	3.4 (4.3, 2.5)
Santa Maria, CA, USA, 2012 (Red Tide)	300 SC	3 (7, 7)	200, 200,200	280, 280, 270	0	4.3 (4.4, 4.3)	0.04 (0.04, 0.04)	< 0.01 (< 0.01, < 0.01)	4.4 (4.4, 4.4)
					1	3.5 (2.8, 4.2)	0.04 (0.04, 0.04)	< 0.01 (< 0.01, < 0.01)	3.5 (2.8, 4.3)
Santa Maria, CA, USA, 2012 (Greenstar)	300 SC	3 (7, 7)	200, 200, 200	280, 280, 270	0	4.5 (4.1, 4.8)	0.02 (0.02, 0.02)	< 0.01 (< 0.01, < 0.01)	4.5 (4.1, 4.8)
					1	4.4 (4.9, 4.0)	0.02 (0.02, 0.02)	< 0.01 (< 0.01, < 0.01)	4.4 (4.9, 4.0)
Guadalupe, CA, USA, 2012 (Berghams Green)	300 SC	3 (7, 7)	200, 200, 210	270, 280, 300	0	3.2 (3.4, 3.0)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	3.2 (3.4, 3.0)
					1	2.7 (2.7, 2.6)	0.01 (0.01, 0.01)	< 0.01 (< 0.01, < 0.01)	2.7 (2.7, 2.7)
					3	0.44 (0.44, 0.44)	0.02 (0.01, 0.02)	< 0.01 (< 0.01, < 0.01)	0.46 (0.45 , 0.46)
					5	0.33 (0.35, 0.31)	0.02 (0.02, 0.01)	< 0.01 (< 0.01, < 0.01)	0.35 (0.37 , 0.32)
					7	0.24 (0.26, 0.22)	0.02 (0.01, 0.02)	< 0.01 (< 0.01, < 0.01)	0.26 (0.27 , 0.24)
Guadalupe, CA, USA, 2012 (Green Thunder)	300 SC	3 (6, 7)	210, 200, 200	280, 270, 270	0	2.1 (2.2, 2.1)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	2.1 (2.2, 2.1)
					1	2.0 (2.0, 1.9)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	2.0 (2.0, 1.9)

Residues were generally undetectable in the untreated control samples, apart from a single detection of parent compound at a level < LOQ

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Mustard greens

A series of trials in mustard greens was conducted in the USA during 2011 (Schreier, 2013-a). Three foliar broadcast applications of a 62.5 g/L EC formulation of fluxapyroxad were made at target rates of 100 g ai/ha and an interval of 7 days. Duplicate treated leaves samples were collected at 0 and 3 days after the last application, with additional decline samples being collected from one site.

Table 28 Residues of fluxapyroxad and its metabolites in mustard greens leaves

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Sycamore, GA, USA, 2011 (Savanna)	3 (7, 7)	100, 100, 100	280, 290, 280	0	4.5 (4.8, 4.3)	< 0.02 (< 0.02, < 0.02)	0.27 (0.28, 0.26)	0.64 (0.65, 0.63)	5.5 (5.7, 5.3)
				1	2.7 (3.1, 2.4)	< 0.02 (< 0.02, < 0.02)	0.29 (0.28, 0.30)	0.64 (0.75, 0.53)	3.7 (4.1, 3.2)
				3	1.7 (1.8, 1.6)	< 0.02 (< 0.02, < 0.02)	0.42 (0.41, 0.43)	0.96 (0.90, 1.0)	3.1 (3.1, 3.1)
				5	1.0 (1.0, 0.95	< 0.02 (< 0.02, < 0.02)	0.30 (0.33, 0.26)	0.87 (0.86, 0.87)	2.2 (2.2, 2.1)
				7	0.83 (0.80, 0.85)	< 0.02 (< 0.02, < 0.02)	0.23 (0.23, 0.23)	0.89 (0.89, 0.88)	1.9 (1.9, 2.0)
Fisk, MO, USA, 2011 (Southern Giant)	3 (7, 7)	100, 100, 100	280, 280, 280	0	3.9 (4.4, 3.3)	< 0.02 (< 0.02, < 0.02)	0.10 (0.10, 0.10)	0.40 (0.38, 0.41)	4.4 (4.9, 3.9)
				3	1.9 (1.9, 1.9)	< 0.02 (< 0.02, < 0.02)	0.36 (0.34, 0.38)	0.45 (0.44, 0.45)	2.7 (2.7, 2.7)
York, NE, USA, 2011 (Green Wave)	3 (7, 7)	100, 100, 110	290, 290, 290	0	3.7 (3.5, 4.0)	< 0.02 (< 0.02, < 0.02)	0.12 (0.12, 0.12)	0.09 (0.10, 0.07)	3.9 (3.7, 4.2)
				3	0.57 (0.55, 0.58)	< 0.02 (< 0.02, < 0.02)	0.19 (0.19, 0.18)	0.18 (0.19, 0.17)	0.93 (0.93, 0.93)
Pilot Point, TX, USA, 2011 (Green Wave)	3 (7, 7)	110, 100, 110	320, 320, 320	0	6.8 (7.1, 6.5)	< 0.02 (< 0.02, < 0.02)	0.57 (0.54, 0.59)	1.3 (1.5, 1.1)	8.7 (9.1, 8.2)
				3	0.48 (0.51, 0.44)	< 0.02 (< 0.02, < 0.02)	0.25 (0.27, 0.22)	0.97 (0.93, 1.0)	1.7 (1.7, 1.7)
Yuba City, CA, USA, 2011 (India)	3 (7, 8)	100, 100, 100	280, 280, 280	0	2.0 (2.2, 1.8)	< 0.02 (< 0.02, < 0.02)	0.08 (0.09, 0.07)	0.14 (0.14, 0.13)	2.2 (2.4, 2.0)
				3	0.90 (0.84, 0.95)	< 0.02 (< 0.02, < 0.02)	0.23 (0.21, 0.24)	0.22 (0.21, 0.23)	1.3 (1.3, 1.4)

Residues were mostly undetectable in the untreated control samples, apart from a single detection of M700F008 below the LOQ

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Radish leaves

A series of trials in radish was conducted in the USA (Norris, 2012). Three applications of fluxapyroxad as a 62.5 g/L EC formulation were made a target rate of 100 g ai/ha and a target interval of 7 days. Radish roots and tops (duplicate samples) were sampled at 7 days after the last application.

Table 29 Residues of fluxapyroxad and its metabolites in radish tops

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Wayne, NY, USA, 2010 (Scarlet Globe)	3 (7, 7)	100, 98, 98	280, 270, 270	7	0.7 (0.7, 0.6)	< 0.02 (< 0.02, < 0.02)	0.3 (0.3, 0.3)	0.2 (0.2, 0.2)	1.2 (1.2, 1.1)
Martin, FL, USA, 2011 (Escala)	3 (7, 7)	99, 100, 100	280, 280, 290	7	0.2 (0.2, 0.2)	< 0.02 (< 0.02, < 0.02)	0.2 (0.2, 0.2)	0.2 (0.2, 0.2)	0.6 (0.6, 0.6)
Palm Beach, FL, USA, 2011 (Escala)	3 (7, 7)	100, 100, 100	290, 280, 290	7	0.2 (0.2, 0.1)	< 0.02 (< 0.02, < 0.02)	0.2 (0.2, 0.1)	0.07 (0.07, 0.07)	0.4 (0.5, 0.3)
Clinton, IL, USA, 2010 (Champion)	3 (6, 7)	100, 100, 100	280, 280, 280	7	4 (4, 4)	< 0.02 (< 0.02, < 0.02)	0.9 (0.8, 0.9)	0.5 (0.5, 0.6)	5 (5, 6)
Tulare, CA, USA, 2010 (Crimson Giant)	3 (7, 7)	100, 100, 100	280, 280, 280	7	1 (1, 1)	< 0.02 (< 0.02, < 0.02)	0.5 (0.5, 0.5)	0.2 (0.2, 0.2)	1.7 (1.7, 1.7)

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad equivalents

Spinach

A series of trials in spinach was conducted in the USA (Schreier, 2013-b). Three foliar broadcast applications of a 62.5 g/L EC or a 300 g/L SC formulation were made at a target rate of 200 g ai/ha and a target interval of 7 days using pressurised backpack sprayers. Duplicate treated samples were collected 0 and 1 day after the last application, with additional decline data samples being collected at one site.

Table 30 Residues of fluxapyroxad and its metabolites in spinach

Location, Year (variety)	Application					Residues, mg/kg parent equivalents			
	Formulation	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F008	M700F048	Total [a]
Guadalupe, CA, USA, 2011 (UniPak 151)	62.5 EC	3 (7, 7)	200, 200, 200	280, 280, 290	0	9.2 (9.6, 8.8)	0.11 (0.11, 0.10)	< 0.01 (< 0.01, < 0.01)	9.3 (9.7, 8.9)
					1	6.0 (6.1, 6.0)	0.23 (0.21,	< 0.01 (< 0.01,	6.3 (6.3,

Location, Year (variety)	Application					Residues, mg/kg parent equivalents			
	Formulation	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F008	M700F048	Total [a]
							0.25)	< 0.01)	6.3)
Guadalupe, CA, USA, 2011 (Avenger)	300 SC	3 (7, 7)	200, 200, 210	250, 250, 250	0	6.2 (6.0, 6.5)	0.07 (0.08, 0.06)	< 0.01 (< 0.01, < 0.01)	6.3 (6.0, 6.6)
					1	1.9 (1.8, 1.9)	0.07 (0.07, 0.07)	< 0.01 (< 0.01, < 0.01)	1.9 (1.9, 2.0)
Germansville, PA, USA, 2011 (Tyee)	62.5 EC	3 (7, 7)	210, 210, 210	310, 300, 310	0	9.8 (9.4, 10.2)	0.41 (0.39, 0.42)	< 0.01 (< 0.01, < 0.01)	10.2 (9.8, 10.6)
					1	8.3 (8.4, 8.2)	0.44 (0.42, 0.46)	< 0.01 (< 0.01, < 0.01)	8.8 (8.8, 8.7)
Lebanon, OK, USA, 2011 (Spargo F1, Tyee F1, Bloomsdale)	62.5 EC	3 (7, 7)	200, 210, 210	320, 320, 320	0	18.0 (19.5, 16.5)	0.81 (0.71, 0.91)	0.03 (0.03, 0.02)	18.8 (20.2, 17.4)
					1	11.5 (11.9, 11.0)	0.76 (0.74, 0.77)	0.02 (0.02, 0.02)	12.2 (12.7, 11.8)
Sycamore, GA, USA, 2011 (Crocodile RZ)	300 SC	3 (7, 7)	200, 200, 200	280, 290, 280	0	6.1 (6.0, 6.3)	0.04 (0.04, 0.04)	< 0.01 (< 0.01, < 0.01)	6.2 (6.0, 6.3)
					1	4.4 (4.1, 4.7)	0.05 (0.05, 0.04)	< 0.01 (< 0.01, < 0.01)	4.4 (4.1, 4.8)
					3	5.2 (4.8, 5.6)	0.06 (0.05, 0.06)	< 0.01 (< 0.01, < 0.01)	5.2 (4.8, 5.6)
					5	3.7 (3.4, 4.0)	0.06 (0.05, 0.06)	< 0.01 (< 0.01, < 0.01)	3.8 (3.5, 4.1)
					7	3.2 (3.3, 3.2)	0.03 (0.03, 0.03)	< 0.01 (< 0.01, < 0.01)	3.3 (3.3, 3.2)
Monte Vista, CO, USA, 2012 (Regiment)	300 SC	3 (7, 7)	200, 200, 200	280, 280, 280	0	7.9 (7.5, 8.3)	0.05 (0.05, 0.04)	< 0.01 (< 0.01, < 0.01)	8.0 (7.6, 8.4)
					1	6.7 (6.6, 6.9)	0.03 (0.03, 0.02)	< 0.01 (< 0.01, < 0.01)	6.8 (6.6, 6.9)

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Root and tuber vegetables

Carrot

A series of trials in carrots was conducted in the USA (Norris, 2012 and Schreier, 2015). Three applications of fluxapyroxad as a 62.5 g/L EC formulation were made a target rate of 100 g ai/ha and a target interval of 7 days. Carrot roots (duplicate samples) were sampled at 7 days after the last application, with additional samples being collected from 0-14 days at one decline trial site.

Table 31 Residues of fluxapyroxad and its metabolites in carrot roots Norris, 2012)

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Hillsborough, FL, USA, 2010 (Imperator 58)	3 (7, 7)	100, 100, 100	280, 280, 280	7	0.1 (0.1, 0.1)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.1 (0.1, 0.1)
Jefferson, IA, USA, 2010 (Nantes Scarlet)	3 (7, 7)	100, 99, 100	280, 280, 290	7	0.05 (0.04, 0.05)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.05 (0.04, 0.05)
Caddo, OK, USA, 2010 (Nantes Scarlet)	3 (7, 6)	100, 97, 100	290, 280, 270	7	0.06 (0.06, 0.06)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.06 (0.06, 0.06)
Tulare, CA, USA, 2010 (Danvers 126)	3 (7, 7)	100, 100, 100	280, 280, 280	7	0.5 (0.5, 0.5)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.5 (0.5, 0.5)
Tulare, CA, USA, 2010 (Danvers 126)	3 (7, 7)	98, 100, 100	270, 280, 280	7	0.1 (0.1, 0.1)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.1 (0.1, 0.1)
Tulare, CA, USA, 2010 (Danvers 126)	3 (7, 7)	100, 100, 100	280, 290, 290	0	0.2 (0.2, 0.2)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.2 (0.2, 0.2)
				3	0.4 (0.3, 0.4)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.4 (0.3, 0.4)
				7	0.3 (0.3, 0.3)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.3 (0.3, 0.3)
				10	0.4 (0.3, 0.4)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.4 (0.3, 0.4)
				14	0.4 (0.4, 0.3)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.4 (0.4, 0.3)
Grant, WA, USA, 2010 (Danvers 126)	3 (7, 7)	100, 100, 100	280, 280, 280	7	0.04 (0.04, 0.04)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.04 (0.04, 0.04)

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad equivalents

Table 32 Residues of fluxapyroxad and its metabolites in carrot roots (Schreier, 2015)

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Madill, OK, USA, 2014 (Danvers)	3 (7, 6)	98, 100, 100	260, 260, 250	0	0.061 (0.054, 0.068) c0.01	< 0.02 (< 0.02, < 0.02)	0.021 (0.022, 0.020) c0.016	< 0.01 (< 0.01, < 0.01)	0.082 (0.076, 0.088)
				3	0.063 (0.065, 0.060)	< 0.02 (< 0.02, < 0.02)	0.022 (0.021, 0.023)	< 0.01 (< 0.01, < 0.01)	0.085 (0.086, 0.083)
				10	0.072 (0.072, 0.071)	< 0.02 (< 0.02, < 0.02)	0.023 (0.023, 0.022)	< 0.01 (< 0.01, < 0.01)	0.094 (0.095, 0.093)
				14	0.066 (0.063, 0.069)	< 0.02 (< 0.02, < 0.02)	0.022 (0.021, 0.022)	< 0.01 (< 0.01, < 0.01)	0.088 (0.084, 0.091)

Except where noted, no residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad equivalents

Potato

A number of residue trials in potatoes were conducted in Europe (Kramm, 2013-a, and Schaufele, 2013). Applications of a 300 g/L SC formulation were made using handheld equipment, at planting. The application was made in two passes, the first in the open furrow prior to sowing the seed potatoes, and the second over the top of the seed potatoes prior to filling in the furrow. The target total rate was 0.25 kg ai/ha. Samples of tubers were collected shortly prior to and at normal harvest maturity (BBCH growth stage 47–49).

Table 33 Residues of fluxapyroxad and its metabolites in potato tubers after in-furrow treatment at planting

Location, Year (variety)	Application			Residues, mg/kg parent equivalents					
	Rate, g ai/ha	Spray volume, L/ha	DALA	Sample	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Waldsee, Germany, 2011 (Berber)	230	140	105	Immature tubers	0.02	< 0.02	< 0.01	< 0.01	0.02
			133	Mature tubers	0.04	< 0.02	< 0.01	< 0.01	0.04
Studernheim, Germany, 2011 (Belana)	260	200	92	Immature tubers	0.02	< 0.02	< 0.01	< 0.01	0.02
			120	Mature tubers	0.01	< 0.02	< 0.01	< 0.01	0.01
Leicestershire, UK, 2011 (Cara)	250	200	88	Immature tubers	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
			116	Mature tubers	0.01	< 0.02	< 0.01	< 0.01	0.01
Derbyshire, UK, 2011 (Maris Piper)	260	210	76	Immature tubers	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
			104	Mature	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01

Location, Year (variety)	Application			Residues, mg/kg parent equivalents					
	Rate, g ai/ha	Spray volume, L/ha	DALA	Sample	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
				tubers					
Ottersum, the Netherlands, 2011 (Presto)	260	100	91	Immature tubers	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
			112	Mature tubers	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Siebengeweld, the Netherlands, 2011 (Cilena)	270	110	98	Immature tubers	0.01	< 0.02	< 0.01	< 0.01	0.01
			114	Mature tubers	0.02	< 0.02	< 0.01	< 0.01	0.02
Marbais, Belgium, 2011 (Ramos)	260	160	110	Immature tubers	0.01	< 0.02	< 0.01	< 0.01	0.01
			134	Mature tubers	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Sirault, Belgium, 2011 (Bintje)	270	160	108	Immature tubers	0.03	< 0.02	< 0.01	< 0.01	0.03
			133	Mature tubers	0.04	< 0.02	< 0.01	< 0.01	0.04
Duras, France, 2012 (Mona Lisa)	280	160	57	Immature tubers	0.01	< 0.02	< 0.01	< 0.01	0.01
			77	Mature tubers	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Bonnieux, France, 2012 (Lisseta)	270	160	68	Immature tubers	0.02	0.02	< 0.01	< 0.01	0.02
			95	Mature tubers	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Nea Magnisia, Greece, 2012 (Jaerla)	250	150	70	Immature tubers	0.01	0.02	< 0.01	< 0.01	0.01
			92	Mature tubers	< 0.01	0.02	< 0.01	< 0.01	< 0.01
Platanos, Greece, 2012 (Agria)	260	150	77	Immature tubers	0.01	< 0.02	< 0.01	< 0.01	0.01
			111	Mature tubers	0.02	< 0.02	< 0.01	< 0.01	0.02
Mulazzano, Italy, 2012 (Desiree)	290	180	121	Immature tubers	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
			126	Mature tubers	0.04	< 0.02	< 0.01	< 0.01	0.04
Caleppio di Settala, Italy, 2012 (Kennebek)	260	150	106	Immature tubers	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
			112	Mature tubers	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Paterna, Spain, 2012 (Nicola)	260	160	80	Immature tubers	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
			90	Mature tubers	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Valencia, Spain, 2012 (Desiree)	250	150	81	Immature tubers	0.02	< 0.02	< 0.01	< 0.01	0.02
			94	Mature	0.03	< 0.02	< 0.01	< 0.01	0.03

Location, Year (variety)	Application			Residues, mg/kg parent equivalents					
	Rate, g ai/ha	Spray volume, L/ha	DALA	Sample	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
				tubers					

No residues were found above the LOQ in the untreated control samples

[a] Sum of parent, M700F008 and M700F048, expressed as parent, as per the residue definition for dietary risk assessment

In another study (Kramm, 2013-b), seed potatoes were treated with a 300 g/L fluxapyroxad SC formulation at a target rate of 0.006 kg ai/100 kg prior to planting. At the planting rate of 2500 kg/ha, this corresponds to a nominal application rate of 150 g ai/ha. Samples of tubers were collected shortly prior to and at normal harvest maturity (BBCH growth stage 47–49).

Table 34 Residues of fluxapyroxad and its metabolites in potato tubers after treatment of seed potatoes prior to sowing

Location, Year (variety)	Application			Residues, mg/kg parent equivalents					
	Rate, g ai/100 kg	Rate, g ai/ha	DALA	Sample	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Sturdenheim, Germany, 2012 (Nicola)	5.6	140	84	Immature tubers	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
			125	Mature tubers	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Waldsee, Germany, 2012 (Nicola)	5.6	160	89	Immature tubers	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
			129	Mature tubers	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Meauzac, France, 2012 (Nicola)	5.6	99	87	Immature tubers	0.02	< 0.02	< 0.01	< 0.01	0.02
			128	Mature tubers	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
Paterna, Spain, 2012 (Nicola)	5.6	140	82	Immature tubers	0.01	< 0.02	< 0.01	< 0.01	0.01
			93	Mature tubers	0.04	< 0.02	< 0.01	< 0.01	0.04

No residues were found above the LOQ in the untreated control samples

[a] Sum of parent, M700F008 and M700F048, expressed as parent, as per the residue definition for dietary risk assessment

Residue trials in potatoes conducted in the USA and Canada (3× 100 g ai/ha foliar applications) was considered by the 2012 Meeting and the data is reproduced below.

Table 35 Residues from the foliar application of fluxapyroxad to potatoes in the USA and Canada (Johnston and Saha 2010, 2009/7003643)

Study No. Trial No. Country Year (Variety)	Application				Matrix	PHI days	Residues (mg/kg)						
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad		M700F002	M700F008	M700F048	Total [a]	
							Mean	Individual				Individual	Mean
GAP, USA	3		97-101			7							
2009/7003643	3	6	100	280	Tuber	7	< 0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	< 0.01

Study No. Trial No. Country Year (Variety)	Application				Matrix	PHI days	Residues (mg/kg)						
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad		M700F002	M700F008	M700F048	Total [a]	
							Mean	Individual				Individual	Mean
RCN R080451 USA (Wayne, New York) 2008 (Superior)		7	101 101 302	282 283				< 0.01	< LOD	< LOD	< LOD	< 0.01	
						14	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< 0.01	< LOD	< LOD	< LOD	< 0.01	
						21	< 0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
2009/7003643 RCN R080452 USA (Wayne, New York) 2008 (Norland)	3	6 7	100 101 101 302	280 281 281	Tuber	7	< 0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< 0.01	< LOD	< LOD	< LOD	< 0.01	
						14	0.02	0.02	< LOD	< LOD	< LOD	0.02	0.02
								0.02	< LOD	< LOD	< LOD	0.02	
						21	0.02	0.02	< LOD	< LOD	< LOD	0.02	0.02
								0.02	< LOD	< LOD	< LOD	0.02	
2009/7003643 RCN R080453 USA (Lehigh, Pennsylvania) 2008 (Dark Red Norland)	3	6 8	104 102 103 309	316 310 314	Tuber	7	< 0.01	< LOD	< 0.01	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						14	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						21	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
2009/7003643 RCN R080454 Canada (Queens, Prince Edward Island) 2008 (Yukon Gold)	3	7 6	102 96 95 293	255 241 238	Tuber	7	< 0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< 0.01	< LOD	< LOD	< LOD	< 0.01	
2009/7003643 RCN R080455 Canada (Queens, Prince Edward Island) 2008 (Shepody)	3	7 6	100 97 98 295	250 242 245	Tuber	7	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< 0.01	< LOD	< LOD	< LOD	< 0.01	
2009/7003643 RCN R080456 USA (Tift, Georgia) 2008 (Red Pontiac)	3	6 7	120 99 100 319	223 236 232	Tuber	7	0.02	(0.01, 0.02) 0.02	< 0.01	< LOD	< LOD	0.02	0.02
								(< 0.01, 0.01) 0.01	< 0.01	< LOD	< LOD	0.01	
						14	0.02	(0.01, 0.02) 0.02	< 0.01	< LOD	< LOD	0.02	0.02
								(0.01, 0.01) 0.01	< 0.01	< LOD	< LOD	0.01	
						21	0.02	(0.01, 0.02) 0.02	< 0.01	< LOD	< LOD	0.02	0.02
								< 0.01	< 0.01	< LOD	< LOD	< 0.01	
2009/7003643 RCN R080457 USA (Seminole, Florida) 2008 (Red Pontiac)	3	7 7	101 100 100 301	284 281 280	Tuber	7	< 0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	< 0.01
								(< LOD, < 0.01) < 0.01	< LOD	< LOD	< LOD	< 0.01	
						14	< 0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	< 0.01
								(< LOD,	< LOD	< LOD	< LOD	< 0.01	

Study No. Trial No. Country Year (Variety)	Application				Matrix	PHI days	Residues (mg/kg)						
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad		M700F002	M700F008	M700F048	Total [a]	
							Mean	Individual				Individual	Mean
								< 0.01) < 0.01					
						21	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
2009/7003643 RCN R080458 USA (Freeborn, Minnesota) 2008 (Cascade)	3	6 7	101 102 101 304	189 192 190	Tuber	7	< 0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< 0.01	< LOD	< LOD	< LOD	< 0.01	
						14	< 0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< 0.01	< LOD	< LOD	< LOD	< 0.01	
						21	< 0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< 0.01	< LOD	< LOD	< LOD	< 0.01	
2009/7003643 RCN R080459 USA (Cass, North Dakota) 2009 (Red Lady)	3	6 8	105 104 105 314	196 194 196	Tuber	7	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						14	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< 0.01	< 0.01	
						21	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						28	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
2009/7003643 RCN R080460 USA (Keokuk, Iowa) 2008 (Kennebec)	3	7 7	101 99 102 302	154 166 192	Tuber	7	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						14	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						21	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< 0.01	< LOD	< 0.01	< 0.01	
2009/7003643 RCN R080461 USA (Dane, Wisconsin) 2008 (Superior)	3	7 7	129 100 94 323	242 262 293	Tuber	7	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						14	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						21	< 0.01	< LOD	< LOD	< LOD	< 0.01	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< 0.01	< 0.01	
2009/7003643 RCN R080462 USA (Pepin, Wisconsin) 2008 (Russet Burbank)	3	7 29	99 100 99 298	278 281 280	Tuber	7	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						14	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						21	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
2009/7003643 RCN R080463 Canada (Taber, Alberta) 2008 (Russet Burbank)	3	7 7	102 99 102 303	154 149 153	Tuber	7	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< 0.01	< LOD	< LOD	< LOD	< 0.01	
						14	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						21	< 0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
2009/7003643 RCN R080464 USA	3	7 7	99 102 101	185 191 189	Tuber	7	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						14	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01

Study No. Trial No. Country Year (Variety)	Application				Matrix	PHI days	Residues (mg/kg)						
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad		M700F002	M700F008	M700F048	Total [a]	
							Mean	Individual				Individual	Mean
(Cache, Utah) 2008 (Klondike Rose)			302					< LOD	< LOD	< LOD	< LOD	< 0.01	
						21	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
2009/7003643 RCN R080465 USA (Sacramento, California) 2008 (1533)	3	7 7	99 99 99 297	187 187 187	Tuber	7	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						14	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						21	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
2009/7003643 RCN R080466 USA (Payette, Idaho) 2008 (Norkotah)	3	6 8	100 102 99 301	234 239 233	Tuber	7	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						14	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						21	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
2009/7003643 RCN R080467 USA (Washington, Idaho) 2008 (Ranger Russet)	3	7 7	102 102 101 305	240 238 236	Tuber	7	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						14	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						21	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
2009/7003643 RCN R080468 USA (Bingham, Idaho) 2008 (Ranger Russet)	3	6 7	103 103 103 309	192 192 192	Tuber	7	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						10	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						15	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						21	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						28	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< 0.01	< LOD	< LOD	< LOD	< 0.01	
2009/7003643 RCN R080469 USA (Power, Idaho) 2008 (Russet Burbank)	3	8 6	98 97 99 294	184 182 186	Tuber	7	< 0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						14	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
						21	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< 0.01	< LOD	< LOD	< LOD	< 0.01	
2009/7003643 RCN R080470 USA (Benton, Oregon) 2008 (Ranger Russet)	3	7 7	98 102 100 300	277 288 283	Tuber	7	< 0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< 0.01	< LOD	< LOD	< LOD	< 0.01	
						14	< 0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< 0.01	< LOD	< LOD	< LOD	< 0.01	
						21	< 0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< LOD	< LOD	< LOD	< LOD	< 0.01	
2009/7003643 RCN R080471	3	7 7	104 103	192 192	Tuber	7	< 0.01	< LOD	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< 0.01	< LOD	< LOD	< LOD	< 0.01	

Study No. Trial No. Country Year (Variety)	Application				Matrix	PHI days	Residues (mg/kg)						
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad		M700F002	M700F008	M700F048	Total [a]	
							Mean	Individual				Individual	Mean
Canada (Strathcona, Alberta) 2008 (Russet Burbank E3)			101 308	189		14	< 0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< 0.01	< LOD	< LOD	< LOD	< 0.01	
						21	< 0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< 0.01	< LOD	< LOD	< LOD	< 0.01	

[a] All analytes are reported in terms of themselves. Total residues (**(**Fluxapyroxad + M700F008 + M700F048**)** are expressed as parent equivalents.

LOQ is 0.01 mg/kg for each of parent fluxapyroxad and metabolites M700F008, M700F002 and M700F048

LOD is 0.002 mg/kg for each of parent fluxapyroxad and metabolites M700F008, M700F002 and M700F048

Radish

A series of trials in radish was conducted in the USA (Norris, 2012). Three applications of fluxapyroxad as a 62.5 g/L EC formulation were made a target rate of 100 g ai/ha and a target interval of 7 days. Duplicate samples of radish roots and tops were collected at 7 days after the last application.

Table 36 Residues of fluxapyroxad and its metabolites in radish roots

Location, Year (variety)	Application				Residues, mg/kg, parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700 F002	M700 F008	M700 F048	Total*
Wayne, NY, USA, 2010 (Scarlet Globe)	3 (7, 7)	100, 98, 98	280, 270, 270	7	0.05 (0.04, 0.05)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.05 (0.04, 0.05)
Martin, FL, USA, 2011 (Escala)	3 (7, 7)	99, 100, 100	280, 280, 290	7	0.04 (0.04, 0.04)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.04 (0.04, 0.04)
Palm Beach, FL, USA, 2011 (Escala)	3 (7, 7)	100, 100, 100	290, 280, 290	7	0.03 (0.03, 0.03)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.03 (0.05, 0.05)
Clinton, IL, USA, 2010 (Champion)	3 (6, 7)	100, 100, 100	280, 280, 280	7	0.1 (0.09, 0.1)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.1 (0.09, 0.1)
Tulare, CA, USA, 2010 (Crimson Giant)	3 (7, 7)	100, 100, 100	280, 280, 280	7	0.1 (0.1, 0.1)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.1 (0.1, 0.1)

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad equivalents

Sugar beet

Residue trials in sugar beet were considered by the 2012 Meeting and the data is reproduced below.

Table 37 Residues in sugar beet roots from the foliar application of fluxapyroxad to sugar beet in the USA and Canada (Johnston and Saha 2010, 2009/7003643)

Study No. Trial No. Country Year (Variety)	Application				Matrix	PHI days	Residues (mg/kg)						
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad		M700F002	M700F008	M700F048	Total [a]	
							Mean	Individual				Individual	Mean
GAP, USA	3		97-101			7							
2009/7003643 RCN R080472 USA (Freeborn, Minnesota) 2008 (Beta 130R)	3	7 7	101 100 100 301	189 188 188	Roots	7	0.06	0.06	< LOD	< LOD	< LOD	0.06	0.06
								0.06	< LOD	< LOD	< LOD	0.06	
					Roots	13	0.04	(0.04, 0.05) 0.05	< LOD	< LOD	< LOD	0.05	0.04
								(0.03, 0.03) 0.03	(< LOD, < 0.01) < 0.01	< LOD	< LOD	0.03	
					Roots	21	0.03	(0.02, 0.04) 0.03	(< LOD, < 0.01) < 0.01	< LOD	< LOD	0.03	0.03
								(0.03, 0.03) 0.03	< LOD	< LOD	< LOD	0.03	
2009/7003643 RCN R080473 USA (Cass, North Dakota) 2008 (539 RR)	3	6 8	99 98 100 297	186 183 187	Roots	7	0.03	0.02	< LOD	< LOD	< LOD	0.02	0.03
								0.03	< LOD	< LOD	< LOD	0.03	
					Roots	14	0.02	0.02	< LOD	< LOD	< LOD	0.02	0.02
								0.02	< LOD	< LOD	< LOD	0.02	
					Roots	21	0.02	0.02	< LOD	< LOD	< LOD	0.02	0.02
								0.02	< LOD	< LOD	< LOD	0.02	
2009/7003643 RCN R080474 USA (Jetterson, Iowa) 2008 (Crystal 539RR)	3	7 7	104 98 101 303	174 157 177	Roots	7	0.04	0.05	< LOD	< LOD	< LOD	0.05	0.04
								0.03	< LOD	< LOD	< LOD	0.03	
					Roots	14	0.06	(0.05, 0.04) 0.05	< LOD	< LOD	< LOD	0.05	0.06
								(0.06, 0.06) 0.06	(< LOD, < 0.01) < 0.01	< LOD	< LOD	0.06	
					Roots	21	0.05	(0.03, 0.04) 0.04	(< LOD, < 0.01) < 0.01	< LOD	< LOD	0.04	0.05
								(0.07, 0.05) 0.06	(< LOD, < 0.01) < 0.01	< LOD	< LOD	0.06	
2009/7003643 RCN R080475 Canada (Strathcona, Alberta) 2008 (Betaseed Beta 1385)	3	7 7	102 103 102 307	190 192 189	Roots	7	0.01	0.01	< LOD	< LOD	< LOD	0.01	0.01
								(0.01, 0.01) 0.01	< LOD	< LOD	< LOD	0.01	
					Roots	14	0.04	(0.03, 0.03) 0.03	< LOD	< LOD	< LOD	0.03	0.04
								(0.04, 0.04) 0.04	< LOD	< LOD	< LOD	0.04	
					Roots	21	0.04	(0.02, 0.03) 0.03	< LOD	< LOD	< LOD	0.03	0.04
								(0.03, 0.04) 0.04	< LOD	< LOD	< LOD	0.04	
2009/7003643 RCN R080476 USA (LaMoure, North Dakota)	3	7 7	102 101 101 304	190 190 189	Roots	7	0.02	0.02	< LOD	< LOD	< LOD	0.02	0.02
								0.02	< LOD	< LOD	< LOD	0.02	
					Roots	13	0.04	0.06	< LOD	< LOD	< LOD	0.06	0.04
								0.02	< LOD	< LOD	< LOD	0.02	

Study No. Trial No. Country Year (Variety)	Application				Matrix	PHI days	Residues (mg/kg)						
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad		M700F002	M700F008	M700F048	Total [a]	
							Mean	Individual				Individual	Mean
2008 (539 RR)					Roots	21	0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	0.01
								0.01	< LOD	< LOD	< LOD	0.01	
2009/7003643 RCN R080477 Canada (Taber, Alberta) 2008 (Beta B85-Pro 15)	3	7 10	99 100 99 298	150 151 150	Roots	8	< 0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< 0.01	< LOD	< LOD	< LOD	< 0.01	
					Roots	15	< 0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	< 0.01
								< 0.01	< LOD	< LOD	< LOD	< 0.01	
					Roots	22	0.01	0.01	< LOD	< LOD	< LOD	0.01	0.01
								< 0.01	< LOD	< LOD	< LOD	< 0.01	
2009/7003643 RCN R080478 USA (Hockley, Texas) 2008 (Phoenix)	3	8 6	102 100 99 301	284 280 277	Roots	7	0.02	(0.02, 0.02) 0.02	< LOD	< LOD	< LOD	0.02	0.02
								(0.01, 0.02) 0.02	< LOD	< LOD	< LOD	0.02	
					Roots	14	0.03	(0.03, 0.03) 0.03	< LOD	< LOD	< LOD	0.03	0.03
								(0.02, 0.03) 0.03	< LOD	< LOD	< LOD	0.03	
					Roots	21	0.03	(0.03, 0.02) 0.03	< LOD	< LOD	< LOD	0.03	0.03
								(0.02, 0.02) 0.02	< LOD	< LOD	< LOD	0.02	
2009/7003643 RCN R080479 USA (Cache, Utah) 2008 (4023 R)	3	7 7	103 103 101 307	192 193 188	Roots	8	0.01	0.01	< LOD	< LOD	< LOD	0.01	0.01
								< 0.01	< LOD	< LOD	< LOD	< 0.01	
					Roots	15	0.01	< 0.01	< LOD	< LOD	< LOD	< 0.01	0.01
								(< 0.01, 0.01) 0.01	< LOD	< LOD	< LOD	0.01	
					Roots	21	0.01	(< 0.01, 0.01) 0.01	< LOD	< LOD	< LOD	0.01	0.01
								(< 0.01, 0.01) 0.01	< LOD	< LOD	< LOD	0.01	
2009/7003643 RCN R080480 USA (Tulare, California) 2008 (Phoenix)	3	7 7	91 100 99 290	286 287 286	Roots	7	0.04	0.03	< LOD	< LOD	< LOD	0.03	0.04
								0.04	< LOD	< LOD	< LOD	0.04	
					Roots	14	0.03	0.03	< LOD	< LOD	< LOD	0.03	0.03
								0.03	< LOD	< LOD	< LOD	0.03	
					Roots	21	0.03	0.02	< LOD	< LOD	< LOD	0.02	0.03
								0.03	< LOD	< LOD	< LOD	0.03	
2009/7003643 RCN R080481 USA (Power, Idaho) 2008 (Hilleshog 9026)	3	7 7	98 101 98 297	185 190 183	Roots	7	0.05	0.07	< LOD	< LOD	< LOD	0.07	0.05
								0.03	< LOD	< LOD	< LOD	0.03	
					Roots	10	0.04	0.03	< LOD	< LOD	< LOD	0.03	0.04
								0.04	< LOD	< LOD	< LOD	0.04	
					Roots	15	0.03	0.05	< LOD	< LOD	< LOD	0.05	0.03
								0.01	< LOD	< LOD	< LOD	0.01	
					Roots	21	0.04	0.04	< LOD	< LOD	< LOD	0.04	0.04
								0.04	< LOD	< LOD	< LOD	0.04	
					Roots	28	0.02	0.02	< LOD	< LOD	< LOD	0.02	0.02
								0.02	< LOD	< LOD	< LOD	0.02	

Study No. Trial No. Country Year (Variety)	Application				Matrix	PHI days	Residues (mg/kg)						
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad		M700F002	M700F008	M700F048	Total [a]	
							Mean	Individual				Individual	Mean
2009/7003643 RCN R080482 USA (Bingham, Idaho) 2008 (BTS 25RR05)	3	7 7	98 103 99 300	183 191 183	Roots	8	0.02	0.01	< LOD	< LOD	< LOD	0.01	0.02
								0.02	< LOD	< LOD	< LOD	0.02	
					Roots	15	0.02	0.02	< LOD	< LOD	< LOD	0.02	0.02
								0.02	< LOD	< LOD	< LOD	0.02	
					Roots	21	0.03	0.02	< LOD	< LOD	< LOD	0.02	0.03
								0.03	< LOD	< LOD	< LOD	0.03	
2009/7003643 RCN R080483 Canada (RM of Portage la Prairie, Manitoba) 2008 (Betaseed Beta 1385)	3	9 7	120 101 105 326	223 189 196	Roots	8	0.05	0.05	< LOD	< LOD	< LOD	0.05	0.05
								0.04	< LOD	< LOD	< LOD	0.04	
					Roots	15	0.03	0.02	< LOD	< LOD	< LOD	0.02	0.03
								0.04	< LOD	< LOD	< LOD	0.04	
					Roots	20	0.03	0.02	< LOD	< LOD	< LOD	0.02	0.03
								0.03	< LOD	< LOD	< LOD	0.03	

[a] All analytes are reported in terms of themselves. Total residues ((Fluxapyroxad + M700F008 + M700F048) are expressed as parent equivalents.

LOQ is 0.01 mg/kg for each of parent fluxapyroxad and metabolites M700F008, M700F002 and M700F048

LOD is 0.002 mg/kg for each of parent fluxapyroxad and metabolites M700F008, M700F002 and M700F048

Celery

A series of trials in celery was conducted in the USA (Schreier, 2013-b). Three applications of a 62.5 g/L EC formulation of fluxapyroxad were made at a target rate of 200 g ai/ha, and an interval of 7 days. Duplicate treated samples were collected at 0 and 1 days after the last application, with additional decline samples being collected at a single site.

Table 38 Residues of fluxapyroxad and its metabolites in celery (untrimmed leaf stalks)

Location, Year (variety)	Application					Residues, mg/kg parent equivalents			
	Formulation	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F008	M700F048	Total [a]
Gregory, MI, USA, 2011 (Tongo)	62.5 EC	3 (7, 7)	200, 200, 200	280, 280, 280	0	1.2 (1.0, 1.4)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.2 (1.0, 1.4)
					1	1.4 (1.4, 1.5)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.4 (1.4, 1.5)
Belle Glade, FL, USA, 2011 (Walt's Pride)	62.5 EC	3 (6, 7)	200, 200, 200	290, 280, 280	0	2.2 (1.8, 2.6)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	2.2 (1.8, 2.6)
					1	1.3 (1.0, 1.6)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.3 (1.0, 1.6)
Lompoc, CA, USA, 2011 (Conquistador)	62.5 EC	3 (7, 7)	200, 200, 210	280, 290, 280	0	2.5 (1.8, 3.2)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	2.5 (1.8, 3.2)
					1	2.7 (2.7, 2.6)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	2.7 (2.7, 2.6)
Lompoc, CA, USA, 2011 (Mission)	62.5 EC	3 (7, 7)	210, 200,	280, 280, 280	0	5.2 (4.4, 6.1)	< 0.01 (< 0.01,	< 0.01 (< 0.01,	5.2 (4.4, 6.1)

Location, Year (variety)	Application					Residues, mg/kg parent equivalents			
	Formulation	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F008	M700F048	Total [a]
			200				< 0.01)	< 0.01)	
					1	5.2 (4.8, 5.5)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	5.2 (4.8, 5.5)
Guadalupe, CA, USA, 2011 (Conquistador)	62.5 EC	3 (7, 7)	200, 200, 200	280, 280, 280	0	1.5 (1.7, 1.2)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.5 (1.7, 1.2)
					1	1.5 (1.1, 1.9)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.5 (1.1, 1.9)
Guadalupe, CA, USA, 2011 (Mission)	62.5 EC	3 (7, 7)	200, 200, 210	280, 280, 280	0	2.0 (1.9, 2.1)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	2.0 (1.9, 2.1)
					1	1.8 (1.7, 2.0)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.8 (1.7, 2.0)
					3	1.4 (1.4, 1.4)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.4 (1.4, 1.4)
					5	1.1 (1.1, 1.1)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.1 (1.1, 1.1)
					7	1.0 (1.1, 0.97)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.0 (1.1, 0.97)

Residues were generally undetectable in the untreated control samples, apart from a single detection of parent compound at a level < LOQ

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad

Cereals

Rice

A series of trials in rice was conducted in the USA (Thiel, 2012). Two foliar broadcast applications of a 300 g/L SC formulation of fluxapyroxad were made using backpack boom sprayers at a target rate of 150 g ai/ha, and a target interval of 7 days. An adjuvant (non-ionic surfactant, fatty acid methyl ester, or crop oil concentrate) was included in the tank mix for all applications. Duplicate treated samples of rice grain with husk were collected 28 days after the last application, with additional decline samples being collected from some sites.

Residue data for rice straw is tabulated in Table 39 below.

Table 39 Residues of fluxapyroxad and metabolites in rice (with husk)

Location, Year (variety)	Application				Residues, mg/kg, parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700 F002	M700 F008	M700 F048	Total [a]
Screeton, AR, USA, 2011 (Jupiter)	2 (7)	150, 150	190, 190	28	0.61 (0.62, 0.59)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.61 (0.62, 0.59)

Location, Year (variety)	Application				Residues, mg/kg, parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700 F002	M700 F008	M700 F048	Total [a]
Lonoke, AR, USA, 2011 (CL142AR)	2 (7)	160, 150	190, 190	28	0.34 (0.34, 0.34)	< 0.02 (< 0.02, < 0.02)	< 0.01 (0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.35 (0.35, 0.34)
Washington, LA, USA, 2011 (Cocodrie)	2 (7)	160, 150	200, 200	28	1.7 (1.6, 1.7)	< 0.02 (< 0.02, < 0.02)	0.02 (0.02, 0.02)	< 0.01 (< 0.01, < 0.01)	1.7 (1.7, 1.7)
Cheneyville, LA, USA, 2011 (Cheniere)	2 (7)	150, 140	130, 140	28	1.1 (1.3, 0.84)	< 0.02 (< 0.02, < 0.02)	0.03 (0.02, 0.03)	< 0.01 (< 0.01, < 0.01)	1.1 (1.4, 0.87)
Delaplaine, AR, USA, 2011 (CLXL 745)	2 (8)	150, 150	190, 190	28	0.80 (0.80, 0.79)	< 0.02 (< 0.02, < 0.02)	0.03 (0.03, 0.03)	< 0.01 (< 0.01, < 0.01)	0.83 (0.83, 0.82)
Delaplaine, AR, USA, 2011 (CLXL 745)	2 (6)	150, 160	47, 47	28	0.47 (0.48, 0.46)	< 0.02 (< 0.02, < 0.02)	0.02 (0.02, 0.02)	< 0.01 (< 0.01, < 0.01)	0.49 (0.50, 0.48)
Pollard, AR, USA, 2011 (CL 111)	2 (6)	150, 150	190, 190	0	5.3 (5.4, 5.2)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	5.3 (5.4, 5.2)
				14	0.61 (0.56, 0.65)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.61 (0.56, 0.65)
				28	0.59 (0.46, 0.71)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.59 (0.46, 0.71)
				30	0.56 (0.55, 0.56)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.56 (0.55, 0.56)
				36	0.54 (0.61, 0.46)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.54 (0.61, 0.46)
Campbell, MO, USA, 2011 (Wells)	2 (8)	150, 150	190, 190	28	0.37 (0.34, 0.40)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.37 (0.34, 0.40)
Fisk, MO, USA, 2011 (CL 151)	2 (8)	150, 150	190, 190	0	4.1 (4.3, 4.0)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	4.1 (4.3, 4.0)
				14	0.98 (1.0, 0.92)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.98 (1.0, 0.92)
				28	0.86 (0.88, 0.83)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.86 (0.88, 0.83)
				30	0.94 (1.0, 0.88)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.94 (1.0, 0.88)
				35	0.78 (0.81, 0.74)	< 0.02 (< 0.02, < 0.02)	0.01 (< 0.01, 0.01)	< 0.01 (< 0.01, < 0.01)	0.78 (0.81, 0.75)
Qulin, MO, USA, 2011 (CLXL 745)	2 (7)	160, 150	47, 47	29	0.60 (0.62, 0.58)	< 0.02 (< 0.02, < 0.02)	0.02 (0.01, 0.02)	< 0.01 (< 0.01, < 0.01)	0.62 (0.63, 0.60)
Glennonville, MO, USA, 2011 (CL 151)	2 (6)	150, 150	47, 47	28	0.26 (0.29, 0.22)	< 0.02 (< 0.02, < 0.02)	0.01 (0.01, 0.01)	< 0.01 (< 0.01, < 0.01)	0.27 (0.30, 0.23)
Dudley, MO, USA, 2011 (CL 111)	2 (7)	150, 150	190, 190	28	0.92 (0.91, 0.93)	< 0.02 (< 0.02, < 0.02)	0.03 (0.03, 0.03)	< 0.01 (< 0.01, < 0.01)	0.95 (0.94, 0.96)

Location, Year (variety)	Application				Residues, mg/kg, parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700 F002	M700 F008	M700 F048	Total [a]
Markham, TX, USA, 2011 (Cocodrie)	2 (7)	160, 150	190, 180	28	0.92 (0.93, 0.91)	< 0.02 (< 0.02, < 0.02)	0.04 (0.04, 0.04)	< 0.01 (< 0.01, < 0.01)	0.96 (0.97, 0.95)
El Campo, TX, USA, 2011 (Cocodrie)	2 (7)	150, 150	190, 180	28	1.2 (1.3, 1.0)	< 0.02 (< 0.02, < 0.02)	0.03 (0.03, 0.03)	< 0.01 (< 0.01, < 0.01)	1.2 (1.3, 1.1)
Porterville, CA, USA, 2011 (Koshihikari)	2 (6)	150, 150	190, 190	29	1.2 (1.2, 1.2)	< 0.02 (< 0.02, < 0.02)	0.03 (0.02, 0.03)	< 0.01 (< 0.01, < 0.01)	1.2 (1.2, 1.3)
Yuba City, CA, USA, 2011 (M206)	2 (7)	150, 150	230, 230	29	3.7 (3.8, 3.6)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	3.7 (3.8, 3.6)

No residues of metabolites were detected in the untreated control samples, while residues of fluxapyroxad at levels < LOQ were found at two of the trial sites

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad equivalents

Sorghum

Residue data in sorghum grain evaluated by the 2012 Meeting is tabulated below. Residue data for sorghum forage and stover is included in Table 46.

Table 40 Residues from the foliar application of fluxapyroxad to grain sorghum in the USA (White 2010, 2010/7003693)

Study No. Trial No. Country Year (Variety)	Application				Matrix	PHI days	Residues (mg/kg)						
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad		M700F002	M700F008	M700F048	Total [a]	
							Mean	Individual				Individual	Mean
2010/7003693 RCN R080440 USA (Butler, Missouri) 2008 (LGX-47)	2	7	101 100 201	188 189	Grain	21	0.13	0.13	< LOD	< 0.01	< LOD	0.13	0.13
								0.12	< LOD	0.01	< LOD	0.13	
2010/7003693 RCN R080441 USA (Ottawa, Michigan) 2008 (9135)	2	7	100 99 199	274 270	Grain	20	0.15	0.15	< LOD	< 0.01	< LOD	0.15	0.15
								0.14	< LOD	< 0.01	< LOD	0.14	
2010/7003693 RCN R080442 USA (Cass, North Dakota) 2008 (WGF)	2	7	100 100 200	187 187	Grain	21	0.15	0.13	< LOD	0.04	< 0.01	0.17	0.20
								0.17	< LOD	0.05	< 0.01	0.22	
2010/7003693 RCN R080443 USA (Caddo,	2	6	99 102 201	178 234	Grain	23	0.19	0.18	< LOD	< 0.01	< LOD	0.18	0.19
								0.19	< LOD	< 0.01	< LOD	0.19	

Study No. Trial No. Country Year (Variety)	Application				Matrix	PHI days	Residues (mg/kg)						
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad		M700F002	M700F008	M700F048	Total [a]	
							Mean	Individual				Individual	Mean
Oklahoma) 2008 (753)													
2010/7003693 RCN R080444 USA (Wharton, Texas) 2008 (84G50)	2	7	100 101 201	134 133	Grain	20	0.31	0.19	< LOD	< 0.01	< LOD	0.19	0.32
								0.43	< LOD	0.01	< 0.01	0.44	
2010/7003693 RCN R080445 USA (Clarke, Georgia) 2008 (82G10)	2	7	99 101 200	273 254	Grain	21	0.40	(< LOD, 0.58, 0.64) 0.41	< LOD	< LOQ	< LOD	0.41	0.40
								(0.22, 0.44, 0.47) 0.38	< LOD	< 0.01	< LOD	0.38	
2010/7003693 RCN R080446 USA (York, Nebraska) 2008 (7R34)	2	7	99 100 199	186 187	Grain	22	0.21	0.21	< LOD	0.01	< 0.01	0.22	0.22
								0.20	< LOD	0.01	< 0.01	0.21	
2010/7003693 RCN R080447 USA (Pawnee, Kansas) 2008 (84G62)	2	7	99 100 199	186 187	Grain	21	0.17	0.16	< LOD	< 0.01	< LOD	0.16	0.17
								0.17	< LOD	< 0.01	< LOD	0.17	
2010/7003693 RCN R080448 USA (Stafford, Kansas) 2008 (84G62)	2	7	104 97 201	194 182	Grain	21	0.24	0.30	< LOD	0.08	< 0.01	0.38	0.30
								0.17	< LOD	0.04	< 0.01	0.21	

[a] All analytes are reported in terms of themselves. Total residues (**(**Fluxapyroxad + M700F008 + M700F048**)** are expressed as parent equivalents.

LOQ is 0.01 mg/kg for each of parent fluxapyroxad and metabolites M700F008, M700F002 and M700F048

LOD is 0.002 mg/kg for each of parent fluxapyroxad and metabolites M700F008, M700F002 and M700F048

Sugar cane

A series of trials in sugar cane (Schreier, 2012-b) was conducted in the USA. Two foliar broadcast applications of a 62.5 g/L EC formulation of fluxapyroxad were made at a target rate and interval of 0.125 kg ai/ha and 14 days using pressurised backpack sprayers. At one of the trial sites, a second treated plot was established, with 2× 0.625 kg ai/ha applications being made in order to generate raw sugar cane for processing (see below for further details of the processing phase of this study). Duplicate treated samples of sugar cane were collected by hand at a target interval of 14 days after the last application.

Table 41 Residues of fluxapyroxad and its metabolites in sugar cane

Location, Year (variety)	Application				Residues, mg/kg, parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Washington, LA, USA, (384)	2 (14)	120, 120	190, 190	14	0.05 (0.05, 0.05)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.05 (0.05, 0.05)
Washington, LA, USA, (384)	2 (14)	120, 120	180, 190	14	0.06 (0.03, 0.09)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.06 (0.03, 0.09)
Washington, LA, USA, (384)	2 (14)	120, 120	190, 190	14	0.04 (0.05, 0.03)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.04 (0.05, 0.03)
Raymondville, TX, USA, 2010 (CP873388)	2 (15)	120, 120	190, 190	14	0.26 (0.19, 0.33)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.26 (0.19, 0.33)
Homestead, FL, USA, 2010 (CP801)	2 (14)	120, 120	190, 190	14	0.56 (0.30, 0.82)	< 0.02 (< 0.02, < 0.02)	0.02 (0.01, 0.02)	< 0.01 (< 0.01, < 0.01)	0.58 (0.31, 0.84)
Belle Glade, FL, USA, 2010 (CP-89-2143)	2 (14)	120, 120	190, 190	14	1.3 (2.2, 0.50)	< 0.02 (< 0.02, < 0.02)	0.01 (< 0.01, 0.02)	< 0.01 (< 0.01, < 0.01)	1.4 (2.2, 0.52)
Belle Glade, FL, USA, 2010 (CP-96-1252)	2 (14)	120, 120	190, 190	14	< 0.01 (< 0.01, < 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
Belle Glade, FL, USA, 2010 (CP-88-1762)	2 (14)	120, 120	190, 190	14	0.73 (1.1, 0.32)	< 0.02 (< 0.02, < 0.02)	0.03 (0.04, 0.02)	< 0.01 (< 0.01, < 0.01)	0.77 (1.2, 0.34)
	2 (14)	640, 630	190, 190	14	2.1 (1.5, 2.7)	< 0.02 (< 0.02, < 0.02)	0.06 (0.10, < 0.01)	< 0.01 (0.01, < 0.01)	2.1 (1.6, 2.7)

No residues of metabolites were detected in the untreated control samples, while residues of fluxapyroxad at levels < LOQ were found at four of the eight trial sites

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad equivalents

Tree nuts

Five trials each in almonds and pecans were conducted in the USA (Wyatt, 2012). Three foliar applications of a 62.5 g/L EC formulation were made at each site using an airblast sprayer. A spray adjuvant was included for all applications. Duplicate samples of treated kernels were collected a target interval of 14 days after the last application, with samples being collected at additional intervals from some sites to generate decline data.

Table 42 Residues of fluxapyroxad and metabolites in almond kernels

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Strathmore, CA, USA, 2011 (Nonpareil)	3 (7, 8)	130, 120, 120	950, 910, 700	14	0.01 (0.01, 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.01 (0.01, 0.01)
				22	0.015 (0.01, 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.015 (0.01, 0.02)

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
				27	0.01 (< 0.01, 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.01 (0.01, < 0.01)
				32	0.015 (0.02, 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.015 (0.02, 0.01)
				38	0.02 (0.02, 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.02 (0.02, 0.02)
Dinuba, CA, USA, 2011 (Carmel)	3 (7, 7)	120, 120, 130	830, 810, 830	14	< 0.01 (< 0.01, < 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
Poplar, CA, USA, 2011 (Carmel)	3 (7, 8)	130, 130, 120	670, 620, 660	13	< 0.01 (< 0.01, < 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
Wasco, CA, USA, 2011 (Price)	3 (8, 6)	130, 120, 120	760, 740, 740	14	0.01 (0.01, 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.01 (0.01, 0.01)
Buttonwillow, CA, USA, 2011 (Monterey)	3 (7, 7)	130, 130, 120	810, 850, 810	14	< 0.01 (< 0.01, < 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad equivalents

Table 43 Residues of fluxapyroxad and metabolites in pecan kernels

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, kg ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Bailey, NC, USA, 2011 (Stuart)	3 (7, 6)	130, 130, 120	660, 680, 650	14	< 0.002 (< 0.002, < 0.002)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
Mystic, GA, USA, 2011 (Sumner)	3 (7, 7)	120, 120, 130	880, 860, 870	14	< 0.002 (< 0.002, < 0.002)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
Alexandria, LA, USA, 2011 (Creek)	3 (7, 7)	140, 130, 130	780, 760, 730	14	< 0.01 (< 0.01, < 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
Pearsall, TX, USA, 2011 (Desirable)	3 (7, 7)	120, 120, 120	620, 650, 780	14	< 0.01 (< 0.01, < 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
				20	< 0.01 (< 0.01, < 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
				29	< 0.01 (< 0.01, < 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
				30	< 0.002 (< 0.002, < 0.002)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
				37	< 0.01 (< 0.01, < 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, kg ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Anton, TX, USA, 2011 (Western Schley)	3 (7, 7)	120, 130, 130	740, 760, 760	14	0.03 (0.03, 0.03)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.03 (0.03, 0.03)

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad equivalents

Cotton

A series of residue trials in cotton were conducted in the USA (Schreier, 2014). Three foliar applications of a 62.5 g/L EC formulation of fluxapyroxad were made at a target rate of 0.1 kg ai/ha and a target interval of 7 days using hand held or tractor-mounted equipment. The plots were harvested at maturity by hand or by mechanical picker, then bolls were ginned to generate undelinted seed samples, with additional gin by-products samples from three sites (see below).

Table 44 Residues of fluxapyroxad and its metabolites in cottonseed

Location, Year (variety)	Application				Residues, mg/kg, parent equivalents			
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F008	M700F048	Total [a]
Sycamore, GA, USA, 2013 (PHY 375)	3 (5, 7)	100, 100, 99	160, 170, 170	30	0.07 (0.05, 0.09)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.07 (0.05, 0.09)
Cheneyville, LA, USA, 2013 (Phytogen 499)	3 (7, 7)	100, 100, 100	170, 160, 150	29	0.11 (0.11, 0.10)	0.01 (0.01, 0.01)	< 0.01 (< 0.01, < 0.01)	0.12 (0.12, 0.11)
Washington, LA, USA, 2013 (PHY 375)	3 (7, 7)	100, 100, 100	150, 150, 140	31	0.01 (< 0.01, 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.01 (< 0.01, 0.02)
St Landry, LA, USA, 2013 (Stoneville 5288)	3 (7, 7)	100, 100, 100	150, 150, 140	31	0.01 (< 0.01, 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.01 (< 0.01, 0.02)
Lebanon, OK, USA, 2013 (FM 2011 GT)	3 (7, 7)	100, 100, 100	140, 140, 140	28	0.13 (0.14, 0.11)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.13 (0.14, 0.11)
Claude, TX, USA, 2013 (FM 9250)	3 (4, 4)	99, 100, 99	140, 140, 140	32	0.09 (0.10, 0.07)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.09 (0.10, 0.07)
Groom, TX, USA, 2013 (FM 2011 GT)	3 (4, 4)	100, 99, 98	140, 140, 140	32	0.11 (0.12, 0.09)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.11 (0.12, 0.09)
Groom, TX, USA, 2013 (FM 2011 GT)	3 (4, 4)	99. 99, 99	140, 140, 140	35	0.07 (0.10, 0.05)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.002)	0.07 (0.10, 0.05)
Groom, TX, USA, 2013 (FM 9250)	3 (4, 4)	100, 99, 99	140, 140, 140	32	0.02 (0.03, 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.02 (0.03, 0.02)
Sanger, CA, USA, 2013 (Pima)	3 (7, 7)	99, 100, 100	140, 140, 150	39	0.03 (0.03, 0.03)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.03 (0.03, 0.03)
Sanger, CA, USA, 2013 (FM 835 LLB 2)	3 (7, 7)	100, 100, 100	150, 140, 150	30	0.02 (0.01, 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.02 (0.01, 0.02)
Fresno, CA, USA, 2013 (Acala)	3 (7, 7)	100, 95, 100	140, 140, 150	31	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad equivalents

Animal feeds

Rice straw

Table 45 Residues of fluxapyroxad and metabolites in rice straw

Location, Year (variety) Dry matter content [%]	Application				Residues, mg/kg, parent equivalents. Residues on a dry weight basis are shown in square brackets for parent compound and total residues only.				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Screeton, AR, USA, 2011 (Jupiter) [27.8]	2 (7)	150, 150	190, 190	28	0.51 (0.36, 0.65 [1.8 (1.3, 2.3)]	< 0.02 (< 0.02, < 0.02)	0.02 (< 0.01, 0.02)	< 0.01 (< 0.01, < 0.01)	0.52 (0.36, 0.67) [1.9 (1.3, 2.4)]
Lonoke, AR, USA, 2011 (CL142AR) [33.8]	2 (7)	160, 150	190, 190	28	2.3 (2.5, 2.1) [6.8 (7.5, 6.1)]	< 0.02 (< 0.02, < 0.02)	0.04 (0.04, 0.04)	0.04 (0.03, 0.04)	2.4 (2.6, 2.1) [7.0 (7.7, 6.3)]
Washington, LA, USA, 2011 (Cocodrie) [32.1]	2 (7)	160, 150	200, 200	28	2.3 (2.7, 2.0) [7.3 (8.4, 6.2)]	< 0.02 (< 0.02, < 0.02)	0.03 (0.03, 0.02)	< 0.01 (0.01, < 0.01)	2.4 (2.7, 2.0) [7.4 (8.5, 6.3)]
Cheneyville, LA, USA, 2011 (Cheniere) [27.5]	2 (7)	150, 140	130, 140	28	2.8 (2.6, 3.0) [10 (9.3, 11)]	< 0.02 (< 0.02, < 0.02)	0.03 (0.02, 0.03)	< 0.01 (< 0.01, < 0.01)	2.8 (2.6, 3.1) [10 (9.4, 11)]
Delaplaine, AR, USA, 2011 (CLXL 745) [68.6]	2 (8)	150, 150	190, 190	28	0.91 (0.85, 0.97) [1.3 (1.2, 1.4)]	< 0.02 (< 0.02, < 0.02)	0.02 (0.01, 0.02)	< 0.01 (< 0.01, < 0.01)	0.93 (0.86, 0.99) [1.4 (1.3, 1.4)]
Delaplaine, AR, USA, 2011 (CLXL 745) [26.7]	2 (6)	150, 160	47, 47	28	0.68 (0.61, 0.74) [2.5 (2.3, 2.8)]	< 0.02 (< 0.02, < 0.02)	0.01 (0.01, 0.01)	< 0.01 (< 0.01, < 0.01)	0.69 (0.62, 0.75) [2.6 (2.3, 2.8)]
Pollard, AR, USA, 2011 (CL 111) [25.8, day 0; 33.1, day 28]	2 (6)	150, 150	190, 190	0	4.7 (4.7, 4.7) [18 (18, 18)]	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	4.7 (4.7, 4.7) [18 (18, 18)]
				14	0.86 (0.93, 0.78) [3.3 (3.6, 3.0)]	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.86 (0.93, 0.78) [3.3 (3.6, 3.0)]
				28	0.95 (0.90, 0.99) [2.9 (2.7, 3.0)]	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.95 (0.90, 0.99) [2.9 (2.7, 3.0)]
				30	0.83 (0.88, 0.77) [2.5 (2.7, 2.3)]	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.83 (0.88, 0.77) [2.5 (2.7, 2.3)]
				36	0.68 (0.68, 0.67) [2.0 (2.1, 2.0)]	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.68 (0.68, 0.67) [2.0 (2.1, 2.0)]
Campbell, MO, USA, 2011 (Wells) [34.6]	2 (8)	150, 150	190, 190	28	0.52 (0.51, 0.52) [1.5 (1.5,	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.52 (0.51, 0.52) [1.5 (1.5,

Location, Year (variety) Dry matter content [%]	Application				Residues, mg/kg, parent equivalents. Residues on a dry weight basis are shown in square brackets for parent compound and total residues only.				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
					1.5)]				1.5)]
Fisk, MO, USA, 2011 (CL 151) [27.2, day 0; 31.5, day 28]	2 (8)	150, 150	190, 190	0	3.6 (3.2, 4.0) [13 (12, 15)]	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	3.6 (3.2, 4.0) [13 (12, 15)]
				14	0.74 (0.82, 0.65) [2.7 (3.0, 2.4)]	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.74 (0.82, 0.65) [2.7 (3.0, 2.4)]
				28	0.56 (0.63, 0.49) [1.8 (2.0, 1.6)]	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.56 (0.63, 0.49) [1.8 (2.0, 1.6)]
				30	0.59 (0.49, 0.69) 1.9 (1.6, 2.2)]	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.59 (0.49, 0.69) [1.9 (1.6, 2.2)]
				35	0.50 (0.47, 0.53) [1.6 (1.5, 1.7)]	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.50 (0.47, 0.53) [1.6 (1.5, 1.7)]
Qulin, MO, USA, 2011 (CLXL 745) [29.5]	2 (7)	160, 150	47, 47	29	2.0 (2.1, 2.0) [6.9 (6.9, 6.8)]	< 0.02 (< 0.02, < 0.02)	0.03 (0.02, 0.03)	< 0.01 (< 0.01, < 0.01)	2.1 (2.1, 2.0) [7.0 (7.1, 6.9)]
Glennonville, MO, USA, 2011 (CL 151) [23.9]	2 (6)	150, 150	47, 47	28	1.0 (1.2, 0.82) [4.2 (5.0, 3.4)]	< 0.02 (< 0.02, < 0.02)	0.02 (0.02, 0.01)	< 0.01 (< 0.01, < 0.01)	1.0 (1.2, 0.82) [4.2 (5.1, 3.4)]
Dudley, MO, USA, 2011 (CL 111) [25.3]	2 (7)	150, 150	190, 190	28	1.0 (1.1, 0.98) [4.0 (4.2, 3.9)]	< 0.02 (< 0.02, < 0.02)	0.02 (0.02, 0.02)	< 0.01 (< 0.01, < 0.01)	1.1 (1.1, 1.0) [4.2 (4.3, 4.0)]
Markham, TX, USA, 2011 (Cocodrie) [80]	2 (7)	160, 150	190, 180	28	2.9 (3.6, 2.2) [3.6 (4.5, 2.7)]	< 0.02 (< 0.02, < 0.02)	0.08 (0.09, 0.06)	0.06 (0.06, 0.05)	3.0 (3.8, 2.3) [3.8 (4.7, 2.9)]
El Campo, TX, USA, 2011 (Cocodrie) [76.9]	2 (7)	150, 150	190, 180	28	2.4 (2.0, 2.8) [3.1 (2.6, 3.6)]	< 0.02 (< 0.02, < 0.02)	0.06 (0.06, 0.05)	0.05 (0.05, 0.05)	2.5 (2.1, 2.9) [3.2 (2.7, 3.7)]
Porterville, CA, USA, 2011 (Koshihikari) [39.1]	2 (6)	150, 150	190, 190	29	2.0 (1.4, 2.7) [5.2 (3.6, 6.8)]	< 0.02 (< 0.02, < 0.02)	0.08 (0.06, 0.10)	< 0.01 (< 0.01, < 0.01)	2.1 (1.5, 2.8) [5.4 (3.8, 7.1)]
Yuba City, CA, USA, 2011 (M206) [34.3]	2 (7)	150, 150	230, 230	29	15 (17, 13) [42 (48, 37)]	< 0.02 (< 0.02, < 0.02)	0.02 (0.02, 0.01)	< 0.01 (< 0.01, < 0.01)	14.6 (16.6, 12.5) [42 (48, 37)]

No residues of metabolites were detected in the untreated control samples, while residues of fluxapyroxad at levels < LOQ were found at one of the trial sites

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad equivalents.

Sorghum forage and stover

Table 46 Residues from the foliar application of fluxapyroxad to grain sorghum in the USA (White 2010, 2010/7003693)

Study No. Trial No. Country Year (Variety)	Application				Matrix (% moisture)	PHI days	Residues (mg/kg) Residues on a dry weight basis are shown in square brackets for mean parent compound and total residues only.						
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad [a]		M700F002	M700F008	M700F048	Total (Fluxapyroxad + M700F008 + M700F048)	
							Individual	Mean				Individual	Mean
2010/7003693 RCN R080440 USA (Butler, Missouri) 2008 (LGX-47)	2	7	101 100 201	190 187	Forage (73.8)	7	0.79	0.72 [2.7]	< LOD	0.01	< 0.01	0.80	0.73 [2.8]
							0.65		< LOD	0.01	< 0.01	0.66	
	2	7	100 101 201	188 189	Stover (66.7)	21	0.44	0.42 [1.3]	< LOD	0.02	< 0.01	0.46	0.45 [1.4]
							0.40		< LOD	0.02	0.02	0.43	
2010/7003693 RCN R080441 USA (Ottawa, Michigan) 2008 (9135)	2	7	99 100 199	275 286	Forage (58.7)	7	1.41	1.4 [3.5]	< LOD	0.02	< 0.01	1.43	1.5 [3.5]
							1.46		< LOD	0.02	< 0.01	1.48	
	2	7	100 99 199	274 270	Stover (70.8)	20	0.89	0.83 [2.8]	< LOD	0.02	< LOD	0.91	0.85 [2.9]
							0.77		< LOD	0.01	< LOD	0.78	
2010/7003693 RCN R080442 USA (Cass, North Dakota) 2008 (WGF)	2	8	100 100 200	187 187	Forage (72.8)	6	0.77	0.79 [2.9]	< LOD	0.03	< 0.01	0.80	0.83 [3.1]
							0.81		< LOD	0.04	< 0.01	0.85	
	2	7	100 100 200	187 190	Stover (77.9)	21	0.34	0.35 [1.6]	< LOD	0.03	< 0.01	0.37	0.39 [1.8]
							0.35		< LOD	0.04	0.02	0.40	
2010/7003693 RCN R080443 USA (Caddo, Oklahoma)	2	8	99 98 197	131 175	Forage (66.0)	7	2.22	2.3 [7.0]	< LOD	0.04	0.02	2.27	2.3 [7.1]
2008							2.37		< LOD	0.04	0.02	2.42	
(753)	2	6	99 102 201	178 234	Stover (75.8)	23	0.46	0.40 [1.6]	< LOD	0.02	0.02	0.49	0.43 [1.8]
							0.33		< LOD	0.02	0.02	0.36	
2010/7003693 RCN R080444 USA (Wharton, Texas) 2008 (84G50)	2	6	99 102 201	129 137	Forage (61.6)	7	1.21	1.2 [3.1]	< LOD	0.04	0.04	1.28	1.2 [3.2]
							1.15		< LOD	0.04	0.03	1.21	
	2	7	100 101 201	134 133	Stover (69.4)	20	0.71	0.75 [2.5]	< LOD	0.03	0.04	0.77	0.81 [2.6]
							0.79		< LOD	0.03	0.03	0.84	
2010/7003693 RCN	2	7	97 101 198	184 193	Forage (85.4)	7	0.70	0.94 [6.4]	< LOD	0.04	0.01	0.75	1.0 [6.8]
							1.18		< LOD	0.06	0.03	1.26	

Study No. Trial No. Country Year (Variety)	Application				Matrix (% moisture)	PHI days	Residues (mg/kg) Residues on a dry weight basis are shown in square brackets for mean parent compound and total residues only.						
	No	Interval Days	g ai/ha	Water (L/ha)			Fluxapyroxad [a]		M700F002	M700F008	M700F048	Total (Fluxapyroxad + M700F008 + M700F048)	
							Individual	Mean				Individual	Mean
R080445 USA (Clarke, Georgia) 2008 (82G10)	2	7	99 101 200	273 254	Stover (59.4)	21	0.89	1.0 [2.5]	< LOD	0.02	0.02	0.92	1.1 [2.6]
							1.17		< LOD	0.03	0.02	1.21	
2010/7003693 RCN R080446 USA (York, Nebraska) 2008 (7R34)	2	7	102 101 203	191 188	Forage (74.7)	6	0.38	0.45 [1.8]	< LOD	0.04	0.01	0.43	0.50 [2.0]
							0.51		< LOD	0.04	0.01	0.56	
	2	7	99 100 199	186 187	Stover (72.1)	22	0.17	0.20 [0.72]	< LOD	< LOD	< LOD	0.17	0.20 [0.72]
							0.23		< LOD	< LOD	< LOD	0.23	
2010/7003693 RCN R080447 USA (Pawnee, Kansas) 2008 (84G62)	2	7	102 100 202	191 188	Forage (68.4)	7	0.43	0.47 [1.5]	< LOD	0.02	< 0.01	0.45	0.49 [1.6]
							0.50		< LOD	0.02	< 0.01	0.52	
	2	7	99 100 199	186 187	Stover (68.7)	21	0.54	0.66 [2.1]	< LOD	0.02	< 0.01	0.56	0.69 [2.2]
							0.77		< LOD	0.03	0.01	0.81	
2010/7003693 RCN R080448 USA (Stafford, Kansas) 2008 (84G62)	2	7	99 101 200	185 189	Forage (75.2)	7	0.54	0.56 [2.3]	< LOD	0.03	< 0.01	0.57	0.59 [2.4]
							0.57		< LOD	0.03	< 0.01	0.60	
	2	7	104 97 201	194 182	Stover (72.6)	21	0.97	0.87 [3.2]	< LOD	0.04	< LOD	1.01	0.91 [3.3]
							0.77		< LOD	0.03	< 0.01	0.80	

[a] All analytes are reported in terms of themselves. Total residues (**(**Fluxapyroxad + M700F008 + M700F048**)** are expressed as parent equivalents

LOQ is 0.01 mg/kg for each of parent fluxapyroxad and metabolites M700F008, M700F002 and M700F048

LOD is 0.002 mg/kg for each of parent fluxapyroxad and metabolites M700F008, M700F002 and M700F048

Moisture content was determined for selected control samples using an infrared moisture determination balance

Almond hulls

Table 47 Residues of fluxapyroxad and metabolites in almond hulls

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Strathmore, CA, USA, 2011 (Nonpareil)	3 (7, 8)	130, 120, 120	950, 910, 700	14	1.2 (1.2, 1.3)	< 0.02 (< 0.02, < 0.02)	0.01 (< 0.01, 0.01)	< 0.01 (< 0.01, < 0.01)	1.2 (1.2, 1.3)

Location, Year (variety)	Application				Residues, mg/kg parent equivalents				
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
				22	1.3 (1.2, 1.4)	< 0.02 (< 0.02, < 0.02)	0.02 (0.02, 0.02)	< 0.01 (< 0.01, < 0.01)	1.3 (1.2, 1.4)
				27	0.75 (0.78, 0.71)	< 0.02 (< 0.02, < 0.02)	0.02 (0.02, 0.01)	< 0.01 (< 0.01, < 0.01)	0.76 (0.80, 0.72)
				32	0.96 (0.99, 0.92)	< 0.02 (< 0.02, < 0.02)	0.02 (0.01, 0.02)	< 0.01 (< 0.01, < 0.01)	0.97 (1.0, 0.94)
				38	1.4 (1.3, 1.4)	< 0.02 (< 0.02, < 0.02)	0.02 (0.02, 0.02)	0.01 (0.01, < 0.01)	1.4 (1.4, 1.4)
Dinuba, CA, USA, 2011 (Carmel)	3 (7, 7)	120, 120, 130	830, 810, 830	14	1.7 (1.7, 1.7)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.7 (1.7, 1.7)
Poplar, CA, USA, 2011 (Carmel)	3 (7, 8)	130, 130, 120	670, 620, 660	13	0.92 (0.86, 0.98)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.92 (0.86, 0.98)
Wasco, CA, USA, 2011 (Price)	3 (8, 6)	130, 120, 120	760, 740, 740	14	1.1 (1.1, 1.1)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	1.1 (1.1, 1.1)
Buttonwillow, CA, USA, 2011 (Monterey)	3 (7, 7)	130, 130, 120	810, 850, 810	14	0.88 (0.74, 1.0)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.88 (0.74, 1.0)

[a]Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad equivalents

Cotton gin by-products

Table 48 Residues of fluxapyroxad and metabolites in cotton gin trash

Location, Year (variety)	Application				Residues, mg/kg, parent equivalents			
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Fluxapyroxad	M700F008	M700F048	Total [a]
Claude, TX, USA, 2013 (FM 9250)	3	99, 100, 99	140, 140, 140	32	6.9 (7.9, 5.9)	0.02 (0.03, 0.02)	< 0.01 (< 0.01, < 0.01)	6.9 (7.9, 5.9)
Groom, TX, USA, 2013 (FM 2011 GT)	3	100, 99, 98	140, 140, 140	32	5.2 (5.0, 5.5)	0.01 (0.01, 0.01)	< 0.01 (< 0.01, < 0.01)	5.3 (5.0, 5.5)
Groom, TX, USA, 2013 (FM 2011 GT)	3	99, 99, 99	140, 140, 140	35	8.0 (7.6, 8.4)	0.03 (0.02, 0.03)	< 0.01 (< 0.01, < 0.01)	8.1 (7.7, 8.5)

No residues were detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad equivalents

Fate of residues in processing

Citrus

A processing study in oranges was conducted in Brazil (Guimaraes, 2014-b). At four field trial sites, three applications of an SC formulation containing 333 g/L pyraclostrobin and 167 g/L fluxapyroxad were made by foliar airblast application at a target rate of 0.5 kg ai/ha pyraclostrobin + 0.25 kg ai/ha fluxapyroxad and a target interval of 28 days. Fruit was collected 14 days after the last application.

Oranges were processed into juice, dried pulp and oil using simulated commercial procedures. Untreated control samples were processed prior to the treated samples. Samples for processing (around 250 kg per sample) were first washed using an industrial water bath equipped with rotary brushes. The cleaned oranges were then juiced using a commercial machine (JBT model HP 391 citrus juice extractor). This juices the oranges by compressing the fruit between two cups with sharpened metal tubes at their bases. A water spray was maintained to separate the oil as an emulsion, with the oil separated from the wash water by centrifuging and decanting. The pulp/juice mixture was separated in a commercial finisher (JBT model UCF 35).

Residues of fluxapyroxad and its metabolites were determined using LC-MS/MS method number L0137/01. Processing was completed within a day of sample collection, and both raw orange and processed commodity samples were frozen within 24 hours of collection. Analyses were completed within 3 months of harvest of the raw oranges.

Table 49 Residues of fluxapyroxad and metabolites in raw oranges and processed fractions

Location, Year (variety)	Application				Residues, mg/kg, parent equivalents					
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Sample	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
San Antonio de Posse, Sao Paolo, Brazil, 2013 (Natal em Swingle)	3 (28, 28)	250, 250, 240	2000, 1980, 1940	14	Raw oranges	0.17	< 0.02	< 0.01	< 0.01	0.17
					Dried pulp	0.02	< 0.02	< 0.01	< 0.01	0.02
					Orange juice	< 0.01	< 0.02	< 0.01 c< 0.01	< 0.01	< 0.01
					Orange oil	9.9 c0.01	< 0.02	0.03	< 0.01	9.9
Aguai, Sao Paolo, Brazil, 2013 (Lima Verde)	3 (28, 28)	240, 230, 240	1890, 1850, 1930	14	Raw oranges	0.23	< 0.02	< 0.01	< 0.01	0.23
					Dried pulp	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
					Orange juice	< 0.01	< 0.02	< 0.01 c< 0.01	< 0.01	< 0.01
					Orange oil	3.2	< 0.02	< 0.01	< 0.01	3.2
Mogi Mirim, Sao Paolo, Brazil, 2013 (Pera Coroa)	3 (28, 28)	250, 250, 250	2000, 1970, 1980	14	Raw oranges	0.40	< 0.02	< 0.01	< 0.01	0.40
					Dried pulp	0.03	< 0.02	< 0.01	< 0.01	0.03
					Orange juice	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01

Location, Year (variety)	Application				Residues, mg/kg, parent equivalents					
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Sample	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
					Orange oil	8.7 c< 0.01	< 0.02	< 0.01	< 0.01	8.7
Limeira, Sao Paolo, Brazil, 2013 (Pera Coroa)	3 (28, 28)	250, 240, 250	2000, 1920, 2030	14	Raw oranges	0.19	< 0.02	< 0.01	< 0.01	0.19
					Dried pulp	0.02	< 0.02	< 0.01	< 0.01	0.02
					Orange juice	< 0.01	< 0.02	< 0.01	< 0.01	< 0.01
					Orange oil	6.2	< 0.02	< 0.01	< 0.01	6.2

Residues were generally not found in the untreated control samples. Where residues were found in the untreated control samples, these are indicated with a 'c' prefix

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad equivalents

Table 50 Processing factors for fluxapyroxad in oranges

Commodity	Processing factor	
	Parent only	Total residues
Dried pulp	< 0.04, 0.08, 0.11, 0.12 (median = 0.095)	< 0.04, 0.08, 0.11, 0.12 (median = 0.095)
Juice	< 0.03, < 0.04, < 0.05, < 0.06 (median = 0.045)	< 0.03, < 0.04, < 0.05, < 0.06 (median = 0.045)
Oil	14, 22, 33, 58 (median = 27.5)	14, 22, 33, 58 (median = 27.5)

Grape

A processing study in grapes was conducted in the USA (Belcher and Riley, 2012-b).

At two sites, grapevines were treated with three foliar airblast applications of a 300 g/L SC formulation of fluxapyroxad at a target rate of 0.4 kg ai/ha and a target interval of 10 days. Two plots, one each of a red and a white grape variety, were treated at each site using the same application regime. Grape samples were collected 14 days after the last application.

Grapes were processed using methods simulating commercial processes as far as possible. The grapes (40–80 kg per sample for processing) were first crushed using a crusher/de-stemmer, and the stems were separated and for red grapes only, the stems and initial crush were sampled. The crush was then subdivided into portions for juice and wine making.

The crushed grapes (approx. 10–25 kg of crush were reserved for juicing) were transferred to a steam-jacketed kettle and heated to 52–57 °C for 8–12 minutes, and then to 60–66 °C for 8–12 minutes. The grape pulp was then pressed using a hydraulic fruit press, and wet pomace was separated. The fresh juice was filtered and pasteurised (79–85 °C for 15–30 seconds). Pasteurised juice was sampled.

For white/rosé winemaking, was approximately 20–35 kg of grape crush were transferred to a kettle, treated with pectic enzyme and potassium metabisulphite and allowed to stand for 1 hour, prior to pressing with a hydraulic press. Primary fermentation was conducted in a 5-gallon container. Yeast was added, and the container allowed to stand overnight at approximately 21 °C. The wine was racked to separate the lees, and transferred to glass carboys for secondary fermentation at approximately 13 °C until the specific gravity reached approximately 1.03. Once

carbon dioxide formation had ceased indicating completion of fermentation, the wine was racked again and gelatin added for fining. The wine was then racked a final time, and filtered through diatomaceous earth before sampling.

For red winemaking, the process was similar to white winemaking, with the addition of a step after the initial crushing and separation of the stems where the juice/pulp mixture was heated to approximately 60 °C to impart colour to the wine, then cooled to approximately 21 °C before addition of the enzyme and sodium metabisulphite. The processing then proceeded as for the white/rosé wine.

For generation of the raisin samples, grapes were harvested and sun dried in the field for 3–13 days before sampling (approx. 1.0–1.3 kg of sun dried grapes per sample). At the processing facility, the raisins were hand sorted to remove loose dirt and debris, stems, panicles and substandard raisins. The cleaned raisins were then spray washed in batches to remove any residual dirt and to raise the water content to $\leq$ 18%. The raisins were drained and dried if necessary to achieve the desired water content.

Residues of fluxapyroxad and its metabolites were determined using LC-MS/MS method number L0137/01. Processing of raw grapes into juice and wine commenced within 1–3 days of harvest, while processing of the sun dried raisins took place around 4–6 weeks after sampling. Raw grape samples for analysis were frozen within 4 hours of collection. Grapes for processing into juice and wine were shipped to the processor at ambient temperature and stored in a cooler pending processing. Raisins were shipped to the processor at ambient temperature, and stored frozen pending further processing. On completion of processing, processed commodity samples were frozen pending analysis. All analyses were completed within 5 months of harvest of the grapes.

Table 51 Residues of fluxapyroxad and metabolites in raw grapes and processed fractions

Location, Year (variety)	Application				Residues, mg/kg, as parent equivalents					
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Sample	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Yates, NY, USA, 2011 (Concord)	3 (10, 11)	400, 400, 400	930, 940, 940	13	Raw grapes (in field)	0.93	< 0.02	< 0.01	< 0.01	0.93
					Raw grapes (pre-processing)	0.53	< 0.02	< 0.01	< 0.01	0.53
					Stalks	2.6	< 0.02	< 0.01	< 0.01	2.6
					Crush	0.41	< 0.02	< 0.01	< 0.01	0.41
					Must	0.09	< 0.02	< 0.01	< 0.01	0.09
					Pomace (wet)	3.8	< 0.02	< 0.01	< 0.01	3.8
					Must deposit	0.42	< 0.02	< 0.01	< 0.01	0.42
					Separated must	0.16	< 0.02	< 0.01	< 0.01	0.16
					Pasteurised juice	0.22	< 0.02	< 0.01	< 0.01	0.22
					Yeast deposit	2.7 (3.7, 1.8)	< 0.02	< 0.01	< 0.01	2.7 (3.7, 1.8)
					Red wine	0.11	< 0.02	< 0.01	< 0.01	0.11
					Raisins	5.4	< 0.02	< 0.01	< 0.01	5.4
Yates, NY, USA, 2011 (Vidal)	3 (10, 9)	400, 400, 400	940, 940, 950	13	Raw grapes (in field)	1.5	< 0.02	< 0.01	< 0.01	1.5
					Raw grapes (pre-processing)	0.81	< 0.02	< 0.01	< 0.01	0.81
					Must	0.24	< 0.02	< 0.01	< 0.01	0.24
					Pomace	4.6	< 0.02	< 0.01	< 0.01	4.6
					Must deposit	1.1	< 0.02	< 0.01	< 0.01	1.1
					Separated must	0.30	< 0.02	< 0.01	< 0.01	0.30
					Pasteurised juice	0.37	< 0.02	< 0.01	< 0.01	0.37
					Yeast deposit	3.4 (3.7, 3.2)	< 0.02	< 0.01	< 0.01	3.4
					Rosé wine	0.18	< 0.02	< 0.01	< 0.01	0.18
					Raisins	4.3	< 0.02	< 0.01	< 0.01	4.3
Madera, CA, USA, 2011 (Ruby Red)	3 (9, 11)	400, 400, 400	470, 470, 470	14	Raw grapes (in	0.60	< 0.02	< 0.01	< 0.01	0.60

Location, Year (variety)	Application				Residues, mg/kg, as parent equivalents					
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Sample	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
					field)					
					Raw grapes (pre-processing)	0.37	< 0.02	< 0.01	< 0.01	0.37
					Stalks	2.6	< 0.02	< 0.01	< 0.01	2.6
					Crush	0.33	< 0.02	< 0.01	< 0.01	0.33
					Must	0.08	< 0.02	< 0.01	< 0.01	0.08
					Pomace (wet)	1.5	< 0.02	< 0.01	< 0.01	1.5
					Must deposit	0.36	< 0.02	< 0.01	< 0.01	0.36
					Separated must	0.07	< 0.02	< 0.01	< 0.01	0.07
					Pasteurised juice	0.10	< 0.02	< 0.01	< 0.01	0.10
					Yeast deposit	0.36	< 0.02	< 0.01	< 0.01	0.36
					Red wine	0.07	< 0.02	< 0.01	< 0.01	0.07
					Raisins	1.2	< 0.02	< 0.01	< 0.01	1.2
Madera, CA, USA, 2011 (Thompson Seedless)	3 (9, 11)	400, 400, 400	460, 470, 470	14	Raw grapes (in field)	0.49	< 0.02	< 0.01	< 0.01	0.49
					Raw grapes (pre-processing)	0.50	< 0.02	< 0.01	< 0.01	0.50
					Must	0.12 (0.12, 0.12)	< 0.02	< 0.01	< 0.01	0.12 (0.12, 0.12)
					Pomace	2.4	< 0.02	< 0.01	< 0.01	2.4
					Must deposit	0.23	< 0.02	< 0.01	< 0.01	0.23
					Separated must	0.11 (0.11, 0.11)	< 0.02	< 0.01	< 0.01	0.11 (0.11, 0.11)
					Pasteurised juice	0.11	< 0.02	< 0.01	< 0.01	0.11
					Yeast deposit	0.65	< 0.02	< 0.01	< 0.01	0.65
					Rosé wine	0.12	< 0.02	< 0.01	< 0.01	0.12
					Raisins	1.4	< 0.02	< 0.01	< 0.01	1.4

Residues were generally not detected in the untreated control samples, except for three detections of parent compound at levels < LOQ

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad equivalents

Table 52 Processing factors for fluxapyroxad in grapes

Commodity	Processing factor	
	Parent compound	Total residues
Stalks	4.9, 7.0 (median = 5.95)	4.9, 7.0 (median = 5.95)
Grape crush	0.77, 0.89 (median = 0.83)	0.77, 0.89 (median = 0.83)
Must	0.17, 0.22, 0.24, 0.30 (median = 0.23)	0.17, 0.22, 0.24, 0.30 (median = 0.23)
Pomace (wet)	4.1, 4.8, 5.7, 7.2 (median = 5.25)	4.1, 4.8, 5.7, 7.2 (median = 5.25)
Must deposit	0.46, 0.79, 0.97, 1.4 (median = 0.88)	0.46, 0.79, 0.97, 1.4 (median = 0.88)
Separated must	0.19, 0.22, 0.30, 0.37 (median = 0.26)	0.19, 0.22, 0.30, 0.37 (median = 0.26)
Pasteurised juice	0.22, 0.27, 0.42, 0.46 (median = 0.345)	0.22, 0.27, 0.42, 0.46 (median = 0.345)
Yeast deposit	0.97, 1.3, 4.2, 5.1 (median = 2.75)	1.0, 1.3, 4.2, 5.1 (median = 2.75)
Red wine	0.19, 0.21 (median = 0.20)	0.19, 0.21 (median = 0.20)
Rosé wine	0.22, 0.24 (median = 0.23)	0.22, 0.24 (median = 0.23)
Raisins	2.8, 3.2, 5.3, 10 (median = 4.25)	2.8, 3.2, 5.3, 10 (median = 4.25)

Sugar cane

A sugar cane processing study was carried out in the USA (Schreier, 2012-b). At a site in Florida, a plot was treated at 2× 0.625 kg ai/ha, with a 14-day re-treatment interval, and sample collection 14 days after the last application.

Sugar cane samples (approximately 40 kg) were processed by expelling the juice by multiple passes through an AUM Enterprise cane crusher. The waste (bagasse) was discarded, while the juice was filtered (100 mesh sieve) and adjusted to pH 7.2–7.4 using calcium oxide solution. After stirring for approximately 15 minutes, the juice was brought to approximately 100 °C and held at that temperature for 3 minutes, then centrifuged to separate the 'mud' from 'thin juice'. The thin juice was concentrated to a solids content of 50–60% using a vacuum evaporator, at a temperature of ≤ 70 °C. Further concentration, to a solids content of 75–80%, was carried out at ≤ 55 °C. The resulting thick juice was seeded with a small amount of pulverised white sugar to commence the crystallisation process, and the batch cooled in a walk-in refrigerator. Molasses and raw sugar were separated by centrifuge, and the raw sugar was washed in the centrifuge to remove molasses by steaming prior to sampling. Refining was carried out by dissolving the raw sugar in an equal amount of water with stirring. The pH was adjusted to 5.5 with phosphoric acid, and the solution rested for 2 minutes prior to addition of calcium oxide under agitation to adjust the pH to 7.2. The batch was heated to 60 °C. Filter aid was added, and the solution was vacuum filtered. Activated charcoal was added to the filtrate, which was heated and then filtered again. The filtrate was seeded with pulverised sugar and evaporated under vacuum at ≤ 55 °C until the boiling slowed. The material was cooled, then dried at 43–55 °C to a moisture content of approximately 3%. Samples of unprocessed cane, molasses, raw sugar and refined sugar were collected and frozen for transport to the laboratory.

The process simulated commercial processing, albeit using a batch rather than a continuous process due to the small amount of material.

Samples of sugar cane and the processed commodities were analysed using LC-MS/MS method number L0137/01. Samples were analysed within 14 months of collection of the raw sugar cane, and within 9 months of finishing the processing phase. Raw sugar cane was stored frozen pending processing, which commenced 4 months after harvest, and was completed within a week (processed samples were frozen after collection).

Table 53 Residues of fluxapyroxad and its metabolites in sugar cane and processed commodities

Location, Year (variety)	Application				Residues, mg/kg, parent equivalents					
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Sample	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]

Location, Year (variety)	Application				Residues, mg/kg, parent equivalents					
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Sample	Fluxapyroxad	M700F002	M700F008	M700F048	Total [a]
Belle Glade, FL, USA, 2010 (CP-88-1762)	2 (14)	640, 630	190, 190	14	Sugar cane	2.1 (1.5, 2.7)	< 0.02 (< 0.02, < 0.02)	0.06 (0.10, < 0.01)	< 0.01 (0.01, < 0.01)	2.1 (1.6, 2.7)
					Sugar cane prior to processing	0.24 (0.27, 0.22)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.24 (0.27, 0.22)
					Molasses	0.04 (0.04, 0.04)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.04 (0.04, 0.04)
					Raw sugar	0.06 (0.06, 0.06)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.06 (0.06, 0.06)
					Refined sugar	< 0.01 (< 0.01, < 0.01)	< 0.02 (< 0.02, < 0.02)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)

Residues of M700F002, M700F008, and M700F048 were not detected in the untreated control samples, while residues of fluxapyroxad were < LOQ

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad equivalents

Table 54 Processing factors in sugar cane commodities

Commodity	Processing factor	
	Parent only	Total residues
Molasses	0.17	0.17
Raw sugar	0.25	0.25
Refined sugar	< 0.04	< 0.04

Cotton

A processing study in cotton was conducted in the USA (Woodard and Brungardt, 2014). Field trials were conducted at two sites. Three foliar broadcast applications of an SC formulation (333 g/L pyraclostrobin and 167 g/L fluxapyroxad) were made at a target rate of 3 L/ha and a target interval of 7 days. A spray adjuvant (non-ionic surfactant) was included in the tank mix for all applications. Cottonseed was harvested 30 days after the last application. Sample of treated and control raw cottonseed from each site were ginned within 1 day of harvest, frozen, and transported to the laboratory. Bulk treated and control seed samples were collected and transported to the processor, either frozen (Hinton site) or at ambient temperature (Sanger site). At the processing site, all samples were stored frozen pending processing, which took place around 4–6 weeks after harvest.

Cottonseed samples (approximately 70 kg per sample) were processed using batch methods simulating commercial processes as far as possible. Control samples were processed prior to treated samples to minimise contamination. Defrosted seed samples were tested for moisture, and dried if necessary to reduce the moisture content below 8%. The seed was passed through a stick/burr extractor to remove gin trash, then ginned to separate the cotton seed and lint. Undelinted cottonseed was sampled at this point. Further delinting was then carried out in a delinter to reduce the remaining lint from 11–15% to 3%. Using a roller mill, the delinted seed was cracked, and the kernel and hulls separated using a screen cleaner. Hulls were sampled at this point.

The moisture content of the kernel was checked, and water added to give a moisture level of ≥ 13.5% if necessary. After moisture equilibration, the kernels were heated to approximately 80–90 °C for approximately 30 minutes, then flaked and fed through an extruder with steam injection to produce collets. The collets were ground, dried in an oven at approximately 65–80 °C for 35–40 minutes, then extracted three times with hexane in stainless steel reactors at approximately 50–60 °C. The residual solvent allowed to evaporate from the meal, and the moisture content of the meal adjusted to ≥ 13.5% if necessary. The meal was then screened, and toasted at approximately 105–115 °C for 45–60 minutes, then cooled and sampled. A vacuum evaporator operating at approximately 90–95 °C was used to separate the crude oil from the extraction solvent.

The free fatty acid content of the crude oil was determined, and the required amount of sodium hydroxide solution was added for refining. Refining was carried out by heating with a water bath at approximately 20–25 °C with high rpm stirring for approximately 15 minutes, followed by approximately 12 minutes at low rpm and approximately 65 °C. The refined oil and soapstock were separated by centrifuge and the soapstock was discarded. The refined oil was filtered and bleached by heating at 40–50 °C with diatomaceous earth under vacuum. The temperature was increased to 85–100 °C for 10–15 minutes, then the oil was cooled and filtered. The bleached oil was deodorised by heating to 220–230 °C under vacuum for approximately 30 minutes, and adding 1 mL 0.5% citric acid solution per 100 mL oil. The deodorised oil was sampled.

All processed samples were frozen immediately after collection.

Residues of fluxapyroxad and its metabolites were determined using LC-MS/MS method number L0137/01. All analyses of cottonseed and processed commodities were completed within

3 months of collection of the seed samples and within approximately 1 month of completion of processing.

Table 55 Residues of fluxapyroxad and metabolites in cottonseed and processed fractions

Location, Year (variety)	Applications					Residues, mg/kg, parent equivalents			
	No. (RTI, days)	Rate, g ai/ha	Spray volume (L/ha)	DALA	Sample	Fluxapyroxad	M700 F008	M700 F048	Total*
Hinton, OK, USA, 2013 (FM9160 B2)	3 (6, 7)	510, 510, 510	190, 190, 200	30	Undelinted seed	0.14	<0.01	<0.01	0.14
					Undelinted seed pre-processing	0.64 (0.54, 0.74)	<0.01 (<0.01, <0.01)	<0.01 (<0.01, <0.01)	0.64 (0.54, 0.74)
					Meal	0.025	<0.01	<0.01	0.025
					Hulls	0.11	<0.01	<0.01	0.11
					Refined oil	0.015	<0.01	<0.01	0.015
Sanger, CA, USA, 2013 (FM835LLB2)	3 (7, 6)	500, 500, 500	190, 190, 190	29	Undelinted seed	0.16 (0.093, 0.21, 0.16, 0.16)[#]	<0.01 (<0.01, <0.01, <0.01)	<0.01 (<0.01, <0.01, <0.01)	0.16 (0.093, 0.21, 0.16, 0.16)
					Undelinted seed pre-processing	0.14	<0.01	<0.01	0.14
					Meal	<0.01	<0.01	<0.01	<0.01
					Hulls	0.028	<0.01	<0.01	0.028
					Refined oil	<0.01	<0.01	<0.01	<0.01

Apart from one of the oil samples, where residues of parent < LOQ were detected, residues were generally not detected in the untreated control samples

[a] Sum of fluxapyroxad, M700F008, and M700F048 (the dietary risk assessment residue definition), expressed as fluxapyroxad equivalents

[b] Control and treated samples of undelinted seed obtained directly from the Sanger trial site appear to have been inadvertently swapped, given that the sample labelled as treated did not contain detectable residues of fluxapyroxad, while the sample labelled as the control contained finite fluxapyroxad residues at a level similar to that observed in the unprocessed seed subsampled from the bulk treated sample for processing from the Sanger site. As a result, the finite residue sample will be regarded as the treated sample.

Table 56 Processing factors for fluxapyroxad in cottonseed

Commodity	Processing factor	
	Parent only	Total residues
Meal	0.04, < 0.07 (median = 0.055)	0.04, < 0.07 (median = 0.055)
Hulls	0.17, 0.2 (median = 0.185)	0.17, 0.2 (median = 0.185)
Refined oil	0.02, < 0.07 (median = 0.045)	0.02, < 0.07 (median = 0.045)

Residues in animal commodities

No new animal feeding studies were supplied to the Meeting.

APPRAISAL

Fluxapyroxad was first evaluated for residues and toxicological aspects by the 2012 JMPR. The 2012 Meeting established an ADI of 0–0.02 mg/kg bw and an ARfD of 0.3 mg/kg bw for fluxapyroxad. The 2012 Meeting recommended a number of maximum residue levels for fluxapyroxad.

The residue definition was established as *fluxapyroxad* for compliance with MRLs for both plant and animal commodities. For estimation of dietary intake, the residue definition was established as *sum of fluxapyroxad, 3-(difluoromethyl)-N-(3',4',5'-trifluoro-1,1'-biphenyl-2-yl)-1H-pyrazole-4-carboxamide (M700F008), and 3-(difluoromethyl)-1-(β-D-glucopyranosyl)-N-(3',4',5'-trifluoro-1,1'-biphenyl-2-yl)-1H-pyrazole-4-carboxamide (M700F048), expressed as fluxapyroxad* for plant commodities and *sum of fluxapyroxad and 3-(difluoromethyl)-N-(3',4',5'-trifluoro-1,1'-biphenyl-2-yl)-1H-pyrazole-4-carboxamide (M700F008),* expressed as *fluxapyroxad* for animal commodities.

Fluxapyroxad was scheduled by the Forty-sixth Session of the CCPR in 2014 for evaluation of residue data for additional crops by the 2015 JMPR.

Methods of analysis

No new methods of analysis were submitted to the Meeting.

Stability of residues in stored analytical samples

No new storage stability studies were submitted to the Meeting.

Results of supervised residue trials on crops

The Meeting received supervised trial data for foliar application of fluxapyroxad to citrus fruit, cherries, grapes, strawberries, blueberries, raspberries, bananas, papaya, mango, bulb vegetables, Brassica vegetables, cucurbits, leafy vegetables, carrots, radish, celery, rice, tree nuts, sugarcane and cotton, as well as data for seed treatment and in-furrow application to potatoes.

It is noted that a number of crops (bulb vegetables, Brassica vegetables, cucurbits, leafy vegetables, celery, rice, sorghum and cotton) for which the critical GAP considered is a foliar application use pattern in the USA also have seed treatment uses registered, and the same crops could be treated with both a seed treatment and foliar application of fluxapyroxad.

All residue data provided was for the foliar use pattern (no seed treatment data was available). The foliar use patterns involve application much closer to harvest, with multiple applications and much shorter pre-harvest intervals. The Meeting noted that residue data for seed treatment of cotton at rates up to 100 g ai/100 kg seed considered by the 2012 Meeting showed no detectable residues of fluxapyroxad in cottonseed or gin by-products at harvest. Seed treatment uses are therefore not expected to contribute significantly to the residues of fluxapyroxad in harvested commodities. The Meeting therefore considered that maximum residue levels recommended based on the foliar use patterns are sufficient to cover residues arising from seed treatment use alone, or combined seed treatment/foliar use.

For dietary intake assessment, the residues are expressed as the sum of fluxapyroxad, M700F008, and M700F048, expressed as fluxapyroxad (total residues). Residues of the metabolites are reported as parent equivalents.

The method LOQ was 0.01 mg/kg for each analyte as measured, or 0.01, 0.02, 0.01 and 0.01 mg/kg as parent equivalents for parent, M700F002, M700F008, and M700F048 respectively. The treatment of residues < LOQ for the purpose of summing residue components is illustrated in the table below.

Residues, mg/kg parent equivalents			Total (sum of fluxapyroxad, M700F008, and M700F048)
Fluxapyroxad	M700F008	M700F048	
0.10	< 0.01	< 0.01	0.10

< 0.01	< 0.01	< 0.01	< 0.01
< 0.01	0.03	< 0.01	0.03

Citrus fruits

The maximum GAP for the citrus fruit group is in Argentina, with 3× 0.0033 kg ai/hL applications, with a maximum spray volume of 5000 L/ha, giving a per hectare rate of 0.165 kg ai/ha, and a pre-harvest interval of 7 days. No trials matching that GAP were available.

The GAP in Brazil is 3× 0.0025 kg ai/hL applications at 7-day intervals, with a spray volume of 2000 L/ha (0.05 kg ai/ha), with a 14-day PHI.

Residue trials in oranges, lemons and limes in accordance with the Brazilian GAP were undertaken in Brazil and Argentina.

Residues of fluxapyroxad (parent only) in oranges (whole fruit) at a 14-day PHI were 0.03, 0.04, 0.05 (2), 0.06 (2), 0.07, 0.14 (2), 0.16, and 0.17 mg/kg.

Total residues in whole oranges were 0.03, 0.04, 0.05 (2), 0.06 (2), 0.07, 0.14 (2), 0.16, and 0.17 mg/kg.

Residue data in peel and pulp were available for some of the trials.

Total residues of fluxapyroxad in pulp (edible portion) in oranges (4 trials) and lemons (2 trials) were < 0.01 (6) mg/kg.

The Meeting concluded that there was sufficient edible portion data on which to estimate the STMR and HR for oranges.

The Meeting estimated a maximum residue level of 0.3 mg/kg for fluxapyroxad in oranges, sweet, sour, together with an STMR and an HR of 0.01 mg/kg (based on the edible portion data).

Residues of fluxapyroxad (parent only and total residues) in whole lemons at a 14-day PHI were 0.09 and 0.13 mg/kg.

Residues of fluxapyroxad (parent only and total residues) in limes at a 14-day PHI were 0.04 and 0.06 mg/kg.

The Meeting concluded that there were insufficient data available to estimate maximum residue levels for fruits other than oranges in the citrus fruit group.

Stone fruits

The critical GAP for the stone fruit group is in the USA, with 3× 0.123 kg ai/ha applications at 7-day intervals, and a 0-day pre-harvest interval.

Residue data in peaches, plums and cherries was considered by the 2012 Meeting in conjunction with the above GAP, and a group maximum residue level of 2 mg/kg was estimated for stone fruit.

A request was received by the present Meeting to reconsider the MRL for cherries, with a view to establishing a higher limit to facilitate trade, noting that the highest residue for stone fruit (in cherries) was 1.9 mg/kg. No new data for stone fruit were provided to the current Meeting: two cherry trials were submitted; however, these were considered by the 2012 Meeting. The 2012-submitted stone fruit data are reconsidered in accordance with the 2013 and 2014 JMPR general considerations relating to group MRLs.

Residues of fluxapyroxad (parent compound) in cherries from supervised trials in accordance with GAP were 0.26, 0.31, 0.55, 0.56, 0.59, 0.82, 1.1, and 1.9 mg/kg.

Total residues in cherries were 0.37, 0.50, 0.72, 0.73, 0.78, 1.1, 1.4, and 2.3 mg/kg.

Residues of fluxapyroxad (parent compound) in peaches from supervised trials in accordance with GAP were 0.28, 0.30, 0.32, 0.33, 0.34, 0.43, 0.45, 0.55, 0.57, 0.58, 0.59, and 0.63 mg/kg.

Total residues in peaches were 0.30, 0.31, 0.33, 0.34, 0.35, 0.45, 0.48, 0.58, 0.62, 0.63, and 0.66 (2) mg/kg.

Residues of fluxapyroxad (parent compound) in plums from supervised trials in accordance with GAP were 0.23, 0.24, 0.27, 0.37, 0.38, 0.49, 0.55, 0.56, 0.64, and 0.95 mg/kg.

Total residues in plums were 0.23, 0.24, 0.27, 0.38, 0.39, 0.49, 0.55, 0.56, 0.64, and 0.95 mg/kg.

The Meeting noted the use in the USA is for the stone fruit crop group. Although the median residues for each fruit differed by less than a factor of five, the Meeting decided to recommend maximum residue levels for the individual sub-groups of stone fruit as there are sufficient trials available for each sub-group. The Meeting estimated a maximum residue level for cherries of 3 mg/kg, together with an STMR and an HR of 0.755 and 2.3 mg/kg respectively. The Meeting estimated a maximum residue level of 1.5 mg/kg for the sub-group peaches, together with an STMR and HR of 0.465 and 0.66 mg/kg respectively. The Meeting estimated a maximum residue level of 1.5 mg/kg for the sub-group plums, together with an STMR and an HR of 0.44 and 0.95 mg/kg. The Meeting withdrew its previous recommendation of 2 mg/kg for stone fruit.

Berries and other small fruits (except grapes)

The critical GAP for bushberries, caneberries, low growing berries, and strawberries is in the USA, with 3× 0.2 kg ai/ha applications at 7-day intervals, and a 0-day pre-harvest interval.

A series of trials in blueberries (highbush type) was conducted in the USA. Residues of fluxapyroxad (parent only) immediately after the last of 3× 0.2 kg ai/ha applications were 1.3, 1.7, 2.4 (2), and 3.8 mg/kg.

Total residues were: 1.3, 1.7, 2.4 (2), and 3.8 mg/kg.

A trial in blackberries was conducted in the USA. Residues of fluxapyroxad (parent only and total residues) immediately after the last of 3× 0.2 kg ai/ha applications were 1.4 mg/kg.

A trial in raspberries was conducted in the USA. Residues of fluxapyroxad (parent only and total residues) immediately after the last of 3 × 0.2 kg ai/ha applications were: 2.0 mg/kg.

In a series of trials in strawberries conducted in the USA, residues of fluxapyroxad (parent only) immediately after the last of 3 × 0.2 kg ai/ha applications were: 0.21, 0.26, 0.76 (2), 0.87, 0.97, 1.0, and 2.3 mg/kg.

Total residues were: 0.22, 0.26, 0.76 (2), 0.87, 0.97, 1.0, and 2.4 mg/kg.

The Meeting noted that the GAPs for the subgroups bushberries, caneberries and low growing berries, and strawberries are the same, and noted that the medians for blueberries and strawberries differed by less than 5× (2.9×) and agreed to consider a group MRL. In determining which datasets to use for estimating the MRL, the Meeting noted that the datasets for blueberries and strawberries were not statistically similar (Mann-Whitney), and, based on the blueberries data set, estimated a maximum residue level of 7 mg/kg for berries and other small fruits (except grapes), together with an STMR and an HR of 2.4 and 3.9 mg/kg (based on the highest residue of duplicate samples) respectively.

Grapes

The critical GAP for grapes is in the USA, with 3× 0.2 kg ai/ha applications at 10-day intervals, and a 14-day pre-harvest interval.

A series of trials was conducted in the USA. Residues of fluxapyroxad (parent only) at a 14-day PHI after 3× 0.2 kg ai/ha applications were 0.11, 0.13, 0.23, 0.43, 0.51, 0.62, 0.71, and 1.4 mg/kg.

Total residues were: 0.11, 0.13, 0.23, 0.43, 0.51, 0.62, 0.71, and 1.4 mg/kg.

The Meeting estimated a maximum residue level of 3 mg/kg for fluxapyroxad in grapes, together with an STMR and an HR of 0.47 and 1.4 mg/kg respectively.

Tropical fruit—inedible peel

Banana

The critical GAP in bananas is 4× 0.15 kg ai/ha applications at 8-day intervals, with a 0-day pre-harvest interval, in Belize, Costa Rica, Dominican Republic, El Salvador, Guatemala, Honduras and Panama. Trials matching GAP and conducted in Brazil, Colombia, and Ecuador were available. Results were reported for both bagged and unbagged fruit for each trial plot; the results for unbagged bananas were considered for estimation of the maximum residue level and dietary risk assessment.

Residues of fluxapyroxad (parent compound) in unbagged bananas (whole fruit) after treatment in accordance with GAP were 0.06, 0.07, 0.08, 0.10, 0.14, 0.15, 0.16, 0.36, 0.66, 0.77, and 1.6 mg/kg.

Total residues of fluxapyroxad in banana pulp (edible portion) were 0.03 (2), 0.05, 0.06, 0.09, and 0.10 mg/kg.

The Meeting estimated a maximum residue level of 3 mg/kg for bananas, based on the whole fruit data, and an STMR and an HR of 0.055 and 0.10 mg/kg, based on the edible portion data.

Mango

The critical GAP for mango is in Brazil, with 4× 0.0067 kg ai/hL applications at 7-day intervals, a spray volume of up to 1000 L/ha (giving a maximum per-hectare rate of 0.067 kg ai/ha), and a pre-harvest interval of 7 days.

In trials conducted at GAP in Brazil, residues of fluxapyroxad (parent compound) at a 7-day PHI were 0.14, 0.16, 0.21, and 0.39 mg/kg. Total residues were 0.14, 0.16, 0.21, and 0.39 mg/kg.

The Meeting concluded that there was insufficient data to estimate a maximum residue level for mango.

Papaya

The critical GAP for papaya is in Mexico, with 2× 0.1 kg ai/ha applications at 14-day intervals, and a 7-day pre-harvest interval.

The Meeting concluded that the residue data did not match the GAP (maximum two sprays GAP versus four sprays in the trials).

Bulb vegetables

The critical GAP for the bulb vegetables group is in the USA (3× 0.2 kg ai/ha applications at 7-day intervals and a 7-day pre-harvest interval).

Residue trials were conducted in bulb onions (dry) and green onions.

Residues of fluxapyroxad (parent only) at a 7-day PHI in bulb onions were 0.03, 0.16, 0.23 (2), and 0.27 mg/kg.

Total fluxapyroxad residues were 0.03, 0.16, 0.23 (2), and 0.27 mg/kg.

The Meeting estimated a maximum residue level of 0.6 mg/kg for bulb onions, together with an STMR and an HR of 0.23 and 0.28 mg/kg respectively.

The Meeting agreed to extrapolate the maximum residue level, STMR and HR values estimated for bulb onions to garlic and shallot.

Residues of fluxapyroxad (parent only) at a 7-day PHI in green onions were 0.24 and 0.56 mg/kg.

Total fluxapyroxad residues were 0.24 and 0.56 mg/kg.

The Meeting concluded that there were insufficient data to estimate maximum residue levels for other crops in the bulb vegetables group.

Brassica vegetables

The critical GAP for Brassica vegetables is in the USA (3× 0.1 kg ai/ha applications, a re-treatment interval of 7 days, and a pre-harvest interval of 3 days).

Residue data in cabbage and broccoli from trials conducted in the USA in accordance with GAP were available to the Meeting.

Fluxapyroxad was accidentally applied at double the label application rate for one of the broccoli trials. The Meeting noted that the application rate was within the acceptable range of 0.3–4× GAP and that other parameters were in accordance with GAP. The Meeting agreed that this result could be scaled to GAP using proportionality.

Residues of fluxapyroxad (parent only) in broccoli (unscaled results) at a 3-day PHI were 0.17, 0.32, 0.35, 0.57, and 1.2 mg/kg. Total residues were 0.17, 0.34, 0.36, 0.61, and 1.5 mg/kg.

Residues of fluxapyroxad (parent only) in broccoli at a 3-day PHI were 0.17, 0.29 (s), 0.32, 0.35, and 1.2 mg/kg, where (s) indicates a result that was scaled to the proposed GAP.

Total residues in broccoli were 0.17, 0.31 (s), 0.34, 0.36, and 1.5 mg/kg.

Residues of fluxapyroxad (parent only) in cabbage (heads with wrapper leaves) at a 3-day PHI were 0.07, 0.11, 0.13, 0.14, 0.22, and 1.2 mg/kg.

Total residues in cabbage (head with wrapper leaves) were 0.07, 0.11, 0.14 (2), 0.22, and 1.3 mg/kg.

Total residues in cabbage heads (without wrapper leaves) were < 0.01, 0.01, 0.04 (2), 0.05, and 0.07 mg/kg.

The Meeting noted that the GAP was for the Brassica vegetables group and considered a group MRL. The Meeting further noted the similarity of the datasets (median for broccoli was 2.6× the median for cabbage, and agreed to consider a group MRL. In determining which datasets to use for estimating the MRL, the datasets were confirmed to be similar by the Mann-Whitney U test) and it was agreed to combine the datasets for the purpose of estimating a group maximum residue level.

Combined dataset for fluxapyroxad (parent only) in broccoli and cabbage (with wrapper leaves): 0.07, 0.11, 0.13, 0.14, 0.17, 0.22, 0.32, 0.35, 0.57, and 1.2 (2) mg/kg.

Combined dataset for total residues in broccoli and cabbage (with wrapper leaves): 0.07, 0.11, 0.14 (2), 0.17, 0.22, 0.31, 0.34, 0.36, 1.3, and 1.5 mg/kg.

The Meeting estimated a maximum residue level for Brassica vegetables of 2 mg/kg. Based on the data for total residues in cabbages with wrapper leaves removed, the Meeting estimated an STMR and an HR of 0.04 and 0.07 mg/kg respectively for cabbage. Based on the combined total residues data set, the Meeting estimated an STMR and an HR of 0.22 and 1.7 mg/kg respectively.

Fruiting vegetables, Cucurbits

The critical GAP for cucurbit fruiting vegetables is in the USA (3× 0.1 kg ai/ha, with a 7-day retreatment interval and a 0-day pre-harvest interval). Residue trials in excess of GAP (3× 0.2 kg ai/ha applications) were conducted in the USA in cucumber, melon (cantaloupe), and summer squash. Trials in melons, including watermelons were also conducted in Brazil, but these did not match the critical GAP (four applications rather than three, and the rate differed by more than ±30%).

Residue data for the crops at the appropriate PHI are summarized below.

Residues of fluxapyroxad (parent only and total residues) in cucumber: 0.03, 0.17 (2), and 0.24 mg/kg.

Residues of fluxapyroxad (parent only and total residues) in whole melons (other than watermelons): 0.05 (2), 0.08, 0.21, and 0.24 mg/kg.

Residues of fluxapyroxad (parent only and total residues) in summer squash: 0.05, 0.07, 0.10, 0.11, and 0.14 mg/kg.

Data for the three crops when scaled to the US GAP (divide by 2) are summarized below:

Residues of fluxapyroxad (parent only and total residues) in cucumber: 0.015, 0.085 (2), and 0.12 mg/kg.

Residues of fluxapyroxad (parent only and total residues) in melons (other than watermelons): 0.025 (2), 0.04, 0.105, and 0.12 mg/kg.

Residues of fluxapyroxad (parent only and total residues) in summer squash: 0.025, 0.035, 0.05, 0.055, and 0.07 mg/kg.

The Meeting noted that the GAP is for the cucurbit fruiting vegetables group and further noted that the datasets are similar (maximum difference in the median was 2.1×). In determining which datasets to use for estimating the MRL, the similarity of the datasets was confirmed by the Kruskal-Wallis test. The Meeting decided to combine the scaled datasets for the purpose of estimating a group maximum residue level.

The combined dataset for residues of fluxapyroxad (parent only) in cucumber, melon and summer squash is 0.015, 0.025 (3), 0.035, 0.04, 0.05, 0.055, 0.07, 0.085 (2), 0.105, and 0.12 (2) mg/kg.

The combined dataset for total residues in cucurbits (whole fruit) is 0.015, 0.025 (3), 0.035, 0.04, 0.05, 0.055, 0.07, 0.085 (2), 0.105, and 0.12 (2) mg/kg.

The Meeting estimated a maximum residue level of 0.2 mg/kg for fruiting vegetables, cucurbits, together with an STMR and an HR of 0.0525 and 0.13 mg/kg respectively.

Leafy vegetables

Brassica leafy vegetables

The critical GAP for Brassica leafy vegetables is in the USA (3× 0.1 kg ai/ha applications, a 7-day retreatment interval, and a 3-day pre-harvest interval).

Residue trials in mustard greens were conducted in the USA in accordance with GAP.

Residues of fluxapyroxad (parent only) at a 3-day PHI were 0.48, 0.57, 0.90, 1.7, and 1.9 mg/kg.

Total residues were 0.93, 1.3, 1.7, 2.7, and 3.1 mg/kg.

The Meeting agreed to extrapolate the residue data for mustard greens to the Brassica leafy vegetables subgroup. The Meeting estimated a maximum residue level of 4 mg/kg for brassica leafy vegetables, together with an STMR and an HR of 1.7 and 3.1 mg/kg respectively.

Leafy vegetables (except Brassica leafy vegetables)

The critical GAP for leafy vegetables other than Brassica leafy vegetables is in the USA (3× 0.2 kg ai/ha applications with a retreatment interval of 7 days, and a 1-day pre-harvest interval).

Residue trials in head lettuce, leaf lettuce, and spinach were conducted in the USA in accordance with the cGAP for leafy vegetables (except Brassica leafy vegetables).

Residues of fluxapyroxad (parent only and total residues) at a 1-day PHI in head lettuce were 0.14, 0.47, 0.51, 0.66, and 1.9 mg/kg.

Residues of fluxapyroxad (parent only) in leaf lettuce at a 1-day PHI were 2.7 and 4.4 mg/kg.

Total residues in leaf lettuce were 2.7 and 4.4 mg/kg.

Two of the residue trials reported as leafy lettuce were for cos lettuce varieties.

Residues of fluxapyroxad (parent only) in cos lettuce at a 1-day PHI were 3.3 and 6.2 mg/kg.

Total residues in cos lettuce were 3.4 and 6.2 mg/kg.

Residues of fluxapyroxad (parent only) in spinach at a 1-day PHI were 5.2, 6.0, 6.7, 8.3, and 11.5 mg/kg.

Total residues in spinach were 5.2, 6.3, 6.8, 8.8, and 12.2 mg/kg.

The Meeting estimated a maximum residue level of 4 mg/kg for head lettuce, together with an STMR and an HR of 0.51 and 2.0 mg/kg respectively.

The Meeting noted that there were insufficient leafy and cos lettuce data for estimation of maximum residue levels.

The Meeting estimated a maximum residue level of 30 mg/kg for spinach, together with an STMR and an HR of 6.8 and 13 mg/kg respectively.

Residue data for radish tops were also available from trials conducted on radish in the USA, in accordance with the GAP for root vegetables (3× 0.1 kg ai/ha, with a 7-day PHI).

Residues of fluxapyroxad (parent only) in radish tops at a 7-day PHI were 0.2 (2), 0.7, 1, and 4 mg/kg.

Total residues in radish tops were 0.4, 0.6, 1.2, 1.7, and 5 mg/kg.

The Meeting estimated a maximum residue level of 8 mg/kg for radish leaves, together with an STMR and HR of 1.2 and 6 mg/kg (based on the highest residue of duplicate samples) respectively.

Short term intake assessment showed that residues in spinach exceed the acute reference dose of 0.3 mg/kg bw, at 180% of the ARfD, for children.

Root and tuber vegetables

The 2012 Meeting considered residue data for potato and sugar beet, in accordance with GAP in the USA (3× 0.1 kg ai/ha foliar applications with 7-day retreatment interval and a 7-day PHI, and maximum residue levels of 0.03 and 0.15 mg/kg were estimated for potato and sugar beet respectively.

The current Meeting received residue data for potato (soil application at planting), carrots and radish (both for foliar applications).

Carrot

The critical GAP for carrots (for the group root and tuber vegetables except sugar beet) is in the USA, at 3× 0.1 kg ai/ha foliar applications, with a 7-day retreatment interval and a 7-day pre-harvest interval.

Trials were conducted in the USA in accordance with GAP.

Residues of fluxapyroxad (parent only and total residues) in carrots at a 7-day PHI were 0.04, 0.05, 0.06, 0.1, and 0.5 mg/kg.

Potato

A series of residue trials was conducted in northern and southern Europe involving a single, at planting, in-furrow application at 0.24 kg ai/ha. However, there are currently no registrations for that GAP. The Meeting therefore was unable to estimate a maximum residue level for potatoes based on at planting soil application.

The 2012 Meeting considered residue data for foliar application to potatoes from trials conducted in accordance with the US GAP for root and tuber vegetables (except sugar beet) group (3× 0.1 kg ai/ha foliar applications, with a 7-day pre-harvest interval).

Residues of fluxapyroxad (parent only and total residues) in potatoes at a 7-day PHI were < 0.01 (17), and 0.02 (2) mg/kg.

Radish

The critical GAP for radish (for the group root and tuber vegetables except sugar beet) is in the USA, at 3× 0.1 kg ai/ha foliar applications, with a 7-day retreatment interval and a 7-day pre-harvest interval.

Trials were conducted in the USA in accordance with GAP.

Residues of fluxapyroxad (parent only and total) in radish roots at a 7-day PHI were 0.03, 0.04, 0.05, and 0.1 (2) mg/kg.

Sugar beet

The critical GAP for sugar beet is in the USA, at 3× 0.1 kg ai/ha foliar applications, with a 7-day retreatment interval and a 7-day pre-harvest interval. Residue data for this GAP was considered by the 2012 Meeting.

Residues of fluxapyroxad (parent only and total residues) in sugar beet roots at a 7-day PHI were 0.01 (2), 0.03 (3), 0.04 (3), 0.05 (2), and 0.06 (2) mg/kg.

The Meeting noted that the critical GAPs for root and tuber vegetables (except sugar beet) and sugar beet were the same, and considered a group maximum residue level.

The Meeting noted that the median residue for potatoes differed from those carrot and radish by > 5-fold (> 6× and > 5× respectively) and concluded that a group maximum residue level was not appropriate. The Meeting confirmed the 2012 recommendation for a maximum residue level, STMR and HR of 0.03, 0.01 and 0.02 mg/kg respectively for fluxapyroxad in potatoes. The Meeting confirmed the 2012 recommendation for a maximum residue level, STMR and HR of 0.15, 0.04, and 0.06 mg/kg respectively for fluxapyroxad in sugar beet.

The Meeting estimated a maximum residue level of 1 mg/kg for fluxapyroxad in carrot, together with an STMR and an HR of 0.06 and 0.5 mg/kg respectively. The Meeting agreed to extrapolate these values to parsnips.

The Meeting estimated a maximum residue level of 0.2 mg/kg for fluxapyroxad in radish, together with an STMR and an HR of 0.05 and 0.1 mg/kg respectively.

Celery

The critical GAP for celery is in the USA, at 3× 0.2 kg ai/ha applications, with a 7-day retreatment interval, and a 1-day pre-harvest interval.

Residues of fluxapyroxad (parent only and total residues) in US trials matching GAP were 1.3, 1.4, 1.8, and 5.2 mg/kg.

The Meeting estimated a maximum residue level of 10 mg/kg for celery, together with an STMR and an HR of 1.6 and 5.5 mg/kg respectively.

Cereals

Rice

The critical GAP for rice is in the USA, with 2× 0.15 kg ai/ha applications, a 7-day retreatment interval, and a 28-day pre-harvest interval. Residue trials matching the GAP were conducted in the USA.

Residues of fluxapyroxad (parent only) in paddy rice (with husks) at a 28-day PHI were 0.26, 0.34, 0.37, 0.59, 0.60, 0.61, 0.80, 0.92 (2), 0.94, 1.1, 1.2 (2), 1.7, and 3.7 mg/kg.

Total residues were 0.35, 0.37, 0.49, 0.59, 0.61, 0.62, 0.83, 0.94, 0.95, 0.96, 1.1, 1.2 (2), 1.7, and 3.7 mg/kg.

The Meeting estimated a maximum residue level of 5 mg/kg for rice, together with an STMR of 0.94 mg/kg.

Sorghum

Residue data for sorghum were provided to the 2012 Meeting, however at the time no maximum residue level was estimated as the data did not match any label GAP. GAPs have now been provided to the Meeting for consideration against the previously submitted data.

The GAP for sorghum in Mexico is 2× 0.1 kg ai/ha applications 14 days apart, with a 10-day pre-harvest interval. No data matching that GAP is available to the Meeting.

The GAP for sorghum in the USA is 2× 0.1 kg ai/ha applications, with a 21-day pre-harvest interval. Data from trials conducted in the USA and submitted to the 2012 Meeting match the US GAP for sorghum.

Residues of fluxapyroxad (parent only) in sorghum at a 21-day PHI were 0.13, 0.15 (2), 0.17, 0.19, 0.21, 0.24, 0.31, and 0.40 mg/kg.

Total residues in sorghum were 0.13, 0.15, 0.17, 0.19, 0.20, 0.22, 0.30, 0.32, and 0.40 mg/kg.

The Meeting estimated a maximum residue level of 0.7 mg/kg for sorghum, together with an STMR of 0.2 mg/kg.

Sugar cane

The critical GAP for sugarcane is in the USA, with 2× 0.125 kg ai/ha applications, a 14-day retreatment interval, and a 14-day pre-harvest interval. Residue trials matching GAP were conducted in the USA.

Residues of fluxapyroxad (parent only) in sugarcane at a 14-day PHI were 0.06, 0.26, 0.56, and 1.3 mg/kg.

Total residues were 0.06, 0.26, 0.58, and 1.4 mg/kg.

The Meeting concluded that there was insufficient data to estimate a maximum residue level for sugarcane.

Tree nuts

The critical GAP for fluxapyroxad in tree nuts is in the USA, with 3× 0.125 kg ai/ha applications, a 7-day retreatment interval, and a 14-day PHI.

Residue trials conducted in the USA in almonds and pecans and matching the US GAP were available to the Meeting.

Residues of fluxapyroxad (parent compound and total residues) in almond kernels at a 14-day PHI were < 0.01 (3), 0.01 and 0.02 mg/kg.

Residues of fluxapyroxad (parent compound and total residues) in pecan kernels at a 14-day PHI were < 0.01 (4), and 0.03 mg/kg.

The Meeting noted that the US GAP was for the tree nuts group and noted the similarity of the datasets for almonds and pecans (the medians were identical at 0.01 mg/kg). The Meeting decided to combine the datasets for almonds and pecans for the purpose of estimating a group maximum residue level.

Parent compound and total residues in almond and pecan kernels were: < 0.01 (7), 0.01, 0.02, and 0.03 mg/kg.

The Meeting estimated a maximum residue level of 0.04 mg/kg for tree nuts, together with an STMR and an HR of 0.01 and 0.03 mg/kg respectively.

Cotton

The 2012 Meeting considered a USA GAP and residue trials for seed treatment application to cotton, and estimated a maximum residue level of 0.01* mg/kg, together with an STMR of 0.

Residue data for foliar application to cotton was presented to the current Meeting.

The GAP for foliar application of fluxapyroxad to cotton in Brazil is 4× 0.058 kg ai/ha applications, with a 12-day retreatment interval and a 14-day pre-harvest interval. No data matching that GAP was available to the Meeting.

The USA GAP for cotton is 3× 0.1 kg ai/ha, with a 7-day retreatment interval and a 30-day pre-harvest interval. A series of trials conducted in the USA in accordance with the GAP was available to the Meeting.

Residues of parent compound in cottonseed after treatment in accordance with GAP were < 0.01, 0.01 (2), 0.03, 0.07, 0.09, 0.11 (2), and 0.13 mg/kg.

Total residues in cottonseed were < 0.01, 0.01 (2), 0.03, 0.07, 0.09, 0.11, 0.12, and 0.13 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg for cottonseed, together with an STMR of 0.07 mg/kg. The Meeting withdrew the previous maximum residue level recommendation of 0.01* mg/kg for fluxapyroxad in cottonseed.

Animal feeds

Rice straw

The critical GAP for rice is in the USA, with 2× 0.15 kg ai/ha applications, and a 28-day pre-harvest interval.

Residues of fluxapyroxad parent compound in rice straw after treatment in accordance with GAP were 1.5, 1.8, 1.9, 2.5, 2.9, 3.1, 3.6, 4.0, 4.2, 5.2, 6.8, 6.9, 7.3, 10, and 42 mg/kg (dry weight basis).

Total residues were 1.5, 1.9 (2), 2.6, 2.9, 3.2, 3.8, 4.2 (2), 5.4, 7.0 (2), 7.4, 10, and 42 mg/kg (dry weight basis).

The Meeting estimated a maximum residue level of 50 mg/kg for rice straw and fodder, dry, together with a median residue and a highest residue of 4.2 and 48 mg/kg respectively.

Sorghum forage and stover

Residue data for sorghum were provided to the 2012 Meeting, but the Meeting was unable to estimate any maximum residue levels due to the data not corresponding with any label GAP. GAPs have now been provided to the Meeting for consideration against the previously submitted data.

The GAP for sorghum in the USA is 2× 0.1 kg ai/ha applications, with a 21-day pre-harvest interval. Data from trials conducted in the USA and submitted to the 2012 Meeting match the US GAP for sorghum.

Residues of fluxapyroxad (parent only) in sorghum forage at a 7-day PHI were 1.5, 1.8, 2.3, 2.7, 2.9, 3.1, 3.5, 6.4, and 7.0 mg/kg (dry weight basis).

Total residues in sorghum forage were 1.6, 2.0, 2.4, 2.8, 3.1, 3.2, 3.5, 6.8, and 7.1 mg/kg (dry weight basis).

The Meeting estimated a median residue and a highest residue of 3.1 and 7.1 mg/kg (dry weight basis) respectively.

Residues of fluxapyroxad (parent only) in sorghum stover at a 21-day PHI were 0.72, 1.3, 1.6 (2), 2.1, 2.5 (2), 2.8, and 3.2 mg/kg (dry weight basis).

Total residues in sorghum stover were 0.72, 1.4,1.8 (2), 2.2, 2.6 (2), 2.9, and 3.3 mg/kg (dry weight basis).

The Meeting estimated a maximum residue level of 7 mg/kg, together with a median residue and a highest residue of 2.2 and 3.3 mg/kg respectively, for sorghum straw and fodder, dry (dry weight basis).

Almond hulls

The critical GAP for fluxapyroxad in tree nuts is in the USA, with 3× 0.125 kg ai/ha applications (maximum two consecutive applications), and a 14-day PHI.

Residues of fluxapyroxad (parent compound and total residues) in almond hulls were 0.88, 0.92, 1.1, 1.4 and 1.7 mg/kg.

The Meeting estimated a median residue of 1.1 mg/kg.

Cotton gin trash

The USA GAP for cotton is 3× 0.1 kg ai/ha, with a 30-day pre-harvest interval.

Residues in cotton gin trash (parent compound) were 6.9 and 8.0 mg/kg, while total residues were 6.9 and 8.1 mg/kg.

The Meeting concluded that there were insufficient data for estimation of a median residue and highest residue for cotton gin trash.

Processing studies

The Meeting received processing studies for oranges, grapes, sugarcane, and cottonseed. The 2012 Meeting received processing studies for plums, rice and sorghum. Processing factors, HR-P, STMR-P and maximum residue levels are summarized in the table below.

Plums

Based on the processing factor of 2.81 for prunes (which was the same for both parent compound and total residues), the STMR and HR of 0.44 and 0.95 mg/kg for plums, the 2012 Meeting estimated an

STMR-P, HR-P and maximum residue level of 1.2, 2.7 and 5 mg/kg respectively for prunes. The current Meeting confirmed those recommendations.

Grapes

Based on the processing factor of 4.25 for raisins (for parent compound and total residues), the STMR of 0.47 mg/kg for grapes, and the HR of 1.4 mg/kg for grapes, the Meeting estimated an STMR-P, an HR-P and a maximum residue level of 2.0, 6.0, and 15 mg/kg respectively for dried grapes.

Using the parent compound and total residues processing factor of 5.25 for grape pomace (wet), the OECD guideline value of 15% for the dry matter content of wet grape pomace, and the above STMR value for grapes, the Meeting estimated a maximum residue level and STMR-P of 150 and 16.5 mg/kg respectively for grape pomace, dry.

Rice

Based on the processing factor of 0.07 for polished rice (which was the same for parent and total residues), the maximum residue level of 5 mg/kg for rice, and the STMR of 0.94 mg/kg, the Meeting estimated a maximum residue level and an STMR-P of 0.4 and 0.066 mg/kg respectively for rice, polished.

Based on the processing factor of 0.59 (for both parent and total residues) for rice, husked produced using the parboiling process, the maximum residue level and STMR of 5 and 0.94 mg/kg respectively, the Meeting estimated a maximum residue level and an STMR-P of 3 and 0.55 mg/kg respectively for rice, husked.

Sugarcane

Although a processing study was provided, there were insufficient data for sugarcane to estimate STMR and HR values, so values for processed commodities were not estimated.

RAC	Processed commodity	PF (parent)	RAC maximum residue level	Processed commodity maximum residue level	PF (total)	RAC STMR	Processed commodity STMR-P	RAC HR	Processed commodity HR-P
Orange	Dried pulp	0.095	0.3	–	0.095	0.06 (whole fruit)	0.006	0.17 (whole fruit)	0.016
	Oil	27.5		–	27.5		1.7		4.7
	Juice	0.045		–	0.045	0.01 (pulp)	0.00045	0.01 (pulp)	0.00045
Plum	Washed plums	0.77	1.5	–	0.77	0.44	0.34	0.95	0.73
	Puree	0.83		–	0.83		0.37		0.79
	Jam	0.41		–	0.41		0.18		0.39
	Dried prunes	2.81		5	2.81		1.23		2.66
Grape	Stalks	5.95	3	–	5.95	0.47	2.8	1.4	8.3
	Grape crush	0.83		–	0.83		0.39		1.2
	Must	0.23		–	0.23		0.11		0.32
	Wet pomace	5.25		–	5.25		2.5		7.4
	Dry pomace	35		150	35		16.5		105
	Must deposit	0.88		–	0.88		0.41		1.2
	Separated must	0.26		–	0.26		0.12		0.36
	Pasteurised juice	0.345		–	0.345		0.16		0.48

RAC	Processed commodity	PF (parent)	RAC maximum residue level	Processed commodity maximum residue level	PF (total)	RAC STMR	Processed commodity STMR-P	RAC HR	Processed commodity HR-P
	Yeast deposit	2.75		–	2.75		1.3		3.9
	Red wine	0.2		–	0.2		0.094		0.28
	Rosé wine	0.23		–	0.23		0.11		0.32
	Raisins	4.25		15	4.25		2		6
Rice	Rice, polished (white rice)	0.07	5	0.4	0.07	0.94	0.066	–	–
	Hulls	4.3		–	4.3		4.04		–
	Bran	3.79		–	3.78		3.55		–
	Rice, husked (brown rice)	0.59		3	0.59		0.55		–
	Flour	0.08		–	0.08		0.08		–
Sorghum	Aspirated grain fractions	14.5	0.7	–	13.8	0.2	2.76	–	–
	Syrup	0.135		–	0.13		0.026		–
Sugar cane	Molasses	0.17	–	–	0.17	–	–	–	–
	Raw sugar	0.25		–	0.25		–		–
	Refined sugar	0.04		–	0.04		–		–
Cotton seed	Meal	0.055	0.3	–	0.055	0.07	0.004	–	–
	Hulls	0.185		–	0.185		0.013		–
	Refined oil	0.045		–	0.045		0.003		–

Residues in animal commodities

Farm animal dietary burden

Dietary burden calculations incorporating all commodities considered by the current and 2012 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	4.73	2.64	22.8	6.81	45.2	12.7	27.3	3.25
Dairy cattle	19.7	4.63	23.3	7.95	40.9	11.9	14.1	2.43
Poultry—broiler	0.985	0.985	1.27	0.898	1.37	1.37	0.35	0.35
Poultry—layer	0.985	0.985	8.53	2.69	1.37	1.37	0.947	0.947

Animal commodity maximum residue levels

The animal commodity maximum residue levels were estimated by the 2012 Meeting based on the following maximum and mean dietary burdens:

Animal (commodities)	Dietary burden (ppm)	
	Maximum	Mean
Beef cattle (mammalian meat and offal)	40.7 (Australia)	11.4 (Australia)
Dairy cattle (milk)	39.2 (Australia)	9.37 (Australia)
Poultry–layers (poultry meat, offal and eggs)	7.14 (EU)	2.10 (EU)

The Meeting noted that the dietary burdens had not changed significantly from those determined by the 2012 Meeting and confirmed its previous recommendations for meat (from mammals other than marine mammals), edible offal (mammalian), milks, poultry meat, poultry, edible offal of, 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 dietary intake assessment.

Definition of the residue (for compliance with the MRL for plant and animal commodities): *Fluxapyroxad.*

Definition of the residue (for estimation of dietary intake for plant commodities): *Sum of fluxapyroxad and 3-(difluoromethyl)- N-(3′,4′,5′-trifluoro[1,1′- biphenyl]-2-yl)-1H-pyrazole-4-carboxamide (M700F008) and 3-(difluoromethyl)- 1-(ß-D-glucopyranosyl)-N-(3′,4′,5′-triflurobipheny-2-yl)-1H-pyrzaole-4- carboxamide (M700F048) and expressed as parent equivalents.*

Definition of the residue (for estimation of dietary intake for animal commodities): *Sum of fluxapyroxad and 3-(difluoromethyl)- N-(3′,4′,5′-trifluoro[1,1′- biphenyl]-2-yl)-1H-pyrazole-4-carboxamide (M700F008) expressed as parent equivalents.*

The residue is fat soluble.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FI 0327	Banana	3		0.055[a]	0.10 [a]
FB 0018	Berries and other small fruits (except grapes)	7		1.3	3.9
VB 0040	Brassica (cole or cabbage) vegetables, Head cabbages, Flowerhead brassicas	2		0.04 (cabbage) 0.22 (others)	0.07 (cabbage) 1.7 (others)
VL 0054	Brassica leafy vegetables	4		1.7	3.1
VR 0577	Carrot	1		0.06	0.5
VS 0624	Celery	10		1.6	5.5
FS 0013	Cherries	3		0.755	2.3
SO 0691	Cotton seed	0.3	0.01*	0.07	
DF 0269	Dried grapes (=Currants, Raisins and Sultanas)	15		2.0	6.0
VC 0045	Fruiting vegetables, Cucurbits	0.2		0.0525	0.13
VA 0381	Garlic	0.6		0.23	0.27
FB 0269	Grapes	3		0.47	1.4
AB 0269	Grape pomace, dry	150		16.5	
VL 0482	Lettuce, head	4		0.51	2.0
VA 0385	Onion (bulb)	0.6		0.23	0.28
FC 0004	Oranges, Sweet, Sour	0.3		0.01[a]	0.01[a]
VR 0588	Parsnip	1		0.06	0.5
FS 2001	Peaches (including nectarine and	1.5		0.465	0.66

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
	apricots)				
FS 0014	Plums (including prunes)	1.5		0.44	0.95
VL 0494	Radish leaves (including radish tops)	8		1.2	6
VR 0494	Radish	0.2		0.05	0.1
GC 0649	Rice	5		0.94	
CM 0649	Rice, husked	3		0.55	
CM 1205	Rice, polished	0.4		0.066	
AS 0649	Rice straw and fodder, dry (dry weight)	50		4.2	48
VA 0388	Shallot	0.6		0.23	0.27
GC 0651	Sorghum	0.7		0.2	
AS 0651	Sorghum straw and fodder, dry (dry weight)	7		2.3	3.3
VL 0502	Spinach [b]	30		6.8	13
FS 0012	Stone fruits	W	2		
TN 0085	Tree nuts	0.04		0.01	0.03
OR 0691	Cotton seed oil, edible			0.003	
JF 0269	Grape juice			0.16	0.48
JF 0004	Orange juice			0.00045	0.00045
CM 1206	Rice bran, Unprocessed			3.55	
	Rice flour			0.08	
	Wine			0.11	0.23
AB 0001	Citrus pulp, dry			0.006	0.016
AB 0691	Cotton seed hulls			0.013	
AB 1203	Cotton seed meal			0.004	
	Grape must			0.11	0.32
CM 1207	Rice hulls			4.04	
AF 1053	Sorghum forage (dry)			3.0	6.9

[a] edible portion

[b] On the basis of information provided to the JMPR, , the Meeting concluded that the short-term intake of residues of fluxapyroxad from consumption of spinach for children may present a public health concern.

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Dietary Intakes (IEDIs) of fluxapyroxad were calculated for the 17 GEMS/food cluster diets using STMRs/STMR-Ps estimated by the current Meeting and by the 2012 JMPR. The results are shown in Annex 3 to the 2015 Report.

The calculated IEDIs of fluxapyroxad were 4–20% of the maximum ADI (0.02 mg/kg bw). The Meeting concluded that the long-term intakes of residues of fluxapyroxad, resulting from the uses considered by the current Meeting and by the 2012 JMPR, are unlikely to present a public health concern.

Short-term intake

The 2012 Meeting estimated an ARfD of 0.3 mg/kg bw for fluxapyroxad. The International Estimated Short Term Intakes were calculated for fluxapyroxad using the recommendations for STMRs and HRs

for raw and processed commodities in combination with consumption data for the corresponding food commodities. The results are shown in Annex 4 to the 2015 Report.

The IESTI for spinach represented 190% of the ARfD for children. On the basis of the information provided to the JMPR, the Meeting concluded that the short-term intake of fluxapyroxad from consumption of spinach may present a public health concern. The Meeting noted that no data for alternative GAPs in spinach were presented.

For the other commodities, the IESTI for fluxapyroxad calculated on the base of recommendations made by JMPR represented 0–60% of the ARfD for children, and 0–60% for the general population.

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IMAZAPIC (266)

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EXPLANATION

Imazapic is an imidazolinone herbicide developed for the control of grasses and broadleaf weeds in a variety of crops. It was first reviewed by the Meeting in 2013. The 2013 JMPR decided the following residue definition and toxicological endpoints:

Definition of the residue for plant and animal commodities (for compliance with MRLs and for estimation of dietary intakes): Imazapic

Residue is not fat-soluble.

The ADI is 0–0.7 mg/kg bw and an ARfD is unnecessary.

The 2013 JMPR received and considered the plant metabolism study and supervised residue trials on transgenic soya beans; and analytical methods, storage stability studies and processing studies on soya beans. However, at the time of the 2013 JMPR, no GAP had been approved for soya bean crops, regardless of whether they are transgenic or not. Due to the lack of approved GAP, it was not possible for the Meeting to estimate maximum residue level for soya beans.

Imazapic was included on the priority list by the CCPR at the 46th Session in 2014 for evaluation for additional MRLs by this Meeting. The current Meeting received information on use patterns now approved in Brazil. The supervised trial data provided to the 2013 Meeting are now reviewed on the basis of the new use pattern.

USE PATTERNS

Imazapic is used to control broad leaf and grassy weeds. It is formulated as a liquid or granular product either as a solo product or in combination with other active substances for use on pulses, cereal grains, grasses for sugar, oilseeds, and straw, forage and fodder of cereal grains. The use of imazapic, in combination with imazapyr, has been approved in Brazil only for soya bean cultivars resistant to imidazolinone herbicides as shown below.

Table 1 Registered use of imazapic relevant to the residue evaluation by the current Meeting.

Country	Formulation Type and g/kg [a] (Other active ingredient)	F/G /P	Application rate				PHI days	Notes Timing
			Method	No. per crop and season	Water L/ha[2/]	Rate kg ai/ha [b]		
Pulses: Soya bean								
Brazil	WG 175 (imazapyr)	F	Ground spraying	1	100-200	0.014 - 0.0175	60	Apply only for soya bean cultivars tolerant to imidazolinone herbicides. Early to normal post-emergence of infesting weeds
Brazil	WG 175 (imazapyr)	F	Aerial spraying	1	40-50	0.014 - 0.0175	60	

[a] In acid equivalents.

[b] Calculated from the dose of the formulation on the label and concentration of the active ingredient (acid equivalents) in the formulation.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The 2013 Meeting received residue data from supervised field trials conducted in Brazil on soya bean cultivars tolerant to imidazolinone herbicides, which were summarized in the Evaluation of the 2013 JMPR and reproduced here with some editorial modification, such as information on the analytical methods and storage, and additional information related to the application of imazapic.

Application rates and residue concentrations were reported as imazapic acid equivalents. 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 average concentration is 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 the higher(est) result from these trials was chosen for the estimation of a maximum residue level. Residues from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels and they are underlined.

Soya beans

The formulation containing imazapic and imazapyr was approved in Brazil for use only on soya bean cultivars tolerant to imidazolinone herbicides. The following trials were conducted on GM soya bean cultivars to which the mutated AHAS gene (CSR1-2) of *Arabidopsis thaliana* was introduced for imidazolinone tolerance.

During the 2006/2007 growing season, eight field trials were carried out in Brazil to determine the residues levels of imazapic in soya bean after treatment with a mixed WG formulation of imazapic and imazapyr. In all trial sites, one trial plot was untreated to provide control samples and one trial plot received one post-emergence application at a rate of 0.0175 kg imazapic/ha (and 0.0525 kg imazapyr/ha) 60 days before harvest (BBCH 24-75). In three trials, the application was performed 40, 60, 80, 100 and 120 days before harvest, each on a separate plot. Samples were taken 60 days after the application (DALA) in all trials; but in three trials, additional samplings were performed 40, 80, 100 and 120 DALA. The soya bean samples were stored frozen until analysis. Soya bean samples were analysed for imazapic using Method SOP-PA.0249.

During the 2007/2008 growing season, a field trial was carried out in Brazil to determine the residues levels of imazapic in soya bean after treatment with a mixed formulation of imazapic and imazapyr. One trial plot was untreated to provide control samples, and one trial plot received one foliar post-emergence spray application at a rate of 0.0175 kg imazapic/ha (and 0.0525 kg imazapyr/ha), either 40, 60, 80, 100 or 120 days before harvest. Samples were taken 40, 60, 80, 100 or 120 days after the application. The soya bean samples were analysed for imazapic and the two metabolites using Method SOP-PA.0288.

During the 2010 growing season, two field trials were carried out in Brazil to determine the residues levels of imazapic in soya bean after treatment with a mixed formulation of imazapic and imazapyr. At both trial sites, one trial plot was untreated to provide control samples and four trial plots received one foliar post-emergence spray application at a rate of 0.0175 kg imazapic/ha (and 0.0525 kg imazapyr/ha), 20, 40, 60 or 80 days before harvest. Samples of soya bean grain were taken 20, 40, 60 and 80 days after the application. Soya bean samples were analysed for residues using Method SOP-PA.0288.

During the 2011 growing season, five field trials were carried out in Brazil to determine the residues levels of imazapic in transgenic soya bean after treatment with a mixed formulation of imazapic and imazapyr. At all trial sites, one trial plot was untreated to provide control samples, and one trial plot received one post-emergence application at a rate of 0.0175 kg imazapic/ha (and 0.0525 kg imazapyr/ha), 60 days before harvest (BBCH 66-73). At one trial with five plots, the application was performed 20, 40, 60, 80 and 100 days before harvest. Samples of soya bean grain were taken 60 days after the application (DALA) at all trials; at one trial, additional samplings were performed 20, 40, 80 and 100 DALA, and at one trial aspirated grain fractions were also sampled. Soya bean samples were analysed for residues using Method SOP-PA.0288.

Table 2 Residues of imazapic in imidazolinone-tolerant soya beans from supervised trials conducted in Brazil

Year Location (Variety)	Application rate				DALT (days)	Imazapic (mg/kg)	Study code Doc ID (Trial No.) BBCH at harvest
	Method	Rate kg ai/ha	No. date	Timing BBCH			
GAP in Brazil	Ground spray	0.014-0.0175	1		PHI 60		-
	Aerial spray	0.014-0.0175	1		PHI 60		-
2006/2007 Santo Antonio de Posse, Sao Paulo (CV 603)	n.r.	0.0175	1 19.02.07 30.01.07 10.01.07 21.12.06 01.12.06	 78 72 65 53 38	 40 60 80 100 120	 0.08 < 0.05 < 0.05 < 0.05 < 0.05	RF-1088-06 2008/1097470 [a] (EC-CD-BRUA/ 1088-06) BBCH 89
2006/2007 Santo Antonio de Posse, Sao Paulo (CV 603)	n.r.	0.0175	1 30.01.07	 24 [b]	 60	 < 0.05	RF-1088-06 2008/1097470 (EC-R-BRUA/ 1088-06) BBCH 89
2006/2007 Santo Antonio de Goias, Goias (CV 603)	n.r.	0.0175	1 25.02.07 05.02.07 16.01.07 27.12.06 07.12.06	 77 71 66 59 39	 40 60 80 100 120	 0.15 0.08 < 0.05 < 0.05 < 0.05	RF-1088-06 2008/1097470 (EC-CD-BRUB/ 1088-06) BBCH 97
2006/2007 Santo Antonio de Goias, Goias (CV 603)	n.r.	0.0175	1 05.02.07	 71	 60	 0.15	RF-1088-06 2008/1097470 (EC-R-BRUB/ 1088-06) BBCH 97
2006/2007 Brasilia Distrito Federal do Brasil (CV 603)	n.r.	0.0175	1 09.02.07	 75	 60	 0.10	RF-1088-06 2008/1097470 (EC-R-BRUC/ 1088-06) BBCH 97
2006/2007 Uberaba, Minas Gerais (CV 603)	n.r.	0.0175	1 04.03.07 13.02.07 23.01.07 03.01.07 14.12.06	 77 73 51 29 19	 40 60 80 100 120	 0.19 0.23 < 0.05 < 0.05 < 0.05	RF-1088-06 2008/1097470 (EC-CD-BRVA/ 1088-06) BBCH 97
2006/2007 Uberaba, Minas Gerais (CV 603)	n.r.	0.0175	1 13.02.07	 73	 60	 0.25	RF-1088-06 2008/1097470 (EC-R-BRVA/ 1088-06) BBCH 97
2006/2007 Londrina, Parana (CV 603)	n.r.	0.0175	1 03.01.07	 67	 60	 < 0.05	RF-1088-06 2008/1097470 (EC-R-BRTA/ 1088-06) BBCH 86
2007/2008 Santo Antonio de Posse, Sao Paulo (CV 127)	spray	0.0175	1 13.02.08 24.01.08 04.01.08 15.12.07 25.11.07	 n.r.[c] 75 66 51 13	 40 60 80 100 120	 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	1273-07 2010/1010261 2010/1079212 (G080102) BBCH 86 Storage: 613 d
2010	spray	0.0175	1				324476

Year Location (Variety)	Application rate				DALT (days)	Imazapic (mg/kg)	Study code Doc ID (Trial No.) BBCH at harvest
	Method	Rate kg ai/ha	No. date	Timing BBCH			
Ponta Grossa, Parana (L 08)			24.03.10 04.03.10 12.02.10 23.01.10	83 75 68 66	20 40 60 80	< 0.01 0.02 0.07 0.03	2010/1127505 (G100005) BBCH 91
2010 Santo Antonio de Posse, Sao Paulo (CV 127)	spray	0.0175	1 13.05.10 23.04.10 04.04.10 14.03.10	 89 87 77 73	 20 40 60 80	 < 0.01 < 0.01 0.05 0.01	324476 2010/1127505 (G100006) BBCH 89
2011 Ponta Grossa, Parana (BRZ 08 200151)	spray	0.0175	1 20.05.11 30.04.11 10.04.11 21.03.11 01.03.11	 79 75 73 64 62	 20 40 60 80 100	 < 0.01 < 0.01 0.05 0.12 < 0.01	324447 2012/3000423 (G100575) BBCH 83
2011 Senador Canedo, Goias (BRZ 5384)	spray	0.0175	1 31.01.11	 66	 60	 < 0.01	324447 2012/3000423 (G100576) BBCH 87
2011 Anapolis, Goias (BRZ 5384)	spray	0.0177	1 04.02.11	 69	 60	 < 0.01	324447 2012/3000423 (G100577) BBCH 85
2011 Santo Antonio de Posse, Sao Paulo (BRZ 08)	spray	0.0175	1 12.05.11	 73	 60	 0.23	324447 2012/3000423 (G100578) BBCH 89
2011 Castro, Parana (BRZ 08 200151)	spray	0.0175	1 22.04.11	 71	 60	 0.07	324447 2012/3000423 (G100579) BBCH 83

[a] Amendment to Doc ID. 2007/1065863

[b] Unlikely value.

[c] From the stage at the time of application for 60 DALA and the stage at harvest, the growth stage at the application for the 60 DALT is speculated to be between BBCH 79 and 83.

BBCH 83: 30% of pods ripe (beans final colour, dry and hard)

BBCH 85: 50% of pods ripe (beans final colour, dry and hard)

BBCH 86: 60% of pods ripe (beans final colour, dry and hard)

BBCH 87: 70% of pods ripe (beans final colour, dry and hard)

BBCH 89: Full maturity: approx. all pods are ripe; beans final colour, dry and hard (= Harvest maturity)

BBCH 91: About 10% of leaves discoloured or fallen

BBCH 97: Above ground parts of plants dead

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

The 2013 Meeting received information on effects of heating in water and processing on imazapic residues in soya bean. The estimated processing factors by the 2013 JMPR are reproduced below.

Table 3 Summary of processing factors for soya bean processing

Processed commodity	N	Processing factor	Mean or best estimate
Meal	3	1.00, 1.00, 1.13	1.04
Defatted meal	1	1.29	1.29
Toasted Meal	1	0.88	0.88
Toasted Defatted Meal	1	1.14	1.14
Oil	2	0.13, 0.14	0.14
Laminated Soya Bean	1	0.71	0.71
Flaked Soya Bean	1	0.50	0.50
Hulls	2	1.00, 1.00	1.00

APPRAISAL

Imazapic is an imidazolinone herbicide for the control of grasses and broadleaf weeds. It was reviewed for the first time by JMPR in 2013 when the residue definition was established for plant and animal commodities to be imazapic for compliance with the MRL and for estimation of dietary intake (The residue is not fat soluble). The Meeting established an ADI of 0–0.7 mg/kg bw and that no ARfD was necessary.

The 2013 JMPR received and considered the plant metabolism study and supervised residue trials on transgenic soya beans; analytical methods, storage stability studies and processing studies on soya beans.

Imazapic was included in the priority list by the CCPR at its Forty-sixth Session in 2014 for evaluation for additional MRLs by this Meeting. The current Meeting received information on the registration of imazapic for application on soya bean cultivars tolerant to imidazolinone herbicides in Brazil. The information on supervised residue trials on imidazolinone-tolerant soya beans provided to the 2013 JMPR is reviewed by the current Meeting against the new GAP in Brazil.

Results of supervised residue trials on crops

The 2013 Meeting received supervised trial data for imazapic on transgenic soya beans. The current Meeting evaluated the data against the new GAP for soya bean cultivars tolerant to imidazolinone herbicides.

Soya bean (dry)

A total of 16 supervised trials were conducted on imidazolinone-tolerant soya beans (transgenic) in different years in Brazil.

The new GAP in Brazil allows a single application of a WG formulation of imazapic (also containing imazapyr) to imidazolinone-tolerant cultivars at the rate of 0.014–0.0175 kg ai/ha (in acid equivalents; for both ground and aerial application) with a PHI of 60 days. For ground applications, the water volume should be 100–200 L/ha and for the aerial application, 40–50 L/ha. The trials employed an application rate of 0.0175 kg ai/ha and the application volume of 200 L/ha.

In one trial in the 2007/2008 growing season, the samples were stored for about 600 days; imazapic was demonstrated to be stable for up to 10 months, the longest storage period tested for imazapic in soya bean. The result of this trial was < 0.01 mg/kg.

Residues arising from the independent supervised residue trials following the critical GAP in Brazil were, in rank order (n=12): < 0.01, < 0.01, < 0.05, < 0.05, 0.05, 0.07, 0.07, 0.10, 0.12, 0.15, 0.23 and 0.25 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.07 mg/kg.

Fate of residues during processing

Processing

The 2013 Meeting received information on processing of soya beans. The processing factor for imazapic in soya bean processed products is described below.

Processed commodity	N	Processing factor	Best estimate	STMR-P mg/kg
Soya bean				0.07 (STMR)
Oil	2	0.13, 0.14	0.14	0.01

The residues of imazapic concentrate marginally in defatted meal (processing factor of 1.29), and toasted defatted meal (1.14).

For the purpose of calculating the animal dietary burden, the Meeting calculated median residues for soya bean meal and hulls to be 0.09 mg/kg and 0.07 mg/kg, respectively, using the STMR of soya bean and the processing factors of 1.29 (highest of similar processed commodities) and 1.00, respectively.

Residues in animal products

Estimation of dietary burdens

The maximum and mean dietary burdens were calculated by the 2013 JMPR using the highest residues or median residues of imazapic estimated at that Meeting on a basis of the OECD Animal Feeding Table. As the highest maximum and mean dietary burden for estimating maximum residue levels and STMRs for foods of bovine origin were calculated on the basis of a ration of 100% grass forage, the inclusion of soya bean feed items, with significantly lower residue levels, would not have any measurable impact on the highest maximum and mean dietary burden.

The addition of soya bean feed items in the calculation of dietary burdens increases by approximately 0.2% the highest maximum and mean dietary burden for poultry. The highest maximum dietary burden calculated at this Meeting (9.65 ppm in feed as compared to 9.63 ppm calculated in 2013) was still lower than the dose of 10.9 ppm in the diet used in the metabolism study in which the TRR in all edible tissues were below the LOQ of 0.01 mg/kg

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed in Annex 1 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 and for estimation of dietary intake): *Imazapic*.

Residue is not fat-soluble.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
VD 0541	Soya bean (dry)	0.5		0.07	
OR 0541	Soya bean oil, refined			0.01	

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
AB 1265	Soya bean meal			0.09	
AB 0641	Soya bean hulls			0.07	

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Dietary Intakes (IEDIs) of imazapic were calculated for the 17 GEMS/Food cluster diets using STMRs estimated by the 2013 JMPR and STMR/STMR-P for soya bean and soya bean oil estimated by the current Meeting (see Annex 3 to the 2015 Report). The ADI is 0–0.7 mg/kg bw and the calculated IEDIs were in the same range as those calculated by the 2013 JMPR using the 13 GEMS/Food Cluster Diet (0% of the maximum ADI). The Meeting confirmed its conclusion in 2013 that the long-term intake of residues of imazapic resulting from the uses considered by the current JMPR is unlikely to present a public health concern.

Short-term intake

The 2013 JMPR decided that an ARfD is unnecessary. The current Meeting therefore concluded that the short-term intake of residues of imazapic is unlikely to present a public health concern.

REFERENCES

Doc. ID	Author(s)	Year	Title, Source, published or not
	FAO/WHO	2014	FAO Plant Production and Protection Paper 220, Pesticide Residues in Food 2013, Evaluations 2013 Part I – Residues, Rome, Italy Published
	FAO/WHO	2014	FAO Plant Production and Protection Paper 219, Pesticide Residues in Food 2013, Report 2013, Rome, Italy Published
2007/1065863	Jones B.	2007	Estudo de residuos de Imazapyr e Imazapic em soja cultivance (graos) apos tratamento com BAS 714 01 H em condicoes de campo no Brasil BASF SA, Guaratingueta, Brazil Unpublished
2008/1097470	Resende G., Souza C.	2008	Amendment report 01 RF-1088-06 - Study of residues of Imazapyr and Imazapic in soybean cultivance (grains) after treatment with BAS 714 01 H, under field conditions in Brazil BASF SA, Guaratingueta, Brazil Unpublished
2008/1097471	Resende G.	2008	Amendment report 02 RF-1088-06 - Study of residues of Imazapyr and Imazapic in soybean cultivance (grains) after treatment with BAS 714 01 H, under field conditions in Brazil BASF SA, Guaratingueta, Brazil Unpublished
2010/1010261	Resende G., Marinho E.	2010	Study of residues of Imazapyr and Imazapic in soybean cultivance (grains) after treatment with BAS 714 01 H under field conditions in Brazil for import tolerance BASF SA, Guaratingueta, Brazil Unpublished
2010/1079212	Resende G.	2010	Amendment 01 - Final report 1273-07 - Study of residues of Imazapyr and Imazapic in soybean cultivance (grains) after treatment with BAS 714 01 H under field conditions in Brazil for import tolerance

Doc. ID	Author(s)	Year	Title, Source, published or not
			BASF SA, Guaratingueta, Brazil Unpublished
2010/1127505	Jones B., Takahashi J.	2011	Study of residues of Imazapyr and Imazapic in cultivance soybean (grains) after treatment with BAS 714 01 H under field conditions in Brazil BASF SA, Guaratingueta, Brazil Unpublished
2012/3000423	Jones B., Cardoso B.	2012	Residue study of Imazapyr and Imazapic in GMO soybean grains and aspirated grain fraction (AGF) after treatment with BAS 714 01 H under field conditions in Brazil BASF SA, Guaratingueta, Brazil Unpublished

IMAZAPYR (267)

First draft prepared by Makoto Irie, Ministry of Agriculture, Forestry and Fisheries, Tokyo, Japan

EXPLANATION

Imazapyr is a broad-spectrum herbicide in the imidazolinone family. It was evaluated at the 2013 JMPR for the first time for toxicology and residues. The 2013 JMPR allocated an ADI of 0-3 mg/kg bw, and ARfD was considered unnecessary. It also determined that the definition of residue was imazapyr for plant and animal commodities (for compliance with MRLs and for estimation of dietary intake). It recommended maximum residue levels for various commodities.

The 2013 JMPR received and considered the plant metabolism study and supervised residue trials on imidazolinone-tolerant soya beans; and analytical methods, storage stability studies and processing studies on soya beans. However, at the time of the 2013 JMPR, no GAP had been approved for soya beans, regardless of transgenic or not. Due to the lack of approved GAP, the Meeting did not estimate a maximum residue level for soya beans.

Imazapry was included on the priority list by the CCPR at the 46th Session in 2014 for evaluation for additional MRLs by this Meeting. The current Meeting received information on analytical methods, use patterns and supervised residue trials to support estimation of maximum residue levels for soya bean and grasses.

RESIDUE ANALYSIS

Analytical methods

The Meeting received information on the analytical method (Method M3023) for the determination of imazapyr in grass, forage and hay (Flatcher, 1999: IZ-244-011).

Residues of imazapyr were extracted from forage and hay of grass with acidic acetone-water (50:148:2 acetone/water/conc. hydrochloric acid). After centrifugation, a 20 mL aliquot was partitioned with dichloromethane. The dichloromethane layer was subsequently cleaned up on a SCX cartridge followed by a RP102 cartridge. The eluted sample was evaporated to dryness and re-dissolved in water for capillary electrophoresis analysis or LC-MS confirmatory analysis.

The M3023 method was validated for the determination of residues of imazapyr in grass, forage and hay. The results were summarized in Table 1. The LOQ for imazapyr was 0.5 mg/kg.

Table 1 Recovery results obtained for the determination of imazapyr by the method M3023

Commodity	Fortification level (mg/kg)	N	Recovery (%)	Mean recovery (%)	Reference Method
Grass, forage	0.5	2	81, 85	83	IZ-244-011
	1.0	2	82, 82	82	M 3023
	5.0	2	85, 86	86	
	50	2	86, 88	87	
Grass, hay	0.5	2	81, 85	83	
	1.0	2	82, 85	84	
	5.0	2	81, 87	84	
	50	2	86, 86	86	

USE PATTERN

The Meeting received labels from Brazil and the USA. The authorized uses relevant to the supervised residue trials data submitted to the current Meeting are summarized in Table 2.

Table 2 Registered uses of imazapyr relevant to the residue evaluation by the current Meeting

Crop	Country	Formulation		Application				PHI, days
		Type	Conc. of imazapyr	Method	Rate kg ai/ha	Volume L/ha	No. max	
Pulses								
Soya bean (imidazolinone-tolerant)	Brazil	WG	525 g/kg	Ground application	0.042-0.053	100-200	1	60
				Aerial application	0.042-0.053	40-50	1	
Straw, fodder and forage of cereal grains and grasses (including buckwheat fodder)								
Bermudagrass and Bahiagrass	USA	SL	278 g/L	Ground application [a]	0.035-0.84[b]	47-935	1	7[c]
				Aerial application [a]	0.035-0.84[b]	19-281	1	7[c]

[a] Spot applications: may not exceed more than 1/10 of the area to be grazed or cut for hay.

[b] Rate per treated hectare

[c] Do not cut forage grass for hay for 7 days after application. There are no grazing restrictions.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on imazapyr supervised field trials for the following crops.

Group	Commodity	Table
Pulses	Soya bean (dry)	Table 3
Straw, fodder and forage of grasses	Grasses	Table 4

Imazapyr formulation was applied by foliar treatment. Each of the field trial sites generally consisted of an untreated control plot and a treated plot. Residues, 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.

Pulses

Soya bean (dry)

The 2013 Meeting received supervised residue trials on imidazolinone-tolerant soya bean conducted in Brazil, which were summarized in the Evaluation of the 2013 JMPR. Table 3 was reproduced to add information related to the application of imazapyr.

Table 3 Imazapyr residues on imidazolinone-tolerant soya bean seeds from supervised trials in Brazil

Soya bean, seed country, year (variety)	Application							DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	Growth Stage [a]		no.			
					applica -tion	harvest				
GAP, Brazil	WG	0.042-0.053		100-200 (ground) 40-50 (aerial)			1	60		
Brazil, 2007 Santo Antônio de Goiás/GO [b] (CV 603)	SL	0.072		200	77 71 66 59 39	97 97 97 97 97	1	40 60 80 100 120	1.8 1.7 2.0 < 0.05 < 0.05	2008/1097472 Resende, 2008
Brazil, 2007 Santo Antônio de Goiás/GOc[c] (CV 603)	SL	0.072		200	67	97	1	60	1.4	Sampling to analysis: 46-87 days
Brazil, 2007 Uberaba/MG [d] (CV 603)	SL	0.072		200	78 73 51 29 19	97 97 97 97 97	1	40 60 80 100 120	1.7 1.3 1.5 0.05 < 0.05	
Brazil, 2007 Uberaba/MG [e] (CV 603)	SL	0.072		200	73	97	1	60	2.0	
Brazil, 2007 Brasilia/DF (CV 603)	SL	0.072		200	75	97	1	60	1.9	
Brazil, 2007 Santo Antônio de Posse/SP [f] (CV 603)	SL	0.072		200	72	89	1	60	0.92	
Brazil, 2007 Santo Antônio de Posse/SP [g] (CV 603)	SL	0.072		200	29 24 18 15 12	89 89 89 89 89	1	40 60 80 100 120	0.06 0.41 0.08 < 0.05 < 0.05	
Brazil, 2007 Londrina/PR (CV 603)	SL	0.072		200	71	86	1	60	< 0.05	
Brazil, 2007 Santo Antônio de Goiás/GO [h] (CV 603)	WG	0.053		200	77 71 66 59 39	97 97 97 97 97	1	40 60 80 100 120	1.4 0.45 0.30 0.07 < 0.05	2008/1097470 Resende, 2008
Brazil, 2007 Santo Antônio de Goiás/GO [i] (CV 603)	WG	0.053		200	71	97	1	60	1.3	Sampling to analysis: 49-65 days
Brazil, 2007 Uberaba/MG [j] (CV 603)	WG	0.053		200	77 73 51 29 19	97 97 97 97 97	1	40 60 80 100 120	2.3 2.5 0.09 < 0.05 < 0.05	
Brazil, 2007 Uberaba/MG [k] (CV 603)	WG	0.053		200	73	97	1	60	3.0	
Brazil, 2007 Brazilia/DF	WG	0.053		200	75	97	1	60	1.3	

Soya bean, seed country, year (variety)	Application							DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	Growth Stage [a]		no.			
					applica -tion	harvest				
(CV 603)										
Brazil, 2007 Santo Antônio de Posse/SP [l] (CV 603)	WG	0.053		200	78 72 65 53 38	89 89 89 89 89	1	40 60 80 100 120	0.85 0.48 0.08 < 0.05 < 0.05	
Brazil, 2007 Santo Antônio de Posse/SP [m] (CV 603)	WG	0.053		200	24	89	1	60	0.27	
Brazil, 2007 Londrina/PR (CV 603)	WG	0.053		200	67	86	1	60	< 0.05	
Brazil, 2008 Santo Antônio de Posse/SP (CV 127)	WG	0.053	0.026	200	79-83 75 66 51 13	86 86 86 86 86	1	40 60 80 100 120	0.10 0.07 0.01 < 0.01 < 0.01	2010/1010261 2010/1079212 Sampling to analysis: 613 days
Brazil, 2010 Ponta Grossa /PR (L 08)	WG	0.053	0.026	200	83 75 68 66	91 91 91 91	1	20 40 60 80	< 0.01 0.07 0.90 1.0	2010/1127505 Jones, 2011
Brazil, 2010 Santo Antônio de Posse/SP (CV 127)	WG	0.053	0.026	200	89 87 77 73	89 89 89 89	1	20 40 60 80	< 0.01 < 0.01 0.35 0.20	Sampling to analysis: 27-78 days
Brazil, 2011 Ponta Grossa /PR (BRZ 08-200151)	WG	0.053	0.026	200	79 75 73 64 62	83 83 83 83 83	1	20 40 60 80 100	< 0.01 < 0.01 0.26 0.83 0.25	2012/3000423 Jones, 2012 Sampling to analysis: 162-273 days
Brazil, 2011 Senador Canedo/PR (BRZ 5384)	WG	0.053	0.026	200	66	87	1	60	0.11	
Brazil, 2011 Anápolis/GO (BRZ 5384)	WG	0.053	0.026	200	69	85	1	60	0.07	
Brazil, 2011 Santo Antônio de Posse/SP (BRZ 5384))	WG	0.053	0.026	200	73	89	1	60	1.3	
Brazil, 2011 Castro/PR (BRZ 08-200151	WG	0.053	0.026	200	71	83	1	60	0.55	

[a] Code of BBCH Scale

[b] Test site: Rodovia Goiânia, km 12 - Nova Veneza. Planting: 7/11/2006 – Harvest 6/4/2007

[c] Test site: Rodovia Goiânia, km 12 - Nova Veneza. Planting: 7/11/2006 – Harvest 6/4/2007

[d] Test site: Rua Afonso Rato, 301. Planting 21/11/2006 – Harvest 13/4/2007

[e] Test site: Rua Afonso Rato, 301. Planting 21/11/2006 – Harvest 14/4/2007

[f] Test site: Rodovia SP 340, km 144. Planting 11/11/2006 – Harvest 31/3/2007

[g] Test site: Rodovia SP 340, km 144. Planting 11/11/2006 – Harvest 31/3/2007

[h] Test site: Rodovia Goiânia, km 12 - Nova Veneza. Planting: 7/11/2006 – Harvest 6/4/2007

[i] Test site: Rodovia Goiânia, km 12 - Nova Veneza. Planting: 7/11/2006 – Harvest 6/4/2007

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[k] Test site: Rua Afonso Rato, 301. Planting 21/11/2006 – Harvest 14/4/2007

[l] Test site: Rodovia SP 340, km 144. Planting 11/11/2006 – Harvest 31/3/2007

[m] Test site: Rodovia SP 340, km 144. Planting 11/11/2006 – Harvest 31/3/2007

Straw, fodder and forage of grasses

Fourteen field trials were conducted in the USA to determine the residue level of imazapyr on grasses. The SL formulation was applied once as broadcast foliar application. Samples of forage were collected at 0 (pre-treatment), 0.1, 7, 14, 28 and 56 days after application. Hay samples were collected on the same day as forage and left to dry before being sampled. Residue concentrations were not adjusted for moisture content and expressed on as received basis.

The Method M 3023 was used for analysis of imazapyr residues in grass forage and hay samples quantifying the analyte by capillary electrophoresis with an LOQ of 0.50 mg/kg.

Table 4 Imazapyr residues on grass from supervised trials

Grass country, year (variety)	Application					DALA Days	Residues[a], mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Analytical portion	no.			
GAP, USA		0.035-0.84			1	7 (hay) no restriction (forage)		
USA, 1996 York/NE (bluegrass)	SL	0.83	185	Forage	1	0.1 7 14 28	65, 66 (66) 6.0, 6.6 (6.3) 4.3, 4.5 (4.4) 2.3, 2.6 (2.5)	IZ-731-029 Khunachak, 1999
				Hay	1	0.1 7 14 28	75, 88 (82) 17, 20 (19) 9.8, 10 (9.9) 4.4, 4.6 (4.5)	Sampling to analysis: 378-409 days
USA, 1996 Newport/AR (common bermudagrass)	SL	0.83	186	Forage	1	0.1 7 14 28	32, 42 (37) 7.4, 7.9 (7.6) 3.9, 4.3 (4.1) 1.5, 1.5 (1.5)	IZ-731-019 Khunachak, 1998
				Hay	1	0.1 7 14 28	112, 115 (113) 18, 18 (18) 8.2, 8.3 (8.3) 2.0, 2.1 (2.1)	Sampling to analysis: 301-323 days
USA, 1996 Hawkinsville/ GA (common bermudagrass)	SL	0.84	243	Forage	1	0.1 7 14 28	50, 57 (54) 6.8, 9.9 (8.3) 4.3, 5.4 (4.8) 0.72, 0.95 (0.84)	IZ-731-022 Khunachak, 1998
				Hay	1	0.1 7 14 28	111, 151 (131) 13, 13 (13) 9.1, 11 (9.9) 2.2, 2.3 (2.2)	Sampling to analysis: 310-339 days
USA, 1996 Payette/ID (tall fescue grass)	SL	0.85	264	Forage	1	0.1 7 14 28 58	38, 38 (38) 3.6, 3.9 (3.7) 1.6, 1.6 (1.6) 1.0, 1.1 (1.1) < 0.50	IZ-731-023 Khunachak, 1998
				Hay	1	0.1 7 14 28 58	132, 168 (150) 9.5, 15 (12) 4.5, 4.9 (4.7) 2.5, 2.6 (2.5) 0.66	Sampling to analysis: 298-350 days
USA, 1996 Sears/MI (bromegrass)	SL	0.83	208	Forage	1	0.1 7 14 28	33, 34 (33) 0.59, 0.81 (0.70) < 0.50, < 0.50 < 0.50, < 0.50	IZ-731-024 Khunachak, 1998

Grass country, year (variety)	Application					DALA Days	Residues[a], mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Analytical portion	no.			
				Hay	1	0.1 7 14 28	65, 65 (65) 0.88, 2.1 (1.5) 0.51, 0.84 (0.67) < 0.50, 0.56	Sampling to analysis: 481-505 days
USA, 1996 Halsey/OR (bluegrass)	SL	0.84	373	Forage	1	0.1 7 14	69, 81 (75) 4.7, 5.6 (5.2) 3.1, 3.5 (3.3)	IZ-731-025 Khunachak, 1999
				Hay	1	0.1 7 14	139, 139 (139) 10, 11 (11) 5.2, 5.3 (5.3)	Sampling to analysis: 319-333 days
USA, 1996 Germansville/ PA (tall fescue grass)	SL	0.87	231	Forage	1	0.1 7 14 28	38, 62 (50) 6.8, 6.9 (6.9) 2.2, 3.4 (2.8) 1.8, 1.9 (1.8)	IZ-731-026 Khunachak, 1999
				Hay	1	0.1 7 14 28	153, 186 (169) 18, 22 (20) 10, 11 (11) 4.1, 4.3 (4.2)	Sampling to analysis: 391-414 days
USA, 1996 Verona/WI (bromegrass)	SL	0.84	198	Forage	1	0.1 7 14 28	64, 71 (68) 3.9, 4.5 (4.2) 1.9, 2.1 (2.0) 0.70, 0.80 (0.75)	IZ-731-027 Khunachak, 1999
				Hay	1	0.1 7 14 28	164, 197 (181) 12, 13 (12) 3.7, 3.9 (3.8) 1.7, 1.9 (1.8)	Sampling to analysis: 315-343 days
USA, 1996 Spearman/TX (tall fescue grass)	SL	0.83	276	Forage	1	0.1 7 14 28	39, 50 (44) 4.1, 4.7 (4.4) 3.0, 4.3 (3.6) 2.0, 2.7 (2.3)	IZ-731-028 Khunachak, 1999
				Hay	1	0.1 7 14 28	189, 196 (193) 11, 14 (13) 9.8, 11 (10) 3.7, 4.1 (3.9)	Sampling to analysis: 286-313 days
USA, 1997 Grand Island/ NE (bluegrass)	SL	0.85	187	Forage	1	0.1 7 14 28	63, 66 (65) 4.4, 4.6 (4.5) 2.6, 2.6 (2.6) 0.81, 0.92 (0.87)	IZ-731-030 Garrett, 1999
				Hay	1	0.1 7 14 28	140, 159 (150) 10, 10 (10) 5.0, 5.1 (5.1) 1.9, 1.9 (1.9)	Sampling to analysis: 192-231 days
USA, 1997 Hillsboro/OR (tall fescue grass)	SL	0.82	205	Forage	1	0.1 7 14 28	34, 39 (36) 4.8, 5.1 (5.0) 1.9, 2.6 (2.2) 1.5, 1.9 (1.7)	IZ-731-031 Garrett, 1999
				Hay	1	0.1 7 14 28	121, 164 (143) 24, 27 (25) 5.8, 6.6 (6.2) 3.6, 3.6 (3.6)	Sampling to analysis: 204-258 days
USA, 1997 Brookshire/TX (common Bermuda grass)	SL	0.86	231	Forage	1	0.1 7 14 28	60, 63 (61) 10, 12 (11) 10, 11 (11) 6.1, 6.4 (6.2)	IZ-731-032 Garrett, 1999
				Hay	0 1	0 0.1 7 14 28	< 0.50, 1.3 129, 149 (139) 23, 24 (24) 16, 20 (18) 7.9, 8.6 (8.2)	Sampling to analysis: 184-224 days

Grass country, year (variety)	Application					DALA Days	Residues[a], mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Analytical portion	no.			
USA, 1997 Noblesville/IN (Bluegrass)	SL	0.85	213	Forage	1	0.1 7 14 28 56	97, 98 (97) 6.0, 6.8 (6.4) 3.1, 3.5 (3.3) 1.5, 1.7 (1.6) < 0.50, < 0.50	IZ-731-033 Garrett, 1999 Sampling to analysis: 85-174 days
				Hay	1	0.1 7 14 28 56	261, 277 (269) 11, 12 (12) 5.6, 6.5 (6.0) 2.3, 2.4 (2.4) < 0.50, < 0.50	
USA, 1997 Read/CO (bromegrass)	SL	0.84	213	Forage	1	0.1 7 14 28	27, 28 (28) 6.5, 7.9 (7.2) 4.9, 5.3 (5.1) 1.8, 1.8 (1.8)	IZ-731-034 Garrett, 1999 Sampling to analysis: 119-163 days
				Hay	0 1	0 0.1 7 14 28	< 0.50, 0.55 65, 78 (71) 22, 22 (22) 12, 12 (12) 3.4, 4.0 (3.7)	

[a] Average in parenthesis

FATE OF RESIDUES IN STORAGE AND PROCESSING

In Processing

The Meeting had received information on the fate of imazapyr residues during the processing of soya bean seeds in 2013. Processing factors were calculated for imazapyr residues in soya bean seeds.

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors*	PF (Mean or best estimate)
Soya bean seeds	Crude oil	< 0.005, < 0.006, < 0.008, < 0.01, < 0.06, < 0.07	< 0.009
	Meal	0.91, 1.0, 1.2, 1,2, 1.3, 1.3, 1.5, 1.8	1.3
	Aspirated grain fractions	0.04	0.04
	Hulls	0.54, 0.79	0.67

APPRAISAL

Imazapyr is a broad-spectrum herbicide in the imidazolinone family. It was evaluated in the 2013 JMPR for the first time for toxicology and for residues. The 2013 JMPR allocated an ADI of 0–3 mg/kg bw; an ARfD was unnecessary. It also determined that the definition of the residue was imazapyr for plant and animal commodities (for compliance with MRLs and for estimation of dietary intake). It recommended maximum residue levels for various commodities.

The 2013 JMPR received and considered the plant metabolism study and supervised residue trials on imidazolinone-tolerant soya beans; analytical methods, storage stability studies and processing studies on soya beans. However, at the time of the 2013 JMPR, no GAP had been approved for soya beans, transgenic or not. Due to the lack of an approved GAP, it was not possible for the Meeting to estimate maximum residue level for soya beans.

Imazapyr was included on the priority list by the CCPR at its Forty-sixth Session in 2014 for evaluation for additional MRLs by this Meeting. The current Meeting received information on analytical methods, use pattern and supervised residue trials to support estimation of maximum residue levels for soya beans and grasses.

Methods of analysis

The Meeting received information on the analytical method used for the determination of imazapyr residues in grass forage and hay. Samples were fortified with imazapyr at 0.5–50 mg/kg and analysed by capillary electrophoresis or LC-MS. The analytical method was validated; the LOQ was 0.5 mg/kg.

The freezer storage stability studies were reported on maize (grain, forage and fodder) and soya bean (seeds and processed fractions) samples in 2013. Storage stability results indicated that imazapyr residues were stable for at least 10 months in soya bean seed, at least 3 months in soya bean processd fractions (laminated soya bean, meal and oil) and at least 27 months in maize (grain, forage and fodder).

Residues resulting from supervised residue trials on crops

The 2013 Meeting received supervised trial data for the foliar application of imazapyr on soya bean (imidazolinone-tolerant) from Brazil and the current Meeting received supervised trial data on grasses from the USA.

Labels were available from Brazil and the USA describing the registered uses of imazapyr.

Soya bean (dry)

Supervised trials were conducted on imidazolinone-tolerant soya bean in Brazil.

The GAP on imidazolinone-tolerant soya bean of Brazil is a foliar application at a maximum rate of 0.053 kg ai/ha with a PHI of 60 days.

Imazapyr residues in soya bean seeds from independent trials in Brazil matching GAP were (n=12): < 0.05, 0.07, 0.11, 0.35, 0.48, 0.55, 0.83, 1.0, 1.3 (3) and 3.0 mg/kg.

Based on the residues for soya bean from trials in Brazil, the Meeting estimated a maximum residue level and an STMR value for imazapyr in soya bean seeds of 5 and 0.69 mg/kg respectively.

Animal feedstuffs

Straw, fodder and forage of grasses

Data were available from supervised trials on grasses in the USA.

The GAP on grasses in the USA is a spot application at a maximum rate of 0.84 kg ai per treated hectare with a PHI of 7 days for hay and no PHI for forage. The spot applications must not exceed more than 1/10 of the area to be grazed or cut for hay.

The trials were conducted with the broadcast foliar application to the whole trial area but the application does not correspond to the GAP. Therefore, the Meeting decided to use a factor of 0.1 to account for the difference between the application in the trials and that in the GAP for the estimation of a maximum residue level.

Calculated residues of imazapyr in forage of grasses were: 2.8, 3.3, 3.6, 3.7, 3.8, 4.4, 5.0, 5.4, 6.1, 6.5, 6.6, 6.8, 7.5 and 9.7 mg/kg.

Based on the calculated residues for forage grasses, the Meeting estimated a median residue value and a highest residue value for imazapyr in forage of grasses of 5.2 and 9.7 mg/kg, respectively on an “as received” basis.

Calculated residues of imazapyr in hay of grasses were: 0.15, 1.0, 1.1, 1.2 (3), 1.3 (2), 1.8, 1.9, 2.0, 2.2, 2.4 and 2.5 mg/kg.

Based on the calculated residues in hay grasses, the Meeting estimated a median residue value of 1.3 mg/kg, a highest residue value of 2.5 mg/kg on an as received basis and after correction for an average 88% dry matter content, estimated a maximum residue level of 6 mg/kg for imazapyr in hay of grasses.

Fate of residues during processing

Residues in processed commodities

The fate of imazapyr residues has been examined in soya bean seeds in processing studies. Estimated processing factors and the derived STMR-Ps are summarized in the Table below.

Processing factors, STMR-P for food and feed

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors*	PF (Mean or best estimate)	RAC STMR (mg/kg)	STMR-P (mg/kg)
Soya bean seeds	Crude oil	< 0.005, < 0.006, < 0.008, < 0.01, < 0.06, < 0.07	< 0.009	0.69	0
	Meal	0.91, 1.0, 1.2, 1,2, 1.3, 1.3, 1.5, 1.8	1.3		0.897
	Aspirated grain fractions	0.04	0.04		0.0276
	Hulls	0.54, 0.79	0.67		0.462

* Each value represents a separate study. The factor is the ratio of the residue in processed commodity divided by the residue in the RAC.

Residue in animal commodities

Farm animal dietary burden

The Meeting estimated the dietary burden of imazapyr in farm animals on the basis of the diets listed in Appendix IX of the FAO Manual 2009. Calculations derived from highest residue, STMR (some bulk commodities) and STMR-P values provide estimations of levels in feed suitable for estimating MRLs, while calculations from STMR and STMR-P values for feed is suitable for estimating STMR values for animal commodities. The percentage dry matter is taken as 100% when the highest residue levels and STMRs are already expressed on a dry weight basis.

Estimated maximum and mean dietary burdens of farm animals

Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are provided in Appendix IX of the FAO manual. The calculations were made according to the animal diets from US-Canada, EU, Australia and Japan in the Table (Appendix IX of the FAO manual).

Since the GAP for grasses is only registered in the USA, median residue value and highest residue value in forage of grasses are used only for the calculation of dietary burden in US-Canada.

Livestock dietary burden, imazapyr, ppm of dry matter diet								
	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0.61	0.40	1.7	1.0	2.8	1.5	1.7	1.2
Dairy cattle	18a	9.6bc	2.0	1.2	2.0	1.2	2.3	1.3
Poultry – broiler	0.43	0.43	0.57	0.57e	0.37	0.37	0.38	0.38
Poultry – layer	0.43	0.43	0.68d	0.54	0.37	0.37	0.33	0.33

[a] Highest maximum cattle dietary burden suitable for MRL estimates for mammalian meat, fat, edible offal and milk

[b] Highest mean 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

[d] Highest maximum poultry dietary burden suitable for MRL estimates for poultry meat, fat, edible offal and eggs

[e] Highest mean poultry dietary burden suitable for STMR estimates for poultry meat, fat, edible offal and eggs

Farm animal feeding studies

The 2013 JMPR received a lactating dairy cow feeding studies using imazapyr, which provided information on likely residues resulting in animal commodities and milk from imazapyr residues in the animal diet.

A poultry feeding study was not submitted as the expected residues of imazapyr in poultry feed were low. A poultry metabolism study at a dose rate of 9.7 ppm imazapyr in feed demonstrated that there was very low transfer to eggs and tissues with all residues of imazapyr less than 0.01 mg/kg.

Animal commodities maximum residue levels

For MRL estimations, the residue in the animal commodities is imazapyr.

Residues in tissues and milk at the expected dietary burden for dairy cattle are shown in the Table below. The mean estimated residue in milk was calculated using the residue values of day 3 to the final day.

	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	58	0.013	58	< 0.05	< 0.05	0.36	< 0.05
Dietary burden and residue estimate	18	0.004	18	< 0.05	< 0.05	0.11	< 0.05
STMR beef or dairy cattle							
Feeding study	58	0.010	58	< 0.05	< 0.05	0.25	< 0.05
Dietary burden and residue estimate	9.6	0.001	9.6	< 0.05	< 0.05	0.041	< 0.05

For beef and diary cattle, the calculated maximum dietary burden is 18 ppm dry weight of feed.

Based on the highest estimated residue in milk (0.004 mg/kg), the Meeting estimated a maximum residue level of 0.01 (*) mg/kg in milk. The Meeting confirmed the previous recommendation for milks.

Based on the highest estimated residue in kidney (0.11 mg/kg), the Meeting estimated a maximum residue level of 0.2 mg/kg in mammalian edible offal to replace the previous recommendation for mammalian edible offal of 0.05 (*) mg/kg.

Based on the mean estimated residues in kidney, the Meeting estimated an STMR value of 0.041 mg/kg in edible offal.

In the lactating dairy cow feeding study, imazapyr residues in meat and fat were less than the LOQ (0.05 mg/kg) at the dose level of 58 and 157 ppm. The mean cattle dietary burden of 9.6 ppm is still lower than the both dose level.

The Meeting confirmed the previous recommendations for mammalian meat and fat.

The maximum dietary burden for broiler and layer poultry is 0.68 ppm and is lower than the dose level in the laying hen metabolism study of 9.7 ppm. In the metabolism study, in which imazapyr equivalent to 9.7 ppm in the diet was dosed to laying hens for 7 consecutive days, no residues of imazapyr exceed 0.01 mg/kg were detected in tissues and eggs.

The Meeting confirmed the previous recommendations for poultry meat, fat, edible offal and eggs.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed in Annex 1 are suitable for estimating maximum residue limits and for IEDI and IESTI assessment.

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

The residue is not fat soluble.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
MO 0105	Edible offal (mammalian)	0.2	0.05*	0.041	
AS 0162	Hay or fodder (dry) of grasses	6		1.3	2.5
VD 0541	Soya bean (dry)	5		0.69	
OC 0541	Soya bean oil, crude		0		
	Forage of grasses			5.2	9.7
	Soya bean asp gr fn [a]			0.028	
AB 0541	Soya bean hulls			0.46	
AB 1265	Soya bean meal			0.9	

[a] aspirated grain fractions

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of imazapyr were calculated for the 17 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the 2013 JMPR and the current Meeting (Annex 3 to the 2015 Report). The ADI is 0–3 mg/kg bw and the calculated IEDIs were 0% of the maximum ADI (3 mg/kg bw). The Meeting concluded that the long-term intakes of residues of imazapyr, resulting from the uses considered by current JMPR, are unlikely to present a public health concern.

Short-term intake

The 2013 JMPR decided that an ARfD is unnecessary. The Meeting therefore concluded that the short-term intake of residues of imazapyr is unlikely to present a public health concern.

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IZ-731-030	Garrett A.D.	1999	CL 243997 (Imazapyr): Residues of CL 243997 in established bluegrass after postemergence treatment with Arsenal 2AS herbicide in Nebraska American Cyanamid Co., Princeton NJ, United States of America IZ-731-030, GLP, Unpublished
IZ-731-031	Garrett A.D.	1999	CL 243997 (Imazapyr): Residues of CL 243997 in established tall fescue grass after postemergence treatment with Arsenal 2AS herbicide in Oregon American Cyanamid Co., Princeton NJ, United States of America IZ-731-031, GLP, Unpublished
IZ-731-032	Garrett A.D.	1999	CL 243997 (Imazapyr): Residues of CL 243997 in established common bermuda grass after postemergence treatment with Arsenal 2AS herbicide in Texas American Cyanamid Co., Princeton NJ, United States of America IZ-731-032, GLP, Unpublished
IZ-731-033	Garrett A.D.	1999	CL 243997 (Imazapyr): Residues of CL 243997 in established bluegrass after postemergence treatment with Arsenal 2AS herbicide in Indiana American Cyanamid Co., Princeton NJ, United States of America IZ-731-033, GLP, Unpublished
IZ-731-034	Garrett A.D.	1999	CL 243997 (Imazapyr): Residues of CL 243997 in established brome grass after postemergence treatment with Arsenal 2AS herbicide in Colorado American Cyanamid Co., Princeton NJ, United States of America IZ-731-034, GLP, Unpublished

IMIDACLOPRID (206)

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EXPLANATION

Imidacloprid is a systemic insecticide which has been used widely in many crops for years. It was first evaluated by JMPR in 2001 (T) and 2002 (R). An ADI of 0-0.06 mg/kg bw and an ARfD of 0.4 mg/kg bw was established. The compound was evaluated for residues in 2006, 2008 and 2012. In 2002 the Meeting agreed that the residue definition for compliance with MRLs and for estimation of dietary intake for plant and animal commodities should be the sum of imidacloprid and its metabolites containing the 6-chloropyridinyl moiety, expressed as imidacloprid. It was listed by the 46th Session of CCPR (2014) for the evaluation of 2015 JMPR for additional MRLs.

The residue studies were submitted by the manufacturer and member countries for additional MRLs for stone fruit, olive, curly kale, soya bean, tea, goji (China) and basil (Thailand).

RESIDUE ANALYSIS

Analytical methods

Samples of cherries, plum and peach were fortified with an equimolar solution of imidacloprid, desnitro imidacloprid (WAK4140, M09), olefin imidacloprid (WAK3745, M06), 5-hydroxyl imidacloprid (WAK4103, M01) and 6-chloronicotinic acid (6-CNA, M14), and were analysed for combined residues of those compounds by GC-MS using a modification of the Bayer Method 00200-reformated, Report No 102624-R1 dated 02/23/94 (see JMPR 2002, 2006 and 2012). At the LOQ of 0.05 mg/kg (expressed as imidacloprid), the recoveries were 98±12% for cherries, 92, 104, 115% for plum and 93±18% for peach.

The Meeting received information on the analytical method (Method 00834) for the determination of imidacloprid residues as well as the total residue of imidacloprid (including parent and all metabolites containing the 6-chloropyridinyl moiety) in plant materials (Schöning, 2003: MR-122/03).

Imidacloprid and related metabolites are extracted with a mixture of methanol/water (3/1, v/v) in the presence of diluted sulphuric acid (10%). Oil samples are dissolved in n-hexane and the residues are extracted twice with water. For the determination of the imidacloprid, an aliquot of the extract is partitioned against cyclohexane/ethyl acetate (1/1, v/v) using a Chromabond XTR column (diatomaceous earth). The organic solution is redissolved in acetonitrile/water (2/8, v/v + 2 mL/L formic acid). Quantitation is performed by reversed phase HPLC-MS/MS. For determination of the total residue of imidacloprid, a corresponding aliquot of the extract is evaporated to the aqueous remainder and dissolved in water. Imidacloprid and all metabolites containing the 6-chloropicolyl moiety are oxidised with alkaline $KMnO_4$ to yield 6-CNA. Following acidification and subsequent neutralisation of the excess oxidant, the 6-CNA is extracted from the aqueous solution using *tert*-butylmethylether (MTBE). The ether phase is dried, the solvent is evaporated and the remainder dissolved in acetonitrile/water (2/8, v/v + 2 mL/L formic acid). These solutions are subjected to analysis by HPLC-MS/MS.

The recoveries for imidacloprid ranged from 83 to 112% at fortification levels of 0.01 and 1.0 mg/kg. The mean recoveries for the parent compound were between 89 and 110% with relative standard deviations (RSD) up to 11.6%. The recoveries for the total residue of imidacloprid fortified as parent compound ranged from 75 to 102% at fortification levels of 0.05 to 2.0 mg/kg. The mean recoveries for the parent compound were between 77 and 93% with RSD values up to 6.1%. The recoveries for the total residue of imidacloprid fortified as a mixture of 6-CNA and desnitro-imidacloprid (1:1, w/w) ranged from 64 to 108% at fortification levels of 0.0567 to 1.134 mg/kg parent equivalents. The mean recoveries for the metabolite mixture were between 73 and 97% with relative standard deviations up to 16%. The LOQ is 0.01 mg/kg for imidacloprid and 0.05 mg/kg for total residue of imidacloprid, expressed as imidacloprid.

The method as modified in 00834/M001 (Schöning, 2004: MR-153/03) contains no changes in the analytical procedure compared to the original method 00834 but it incorporates an internal standard procedure to the method. Method 00834/M001 was validated for the determination of residues of imidacloprid parent compound as well as the total residue of imidacloprid (including parent and all metabolites containing the 6-chloropyridinyl moiety) in plant materials. The recoveries for imidacloprid ranged from 80 to 104% at fortification levels of 0.01 and 2.0 mg/kg (mean recoveries: 88 to 99%, RSDs: 1.5 to 7.4%). The recoveries for the total residue of imidacloprid ranged from 66 to 106% at fortification levels of 0.05 (0.0567 mg/kg as mixture of 6-CNA and desnitro metabolite (1:1, w/w) calculated as imidacloprid) to 2.0 mg/kg (mean recoveries: 75 to 101%, RSDs: 1.1 to 9.8%).

The analytical method 00834/M002 (Schöning, 2010: MR-09/169) was developed for the determination of residues of imidacloprid, 5-hydroxyl imidacloprid (WAK4103, M01) and olefin imidacloprid (WAK3745, M06) in plant materials. Imidacloprid and its metabolites are extracted from tomato (fruit), bean (bean with pod), orange (fruit), rape (seed), cereals (grain) and tobacco (green leaf and dried leaf) with methanol/water (3/1, v/v) using a blender. After filtration an aliquot of the extract was evaporated to the aqueous remainder and further the stable isotopically labelled analytes are added for tomato (fruit), bean (bean with pod), orange (fruit), rape (seed), cereals (grain) and tobacco (green leaf). Parts of the solutions are transferred into an HPLC vial and subjected to reversed phase HPLC-MS/MS in the positive ion mode without further clean-up. Recoveries were determined at fortification levels of 0.01 mg/kg (LOQ level, 0.05 mg/kg for tobacco), and 0.10 mg/kg (0.5 mg/kg for tobacco) (each compound expressed as parent equivalent). Mean recoveries for each fortification level ranged from 70 to 107% with RSD up to 12% for all matrices.

The supplemental method 00300/E007 (Schöning, 2010: MR-158/00) has no changes in the analytical procedure compared to the original method 00300. The method was validated for additional matrices of olive fruit, grape pomace and cacao bean. For imidacloprid, recoveries were determined by spiking control samples with imidacloprid at fortification levels of 0.01 and 0.20 mg/kg. The recoveries were in the range from 68 to 110%, the mean recoveries for each matrix ranged from 74 to 90% with a mean RSD ranging from 3.3 to 17.3%. For the total residue of imidacloprid, recoveries were determined by spiking control samples with imidacloprid (fortification levels of 0.05 and 0.5 mg/kg) or with a mixture of 6-CNA and desnitro imidacloprid (0.02 mg/kg each corresponding to 0.0567 mg/kg calculated as imidacloprid). The recoveries were in the range from 64 to 98%, the mean recoveries for each matrix ranged from 70 to 96% with a mean RSD ranging from 2.2 to 12%.

The results for olive are summarized in Tables 1 and 2.

Table 1 Recovery results obtained for the determination of imidacloprid from olive and its processed commodities

Commodity	Fortification level (mg/kg)	N	Recovery range (%)	Mean recovery (%)	% RSD	Reference Method
Olive, fruit	0.01 1.0	5 5	83 – 96 106 – 111	92 108	5.6 1.7	MR-122/03 00834 (m/z 258→175)
Olive, oil	0.01 1.0	5 5	97 – 98 96 – 100	98 99	0.5 1.8	
Olive, pomace	0.01 1.0	5 5	94 – 100 109 – 112	97 110	2.5 1.1	
Olive, fruit	0.01 2.0	5 5	80 – 96 96 – 99	88 97	7.4 1.5	MR-153/03 00834/M001
Olive, fruit	0.01 0.20	3 3	78 – 104 87 – 93	87 90	17 3.3	MR-158/00 00300/E007

Table 2 Recovery results obtained for the determination of total residue of imidacloprid from olive and its processed commodities

Commodity	Fortification level (mg/kg)	N	Recovery range (%)	Mean recovery (%)	% RSD	Reference Method
Olive, fruit	0.05	5	80 – 86	83	2.9	MR-122/03
	2.0	5	78 – 80	79	1.1	00834
	0.0567*	5	75 – 91	82	7.2	(m/z 158→122
	1.134*	5	70 – 79	74	5.8	
Olive, oil	0.05	5	83 – 85	84	1.2	
	2.0	5	78 – 92	84	6.1	
	0.0567*	5	77 – 83	80	3.4	
	1.134*	5	79 – 84	82	2.5	
Olive, pomace	0.05	5	88 – 95	91	3.3	
	2.0	5	77 – 82	80	2.6	
	0.0567*	5	85 – 95	92	4.6	
	1.134*	5	86 – 94	90	3.4	
Olive, fruit	0.05	5	81 – 89	85	3.9	MR-153/03
	2.0	5	76 – 95	88	9.8	00834/M001
	0.0567*	5	80 – 97	91	7.3	
Olive, fruit	0.05	3	64 – 79	73	11	MR-158/00
	0.50	3	73 – 82	78	5.9	00300/E007
	0.0567*	3	68 – 72	70	2.9	

* Mixture of 6-CNA (0.02 mg/kg) and desnitro metabolite (0.02 mg/kg), (1/1, w/w) calculated as imidacloprid

The Meeting has received information on the analytical method (NY/T 1275-2007) for the detection, quantitative analysis and confirmation of imidacloprid residues in fresh and dried goji berries (Niu, 2014: IG-01).

Imidacloprid is extracted from goji samples by homogenizing with acetonitrile. After adding sodium chloride, the sample is shaked and centrifuged. An aliquot is concentrated, and purified by solid phase extraction using amino cartridges. Imidacloprid residues were analysed by reversed-phase HPLC-UV (275 nm). The method was validated in fresh or dried goji samples. Control samples were spiked with a standard solution of imidacloprid at fortified level of 0.02, 0.05 and 0.1 mg/kg, and recoveries of imidacloprid with this method ranged from 69–87% (mean: 72–84% with RSD of 2.6–3.5%) in fresh goji samples, while recoveries ranged from 76–100% (mean: 79–100% with RSD of 0–11%) in dried goji samples. The LOQ is 0.02 mg/kg for both matrices.

Table 3 Recovery results obtained for the determination of imidacloprid residue from goji berries

Commodity	Fortification level (mg/kg)	N	Recovery range (%)	Mean recovery (%)	% RSD	Reference Method
Goji, fresh	0.02	5	70 – 76	72	2.6	IG-01
	0.05	5	69 – 78	73	4.1	Yan Niu, 2014
	0.10	5	78 – 87	84	3.5	
Goji, dried	0.02	5	100	100	0.0	
	0.05	5	70 – 100	85	11	
	0.10	5	76 – 82	79	3.0	

The Meeting has also received information on the analytical method (NT-001-P04-01) used for the determination of residues of imidacloprid in soya bean matrices (seed, forage, hay, meal, hull, refined oil, defatted flour and aspirated grain fractions) (Gould *et al*., 2005: 201591).

This analytical method is based on earlier methods 00200 and 00834 and is designed to make use of the equipment and techniques available at the analytical laboratory. The total residue of imidacloprid is analysed by a common moiety method and quantified by using isotopically-labelled internal standards and HPLC-MS/MS. The method is validated by measuring the concurrent recoveries of each analyte (imidacloprid, desnitro imidacloprid, 5-hydroxy imidacloprid, olefin imidacloprid, and 6-CNA) individually in separate control samples of soya bean seed, forage, and

hay, as well as processed commodities of meal, hull, refined oil, defatted flour and aspirated grain fractions. Additionally, the method is further validated by measuring the concurrent recoveries of an imidacloprid/desnitro mixture (1:1, w/w) in these same matrices at various fortification levels. The validation was performed concurrently during the studies RANTY002 and RANTY003-1 (see Table 4).

Table 4 Recovery results obtained for the determination of imidacloprid from soya bean matrices

Analyte	Fortification level (mg/kg)	N	Recovery range (%)	Mean recovery (%)	% RSD	Reference
Seed						RANTY002 Mackie, 2006
Imaidacloprid	0.050	1	72			
Desnitro imidacloprid	0.050	1	59			
5-hydroxy imidacloprid	0.050	1	67			
Olefin imidacloprid	0.050	1	58			
6-CNA	0.050	1	79			
Imidacloprid/desnitro imidacloprid mixture (1:1)*	0.050 0.10 2.0	5 5 3	75 – 93 67 – 99 69 – 79	85 82 74	9.3 15 6.8	
Forage						
Imaidacloprid	0.025	2	69, 92	81		
Desnitro imidacloprid	0.025	2	72, 75	74		
5-hydroxy imidacloprid	0.025	2	83, 87	85		
Olefin imidacloprid	0.025	2	83, 95	89		
6-CNA	0.025	2	66, 81	74		
Imidacloprid/desnitro imidacloprid mixture (1:1)*	0.025 2.0 7.5 10	4 10 3 3	60 – 88 74 – 90 81 – 86 74 – 78	75 81 84 77	16 7.2 3.0 3.0	
Hay						
Imaidacloprid	0.010	2	80, 85	83		
Desnitro imidacloprid	0.010	2	89, 92	91		
5-hydroxy imidacloprid	0.010	2	77, 99	88		
Olefin imidacloprid	0.010	2	74, 85	79		
6-CNA	0.010	2	78, 94	86		
Imidacloprid/desnitro imidacloprid mixture (1:1)*	0.010 2.0 30	4 8 3	57 – 85 72 – 95 87 – 90	75 80 88	17 9.5 1.7	
Seed						RANTY003 Krolski, 2006
Imidacloprid/desnitro imidacloprid mixture (1:1)*	0.20 2.0	3 3	73 – 87 79 – 88	81 84	8.9 5.4	
Meal						
Imaidacloprid	0.20	1	72			
Desnitro imidacloprid	0.20	1	76			
5-hydroxy imidacloprid	0.20	1	79			
Olefin imidacloprid	0.20	1	79			
6-CNA	0.20	1	84			
Imidacloprid/desnitro imidacloprid mixture (1:1)*	0.20 2.0	4 6	64 – 74 74 – 84	71 80	6.7 4.6	
Hull						

Analyte	Fortification level (mg/kg)	N	Recovery range (%)	Mean recovery (%)	% RSD	Reference
Imaidacloprid	0.20	1	91			
Desnitro imidacloprid	0.20	1	92			
5-hydroxy imidacloprid	0.20	1	92			
Olefin imidacloprid	0.20	1	94			
6-CNA	0.20	1	78			
Imidacloprid/desnitro imidacloprid mixture (1:1)*	0.20 2.0	4 6	69 – 78 70 – 84	75 77	5.7 6.5	
Oil						
Imaidacloprid	0.20	1	87			
Desnitro imidacloprid	0.20	1	72			
5-hydroxy imidacloprid	0.20	1	84			
Olefin imidacloprid	0.20	1	82			
6-CNA	0.20	1	92			
Imidacloprid/desnitro imidacloprid mixture (1:1)*	0.10 2.0	3 2	82 – 93 71, 73	87 72	6.6	
Flour						
Imaidacloprid	0.20	1	86			
Desnitro imidacloprid	0.20	1	88			
5-hydroxy imidacloprid	0.20	1	87			
Olefin imidacloprid	0.20	1	84			
6-CNA	0.20	1	79			
Imidacloprid/desnitro imidacloprid mixture (1:1)*	0.20 2.0	4 6	64 – 76 78 – 84	71 81	8.1 2.7	
AGF						
Imidacloprid/desnitro imidacloprid mixture (1:1)*	30 150	2 2	72, 79 60, 74	75 67		

* The fortification level given is the total mg/kg of both analytes in the mixture.

The analytical method 01389 was developed for the determination of residues of imidacloprid, its 2 metabolites 5-hydroxy imidacloprid and olefin imidacloprid, and of the total residue of imidacloprid determined as 6-CNA in/on plant materials (Richter, 2014: P 3009 G). Imidacloprid and its metabolites are extracted from whole orange fruit, tomato fruit, wheat grain, dry beans, olive fruit, tea (green tea and black tea), hop cones (green and dried), tobacco (green leaves and fermented tobacco), coffee (green beans and roasted coffee), and cocoa (green beans and roasted beans) with methanol/water (3/1, v/v). For the individual analytes, an aliquot of the extract is cleaned-up with liquid/liquid SPE. For the common moiety analysis, an aliquot of the extract is made by alkaline oxidation under reflux and liquid/liquid partition. Final extracts of both branches are subjected to reversed phase HPLC-MS/MS.

The LOQ (expressed as imidacloprid equivalents) for each analyte is 0.01 mg/kg. For dried, fermented and roasted difficult matrices (dried hop cones, fermented tobacco leaves, roasted cocoa beans, roasted coffee beans, black tea) the LOQ increased to 0.05 mg/kg, because validation attempts for dried hop cones and roasted coffee beans at 0.01 mg/kg failed. For the total residue of imidacloprid, the LOQ is 0.05 mg/kg for all matrices.

Table 5 Recovery results obtained for the determination of imidacloprid from tea (green tea and black tea)

Analyte	Fortification level (mg/kg)	N	Recovery range (%)	Mean recovery (%)	% RSD
Green tea					
Imidacloprid	0.01 0.10	5 5	84 – 117 82 – 109	100 100	16 12
5-hydroxy imidacloprid	0.01 0.10	5 5	63 – 99 67 – 86	79 76	20 9.3
Olefin imidacloprid	0.01 0.10	5 5	70 – 112 77 – 101	91 90	19 12
6-chloronicotinic acid	0.05 0.50	5 5	70 – 82 79 – 101	76 87	5.7 10
Black tea					
Imidacloprid	0.05 0.50	5 5	80 – 86 79 – 96	83 86	3.3 7.9
5-hydroxy imidacloprid	0.05 0.50	5 5	90 – 95 89 – 97	93 92	2.5 3.1
Olefin imidacloprid	0.05 0.50	5 5	78 – 95 84 – 93	89 88	9.2 3.7
6-chloronicotinic acid	0.05 0.50	5 5	74 – 95 69 – 84	82 75	10 8.1

Stability of pesticide residues in stored analytical samples

The storage stability of imidacloprid and various important metabolites was tested in various plant and animal materials. Tests on animal samples were carried out to assess the stability of the total residue. For plants, tests were carried out to assess the stability of the total residue and on plants to assess the stability of residues of the active substance and of the total residue. The results indicate that imidacloprid and the tested metabolites are stable for a minimum of approximately 2 years in plants and for at least 1 year in animal commodities (see JMPR 2002, 2006, 2008 and 2012).

The Meeting has received data on the storage stability of imidacloprid, 5-hydroxy imidacloprid and olefin imidacloprid in various plant matrices for a period of 36 months (Schoening and Diehl, 2014: MR-09/182, P642094733). Samples of wheat (grain), orange (fruit), tomato (fruit), bean (seed) and rape seed were fortified with imidacloprid and its metabolites 5-hydroxy imidacloprid and olefin imidacloprid at a level of 0.1 mg/kg. The samples stored at an average temperature of -18°C or below were analysed at the nominal storage interval of 0, 30, 90, 180, 360, 540, 720, 900 and 1080 days.

At each storage interval imidacloprid and its metabolites 5-hydroxy and olefin were determined in the stored control samples and in the stored spiked samples according to the analytical method 00834/M002. Procedual recovery experiments at fortification levels of 0.10 mg/kg (0.01 mg/kg for 0 day storage interval) were also performed for each analyte at each storage interval. For all matrices the LOQ was 0.01 mg/kg for imidacloprid and its metabolite 5-hydroxy and olefin expressed as imidacloprid equivalent.

Table 6 Recovery of imidacloprid from stored fortified samples of plant matrices

Storage interval (days)	Recovery (%) [0.10 mg/kg fortification]		
	Procedual	% remaining	Mean
Wheat, grain			
0	91, 94	90, 91, 94, 95, 101	94
38	83, 90	85, 87, 96	89
90	88, 92	88, 92, 93	91

Storage interval (days)	Recovery (%) [0.10 mg/kg fortification]		
	Procedual	% remaining	Mean
180	63, 80	81, 85, 102	89
361	93, 94	96, 98, 100	98
542	90, 95	99, 104, 105	103
719	77, 89	86, 87, 94	89
908	107, 110	91, 104, 129	108
1082	99, 106	94, 110, 110	105
Orange, fruit			
0	87	82, 82, 90, 92, 93	88
35	92, 94	75, 92, 96	88
91	84, 89	95, 100, 101	99
182	106, 113	93, 100, 112	102
366*/360	97, 101	106, 112, 117	112
540	96, 106	105, 110, 114	110
721	83, 88	82, 87, 93	87
912	107, 109	90, 115, 117	107
1080	97	106, 107, 107	107
Tomato, fruit			
0	90, 95	98, 98, 101, 102, 113	102
35	95, 100	88, 100, 100	96
90	93, 101	105, 107, 112	108
181	95, 99	106, 112, 113	110
360	94, 100	98, 105, 109	104
540	102, 105	109, 112, 116	112
720	86, 92	74, 79, 85	79
903	105, 112	108, 112, 120	113
1078	100, 108	110, 116, 124	117
Bean, seed			
0	74, 75	89, 91, 94, 95, 96	93
34	90, 102	85, 87, 88	87
90	85, 93	81, 82, 84	82
180	95, 96	101, 105, 111	106
359	87, 92	94, 97, 97	96
540	102, 109	94, 103, 106	101
720	90, 93	87, 95, 95	92
910	92, 95	89, 100, 106	98
1077	92, 93	97, 98, 104	100
Rape, seed			
0	79, 83	73, 90, 91, 91, 93	88
33	77, 82	73, 74, 80	76
90	84, 85	70, 75, 87	77
180	104, 107	103, 104, 111	106
361	99, 100	86, 87, 89	87
540	82, 85	59, 64, 66	64
719	85, 90	76, 89, 95	87
901	84, 85	78, 90, 95	88
1076	89, 100	82, 85, 92	86

* for procedual recoveries

Table 7 Recovery of 5-hydroxy imidacloprid from stored fortified samples of plant matrices

Storage interval (days)	Recovery (%) [0.10 mg/kg fortification]		
	Procedual	% remaining	Mean
Wheat, grain			
0	70, 73	77, 95, 96, 96, 102	93
38	87, 98	96, 100, 102	99
90	90, 91	70, 80, 86	79
180	90, 92	73, 83, 85	80
361	97, 98	100, 105, 105	103
542	89, 96	98, 101, 102	100

Storage interval (days)	Recovery (%) [0.10 mg/kg fortification]		
	Procedual	% remaining	Mean
719	83, 96	94, 97, 103	98
908	103, 104	101, 102, 105	103
1082	100, 103	102, 105, 108	105
Orange, fruit			
0	79	99, 101, 103, 109, 112	105
35	98, 104	103, 105, 109	106
91	89, 98	98, 99, 99	99
182	109, 114	73, 76, 89	79
366*/360	93, 100	95, 101, 101	99
540	95, 106	95, 99, 103	99
721	100, 109	79, 103, 106	96
912	97, 99	96, 103, 111	103
1080	101	76, 92, 103	90
Tomato, fruit			
0	93, 109	98, 98, 99, 102, 104	100
35	105, 106	94, 99, 105	99
90	99, 102	95, 99, 104	99
181	106, 112	105, 106, 109	107
360	97, 105	91, 93, 94	93
540	107, 108	108, 115, 120	114
720	93, 94	95, 98, 99	97
903	91, 95	95, 101, 102	99
1078	84, 91	89, 94, 101	95
Bean, seed			
0	73, 73	72, 75, 80, 82, 86	79
34	99, 100	83, 84, 85	84
90	78, 94	83, 84, 84	84
180	101, 103	83, 97, 99	93
359	79, 84	93, 95, 97	95
540	115, 117	68, 83, 97	83
720	92, 93	91, 94, 95	93
910	89, 91	90, 93, 103	95
1077	107, 111	104, 108, 111	108
Rape, seed			
0	86, 86	86, 88, 89, 91, 96	90
33	89, 94	78, 85, 92	85
90	90, 93	76, 78, 78	77
180	105, 105	96, 99, 103	99
361	94, 98	84, 87, 92	88
540	103, 111	77, 86, 93	85
719	91, 91	88, 101, 103	97
901	91, 98	77, 81, 85	81
1076	89, 95	95, 96, 99	97

* for procedual recoveries

Table 8 Recovery of olefin imidacloprid from stored fortified samples of plant matrices

Storage interval (days)	Recovery (%) [0.10 mg/kg fortification]		
	Procedual	% remaining	Mean
Wheat, grain			
0	108, 114	79, 80, 82, 87, 90	84
38	77, 80	84, 85, 90	86
90	87, 91	86, 88, 96	90
180	90, 93	84, 85, 88	86
361	109, 110	92, 93, 95	93
542	90, 98	97, 105, 113	105
719	85, 90	93, 88, 93	88
908	103, 107	96, 99, 105	100
1082	98, 106	105, 109, 110	108

Storage interval (days)	Recovery (%) [0.10 mg/kg fortification]		
	Procedual	% remaining	Mean
Orange, fruit			
0	92	86, 87, 91, 92, 95	90
35	87, 94	87, 89, 97	91
91	80, 87	92, 95, 98	95
182	78, 89	60, 70, 75	68
366*/360	104, 107	88, 92, 92	91
540	93, 107	111, 112, 119	114
721	89, 97	89, 92, 99	93
912	86, 90	100, 102, 104	102
1080	102	67, 70, 105	81
Tomato, fruit			
0	85, 87	90, 101, 101, 102, 103	99
35	95, 97	90, 92, 96	93
90	85, 87	96, 96, 97	96
181	83, 86	85, 91, 95	90
360	105, 117	92, 96, 105	98
540	111, 113	93, 94, 98	95
720	94, 96	91, 92, 93	92
903	99, 104	108, 112, 120	113
1078	79, 84	74, 74, 95	81
Bean, seed			
0	69, 73	67, 70, 82, 83, 87	78
34	88, 96	70, 70, 71	70
90	80, 82	76, 77, 80	78
180	73, 76	71, 78, 81	77
359	81, 88	66, 80, 86	77
540	106, 108	99, 100, 104	101
720	87, 90	81, 87, 93	87
910	88, 89	79, 83, 101	88
1077	98, 100	101, 102, 111	105
Rape, seed			
0	84, 87	67, 71, 72, 72, 76	72
33	80, 86	79, 83, 91	84
90	94, 109	73, 76, 97	82
180	80, 83	65, 67, 72	68
361	97, 101	83, 85, 90	86
540	81, 86	74, 80, 94	83
719	81, 86	81, 84, 86	84
901	97, 103	83, 84, 85	84
1076	70, 81	70, 77, 79	75

* for procedual recoveries

Storage stability results indicated that residues of imidacloprid and its metabolites 5-hydroxy imidacloprid and olefin imidacloprid were stable for at least 36 months under freezer conditions at about -18°C or below in wheat (grain), orange (fruit), tomato (fruit), bean (seed) and rape seed.

The Meeting has also received data on the storage stability of imidacloprid, olefin imidacloprid and 6-CNA in basil for a period of 9 months. Samples were fortified with imidacloprid, olefin imidacloprid and 6-CNA at a level of 0.50 mg/kg. The samples stored at -20°C were analysed at the storage interval of 0, 3, 6 and 9 months.

Imidacloprid, olefin imidacloprid and 6-CNA were determined in the control samples and in the stored fortified samples according to the analytical method 01389.

Table 9 Recovery of imidacloprid and its metabolites from stored fortified samples of basil

Storage interval (months)	Recovery (%) [0.50 mg/kg fortification]		
	Procedural	% remaining	Mean
Imidacloprid			

Storage interval (months)	Recovery (%) [0.50 mg/kg fortification]		
	Procedural	% remaining	Mean
0	78	76, 80	78
3	80	74, 82	78
6	77	70, 91	81
9	89	72, 79	76
Olefin imidacloprid			
0	79	77, 81	79
3	95	93, 96	95
6	73	76, 82	79
9	96	79, 90	85
6-CNA			
0	83	79, 87	83
3	95	93, 95	94
6	88	79, 85	82
9	82	79, 84	81

USE PATTERN

The Meeting received labels from Italy, Japan, Spain and the USA. The authorized uses relevant to the supervised residue trials data submitted to the current Meeting are summarized in Table 10.

Table 10 Registered uses of imidacloprid relevant to the residue evaluation by the current Meeting

Crop	Country	Formulation		Application					PHI, days
		Type	Conc. of imidacloprid	Method	kg ai/ha	kg ai/hL	L/ha	No. max	
Stone fruits									
Stone fruits	USA	SC	550 g/L	Soil	0.28-0.43 (max 0.43/year)			1	21
				Pre-plant, root dip	14.3 mL/38 L root dip solution			1	-
Stone fruits (Apricot, Nectarine, Peach)				Foliar	0.056-0.11 (max 0.34/year)		468 (G) 234 (A)	3-6	0 (7 days interval)
Stone fruits (Cherries, Plums, Plumcot, Prune)					0.056-0.11 (max 0.56/year)		468 (G) 234 (A)	5-10	7 (10 days interval)
Assorted tropical and sub-tropical fruits – edible peel									
Olive	Italy	OD	200 g/L	Foliar		0.01-0.013		1	28
Olive	Spain	SL	200 g/L	Foliar		0.01		2	7 (30 days interval)
				Foliar (a)	0.01-0.02		50-100	4	7 (7-10 days interval)
Brassica vegetables									
Cabbages (including cauliflower, broccoli and other brassica cabbage, cabbage head, leafy brassica, kohlrabi)	Italy	OD	200 g/L	Foliar		0.01		1	14
Cabbage (cabbage head, leafy brassica)	Italy	OD	75 g/L	Foliar	0.075-0.094			1-2	7
Fruiting vegetables, other than Cucurbits – subgroup Tomatoes									

Crop	Country	Formulation		Application					PHI, days
		Type	Conc. of imidacloprid	Method	kg ai/ha	kg ai/hL	L/ha	No. max	
Goji berry	China	EC	50 g/L	Foliar		0.003-0.005		3	3
Pulses									
Soya bean	USA	FS	480 g/L	Seed treatment	63-125 g ai/100 kg seed			1	-
		SC	550 g/L	Foliar	0.053 (max 0.16/year)			3	21 (7 days interval)
Herbs									
Basil	Thailand	WG	700 g/kg	Foliar		0.021-0.042		(b)	7
Teas									
Tea	Japan	WG	500 g/kg	Foliar		0.005-0.01	2000-4000	1	7

(a) spray solution containing a hydrolysed protein mixture at 1-2%, coarse drop application to parts facing south. Use only one of the two authorized methods (spray or bait) during the growing season of one crop.

(b) apply when infested

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on imidacloprid supervised field trials for the following crops.

Group	Commodity	Table
Stone fruits	Cherries	Table 11
	Plum	Table 12
	Peach	Table 13
Assorted tropical and sub-tropical fruits–edible peel	Olive	Table 14–16
Leafy vegetables	Kale	Table 17
Fruiting vegetables, other than Cucurbits	Goji berry	Table 18
Pulses	Soya bean	Table 19
Herbs	Basil	Table 20
Teas	Tea	Table 21, 22
Legume animal feeds	Soya bean fodder and forage	Table 23

Imidacloprid formulations were applied by foliar treatment. Each of the field trial sites generally consisted of an untreated control plot and treated plots. Residues, 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.

Stone fruits

Cherries

Twelve residue trials for cherries were conducted in the USA (Dorschner, 2002: 111045). The 192 g/L SC formulation was applied five or six times as foliar spray at application rates 0.11-0.13 kg ai/ha. The total residue of imidacloprid was determined according to method 102624-R1 (based on the method 00200). The LOQ was 0.050 mg/kg.

Table 11 Imidacloprid residues on cherries from supervised trials in USA

Cherries country, year (variety)	Application					DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Treatment	no.			
GAP, USA	*SC*	*Max 0.56 kg ai/ha /year*				*7*		
USA, 1999 Bridgeton/NJ (Montmorency tart cherry) 99-NJ17	SC	0.11 0.11 0.11 0.11 0.11	1031 1040 1025 1022 1027	100% petal fall Fruiting Green fruit First red fruit Ripening fruit	5	6	2.4, 2.5 Mean 2.5	111045 IR-4 PR. 07202 Dorschner, 2002
USA, 1999 Fennville/MI (Montmorency tart cherry) 99-MI09[a]	SC	0.11 0.11 0.11 0.11 0.11	953 931 944 939 935	Immature fruit Immature fruit Immature fruit Immature fruit Immature fruit	5	0 3 7 14	1.2, 1.2 1.0, 1.1 1.0, 1.1 Mean 1.1 0.94, 1.0	Sampling to analysis: 189-250 days
USA, 1999 Fennville/MI (Montmorency tart cherry) 99-MI10[b]	SC	0.11 0.11 0.11 0.11 0.11	934 955 937 957 942	Immature fruit Immature fruit Immature fruit Immature fruit Immature fruit	5	7	1.2, 1.5 Mean 1.4	
USA, 1999 Fennville/MI (Montmorency tart cherry) 99-MI11[c]	SC	0.11 0.11 0.11 0.11 0.11	936 950 943 931 941	Immature fruit Immature fruit Immature fruit Immature fruit Immature fruit	5	7	0.88, 0.93 Mean 0.90	
USA, 1999 Traverse City/MI (Emperor Francis sweet cherry) 99-MI12[d]	SC	0.11 0.12 0.11 0.11 0.12	555 584 564 563 584	Pea-sized fruit 14-mm fruit 15-mm fruit 16-mm fruit 22-mm fruit	5	7	0.33, 0.34 Mean 0.34	
USA, 1999 Traverse City/MI (Hedelfingen sweet cherry) 99-MI13[e]	SC	0.12 0.11 0.11 0.12 0.12	579 562 564 578 579	- 14-mm fruit 17-mm fruit 22-mm fruit 24-25-mm fruit	5	7	0.39, 0.43 Mean 0.41	
USA, 1999 Grandview/WA (Bing sweet cherry) 99-WA19	SC	0.11 0.11 0.11 0.11 0.11	618 1149 1041 1094 1221	Bloom Fruiting Small fruit Fruiting Fruiting	5	8	0.24, 0.24 Mean 0.24	
USA, 1999 Buhl/ID (Bing sweet cherry) 99-ID07	SC	0.12 0.13 0.13 0.13 0.13 0.13	930 943 938 943 940 946	Late bloom Fruiting Fruiting Fruiting Fruiting Fruiting	6	7	0.55, 0.60 Mean 0.57	
USA, 1999 Caldwell/ID (Lambert sweet cherry) 99-ID08	SC	0.11 0.11 0.11 0.12 0.12 0.12	915 924 928 935 931 933	Bloom Part bloom Fruiting Fruiting Fruiting Maturing	6	7	0.62, 0.63 Mean 0.63	
USA, 1999 Hood River/OR (Bing sweet	SC	0.11 0.11 0.11	1890 1777 1833	Fruiting Fruiting A few turning pink	5	7	0.35, 0.36 Mean 0.36	

Cherries country, year (variety)	Application					DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Treatment	no.			
cherry) 99-OR02		0.11 0.11	1813 1828	Fruit ripening Red fruit				
USA, 1999 Stockton/CA (Bing sweet cherry) 99-CA115[f]	SC	0.11 0.11 0.11 0.11 0.11	946 936 944 932 933	99% petal fall Fruiting Fruiting Immature fruit Immature fruit	5	7	0.22, 0.28 Mean 0.25	
USA, 1999 Stockton/CA (Dawson sweet cherry) 99-CA116[g]	SC	0.11 0.11 0.11 0.12 0.11	939 926 931 949 935	99% petal fall Fruiting Fruiting Immature fruit Immature fruit	5	7	0.45, 0.62 Mean 0.53	

Portion analysed: Fruit

[a] Application date: 19 May–29 June 1999, Trial site: Trevor Nochols Research Complex, 124th Ave., Fennville

[b] Application date: 26 May–2 July 1999, Trial site: Trevor Nochols Research Complex, 124th Ave., Fennville

[c] Application date: 25 May – 5 July 1999, Trial site: Trevor Nochols Research Complex, 124th Ave., Fennville

[d] Application date: 21 May–1 July 1999,

Trial site: NW Michigan Horticultural Research Station, 6686 S. Center Highway, Traverse City

[e] Application date: 1 June – 12 July 1999, Trial site: NW Michigan Horticultural Research Station, 6686 S. Center Highway, Traverse City

[f] Application date: 16 April – 20 May 1999, Trial site: 7700 Cherokee Lane, Stockton

[g] Application date: 16 April – 20 May 1999, Trial site: 7700 Cherokee Lane, Stockton

Plum

Eight residue trials were conducted in the USA on plums according to the US GAP (Dorschner, 2002: 111044). The 192 g/L SC formulation was applied 5 times at the rate of 0.11 kg ai/ha with an application interval of 8-12 days. The total residue of imidacloprid were quantified with method 102624-R1 (based on the method 00200) at an LOQ of 0.05 mg/kg.

Table 12 Imidacloprid residues on plums from supervised trials in USA

Plum country, year (variety)	Application					DALA Days	Residues, mg/kg [a]	Ref
	Form	kg ai/ha	water, L/ha	Treatment	no.			
GAP, USA	*SC*	*Max 0.56 /year*				*7*		
USA, 1999 Bridgeton/NJ (Superior plum) 99-NJ16	SC	0.11 0.11 0.11 0.11 0.11	738 727 724 726 736	Fruiting Green sizing fruit Fruiting Fruiting Fruit enlarging	5	7	0.64, 0.70 Mean 0.67	111044 IR-4 PR. 07279 Dorschner, 2002
USA, 1999 Fennville/MI (Ealy Golden plum) 99-MI08	SC	0.11 0.11 0.11 0.11 0.11	922 939 942 917 927	Immature fruit Immature fruit Immature fruit Immature fruit Immature fruit	5	7	0.38, 0.46 Mean 0.42	Sampling to analysis: 75-235 days
USA, 1999 Gervais/OR (Brooks plum) 99-OR21	SC	0.11 0.11 0.11 0.11 0.11	1024 974 987 964 971	Green fruit Green fruit Fruit growth Ripening fruit Fruit maturing	5	6	0.089, 0.10 Mean 0.095	
USA, 1999 Gervais/OR (Brooks plum) 99-OR22	SC	0.12 0.11 0.11 0.11 0.11	770 756 766 747 746	Growing fruit, green Fruit growth Fruit growth Beginning to ripen Fruit ripening	5	7	0.077, 0.086 Mean 0.082	
USA, 1999 Buhl/ID (Simca Rosa	SC	0.11 0.11 0.11	924 936 938	Fruiting Fruiting Fruiting	5	7	0.16, 0.27 Mean 0.22	

Plum country, year (variety)	Application					DALA Days	Residues, mg/kg [a]	Ref
	Form	kg ai/ha	water, L/ha	Treatment	no.			
plum) 99-ID05		0.11 0.11	933 933	Fruiting Fruiting				
USA, 1999 Caldwell/ID (Empress plum) 99-ID06	SC	0.11 0.11 0.11 0.11 0.11	929 940 929 940 930	Fruiting Fruiting Fruiting Fruiting Fruiting	5	7	0.32, 0.35 Mean <u>0.34</u>	
USA, 1999 Kerman/CA (French prunes) 99-CA79	SC	0.11 0.11 0.11 0.11 0.11	1403 1417 1430 1398 1395	Small green prunes Fruit 0.5-1 inch Fruit 1-1.5 inch Coloring prunes Fruiting	5	0 3 6 13	0.44, 0.52 0.44, 0.46 0.30, 0.41 Mean 0.36 0.39, 0.39 Mean <u>0.39</u>	
USA, 1999 Chowchilla/CA (Fortune plums) 99-CA80	SC	0.11 0.11 0.11 0.11 0.11	1409 1415 1401 1407 1416	Fruit 0.75-1 inch Fruit 1.5-2 inch Fruit 1.5-2.5 inch Coloring fruit Fruiting	5	7	0.12, 0.19 Mean <u>0.15</u>	

[a] Portion analysed: Fruit without pit and stem

Peach

Sixteen side-by-side residue trials were conducted in the USA on peaches according to the US GAP (Harbin & Woodard, 2000: 109238). The 192 g/L SC formulation was applied 3 times at the rate of 0.11 kg ai/ha with application intervals of 7 days. Two different application scenarios (concentrated and dilute spraying) were tested within the same location. The total residue of imidacloprid were quantified with method 102624-R1 (based on the method 00200) at an LOQ of 0.05 mg/kg.

Table 13 Imidacloprid residues on peaches from supervised trials in USA

Peach country, year (variety)	Application							DALA Days	Residues, mg/kg [a]		Ref
	Form	kg ai/ha	kg ai/hL		L/ha		no.				
			dil	conc	dil	conc			dil	conc	
GAP, USA	*SC*	*Max 0.34 /year*						*0*			
USA, 1998 Fresno/CA (Red top) FCA-PO001-98D	SC	0.11 0.11 0.11	0.0030 0.0029 0.0027	0.023 0.023 0.028	3714 3770 4022	485 485 391	3	0 7 14 21	<u>0.10</u> 0.099 0.066 0.059	0.094 0.058 0.074 0.051	109238 Harbin & Woodard, 2000
USA, 1998 Tulare/CA (Carson) BAY-PO002-98H	SC	0.11 0.11 0.11	0.0047 0.0048 0.0047	0.018 0.018 0.019	2338 2271 2324	615 628 575	3	0	<u>0.34</u>	0.25	Sampling to analysis: 378-414 days
USA, 1998 Porterville/CA (Red sun) BAY-PO003-98H	SC	0.11 0.11 0.11	0.0042 0.0052 0.0042	0.016 0.020 0.020	2605 2118 2598	684 561 549	3	0	<u>0.25</u>	0.15	
USA, 1998 Gridley/CA (Lodell 19440 ex erly) BAY-PO004-98H	SC	0.11 0.11 0.11	0.0056 0.0052 0.0052	0.020 0.019 0.020	1962 2104 2106	541 574 542	3	0	0.36	<u>0.37</u>	
USA, 1998 Colony/OK (Glohaven) BAY-PO005-98H	SC	0.11 0.11 0.11	0.0047 0.0045 0.0041	0.020 0.019 0.017	2327 2418 2715	543 572 633	3	0	0.48	<u>0.77</u>	
USA, 1998 Centralia/IL (Crest haven BAY-PO006-98H	SC	0.11 0.11 0.11	0.0032 0.0033 0.0033	0.018 0.018 0.018	3391 3328 3374	618 613 595	3	0	<u>0.38</u>	0.33	

Peach country, year (variety)	Application						no.	DALA Days	Residues, mg/kg [a]		Ref
	Form	kg ai/ha	kg ai/hL		L/ha						
			dil	conc	dil	conc			dil	conc	
USA, 1998 Morven/GA (Gold prince) BAY-PO007-98H	SC	0.11 0.11 0.11	0.0047 0.0045 0.0046	0.020 0.021 0.018	2334 2430 2372	540 525 601	3	0	0.38	0.32	
USA, 1998 Hereford/PA (Glohaven) BAY-PO008-98H	SC	0.11 0.11 0.11	0.0034 0.0034 0.0034	0.018 0.018 0.018	3224 3222 3237	623 603 615	3	0	0.28	0.19	

[a] Portion analysed: Fruit

Assorted tropical and sub-tropical fruits–edible peel & Oilseed

Olives

Eight trials on olives were conducted in Spain, Portugal, Italy and Greece (Schöning & Berkum, 2009: RA-2032/07, Schöning, Reneke & Krusell, 2011: 08-2001). The 200 g/L OD formulation was applied 5 times as a low pressure bait application with 0.020 kg ai/ha, corresponding to a concentration of 0.02 kg ai/hL and a spray volume of 100 L/ha. Only the south side (25% of the whole trees) was treated but samples were taken randomly from the whole trees. The application rate was related to the size of the plot and not just to the area actually treated. At each application the additive Buminal (hydrolyzed protein) was used (1.5%). The application intervals were 9 to 13 days.

All trials were analysed for imidacloprid parent compound and the total residue of imidacloprid according to method 00834/M001. Additionally, the samples taken in 2008 were analysed for imidacloprid parent compound and the metabolites 5-hydroxy imidacloprid and olefin imidacloprid according to method 00834/M002 (not be shown in Table 5).

Table 14 Imidacloprid residues on olives from supervised trials in Southern Europe

Olive country, year (variety)	Application						DALA Days [b]	Residues, mg/kg		Ref
	Form	kg ai/ha	kg ai/hL	L/ha	Growth stage[a]	no.		Parent	Total	
GAP, Spain	*SL*	*0.01-0.02*		*50-100*		*4*	*7*			
Spain, 2007 Cataluña (Morrut) R2007 0408/9	OD	0.02	0.02	100	81-85	5	-0 0 4 7	0.27 0.45 - 0.40	0.47 0.64 0.56 0.71	RA-2032/07 Schöning & Berkum, 2009
Portugal, 2007 Ribatejo e Oeste (Galega) R2007 0409/7	OD	0.02	0.02	100	81-88	5	-0 0 7	0.33 0.70 0.40	0.81 1.3 1.1	Sampling to analysis: 63-141 days
Italy, 2007 Sicilia (Nocellara Etnea) R2007 0439/9	OD	0.02	0.02	100	78-80	5	-0 0 3 7	0.03 0.04 - 0.03	0.15 0.14 0.17 0.14	
Italy, 2007 Puglia (Corato) R2007 0440/2	OD	0.02	0.02	100	75-80	5	-0 0 7	0.13 0.30 0.30	0.33 0.57 0.63	
Spain, 2008 Cataluña (Vera) 08-2001-01	OD	0.02	0.02	100-114	80-88	5	-0 0 4 8	0.04 0.17 0.07 0.05	0.12 0.24 0.12 0.11	08-2001 Schöning, Reineke & Krusell, 2011
Italy, 2008 Sicilia (Bella di Spagna) 08-2001-02	OD	0.02	0.02	100	80-85	5	-0 0 3 7	< 0.01 0.04 0.02 0.02	< 0.05 0.06 < 0.05 < 0.05	Sampling to analysis: 484-

Olive country, year (variety)	Application						DALA Days [b]	Residues, mg/kg		Ref
	Form	kg ai/ha	kg ai/hL	L/ha	Growth stage[a]	no.		Parent	Total	
Portugal, 2008 Ribatejo e Oeste (Galega) 08-2001-03	OD	0.02	0.02	100	79-88	5	0 7	0.37 0.42	0.45 0.49	534 days
Greece, 2008 Katerini (Megaron) 08-2001-04	OD	0.02	0.02	100	76-81	5	0 7	0.22 0.11	0.34 0.22	

Portion analysed: Fruit

[a] Code of BBCH scale

[b] -0: the date before last treatment

Eight trials on olives were conducted in Spain, Italy, Portugal and Greece (Anderson & Eberhardt, 2002: RA-2065/00, Schöning, 2002: RA-2034/01). The 200 g/L SL formulation was applied twice as a spray application with 0.10 kg ai/ha, corresponding to a concentration of 0.0125 kg ai/hL and a spray volume of 800 L/ha. The application intervals were 28 to 32 days.

Fruits taken at day 0 at and the PHI of 28 days after the last application were analysed for parent compound whereas fruits of all sampling dates were analysed for the total residue of imidacloprid. Both analytes were either analysed according to method 00300/E007 or method 00834.

Table 15 Imidacloprid residues on olives from supervised trials in Southern Europe

Olive country, year (variety)	Application						DALA Days	Residues, mg/kg		Ref
	Form	kg ai/ha	kg ai/hL	L/ha	Growth stage*	no.		Parent	Total	
GAP, Spain	*SL*		*0.01*			*2*	*28*			
Spain, 2000 (Vera) R2000 0073/1	SL	0.10 0.10	0.013 0.013	800 800	81 85	2	0 6 11 21 28 35	0.12 0.02 	0.25 0.27 0.28 0.29 0.25 0.22	RA-2065/00 Anderson & Eberhardt, 2002
Italy, 2000 (Nocellara Etnea) R2000 0313/7	SL	0.10 0.093	0.013 0.013	800 743	87 87/88	2	0 7 14 22 28 35	0.10 < 0.01 	0.11 0.11 0.08 < 0.05 0.05 0.05	Sampling to analysis: 243-354 days
Portugal, 2000 (Blanqueta) R2000 0314/5	SL	0.10 0.10	0.013 0.013	800 800	No data 82	2	0 6 14 21 28 35	0.18 0.03 	0.60 0.36 0.19 0.16 0.21 0.07	
Greece, 2000 (Manaki) R2000 0315/3	SL	0.10 0.10	0.013 0.013	800 800	79 85	2	0 7 14 21 28 35	0.32 0.14 	0.59 0.71 0.51 0.67 0.73 0.43	
Spain, 2001 (Vera) R2001 0090/6	SL	0.11 0.10	0.013 0.013	904 800	79 79-81	2	0 6 14 19 27 35	0.14 0.09 0.05 0.02 	0.28 0.26 0.26 0.26 0.22 0.24	RA-2034/01 Schöning, 2002 Sampling to analysis: 134-195 days
Italy, 2001 (Nocellara Etnea) R2001 0091/4	SL	0.10 0.10	0.013 0.013	800 800	78 78-80	2	0 7 14	0.14 0.04 0.02	0.22 0.14 0.12	

Olive country, year (variety)	Application						DALA Days	Residues, mg/kg		Ref
	Form	kg ai/ha	kg ai/hL	L/ha	Growth stage*	no.		Parent	Total	
							20 28 35	 0.01	0.11 0.15 0.13	
Portugal, 2001 (Picual) R2001 0092/2	SL	0.10 0.10	0.013 0.013	800 800	75/76 79/80	2	0 7 14 21 28 35	0.31 0.11 0.09 0.06	0.39 0.22 0.19 0.10 0.16 0.16	
Greece, 2001 (Manaki) R2001 0093/0	SL	0.10 0.10	0.013 0.013	800 800	79 82	2	0 8 15 22 28 35	0.22 0.01 < 0.01 < 0.01	0.29 0.08 0.06 0.05 0.06 0.06	

Portion analysed: Fruit

* Code of BBCH scale

Eight trials on olives were conducted in Italy, Spain, Portugal and Greece (Schöning & Krusell, 2011: 09-2087, Schöning & Bauer, 2011: 10-2151). The 200 g/L OD formulation was applied once as a spray application with 0.15 kg ai/ha, corresponding to a concentration of 0.0125–0.0188 kg ai/hL and a spray volume of 800–1200 L/ha.

The samples were analysed for imidacloprid parent compound and the metabolites 5-hydroxy imidacloprid and olefin imidacloprid according to method 00834/M002 as well as for the total residue of imidacloprid according to method 00834/M001.

Table 16 Imidacloprid residues on olives from supervised trials in Southern Europe

Olive country, year (variety)	Application						DALA Days [b]	Residues, mg/kg		Ref
	Form	kg ai/ha	kg ai/hL	L/ha	Growth stage [a]	no.		Parent	Total	
GAP, Italy	*OD*		*0.01-0.013*			*1*	*28*			
Italy, 2009 (Nocellara Etnea) 09-2087-01	OD	0.15	0.015	1000	78	1	-0 0 7 14 28 35	< 0.01 0.33 0.16 0.06 < 0.01 < 0.01	< 0.05 0.31 0.29 0.30 0.23 0.28	09-2087 Schöning & Krusell, 2011 Sampling to analysis: 266-428 days
Spain, 2009 (Arbequina) 09-2087-02	OD	0.15	0.015	1000	85	1	-0 0 7 13 28 35	< 0.01 1.02 0.44 0.27 0.14 0.10	< 0.05 0.93 0.79 0.74 0.75 0.77	
Portugal, 2009 (Cobrançosa) 09-2087-03	OD	0.15	0.019	800	80	1	0 28	0.70 0.51	0.51 0.81	
Italy, 2009 (Nocellara Etnea) 09-2087-04	OD	0.15	0.013	1200	81	1	0 28	0.32 0.01	0.29 0.23	
Italy, 2010 (Bella di Spagna) 10-2151-01	OD	0.15	0.015	1000	78	1	0 7 14 28 35	0.21 0.16 0.07 < 0.01 < 0.01	0.16 0.18 0.13 0.26 0.20	10-2151 Schöning & Bauer, 2011
Spain, 2010 (Arbequina) 10-2151-02	OD	0.15	0.019	800	81	1	0 8 14	0.40 0.20 0.16	0.16 0.35 0.38	Sampling to analysis: 79-129 days

Olive country, year (variety)	Application						DALA Days [b]	Residues, mg/kg		Ref
	Form	kg ai/ha	kg ai/hL	L/ha	Growth stage [a]	no.		Parent	Total	
							28 35	0.04 0.04	0.41 0.43	
Portugal, 2010 (Cobrançosa) 09-2087-03	OD	0.15	0.019	800	81	1	0 30	0.77 0.43	0.68 0.61	
Greece, 2010 (Amphisses) 10-2151-04	OD	0.15	0.019	800	79	1	0 28	0.34 < 0.01	0.24 0.12	

Portion analysed: Fruit

[a] Code of BBCH scale

[b] -0: The date before last treatment

Leafy vegetables (including Brassica leafy vegetables)

Kale

Four trials were conducted on curly kale in Spain and Italy (Schmeer, Krusell & Bauer, 2010: 08-2029, Ballesteros, 2011: 09-2002). The OD formulation containing 75 g/L imidacloprid and 10 g/L deltamethrin was applied twice as a spray application with 0.094 kg ai/ha, corresponding to a concentration of 0.012–0.016 kg ai/hL and a spray volume of 600–800 L/ha. The application interval was 13–18 days.

The samples were analysed for the parent imidacloprid and its metabolites 5-hydroxy imidacloprid and olefin imidacloprid according to method 00834/M002. The total residue of imidacloprid was determined as 6-CNA common moiety according to method 00834/M001.

Table 17 Imidacloprid residues on curly kale from supervised trials in Spain and Italy

Kale country, year (variety)	Application						DALA Days [b]	Residues, mg/kg		Ref
	Form	kg ai/ha	Kg ai/hL	L/ha	Growth stage [a]	no.		Parent	Total	
GAP, Italy	*OD*	0.075-0.094				*2*	*7*			
Spain, 2008 (Reflex F1) 08-2029-01	OD	0.094 0.094	0.016 0.012	600 800	47 49	2	-0 0 3 6 13	< 0.01 2.9 0.46 0.09 0.02	0.46 3.7 1.8 <u>1.1</u> 0.82	08-2029 Schmeer, Krusell & Bauer, 2010
Italy, 2008 (Nero di Toscana) 08-2029-02	OD	0.094 0.094	0.012 0.012	800 800	42 46	2	-0 0 3 7 14	0.03 2.5 0.64 0.20 0.10	0.34 3.3 2.2 <u>1.5</u> 0.99	Sampling to analysis: 307-596 days
Spain, 2009 (Reflex F1) 09-2002-01	OD	0.094 0.094	0.016 0.016	600 600	42 45	2	-0 0 3 7 15	0.16 2.9 0.99 0.34 0.04	0.93 4.1 2.8 <u>2.0</u> 1.1	09-2002 Ballesteros, 2011
Italy, 2009 (Nero di Toscana) 09-2002-02	OD	0.094 0.094	0.012 0.012	800 800	41 43	2	-0 0 3 6 14	0.02 1.5 0.50 0.31 0.09	0.36 2.3 1.0 <u>1.0</u> 0.64	Sampling to analysis: 85-198 days

Portion analysed: Leaf

[a] Code of BBCH scale

[b] 0: The date before last treatment

Fruiting vegetables, other than Cucurbits–subgroup Tomatoes

Goji berry

Six trials were conducted on goji in China, using the EC formulation containing 50 g/L imidacloprid. The EC formulation was applied with three foliar applications at a concentration of 0.005 kg ai/hL. The application interval was 10 days.

After fresh goji were collected from field trial, 5 g potassium carbonate (0.5% of the weight of fresh goji sample) was added per 1000 g sample, then well mixed and stood for 30 min. The sample was dried in sunshine or under blast drying under 45-50°C. The water content of goji is 70-80%. So the weight of dried goji is about 20-30% of that before drying.

The analytical method NY/T 1275-2007 & GB/T 23201-2008 was used to determine the residue of imidacloprid on goji. The LOQ was 0.02 mg/kg.

Table 18 Imidacloprid residues on goji from supervised trials in China

Goji country, year (variety)	Application			DALA Days	Portion analysed	Residues, mg/kg* Imidacloprid	Ref
	Form	kg ai/hL	no.				
GAP, China	*EC*	*0.003-0.005*	*3*	*3*			
China, 2010 Yinchuan/ Ningxia Hui (Ningqi No. 1) NX-01	EC	0.005	3	1 2 3 5 7 10 14	Fresh fruits	0.078, 0.099, 0.11 (0.096) 0.052, 0.054, 0.082 (0.063) 0.021, 0.032, 0.067 (0.040) < 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 (< 0.02)	R-IG-03 Niu, 2014 Sampling to analysis: 131-150 days
				5 7 10 14	Dried fruits	< 0.02, 0.023, 0.038 (0.027) 0.059, 0.062 (0.061) 0.054, 0.055 (0.055) 0.025, 0.027, 0.027 (0.026)	
China, 2010 Zhongning/ Ningxia Hui (Ningqi No. 1) NX-02	EC	0.005	3	1 2 3 5 7 10 14 21	Fresh fruits	0.32, 0.34, 0.69 (0.45) 0.54, 0.64, 0.69 (0.62) 0.36, 0.43, 0.47 (0.42) 0.033, 0.035, 0.036 (0.035) 0.029, 0.030, 0.030 (0.030) 0.051, 0.055, 0.058 (0.055) < 0.02, < 0.02, 0.024 (0.021) < 0.02, < 0.02, < 0.02 (< 0.02)	R-IG-04 Niu, 2014 Sampling to analysis: 129-149 days
				5 7 10 14	Dried fruits	0.063, 0.092, 0.10 (0.085) < 0.02, 0.025, 0.034 (0.026) < 0.02, < 0.02, < 0.02 (< 0.02) < 0.02, < 0.02, 0.024 (0.021)	
China, 2010 Bayannaoer/ Inner Mongolia (Ningqi No. 1) IM-01	EC	0.005	3	1 2 3 5 7 10 14 21	Fresh fruits	0.77, 0.84, 0.99 (0.87) 0.57, 0.61, 0.67 (0.62) 0.57, 0.59, 0.78 (0.65) 0.36, 0.39, 0.41 (0.39) 0.28, 0.31, 0.35 (0.31) 0.25, 0.26, 0.26 (0.26) 0.072, 0.094, 0.12 (0.095) < 0.02, < 0.02, < 0.02 (< 0.02)	R-IG-05 Zhang, 2014 Sampling to analysis: 130-150 days
				5 7 10 14	Dried fruits	0.42, 0.64 (0.53) 0.49, 0.54 (0.52) 0.29, 0.30, 0.36 (0.32) 0.24, 0.31 (0.28)	

Goji country, year (variety)	Application Form	kg ai/hL	no.	DALA Days	Portion analysed	Residues, mg/kg* Imidacloprid	Ref
China, 2010 Baiyin/ Gansu (Ningqi No. 1) GS-01	EC	0.005	3	1 2 3 5 7 10 14 21	Fresh fruits	0.38, 0.44, 0.85 (0.56) 0.25, 0.45, 0.47 (0.39) 0.26, 0.32, 0.48 (0.35) 0.17, 0.19, 0.20 (0.19) 0.10, 0.22 (0.16) 0.054, 0.066, 0.13 (0.083) 0.012, 0.030 (0.021) < 0.02, < 0.02, < 0.02 (< 0.02)	R-IG-06 Liu, 2014 Sampling to analysis: 124-144 days
				5 7 10 14	Dried fruits	0.10, 0.11, 0.14 (0.12) 0.049, 0.050, 0.17 (0.090) < 0.02, 0.029, 0.040 (0.030) < 0.02, < 0.02, 0.026 (0.022)	
China, 2010 Xinjiang Uygur (Ningqi No. 1) XJ-01	EC	0.005	3	1 2 3 5 7 10 14 21	Fresh fruits	0.29, 0.34, 0.41 (0.35) 0.30, 0.31, 0.41 (0.34) 0.30, 0.31, 0.39 (0.33) 0.24, 0.28, 0.31 (0.28) 0.28, 0.30, 0.37 (0.32) 0.22, 0.27, 0.30 (0.26) 0.25, 0.27, 0.28 (0.27) 0.020, 0.14, 0.14 (0.10)	R-IG-07 Gou, 2014 Sampling to analysis: 122-142 days
				5 7 10 14	Dried fruits	0.044, 0.047, 0.086 (0.059) 0.059, 0.14, 0.17 (0.12) 0.054, 0.075, 0.076 (0.068) 0.051, 0.068, 0.070 (0.063)	
China, 2010 Haixi, Qinghai (Ningqi No. 1) QH-01	EC	0.005	3	1 2 3 5 7 10 14 21	Fresh fruits	0.40, 0.47, 0.51 (0.46) 0.17, 0.37, 0.56 (0.37) 0.23, 0.30, 0.44 (0.32) 0.16, 0.21, 0.25 (0.21) 0.17, 0.17, 0.17 (0.17) 0.13, 0.17, 0.20 (0.17) 0.27, 0.44 (0.36) 0.083, 0.18, 0.39 (0.22)	R-IG-08 Gou, 2014 Sampling to analysis:
				5 7 10 14	Dried fruits	0.74, 0.74, 0.78 (0.75) 0.22, 0.46, 0.46 (0.38) 0.62, 0.63 (0.63) 0.28, 0.28, 0.29 (0.28)	

* Average in parentheses

Pulses

Soya bean (dry)

Twenty-one field residue trials were carried out with imidacloprid in soya beans in the USA and Canada using the 480 g/L SC formulation (Mackie, 2006: RANTY002). Soya bean seeds were treated at a rate of 0.125 kg ai/100 kg seed. The growing soya bean plants were subsequently treated with three foliar applications at a target rate of 0.053 kg ai/ha for a total seasonal application of 0.16 kg ai/ha. In each of the 21 residue trials, the treated plot was divided in two sub-plots A and B; from sub-plot A, forage and hay was harvested, and from sub-plot B, soya bean seeds. The application intervals, once foliar treatment was initiated, generally ranged between 5 and 7 days.

The analytical method NT-001-P04-01 (common moiety method) was used to determine the total residue of imidacloprid in soya bean seeds.

Table 19 Imidacloprid residues on soya bean seeds from supervised trials in USA and Canada

Soya bean seed country, year (variety)	Application Form	kg ai/ha	Seeding density water, L/ha	no.	DALA Days	Residues, mg/kg* Total imidacloprid	Ref
GAP, USA	*SC*	*63-125 g ai/100 kg seed*		*1*	*21*		

Soya bean seed country, year (variety)	Application				DALA Days	Residues, mg/kg* Total imidacloprid	Ref
	Form	kg ai/ha	Seeding density water, L/ha	no.			
		0.053 *Max 0.16 kg ai/ha /year*		*3*			
USA, 2004	SC	0.070	56.3 kg/ha	1	7	0.035, 0.036 (0.035)	RANTY002 Mackie, 2006
Tifton/GA		0.053	141	3	14	0.022, 0.031 (0.027)	
(DK 5386)		0.053	141		21	0.047, 0.054 (0.050)	
NT001-04D		0.053	140		28	0.037, 0.039 (0.038)	
					34	0.042, 0.043 (0.042)	Sampling to analysis: max 450 days
USA, 2004	SC	0.082	65.9 kg/ha	1	19	0.17, 0.25 (0.21)	
Bumpass/VA		0.052	162	3			
(Pioneer 9492RR)		0.053	163				
NT002-04H		0.053	163				
USA, 2004	SC	0.091	72.5 kg/ha	1	20	0.35, 0.41 (0.38)	
Leland/MS		0.054	151	3			
(Pioneer 9492RR)		0.052	151				
NT003-04H		0.052	157				
USA, 2004	SC	0.098	78.5 kg/ha	1	21	0.64, 0.71 (0.67)	
Proctor/AR		0.054	140	3			
(DK 5386)		0.054	140				
NT004-04H		0.053	139				
USA, 2004	SC	0.087	69.4 kg/ha	1	21	0.32, 0.43 (0.38)	
Newport/AR		0.053	185	3			
(DK 5386)		0.054	189				
NT005-04H		0.054	188				
USA, 2004	SC	0.10	79.9 kg/ha	1	8	0.039, 0.041 (0.040)	
Stilwell/KS		0.055	153	3	14	0.038, 0.042 (0.040)	
(Pioneer 93B68)		0.056	153		20	0.024, 0.030 (0.027)	
NT006-04D		0.053	145		27	0.030, 0.031 (0.031)	
					34	0.033, 0.037 (0.035)	
USA, 2004	SC	0.090	72.2 kg/ha	1	19	0.17, 0.19 (0.18)	
Seymour/IL		0.054	132	3			
(Pioneer 93B68)		0.054	132				
NT007-04H		0.054	132				
USA, 2004	SC	0.093	74.2 kg/ha	1	20	0.066, 0.15 (0.11)	
Springfield/NE		0.054	139	3			
(S2802-4)		0.053	140				
NT008-04H		0.053	133				
USA, 2005	SC	0.096	76.9 kg/ha	1	21	0.18, 0.20 (0.19)	
Sabin/MN		0.053	153	3			
(Northrup King)		0.055	158				
NT009-04HA		0.054	152				
USA, 2004	SC	0.092	74.0 kg/ha	1	20	0.40, 0.46 (0.43)	
Carlock/IL		0.053	121	3			
(S2802-4)		0.052	119				
NT010-04H		0.055	124				
USA, 2004	SC	0.15	122 kg/ha	1	19	0.45, 0.52 (0.48)	
Bagley/IA		0.053	130	3			
(S2802-4)		0.051	132				
NT011-04H		0.051	134				
USA, 2004	SC	0.084	67.3 kg/ha	1	19	0.61, 0.65 (0.63)	
Marysville/OH		0.053	140	3			
(S2802-4)		0.053	141				
NT012-04H		0.053	140				
USA, 2004	SC	0.090	71.6 kg/ha	1	21	0.94, 2.0 (1.5)	
Dumfries/MN		0.054	176	3			
(Pioneer 92B13)		0.055	179				
NT013-04H		0.053	178				
USA, 2004	SC	0.071	57.2 kg/ha	1	20	0.56, 0.65 (0.61)	
Northwood/ND		0.052	185	3			
(S02-G2)		0.052	186				
NT014-04H		0.053	187				
USA, 2004	SC	0.092	73.8 kg/ha	1	21	0.73, 0.73 (0.73)	

Soya bean seed country, year (variety)	Application				DALA Days	Residues, mg/kg* Total imidacloprid	Ref
	Form	kg ai/ha	Seeding density water, L/ha	no.			
Gardner/ND (Pioneer 90B51) NT015-04H		0.052 0.053 0.053	182 181 161	3			
USA, 2004 New Holland/OH (Pioneer 93B68) NT016-04H	SC	0.094 0.053 0.054 0.054	74.9 kg/ha 148 153 150	1 3	32	0.025, 0.029 (0.027)	
USA, 2004 Kirksville/MO (Pioneer 93B68) NT017-04H	SC	0.085 0.053 0.052 0.054	68.3 kg/ha 165 170 178	1 3	21	1.4, 1.6 (1.5)	
USA, 2004 Ellendale/MN (Pioneer 92B13) NT018-04H	SC	0.11 0.054 0.055 0.054	83.9 kg/ha 154 156 160	1 3	21	0.57, 0.67 (0.62)	
USA, 2004 Carlyle/IL (Pioneer 93B68) NT019-04H	SC	0.087 0.053 0.053 0.052	69.3 kg/ha 92 137 93	1 3	21	0.039, 0.065 (0.052)	
USA, 2004 Rockwood/ON (S02-G2) NT020-04H	SC	0.094 0.053 0.053 0.053	75.2 kg/ha 94 95 97	1 3	25	0.034, 0.069 (0.052)	
USA, 2004 Bright/ON (Pioneer 90B51) NT021-04H	SC	0.094 0.054 0.053 0.053	75.2 kg/ha 92 101 89	1 3	25	0.093, 0.096 (0.094)	

* Average in parentheses

Herbs

Basil

Four field residue trials were carried out with imidacloprid in basil in Thailand using the 700 g/kg WG formulation. The growing basil plants were treated with two foliar applications at a target concentration of 0.042 kg ai/hL. The application intervals were 7 or 8 days.

The on-line multi residue methods applied for the determination of imidacloprid residues was based on extraction with a mixture of acetone, dichloromethane and sodium chloride water solution. The concentrated extract is cleaned up on silica gel column and detection with HPLC-MS/MS (Steinwandter, 1985). The LOQ for imidacloprid was 0.01 mg/kg. The total residue of imidacloprid (6-CNA common moiety analysis) was determined according to method 01389. The LOQ for total imidacloprid was 0.05 mg/kg.

Table 20 Imidacloprid residues on basil from supervised trials in Thailand [a]

Basil country, year (variety)	Application				DALA Days	Residues, mg/kg*		Ref.
	Form	kg ai/ha	kg ai/hL	no.		Imidacloprid	Total imidacloprid	
GAP, Thailand	*WG*		*0.021-0.042*		*7*			
Thailand, 2014 Nakornpratom (White Holy basil)	WG	0.34	0.042	2	0 1 3 5 7 10 14	16, 24, 26 (22) 8.4, 14, 22 (15) 4.7, 5.0, 5.4 (5.0) 1.5, 2.8, 3.0 (2.4) 0.94, 1.1, 1.4 (1.1) 0.34, 0.41, 0.55 (0.43) 0.04, 0.07, 0.20 (0.10)	16, 28, 38 (27) 16, 25, 40 (27) 4.2, 4.8, 5.8 (4.9) 2.8, 3.5, 5.1 (3.8) 2.5, 4.3, 5.3 (4.0) 3.3, 5.6, 6.5 (5.1) 3.3, 3.8, 3.8 (3.6)	Imida- basil-1 Chaiyanboon, 2014 Sampling to analysis: 268-284 days
Thailand, 2014	WG	0.32	0.042	2	0	31, 46, 49 (42)	40, 49, 50 (46)	Imida-basil-2

Basil country, year (variety)	Application				DALA Days	Residues, mg/kg*		Ref.
	Form	kg ai/ha	kg ai/hL	no.		Imidacloprid	Total imidacloprid	
Saraburi (Red Holy basil)					1 3 5 7 10 14	10, 11, 12 (11) 1.1, 2.8, 3.4 (2.4) 1.2, 1.2, 1.3 (1.2) 0.41, 0.43, 0.49 (0.44) 0.15, 0.21, 0.21 (0.19) < 0.01,< 0.01, 0.03 (0.017)	19, 21, 21 (20) 23, 23, 25 (23) 8.1, 10, 13 (11) 6.1, 6.2, 7.3 (6.5) 4.4, 4.5, 4.8 (4.6) 2.7, 3.4, 3.6 (3.2)	Thongsam, 2014 Sampling to analysis: 256-271 days
Thailand, 2014 Nakhonpathom (Sweet basil)	WG	0.32	0.042	2	0 1 3 5 7 10 14	24, 24, 25 (24) 13, 15, 15 (14) 4.1, 4.3, 4.8 (4.4) 2.1, 2.2, 2.3 (2.2) 0.98, 1.1, 1.2 (1.1) 0.29, 0.32, 0.37 (0.33) 0.17, 0.18, 0.26 (0.20)	23, 25, 29 (26) 18, 19, 20 (19) 8.4, 8.7, 9.7 (8.9) 7.3, 7.3, 8.3 (7.6) 4.2, 4.7, 5.7 (4.9) 2.4, 2.5, 2.9 (2.6) 0.95, 1.2, 1.9 (1.3)	Imida-basil-3 Pongpinyo 2014 Sampling to analysis: 215-232 days
Thailand, 2014 Supanburi (Sweet basil)	WG	0.26	0.042	2	0 1 3 5 7 10 14	18, 19, 21 (19) 3.9, 4.1, 4.1 (4.0) 1.5, 1.7, 1.7 (1.6) 0.64, 0.70, 0.74 (0.69) 0.19, 0.22, 0.23 (0.21) 0.06, 0.07, 0.07 (0.07) 0.02, 0.03, 0.06 (0.04)	21, 24, 30 (25) 10, 11, 14 (12) 6.0, 6.1, 6.4 (6.1) 4.7, 6.0, 6.2 (5.6) 3.9, 4.5, 4.7 (4.3) 1.8, 1.9, 2.3 (2.0) 0.42, 0.54, 0.96 (0.64)	Imida-basil-4 Phaikaew 2014 Sampling to analysis: 264-280 days

[a] Portion analysed: whole commodity

* Average in parentheses

Tea, Green, Black

A total of eight trials (four decline and four harvest trials) were conducted at four different trial locations during the dry season (Manikandan, 2015: RANTN021). Four trials were conducted in spring and the other remaining four trials in autumn. Imidacloprid 700 g/kg WG formulation was applied in a spray application once at 0.40 kg ai/ha. In decline trials tea shoots (two to three leaves and a bud) were harvested 0, 3, 7 and 10 days after the application. In three of them duplicate composite samples were taken and in one decline trial only single samples were taken. In harvest trials, tea shoots were harvested immediately after the application and 7 days thereafter. In all the decline and harvest trials a portion of the tea shoots harvested 7 days after application was used to manufacture green and black tea.

For green tea production, tea shoots comprising of two to three leaves and bud harvested from experimental plots were subjected for steaming for about 10 min. Then the leaves were allowed to cool down to room temperature and subjected to CTC (Crush, Tear and Curl) manufacturing process. The tea leaves were dried for about 20 min in a fluidized bed drier, and cooled to ambient temperature.

For black tea production, the tea leaves were spread to a thickness of about 6.4 cm in a miniature withering trough and allowed to wither for 15-16 hours. The withered leaves were put into a rolling machine and rolled. The rolled leaves were then passed through a CTC machine. Afterwards, the tea was fermented at a humidity of 90%. The fermented tea was dried in a fluid bed drier. The dried tea was allowed to cool at ambient conditions.

All samples were analysed for imidacloprid parent compound and its metabolites and the total residue of imidacloprid according to method 01389. The LOQ was 0.01 mg/kg for imidacloprid in fresh and green tea leaves and 0.05 mg/kg in black tea. The LOQ of the total residue of imidacloprid was 0.05 mg/kg in all sample materials.

Table 21 Imidacloprid residues on tea (fresh leaves) from supervised trials in India

Tea	Application	DALA	Residues, mg/kg*	Ref

country, year (variety)	Form	kg ai/ha	kg ai/hL	L/ha	no.	Days	Parent	Total	
GAP, Japan	*WG*		0.005-0.01	*2000-4000*	*1*	*7*			
India, 2013 Gudalur (Assam Jat/ Seedling tea) S1	WG	0.40	0.089	450	1	0 3 7 10	54, 58 (56) 0.77, 0.99 (0.88) 0.40, 0.52 (0.46) 0.32, 0.37 (0.35)	53, 59 (56) 1.3, 1.4 (1.4) 0.70, 0.82 (0.76) 0.58, 0.65 (0.62)	Sampling to analysis: 207-370 days
India, 2012 Meppadi (Assam Jat/ Seedling tea) S2	WG	0.40	0.089	450	1	0 3 7 10	43, 46 (45) 7.7, 8.6 (8.2) 0.93, 1.1 (1.0) 0.95, 1.0 (0.98)	48, 51 (50) 12, 13 (13) 2.7, 2.8 (2.8) 2.8, 2.8 (2.8)	Sampling to analysis: 189-298 days
India, 2012 Coonoor (Assam Jat/ UPASI-9) S3	WG	0.40	0.089	450	1	0 7	21 c:0.038 1.5	25 2.3	Sampling to analysis: 175-291 days
		1.2	0.27	450	1	0 7	116 4.4, 4.5 (4.5)	151 7.1, 7.2 (7.2)	
India, 2013 Valparai (Assam Jat/ Seedling tea) S4	WG	0.40	0.089	450	1	0 7	39 0.64	48 1.2	Sampling to analysis: 175-281 days
		1.2	0.27	450	1	0 7	124 1.4, 1.4 (1.4)	155 2.6, 3.2 (2.9)	
India, 2012 Valparai (Assam Jat/ Mixed seedling tea) N1	WG	0.40	0.089	450	1	0 3 7 10	46, 51 (49) c:0.040, 0.26 7.2, 7.4 (7.3) 2.8, 4.2 (3.5) c:0.20 2.8, 3.1 (3.0)	51, 56 (54) c:0.29 10, 11 (11) 4.3, 6.1 (5.2) c:0.36 4.0, 4.0 (4.0)	Sampling to analysis: 39-96 days
India, 2011 Coonoor (Assam Jat/ UPASI-9) N2	WG	0.40	0.089	450	1	0 3 7 10	7.5 0.88 0.77 0.57	7.9 0.92 0.87 0.76	Sampling to analysis: 46-100 days
India, 2010 Meppadi (Assam Jat/ Seedling tea) N3	WG	0.40	0.089	450	1	0 7	54 0.48	59 1.0	Sampling to analysis: 56-102 days
India, 2011 Gudalur (Assam Jat/ TRI-2024) N4	WG	0.40	0.089	450	1	0 7	31 1.4	32 2.4	Sampling to analysis: 54-101 days

* Average in parentheses

c: control sample

Table 22 Imidacloprid residues on tea (Green tea and Black tea) from supervised trials in India

Tea country, year (variety)	Application					DALA Days	a	Residues, mg/kg*		Ref
	Form	kg ai/ha	kg ai/hL	L/ha	no.			Parent	Total	
GAP, Japan	*WG*		0.005-0.01	*2000-4000*	*1*	*7*				Sampling to analysis
India, 2013 Gudalur (Assam Jat/ Seedling tea) S1	WG	0.40	0.089	450	1	7	G	1.7, 1.7 (1.7) c:0.011, 0.013	2.7, 3.1 (2.9)	269-310 days
							B	1.3, 1.6 (1.5)	3.1, 3.4 (3.3)	264-313 days
India, 2012 Meppadi	WG	0.40	0.089	450	1	7	G	4.7, 4.8 (4.8) c:0.018, 0.023	11, 12 (12) c:0.40	253-294

Tea country, year (variety)	Application					DALA Days	[a]	Residues, mg/kg*		Ref
	Form	kg ai/ha	kg ai/hL	L/ha	no.			Parent	Total	
(Assam Jat/ Seedling tea) S2										days
							B	5.1, 5.1 (5.1)	12, 12 (12)	248-297 days
India, 2012 Coonoor (Assam Jat/ UPASI-9) S3	WG	0.40	0.089	450	1	7	G	5.4 c:0.13	11 c:0.18	236-283 days
							B	4.0 c:0.13	12 c:0.47	231-286 days
		1.2	0.27	450	1	7	G	16, 16 (16)	34, 34 (34)	
							B	13, 14 (14)	33, 35 (34)	
India, 2013 Valparai (Assam Jat/ Seedling tea) S4	WG	0.40	0.089	450	1	7	G	2.0 c:0.012	5.5 c:0.099	236-280 days
							B	2.2 c:0.54	5.1 c:0.96	231-280 days
		1.2	0.27	450	1	7	G	4.8, 4.8 (4.8)	12, 12 (12)	
							B	4.1, 4.9 (4.5)	13, 13 (13)	
India, 2012 Valparai (Assam Jat/ Mixed seedling tea) N1	WG	0.40	0.089	450	1	7	G	16, 16 (16) c:0.77, 0.90	22, 23 (23) c:0.91, 1.1	56-100 days
							B	15, 15 (15) c:0.33, 0.51	27, 29 (28) c:0.70, 0.86	62-103 days
India, 2011 Coonoor (Assam Jat/ UPASI-9) N2	WG	0.40	0.089	450	1	7	G	2.8 c:0.12	3.0 c:0.21	64-107 days
							B	1.8	2.7	70-110 days
India, 2010 Meppadi (Assam Jat/ Seedling tea) N3	WG	0.40	0.089	450	1	7	G	1.0 c:0.056	2.9 c:0.14	68-111 days
							B	0.90	2.7	72-112 days
India, 2011 Gudalur (Assam Jat/ TRI-2024) N4	WG	0.40	0.089	450	1	7	G	5.2 c:0.031	7.3 c:0.077	65-108 days
							B	2.7 c:0.070	5.1 c:0.19	71-111 days

* Average in parentheses

[a] commodity, G: green tea, B: black tea

c: control sample

Legume animal feeds

Soya bean fodder and forage (green)

Twenty-one field residue trials were carried out with imidacloprid in soya beans in the USA and Canada using the 480 g/L SC formulation (Mackie, 2006: RANTY002). Soya bean seeds were treated at a rate of 0.125 kg ai/100 kg seed. The growing soya bean plants were subsequently treated with three foliar applications at a target rate of 0.053 kg ai/ha for a total seasonal application of 0.16 kg ai/ha. Sample materials were forage and hay. In each of the 21 residue trials, the treated plot was divided in two sub-plots A and B; from sub-plot A, forage and hay was harvested, and from sub-

plot B, soya bean seeds. The application intervals, once foliar treatment was initiated, generally ranged between 5 and 7 days.

The analytical method NT-001-P04-01 (common moiety method) was used to determine the total residue of imidacloprid in soya bean forage and hay.

Table 23 Imidacloprid residues on soya bean forage and hay from supervised trials in USA and Canada

Soya bean forage/hay country, year (variety)	Application				DALA Days	Portion analysed	Residues, mg/kg *	Ref
	Form	kg ai/ha	Seeding density water, L/ha	no.				
GAP, USA	*SC*	*63-125 g ai/100 kg seed*		*1*	*0*			
		0.053		*3*				
		Max 0.16 kg ai/ha /year						
USA, 2004 Tifton/GA (DK 5386) NT001-04D	SC	0.067	53.8 kg/ha	1	0	Forage	3.5, 4.2 (3.9)	RANTY002 Mackie, 2006 Sampling to analysis: max 325 days for forage, max 336 days for hay
		0.053	140	3	1		1.7, 1.8 (1.7)	
		0.053	143		3		1.4, 1.5 (1.5)	
		0.053	138		7		0.89, 1.1 (1.0)	
					10		0.86, 0.90 (0.88)	
					0	Hay	9.2, 9.7 (9.4)	
					1		4.3, 4.4 (4.4)	
					3		4.1, 5.1 (4.6)	
					7		3.2, 4.0 (3.6)	
					10		1.9, 2.2 (2.0)	
USA, 2004 Bumpass/VA (Pioneer 9492RR) NT002-04H	SC	0.082	65.9 kg/ha	1	0	Forage	1.5, 1.8 (1.6)	
		0.055	135	3				
		0.054	134			Hay	8.0, 11 (9.6)	
		0.055	135					
USA, 2004 Leland/MS (Pioneer 9492RR) NT003-04H	SC	0.064	51.0 kg/ha	1	0	Forage	4.2, 4.6 (4.4)	
		0.053	151	3				
		0.056	147			Hay	17, 24 (21)	
		0.053	148					
USA, 2004 Proctor/AR (DK 5386) NT004-04H	SC	0.098	78.5 kg/ha	1	0	Forage	2.5, 3.0 (2.7)	
		0.055	42	3				
		0.052	136			Hay	5.4, 6.0 (5.7)	
		0.052	140					
USA, 2004 Newport/AR (DK 5386) NT005-04H	SC	0.087	69.4 kg/ha	1	0	Forage	3.5, 4.2 (3.8)	
		0.054	186	3				
		0.053	185			Hay	21, 21 (21)	
		0.055	190					
USA, 2004 Stilwell/KS (Pioneer 93B68) NT006-04D	SC	0.11	86.1 kg/ha	1	0	Forage	1.1, 1.2 (1.1)	
		0.056	149	3	1		0.79, 1.4 (1.1)	
		0.056	143		3		0.90, 0.94 (0.92)	
		0.051	134		7		0.62, 0.78 (0.70)	
					10		0.61, 0.78 (0.69)	
					0	Hay	3.5, 4.3 (3.9)	
					1		3.0, 3.6 (3.3)	
					3		3.4, 4.7 (4.0)	
					7		1.7, 2.0 (1.8)	
					10		2.8, 3.0 (2.9)	
USA, 2004 Seymour/IL (Pioneer 93B68) NT007-04H	SC	0.089	71.1 kg/ha	1	0	Forage	2.3, 2.8 (2.6)	
		0.055	129	3				
		0.055	128			Hay	9.0, 9.4 (9.2)	
		0.054	128					
USA, 2004 Springfield/NE (S2802-4) NT008-04H	SC	0.093	74.2 kg/ha	1	0	Forage	1.9, 2.3 (2.1)	
		0.053	134	3				
		0.053	135			Hay	6.1, 6.9 (6.5)	
		0.053	131					
USA, 2005 Sabin/MN (Northrup King) NT009-04HA	SC	0.096	76.9 kg/ha	1	0	Forage	1.7, 1.9 (1.8)	
		0.055	163	3				
		0.053	154			Hay	3.8, 5.3 (4.5)	
		0.053	149					

Soya bean forage/hay country, year (variety)	Application				DALA Days	Portion analysed	Residues, mg/kg *	Ref
	Form	kg ai/ha	Seeding density water, L/ha	no.				
USA, 2004 Carlock/IL (S2802-4) NT010-04H	SC	0.092 0.055 0.053 0.054	74.0 kg/ha 121 118 119	1 3	0	Forage Hay	3.2, 3.3 (3.2) 12, 15 (14)	
USA, 2004 Bagley/IA (S2802-4) NT011-04H	SC	0.15 0.051 0.052 0.052	122 kg/ha 106 155 156	1 3	0	Forage Hay	1.9, 2.3 (2.1) 8.3, 10 (9.1)	
USA, 2004 Marysville/OH (S2802-4) NT012-04H	SC	0.084 0.053 0.053 0.053	67.3 kg/ha 160 141 141	1 3	0	Forage Hay	4.1, 8.9 (6.5) 5.8, 16 (11)	
USA, 2004 Dumfries/MN (Pioneer 92B13) NT013-04H	SC	0.090 0.053 0.054 0.053	71.6 kg/ha 174 176 175	1 3	0	Forage Hay	2.3, 2.5 (2.4) 15, 15 (15)	
USA, 2004 Northwood/ND (S02-G2) NT014-04H	SC	0.071 0.052 0.052 0.052	57.2 kg/ha 140 140 139	1 3	0	Forage Hay	3.7, 4.5 (4.1) 8.2, 8.9 (8.5)	
USA, 2004 Gardner/ND (Pioneer 90B51) NT015-04H	SC	0.092 0.054 0.053 0.054	73.8 kg/ha 200 171 172	1 3	0	Forage Hay	4.3, 4.8 (4.6) 21, 24 (22)	
USA, 2004 New Holland/OH (Pioneer 93B68) NT016-04H	SC	0.094 0.054 0.054 0.053	74.9 kg/ha 145 145 146	1 3	0	Forage Hay	3.2, 3.8 (3.5) 5.7, 9.3 (7.5)	
USA, 2004 Kirksville/MO (Pioneer 93B68) NT017-04H	SC	0.085 0.051 0.052 0.053	68.3 kg/ha 149 169 173	1 3	0	Forage Hay	3.0, 4.0 (3.5) 11, 14 (13)	
USA, 2004 Ellendale/MN (Pioneer 92B13) NT018-04H	SC	0.11 0.054 0.054 0.053	83.9 kg/ha 152 147 146	1 3	0	Forage Hay	3.3, 4.3 (3.8) 9.5, 10 (9.9)	
USA, 2004 Carlyle/IL (Pioneer 93B68) NT019-04H	SC	0.087 0.054 0.052 0.054	69.3 kg/ha 145 120 150	1 3	0	Forage Hay	3.5, 5.0 (4.2) 12, 14 (13)	
USA, 2004 Rockwood/ON (S02-G2) NT020-04H	SC	0.094 0.054 0.053 0.053	75.2 kg/ha 101 108 104	1 3	0	Forage Hay	2.8, 3.2 (3.0) 15, 20 (18)	
USA, 2004 Bright/ON (Pioneer 90B51) NT021-04H	SC	0.094 0.053 0.054 0.055	75.2 kg/ha 105 113 107	1 3	0	Forage Hay	2.7, 3.4 (3.1) 14, 16 (15)	

* Average in parentheses

FATE OF RESIDUES IN STORAGE AND PROCESSING

In Processing

The Meeting has received information on the fate of imidacloprid residues during the processing of plum, olive, soya bean seeds and tea. Processing factors have been calculated for imidacloprid residues in olive, soya bean seeds and tea.

The processing trials for cherry and peach were submitted in 2002.

Plum

The trial was conducted in the USA on plums according to the US GAP (Dorschner, 2002: 111044). The 192 g/L SC formulation was applied 5 times at the rate of 0.11 kg ai/ha with an application interval of 8-12 days. The plums were taken to a commercial drier and dried for 25.5 hours. The total residue of imidacloprid were quantified with method 102624-R1 (based on the method 00200) at an LOQ of 0.05 mg/kg.

Table 24 Imidacloprid residues in processed commodities of plum from supervised trials

country, year (variety)	Application			DALA Days	Commodity	Residues, mg/kg	
	kg ai/ha	water, L/ha	no.			Total	
						mg/kg	PF
USA, 1999 Kerman/CA (French prunes) 99-CA79	0.11	1395-1430	5	6	Fruit (RAC) Dried (prunes)	0.30, 0.41 mean 0.36 1.0, 1.1 mean 1.1	 3.1

Portion analysed: Fruit without pit and stem

Olive

Processing studies for olive were conducted in Germany to determine the concentration of residues of imidacloprid in/on olive fruits and processing products of olive (Schöning and Eberhardt, 2002: RA-3034/01, Schoening, *et al*., 2003: RA-3155/02). The SL formulation containing 200 g/L of imidacloprid was sprayed twice to olive trees with an application rate of 0.10–0.11 kg ai/ha, corresponding to a concentration of 0.013 kg ai/hL and a spray volume of 800–904 L/ha. The olives were harvested after two spray applications of the SL formulation in Spain, Italy, Greece and Portugal (RA-2034/01 and RA-2155/02). Samples in 2001 were taken from the treated and the control plots on day 6 or 7 after the last treatment, while in 2002, at day 28 or 30. The samples from the treated plots were processed. Imidacloprid and the total residue of imidacloprid were analysed according to methods 00300/E007, 00300/E010 and 00834. The LOQ was 0.01 mg/kg for imidacloprid. For the total residue of imidacloprid the LOQ was 0.2 mg/kg for press cake and 0.05 mg/kg for all other sample materials.

Preparation of washed olives, washing water, press cake, separation water and crude oil

The olives were washed in standing water, one part of the olives was weighed and stored deep-frozen. The remaining part of the washed olives was crushed into olive pulp using a cutter. After addition of NaCl (1%, w/w), the olive pulp was pressed to obtain press cake and a water/oil emulsion. A sample of press cake was taken. The water/oil emulsion was separated into crude oil and separation water using a centrifuge; both fractions were taken for analysis.

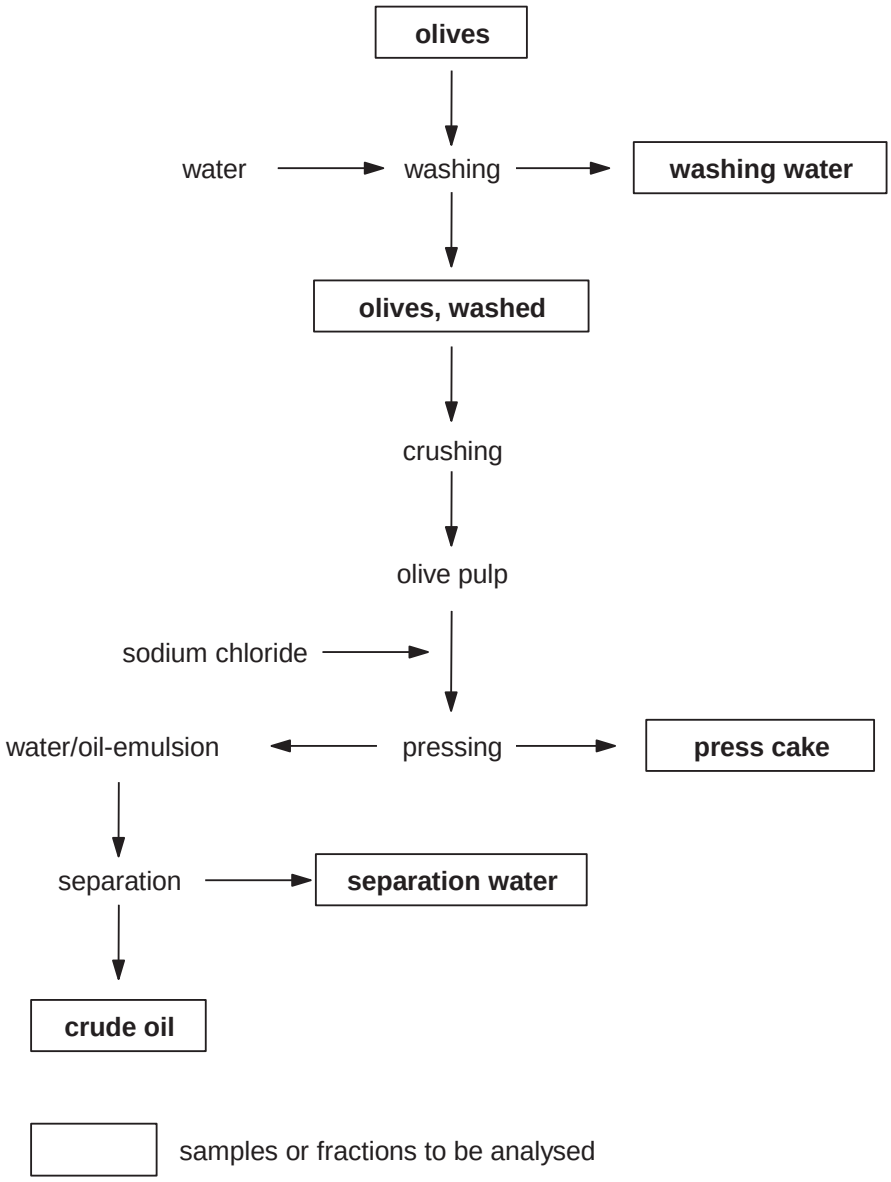

Figure 1 Flow chart of the preparation of washed olive, washing water, press cake, separation water and crude oil

Preparation of refined oil

The crude oil was preclarified by heating, removal of precipitated compounds and one part of the oil was taken for analysis. The remaining part of the preclarified oil was neutralized. After neutralisation, one part of the neutralised crude oil was taken for analysis.

The subsequent processes (bleaching, filtration and steaming) were all carried out in a vacuum. A sample of refined oil was taken for analysis

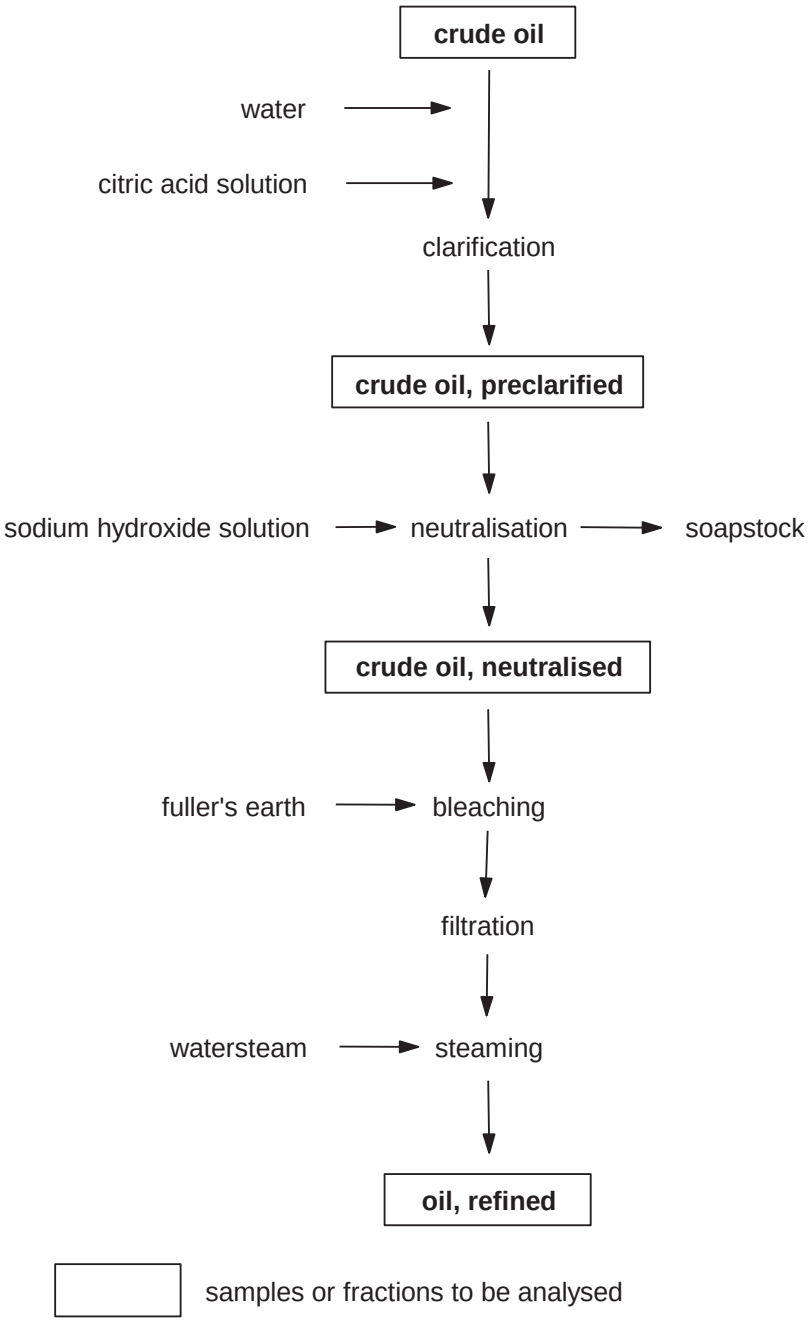

Figure 2 Flow chart of the preparation of refined oil

Table 25 Imidacloprid residues in processed commodities of olive from supervised trials

country, year (variety)	Application				DALA Days	Commodity	Residues, mg/kg			
	kg ai/ha	kg ai/hL	water, L/ha	no.			Parent		Total	
							mg/kg	PF	mg/kg	PF
Spain, 2001 (Vera) R2001 0090/6	0.11 0.10	0.013 0.013	904 800	2	6	Fruit (RAC) Fruit, washed Press cake Crude oil Washing water Separation water	0.09 0.048 0.13 0.01 < 0.01 0.04	 0.53 1.4 0.1 < 0.1 0.44	0.26 0.18 0.21 < 0.05 < 0.05 0.13	 0.69 0.81 < 0.1 9 < 0.1 9 0.50

country, year (variety)	Application				DALA Days	Commodity	Residues, mg/kg			
	kg ai/ha	kg ai/hL	water, L/ha	no.			Parent		Total	
							mg/kg	PF	mg/kg	PF
Italy, 2001 (Nocellara Etnea) R2001 0091/4	0.10 0.10	0.013 0.013	800 800	2	7	Fruit (RAC) Fruit, washed Press cake Crude oil Crude oil, preclarified Crude oil, neutralized Refined oil Washing water Separation water	0.04 0.03 0.04 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	 0.75 1.0 < 0.25 < 0.25 < 0.25 < 0.25 < 0.25 0.25	0.14 0.09 < 0.2 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	 0.64 <1.4 < 0.3 6 < 0.3 6 < 0.3 6 < 0.3 6 < 0.3 6
Portugal, 2001 (Picual) R2001 0092/2	0.10 0.10	0.013 0.013	800 800	2	7	Fruit (RAC) Fruit, washed Press cake Crude oil Crude oil, preclarified Crude oil, neutralized Refined oil Washing water Separation water	0.11 0.10 0.14 0.02 0.01 < 0.01 < 0.01 < 0.01 0.06	 0.91 1.3 0.18 0.091 < 0.09 < 0.09 < 0.09 0.55	0.22 0.19 0.24 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 0.08	 0.86 1.1 < 0.2 3 < 0.2 3 < 0.2 3 < 0.2 3 0.36
Portugal, 2002 (Cobrancosa) R2002 0697/6	0.10 0.10	0.013 0.013	800 800	2	28	Fruit (RAC) Fruit, washed Press cake Crude oil Crude oil, preclarified Crude oil, neutralized Refined oil Washing water Separation water	0.02 0.03 0.04 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.02	 1.5 2.0 < 0.5 < 0.5 < 0.5 < 0.5 < 0.5 1.0	0.05 < 0.05 0.06 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 0.05	 <1.0 1.2 <1.0 <1.0 <1.0 <1.0 <1.0 1.0
Greece, 2002 (Megaritiki) R2002 0698/4	0.10 0.10	0.013 0.013	800 800	2	30	Fruit (RAC) Fruit, washed Press cake Crude oil Crude oil, preclarified Crude oil, neutralized Refined oil Washing water Separation water	0.18 0.14 0.18 0.03 0.02 < 0.01 < 0.01 < 0.01 0.12	 0.78 1.0 0.17 0.11 < 0.06 < 0.06 < 0.06 0.67	0.76 0.67 0.62 0.09 0.07 < 0.05 < 0.05 < 0.05 0.77	 0.88 0.82 0.12 0.092 < 0.0 7 < 0.0 7 < 0.0 7 1.0

Soya bean seeds

A field trial was conducted to measure the magnitude of imidacloprid residue on soya beans treated with three foliar applications of the SC formulation containing 480 g/L of imidacloprid at a target rate of 0.263 kg ai/ha/application with 7 days between applications. Each application was made at a concentrated spray volume, 160–170 L/ha. The treatment rate is equivalent to a 5× the maximum recommended label use rate on soya beans. Single control and treated samples of soya bean seed were collected at normal commercial harvest (BBCH 89), corresponding to a 20-day PHI. Soya bean seed was processed into the commodities of meal, hulls, refined oil, and defatted flour. Aspirated grain

fractions were also collected. Processing was performed using procedures which simulated commercial processing practices (Krolski, 2006: RANTY003).

The total imidacloprid residue was quantitated as 6-chloronicotinic acid (6-CNA) by HPLC-MS/MS using isotopically labeled internal standards (method NT-001-P04-01). Method validation and concurrent recoveries were performed to demonstrate acceptable method performance. The LOQ for the total residue of imidacloprid was 0.05 mg/kg in soya bean seed, 0.10 mg/kg in soya bean refined oil, 0.20 mg/kg in soya bean meal, hulls, and defatted flour, and 30 mg/kg in soya bean aspirated grain fractions.

Preparation of aspirated grain fractions

After determining the moisture content of soya bean (RAC), the samples were dried to a moisture content of 10–13%. The samples were then placed in a dust generation room and moved in the system. Aspiration was used to remove light impurities. The light impurities were classified by sieving.

Preparation of hull, meal, defatted flour and refined oil

Soya beans were fed to a disc mill to crack the hull and liberate the kernel. After hulling, the material was passed through an aspirator to separate hull and kernel. The kernel material was flaked and heated. After expansion, the collets were dried and promptly taken for solvent extraction. The material was washed several times with hexane. Then the defatted flakes were ground and screened to produce defatted flour and the crude oil was heated for hexane removal and afterwards alkali refined. The refined oil was bleached and deodorised.

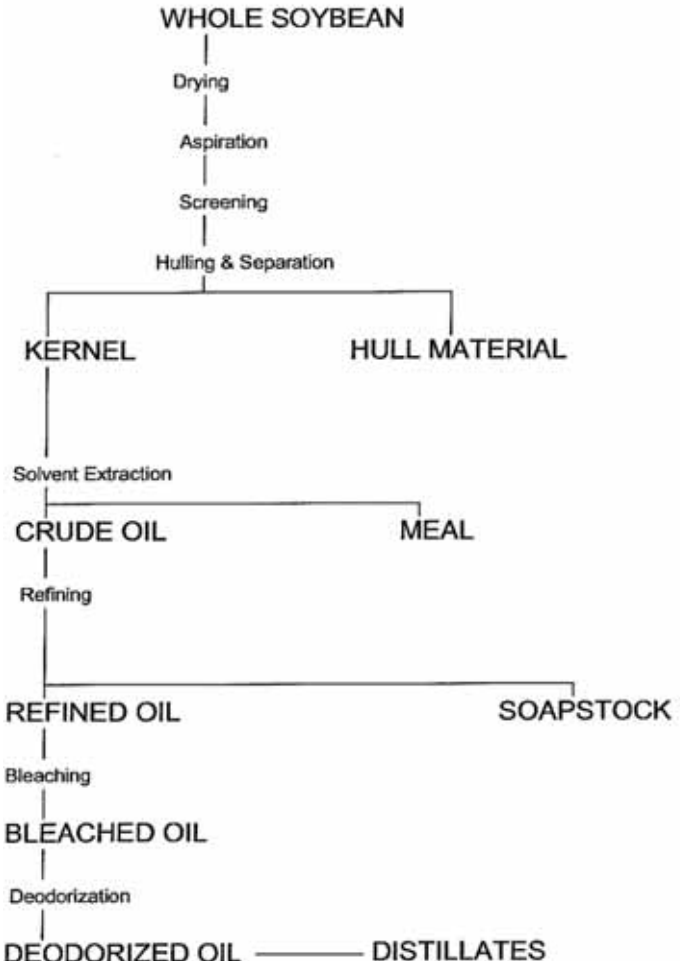

Figure 3 Flow chart for soya bean processing

Table 26 Imidacloprid residues in processed commodities of soya bean seeds from supervised trials

country, year (variety)	Application				DALA Days	Commodity	Total imidacloprid Residues, mg/kg	
	kg ai/ha	water, L/ha	GS (BBCH)	no.			mg/kg	PF
USA, 2004	0.263	160	89	3	20	seed	0.42	
Leland/MS	0.263	170				meal	0.36	0.86
(Pioneer 9492PR)	0.263	162				hulls	0.31	0.72
NT022-04P						refined oil	< 0.10	< 0.24
						defatted flour	0.34	0.80
						aspirated grain fractions	68	160

Tea

Two processing trials were performed in southern India with the imidacloprid 700 g/kg WG formulation. The formulation was applied once at a triple rate of 1.2 kg ai/ha. Tea shoots (two to three leaves and a bud) harvested 7 days after the application were used to manufacture green and black tea. Green and black tea was further processed into infusion using household practices whereas the preparation of instant tea simulated the industrial practice (Manikandan, 2014: RANTN021).

Preparation of infusion

100 g of green or black tea were infused into 5 L of boiling water for approximately 10 min. Infusion solution (liquid part) was separated with a sieve from wastes (infused tea). Wastes were weighed and discarded.

Preparation of instant tea

Two 0.5 L infusion solution subspecimens were collected and deep-frozen (below -18 °C). The Brix degree of the infusion solution was measured. The dry matter of solution determined by its Brix degree was increased with an addition of food additive to obtain between 8 to 9%. The pH of this preparation was measured and corrected with an addition of citric acid to obtain between a pH of 3.0 to 3.2. Afterwards the solution was placed in a vacuum chamber, where the water frozen in the solution was evaporated through sublimation.

All samples were analysed for imidacloprid parent compound and its metabolites and the total residue of imidacloprid according to method 01389. The LOQ was 0.01 mg/kg for imidacloprid in processed commodities (green and black tea infusion and instant green and black tea). The LOQ of the total residue of imidacloprid was 0.05 mg/kg in all sample materials.

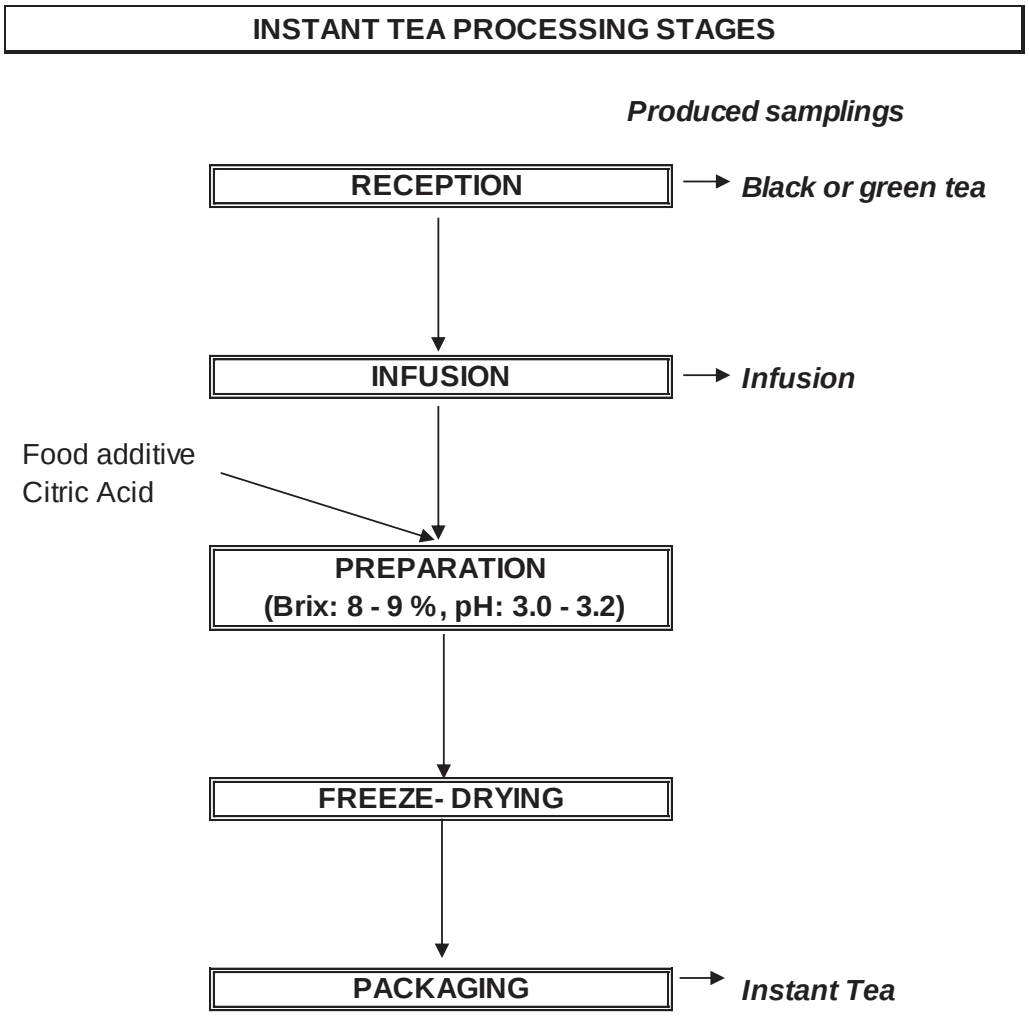

Figure 4 Flow chart for tea processing

Table 27 Imidacloprid residues in processed commodities of tea from supervised trials

country, year (variety)	Application				DALA Days	Commodity	Residues, mg/kg			
							Parent		Total	
	kg ai/ha	kg ai/hL	L/ha	no.			mg/kg	PF	mg/kg	PF
India, 2012 Coonoor (Assam Jat/ UPASI-9) S3	1.2	0.27	450	1	7	Green tea	16		34	
						Infusion green tea	0.41	0.026	0.81	0.024
						Instant green tea	3.6	0.23	8.0	0.24
						Black tea	14		34	
						Infusion black tea	0.34	0.024	0.57	0.017
						Instant black tea	3.0	0.21	6.4	0.19
India, 2013 Valparai (Assam Jat/ Seedling tea) S4	1.2	0.27	450	1	7	Green tea	4.8		12	
						Infusion green tea	0.12	0.025	0.30	0.025
						Instant green tea	1.1	0.23	3.0	0.25
						Black tea	4.5		13	
						Infusion black tea	0.13	0.029	0.30	0.023
						Instant black tea	1.3	0.29	3.6	0.28

APPRAISAL

Imidacloprid is a systemic insecticide which has been used widely in many crops for years. It was first evaluated by JMPR in 2001 (T) and 2002 (R). An ADI of 0–0.06 mg/kg bw and an ARfD of

0.4 mg/kg bw were established. The compound was evaluated for residues in 2006, 2008 and 2012. In 2002 the Meeting agreed that the residue definition for compliance with MRLs and for estimation of dietary intake for plant and animal commodities should be the sum of imidacloprid and its metabolites containing the 6-chloropyridinyl moiety, expressed as imidacloprid. It was listed by the Forty-sixth Session of CCPR (2014) for the evaluation of 2015 JMPR for additional MRLs.

The residue studies were submitted by the manufacturer and member countries for additional MRLs for stone fruit, olive, curly kale, soya bean, tea, goji berry (China) and basil (Thailand).

Methods of analysis

The Meeting received information on analytical methods used for the determination of imidacloprid residues in samples derived from supervised trials on olive, kale and soya bean (dry). Samples were fortified with imidacloprid and its metabolites desnitro-imidacloprid and 6-chloronicotinic acid. Imadacloprid and all metabolites containing 6-chloropyridinyl moiety were oxidised with alkaline $KMnO_4$ to yield 6-chloronicotinic acid. The 6-chloronicotinic acid was extracted from the aqueous solution using *tert*-butylmethylether (MTBE) and analysed by HPLC-MS/MS. The LOQ was 0.05 mg/kg (expressed in parent equivalents) for the commodities mentioned above.

The analytical method was developed for the determination of residues of imidacloprid, its 2 metabolites 5-hydroxy imidacloprid and olefin imidacloprid, and for the total residue of imidacloprid determined as 6-chloronicotinic acid in tea. Imidacloprid and its metabolites were extracted from tea (green tea and black tea) with methanol/water (3/1, v/v). For the individual analytes, an aliquot of the extracts was cleaned-up with liquid/liquid SPE. For the common moiety analysis, an aliquot of the extracts was made by alkaline oxidation under reflux and liquid/liquid partition. Final extracts of both branches were subjected to reversed phase HPLC-MS/MS. The LOQ (expressed as imidacloprid equivalents) for the total residue of imidacloprid was 0.05 mg/kg.

The Meeting received information on the analytical method for the determination of imidacloprid residues in fresh and dried goji berries. Imidacloprid was extracted from goji berries with acetonitrile. After adding sodium chloride, an aliquot was concentrated and purified by solid phase extraction using amino cartridges. Imidacloprid residues were analysed by reversed-phase HPLC-UV (275 nm). The LOQ was 0.02 mg/kg for both matrices.

The Meeting received data on the storage stability of imidacloprid, 5-hydroxy imidacloprid and olefin imidacloprid in various plant matrices. Storage stability results indicated that residues of imidacloprid and its metabolites 5-hydroxy imidacloprid and olefin imidacloprid were stable for at least 36 months under freezer conditions at about -18 °C or below in wheat (grain), orange (fruit), tomato (fruit), bean (seed) and rape seed.

Residues resulting from supervised residue trials on crops

The Meeting received supervised trial data for the foliar application of imidacloprid on cherries, plum, peach, olive, kale, goji berry, soya bean, basil and tea. Residue trial data was made available from Canada, China, India, Southern Europe, Thailand and the USA.

Labels were available from China, Italy, Japan, Spain, Thailand and the USA describing the registered uses of imidacloprid.

Stone fruits

The 2002 JMPR evaluated residue supervised trials data for imidacloprid on sweet cherries, plums, peaches and nectarines conducted in southern Europe. New residue data were submitted to the current Meeting for cherries, plums and peaches.

Cherries

Data were available from supervised trials on cherries in the USA.

The GAP of the USA is foliar applications of 0.056-0.11 kg ai/ha at a maximum rate of 0.56 kg ai/ha per year with a PHI of 7 days.

Imidacloprid residues in whole fruits of cherries from independent trials in the USA matching GAP were (n=8): 0.24, 0.36, 0.41, 0.53, 0.57, 0.63, 1.4 and 2.5 mg/kg.

Based on the residues for cherries from trials in the USA, the Meeting estimated a maximum residue level of 4 mg/kg, an STMR value of 0.55 mg/kg and an HR value of 2.5 mg/kg for the cherries subgroup. The Meeting withdrew the previous recommendation for Cherry, Sweet.

Plums

Data were available from supervised trials on plums in the USA.

The GAP of the USA is foliar applications of 0.056–0.11 kg ai/ha at a maximum rate of 0.56 kg ai/ha per year with a PHI of 7 days.

Imidacloprid residues in fruits without stone of plums from independent trials in the USA matching GAP were (n=8): 0.082, 0.095, 0.15, 0.22, 0.34, 0.39, 0.42 and 0.67 mg/kg.

Since the weight of stone does not significantly affect the residue level in plum fruits, the Meeting agreed to use the residues in the edible portion of plums to estimate a maximum residue level.

Based on the residues in the edible portion of plums from trials in the USA, the Meeting estimated a maximum residue level of 1.5 mg/kg, an STMR value of 0.28 mg/kg and an HR value of 0.70 mg/kg (based on a highest residue of duplicate samples) for imidacloprid in the plums (including prunes) subgroup, to replace the previous recommendation for plums (including prunes).

Peaches

Data were available from supervised trials on peaches in the USA.

The GAP in the USA is foliar applications of 0.056-0.11 kg ai/ha at a maximum rate of 0.34 kg ai/ha per year with a PHI of 0 days.

Imidacloprid residues in whole fruit peaches from trials in the USA, matching GAP, were (n=8): 0.10, 0.25, 0.28, 0.34, 0.37, 0.38 (2) and 0.77 mg/kg.

Based on the residues for peaches from trials in the USA, the Meeting estimated a maximum residue level of 1.5 mg/kg, an STMR value of 0.355 mg/kg and an HR value of 0.77 mg/kg for imidacloprid in the Peaches (including nectarine and apricots) subgroup. The Meeting withdrew the previous recommendations for peach, nectarine and apricot.

Olives

Data were available from supervised trials on olives from Southern Europe.

The GAP of Italy is for a foliar application at a maximum concentration of 0.013 kg ai/hL, with a PHI of 28 days. Imidacloprid residues in olives, from trials in Southern Europe matching GAP, were (n=8): 0.12, 0.23, 0.26, 0.28, 0.43, 0.61, 0.77 and 0.81 mg/kg.

The GAP of Spain is a maximum of four foliar applications at a maximum rate of 0.02 kg ai/ha with a PHI of 7 days. Imidacloprid residues in olive from independent trials in Southern Europe matching GAP were (n=8): < 0.05, 0.11, 0.14, 0.22, 0.49, 0.63, 0.71 and 1.1 mg/kg.

Based on the residues for olive from trials with the highest residue levels matching Spanish GAP, the Meeting estimated a maximum residue level of 2 mg/kg, an STMR value of 0.355 mg/kg and an HR value of 1.1 mg/kg for imidacloprid in olives.

Kale

Data were available from supervised trials on curly kale in Italy and Spain.

The GAP of Italy is a maximum two foliar applications at a maximum rate of 0.094 kg ai/ha with a PHI of 7 days.

Imidacloprid residues in curly kale from independent trials in Italy and Spain matching GAP were (n=4): 1.0, 1.1, 1.5 and 2.0mg/kg.

Based on the residues for curly kale from trials in Italy and Spain, the Meeting estimated a maximum residue level of 5 mg/kg, an STMR value of 1.3 mg/kg and an HR value of 2.0 mg/kg for imidacloprid in kale.

Goji berry

The GAP of China is a maximum three foliar applications at a maximum concentration of 0.005 kg ai/hL with a PHI of 3 days. Six trials were conducted on goji berries in China in 2010 with foliar treatment by 3 × 0.005 kg ai/hL. Samples were taken at 1–21 days after the last treatment. The data were submitted as separate trials but the analyte was parent imidacloprid only.

As the residue definition of imidacloprid is the sum of imidacloprid and its metabolites containing the 6-chloropyridinyl moiety, expressed as imidacloprid, the Meeting could not estimate a maximum residue level for imidacloprid in goji berry.

Soya bean (dry)

Data were available from supervised trials on soya bean in the USA.

The GAP on soya bean of the USA is seed treatment at a maximum rate of 0.125 kg ai/100 kg seed, and/or maximum three foliar applications at a maximum rate of 0.053 kg ai/ha with a PHI of 21 days.

Imidacloprid residues in soya bean seeds from independent trials in the USA matching GAP were (n=20): 0.035, 0.050, 0.052 (2), 0.094, 0.11, 0.18, 0.19, 0.21, 0.38 (2), 0.43, 0.48, 0.61, 0.62, 0.63, 0.67, 0.73 and 1.5 (2) mg/kg.

Based on the residues for soya bean from trials in the USA, the Meeting estimated a maximum residue level of 3 mg/kg and an STMR value of 0.38 mg/kg for imidacloprid in soya bean seed (dry).

Basil

Data were available from supervised trials on basil in Thailand.

The GAP of Thailand is foliar applications when the crop is infested at a maximum concentration of 0.042 kg ai/hL with a PHI of 7 days.

Imidacloprid residues in fresh basil from independent trials in Thailand matching GAP were (n=4): 4.3, 4.9, 5.1 and 6.5 mg/kg.

Based on the residues for basil from trials in Thailand, the Meeting estimated a maximum residue level of 20 mg/kg, an STMR value of 5.0 mg/kg and an HR value of 7.3 mg/kg (based on a highest residue of replicate samples) for imidacloprid in basil.

Tea, Green, Black

Data were available from supervised trials on tea in India.

The GAP on tea of Japan is a foliar application at a maximum concentration of 0.01 kg ai/hL with a PHI of 7 days.

Imidacloprid residues in green tea from independent trials in India matching Japanese GAP were (n=8): 2.9 (2), 3.0, 5.5, 7.3, 11, 12 and 23 mg/kg.

Imidacloprid residues in black tea from independent trials in India matching Japanese GAP were (n=8): 2.7 (2), 3.3, 5.1 (2), 12 (2) and 28 mg/kg.

The samples of green tea and black tea were produced from fresh tea leaves harvested 7 days after application at the same plot.

The Meeting recognized that the residue populations from trials on green tea and black tea were not different according to statistical tests (Mann-Whitney U-test). The Meeting agreed to use highest residues of green tea and black tea samples in each trial to estimate a maximum residue level for tea, green and black.

The residues in green tea and black tea were in rank order (n=8): 2.9, 3.0, 3.3, 5.5, 7.3, 12 (2) and 28 mg/kg.

Based on the residues for green tea and black tea from trials in India, the Meeting estimated a maximum residue level of 50 mg/kg and an STMR value of 6.4 mg/kg for imidacloprid in tea, green and black.

Animal feedstuffs

Soya bean fodder and forage (green)

Data were available from supervised trials on soya bean in the USA.

The GAP on soya bean in the USA isa seed treatment at a maximum rate of 0.125 kg ai/100 kg seed, and/or maximum three foliar applications at a maximum rate of 0.053 kg ai/ha for forage grass for hay.

Imidacloprid residues in soya bean forage from independent trials in the USA matching GAP were (n=21): 1.1, 1.6, 1.8, 2.1 (2), 2.4, 2.6, 2.7, 3.0, 3.1, 3.2, 3.5 (2), 3.8 (2), 3.9, 4.1, 4.2, 4.4, 4.6 and 6.5 mg/kg.

Based on the trials for soya bean forage from trials in the USA, the Meeting estimated a median residue value and a highest residue value for imidacloprid in soya bean forage of 3.2 and 6.5 mg/kg, respectively as received basis.

Imidacloprid residues in soya bean hay from independent trials in the USA matching GAP were (n=21): 4.0, 4.5, 5.7, 6.5, 7.5, 8.5, 9.1, 9.2, 9.4, 9.6, 9.9, 11, 13 (2), 14, 15 (2), 18, 21 (2) and 22 mg/kg.

Based on the residues in soya bean hay from trials in the USA, the Meeting estimated a median residue value of 9.9 mg/kg, a highest residue value of 22 mg/kg on an as received basis and after correction for an average 85% dry matter content, estimated a maximum residue level of 50 mg/kg for imidacloprid in soya bean hay.

Fate of residues during processing

Residues in processed commodities

The fate of imidacloprid residues has been examined in plum, olive, soya bean seeds and tea processing studies. Estimated processing factors and the derived STMR-Ps are summarized in the Table below.

Processing factors, STMR-P for food and feed

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors*	PF (Mean or best estimate)	RAC STMR (mg/kg)	STMR-P (mg/kg)	RAC HR (mg/kg)	HR-P (mg/kg)
Cherry	Canned fruit	< 0.56, < 0.56, < 0.63 < 0.63	< 0.60	0.55	< 0.33	2.5	<1.5
Plum	Dried (prunes)	3.1	3.1	0.28	0.87	0.70	2.2
Peach	Canned fruit	< 0.38	< 0.38	0.32	< 0.12	0.77	< 0.092
	Jam	< 0.38	< 0.38		< 0.12		
Olive	Crude oil	< 0.19, < 0.36, < 0.23, < 1.0, 0.12	0.12	0.36	0.04		
Soya bean seeds	Refined oil	< 0.24	< 0.24	0.38	< 0.09		

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors*	PF (Mean or best estimate)	RAC STMR (mg/kg)	STMR-P (mg/kg)	RAC HR (mg/kg)	HR-P (mg/kg)
	Meal	0.86	0.86		0.33		
	Aspirated grain fractions	160	160		61		
	Hulls	0.72	0.72		0.27		
Green tea	Infusion	0.024, 0.025	0.025	6.4	0.16		
	Instant	0.24, 0.25	0.25		1.6		
Black tea	Infusion	0.017, 0.023	0.02	6.4	0.13		
	Instant	0.19, 0.28	0.24		1.5		

* Each value represents a separate study. The factor is the ratio of the residue in processed commodity divided by the residue in the RAC.

The Meeting estimated a maximum residue level of 5 mg/kg (1.5 × 3.1 = 4.65 mg/kg) for dried plums.

Residue in animal commodities

The 2015 JMPR evaluated residues of imidacloprid in soya bean (dry), which is listed in the OECD feeding table. The Meeting noted that the estimation did not result in a significant change of the dietary burdens of farm animals. The previous recommendations of maximum residue level for animal commodities were maintained.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed in Annex 1 are suitable for estimating maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for plant and animal commodities (for compliance with the MRL and for estimation of dietary intake): *Sum of imidacloprid and its metabolites containing the 6-chloropyridinyl moiety, expressed as imidacloprid*

CCN	Commodity	Recommended Maximum residue level (mg/kg)		Recommended Maximum residue level (mg/kg)	HR or HR-P mg/kg
		New	Previous		
FS 0240	Apricot	W	0.5		
HH 0722	Basil	20		5.0	7.3
FS 0013	Cherries	4		0.55	2.5
FS 0244	Cherry, Sweet	W	0.5		
DF 0014	Prunes	5		0.87	2.2
VL 0480	Kale	5		1.3	2.0
FS 0247	Nectarine	W	0.5		
SO 0305	Olives for oil production	2		0.355	1.1
FS 0247	Peach	W	0.5		
FS 2001	Peaches (including nectarines and apricots)	1.5		0.355	0.77
FS 0014	Plums (including Prunes)	1.5	0.2	0.28	0.7
VD 0541	Soya bean (dry)	3		0.38	
AL 0541	Soya bean fodder	50		9.9	22
FT 0305	Table olives	2		0.355	1.1
DT 1114	Tea, Green, Black (black, fermented and dried)	50		6.4	
	Apricot, canned			0.12	0.092
	Apricot jam			0.12	
	Cherries, canned			0.33	1.5
	Nectarine, canned			0.12	0.092

CCN	Commodity	Recommended Maximum residue level (mg/kg)		Recommended Maximum residue level (mg/kg)	HR or HR-P mg/kg
		New	Previous		
	Nectarine, jam			0.12	
OC 0305	Olive oil, virgin oil			0.04	
	Peaches, canned			0.12	0.092
	Peaches, jam			0.12	
OR 0541	Soya bean oil, refined			0.09	
	Tea, infusion			0.16	
	Tea instant			1.6	
AL 1265	Soya bean forage (green)			3.2	6.5
	Soya bean asp gr fn[a]			61	
AB 0541	Soya bean hulls			0.27	
AB 1265	Soya bean meal			0.33	

[a] aspirated grain fractions

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of imidacloprid were calculated for the 17 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the 2002, 2006, 2008, 2012 and current Meeting (Annex 3). The ADI is 0–0.06 mg/kg bw and the calculated IEDIs were 2–5% of the maximum ADI (0.06 mg/kg bw). The Meeting concluded that the long-term intake of residues of imidacloprid, resulting from the uses considered by the current JMPR, were unlikely to present a public health concern.

Short-term intake

The International Estimated Short-Term Intakes (IESTI) of imidacloprid were calculated for food commodities and their processed commodities using HRs/HR-Ps or STMRs/STMR-Ps estimated by the current Meeting (Annex 4). The ARfD is 0.4 mg/kg bw and the calculated IESTIs were a maximum of 10% of the ARfD. The Meeting concluded that the short-term intake of residues of imidacloprid, when used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

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LAMBDA-CYHALOTHRIN (146)

First draft prepared by Makoto Irie, Ministry of Agriculture, Forestry and Fisheries, Tokyo, Japan

EXPLANATION

Lambda-cyhalothrin consists of two of the four enantiomers of cyhalothrin. It was first evaluated by JMPR in 1984 (T, R) and periodic re-evaluation conducted in 2007 (T) and 2008 (R). A group of ADI for cyhalothrin and lambda-cyhalothrin was established as 0–0.02 mg/kg bw and an ARfD was estimated at 0.02 mg/kg bw. In 2008 the Meeting agreed that the residue definition for compliance with MRLs and for estimation of dietary intake for plant and animal commodities should be cyhalothrin, sum of isomers. It was listed by the 46th Session of the CCPR (2014) for the evaluation by the 2015 JMPR for additional MRLs.

The residue studies were submitted by the manufacturer and member countries for additional MRLs for basil (Thailand) and coffee.

RESIDUE ANALYSIS

Analytical methods

The Meeting received information on the analytical method (POPIT MET.044 Rev.31) for the determination of residues of lambda-cyhalothrin in plant materials (Reigada, 2009).

Lambda-cyhalothrin is extracted from samples with acetone/hexane (1:1 v/v). For coffee, deionised water is added to achieve phase separation and the upper (organic) phase is removed and evaporated to dryness. The evaporated residue is diluted with hexane and purified with a silica SPE column. The solvent is evaporated and the residue is dissolved in the internal standard (dicyclohexyl phthalate) and quantification was achieved by GC-ECD.

The LOQ is 0.01 mg/kg for lambda-cyhalothrin in coffee beans.

Table 1 Recovery results obtained for the determination of lambda-cyhalothin from coffee beans

Commodity	Fortification level (mg/kg)	N	Recovery range (%)	Mean recovery (%)	% RSD
Coffee beans	0.01 0.1	7 5	79–97 83–110	89 100	7.6 13

Stability of pesticide residues in stored analytical samples

Information on the freezer storage stability of lambda-cyhalothrin residues in plant commodities was submitted to the 2008 JMPR. Lambda-cyhalothrin residues were stable in the commodities apple and cabbage for 16 months and were stable for 26 months in peach, cabbage, pea, potato, rape seeds, wheat grain, sugar beet roots and cotton seed.

The periods of freezer storage between sampling and analysis for the residue trials of coffee beans submitted to the current Meeting were covered by the period of the freezer storage stability studies.

USE PATTERN

The Meeting received labels from Brazil and Thailand. The authorised uses relevant to the supervised residue trials data submitted to the current Meeting are summarized in Table 2.

Table 2 Registered uses of lambda-cyhalothrin relevant to the residue evaluation by the current Meeting

Crop	Country	Formulation		Application					PHI, days
		Type	Conc. of lambda-cyhalothin	Method	kg ai/ha	kg ai/hL	L/ha	No. max	
Seed for beverages and sweets									
Coffee	Brazil	CS	50 g/L	Foliar	0.005		100–150	2	1 (45 days interval)
Herbs									
Basil	Thailand	CS	25 g/L	Foliar		0.0025		[a]	7

[a] Apply when infested

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on lambda-cyhalothrin supervised field trials for the following crops.

Group	Commodity	Table
Seed for beverages and sweets	Coffee beans	3
Herbs	Basil	4

The lambda-cyhalothrin formulation was applied by foliar treatment. Each of the field trial sites generally consisted of an untreated control plot and treated plot. Residues, 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.

Seed for beverages and sweets

Coffee beans

Four residue field trials for coffee were carried out in Brazil (Marconi, 2009: M09068). Coffee plants were treated twice with the 50 g/L CS formulation at a rate of 0.005 kg ai/ha. The first application was done 50 days before harvest time followed by one application 45 days after the first application. The water volume used was 250 L/ha.

Coffee cherries were collected 0, 1, 7, 14 and 21 days after the last application. After collection, coffee cherries were placed in the sun to dry and coffee beans were separated from the shells with electric machinery. Residues of lambda-cyhalothrin in green coffee beans were determined according to the method POPIT MET.044 Rev31. The LOQ was 0.01 mg/kg.

Table 3 Lambda-cyhalothrin residues on coffee beans from supervised trials in Brazil

Coffee country, year (variety)	Application						DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Growth stage [a]		no.			
				Appli.	Coll.				
GAP, Brazil	CS	0.005	100–150			2	1		
Brazil, 2009 Monte Carmelo/MG (Mundo Novo) M09068-JJB1	SC	0.005	250	79 87	87 87 88 89 89	2	0 1 7 14 21	< 0.01 <u>< 0.01</u> < 0.01 < 0.01 < 0.01	M09068 Marconi, 2009 Sampling to analysis: 127–159 days
Brazil, 2009 Indianópolis/MG (Catuaí) M09068-JJB2	CS	0.005	250	79 87	87 87 88 89 89	2	0 1 7 14 21	< 0.01 <u>< 0.01</u> < 0.01 < 0.01 < 0.01	
Brazil, 2009 Careaçú/MG (Catuaí) M09068-JJB3	CS	0.005	250	85 88	88 88 88 88 88	2	0 1 7 14 21	< 0.01 <u>< 0.01</u> < 0.01 < 0.01 < 0.01	
Brazil, 2009 Bandeirantes/PR (IAPAR 59) M09068-LZF	CS	0.005	250	81 89	89 89 89 89 89	2	0 1 7 14 21	< 0.01 <u>< 0.01</u> < 0.01 < 0.01 < 0.01	

Portion analysed: Beans

[a] Code of BBCH scale

Herbs

Basil

Four field residue trials were carried out with lambda-cyhalothrin on <u>basil</u> in Thailand using the 25 g/L CS formulation. The basil plants were treated with two foliar applications at a target concentration of 0.025 kg ai/hL. The application interval was 6 or 7 days. The residue analysis was performed within 24 hours after sample collection.

The on-line method applied for the determination of lambda-cyhalothrin residues was based on extraction with a mixture of acetone, dichloromethane and sodium chloride water solution. The concentrated extract is cleaned up on silica gel column and detection with GC-ECD (Steinwandter, 1985). The recoveries for lambda-cyhalothrin ranged from 86–114% at fortification level of 0.02 mg/kg, 85–105% at 0.05 mg/kg, 94–110% at 0.1 mg/kg and 91–98% at 1.0 mg/kg. The LOQ for lambda-cyhalothrin was 0.01 mg/kg.

Table 4 Lambda-cyhalothrin residues on basil from supervised trials in Thailand

Basil country, year (variety)	Application				DALA Days	Residues, mg/kg*	Ref.
	Form	kg ai/ha	kg ai/hL	no.			
GAP, Thailand	CS		0.0025		7		
Thailand, 2011 Nakhon Pathom (Sweet basil)	CS	0.019	0.0025	2	0 1 3 5 8 10 14	1.8, 2.0, 2.5 mean 2.1 1.4, 1.9, 2.5 mean 1.9 0.30, 0.30, 0.39 mean 0.33 0.14, 0.19, 0.20 mean 0.18 0.07, 0.08, 0.09 mean <u>0.08</u> 0.02, 0.03, 0.03 mean 0.03 0.01, 0.01, 0.02 mean 0.01	LCY-BS-01 Palakul, 2011
Thailand, 2011 Dunneonsaduak, Ratchaburi (Sweet basil)	CS	0.019	0.0025	2	0 1 3 5	1.2, 1.3, 1.3 mean 1.3 0.67, 0.71, 0.85 mean 0.74 0.34, 0.36, 0.45 mean 0.38 0.27, 0.29, 0.31 mean 0.29	LCY-BS-02 Phaikaew, 2011

Basil country, year (variety)	Application				DALA Days	Residues, mg/kg*	Ref.
	Form	kg ai/ha	kg ai/hL	no.			
					7 8 10 14	0.20, 0.20, 0.21 mean 0.20 0.13, 0.14, 0.14 mean 0.14 0.04, 0.06, 0.06 mean 0.05 0.01, 0.01, 0.02 mean 0.01	
Thailand, 2011 Ratchaburi (Holly basil)	CS	0.017	0.0023	2	0 1 3 5 7 8 10 14	3.6, 4.0, 4.0 mean 3.9 0.93, 0.99, 1.1 mean 1.0 0.32, 0.53, 0.62 mean 0.49 0.17, 0.21, 0.35 mean 0.24 0.16, 0.16, 0.18 mean 0.17 0.08, 0.09, 0.10 mean 0.09 0.06, 0.06, 0.08 mean 0.07 0.03, 0.04, 0.04 mean 0.04	LCY-BS-03 Akcaboot, 2011
Thailand, 2014 Nakornprathom (Holly basil)	CS	0.019	0.0025	2	0 1 3 5 7 8 10 14	2.4, 2.8, 3.8 mean 3.0 2.5, 2.6, 2.8 mean 2.7 0.93, 0.96, 1.2 mean 1.0 0.49, 0.72, 0.75 mean 0.65 0.34, 0.38, 0.40 mean 0.37 0.30, 0.34, 0.36 mean 0.33 0.16, 0.21, 0.23 mean 0.20 0.10, 0.11, 0.11 mean 0.11	LCY-BS-04 Buasri, 2011

Portion analysed: whole commodity

APPRAISAL

Lambda-cyhalothrin consists of two of the four enantiomers of cyhalothrin. It was first evaluated by JMPR in 1984 (T, R) and subsequently under the periodic re-evaluation programme in 2007 (T) and 2008 (R). A group ADI for cyhalothrin and lambda-cyhalothrin was established at 0–0.02 mg/kg bw and a group ARfD, 0.02 mg/kg bw. In 2008 the Meeting agreed that the residue definition for compliance with the MRL and for estimation of dietary intake for plant and animal commodities should be cyhalothrin, sum of isomers. It was listed by the Forty-sixth Session of the CCPR (2014) for the evaluation by the 2015 JMPR for additional MRLs.

The residue studies were submitted by the manufacturer and member countries for additional MRLs for basil (Thailand) and coffee.

Methods of analysis

The Meeting received new information on the analytical method (POPIT MET.044 Rev.31) for the determination of residues of lambda-cyhalothrin in plant materials including coffee beans. Lambda-cyhalothrin is extracted from samples with acetone/hexane (1:1 v/v). For coffee beans, deionised water is added to achieve phase separation and the upper (organic) phase is removed and evaporated to dryness. The evaporated residue is diluted with hexane and purified with a silica SPE column. The solvent is evaporated and the residue is dissolved in the internal standard (dicyclohexyl phthalate) and quantification is achieved by GC-ECD. The LOQ is 0.01 mg/kg for lambda-cyhalothrin in coffee beans.

For the determination of lambda-cyhalothrin in basil, a method[2] available from the scientific literature was used. The recoveries for lambda-cyhalothrin in basil tested concurrently with the analysis of trial samples ranged between 85 and 114%. The LOQ is 0.01 mg/kg for lambda-cyhalothrin in basil.

[2] H. Steinwandter, 1985, Universal 5-min on-line method for extracting and isolating pesticide residues and industrial chemicals

Residues resulting from supervised residue trials on crops

The Meeting received supervised trial data for the foliar application of lambda-cyhalothrin on coffee and basil. Residue trial data was made available from Brazil and Thailand.

Labels were available from Brazil and Thailand describing the registered uses of lambda-cyhalothrin.

Coffee beans

Data were available from supervised trials on coffee in Brazil.

The GAP of Brazil is maximum two foliar applications at a maximum rate of 0.005 kg ai/ha with a PHI of 1 day.

Lambda-cyhalothrin residues in green coffee beans from independent trials in Brazil matching GAP were (n=4): < 0.01 (4) mg/kg.

Based on the residues for coffee beans from trials in Brazil, the Meeting estimated a maximum residue level of 0.01 (*) mg/kg and an STMR value of 0.01 mg/kg for lambda-cyhalothrin in coffee beans.

Basil

Data were available from supervised trials on basil in Thailand.

The GAP of Thailand is foliar applications when crop is infested at a maximum concentration of 0.0025 kg ai/hL with a PHI of 7 days.

Lambda-cyhalothrin residues in basil from independent trials in Thailand matching GAP were (n=4): 0.08, 0.17, 0.20 and 0.37 mg/kg.

Based on the residues for basil from trials in Thailand, the Meeting estimated a maximum residue level of 0.7 mg/kg, an STMR value of 0.19 mg/kg and an HR value of 0.40 (based on a highest residue of replicate samples) mg/kg for lambda-cyhalothrin in basil.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels assessed were suitable for estimating maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for plant and animal commodities (for compliance with the MRL and for estimation of dietary intake): *Cyhalothrin, sum of isomers*

The residue is fat soluble.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
HH 0722	Basil	0.7		0.19	0.40
SB 0716	Coffee beans	0.01*		0.01	

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of lambda-cyhalothrin were calculated for the 17 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the 2008 JMPR and the current Meeting (Annex 3). The ADI is 0-0.02 mg/kg bw and the calculated IEDIs were 2-9% of the maximum ADI (0.02 mg/kg bw). The Meeting concluded that the long-term intakes of residues of

lambda-cyhalothrin, arising from the uses considered by the current Meeting, are unlikely to present a public health concern.

Short-term intake

The International Estimated Short-Term Intakes (IESTI) of lambda-cyhalothrin were calculated for food commodities and their processed commodities using HRs/HR-Ps or STMRs/STMR-Ps estimated by the current Meeting (Annex 4). The ARfD is 0.02 mg/kg bw and the calculated IESTIs were a maximum of 2% of the ARfD. The Meeting concluded that the short-term intake of residues of lambda-cyhalothrin, when used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

REFERENCES

Code	Author	Year	Title, Institution, Report reference
POPIT MET.044.Rev31	Reigada, J	2009	Determination of Residues of Lambda-Cyhalothrin in Vegetable Samples through GC/μECD Syngenta Crop Protection AG, Basel, CH, POPIT MET.044.Rev31 Not GLP, not published Syngenta File No PP321_11675
M09068	Marconi, F & Terada, R	2009	Magnitude of Residues of Lambda-Cyhalothrin in coffee—Brazil, 2008–09 Syngenta Crop Protection Ag, Basel, CH, Syngenta Proteção de Cultivos Ltd.a, São Paulo, Brazil, M09068 GLP, not published Syngenta File No A12688B_10000
On-line method	Steinwandter	1985	Universal 5-min on-line method for extracting and isolating pesticide residues and industrial chemicals Published
LCY-BS-01	Somsamai Palakul	2011	Report on pesticide residue trial. Pesticide Research Group, Agricultural Production Science Research, Development Office, Department of Agriculture, Thailand. Trial No.: LCY-BS-01. Unpublished.
LCY-BS-02	Yongyuth Phaikaew	2011	Report on pesticide residue trial. Pesticide Research Group, Agricultural Production Science Research, Development Office, Department of Agriculture, Thailand. Trial No.: LCY-BS-02. Unpublished.
LCY-BS-03	Piyasak Akcaboot	2011	Report on pesticide residue trial. Pesticide Research Group, Agricultural Production Science Research, Development Office, Department of Agriculture, Thailand. Trial No.: LCY-BS-03. Unpublished.
LCY-BS-04	Wittaya Buasri	2011	Report on pesticide residue trial. Pesticide Research Group, Agricultural Production Science Research, Development Office, Department of Agriculture, Thailand. Trial No.: LCY-BS-04. Unpublished.

LINDANE (048)

The first draft was prepared by Professor Arpad Ambrus, Hungarian Food Chain Safety Office, Budapest, Hungary

EXPLANATION

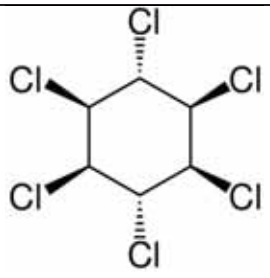
Lindane was first evaluated by the Joint Meeting in 1966 (T,R). It had been last re-evaluated within the periodic review programme in 2002 (T) and 2003 (R). The Meeting agreed that the definition of the residue for compliance with MRLs and for estimation of dietary intake should be: lindane, for both plant and animal commodities. The residue is fat-soluble.

Since lindane was currently listed in Annex A of the Stockholm Convention by which Parties must take measures to eliminate the production and use of such chemicals, and there was no information on existing national registrations for lindane uses, the 46th CCPR (2014) requested a periodic review in 2015 to convert the existing CXLs for sweet corn, cereals, eggs, pountry and meats into Codex EMRLs.

Lindane has no use for crop protection. According to the Stockholm Convention, as a specific exemption, it may be used as a human health pharmaceutical for control of head lice and scabies as second line treatment (decision SC-4/15 under the Stockholm Convention

Subsequently, monitoring data were submitted by EFSA for the period of 2009-2013, the GEMS Food programme (2000-2011) In addition, individual residue data were provided by the Netherlands, and summarized results from India and the USA.

IDENTITY

Common name	Lindane;(for material containing ≥99% gamma stereoisomer)
Chemical name	
IUPAC:	1α,2α,3β,4α,5α,6β-hexachlorocyclohexane (gamma stereoisomer)
CAS:	(1α,2α,3β,4α,5α,6β)-hexachlorocyclohexane (gamma stereoisomer)
Other names	Gamma-BHC; Gamma-HCH;
CAS number:	58-89-9 (for the gamma isomer)
CIPAC Code:	488
Molecular formula:	$C_6H_6Cl_6$
Molecular weight:	290.82984 g/mol
Structural formula:	

PHYSICAL AND CHEMICAL PROPERTIES

The physical and chemical properties, metabolism and environmental fate were evaluated by the 2003 JMPR as part of the periodic review programme.

METHODS OF RESIDUE ANALYSIS

Analytical methods

Lindane is typically analysed with multi residue procedures enabling detection of a large number of samples whose pesticide treatment history is usually unknown. In the screening procedure the emphasis is to detect residues which are around the legal limit; achieving the lowest detectable concentration is not of the primary goal.

No analytical methods were referenced in the submissions of monitoring data. The reported LOQ values varied to a large extent in case of individual commodities and among commodities. The reported ranges of LOQs, where available, are mentioned together with the results of monitoring data. If the LOQ exceeded the present CXL values, for the evaluation of data, the residues were taken as non-detected.

Similarly, no information was provided on the design of sampling programmes or on the size of samples collected. In view of the very large number of samples analysed, the potential deviation from the principles of random sampling or the size of samples do not affect the applicability of the data for estimation of EMRLs.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Residue data derived from the European monitoring programmes

For the period of 2009-2013, the European Food Safety Authority (EFSA) provided approximately 25000 monitoring results on lindane residues in unprocessed food products reported by EU Member States, as well as Norway and Iceland for the products for which currently Codex has established CXLs. The tested products were obtained from more than 60 different countries. However, the majority of the results (approximately 24,000 samples) refer to samples originating from the reporting countries. More specifically, the samples originated from Germany (5,261), the United Kingdom (2,508), Ireland (2,180), Denmark (,1856), France (1,738), Spain (1,385), Romania (1,336) and Poland (1,049); for the remaining testing countries the number of samples analysed amounted to less than 1000. The data compilation includes data on all sampling strategies (surveillance data and data reflecting targeted sampling strategies).

It is noted that no specific results for straw and fodder (dry) of cereal grains are available in the EFSA pesticide monitoring database.

Barley

In total, 630 results on lindane in barley were submitted by 17 European reporting countries. The samples originated from 21 different countries. In none of the samples detectable residues at or above the LOQ were found (LOQ ranged from 0.002 mg/kg to 0.05 mg/kg).

Maize

In total, 642 results on lindane in maize were submitted by 15 reporting countries. The samples originated from 25 different countries. Detectable residues at or above the LOQ were found in none of the samples (LOQ ranged from 0.001 mg/kg to 0.1 mg/kg).

Oats

In total, 898 results on lindane in oats were submitted by 20 reporting countries. The samples originated from 26 different countries. None of the samples contained detectable residues at or above the LOQ (LOQ ranged from 0.001 mg/kg to 0.05 mg/kg).

Rye

In total, 1,658 results on lindane in rye were submitted by 21 reporting countries. The samples originated from 25 different countries. None of the samples contained detectable residues at or above the LOQ (LOQ ranged from 0.0004 mg/kg to 0.05 mg/kg).

Sorghum

In total, 36 results on lindane in sorghum were submitted by two reporting countries. The samples originated from three different countries. None of the samples contained detectable residues above the LOQ were found (LOQ ranged from 0.01 to 0.02 mg/kg).

Wheat

In total, 4942 results on lindane in wheat were submitted by 25 reporting countries. The samples originated from 45 different countries. In only one sample, originating from France, lindane was quantified above the LOQ (0.078 mg/kg). The LOQ values ranged from 0.001 mg/kg to 0.05 mg/kg.

Sweet corn (kernels)

In total, 424 results on lindane in sweet corn were submitted by 15 reporting countries. The samples originated from 27 different countries. None of the samples contained detectable residues at or above the LOQ (LOQ ranged from 0.002 to 0.02 mg/kg).

Milks

Altogether, 4,319 results of lindane residue data in milk of different species (cattle, sheep, goat and horses) were submitted by 25 reporting countries. The samples included only unprocessed, frozen and pasteurised milk. The samples originated from 28 different countries. The LOQ values ranged from 0.00004 to 0.001 mg/kg. It is noted that the results concerning 379 samples were reported on a fat basis. The detectable residues are summarized in Table 1.

Table 1 Lindane residues detected in milk samples.

Commodity	LOQ [mg/kg]	Expression of residues	Residue detected [mg/kg]
Cattle milk	0.00004	Whole product basis	0.00004, 0.00004,0.00004, 0.00008
	0.00005	Whole product basis	
	0.0001	Whole product basis	0.0001
		Fat basis	0.0006
Sheep milk	0.001	Whole product basis	0.002
Goat milk	0.0003	Whole product basis	0.0003
	0.0001	Whole product basis	0.0006

Meat (from mammals other than marine mammals)

Overall, 3,360 samples of meat and 2,657 samples of fat of mammals (swine, bovine, sheep, goat and equine) were analysed for lindane residues. These samples originated from 42 different countries and were tested by 27 EU countries. The LOQ values ranged from 0.0001 to 0.005 mg/kg. Overall, 40 samples contained measurable residues at or above the LOQ. For 2,957 meat samples the results were expressed on whole weight basis, which were converted to a fat basis by applying a default fat content of 20 % unless the actual fat content of the sample was reported. For 403 meat samples the results were expressed on a fat basis. It is noted that for 15 of the fat samples, where the results were reported on whole weight basis, a specific fat content was reported which was taken into account for the evaluation of the data.

Table 2 Lindane resides detected in animal meats

Commodity	LOQ [mg/kg]	Residues expressed	Residue detected [mg/kg]
Swine meat	0.0001	Wpb [a]	0.0002
	0.0005	Fat basis	0.0005, 0.0006, 0.0008, 0.001 (2), 0.002 (2), 0.003 (2)
	0.005	Fat basis	0.007, 0.009, 0.013, 0.015, 0.017
	0.001	Fat basis	0.001,0.001
	0.002	Wpb	0.002
Bovine meat	0.0005	Fat basis	0.0005, 0.0006, 0.0007,0.0008, 0.001, 0.002,0.003
	0.001	Fat basis	0.001 (3), 0.005
	0.002	Fat basis	0.0037
Sheet meat	0.001	Fat basis	0.001 (3)
Swine fat	0.005	Fat	0.007
Bovine fat	0.01	Fat	0.015
	0.005		0.006
Sheep fat	0.005		0.005, 0.006, 0.006, 0.009, 0.01, **0.53**

[a:] Wpb: Whole product basis

Mammalian edible offal

In total, 680 results on lindane residues in mammalian edible offal of different species (swine, bovine, sheep, goat and equines) were submitted by 23 reporting countries. It is noted that for 71 samples the results were expressed on a fat basis. The samples originated from 25 different countries. The LOQ values ranged from 0.0001 to 0.02 mg/kg. All but four samples were free of detectable residues (residues below the LOQ). The only detectable residues were measured in one sample of sheep edible offal (0.018 mg/kg on fat basis) and in three samples of bovine liver (0.0008 mg/kg, 0.001 mg/kg, 0.002 mg/kg on fat basis).

Poultry meat

Overall, 1,760 samples of poultry (chicken, geese, duck, turkey, and Guinea fowl) meat and poultry fat were reported (700 samples of poultry fat and 1,060 samples of poultry meat). These samples originated from 32 countries and were taken in 23 countries. The LOQ values ranged from 0.00005 to 0.02 mg/kg. For 931 poultry meat samples the results were reported on whole product basis. Thus, the results had to be recalculated on a fat basis using a default fat content of 10 % unless the specific fat content of the sample was reported. The LOQ was 0.0005 when the following residues were detected on a fat basis: 0.0006 (2), 0.0007 (3), 0.0008, 0.0009, 0.001 (5), 0.002 (11), 0.004 (2). The residues measured on whole product basis were recalculated assuming 10% fat. They were: 0.001, 0.002 and 0.004 mg/kg.

Poultry offal

In total, 406 results of poultry offal were reported; 402 thereof concerned poultry liver. The results for 13 samples of poultry edible offal were reported on a fat basis; the specific fat content of the samples were also reported. The samples originated from 18 different countries and tested by 15 countries. The LOQ values ranged from 0.0005 to 0.01 mg/kg. Four samples of poultry liver were reported at or above the LOQ. They were on a fat basis: 0.0009, 0.001, 0.0045, 0.1 mg/kg

Eggs

Overall, 2,465 results of lindane in eggs of different species (chicken, duck and quail) were submitted by 26 reporting countries. The samples included only unprocessed, frozen and pasteurised eggs. These samples originated from 28 countries. For 261 samples the results were expressed on a fat basis. The detectable residues at or above the LOQ are summarized in Table 3. (LOQs ranged from 0.00008 to 0.05 mg/kg).

Table 3 Lindane resides detected in eggs

LOQ [mg/kg]	Residues expressed	Residue detected [mg/kg]
0.0001	Wpb[1]	0.0001 (4), 0.0002 (2)
0.0005	Wpb	0.01
0.001	Wpb	0.001
0.005	Wpb	0.006, 0.007,
0.01	Wpb	0.25, 0.30
0.0005	Fat basis	0.0005 (2), 0.0006, 0.0007 (2), 0.0008, 0.001(4), 0.002
0.001	Fat basis	0.001 (4), 0.002
0.005	Fat basis	0.006

Wpb: whole product basis

GEMS/Food data

The GEMS/Food data package contained 4,110 individual results collected during 2000-2011 in Australia, New Zealand, China HK SAR, Germany, Slovakia and Denmark.

The summary of relevant results is given in Table 4. In addition, the results of analysis of other commodities are given in Table 5.

Table 4 Summary of the results of analyses for lindane residues in eggs, milk and meat samples

	N	LOD mg/kg	LOQ mg/kg	Residues detected
Chicken eggs	163	0.003	0.007	0
Eggs and egg products NS	37	0.003	0.007	0
Eggs	**200**			0
Cattle milk	341	0.002	0.0035	0
Milks, NS	19	0.001	0.003	0
Goat milk	1	0.0014	0.0034	0
Milks	**361**			0
Chicken meat	7	0.0007	0.003	0.0034
Turkey meat	4	0.001	0.003	0
Poultry meat	4	0.0007	0.003	0
Poultry meat	**15**			1
Cattle meat	5	0.001	0.005	0
Swine meat	4	0.001	0.02	0
Mammalian meat NS	7	0.0003	0.001	0
Mammalian meats	**16**			0
Poultry fat	206	0.0007	0.002	0
Fats and oils NS	1054	0.0007	0.002	0
	1260			0

Table 5 Summary of the results of analyses for lindane residues in fruits, vegetables, fish and seafood samples

	N	LOD mg/kg	LOQ mg/kg	Residues detected
Almonds	4	0.02	0.1	
Apple	12	0.001	0.005/0.1	
Avocado	7	0.02	0.1	
Banana	12	0.0001	0.01	

Dragon fruits	4	0.001	0.005	
Grapes	13	0.02	0.5	
Kiwi fruit	13	0.02	0.1	
Longan	4	0.001	0.005	
Mango	8	0.02	0.1	
Melons	4	0.1	0.5	
Nectarine	8	0.02	0.1	
Orange	12	0.02	0.1	
Papaya	4	0.0001	0.0005	
Peach	4	0.0001	0.0005	
Pear	4	0.0001	0.0005	
Pineapple	4	0.0001	0.0005	
Plum	4	0.0001	0.0005	
Pumelo/grapefruits	4	0.0001	0.0005	0.0028
Strawberries	8	0.02	0.1	
Watermelon	12	0.02	0.1	
Fruits	**145**			
Celery	8	0.002	0.1	
Cucumber	11	0.02	0.1	
Lettuce	13	0.02	0.1	
Mushrooms	8	0.02	0.1	
Onions	8	0.02	0.1	
Peppers sweet	8	0.02	0.1	
Persimmon	4	0.0001	0.0005	
Tomato	8	0.02	0.1	
Lambs lettuce	1	0.001	0.003	
Beans, dry	1	0.003	0.005	
Vegetables	**67**			
Cod	5	0.0004	0.002	
Eels	42	0.0003	0.001	
Herring	241	0.0001	0.0005	
Mackerel	36	0.0002	0.001	
Salmon	500	0.0001	0.0007	0.0019, 0.003, 0.0045, 0.0095
Sardines	7	0.1	0.4	
Fishes NS	1818	0.0002	0.0007	0.0028, 0.0029 (6), 0.0031, 0.0032 (6), 0.0033, 0.0036 (5), 0.0037 (2), .0038 (2), 0.004, 0.0042 (2), 0.0043 (2), 0.0045, 0.0051 (2), 0.0063 (4), 0.0083
Fish and sea food NS	119	0.0002	0.002	
Fish and sea food	**2768**			

Monitoring data from India

Monitoring data for lindane residues in cereals, eggs, poultry and meat obtained in India under "Monitoring of Pesticide Residues at National Level" during 2009-14 were reported in summarized form. They are shown in Tables 6 and 7.

Table 6 Summary results of monitoring lindane residues in cereals, meat and eggs in India

Year	Commodity	Number of samples	LOQ mg/kg	Detected residues [mg/kg]
2009-2014	Cereals (Rice & Wheat)	7650	0.01	0.01,0.02 (2), 0.04, 0.05, 0.06, 0.08, 0.16
	Meat & Eggs	2361	0.01	0

The Netherlands

Fifty seven positive results derived from monitoring programmes carried out between 2004–2013 were provided. The relevant commodities and the detectable residues found were: maize whole meal (0.003 mg/kg), maize grits (0.012 mg/kg), wheat wholegrain floor (0.003, 0.006 mg/kg). The total number of samples analysed were not reported.

United States

The Pesticide Data Program (PDP) is directed at raw agricultural products and various processed foods originated from domestic production and import. Although processed foods are also included, the emphasis is on the raw agricultural product, which is typically analysed as the unwashed, whole (unpeeled), raw commodity. In addition to monitoring foods for human consumption, FDA also samples and analyses domestic and imported animal feeds for pesticide residues (US FDA).

None of the 80,224 samples analysed between 2007–2012 contained detectable amounts of lindane in the commodities relevant to the present evaluation. Only 14 samples, comprising frozen potato, ginseng and ginseng products, chick pea, dried mushroom and panax root powder contained lindane residues in the range of 0.003 and 0.7 mg/kg.

APPRAISAL

Lindane was first evaluated by the Joint Meeting in 1966 (T, R). It had been last re-evaluated within the periodic review programme in 2002 (T) and 2003 (R). The Meeting established an ADI of 0-0.005 mg/kg bw and ARfD of 0.06 mg/kg bw. The Meeting agreed that the definition of the residue for compliance with MRLs and for estimation of dietary intake should be: lindane for both plant and animal commodities. The residue is fat-soluble.

Since lindane is currently listed in Annex A of the Stockholm Convention by which Parties must take measures to eliminate the production and use of the chemical, and there was no information on existing national registrations for lindane uses, the Forty-sixth Session of the CCPR (2014) requested a periodic review in 2015 to convert the CXLs into Codex EMRLs.

Monitoring data were submitted by the European Food Safety Authority (EFSA) for the period of 2009-2013 and from the GEMS/Food programme (2000-2011) to the Meeting. In addition, individual residue studies were provided by the Netherlands in processed maize and wheat, and summarized results from India and the USA.

Methods of residue analysis

Lindane can be recovered using numerous multi residue procedures. The sensitivity of the detection depends on the extraction and cleanup procedures, and the instrumentation available for qualitative and quantitative determination. No information was provided on the methods of analyses of samples for which lindane residues were reported. However, in the screening procedures, the objective is to detect residues which are around the legal limit, and to achieve the lowest concentration is not the primary goal. The reported LOQ values varied significantly in cases of individual commodities and among different commodities. The median reported LOQ values reported by EFSA and GEMS/Food were: cereal grains (0.01 mg/kg), mammalian and poultry meat (0.001 mg/kg), mammalian and poultry edible offal (0.001 mg/kg), milks (0.0004 mg/kg) and eggs (0.001 mg/kg). The Meeting assumed that these values can be realistically achieved applying current instrumental detection techniques and they were taken into consideration in estimation of EMRL values. If the LOQ exceeded the present CXL values, the reported <LOQ values were considered as non-detected.

Residues reported from monitoring programmes

The EFSA submitted the results of analyses of about 25,000 individual samples relevant to the present evaluation. The results originated from 60 different countries with the majority (96%) from the EU Member States, Iceland and Norway. In addition the Netherlands reported detected residues in some samples.

The GEMS/Food data package contained 4,110 individual results collected in Australia, New-Zealand, China HK SAR, Germany, Slovakia and Denmark. The data package included several commodities for which no CXL had been established. When sufficient numbers of results were available, these data were also considered for estimation of EMRLs.

India provided the summarized results of analyses of 7,650 cereal grain samples, including rice and wheat, and 2,361 meat and egg samples.

The summary results of the US FDA Pesticide Data Programme (2007-2012) were provided, which included over 80,000 residue measurements obtained from a large variety of commodities. None of the samples analysed between 2007 and 2012 contained lindane residues above the LOQ in the commodities relevant to the present evaluation.

The above data sets including the results of analyses of large numbers of samples did not indicate differences among geographical regions; therefore it was assumed that they provide information on the lindane concentration resulting from environmental contamination present around the world. Consequently, they were considered together for estimating EMRLs. According to previous practice of the JMPR, EMRLs should cover a minimum of 99 percentile of the relevant residue data population with a 99% probability (FAO Manual sub-chapter 6.11.2). To meet this criterion a

minimum of 459 valid results are required. For covering 99.9 percent of the likely residues present with 99 percent probability, 4,603 results would be needed.

Sweet corn

None of the 424 samples, reported by EFSA originating from 27 different countries, contained detectable lindane residues.

Using the mature maize residue data (642) as supporting evidence, the Meeting concluded that the database is sufficient to recommend an EMRL of 0.01 mg/kg for sweet corn kernels.

The Meeting withdraws its previous recommendation of 0.01(*) mg/kg.

Cereal grains

Individual residue analyses are available from European countries for barley (630), maize (642) oat (898), rye (1,658), sorghum (36), and wheat (4,942). Quantified residues were reported by France in wheat (0.078 mg/kg), and The Netherlands in whole maize flour (0.003 mg/kg), maize grits (0.012 mg/kg), and whole wheat flour (0.003 and 0.006 mg/kg). Of a total of 8,806 raw cereal grain samples, only one wheat sample (0.078 mg/kg) and one maize grit sample (0.012 mg/kg) contained residues above the current CXL of 0.01 mg/kg (0.022%). Based on this result it can be stated that at least 99.8% of the expectable residues are below the current CXL with a 99% probability. This conclusion is supported by the large number of results reported by India and USA.

The Meeting recommended an EMRL of 0.01 mg/kg for cereal grains.

The Meeting withdraws its previous recommendations of 0.01 (*) mg/kg for maximum residue levels in barley, maize, oats, rye, sorghum and wheat.

Straw and fodder of cereal grains

Based on the results reported by the 2003 JMPR (Pesticide Residues in Food - 2003 Evaluations Part I P583, pp 177) indicating similar, generally non-detected, residues in wheat grains, hay and straw, supported by the summarized US FDA data package, the Meeting concluded that residues above 0.01 mg/kg are unlikely to occur in straw and fodder, dry, from environmental contamination.

The Meeting recommended an EMRL of 0.01 mg/kg for straw and fodder of cereal grains.

The Meeting withdraws its previous recommendation of 0.01 (*) mg/kg.

Meat (from mammals other than marine mammals)

Overall, 3,360 samples of meat and 2,657 samples of fat of mammals (pig, cattle, sheep, goat and horse) were analysed for lindane residues. These samples, reported by EFSA, originated from 42 different countries. Overall, 40 samples contained residues at or above the LOQ. However, only one sheep fat sample contained residue (0.53 mg/kg) above the current CXL of 0.1 mg/kg (0.016%). The next two highest values were in swine meat (fat) 0.017 mg/kg and 0.015 in beef fat.

Sixteen mammalian meat samples reported from the GEMS/Food database contained non-detected residues

The Meeting concluded that the residue level reported is much lower than that which was reported at the time of the estimation of the current CXL of 0.1 mg/kg.

The Meeting recommended an EMRL of 0.01 mg/kg (fat) for meat (from mammals other than marine mammals)

The Meeting withdraws its previous recommendation of 0.1 mg/kg (fat).

Edible offal (mammalian)

Overall, 680 samples of mammalian edible offal of different species (pig, cattle, sheep, goat and horse) were analysed for lindane residues. These samples originated from 25 different countries.

Four samples contained residues but none of them exceeded the current CXL of 0.01 mg/kg. Three cattle liver samples contained residues (0.0008 mg/kg, 0.001 mg/kg, 0.002 mg/kg on a fat basis), and one sheep edible offal (0.018 mg/kg on a fat basis). The residues expressed on a whole product basis would be about 20 times lower.

The Meeting concluded that there was sufficient information to recommend an EMRL of 0.001 mg/kg for edible offal (mammalian).

The Meeting withdraws its previous recommendation of 0.01 (*) mg/kg.

Milks

Altogether 4,319 lindane residues in unprocessed, frozen and pasteurised milk samples of different species (cattle, sheep, goat and horses) were reported by EFSA. Overall, detected residues were ≥ LOQ (0.00004 (3), 0.00008, 0.0001, 0.0003, 0.0006 and 0.002 mg/kg on a whole product basis and 0.0006 mg/kg on a fat basis. None of them exceeded the current CXL.

Cattle (341) and goat (1) samples obtained from GEMS/Food database contained non-detected residues (< 0.002 mg/kg).

Based on the extensive data base, the Meeting recommended an EMRL of 0.001 mg/kg for milks.

The Meeting withdraws its previous recommendation of 0.01 (*) mg/kg.

Poultry meat

Overall, 700 samples of poultry fat and 1,060 samples of poultry meat (chicken, geese, duck, turkey, and Guinea fowl) were derived from 32 countries. The LOQ was 0.0005 when the following residues [mg/kg] were detected on a fat basis: 0.0006 (2), 0.0007 (3), 0.0008, 0.0009, 0.001 (5), 0.002 (11), 0.004 (2). The residues measured on a whole product basis were recalculated assuming 10% fat. The values were: 0.001, 0.002 and 0.004 mg/kg.

One chicken meat (fat) sample of the 15 poultry meat samples obtained from GEMS/Food database contained residues of 0.0034 mg/kg lindane. None of the samples contained residues above the current CXL. The results indicate that 99.5% of the samples would unlikely contain residues above 0.004 mg/kg (fat) in 99.9% of the cases.

Based on the data available the Meeting concluded that 0.005 mg/kg residue level would sufficiently cover the residues carried over from environmental contamination, and recommended it as the EMRL for poultry meat (on fat basis).

The Meeting withdraws its previous recommendation of 0.05 mg/kg.

Poultry, edible offal of

In total, 406 results of poultry offal were reported by EFSA of which 402 were poultry liver. Four samples contained detected residues. They were on a fat basis: 0.0009, 0.001, 0.0045, 0.1 mg/kg. The residues expressed on a whole product basis would be at least 20 times lower.

Based on the 406 residue dataset, it can be assumed that 99% of the sampled lot would contain less than 0.01 mg/kg lindane residues with at least 98% probability.

Based on the available data the Meeting recommended an EMRL of 0.005 mg/kg for poultry, edible offal.

The Meeting withdraws its previous recommendation of 0.01 (*) mg/kg.

Eggs

Altogether 2,665 residue determinations were conducted in eggs on a whole product or fat basis as reported by EFSA and obtained from the GEMS Food database. The samples originated from more

than 26 countries. Of the 2,665 samples only 2 (0.075%) contained residues (0.25 and 0.3 mg/kg) above the current CXL.

Based on the available data the Meeting recommended an EMR of 0.001 mg/kg for eggs.

The Meeting withdraws its previous recommendation of 0.01 mg/kg.

Fish and diadromous fish

Lindane residues were reported from the GEMS/Food data base. Residues were detected in 41 of 2,649 samples. They were in rank order: 0.0019, 0.0028, 0.0029 (6), 0.003, 0.0031, 0.0032 (6), 0.0033, 0.0036 (5), 0.0037 (2), 0.0038 (2), 0.004, 0.0042 (2), 0.0043 (2), 0.0045 (2), 0.0051 (2), 0.0063 (4), 0.0083 and 0.0095 mg/kg.

The Meeting considered that the residues in fish are a suitable indicator of environmental contamination. The Meeting concluded that the residue data on fish derived from the GEMS/Food database would provide sufficient basis (99.8% of residues with 99.5% probability) for estimation of likely maximum residue levels of lindane in fish.

Based on the data available the Meeting recommended an EMRL of 0.01 mg/kg for fish and diadromous fish.

RECOMMENDATION

The Meeting noted that there are no authorised uses of lindane for crop protection and withdraws its previous recommendations for maximum residue levels and recommends the following extraneous residue levels for use as EMRLs.

Definition of residue is unchanged.

Definition of reside for compliance with EMRLs and for estimation of dietary intake: lindane.

The residue is fat soluble.

		Estimated residue levels mg/kg			Recommendation [a]	
CCN	Commodity	EMRL [b]	*Median*	*Highest*	New	Previous
GC 0640	Barley				W	0.01*
GC 0051	Cereal grains, except rice	0.01	*0.005*	*0.005*		
WD 0120	Diadromous fish	0.01	*0.0036*	*0.0095*		
MO 0105	Edible offal (mammalian)	0.001	*0.00002*	*0.0002*	W	0.01*
PE 0112	Eggs	0.001	*0.0007*	*0.002*	W	0.01*
GC 0645	Maize				W	0.01*
WS 0125	Marine fish	0.01	*0.0036*	*0.0095*		
MM 0095	Meat (from mammals other than marine mammals)	0.01 (F)	*0.00007 (0.0005)*	*0.0005 (0.006)*	W	0.1
ML 0106	Milks	0.001	*0.00003*		W	0.01*
GC 0647	Oats				W	0.01*
PM 0110	Poultry meat	0.005 (F)	*0.0006 (0.0008)*	*0.001 (0.016)*	W	0.05
PO 0111	Poultry, edible offal of	0.005	*0.00008*	*0.0002*	W	0.01*
GC 0650	Rye				W	0.01*
GC 0651	Sorghum				W	0.01*
AS 0161	Straw and fodder of cereal grains	0.01			W	0.01*
VO 1275	Sweet corn (kernels)	0.01	*0.005*	*0.005*	W	0.01*
GC 0655	Wheat				W	0.01*

[a] Lindane was recently classified as 2A (Probable carcinogen) by IARC. Since lindane is listed in annex A of the Stockholm convention and should be eliminated from production and use no toxicological re-evaluation is requested.

[b] Extraneous Maximum Residue Limit (EMRL) is the maximum concentration of a pesticide residue arising from environmental sources due to former agricultural uses, not from the use of the pesticide directly or indirectly on the food or feed.

Estimation of dietary intake

Cereal grains

The median LOQ value reported for barley, maize, oats, rye, sorghum and wheat is 0.01 mg/kg. For dietary intake calculations the 2003 JMPR estimated an STMR and HR of 0.005 mg/kg based on the results of supervised trials. As the estimated EMRL is at the same level as the previous CXL value, the Meeting concluded that the best estimates of the STMR and HR for these commodities and sweet corn are those recommended by the 2003 JMPR.

Animal commodities

Based on animal feeding studies taking into account the expected residue levels in feed commodities deriving from the use of lindane, the 2003 JMPR recommended HR and STMR values for muscle (0.005 mg/kg and 0.0007 mg/kg), fat (0.06 mg/kg and 0.005 mg/kg) edible offal (0.002 mg/kg and 0.0002 mg/kg) from mammals other than marine mammals, and STMR of 0.0003 mg/kg for milks.

Based on the monitoring data, the current residue level in mammalian meat and poultry meat is 10 times lower; the Meeting applied the 10 times lower factor in the corresponding commodities, compared with those estimated by the 2003 JMPR.

The Meeting estimated highest and median residue values for muscle (0.0005 mg/kg and 0.00007 mg/kg), fat (0.006 mg/kg and 0.0005 mg/kg) edible offal (0.0002 mg/kg and 0.00002 mg/kg) from mammals other than marine mammals, and a median residue of 0.00003 mg/kg for milks.

The 2003 Meeting recommended HR and STMR values for poultry meat (0.001 mg/kg and 0.0006 mg/kg), poultry fat (0.016mg/kg and 0.008 mg/kg), eggs (0.002 mg/kg and 0.0007 mg/kg) and edible offal (0.001 mg/kg and 0.0004 mg/kg).

For poultry meat and edible offal the residue levels are about 5–10 times lower, respectively, than those estimated in 2003.

The Meeting estimated highest and median residue values for poultry meat (0.0001 mg/kg and 0.00006 mg/kg), poultry fat (0.0016 mg/kg and 0.0008 mg/kg), poultry edible offal (0.0002 mg/kg and 0.00008 mg/kg) and eggs (0.0002 mg/kg and 0.00007 mg/kg

The fish consumption data was provided by the GEMS/Food database. The long-term intake is 0.43 g/kg bw and the short-term intake (97.5^{th} percentile of 1,043 consumption days) is 10 g/kg bw. The short-term intake was calculated with the highest residue observed in fish (0.0095 mg/kg) and the long term intake was calculated with the median of LOQ values (0.0036 mg/kg) reported for analyses of fish samples.

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of lindane were calculated for the 17 GEMS/Food cluster diets using STMRs estimated by the Meeting. The results are shown in Annex 3 to the 2015 Report.

The ADI is 0–0.005 mg/kg bw and the calculated IEDIs were 0–1% of the maximum ADI. The fish consumption contributes to < 0.001% of the max ADI. The Meeting concluded that the long-term intake of residues of lindane from the environmental contamination of commodities considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The ARfD is 0.06 mg/kg bw. The short-term intake calculated using the HR and STMR values estimated by the Meeting were 0% of the ARfD for children and the general population. The fish consumption contributes to 0.016% of the ARfD. The Meeting concluded that the short-term intake of residues of lindane from the environmental contamination of commodities considered by the JMPR is

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http://www.fda.gov/downloads/Food/FoodborneIllnessContaminants/Pesticides/UCM432758.pdf

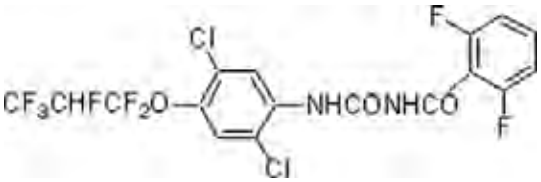
LUFENURON (286)

The first draft was prepared by Mr Christian Sieke, Federal Institute for Risk Assessment, Berlin, Germany

EXPLANATION

Lufenuron is an insect growth inhibitor that is active against larvae of Lepidoptera and Coleoptera. When ingested, lufenuron interferes with chitin synthesis, and prevents larvae from moulting. It was considered for the first time by the 2015 JMPR for toxicology and residues.

IDENTITY

ISO common name	Lufenuron
Chemical name	
IUPAC	(*RS*)-1-[2,5-dichloro-4-(1,1,2,3,3,3-hexafluoropropoxy)phenyl]-3-(2,6-difluorobenzoyl)urea
CA	N-[[[2,5-dichloro-4-(1,1,2,3,3,3-hexafluoropropoxy)phenyl]amino]carbonyl]-2,6-difluorobenzamide
CAS No.	103055-07-8
CIPAC No.	704
Structural formula	
Molecular formula	$C_{17}H_8Cl_2F_8N_2O_3$
Molecular mass	511.15 g/mol

Lufenuron consists of a pair of enantiomers. A chiral centre exists at the 2-position of the hexafluoropropoxy side-chain. Lufenuron technical active ingredient is manufactured under non-stereospecific conditions giving a racemate (R:S 50:50).

Specifications

Specifications for lufenuron were not yet developed by FAO.

PHYSICAL AND CHEMICAL PROPERTIES

Property	*Results*	*Method (test material)*	*Reference*
Melting point	168.7–169.4 °C	OECD 102 (Batch AMS 266/102, 99.7% purity)	Das, R, 1998 LUFEN_001
Boiling point & temperature of decomposition	Not measurable (decomposes) Decomposition starts to occur at about 242 °C	OECD 103 (Batch AMS 266/102, 99.7% purity)	Das, R, 2000 LUFEN_002
Appearance	Appearance—pure active substance: white fine powder (PAI)	Visual inspection (Batch AMS 266/102, 99.7% purity)	Das, R, 1998 LUFEN_003
Relative density	1.67 g cm^{-3} at 20 °C	OECD Guideline for Testing of Chemicals 109 (Batch AMS 266/102, 99.7% purity)	Fueldner, 1998, LUFEN_004

Property	*Results*	*Method (test material)*	*Reference*
Vapour pressure	$< 4 \times 10^{-6}$ Pa at 25 °C	OECD Guideline for Testing of Chemicals 104A (Batch AMS 266/101, 99.7% purity)	Geoffroy, 1992, LUFEN_005
Henry´s Law Coefficient	$< 4.4 \times 10^{-2}$ Pa m^3 mol^{-1}	Calculation	Born, 2008, LUFEN_006
Solubility in water including effect of pH	pH 5: 54 µg/L (25 °C) pH 7: 46 µg/L (25 °C) pH 9: 64 µg/L (25 °C)	OECD Guideline for Testing of Chemicals 105 (Batch AMS 266/102, 99.7% AI)	Das, R, 2002, LUFEN_007
Partition coefficient n-octanol / water	log P_{OW}=5.12 (25 °C, pure water)	OECD Guideline for Testing of Chemicals 117 (Batch AMS266/102, 99.7% AI)	Rodler, 1992, LUFEN_009
Dissociation constant	$pK_{a,1}$=10.18 at 20 °C in methanol:water mixtures	OECD Guideline for Testing Chemicals 112 (Batch AMS266/102, 99.7% AI)	Martin, 2002, LUFEN_010
UV/VIS absorption (max.) incl. ε	Wavelength [nm] / molar extinction coefficient [L/mol · cm] neutral solution 210 / 37293 255 / 16417 295 / 1648 acidic solution 210 / 30588 255 / 15165 295 / 2220 basic solution 230 / 20658 267 / 22440 295 / 4871 No absorption maximum between 350 nm and 750 nm was observed	OECD Guideline for Testing Chemicals 101 (Batch AMS266/102, 99.7% AI)	Oggenfuss, 2002, LUFEN_011
	Wavelength [nm] / molar extinction coefficient [L/mol · cm] methanol 290 / 5212 305 / 499 Absorption levels out above 300 nm	JMAFF Agchem Test Guidelines 12 (Batch ILA-178.3, 98.9% AI)	Mamouni, 2004, LUFEN_012
Photochemical degradation in water	pH 7, 25 °C (buffer) $t_{1/2}$ 11.2 ± 1.3 days (natural sunlight at 30–50 °N, 12:12 photocycle)	JMAFF Agchem Test Guidelines 12 (Batch ILA-178.3, 98.9% AI)	Mamouni, 2004, LUFEN_012
	Sterile buffer pH 7, 25 °C (Xenon arc light, λ≥ 290 nm) DT_{50}: 16 d continuous Xenon arc light equivalent to ca. 34 d clear summer sunlight at 30–40 °N)	EPA 540/9-82-021 ([^{14}C-dichlorophenyl]-label, AMS 266/101, 99.5% AI)	Ellgehausen, 1994, LUFEN_013
	Sterile buffer pH 7, 25 °C (Xenon arc light, λ≥ 290 nm) DT_{50}: 10.3 d continuous Xenon arc light equivalent to ca. 18 d clear summer sunlight at 30–40 °N)	EPA 540/9-82-021 ([^{14}C-dichlorophenyl]-label, AMS 266/101, 99.5% AI)	Ellgehausen, 1994, LUFEN_014
Quantum yield of direct photo-transformation	ϕ=0.0026 in 0.01 M phosphate buffer/ethanol mixture (1:1 v/v), λ=290 nm	UBA Draft Test Guideline "Phototrans-formation of Chemicals in Water, Part A, Direct Phototransformation", Berlin, FRG 1990 (Batch AMS 266/101, 99.5% AI)	Abildt, 1995, PYMET_015

Property	*Results*	*Method (test material)*	*Reference*
Solubility in organic solvents	The solubility in different organic solvents at 25 °C was determined to be : acetone 460 g/lL dichloromethane 84 g/lL ethyl acetate 330 g/lL hexane 0.10 g/lL methanol 52 g/lL octanol 8.2 g/lL toluene 66 g/lL	In-house method (Batch P.704809, 99.5% AI)	Kettner, 2000, LUFEN_008

Formulations

Lufenuron is primarily available as the following EC formulations:

Formulations registered containing lufenuron as active ingredient.

Formulation	*Content of active ingredients*	*Trade names*
EC	50 g/L	Match EC, Match 5 EC, Curyom 550 EC

METABOLISM AND ENVIRONMENTAL FATE

Metabolism studies were conducted using [dichlorophenyl-^{14}C]-lufenuron (dichlorophenyl-label) and [difluorophenyl-^{14}C]-lufenuron (difluorophenyl-label). The position of the label for both substances is presented in the following figures:

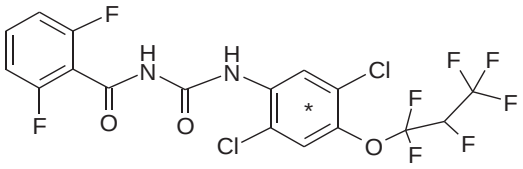


Figure 1 [dichlorophenyl -^{14}C]-lufenuron

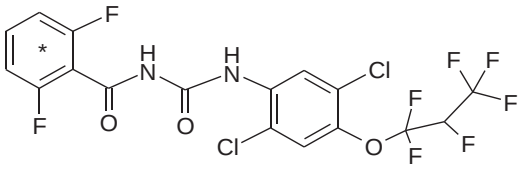
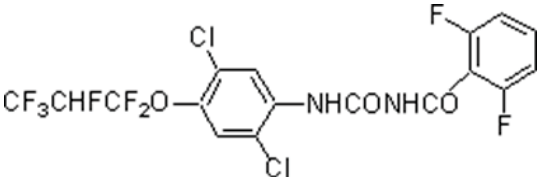
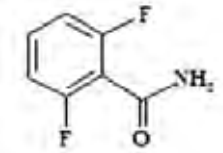
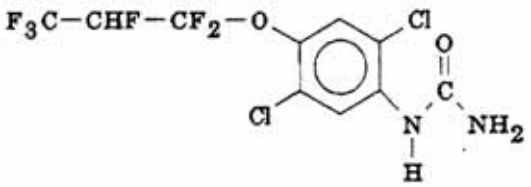
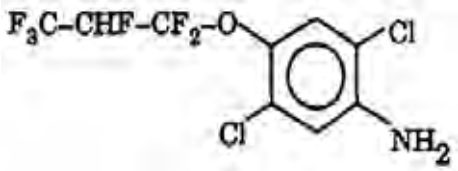
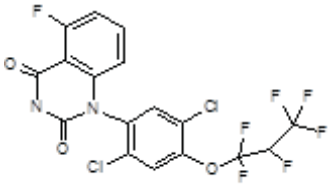


Figure 2 [difluorophenyl-^{14}C]-lufenuron

Chemical names, structures and code names of metabolites and degradation products of lufenuron are shown below.

Code Names	*Chemical Abstracts Name (IUPAC Name), molecular formula, molar mass*	*Structure*	*Where found*
Parent lufenuron, CGA 184699	(RS)-1-[2,5-dichloro-4-(1,1,2,3,3,3-hexafluoropropoxy) phenyl] -3-(2,6-difluorobenzoyl)urea		Cabbage leaves tomato fruit Goat—kidney, urine and

Code Names	*Chemical Abstracts Name (IUPAC Name), molecular formula, molar mass*	*Structure*	*Where found*
			faeces Hen—kidney, egg white, excreta
CGA149776	2,6-Difluoro-benzoic acid		Goat—faeces Hen—excreta Soil
CGA149772	2,6-Difluoro-benzamide		Goat—faeces Hen—egg white Soil
CGA238277	2,5-Dichloro-4-(1,1,2,3,3,3-hexafluoropropoxy) -phenyl-urea		Goat—faeces, Hens—kidney
CGA224443	N-[2,5-dichloro-4-(1,1,2,3,3,3-hexafluoropropoxy) -benzenamine		Soil
CGA301018			Water

Environmental fate in soil

For the investigation of the environmental fate of lufenuron the Meeting received studies on soil photolysis, hydrolysis, aerobic soil metabolism and the behaviour in confined rotational crops.

Soil photolysis

The soil surface photolytic behaviour of lufenuron on moist and dry soil was investigated by Ellgehausen (1994, LUFEN_026) using [^{14}C]dichlorophenyl ring-labelled lufenuron.

Moist and dry soil was dosed with radio-labelled lufenuron at 5 µg/cm^2 (equivalent to 500 g ai/ha). The samples were irradiated continuously at 25 °C for up to 17 days. Samples were taken at 0, 5, 9, 14, 19 and 26 days. One dark control sample was prepared in parallel.

For analysis, the soil layer was extracted by shaking with acetone (twice) followed by a mixture of acetone:water (80:20 v/v). Following each extraction step, the samples were centrifuged and the supernatants combined. The supernatants were concentrated, partitioned with dichloromethane and the radioactivity in each phase quantified by LSC. Characterisation and quantification of the photo-degradation products was conducted by HPLC.

The percentage recovery of the applied radioactivity is presented in Tables 1 and 2 and ranged from 99.4–103.8%. The recovery from the dark control plates was > 99% at the end of the study.

Table 1 Distribution of Applied Recovery in dry soil after Continuous Irradiation and results of the dark control sample

Degradate	Incubation period (hours)						Dark control
	0	168	240	288	336	408	
Lufenuron	99.55	91.21	87.97	88.71	85.23	85.46	99.3
CO_2	0	3.17	4.24	5.34	6.47	7.79	0.0
Unidentified degradates [a]	0.86	2.38	5.68	3.49	6.03	6.28	0.72
Unextracted	0.06	4.91	4.62	4.11	5.15	4.2	0.93
Organic volatiles	0	0.02	0.03	0.04	0.05	0.07	0.0
Total	100.5	101.7	102.5	101.7	102.9	103.8	100.9

[a] At least three components, none of which exceeded 3.8% AR

Table 2 Distribution of Applied Recovery in moist soil after Continuous Irradiation

Degradate	Incubation period (hours)					
	0	120	216	336	456	624
Lufenuron	96.95	93.99	93.25	91.76	90.9	90.05
CO_2	0	0.27	0.55	0.85	1.19	1.76
Unidentified degradates [a]	2.39	4.15	3.85	4.63	4.51	4.29
Unextracted	0.05	2.56	3.18	4.04	4.11	5.02
Organic volatiles	0	0	0	0	0	0
Total	99.4	101.0	100.8	101.3	100.7	101.1

[a] At least five components, none of which exceeded 1.9% AR

In a second experiment conducted by Ellgehausen (1994, LUFEN_027) [^{14}C]difluorophenyl-labelled lufenuron was used to investigate its behaviour under soil photolysis. The experimental conditions and analytical methods were identical to the ones used in the previous study for the [^{14}C]dichlorophenyl-label, however only dry soil was investigated.

The percentage recovery of applied radioactivity is presented in the following table and ranged from 99.7 to 102.1%. The recovery from the dark control plates was 101% at the end of the study.

Table 3 Distribution of Applied Recovery in Dry soil after Continuous Irradiation

Degradate	Incubation period (hours)						Dark control
	0	120	292	309	381	453	
Lufenuron	94.2	90.7	89.6	88.6	81.7	84.0	97.2
CGA149772 [a]	2.23	6.07	6.50	7.08	11.2	7.14	1.4
CO_2	0	1.32	2.06	3.85	4.85	6.34	0.0

Unidentified degradates [b]	3.2	1.7	1.4	0.51	1.9	1.8	1.5
Unextracted	0.04	2.25	1.77	1.94	2.37	2.14	0.96
Organic volatiles	0	0.01	0.02	0.04	0.05	0.07	0.0
Total	99.7	102.0	101.4	102.0	102.1	101.5	101.0

[a] The values in this row have not been adjusted for the 1.1% present in the starting material

[b] At least five components, none of which exceeded 1.6% AR

The amounts of lufenuron recovered decreased very slowly from 94.2% AR to 84.0% AR after 18.9 days continuous irradiation. CGA149772, the difluorobenzamide metabolite, reached a maximum of 11.2% AR after 15.8 days then decreased to 7.1% AR at the end of the study. A maximum 6.3% of carbon dioxide was evolved.

Hydrolysis

The stability of lufenuron in sterile buffer solutions was investigated using [dichlororphenyl-^{14}C] and [difluorophenyl-^{14}C]-lufenuron (Ellgehausen, 1992, LUFEN_025).

The test compounds were incubated under sterile conditions in buffer solutions contained in brown glass test tubes. A range of pH (5, 7 and 9) and temperature (25 ºC) conditions were applied to both difluorophenyl-labelled and dichlorophenyl-labelled lufenuron. In addition, a few experiments were conducted under more extreme conditions (pH 1 and 13) and temperature (50 and 70 °C) although not every combination was tested. Lufenuron and its degradation products were partitioned with dichloromethane and the amounts in each phase quantified by LSC and HPLC. Degradates were characterized, after derivatisation where necessary, by MS or GC-MS.

For the samples incubated at 25 °C, both labels showed virtually no degradation at pH 5, 7 and 9. Over 93% of the initial radioactivity was recovered as unchanged lufenuron. Only at pH 9, minor amounts of CGA238277 (3.9% AR) and CGA224443 (1.8% AR) for the dichlorophenyl-label and CGA149776 (3.8% AR) for the difluorophenyl-label were found.

Under more extreme conditions the parent substance was stable at pH 1 and 70 °C, representing more than 90% of the radioactivity after up to 168 hours. At pH 9 an accelerated degradation was observed. An overview of the degradation for the dichlorophenyl-label is presented in Tables 4 and 5, while the difluorophenyl-label results are presented in Tables 6 and 7.

Table 4 Hydrolysis of [^{14}C]dichlorophenyl-lufenuron at pH 9 and 50 °C (%AR)

Time (hours)	Lufenuron	CGA224443	CGA301018	CGA238277	Total
0	101.04	0	0	0	101.04
4	97.07	0	0	3.42	100.49
6	96.58	3.07	0	2.77	102.42
8	96.96	1.42	0	3.04	101.42
24	87.59	5.15	2.02	6.16	100.92
32	85.74	5.47	2.33	7.80	101.34
48	81.94	6.16	3.63	9.18	100.91
72	71.02	9.53	4.01	15.75	100.31
78	68.86	9.28	5.95	16.74	100.83
102	60.83	13.83	5.59	17.86	98.11
150	57.85	15.05	5.96	18.86	97.72
174	53.47	15.99	7.36	21.29	98.11

Table 5 Hydrolysis of [^{14}C]dichlorophenyl-lufenuron at pH 9 and 70 °C (%AR)

Time (hours)	Lufenuron	CGA224443	CGA301018	CGA238277	Unresolved	Total
0	99.12	0	0	0	0.88	100
2	73.16	11.27	4.35	10.08	1.12	99.98
4	46.99	24.14	9.16	18.22	1.22	99.73
7	30.3	33.56	10.76	24.08	1.38	100.08

24	10.5	46.74	14.97	24.98	1.77	98.96
48	7.29	53.11	14.67	13.68	3.51	92.26
72	8.75	49.03	16.09	19.16	1.8	94..83
96	10.95	51.62	15.75	10.68	2.09	91.09
120	1.77	60.94	15.35	8.96	4.44	91.46

Table 6 Hydrolysis of [^{14}C]difluorophenyl-lufenuron at pH 9 and 50 °C (%AR)

Time (hours)	Lufenuron	CGA301018	CGA149776	CGA149772	Total
0	98.46	0	0	0	98.46
24	70.40	3.43	13.63	10.59	98.05
48	56.98	4.67	21.54	16.08	99.27
72	33.42	7.47	32.63	24.77	98.29
96	18.63	8.82	41.33	30.33	99.11
120	24.05	7.89	39.13	27.5	98.57
144	18.86	9.38	40.29	30.22	98.75
168	7.33	10.89	45.95	34.86	99.03
192	14.65	11.01	41.5	31.17	98.33
216	14.5	11.15	39.55	33.44	98.64

Table 7 Hydrolysis of [^{14}C]difluorophenyl-lufenuron at pH 9 and 70 °C (%AR)

Time (hours)	Lufenuron	CGA301018	CGA301020	CGA149776	CGA149772	Total
0	99.97	0	0	0	0	99.97
2	21.81	11.25	0	24.72	42.71	100.49
4	17.33	11.99	0	25.69	45.74	100.75
7	6.44	11.27	0	30.4	52.54	100.65
24	0	15.08	0	29.57	55.24	99.89
48	0	14.4	0	32.29	53.29	99.98
72	0	14.57	0	30.76	55.18	100.51
96	0	12.97	1.77	31.84	51.68	98.27
120	0	12.43	1.29	31.22	54.49	99.43

In the experiments conducted at a pH of 13 with up to 70 °C incubation temperature, lufenuron was completely degraded within the first 24 hours. The primary hydrolysis products formed were CGA239786 (up to 51% AR after 96 h) and CGA301020 (up to 19% AR after 32 h) for the [^{14}C]dichlorophenyl-label and CGA149776 (up to 49% AR after 2.5 h) for the [^{14}C]difluorophenyl-label.

Aerobic soil metabolism

In a first set of studies the aerobic soil metabolism of lufenuron was investigated in two microbial active soil types and in their sterilised form.

Ref.: Ellgehausen (1991, LUFEN_028)

Test material: [^{14}C]dichlorophenyl-lufenuron Dose rate: 1 mg/kg

Duration: 361 days Temp: 20 °C

Moisture: 44.8% Soil: Collombey (sandy loam, micro. active)

pH 7.2 Organic carbon: 3.0%

Half-live (parent): 24 days two 1st order compartment model) ^{14}C accountability: 99–107%

% lufenuron remaining: 8.2% after 361 days

% mineralisation: up to 9.9% after 361 days

% unextracted: up to 70.7% after 240 days

Metabolites	Max (% TRR)	Day
CGA238277	24.3	14
CGA224443	26.9	59

Ref.: Ellgehausen (1991, LUFEN_028)

Test material: [^{14}C]dichlorophenyl-lufenuron — Dose rate: 1 mg/kg

Duration: 361 days — Temp: 20 °C

Moisture: 83.6% — Soil: Les Evouettes (loam, microbial active)

pH 6.8 — Organic carbon: 3.8%

Half-live (parent): 16 days two 1st order compartment model) — ^{14}C accountability: 100–110%

% lufenuron remaining: 4.2% after 361 days

% mineralisation: up to 15.1% after 361 days

% unextracted: up to 78.6% after 240 days

Metabolites	Max (% TRR)	Day
CGA238277	23.1%	14
CGA224443	21.6%	59

Ref.: Ellgehausen (1991, LUFEN_028)

Test material: ^{14}C-difluorophenyl-lufenuron — Dose rate: 1.2 mg/kg

Duration: 361 days — Temp: 20 °C

Moisture: 83.6% — Soil: Les Evouettes (loam, microbial active)

pH 6.8 — Organic carbon: 3.8%

Half-live (parent): 24 days two 1st order compartment model) — ^{14}C accountability: 80–103%

% lufenuron remaining: 1.8% after 361 days

% mineralisation: up to 58.6% after 361 days

% unextracted: up to 36.1% after 60 days

Metabolites	Max (% TRR)	Day
None		

The aerobic soil metabolism was also investigated in the same soil types as above without microbial activity (sterile soil). After up to 90 days only unchanged lufenuron was recovered for both radiolabels without significant mineralisation or an increase of unextracted residues.

In a second study Gonzalez-Valero (1991, LUFEN_030) investigated the degradation of [^{14}C]dichlorophenyl-lufenuron in two soil types.

Ref.: Gonzalez-Valero (1991, LUFEN_030)

Test material: [^{14}C]dichlorophenyl-lufenuron — Dose rate: 0.1 mg/kg dry soil

Duration: 149 days
Moisture: 40% MWC
pH 5.0
Half-live (parent): 83 days
% lufenuron remaining: 32.4% after 149 days
% mineralisation: 2.0% after 149 days
% unextracted: 24.6% after 149 days

Temp: 20 °C
Soil: Neuhofen (sand, sterilised)
Organic carbon: 1.78
^{14}C accountability: 95.4–101.5%

Metabolites	Max (% TRR)	Day
CGA238277	10.1%	82
CGA224443	32.8%	149

Ref.: Gonzalez-Valero (1991, LUFEN_030)

Test material: [^{14}C]dichlorophenyl-lufenuron Dose rate: 0.1 mg/kg dry soil

Duration: 100 days Temp: 20 °C

Moisture: 40% MWC Soil: Mosimann (sandy loam, sterilised)

pH: 7.3 Organic carbon: 1.08

Half-live (parent): 17 days ^{14}C accountability: 89.9–101.8%

% lufenuron remaining: 8.1% after 82 days

% mineralisation: 5.0% after 100 days

% unextracted: 56.8% after 100 days

Metabolites	Max (% TRR)	Day
CGA238277	31.8%	30
CGA224443	28.0%	61

The nature of the unextracted radioactivity was further investigated by van der Gaauw (2004, LUFEN_029). After 90 day incubation of two soil types (silt loam “Les Evouettes”; loamy sand “Collombey”) the samples were extracted with three different extractants: acetonitrile: water (4:1 v/v, “solvent”), 40 mM aqueous solution of hydroxypropyl-ß-cyclodextrin (“HPCD”) or 0.02 M aqueous calcium chloride solution ($CaCl_2$). The radioactive residues, CO_2 and biomass were investigated during the experiment. In the following table the mass balance for each of the soils and its extraction efficiencies are summarized:

Table 8 Mass balance of radioactivity in soil

		Recovered Radioactivity (% applied)				
Soil Type		Days after treatment / extraction system				
		0	90/solvent	90/HPCD	90/$CaCl_2$	121/solvent
Collombey	Extractable	96.4	19.9	23.3	1.0	16.0
	Soxhlet	–	5.9	10.5	–	4.0
	Reflux	–	5.7	–	–	6.0
	CO_2	–	13.7	12.7	12.8	13.8
	Unextracted	3.1	49.1	51.4	89.6	52.6
	TOTAL	99.6	94.2	97.9	103.4	92.5
Les Evouettes	Extractable	97.1	19.7	10.9	0.35	16.9
	Soxhlet	–	6.7	15.2	–	4.0
	Reflux	–	3.2	–	–	4.2
	CO_2	–	20.0	19.0	18.8	20.3
	Unextracted	4.6	44.5	56.9	75	49.6
	TOTAL	101.7	94.1	102.0	94.2	95.0

In the “solvent” and “HPCD” extracts the composition of the radioactivity was analysed. In addition the radioactivity associated to the biomass was characterized.

Table 9 Distribution of radioactivity

Soil	Degradate	Days after treatment/extraction system			
	(% of applied)	0	90/solvent	90/HPCD	121/solvent
Collombey	Lufenuron	96.4	10.8	6.7	10.6
	CGA238277	< 0.1	4.7	6.2	7.7
	CGA224443		1.2	11.6	
	Unknown M3		2.5	5.8	
	Unknown M4		6.5		1.6
	Unknowns (2)		0.1	3.6	
	TOTAL	96.4	25.8	33.9	19.9
Les Evouettes	Lufenuron	97.0	9.3	3.4	7.6
	CGA238277		9.3	5.2	6.8
	CGA224443		0.8	7.3	0.1
	Unknown M3		1.9	7.5	
	Unknown M4		4.0		4.9
	Unknowns (4)		1.1	2.7	1.3
	TOTAL	97.4	26.4	26.1	20.7

Table 9 Organic matter fractionation of the residue remaining from solvent extraction

	Recovered Radioactivity (% applied)	
Soil fraction	Collombey	Les Evouettes
Fulvic	5.9	5.0
Humic	15.6	10.9
Humin	27.7	28.7
Total	49.1	44.6

In addition the influence of the application technique was investigated by Ellgehausen (1994, LUFEN_033). In this study [^{14}C]difluorophenyl ring-labelled lufenuron was applied at 0.1 mg/kg to a silt loam soil (60% MHC) under three test conditions involving surface treatment, incorporation and surface treatment following incorporation after 14 days. For each of the three conditions the remaining residues of the parent substance were measured.

In the following tables the mass balance and the recovered parent substance at various sampling intervals are summarized.

Table 11 Mass balance for the applied radioactivity following three different treatment conditions

	% AR						
	0 d	7 d	14 d	21 d	34/35 d	71/72 d	91/92 d
Incorporated							
Extractable	96.3	58.6	36.9	26.0	16.9	9.8	8.8
CO2	–	14.5	27.5	35.0	42.4	50.2	52.0
Unextracted	4.4	27.2	35.3	37.9	37.6	35.8	37.2
Total	100.7	100.3	99.7	98.9	96.9	95.8	98.1
No incorporation							
Extractable	94.3	81.1	73.5	59.4	44.4	24.9	36.8
CO2	–	4.4	9.1	12.6	16.7	20.3	20.7
Unextracted	3.6	13.7	17.6	25.2	33.2	47.5	36.4
Total	98.0	99.2	100.2	97.3	94.3	92.6	93.8
Surface then mixing after 14 d							
Extractable	96.0	80.3	71.5	52.9	31.0	17.4	12.8
CO2	–	4.7	9.8	15.2	21.2	25.6	26.2
Unextracted	3.0	14.1	18.9	28.8	44.1	55.5	53.3
Total	99.0	99.1	100.2	96.9	96.3	98.5	92.2

Table 12 Parent lufenuron remaining and calculated DT_{50} values

	% AR							DT_{50}
Test Conditions	0 d	7 d	14 d	21 d	34/35 d	71/72 d	91/92 d	
Incorporated	93.3	57.4	36.9	26.0	15.0	8.9	7.9	9.4 d
No incorporation	94.3	81.1	73.5	59.4	44.4	30.1	35.2	32.5 d
No Incorporation (14 days) then mixing	96.0	80.3	71.5	52.9	31.0	16.2	11.8	32.3 d (0–14 d) 13.8 d (14–92 d)

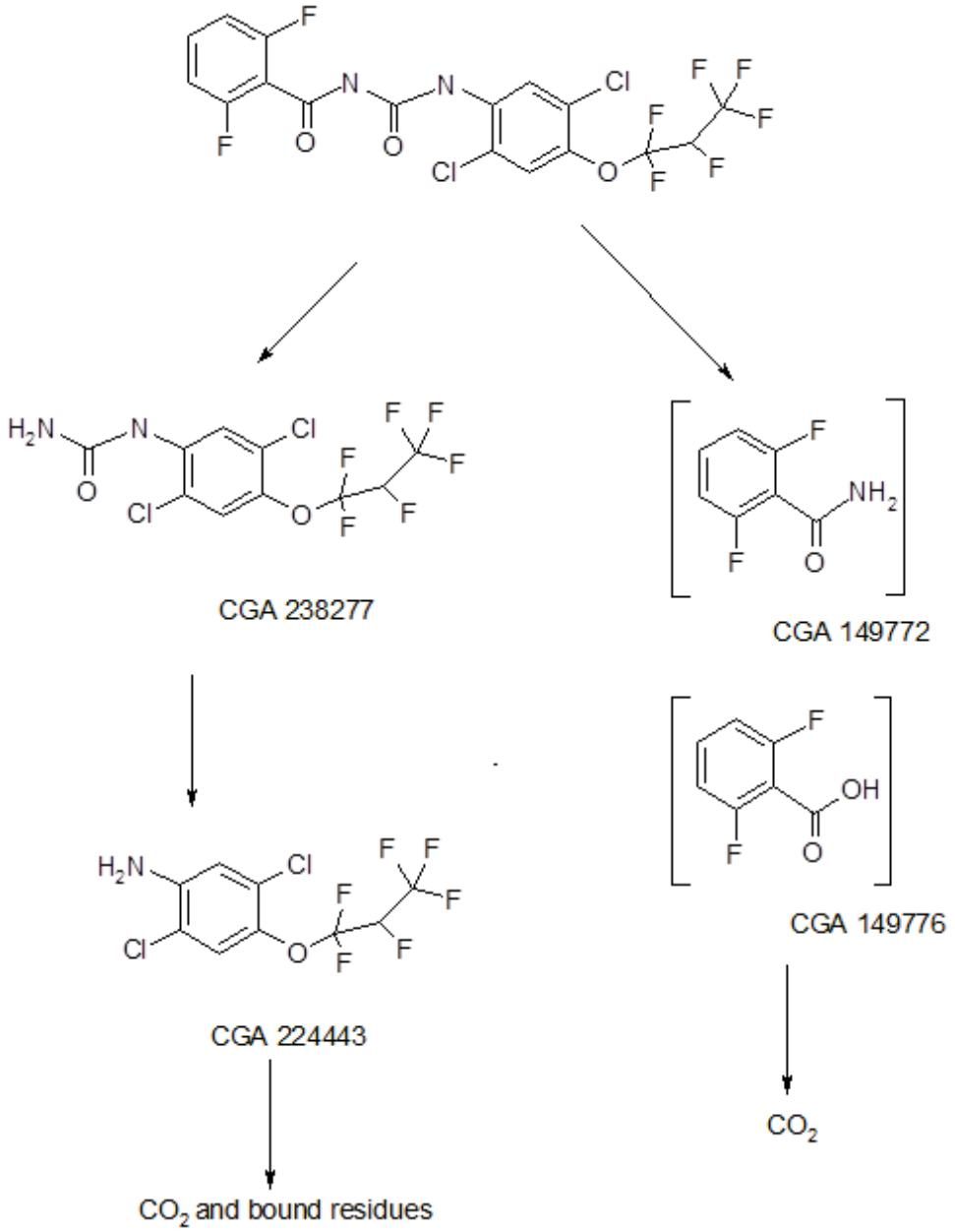


Figure 3 Proposed metabolic pathway of lufenuron in soil (aerobic)

Besides the parent substance the behaviour of the soil metabolite CGA149772 (2,6-difluorobenzamide) under aerobic conditions was investigated by Slangen (2003, LUFEN_034) in three different soil types using ^{14}C-phenyl ring-labelled CGA149772.

Soil was extracted by shaking with acetonitrile: acetic acid 98:2 following two more extractions with a mixture of acetonitrile: water 80:20 (v/v). Finally, the soil was extracted with water. The remaining soil debris was extracted with acetonitrile in a Soxhlet for six hours. All the supernatants were evaporated to aqueous and analysed by LSC followed by two different normal phase TLC methods and HPLC where possible. The sample of each soil type with the highest bound residue remaining after extraction was subjected to organic matter fractionation.

Ref.: Slangen (2003, LUFEN_034)

Test material: ^{14}C-phenyl-2,6-difluorobenzamide — Dose rate: 0.4 mg/kg

Duration: 120 days — Temp: 20 °C

Moisture: 45% MHC — Soil: Borstel

pH 5.14 — Organic carbon: 1.0

Half-live (CGA149772): 4.8 days — ^{14}C accountability: 87.6–104.5%

% CGA149772 remaining: < 0.1% after 120 days

% mineralisation: max. 59.5% after 56 days

% unextracted: max. 37.9% after 56 days

Metabolites	Max (% TRR)	Day
CGA149776	50.9	14

Ref.: Slangen (2003, LUFEN_034)

Test material: ^{14}C-phenyl-2,6-difluorobenzamide — Dose rate: 0.4 mg/kg

Duration: 120 days — Temp: 20 °C

Moisture: 45% MHC — Soil: Gartenacker

pH 7.23 — Organic carbon: 2.35

Half-live (CGA149772): 2.7 days — ^{14}C accountability: 90.5–102.8%

% CGA149772 remaining: < 0.1% after 120 days

% mineralisation: max. 62.8% after 120 days

% unextracted: max. 39.1% after 28 days

Metabolites	Max (% TRR)	Day
CGA149776	29.3	7

Ref.: Slangen (2003, LUFEN_034)

Test material: ^{14}C-phenyl-2,6-difluorobenzamide — Dose rate: 0.4 mg/kg

Duration: 120 days — Temp: 20 °C

Moisture: 45% MHC — Soil: Weide

pH 7.58 — Organic carbon: 1.94

Half-live (CGA149772): 4.0 days ^{14}C accountability: 93.6–103.1%

% CGA149772 remaining: < 0.1% after 120 days

% mineralisation: max. 64.6% after 120 days

% unextracted: max. 41.4% after 28 days

Metabolites	Max (% TRR)	Day
CGA149776	24.7	14

Soil degradation

The soil degradation of [^{14}C]dichlorophenyl-lufenuron and its primary metabolites CGA224443 and CGA238277 under varying moisture and temperature was investigated by Gonzalez-Valero (1991, LUFEN_031). A silt loam soil type (Les Evouettes) was incubated under different conditions described in the following table. For each condition, an amount of 0.1 mg ai/kg or 1 mg ai/kg soil was applied.

Table 13 Incubation conditions

Moisture content	Temperature (°C)	Concentration
30% field capacity	20	0.1 and 1.0 mg/kg
60% field capacity	20	0.1 and 1.0 mg/kg
60% field capacity	10	0.1 and 1.0 mg/kg

Based on these conditions, the following amounts of lufenuron, CGA224443 and CGA238277 were recovered.

Table 14 Radioactivity recovered as lufenuron in% AR (mean of both application rates)

	Sampling Interval (days)									
Test Conditions	0	7	14	21	28	42	60	90	120	180
60% FC, 20 °C	98.1	73.3	49.0	35.9	29.4	15.0	13.5	13.6	10.7	–
60% FC, 10 °C	98.8	89	76.9	–	52.8	42.1	30.2	23.3	18.3	13.3
30% FC, 20 °C	96.5	84.5	73.2	58.8	53.4	37.4	31.8	22.2	17.9	13.4

Table 15 Radioactivity recovered as CGA238277 in% AR (mean of both application rates)

	Sampling Interval (days)									
Test Conditions	0	7	14	21	28	42	60	90	120	180
60% FC, 20 °C	0	18.9	29.0	30.2	24.3	12.6	8.0	7.3	3.9	–
60% FC, 10 °C	0	7.25	14.4	–	24.7	26.5	27.5	21.2	15.8	10.6
30% FC, 20 °C	0	9.4	11.9	12.2	12.2	12.6	8.25	6.4	5	2.8

Table 16 Radioactivity recovered as CGA224443 in% AR (mean of both application rates)

	Sampling Interval (days)									
Test Conditions	0	7	14	21	28	42	60	90	120	180
60% FC, 20 °C	0	6.3	13.5	17.0	19.5	26.3	23.3	17.7	17.4	–
60% FC, 10 °C	0	2.8	5.8	–	9.1	16.8	23.0	26.0	28.3	24.7
30% FC, 20 °C	0	4.1	7.75	14.1	12.8	15.5	17.1	16.8	12.8	11.6

The modelling of DT_{50}- and DT_{90}-values based on this study was conducted by Sapiets (2003, LUFEN_032). By using first-order compartment models (FOMC) the following values were estimated:

Table 17 Calculated DT_{50}- and DT_{90}-values for lufenuron, CGA224443 and CGA238277

Compound	Model	DT_{50} (days)	DT_{90} (days)
Lufenuron	FOMC	13.7	81.1
CGA238277	FOMC	12.8	42.5
CGA224443	FOMC	35.8	118.8

Plant metabolism

The fate of lufenuron in plants was investigated following foliar spray application of [dichlorophenyl-14C]- and/or [difluorophenyl-14C]-radiolabelled active substance to tomato, cabbage and cotton.

In all samples unchanged lufenuron was the only residue compound detected, mainly present on the surface of the treated plant parts. No significant translocation was observed after treatment or direct stem injection. After several weeks, an uptake of the residue in treated leaves was observed, however the extracts contained lufenuron solely. In very minor amounts CGA238277 was detected at levels of 3.3% TRR or less. A proposed metabolic pathway scheme is presented in Figure 4.

Tomato

The metabolism of lufenuron was investigated in tomatoes after three spray applications with [dichlorophenyl-^{14}C]-lufenuron by Stingelin (1992, LUFEN_019). Fruit bearing plants were treated with rates equivalent to 0.03 kg ai/ha per application with one week intervals. The plants were kept in protected environments. Samples were collected from the same four plants 1 h after the first treatment, and 1 h, 12 d and 28 d after the final application (dissipation experiment). Foliage and mature fruits of four additional plants were collected 28 days after the final treatment to investigate the distribution and degradation of lufenuron.

In a second experiment four single fruits were treated by injection of 34 µg lufenuron. The fruits were sampled after 18 and 33 days.

The tomato fruits were washed three times (1 minute) in acetone (250 mL) to solubilise surface radioactivity; the levels of radioactivity in the washing were determined by liquid scintillation counting (LSC). The washed tomato fruits were frozen and homogenised under liquid nitrogen and the total radioactive residues (TRR) determined by combustion and LSC.

Extraction of the radioactive residues in the homogenised plant material was carried out using methanol-water (80:20, v/v) for two hours. This procedure was repeated until the radioactivity of the last extract was less than 5% of the first extract (maximum five extraction steps). Any remaining residues were subjected to Soxhlet extraction and finally unextracted residues were determined by combustion.

Extracts and washings were analysed by thin layer chromatography. Reference markers were visualized under UV light and areas of radioactivity detected using a radiochromatogram camera.

In all fruit and leaves samples from the foliar spray experiments most of the radioactivity was recovered in the surface wash, presenting 74–100% of the TRR. Minor amounts were also recovered primarily by methanol/water extraction, adding to total recoveries of radioactivity of 96–118% TRR. Lufenuron was the major residue identified in the combined surface wash and extracts, representing 93–99% of the TRR. In the extracts of fruits sampled 28 DALT, traces of CGA238277 were identified at 0.2% of the TRR (see Tables 18 Table and 19).

In mature fruits receiving a direct injection of lufenuron the results were comparable, with 90–95% of the radioactivity identified as unchanged lufenuron. Again CGA238277 was identified in minor amounts up to 2% of the TRR, and 5% of the total radioactivity remained unextracted.

Table 18 Summary of the distribution of radioactivity and residual [dichlorophenyl-^{14}C]-lufenuron in tomato fruits (dissipation experiment)

	1 hour after Application 1	1 hour after Application 3	12 days after Application 3	28 days after Application 3
TRR	0.58 mg eq/kg	1.216 mg eq/kg	0.84 mg eq/kg	0.694 mg eq/kg
Surface wash (surf.)	99.6% TRR	98.6% TRR	95.9% TRR	93.6% TRR
Methanol/water extraction (extr.)	Not analysed	3.5% TRR	10.0% TRR	1.7% TRR
Soxhlet extraction (extr.)	Not analysed	< 0.1% TRR	0.1% TRR	0.1% TRR
Lufenuron in combined extracts (surf.+extr.)	Not analysed	1.209 mg eq/kg (99.4% TRR)	0.822 mg eq/kg (97.9% TRR)	0.644 mg eq/kg (92.8% TRR)
CGA238277 (extr. only)	Not detected	Not detected	Not detected	0.2% TRR
Unextracted	Not analysed	0.1% TRR	0.1% TRR	0.2% TRR
Total (surf. + extr. + unextr.)	100% TRR	102.2% TRR	106.1% TRR	95.9% TRR

Table 19 Summary of the distribution of radioactivity and residual [dichlorophenyl-^{14}C]-lufenuron in tomato foliage and fruits (distribution and degradation experiment)

	Foliage (28 d DALT)	Green fruits (28 d DALT)	Red fruits (28 d DALT)	Combined fruits (28 d DALT)
TRR	0.467 mg eq/kg	0.03 mg eq/kg	0.44 mg eq/kg	0.199 mg eq/kg
Surface wash	Not determined	73.7% TRR	89.9% TRR	88.5% TRR
Methanol/water extraction	116.9% TRR	Not analysed	12.2% TRR	Not analysed
Soxhlet extraction	0.7% TRR	Not analysed	0.5% TRR	Not analysed
Lufenuron in combined extracts	0.444 mg eq/kg (95.1% TRR)	0.028 mg eq/kg (93.3% TRR)	0.43 mg eq/kg (97.7% TRR)	0.194 mg eq/kg (97.5% TRR)
Unextracted	0.6% TRR	Not analysed	0.2% TRR	Not analysed
Total (surf. + extr. + unextr.)	118.2% TRR	100% TRR	102.8% TRR	Not analysed

Cabbage

Cabbage plants (white cabbage) in a greenhouse were treated by Krauss (1994, LUFEN_020) with three spray applications of 0.02 kg ai/ha each (0.06 kg ai/ha total) in two week intervals using [dichlorophenyl-^{14}C]-lufenuron. Samples were taken one hour after the first and last application, and at crop maturity, 28 days after the last application. At each sampling the heads were separated into old/wrapper leaves and remaining heads.

Homogenised plant material was extracted five times with methanol-water (80:20, v/v) or until the radioactivity of the last extract was less than 5% of first extraction. Further extraction of the plant material was carried out using Soxhlet extraction with methanol. The amount of radioactivity in extracts was determined using liquid scintillation counting (LSC) and by combustion LSC of solid materials.

The nature of the residues in cabbage extracts was elucidated using normal and reverse phase thin layer chromatography. Reference markers were visualised under UV light and areas of radioactivity detected using a radiochromatogram camera.

In cabbage samples most of the radioactivity was present in part of the heads directly affected by the spray solution. Whole cabbage and older leaves gave TRR levels between 0.5–1.8 mg eq/kg, while the inner head contained lower radioactive residues of 0.2–0.3 mg eq/kg, and 89–101% of the TRR were extracted by methanol/water. In the extracts, unchanged parent lufenuron was the only major residue representing 88–98% of the TRR. The only other metabolite identified was CGA238277, representing up to 3.3% of the TRR (see Table 20).

Table 20 Summary of the distribution of radioactivity and residual [dichlorophenyl-^{14}C]-lufenuron in cabbage

	1 hour after Appl. 1	1 hour after application 3 (last application)		28 days after application 3 (last application)	
	Whole cabbage	Head cabbage	Old leaves	Head cabbage	Old leaves

	1 hour after Appl. 1	1 hour after application 3 (last application)		28 days after application 3 (last application)	
	Whole cabbage	Head cabbage	Old leaves	Head cabbage	Old leaves
TRR	0.501 mg eq/kg	0.301 mg eq/kg	1.659 mg eq/kg	0.195 mg eq/kg	1.790 mg eq/kg
Methanol/water extraction	90.1% TRR	100.7% TRR	89.3% TRR	96.9% TRR	96.3% TRR
Soxhlet extraction	0.9% TRR	2.3% TRR	1.7% TRR	4.7% TRR	3.0% TRR
Total extracts					
Start	1.0% TRR [a]	3.0% TRR [a]	–	–	1.3% TRR [a]
CGA238277	–	–	–	0.6% TRR [a]	3.3% TRR [a]
Lufenuron	0.446 mg eq/kg (89.0% TRR)	0.296 mg eq/kg (97.9% TRR)	1.46 mg eq/kg (88.0% TRR)	0.19 mg eq/kg (97.5% TRR)	1.702 mg eq/kg (95.1% TRR)
Unresolved	0.5% TRR [a]	1.1% TRR [a]	1.5% TRR [a]	1.6% TRR [a]	1.3% TRR [a]
Unextracted	0.1% TRR	0.2% TRR	0.1% TRR	0.5% TRR	0.4% TRR
Total (surf. + extr. + unextr.)	91.1% TRR	103.2% TRR	91.1% TRR	102.7% TRR	103.1% TRR

[a] Concentration not quantified in TLC system

Cotton

The investigation on the metabolism of lufenuron in cotton under glasshouse conditions was reported in two studies. In the first study by Stingelin (1991, LUFEN_021) [dichlorophenyl-^{14}C]-lufenuron formulated as EC50 product was applied with three spray applications at a rate equivalent to 0.03 kg ai/ha (total seasonal application rate 0.09 kg ai/ha). The first application was made at the beginning of flowering and further applications made at 14-day intervals. Sampling of leaves took place 1 hour, 1 day, 3 and 7 days after the first application and 14 days, 28 and 84 days (maturity) after the last application. At maturity, plants were also separated into stalks, leaves (old and new), green bolls, hulls, fibre and seeds

In addition, four cotton plants were injected (into the stalks) with radiolabelled lufenuron (100 µg) dissolved in acetone (2 µL). Two further injections were made at 14-day intervals. Harvested cotton plants from the injection experiment were separated into similar components, i.e. stalks, (region of the injection and remainder) leaves (old and new), green bolls, hulls, fibre and seeds.

All plants were kept in plastic containers in greenhouse.

At each interval, from the foliar application, the leaves were washed three times with a mixture of acetone-water (50:50; v/v). The washed leaves were then homogenized in the presence of methanol water (80:20, v/v).

The components from the mature cotton plants were homogenized in the presence of “dry ice” or after freezing with liquid nitrogen; in the case of dry hulls the samples were homogenized in a mill. For extraction of the radioactive residues, the homogenised plant material was suspended in a mixture of methanol-water (80:20; v/v). This procedure was repeated until the radioactivity of the last extract was equal or less than 5% of the radioactivity contained in the first extract.

The amount of radioactivity in extracts and post-extraction solids was determined using liquid scintillation counting (LSC) and by combustion LSC. The nature of the residues in extracts was elucidated using silica gel 60 F thin layer chromatography. Reference markers were visualised under UV light (254 nm) and areas of radioactivity detected using a radiochromatogram camera.

In cotton leaves most of the residue was recovered in the surface wash, however at the end of the experiment (84 DALT) approximated half of the radioactivity was present in the washed leaf extracts. In total, the extraction rates of leaves and other plant parts was high, leaving less than 3% unextracted. In the combined extracts, unchanged lufenuron was the only

residue identified in leaves, stalks and hulls, representing 89–100% of the TRR. Fibre, seeds and bolls did not contain sufficient radioactivity for identification (TRR ≤ 0.001 mg eq/kg).

Table 21 Summary of the distribution of radioactivity and residual [dichlorophenyl-^{14}C]-lufenuron in cotton foliage

	Leaves (1 hour after Appl. 1)	Leaves (1 day after Appl. 1)	Leaves (3 days after Appl. 1)	Leaves (7 days after Appl. 1)	Leaves (14 DALT)	Leaves (28 DALT)	Leaves (84 DALT)
TRR	2.453 mg eq/kg	2.374 mg eq/kg	1.79 mg eq/kg	0.64 mg eq/kg	3.334 mg eq/kg	2.74 mg eq/kg	4.912 mg eq/kg
Surface wash (surf.)	98.0% TRR	86.5% TRR	71.5% TRR	76.9% TRR	62.9% TRR	45.2% TRR	42.5% TRR
Methanol/water extraction (extr.)	1.9% TRR	13.2% TRR	28.1% TRR	22.8% TRR	35.6% TRR	52.6% TRR	54.3% TRR
Lufenuron in combined extracts (surf.+extr.)	2.406 mg eq/kg (98.1% TRR)	2.251 mg eq/kg (94.8% TRR)	1.646 mg eq/kg (91.9% TRR)	0.593 mg eq/kg (92.7% TRR)	3.102 mg eq/kg (93.0% TRR)	2.491 mg eq/kg (90.9% TRR)	4.364 mg eq/kg (88.8% TRR)
Unextracted	0.1% TRR	0.3% TRR	0.4% TRR	0.4% TRR	1.4% TRR	2.2% TRR	3.2% TRR
Total (surf. + extr. + unextr.)	100.0% TRR	100.0% TRR	100.0% TRR	100.1% TRR	99.9% TRR	100.0% TRR	100.0% TRR

Table 22 Summary of the distribution of radioactivity and residual [dichlorophenyl-^{14}C]-lufenuron in various cotton plant parts at maturity (84 DALT)

	Old Leaves	New Leaves	Stalks	Hulls	Fibre	Seeds	Green Bolls
TRR	1.487 mg eq/kg	0.014 mg eq/kg	0.026 mg eq/kg	0.092 mg eq/kg	< 0.001 mg eq/kg	< 0.001 mg eq/kg	0.001 mg eq/kg
Surface wash (surf.)	43.6% TRR	–	–	–	–	–	–
Methanol/water extraction (extr.)	58.7% TRR	109.4% TRR	116.2% TRR	103.9% TRR	n.a.	n.a.	n.a.
Soxhlet (extr.)	0.9% TRR	4.0% TRR	1.9% TRR	1.2% TRR	n.a.	n.a.	n.a.
Lufenuron in combined extracts (surf.+extr.)	1.415 mg eq/kg (95.2% TRR)	0.014 mg eq/kg (100% TRR)	0.026 mg eq/kg (100% TRR)	0.091 mg eq/kg (98.9% TRR)	n.a.	n.a.	n.a.
Unextracted	1.6% TRR	2.7% TRR	2.1% TRR	1.6% TRR	n.a.	n.a.	n.a.
Total (surf. + extr. + unextr.)	104.8% TRR	116.1% TRR	120.2% TRR	106.7% TRR	–	–	–

n.a.=Not analysed

The translocation experiment following stem injection showed that most of the applied radioactivity remained at the injection site (81.2% AR). Into close stalks (13.3% AR) and leaves (1.6-3.9% AR) a minor translocation was observed. In all samples the unchanged parent was the only residue identified (approximately 95–98% TRR).

In a second study conducted by Gentile (1991, LUFEN_022) cotton grown in greenhouse was treated with [^{14}C]difluorophenyl-lufenuron formulated as an EC50 product. Eight cotton plants were separately treated with three spray applications at a rate equivalent to 0.03 g ai/ha each (total seasonal application rate 0.09 g ai/ha). The first application was made at two months after sowing (no growth stage reported) and further applications made two and four weeks after the first application.

Sampling (three leaves from four plants) took place 2 hours after each application. At maturity, 52 days after the last application, plants were separated into stems, leaves (old and new), hulls, fibre and seeds.

At each interval from the foliar application, the leaves were washed twice with acetonitrile (surface wash). The washed leaves were then homogenized in the presence of acetonitrile-water (80:20, v/v). The unextracted radioactive residues were determined by combustion and liquid scintillation counting (LSC).

The components from the mature cotton plants were homogenized in the presence of liquid nitrogen. Radioactive residues in the homogenised plant material were extracted with acetonitrile-water (80:20, v/v). The procedure was repeated until the radioactivity of the last extract was equal or less than 5% of the radioactivity contained in the first extract. Residues remaining in the plant material were solubilised using Soxhlet extraction with acetonitrile. The amount of radioactivity in extracts was determined using liquid scintillation counting (LSC) and by combustion LSC in solid materials. The nature of the residues in extracts was elucidated using silica gel 60 F thin layer chromatography. Reference markers were visualised under UV light (254 nm) and areas of radioactivity detected using a TLC scanner.

In the leaves sampled at each interval at least 49% of the radioactivity was found in the surface wash. The total recovery of radioactivity was high, leaving less than 2% of the TRR unextracted. In the combined extracts unchanged lufenuron was the only residue identified, representing at least 92% of the TRR.

In other matrices (old leaves, stems, hulls and fibre) the methanol/water extract released the major part of the residue. Again, only unchanged lufenuron was present in the extracts at levels of 78.7–83.1% TRR. In seeds and new grown leaves the TRR was too low for further identification (0.003–0.005 mg eq/kg).

For a summary of the results please refer to Table 23.

Table 23 Summary of the distribution of radioactivity and residual [difluorophenyl-^{14}C]-lufenuron in various cotton plant parts at maturity (52 DALT)

Interval	Matrix	Total Residues [mg eq/kg]	Parent		Surface wash [% TRR]	Extracts			Total Rad. [% TRR]
			[mg eq/kg]	[% TRR]		Met./Water extract [% TRR]	Soxhlet extract [% TRR]	PES [% TRR]	
2 hours after Appl. 1	Leaves Plant 1	1.907	–	96.8	91.4	8.2	0.0	0.3	100
	Leaves Plant 2	2.379	–	96.6	92.2	7.5	0.1	0.2	100
	Leaves Plant 3	4.068	–	97.0	89.6	10.0	0.1	0.3	100
	Leaves Plant 4	4.592	–	96.1	92.4	7.1	0.1	0.3	100
	Leaves Mean	3.237	–	–	–	–	–	–	–
2 hours after Appl. 2	Leaves Plant 1	2.434	–	95.8	63.0	35.9	0.3	0.8	100
	Leaves Plant 2	5.103	–	97.0	83.2	16.0	0.1	0.6	100
	Leaves Plant 3	4.715	–	95.6	77.6	21.5	0.2	0.6	100
	Leaves Plant 4	6.233	–	95.3	79.1	20.1	0.2	0.6	100
	Leaves Mean	4.621	–	–	–	–	–	–	–
2 hours after Appl. 3	Leaves Plant 1	3.153	–	97.3	88.0	11.3	0.1	0.6	100
	Leaves Plant 2	3.777	–	97.3	76.1	22.5	0.2	1.2	100
	Leaves Plant 3	2.663	–	96.6	90.0	8.9	0.2	0.9	100
	Leaves Plant 4	2.342	–	96.2	81.4	17.6	0.1	0.8	100
	Leaves Mean	2.984	–	–	–	–	–	–	–
Maturity 52 DALT	Leaves Plant 1	1.85	–	92.1	49.2	48.8	0.8	1.2	100
	Leaves Plant 3	5.95	–	93.0	57.7	39.8	1.0	1.5	100
	Old leaves	2.089	1.95	93.3	n.p.	98.8	1.3	1.6	101.7
	New leaves	0.005	n.a.	n.a.	n.p.	n.a.	n.a.	n.a.	n.a.
	Stems	0.124	0.103	83.1	n.p.	91.7	1.5	1.2	94.4
	Hulls	0.687	0.541	78.7	n.p.	84.0	1.4	1.3	86.7
	Fibre	0.028	0.023	82.1	n.p.	91.7	1.7	5.5	98.9
	Seeds	0.003	n.a.	n.a.	n.p.	n.a.	n.a.	n.a.	n.a.

PES=Post-extraction solids

n.a. = Not analysed
n.p. = Not performed

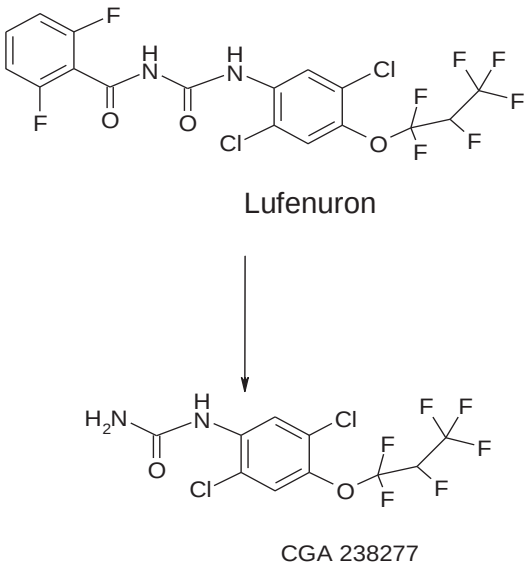


Figure 4 Proposed metabolic pathway of lufenuron in plants

Confined rotational crop studies

For the investigation of lufenuron in rotational crops two studies were conducted involving application of either [^{14}C]difluorophenyl- or [^{14}C]dichlorophenyl-lufenuron. The experiments using [^{14}C]difluorophenyl-lufenuron was conducted by Gentile (1992, LUFEN_023). Plant containers kept in a glasshouse received application to bare soil equivalent to 0.15 kg ai/ha. Lettuce, spring wheat, maize and carrots were planted in the treated soil 63 days after test substance application. Immature and mature samples of the crops were taken throughout the study and soil samples were taken at each sampling.

Fresh samples were homogenised in the presence of liquid nitrogen and dry plant parts, e.g. grain, were homogenised in a mill. For extraction of the radioactive residues the homogenised plant material was suspended in a mixture of acetonitrile-water (80:20; v/v). This procedure was repeated until the radioactivity of the last extract was equal or less than 5% of the radioactivity contained in the first extract. Non-extracted residues were solubilised using Soxhlet extraction with acetonitrile. The amount of radioactivity in extracts was determined using liquid scintillation counting (LSC) and by combustion LSC in solid materials.

The nature of the residues in extracts was elucidated using silica gel 60 F thin-layer chromatography. Reference markers were visualised under UV light (254 nm) and areas of radioactivity detected using a radiochromatogram camera.

The transfer of radioactivity into lettuce, wheat, maize and carrots grown as succeeding crops was very limited. In mature lettuce (126 d after treatment) the highest TRR levels of 0.047 mg eq/kg were found. 53% of the TRR was identified as unchanged parent (0.025 mg/kg). In other matrices only wheat straw (0.023 mg eq/kg, 0.007 mg lufenuron/kg) and immature carrots roots (0.023 mg eq/kg, no identification conducted) showed total radioactive residues above 0.01 mg eq/kg. No further identification was conducted for these matrices. In soil, nearly the entire extracted radioactivity was attributed to lufenuron. No further metabolites could be identified against the reference compounds CGA149772 or CGA149776.

Table 24 Distribution of total radioactivity and residues of lufenuron in succeeding lettuce grown in soil treated at a rate equivalent to 0.15 kg [^{14}C]difluorophenyl-lufenuron per ha

Days after treatment	Soil layer	Total residues		Parent	Extracted radioactivity		Unextracted	Total
					Cold	Soxhlet		
(PBI: 63 d)		[mg eq/kg]	[% TRR]	[mg eq/kg (% TRR)]	[% TRR]	[% TRR]	[% TRR]	[% TRR]
63	SOIL							
	0–5 cm	0.206	93.9	0.146	76.3	0.9	25.1	102.3
	5–10 cm	0.009	3.9	(70.8)	n.a.	n.a.	n.a.	n.a.
	10–20 cm	0.003	2.1	n.a.	n.a.	n.a.	n.a.	n.a.
	Total	0.066	100	n.a.	–	–	–	–
				–				
99	SOIL							
	0–5 cm	0.239	99.1	0.151	70.0	0.9	26.8	97.7
	5–10 cm	0.002	0.6	(63.2)	n.a.	n.a.	n.a.	n.a.
	10–20 cm	< 0.001	0.3	n.a.	n.a.	n.a.	n.a.	n.a.
	Total	0.087	100	n.a.	–	–	–	–
				n.a.				
	HEADS	0.004	100	n.a.	n.a.	n.a.	n.a.	n.a.
126	SOIL							
	0–5 cm	0.269	89.0	0.176	69.5	1.0	27.6	98.1
	5–10 cm	0.044	10.0	(65.4)	65.9	1.1	31.8	98.8
	10–20 cm	0.005	1.0	0.027	n.a.	n.a.	n.a.	n.a.
	Total	0.134	100	(61.4)	–	–	–	–
				n.a.				
				n.a.				
	HEADS	0.047	100	0.025 (53.2)	75.0	1.7	43.4	120.1

n.a.=Not analysed

Table 25 Distribution of total radioactivity and residues of lufenuron in succeeding wheat grown in soil treated at a rate equivalent to 0.15 kg [^{14}C]difluorophenyl-lufenuron per ha

Days after treatment	Soil layer	Total residues		Parent	Extracted radioactivity		Unextracted	Total
					Cold	Soxhlet		
(PBI: 63 d)		[mg eq/kg]	[% TRR]	[mg eq/kg (% TRR)]	[% TRR]	[% TRR]	[% TRR]	[% TRR]
63	SOIL							
	0–5 cm	0.221	94.4	0.155	75.0	1.0	26.1	102.1
	5–10 cm	0.012	4.3	(70.1)	75.9	1.4	29.2	106.5
	10–20 cm	0.002	1.4	0.009 (75)	n.a.	n.a.	n.a.	n.a.
	Total	0.071	100	n.a.	–	–	–	–
				–				
99	SOIL							
	0–5 cm	0.128	95.4	0.087 (68)	72.2	1.1	23.0	96.3
	5–10 cm	0.006	4.2	n.a.	n.a.	n.a.	n.a.	n.a.
	10–20 cm	< 0.001	0.4	n.a.	n.a.	n.a.	n.a.	n.a.
	Total	0.046	100	–	–	–	–	–
	WHOLE TOPS	0.005	100	n.a.	n.a.	n.a.	n.a.	n.a.
126	SOIL							
	0–5 cm	0.212	94.7	0.127	63.8	0.9	26.7	91.4
	5–10 cm	0.01	4.4	(59.9)	57.4	1.7	42.3	101.4
	10–20 cm	0.001	1.0	0.005 (50)	n.a.	n.a.	n.a.	n.a.
	Total	0.063	100	n.a.	–	–	–	–
				–				
	WHOLE TOPS	0.002	100	n.a.	n.a.	n.a.	n.a.	n.a.

Days	Soil layer	Total residues		Parent	Extracted radioactivity		Unextracte	Total
161	SOIL							
	0–5 cm	0.167	99.6	0.114	73.4	0.7	26.0	100.1
	5–10 cm	< 0.001	0.3	(68.3)	n.a.	n.a.	n.a.	n.a.
	10–20 cm	< 0.001	0.1	n.a.	n.a.	n.a.	n.a.	n.a.
	Total	0.063	100	n.a.	–	–	–	–
				–				
	STALKS	0.023	100	0.007	65.8	0.5	32.3	98.6
				(30.4)				
	HUSKS	0.002	100	n.a.	n.a.	n.a.	n.a.	n.a.
	GRAIN	0.007	100	n.a.	n.a.	n.a.	n.a.	n.a.

n.a.=Not analysed

Table 26 Distribution of total radioactivity and residues of lufenuron in succeeding maize grown in soil treated at a rate equivalent to 0.15 kg [^{14}C]difluorophenyl-lufenuron per ha

Days after treatment	Soil layer	Total residues		Parent	Extracted radioactivity		Unextracte d	Total
					Cold	Soxhlet		
(PBI: 63 d)		[mg eq/kg]	[% TRR]	[mg eq/kg (% TRR)]	[% TRR]	[% TRR]	[% TRR]	[% TRR]
63	SOIL							
	0–5 cm	0.405	96.4	0.311	81.6	0.6	18.6	100.8
	5–10 cm	0.019	3.4	(76.8)	79.3	1.1	23.0	103.4
	10–20 cm	< 0.001	0.2	0.014	n.a.	n.a.	n.a.	n.a.
	Total	0.14	100	(73.7)	–	–	–	–
				n.a.				
				–				
99	SOIL							
	0–5 cm	0.186	98.0	0.148	83.7	1.1	23.0	107.8
	5–10 cm	0.003	1.3	(79.6)	n.a.	n.a.	n.a.	n.a.
	10–20 cm	0.001	0.7	n.a.	n.a.	n.a.	n.a.	n.a.
	Total	0.066	100	n.a.	–	–	–	–
				–				
	TOPS	< 0.001	100	n.a.	n.a.	n.a.	n.a.	n.a.
126	SOIL							
	0–5 cm	0.23	97.8	0.138	63.9	1.1	31.3	96.3
	5–10 cm	0.004	1.6	(60.0)	n.a.	n.a.	n.a.	n.a.
	10–20 cm	< 0.001	0.6	n.a.	n.a.	n.a.	n.a.	n.a.
	Total	0.069	100	n.a.	–	–	–	–
				–				
	TOPS	0.002	100	n.a.	n.a.	n.a.	n.a.	n.a.
197	SOIL							
	0–5 cm	0.107	97.7	0.063	63.6	1.2	36.5	101.3
	5–10 cm	< 0.001	0.6	(58.9)	n.a.	n.a.	n.a.	n.a.
	10–20 cm	0.003	1.8	n.a.	n.a.	n.a.	n.a.	n.a.
	Total	0.047	100	n.a.	–	–	–	–
				–				
	STALKS	0.008	100	n.a.	n.a.	n.a.	n.a.	n.a.
	COBS	0.003	100	n.a.	n.a.	n.a.	n.a.	n.a.
	GRAIN	0.004	100	n.a.	n.a.	n.a.	n.a.	n.a.

n.a.=Not analysed

Table 27 Distribution of total radioactivity and residues of lufenuron in succeeding carrots grown in soil treated at a rate equivalent to 0.15 kg [^{14}C]difluorophenyl-lufenuron per ha

Days after treatment	Soil layer	Total residues		Parent	Extracted radioactivity		Unextracte d	Total
					Cold	Soxhlet		
(PBI:		[mg	[% TRR]	[mg eq/kg	[% TRR]	[% TRR]	[% TRR]	[% TRR]

Days	Soil layer	Total residues		Parent	Extracted radioactivity		Unextracte	Total
63 d)		eq/kg]		(% TRR)]				
63	SOIL 0–5 cm 5–10 cm 10–20 cm Total	 0.169 0.015 0.001 0.006	 92.1 7.2 0.6 100	 0.128 (75.7) 0.009 (60.0) n.a. –	 79.9 71.6 n.a. –	 1.0 1.0 n.a. –	 24.5 30.0 n.a. –	 105.4 102.6 n.a. –
99	SOIL 0–5 cm 5–10 cm 10–20 cm Total	 0.111 < 0.001 < 0.001 0.041	 99.0 0.5 0.5 100	 0.068 (61.3) n.a. n.a. –	 64.7 n.a. n.a. –	 1.3 n.a. n.a. –	 33.5 n.a. n.a. –	 99.5 n.a. n.a. –
	WHOLE TOPS	0.008	100	n.a.	n.a.	n.a.	n.a.	n.a.
126	SOIL 0–5 cm 5–10 cm 10–20 cm Total	 0.136 0.001 0.002 0.047	 97.7 0.9 1.4 100	 0.077 (56.6) n.a. n.a. –	 62.3 n.a. n.a. –	 1.2 n.a. n.a. –	 36.0 n.a. n.a. –	 99.5 n.a. n.a. –
	WHOLE TOPS	0.008	100	n.a.	n.a.	n.a.	n.a.	n.a.
	ROOTS	0.023	100	n.a.	n.a.	n.a.	n.a.	n.a.
197	SOIL 0–5 cm 5–10 cm 10–20 cm Total	 0.184 0.002 0.001 0.06	 97.8 1.4 0.8 100	 0.085 (46.2) n.a. n.a. –	 50.4 n.a. n.a. –	 0.9 n.a. n.a. –	 45.3 n.a. n.a. –	 96.6 n.a. n.a. –
	WHOLE TOPS	0.005	100	n.a.	n.a.	n.a.	n.a.	n.a.
	ROOTS	0.005	100	n.a.	n.a.	n.a.	n.a.	n.a.

n.a.=Not analysed

In a second confined study in the field conducted by Stingelin (1992, LUFEN_024) [^{14}C]dichlorophenyl-lufenuron was applied to bare soil one at a rate equivalent to 0.13 kg ai/ha. After different plant-back intervals (PBI) lettuce (PBI 76 d), winter wheat (PBI 126 d), sugar beets (PBI 306 d) and maize (PBI 331 d) were planted/sown and grown to maturity. In addition soil samples from layers up to 30 cm depth were collected and analysed for residues.

Fresh samples were homogenised in the presence of liquid nitrogen and dry plant parts, e.g. grain, were homogenised in a mill. After homogenisation samples were combusted and the levels of radioactivity were measured by liquid scintillation counting (LSC).

None of the plant samples were extracted since the radioactive residues were < 0.01 mg/kg.

In soil samples most of the radioactivity was recovered in the first 5 cm soil layer (55–96% AR). At the end of the study (519 days after treatment) up to 27.9% AR moved into the 5–10 cm layer and up to 24.7% to the 10–20 cm layer. The transfer into even lower layers was minimal (< 7% AR). The analysis of the upper layers revealed lufenuron as the major residue. The only metabolites identified were CGA238277 and CGA224443, both not exceeding 0.014 mg eq/kg.

Table 28 Distribution of total radioactivity of lufenuron in succeeding crops grown under field conditions in soil treated at a rate equivalent to 0.13 kg [^{14}C]dichlorophenyl-lufenuron per ha

Crop/Plant-back interval	Matrix	Days after soil treatment	Days after planting/sowing	TRR in mg eq/kg

Lettuce (PBI 76 d)	Heads, immature	30	106	0.004
	Heads, mature	62	138	0.001
Wheat (PBI 126 d)	Whole tops	182	56	0.003
	Whole tops	307	181	< 0.001
	Whole tops	363	237	< 0.001
	Stalks	418	292	0.004
	Husks	418	292	0.001
	Grain	418	292	< 0.001
Sugar beets	Immature roots	363	57	0.002
(PBI 306 d)	Immature tops	363	57	0.002
	Immature roots	418	112	0.001
	Immature tops	418	112	< 0.001
	Roots	519	213	< 0.001
	Tops	519	213	< 0.001
Maize (PBI 331 d)	Whole tops	363	32	0.002
	Whole tops	418	87	< 0.001
	Stalks	495	164	0.003
	Cobs	495	164	< 0.001
	Grain	495	164	< 0.001

Animal metabolism

The Meeting received metabolism studies on laboratory animals, poultry and lactating goats using the difluorophenyl- and the dichlorophenyl-label of lufenuron.

The metabolism of lufenuron in livestock animals was minimal, showing only unchanged parent substance in all goat matrices. In poultry minor amounts of CGA149772 and CGA238277 were found in edible commodities, however at levels below 10% TRR or 0.01 mg eq/kg. Most of the radioactive residue was present in fat tissue, egg yolk and milk.

Laboratory animals

Lactating goats

The metabolic fate of lufenuron in lactating goats was investigated using [^{14}C]difluorophenyl- or [^{14}C]dichlorophenyl-lufenuron (Cameron, 1992, LUFEN_018 & Schulze-Aurich, 1992, LUFEN_017). The compound was administered to one lactating goat for each label in gelatine capsules at 5.4 ppm for the difluorophenyl-label (0.135 mg/kg body weight) and 6.0 ppm for the dichlorophenyl-label (0.15 mg/kg body weight) for ten consecutive days. Excreta and milk were collected daily. The animals were slaughtered approximately 24 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 by combustion and liquid scintillation counting. The composition of samples was investigated two months after sampling. Thin-layer chromatography was used to identify and characterize radioactive components in sample extracts.

The total recovery of the administered radioactivity was 95% for both labels. The majority of the radioactivity (73–74%) was found in the faeces. Radioactive residues in the edible tissues were 0.8–1.6% AR in muscle, 4.2–5.4% AR in fat, 0.28–0.3% AR in liver, 0.01–0.02% AR in kidney and 5.8–6.8% AR in milk. A summary of the recovered radioactivity is presented in Table 29.

Table 29 Radioactive residues in milk and tissues after oral administration of [^{14}C]difluorophenyl- (5.4 ppm) or [^{14}C]dichlorophenyl-lufenuron (6.0 ppm) for 10 consecutive days

	[^{14}C]\difluorophenyl-label (5.4 ppm)		[^{14}C]dichlorophenyl-label (6.0 ppm)	
Tissue	Mean radioactivity (mg/kg or mg/L lufenuron eq.)	Mean radioactivity (% of total dose)	Mean radioactivity (mg/kg or mg/L lufenuron eq.)	Mean radioactivity (% of total dose)

	[^{14}C]\difluorophenyl-label (5.4 ppm)		[^{14}C]dichlorophenyl-label (6.0 ppm)	
Tissue	Mean radioactivity (mg/kg or mg/L lufenuron eq.)	Mean radioactivity (% of total dose)	Mean radioactivity (mg/kg or mg/L lufenuron eq.)	Mean radioactivity (% of total dose)
Total milk	–	6.76	–	5.76
Muscle, hindquarter	0.066		0.039	
Muscle, forequarter	0.08	1.6	0.038	0.77
Muscle, Tenderloin	0.071		0.04	
Fat, omental	2.288		2.411	
Fat, subcutaneous	0.883	5.4	0.821	4.22
Fat, renal	2.434		1.64	
Liver	0.417	0.297	0.367	0.28
Kidney	0.114	0.017	0.118	0.014
Rumen and intestinal contents	0.35	5.04	0.75	10.1
Faeces	–	73.8	–	72.8
Cage wash	–	0.25	–	0.27
Total recovery	–	94.6	–	94.8

In milk radioactive residues approximated a plateau after one week of dosing. In the following table the total radioactivity recovered from milk is summarized.

Table 30 Mean radioactive residues in goat milk following 10 consecutive doses of [^{14}C]dichlorophenyl or [^{14}C]difluorophenyl lufenuron to lactating goats

	TRR mg eq/kg			
	[^{14}C]difluorophenyl-label (5.4 ppm)		[^{14}C]dichlorophenyl-label (6.0 ppm)	
Days of dosing	am milk	pm milk	am milk	pm milk
1	0.000	0.043	0.000	0.030
2	0.303	0.381	0.315	1.270
3	0.560	0.622	1.042	0.850
4	0.848	0.594	0.752	0.823
5	0.646	0.601	0.719	0.792
6	0.766	0.802	0.875	1.186
7	0.878	0.892	0.850	1.049
8	0.998	0.940	1.037	0.798
9	1.001	0.979	0.711	0.790
10	0.997	0.706	0.786	0.791
11	0.690	–	0.674	–

For both labels unchanged lufenuron was the only residue in tissues and milk, representing 73–94% of the TRR. Highest concentrations were present in fat tissue and milk. No separation between milk fat and skim milk was conducted.

In goat faeces and urine the majority of the residue also comprised of lufenuron. Varying levels, depending on the sampling period, of CGA238277, CGA149772 and CGA149776 were also found.

For the composition of radioactive residues in milk and tissues please see Tables 31 and 32.

Table 31 Extraction and analysis of radioactive residues in goats tissues and milk treated with [^{14}C]difluorophenyl labelled lufenuron (5.4 ppm)

	Metabolite Fractions in mg eq/kg (% TRR)				
	Fat	Muscle	Liver	Kidney	Milk
TRR	1.67	0.07	0.417	0.114	0.993
Identified					
Lufenuron (parent)	1.502 (89.9)	0.061 (87.0)	0.305 (73.1)	0.095 (83.3)	0.922 (92.8)
Unknown [a]	0.099 (6.0)	0.007 (9.5)	0.078 (18.8) [b]	0.014 (12.5)	0.066 (6.6)

	Metabolite Fractions in mg eq/kg (% TRR)				
	Fat	Muscle	Liver	Kidney	Milk
Unextracted	0.068 (4.1)	0.002 (3.5)	0.034 (8.1)	0.005 (4.2)	0.006 (0.6)

[a] Unresolved radioactivity in TLC system

[b] Two unresolved fractions

Table 32 Extraction and analysis of radioactive residues in goat tissues and milk treated with [^{14}C]difluorophenyl labelled lufenuron (6.0 ppm)

	Metabolite Fractions in mg eq/kg (% TRR)				
	Fat	Muscle	Liver	Kidney	Milk
TRR	2.02	0.039	0.367	0.118	0.737
Identified					
Lufenuron (parent)	1.817 (90)	0.035 (89.5)	0.291 (79.4)	0.105 (88.6)	0.689 (93.5)
Unknown [a]	0.14 (6.9)	0.003 (7.8)	0.043 (11.7) [b]	0.011 (9.1)	0.041 (5.6)
Unextracted	0.063 (3.1)	0.001 (2.7)	0.033 (8.9)	0.003 (2.3)	0.007 (0.9)

[a] Unresolved radioactivity in TLC system

[b:] Two unresolved fractions

Laying hens

The metabolic fate of lufenuron was investigated using [^{14}C]difluorophenyl- or [^{14}C]dichlorophenyl-lufenuron (Cameron, 1992, LUFEN_016 & Schulze-Aurich, 1992, LUFEN_017). For each label the compound was administered in gelatine capsules to three laying hens at doses of 3.4 ppm for the difluorophenyl-label (representing 2.6 mg/kg body weight) and of 5.2 ppm for the dichlorophenyl-label (representing 3.5 mg/kg body weight) for fourteen consecutive days. Excreta and eggs were collected daily. The animals were slaughtered approximately 24 hours after the last dose. Muscle, skin with attached fat, peritoneal fat, liver, kidney and content of gastrointestinal tract were collected.

Radioactivity was measured by combustion and liquid scintillation counting. The composition of samples was investigated two months after sampling. Thin-layer chromatography was used to identify and characterize radioactive components in sample extracts.

The total recovery of the administered radioactivity was 75–79%. The majority of the radioactivity (54–62%) was found in the excreta. Radioactive residues in the edible tissues were 0.55–1.15% AR in lean meat, 5.1–9.9% AR in fat, 0.4–0.58% AR in liver, 0.07% AR in kidney and 8.7–9.6% AR in eggs. A summary of the recovered radioactivity is presented in Table 33.

Table 33 Radioactive residues in eggs and tissues after oral administration of [^{14}C]difluorophenyl- (3.4 ppm) or [^{14}C]dichlorophenyl-lufenuron (5.2 ppm) for 14 consecutive days

	[^{14}C]difluorophenyl-label (3.4 ppm)		[^{14}C]dichlorophenyl-label (5.2 ppm)	
Tissue	Mean radioactivity (mg/kg or mg/L lufenuron eq.)	Mean radioactivity (% of total dose)	Mean radioactivity (mg/kg or mg/L lufenuron eq.)	Mean radioactivity (% of total dose)
Total egg	–	8.69	–	9.64
Lean meat	0.237	1.15	0.104	0.55
Skin + fat	2.56	Not calculated	1.296	Not calculated
Peritoneal fat	13.04	8.83	7.189	5.09
Liver	1.45	0.64	0.828	0.4
Kidney	0.737	0.09	0.524	0.07
Blood	0.292	0.14	0.189	0.1
Intestinal contents	–	0.15	–	0.21
Excreta	–	62.18	–	53.5
Cage wash	–	1.45	–	1.27

	[^{14}C]difluorophenyl-label (3.4 ppm)		[^{14}C]dichlorophenyl-label (5.2 ppm)	
Tissue	Mean radioactivity (mg/kg or mg/L lufenuron eq.)	Mean radioactivity (% of total dose)	Mean radioactivity (mg/kg or mg/L lufenuron eq.)	Mean radioactivity (% of total dose)
Total recovery	–	78.95	–	75.49

In eggs radioactive residues were mainly present in the egg yolk for both labels. A plateau was approximated at the end of the 14 days dosing period in the yolk while residues in egg white remained stable after more than 4 days. In the following table the total radioactivity recovered from egg white and egg yolk is summarized:

Table 34 Mean radioactive residues in hen egg following 14 consecutive doses of [^{14}C]dichlorophenyl or [^{14}C]difluorophenyl lufenuron to laying hens

	TRR mg eq/kg			
	[^{14}C]difluorophenyl-label (3.4 ppm)		[^{14}C]dichlorophenyl-label (5.2 ppm)	
Days of dosing	Egg white	Egg yolk	Egg white	Egg yolk
1	0.000	0.000	0.001	0.000
2	0.000	0.158	–	–
3	0.001	0.566	0.003	0.474
4	0.003	1.635	0.005	1.065
5	0.003	2.301	–	–
6	0.003	3.802	0.005	2.419
7	0.005	6.334	0.011	3.966
8	0.002	5.258	0.009	3.973
9	0.005	5.507	0.016	6.133
10	–	–	0.007	4.766
11	0.002	7.441	0.015	6.565
12	0.008	7.110	0.008	7.585
13	0.003	6.555	0.008	8.048
14	0.002	6.470	0.008	8.479

For both labels unchanged lufenuron was the major residue in all tissues and eggs, representing at least 79.3% of the TRR. Highest concentrations were present in poultry fat and egg yolk.

The only other metabolites identified were CGA149772 for the difluorophenyl-label (egg white, 0.001 mg eq/kg, 17.3% TRR) and CGA238277 for the dichlorophenyl-label (kidney and egg white, < 0.001–0.028 mg eq/kg, 5.3–7% TRR).

In hen excreta > 90% TRR was extracted. Lufenuron was the major component of the residue, i.e. > 82%. No other component accounted for > 5% TRR, CGA238277 represented 3% TRR and CGA149776 for < 4.3% TRR.

For the composition of radioactive residues in eggs and tissues please refer to Tables 35 and 36.

Table 35 Extraction and analysis of radioactive residues in hen tissues and eggs treated with [^{14}C]difluorophenyl labelled lufenuron (3.4 ppm)

	Metabolite Fractions in mg eq/kg (% TRR)					
	Fat	Liver	Kidney	Lean meat	Egg yolk	Egg white
TRR	9.763	1.451	0.737	0.237	8.048	0.008
Identified						
Lufenuron (parent)	9.148 (93.7	1.337 (92.1)	0.588 (79.8)	0.196 (82.6)	7.179 (89.2)	0.003 (37.6)
CGA149772	–	–	–	–	–	0.001 (17.3)
Unknown [a]	0.469 ((4.8)	0.087 (6.0)	0.128 (17.4)	0.029 (12.4)	0.249 (3.1)	0.003 (42.1)
Unextracted	0.146 (1.5)	0.028 (1.9)	0.021 (2.8)	0.012 (5.0)	0.62 (7.7)	< 0.001 (3.0)

[a] Unresolved radioactivity in TLC system

Table 36 Extraction and analysis of radioactive residues in hen tissues and eggs treated with [^{14}C]dichlorophenyl labelled lufenuron (5.2 ppm)

	Metabolite Fractions in mg eq/kg (% TRR)					
	Fat	Liver	Kidney	Lean meat	Egg yolk	Egg white
TRR	4.148	0.828	0.524	0.104	6.555	0.003
Identified						
Lufenuron (parent)	3.795 (91.5)	0.705 (85.1)	0.415 (79.3)	0.089 (85.7)	6.135 (93.6)	0.001 (44.1)
CGA238277	–	–	0.028 (5.3)	–	–	< 0.001 (7.0)
Unknown [a]	0.262 (6.3)	0.069 (8.3)	0.055 (10.6)	0.011 (10.7)	0.197 (3.0)	0.001 (37.4)
Unextracted	0.091 (2.2)	0.055 (6.6)	0.026 (4.9)	0.004 (3.6)	0.223 (3.4)	< 0.001 (11.4)

[a] Unresolved radioactivity in TLC system

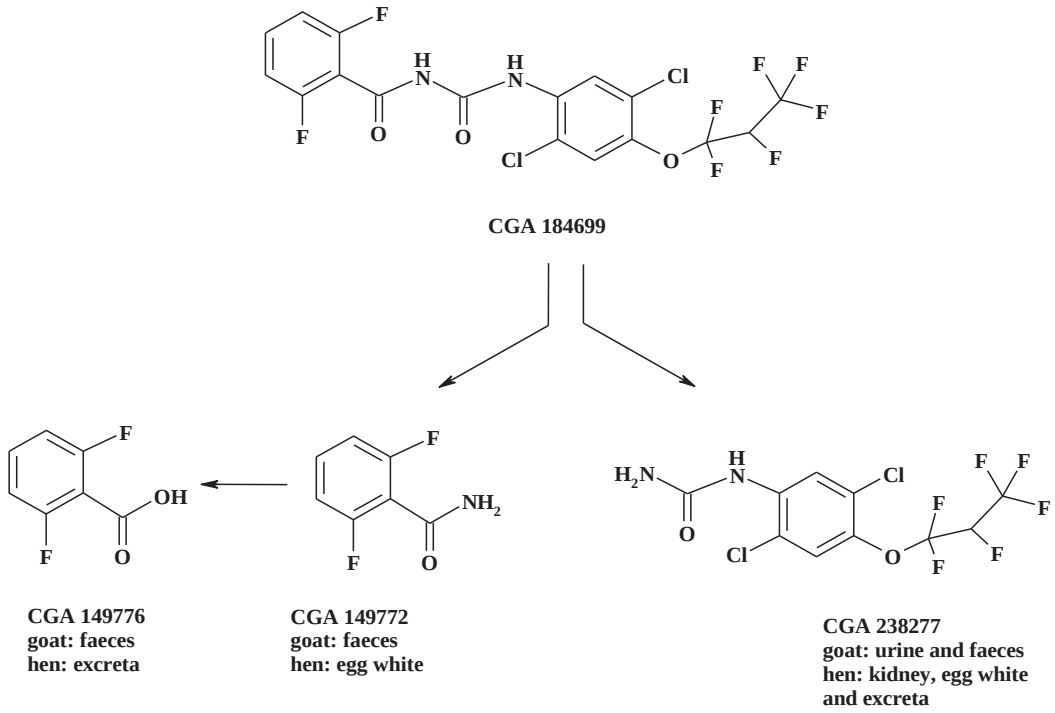


Figure 5 Metabolic pathway of lufenuron (CGA184699) in animals

RESIDUE ANALYSIS

Analytical methods

For lufenuron analytical methods were provided for plant and animal matrices. All plant matrices were validated with an LOQ of 0.01 mg/kg. For animal commodities a general LOQ of 0.02 mg/kg was validated.

The applicability of multi residue methods was confirmed on basis of DFG S19 for plant and animal matrices (LOQ 0.02 mg/kg for all commodities).

Table 37 Overview of analytical methods for lufenuron

Method	Matrix	Extraction	Clean-Up	Detection, LOQ

Method	Matrix	Extraction	Clean-Up	Detection, LOQ
REM 118.01 & modification REM 118.07	high water acidic	Methanol, partitioning against hexane/diethyl ether (9:1,v:v)	cyano SPE	REM 118.01: HPLC-UV (255 nm), LOQ: 0.02 mg/kg REM 118.07: HPLC-MS/MS m/z: 509.1 → 326.0 LOQ: 0.01 mg/kg
POPIT MET.015	High oil Difficult (coffee)	water, saturated sodium chloride solution, and hexane: ethyl ether (9:1,v:v)	C_{18} SPE	HPLC-UV, 255 nm LOQ: 0.02 mg/kg
POPIT MET.077.Rev05	High water High oil Difficult (coffee)	water, saturated sodium chloride solution, and hexane: ethyl ether (9:1,v:v)	C_{18} SPE	HPLC-MS/MS m/z 511.09 → 158.2 LOQ: 0.01 mg/kg
POP PAT 004 V01/V04	Dry High oil	water, saturated sodium chloride solution, and hexane: ethyl ether (9:1,v:v)	none	HPLC-MS/MS m/z 511.09 → 158.2 LOQ: 0.01 mg/kg
MRM DFG S19	High water Acidic Dry High oil	See DFG S19	See DFG S19	HPLC-MS/MS m/z 509 → 326 & 509 → 175 LOQ: 0.02 mg/kg
REM 118.04	Animal tissues Milk Blood	Fat/milk: acetonitrile Others: methanol	Silica gel SPE	HPLC-UV, 255 nm LOQs: Milk: 0.001 mg/kg Blood: 0.002 mg/kg Liver, kidney: 0.01 mg/kg Meat: 0.02 mg/kg Fat: 0.1 mg/kg
MRM DFG S19	Milk Eggs Animal tissues	See DFG S19	See DFG S19	HPLC-MS/MS m/z 509 → 326 & 509 → 175 LOQ: 0.02 mg/kg

Plant materials

Method REM 118.01 (Altenburger, 1988, LUFEN_035; Clarke, 2004, LUFEN_036) and Method REM 118.07 (Clarke, 2005, LUFEN_037)

Lufenuron residues were extracted from plant material by maceration in the presence of methanol. The extract filtered and diluted with water and sodium chloride solution. Lufenuron is partitioned into hexane/diethyl ether (9/1; v/v); the organic phase is reduced in volume, redissolved in hexane, and "cleaned up" using solid phase extraction on a cyano SPE cartridge (REM 118.01 only). The concentration of lufenuron is determined using HPLC-UV detection at 255 nm (REM 118.01) and in the current version HPLC-MS/MS (REM 118.07).

Table 38 Recovery data for method REM 118.01 (HPLC-UV: 255 nm) and its modification REM 118.01 (LC-MS/MS: m/z: 509.1 → 326.0) measuring lufenuron in plant matrices

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Reference, MRM transition
Tomato	0.01	5	92–108	98	10	Clarke (2004, LUFEN_035; 2005,
	0.1	5	87–109	96	11	LUFEN_037)
Oranges	0.01	5	71–85	78	8	m/z: 509.1 → 326.0
	0.1	5	69–84	77	9	
Grapes	0.01	5	82–94	88	6	
	0.1	5	83–89	85	3	
Tomato	0.02	5	77–95	86	9	Altenburger (1988, LUFEN_035)
	0.2	5	77–110	90	13	UV: 255 nm
Grapes	0.02	5	74–102	89	14	
	0.2	5	82–111	102	12	

Method POPIT MET.015 (Anonymous, 2002, LUFEN_038)

Method POPIT MET.015 provides for the determination of lufenuron in coffee beans and soybeans. The frozen raw sample is prepared by milling the whole sample with dry ice until the complete homogenization. 10 g of the sample is homogenized with 80 mL of methanol by milling. An 8 mL aliquot of the sample is mixed with 8 mL of water, 4 mL of saturated sodium chloride solution, and 4 mL of hexane: ethyl ether (9:1) solution, for a final volume of 24 mL. The upper layer is transferred to another vessel, evaporated at 40 °C, and re-dissolved in 2.5 mL hexane. The sample is cleaned up by silica solid phase extraction (SPE). The sample solution collected is evaporated at 40 °C, and dissolved in 2 mL of hexane: isopropanol: methanol (90:5:5) solution. The final sample solution is analysed by LC UV (255 nm).

Table 39 Recovery data for method POPIT MET.015 in coffee and soybeans

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Reference
Coffee beans	0.02	8	93–107	101	6	Anonymous (2002, LUFEN_038)
	0.2	4	86–92	90	3	HPLC-UV: 255 nm
Soybeans	0.02	7	82–87	84	3	
	0.2	5	71–76	75	3	

Method POPIT MET.077.Rev05 (Anonymous, 2008, LUFEN_039)

Method POPIT MET.077 provides for the determination of lufenuron in cotton, coffee, sunflowers, peaches sugarcane and sugar cane litter. The frozen sample is prepared by milling the whole sample with dry ice until the complete homogenization. 5 g of the sample is homogenized with 40 mL of methanol by milling. An 8 mL aliquot of the sample is mixed with 8 mL of water, 4 mL of saturated sodium chloride solution, and 4 mL of hexane: ethyl ether (9:1) solution, for a final volume of 24 mL. The upper layer is transferred to another vessel. The clean-up is repeated and a 4 mL aliquot of the hexane: ethyl ether (9:1) solution is added to the remaining layer. The upper layer is combined with the initial extract. This extract is evaporated at 40 °C, and re-dissolved in 2.5 mL hexane. The sample is then cleaned up by silica solid phase extraction (SPE) and analysed by LC-MS/MS.

Table 40 Recovery data for method POPIT MET.077.Rev05 in plant matrices

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Reference, MRM transition
Cotton seed	0.01	7	75–82	80	3	Anonymous (2008, LUFEN_039)
	0.1	6	75–82	79	3	m/z 511.09 → 158.2
Coffee beans	0.01	8	91–109	100	7	
	0.1	6	96–106	101	4	
Sunflower seed	0.01	8	87–102	95	5	
	0.1	6	82–93	89	6	
Peach	0.01	7	83–91	86	3	
	0.1	5	101–106	104	2	
	2.5	5	82–84	83	1	
Sugar cane	0.01	7	83–104	93	8	
	0.1	5	103–108	106	2	
Sugar cane litter	0.01	8	70–83	76	6	
	0.1	6	86–92	89	2	

Method POP PAT 004 V01/V04 (Anonymous, 2010, LUFEN_040)

Method POP PAT 004 provides for the determination of lufenuron in maize and soy. The frozen raw sample is prepared by milling the whole sample with dry ice until homogenous. The sample is homogenized with 20 mL of methanol by milling. A 4 mL aliquot of the sample is mixed with 4 mL of water, 4 mL of saturated sodium chloride solution, and 4 mL of hexane: ethyl ether (9:1) solution, to a final volume of 16 mL. The upper layer is transferred to another vessel. The clean-up is repeated, and a 4 mL aliquot of the hexane:ethyl ether (9:1) solution is added to the remaining layer. The upper

layer is combined with the initial extract. The combined sample is evaporated at 40 °C, re-dissolved in 1 mL methanol and analysed by LC-MS/MS.

Table 41 Recovery data for method POP PAT 004 V01/V04

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Reference, MRM transition
Maize grain	0.01	5	74–97	81	11	Anonymous (2010, LUFEN_040)
	0.1	5	71–73	72	1	m/z: 511 → 158 & m/z: 511 → 141
Soybean seeds	0.01	5	77–95	86	9	
	0.1	5	72–104	84	15	

Multi-residue method DFG S19 (extended revision) (Anspach, 2002, LUFEN_042 & Schulz, 2003, LUFEN_043)

A method, based on the DFG S19 (extended revision) multi-method, for routine monitoring of lufenuron in samples of plant material has been validated.

Lufenuron residues are extracted using module E1 for orange and tomato, E2 for wheat grain followed by clean up procedures according to module GPC (gel permeation chromatography). All samples are analysed by high performance liquid chromatography with tandem mass spectrometric detection, HPLC-MS/MS (m/z: 509 → 326 & m/z: 509 → 175).

Table 42 Recovery data for the multi-residue method DFG S19 in plant commodities

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Reference, MRM transition
Tomato	0.02	5	75–82	79	3	Anspach (2002, LUFEN_042)
	0.2	5	78–86	84	4	m/z: 509 → 326
Orange	0.02	5	69–85	79	8	
	0.2	5	74–88	80	7	
Maize grain	0.02	5	65–79	75	8	
	0.2	5	81–93	86	6	
Tomato	0.02	5	92–106	100	6	Schulz (2003, LUFEN_043)
	0.2	5	81–106	98	6	m/z: 509 → 326
Oilseed rape seeds	0.02	5	91–110	100	7	
	0.2	5	94–110	104	6	
Orange	0.02	5	77–93	86	6	
	0.2	5	81–90	86	3	
Maize grain	0.02	5	79–86	83	4	
	0.2	5	82–90	86	4	

Animal materials

Method REM 118.04 (Tribolet, 1995, LUFEN_041)

REM 118.04 provides for the determination of lufenuron in tissues, fat and milk. Tissue samples are extracted by maceration (liver and kidney) or shaking (meat) with methanol. In the case of fat, the sample is melted and lufenuron residues are extracted by shaking with acetonitrile. The extract is filtered and the acetonitrile reduced in volume. The residue is re-dissolved in methanol.

Milk samples are diluted with acetonitrile to precipitate proteins, filtered and the acetonitrile reduced in volume. The residue is re-dissolved in methanol.

The extracts, for all commodities, are diluted with water and sodium chloride solution. Lufenuron is partitioned into hexane/diethyl ether (9/1; v/v) and the organic phase is reduced in volume, re-dissolved in hexane, and “cleaned up” using solid phase extraction on a silica gel cartridge.

The concentration of lufenuron is determined by HPLC-UV at 255 nm.

Table 43 Recovery data for method REM 118.04 in animal matrices

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Reference
Milk	0.001	12	74–112	96	11	Tribolet (1995, LUFEN_041)
	0.01	9	86–116	94	11	HPLC-UV: 255 nm
Meat	0.01	7	61–112	82	25	
	0.02	2	86–89	88	–	
	0.1	4	81–84	83	2	
Liver	0.01	4	87–100	92	7	
	0.02	2	63–86	75	–	
	0.1	4	87–111	94	12	
Kidney	0.01	4	105–144	119	15	
	0.02	2	108–113	111	–	
	0.1	4	103–106	104	1	
Fat	0.01	11	53–109	76	23	
	0.1	4	68–80	72	8	
Blood	0.002	12	68–97	85	11	
	0.02	9	83–105	89	10	

Multi-residue method DFG S19 (extended revision) (Anspach, 2003, LUFEN_044 & Schulz, 2003, LUFEN_045)

A method, based on the DFG S19 (extended revision) multi-method, for samples of tissues, milk and eggs has been validated. For samples of milk, meat and eggs, samples are extracted with acetone. Water is added prior to extraction to maintain a ratio of 2/1 (v/v), taking into account the natural water content of the matrices. Ethyl acetate/cyclohexane (1/1; v/v) and sodium chloride are added and the mixture homogenised. An aliquot of the organic phase is applied and cleaned up using gel permeation chromatography. In the case of fat, samples are mixed with synthetic calcium silicate after addition of acetone and acetonitrile. An aliquot of the organic phase was cleaned up on gel permeation chromatography.

All samples were analysed for residues of lufenuron by high performance liquid chromatography with tandem mass spectrometric detection, HPLC-MS/MS (m/z: 509 → 326 & m/z: 509 → 175).

Table 44 Recovery data for the multi-residue method DFG S19 in animal commodities

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Reference, MRM transition
Milk	0.02	5	79–101	87	10	Anspach (2002, LUFEN_042)
	0.2	5	69–94	83	13	m/z: 509 → 326
Meat	0.02	5	69–84	78	7	
	0.2	5	71–93	79	11	
Eggs	0.02	5	78–129	104	17	
	0.2	5	79–101	88	11	
Fat	0.02	5	67–78	72	6	
	0.2	5	80–91	86	6	
Milk	0.02	5	76–102	87	14	Schulz (2003, LUFEN_043)
	0.2	5	105–117	109	5	m/z: 509 → 326
Meat	0.02	5	62–84	76	13	
	0.2	5	76–91	85	7	

Stability of pesticides in stored analytical samples

Plant matrices

Tribolet (1993, LUFEN_046)

Samples of cotton seed, cabbage and orange were fortified with lufenuron at a concentration of 0.5 mg/kg and stored under –18 °C. The samples were stored in plastic and glass vessels, however no difference between both materials was observed. Samples were taken for analysis at intervals up to 24 months in parallel to freshly fortified samples to estimate the procedural recovery. Analysis of the samples was performed according to the method REM 118.01.

In the study report the results for the stored samples are only reported as percentage of the fortified level corrected by the procedural recoveries. No measured concentrations were described.

Table 45 Recovered lufenuron residues in stored plant commodities after storage up to 24 months (Tribolet, 1993, LUFEN_046)

Matrix	Fortification level (mg/kg)	Storage period (months)	Residue level in stored samples corrected by procedural recoveries		Procedural recovery	
			Individual corrected values (% fortified)	Mean (%)	Individual values (%)	Mean (%)
Cottonseed	0.5	0	–	–	94, 92	93
		0.5	96, 97, 99, 100, 102, 105	100	94, 92	93
		1	94, 98, 98, 106	99	94, 92	93
		3	100, 103, 103, 104	103	89, 88	89
		6	102, 103, 108, 110	106	78, 89	84
		12	93, 95, 97, 98	96	91, 92	92
		24	98, 101, 103, 106	102	90, 90	90
Cabbage	0.5	0	–	–	83, 93	88
		0.5	89, 97, 98, 98, 100, 102	97	87, 87	87
		1	104, 107, 109, 110	108	85, 84	85
		3	101, 101, 102, 102	102	92, 92	92
		6	99, 100, 101, 102	101	87, 89	88
		12	95, 97, 98, 106	99	94, 94	94
		24	99, 99, 114, 115	107	79, 83	81
Orange	0.5	0	–	–	91, 92	92
		0.5	95, 97, 97, 98, 98, 106	99	90, 94	92
		1	97, 97, 100, 102	99	91, 92	92
		3	101, 103, 106, 112	106	91, 94	93
		6	88, 93, 95, 95	93	88, 89	89
		12	99, 99, 108, 108	104	89, 91	90
		24	99, 101, 103, 116	105	88, 89	89

Animal matrices

Tribolet (1995, LUFEN_047)

Storage stability of residues of lufenuron in bovine tissues and milk were conducted to support the data from the livestock feeding study. Samples of bovine muscle, liver, kidney, fat, milk and blood were fortified with lufenuron at a concentration of 0.2 mg/kg in tissues, 0.02 mg/kg in milk and 0.04 mg/kg in blood. Samples were stored at –18 °C for a period of 9 months, which covered the sample storage time in the study. Analysis of the samples (in triplicate) was performed according to the method REM 118.04.

Table 46 Residues of lufenuron in animal commodities after storage at –18 ºC (Tribolet, 1995, LUFEN_047)

Matrix	Forti	Storage	Lufenuron

	fication level (mg/kg)	period (months)	Residue level in stored samples		Procedural recovery
			Individual values in mg/kg (mean)	% nominal	%
Muscle	0.2	9	0.13, 0.14, 0.14 (0.14)	70	75
Liver	0.2	9	0.14, 0.14, 0.16 (0.15)	75	84
Kidney	0.2	9	0.14, 0.15, 0.16 (0.15)	75	90
Fat	0.2	9	0.14, 0.16, 0.17 (0.16)	80	77
Milk	0.02	9	0.015, 0.016, 0.016 (0.016)	80	79
Blood	0.04	9	0.032, 0.037, 0.041 (0.037)	93	90

USE PATTERN

Lufenuron is an insect growth inhibitor that is active against larvae of Lepidoptera and Coleoptera. It is used in a vegetable crops, oilseeds, root crops maize, sugarcane and coffee close to harvest.

Table 47 List of uses of lufenuron

Crop	Country	Application detail					
		Indoor/ Outdoor	Type	kg ai/ha	Growth stage at last treatment	No	PHI
Citrus fruit							
Citrus fruit	BR	Outdoor	Foliar spray	0.004 kg ai/hL	At infestation	1	28
Citrus fruit	CN	Outdoor	Foliar spray	0.033	At infestation	2	28
Pome fruit							
Apple	BR	Outdoor	Foliar spray	0.005 kg ai/hL	At infestation	4	14
Apple	CN	Outdoor	Foliar spray	0.05	At infestation	3	14
Stone fruit							
Peaches	BR	Outdoor	Foliar spray	0.005 kg ai/hL	At infestation	3	10
Brassica vegetables							
Cabbage	BR	Outdoor	Foliar spray	0.005 kg ai/hL	At infestation	2	7
Cabbage	CN	Outdoor	Foliar spray	0.03	At infestation	2	14
Fruiting vegetables—cucurbits							
Cucumber	BR	Outdoor	Foliar spray	0.0025 kg ai/hL	At infestation	4	7
Cucumber	ES	Indoor	Foliar spray	0.1	At infestation	2	7
Melon	ES	Indoor	Foliar spray	0.1	At infestation	3	7
Watermelon	ES	Indoor	Foliar spray	0.1	At infestation	3	7
Fruiting vegetables—other than cucurbits							
Pepper	ES	Indoor	Foliar spray	0.1	At infestation	3	7
Tomato	BR	Outdoor	Foliar spray	0.004 kg ai/hL	At infestation	4	10
Tomato	CN	Outdoor	Foliar spray	0.045	At infestation	2	7
Tomato	ES	Indoor	Foliar spray	0.1	At infestation	3	7
Leafy vegetables							
Lettuce	ES	Indoor	Foliar spray	0.03	At infestation	3	7

Crop	Country	Application detail					
		Indoor/ Outdoor	Type	kg ai/ha	Growth stage at last treatment	No	PHI
Pulses							
Beans	CN	Outdoor	Foliar spray	0.038	At infestation	3	7
Soybean	BR	Outdoor	Foliar spray	0.02	At infestation	2	35
Root and tuber crops							
Cassava	BR	Outdoor	Foliar spray	0.015	At infestation	3	7
Potato	BR	Outdoor	Foliar spray	0.04	At infestation	4	14
Potato	BR	Outdoor	Foliar spray	0.002	At infestation	3	14
Cereal grains							
Maize	BR	Outdoor	Foliar spray	0.015	At infestation	1	35
Wheat	BR	Outdoor	Foliar spray	0.005	At infestation	2	14
Grasses for sugar or syrup productions							
Sugar cane	BR	Outdoor	Foliar spray	0.02	At infestation	2	14
Tree nuts							
Coconut	BR	Outdoor	Foliar spray	0.0025 kg ai/h L	At infestation	1	14
Oilseeds							
Cotton	BR	Outdoor	Foliar spray	0.05	At infestation	1	28
Cotton	CN	Outdoor	Foliar spray	0.045	At infestation	2	28
Sunflower	BR	Outdoor	Foliar spray	0.015	At infestation	3	14
Seed for beverages and sweets							
Coffee	BR	Outdoor	Foliar spray	0.04	At infestation	2	7

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Residue levels were reported as measured. Application rates were always reported as lufenuron equivalents. When residues were not detected they are shown as below the LOQ, e.g., < 0.01 mg/kg. Application rates, spray concentrations and mean residue results have generally been rounded to the even with 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.

Lufenuron—supervised residue trials

Commodity	Indoor/Outdoor	Treatment	Countries	Table
Cucumber	Indoor	Foliar	France, Greece, Spain	48
Melons	Indoor	Foliar	Spain	49

Commodity	Indoor/Outdoor	Treatment	Countries	Table
Sweet Pepper	Indoor	Foliar	Greece, Italy, Spain	50
Tomato	Indoor	Foliar	Greece, Spain, Switzerland	51
Sweet corn	Outdoor	Foliar	Brazil	52
Soybeans	Outdoor	Foliar	Brazil	53
Potatoes	Outdoor	Foliar	Brazil	54
Maize	Outdoor	Foliar	Brazil	55
Sugarcane	Outdoor	Foliar	Brazil	56
Cotton	Outdoor	Foliar	China	57
Coffee	Outdoor	Foliar	Brazil	58

Table 48 Residues of lufenuron following foliar application to protected cucumbers

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	lufenuron	Report/Trial No., Reference, analytical method, validation data, storage period
France, Montfavet 2003 (Defens)	EC	2	0.12	0.01	89	Fruits	0 1 3 7 10	0.05 0.03 0.02 <u>0.01</u> < 0.01	03-5064, Osborne (2005, LUFEN_051) REM. 118.07, LOQ : 0.01 mg/kg, 75–79% Recovery (n=2), Storage: 13 months
France, Saint Andiol 2003 (Tyria)	EC	2	0.12	0.01	72	Fruits	0 7	0.10 <u>0.06</u>	03-5065, Osborne (2005, LUFEN_052) REM. 118.07, LOQ : 0.01 mg/kg, 74–92% Recovery (n=2), Storage: 12 months
Greece, Kenourgio 2000 (Hana)	EC	2	0.15	0.01	88	Fruits	0 7	0.02 <u>0.03</u> (< 0.02, 0.03)	Report 1048/00, Salvi (2001, LUFEN_053) REM. 118.01, LOQ : 0.02 mg/kg, 95–96% Recovery (n=2), Storage: 4 months
Greece, Kenourgio 2001 (Aris)	EC	2	0.15	0.01	89	Fruits	0 1 3 7 14	0.17 0.19 0.12 <u>0.04</u> (0.06, 0.03) < 0.02	Report 1063/01, Gasser (2001, LUFEN_054) REM. 118.01, LOQ : 0.02 mg/kg, 97–113% Recovery (n=2), Storage: 3 months
Greece, Kenourgio 1999 (Aris)	EC	2	0.15	0.01	89	Fruits	0 1 3 7	0.04 0.06 0.02 <u>0.02</u> (< 0.02, 0.02)	Report 1096/99, Tribolet (2000, LUFEN_055) REM. 118.01, LOQ : 0.02 mg/kg, 96–105% Recovery (n=2), Storage: 4 months
Spain, Motril 2004 (Baya)	EC	2	0.15	0.01	74	Fruits	0 1 3	0.06 0.06 0.04	04-5005, ES-IR-04-003, Gardinal (2006, LUFEN_050)

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	lufenuron	Report/Trial No., Reference, analytical method, validation data, storage period
							7 10	0.03 0.02	REM. 118.07, LOQ : 0.01 mg/kg, 90% Recovery (n=2), Storage: 12 months
Spain, Los Palacios 2000 (Torres)	EC	2	0.1	0.01	87	Fruits	0 3 7 10	0.15 0.09 0.06 (0.05, 0.06) < 0.02	Report 1042/00, Salvi (2001, LUFEN_048) REM. 118.01, LOQ : 0.02 mg/kg, 100–107% Recovery (n=2), Storage: 9 months
Spain, Los Palacios 2000 (Edona)	EC	2	0.11	0.01	87	Fruits	0 3 7 10	0.12 0.07 0.02 < 0.02	Report 1043/00, Salvi (2001, LUFEN_049) REM. 118.01, LOQ : 0.02 mg/kg, 97–110% Recovery (n=2), Storage: 8 months
Spain, Los Palacios 2001 (Darina)	EC	2	0.15	0.01	81	Fruits	0 1 3 7	0.09 0.07 0.06 0.02 (0.02, 0.02)	Report 1094/01, Gasser (2003, LUFEN_056) REM. 118.01, LOQ : 0.02 mg/kg, 85–99% Recovery (n=2), Storage: 9 months
Spain, Carchuna 2001 (Marumba)	EC	2	0.15	0.01	86	Fruits	0 7	0.06 0.03 (0.03, 0.03)	Report 1095/01, Gasser (2003, LUFEN_057) REM. 118.01, LOQ : 0.02 mg/kg, 92–129% Recovery (n=2), Storage: 7 months
Spain, Calahonda 2001 (Marumba)	EC	2	0.15	0.01	86	Fruits	0 7	0.08 0.02 (< 0.02, 0.02)	Report 1096/01, Gasser (2003, LUFEN_058) REM. 118.01, LOQ : 0.02 mg/kg, 75–79% Recovery (n=2), Storage: 7 months

DAT = Days after last treatment

BBCH 71–79 = 1st–9th fruit has reached typical size

BBCH 81–88=10–80% of fruits show typical fully ripe colour

BBCH 89=Fully ripe: fruits have typical fully ripe colour

Table 49 Residues of lufenuron following foliar application to protected melons

Location, Year (variety)	Application Form.	no	kg ai/ha	kg ai/hL	BBCH	Residues, mg/kg Sample	DAT	lufenuron	Report/Trial No., Reference, analytical method, validation data, storage period
Spain, Sanlúcar de Barrameda 2000 (Prima)	EC	3	0.1	0.01	89	Whole fruit [a]	0 3 7 10	0.09 0.06 0.07 0.04	1017/00, Salvi (2001, LUFEN_059) REM. 118.01, LOQ : 0.02 mg/kg, 70–108% Recovery (n=2), Storage: 8 months Sample segmented before storage
	EC	3	0.1	0.01	89	Whole fruit [a]	0 3	0.14 0.09 (0.09, 0.09)	1019/00, Salvi (2001, LUFEN_061)

Location,	Application					Residues, mg/kg			Report/Trial No., Reference,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	analytical method, validation data, storage period
							7	0.06 (0.05, 0.06)	REM. 118.01, LOQ : 0.02 mg/kg, 109–110% Recovery (n=2), Storage: 8 months Sample segmented before storage
Spain, Sanlúcar de Barrameda 2000 (Melisa)	EC	3	0.1	0.01	89	Whole fruit [a]	0 3 7 10	0.16 0.12 (0.09, 0.15) <u>0.19</u> 0.1	1018/00, Salvi (2001, LUFEN_060) REM. 118.01, LOQ : 0.02 mg/kg, 75–91% Recovery (n=4), Storage: 7 months Sample segmented before storage
	EC	3	0.1	0.01	89	Whole fruit [a]	0 3 7	0.14 0.14 (0.105, 0.175) 0.15 (0.14, 0.16)	1020/00, Salvi (2001, LUFEN_062) REM. 118.01, LOQ : 0.02 mg/kg, 75–96% Recovery (n=4), Storage: 7 months Sample segmented before storage
Spain, Vistabella 2001 (Solarquin)	EC	3	0.1	0.01	78	Whole fruit [a] Peel Pulp	0 3 3 3	0.14 <u>0.06</u> [b] (0.06, 0.06) 0.09 (0.09, 0.09) < <u>0.02</u> (< 0.02, < 0.02)	1049/01, Gasser (2003, LUFEN_063) REM. 118.01, LOQ : 0.02 mg/kg, 95–102% Recovery (n=6), Storage: 3 months Whole fruit sample segmented before storage, peel/pulp samples separated in the field
Spain, Sanlúcar de Barrameda 2001 (Galia)	EC	3	0.1 0.11 0.1	0.01	87	Whole fruit [a] Peel Pulp	0 3 3 3	0.07 <u>0.02</u> [b] (0.02, 0.03) 0.04 (0.03, 0.05) < <u>0.02</u> (< 0.02, < 0.02)	1050/01, Gasser (2003, LUFEN_064) REM. 118.01, LOQ : 0.02 mg/kg, 95–102% Recovery (n=6), Storage: 3 months Whole fruit sample segmented before storage, peel/pulp samples separated in the field
Spain, Chipiona 2001 (Galia-F1)	EC	3	0.1	0.01	79	Whole fruit [a] Peel Pulp	0 3 7 10 3 3	0.03 0.02 [b] (0.02, 0.03) <u>0.03</u> 0.02 0.05 (0.04, 0.06) < <u>0.02</u> (< 0.02, < 0.02)	1051/01, Gasser (2003, LUFEN_065) REM. 118.01, LOQ : 0.02 mg/kg, 96–118% Recovery (n=6), Storage: 4 months Whole fruit sample segmented before storage, peel/pulp samples separated in the field
Spain, El Ejido 2001 (Siglo)	EC	3	0.1 0.09 0.1	0.01	85	Whole fruit [a]	0 3 7 10	0.13 0.12 [b] (0.1, 0.14) 0.07 <u>0.13</u>	1052/01, Gasser (2003, LUFEN_066) REM. 118.01, LOQ : 0.02 mg/kg, 96–118%

Location,	Application					Residues, mg/kg			Report/Trial No., Reference,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	analytical method, validation data, storage period
						Peel Pulp	3 3	0.18 (0.15, 0.21) < 0.02 (< 0.02, < 0.02)	Recovery (n=6), Storage: 4 months Whole fruit sample segmented before storage, peel/pulp samples separated in the field

[a] Calculated based on segment weight or peel/pulp ratio

DAT=Days after last treatment

BBCH 71–79 = 1st–9th fruit has reached typical size

BBCH 81–88=10–80% of fruits show typical fully ripe colour

BBCH 89 = Fully ripe: fruits have typical fully ripe colour

Table 50 Residues of lufenuron following foliar application to protected sweet peppers

Location,	Application					Residues, mg/kg			Report/Trial No., Reference,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	analytical method, validation data, storage period
Greece, Tyrnavos 2000 (Sammy RZ)	EC	3	0.15	0.1	89	Fruits	0 3	0.64 0.52 (0.37, 0.66)	1050/00, Salvi (2001, LUFEN_073) REM. 118.01, LOQ : 0.02 mg/kg, 94–105% Recovery (n=2), Storage: 7 months
(35-70 RZ)	EC	3	0.15	0.1	89	Fruits	0 3	0.98 0.74 (0.59, 0.88)	1051/00, Salvi (2001, LUFEN_074) REM. 118.01, LOQ : 0.02 mg/kg, 94–105% Recovery (n=2), Storage: 7 months
Greece, Tyrnavos 2001 (Sammy RZ)	EC	3	0.15	0.1	89	Fruits	0 1 3 7 14	0.21 0.34 0.25 0.18 (0.16, 0.21) 0.06	1064/01, Gasser (2003, LUFEN_075) REM. 118.01, LOQ : 0.02 mg/kg, 98–109% Recovery (n=2), Storage: 3 months
Greece, Tyrnavos 2001 (Sammy RZ)	EC	3	0.15	0.1	89	Fruits	0 7	0.67 0.42 (0.36, 0.49)	1065/01, Gasser (2003, LUFEN_076) REM. 118.01, LOQ : 0.02 mg/kg, 98–105% Recovery (n=2), Storage: 2 months
Italy, Bagnarola of Budrio 2001 (Sienor)	EC	3	0.15	0.1	82	Fruits	0 3 7 14	0.37 0.2 0.36 (0.29, 0.44) 0.23	1045/01, Gasser (2003, LUFEN_072) REM. 118.01, LOQ : 0.02 mg/kg, 93–106% Recovery (n=4), Storage: 4 months
Spain, El Mirador 1996 (Sonar)	EC	3	0.1	0.01	83	Fruits	–0 0 3 7 14 21	0.11 0.17 0.18 0.13 0.1 0.05	1013/97, Tribolet (1998, LUFEN_067) REM. 118.01, LOQ : 0.02 mg/kg, 84–108% Recovery (n=2), Storage: 3 months

Location,	Application					Residues, mg/kg			Report/Trial No., Reference,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	analytical method, validation data, storage period
Spain, El Ejido 1997 (Dulce Italiano)	EC	3	0.1	0.16 0.12 0.12	89	Fruits	–0 0 3 7 14 21	0.04 0.1 0.1 <u>0.08</u> 0.04 0.03	1015/97, Tribolet (1998, LUFEN_068) REM. 118.01, LOQ : 0.02 mg/kg, 67–95% Recovery (n=3), Storage: 9 months
Spain, El Ejido 1997 (Taranto)	EC	3	0.1	0.009	89	Fruits	–0 0 3 7 14 21	0.02 0.08 0.09 0.05 0.11 <u>0.17</u>	1016/97, Tribolet (1998, LUFEN_069) REM. 118.01, LOQ : 0.02 mg/kg, 81–92% Recovery (n=2), Storage: 4 months
(Mazurca)	EC	3	0.1	0.009	89	Fruits	–0 0 3 7 14 21	0.02 0.05 0.09 0.06 0.03 0.07	1017/97, Tribolet (1998, LUFEN_070) REM. 118.01, LOQ : 0.02 mg/kg, 65–94% Recovery (n=2), Storage: 4 months
Spain, El Ejido 1997 (Cadia)	EC	3	0.1	0.009 0.008 0.008	89	Fruits	–0 0 3 7 14 21	0.09 0.15 0.1 <u>0.13</u> 0.12 0.13	1018/97, Tribolet (1998, LUFEN_070) REM. 118.01, LOQ : 0.02 mg/kg, 83–94% Recovery (n=2), Storage: 8 months
Spain, Adra 1998 (Genil)	EC	3	0.1	0.01	89	Fruits	3 7	0.18 (0.17, 0.18) <u>0.18</u> (0.16, 0.19)	1139/98, Tribolet (1999, LUFEN_077) REM. 118.01, LOQ : 0.02 mg/kg, 85–86% Recovery (n=2), Storage: 3 months
Spain, Motril 1998 (Ciclon)	EC	3	0.1	0.01	89	Fruits	3 7	<u>0.54</u> (0.51, 0.56) 0.47 (0.47, 0.47)	1140/98, Tribolet (1999, LUFEN_078) REM. 118.01, LOQ : 0.02 mg/kg, 85–86% Recovery (n=2), Storage: 3 months

–0=Sampling before last application

DAT=Days after last treatment

BBCH 81–88 = 10–80% of fruits show typical fully ripe colour

BBCH 89=Fully ripe: fruits have typical fully ripe colour

Table 51 Residues of lufenuron following foliar application to protected tomatoes

Location,	Application					Residues, mg/kg			Report/Trial No., Reference,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	analytical method, validation data, storage period
Greece, Kenurgio 1999 (Noa)	EC	3	0.15	0.01	81	Fruits	0 7	0.04 (0.03, 0.05) 0.04 (0.03, 0.04)	1097/99, Tribolet (2000, LUFEN_090) REM. 118.01, LOQ : 0.02 mg/kg, 83–87% Recovery (n=2), Storage: 5 months
Greece, Kenourio	EC	3	0.1	0.01	89	Fruits	0 7	0.03 <u>0.02</u>	1049/00, Salvi (2001, LUFEN_085)

Location,	Application					Residues, mg/kg			Report/Trial No., Reference,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	analytical method, validation data, storage period
2000 (Noa)								(0.02, 0.02)	REM. 118.01, LOQ : 0.02 mg/kg, 79–98% Recovery (n=2), Storage: 3 months
Greece, Kenourigio 2001 (Noa)	EC	3	0.1	0.01	89	Fruits	0 7	0.23 <u>0.24</u> (0.2, 0.29)	1066/01, Gasser (2003, LUFEN_087) REM. 118.01, LOQ : 0.02 mg/kg, 100–111% Recovery (n=2), Storage: 2 months
Spain, Perellonet 1998 (Marmanda)	EC	3	0.1	0.02 0.015 0.012	72	Fruits	0 3 7 15 22	0.06 0.06 (0.05, 0.07) 0.05 <u>0.06</u> 0.06	1013/99, Tribolet (1999, LUFEN_079) REM. 118.01, LOQ : 0.02 mg/kg, 76–84% Recovery (n=2), Storage: 6 months
Spain, Cullera 1998 (Welkor)	EC	3	0.1	0.02 0.015 0.012	72	Fruits	0 3 7 15 22	0.03 0.05 (0.04, 0.06) 0.07 <u>0.08</u> 0.03	1014/99, Tribolet (1999, LUFEN_081) REM. 118.01, LOQ : 0.02 mg/kg, 83–91% Recovery (n=2), Storage: 7 months
Spain, Los Palacios 1998 (Genaro)	EC	3	0.1	0.01	81	Fruits	0 3 7 14 21	0.12 0.06 0.07 <u>0.09</u> 0.05	1051/98, Tribolet (1999, LUFEN_086) REM. 118.01, LOQ : 0.02 mg/kg, 85–95% Recovery (n=2), Storage: 5 months
Spain, Los Palacios 1999 (Bond)	EC	3	0.1	0.01	89	Fruits	0 7	0.09 (0.07, 0.11) <u>0.04</u> (0.02, 0.05)	1126/99, Tribolet (1999, LUFEN_091) REM. 118.01, LOQ : 0.02 mg/kg, 88–116% Recovery (n=2), Storage: 5 months
Spain, Los Palacios 1999 (Genaro)	EC	3	0.1	0.01	89	Fruits	0 7	0.08 (0.06, 0.09) <u>0.08</u> (0.06, 0.09)	1127/99, Tribolet (1999, LUFEN_092) REM. 118.01, LOQ : 0.02 mg/kg, 88–116% Recovery (n=2), Storage: 5 months
Spain, Perellonet 2000 (Marmanda)	EC	3	0.11	0.011	72	Fruits	0 3 7 10	0.05 0.09 <u>0.1</u> (0.07, 0.13) 0.1	1014/00, Salvi (2001, LUFEN_080) REM. 118.01, LOQ : 0.02 mg/kg, 70–110% Recovery (n=2), Storage: 9 months
Spain, Los Palacios 2000 (Bond)	EC	3	0.11 0.11 0.1	0.01	75	Fruits	0 3 7 10	0.05 0.04 <u>0.05</u> (0.04, 0.06) 0.03	1015/00, Salvi (2001, LUFEN_082) REM. 118.01, LOQ : 0.02 mg/kg, 100–108% Recovery (n=2), Storage: 9 months
Spain, Los Palacios	EC	3	0.11 0.1	0.01	75	Fruits	0 7	0.04 <u>0.04</u>	1016/00, Salvi (2001, LUFEN_083)

Location,	Application					Residues, mg/kg			Report/Trial No., Reference,
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	analytical method, validation data, storage period
2000 (Bond)			0.1					(0.03, 0.04)	REM. 118.01, LOQ : 0.02 mg/kg, 76–83% Recovery (n=2), Storage: 9 months
Spain, Los Palacios 2001 (Genaro)	EC	3	0.1	0.01	83	Fruits	0 7	0.1 <u>0.08</u> (0.07, 0.1)	1092/01, Gasser (2003, LUFEN_088) REM. 118.01, LOQ : 0.02 mg/kg, 98–100% Recovery (n=2), Storage: 3 months
Spain, Penaflor 2001 (Bond)	EC	3	0.09 0.1 0.1	0.01	74	Fruits	0 1 3 7	0.08 0.07 0.05 <u>0.08</u> (0.07, 0.08)	1093/01, Gasser (2003, LUFEN_089) REM. 118.01, LOQ : 0.02 mg/kg, 98–102% Recovery (n=2), Storage: 3 months
Switzerland, Chessel 1998 (Paola)	EC	3	0.1	0.005	72	Fruits	0 3 7 14	0.13 0.15 <u>0.11</u> 0.07	1024/98, Tribolet (1998, LUFEN_084) REM. 118.01, LOQ : 0.02 mg/kg, 87–91% Recovery (n=2), Storage: 3 months

DAT=Days after last treatment

BBCH 71–79=1st–9th fruit has reached typical size

BBCH 81–88=10–80% of fruits show typical fully ripe colour

BBCH 89=Fully ripe: fruits have typical fully ripe colour

Table 52 Residues of lufenuron following foliar application to sweet corn (Method POP-PAT-004 v.03, LOQ: 0.01 mg/kg, 94–119% Recovery (n=6), Storage: 7 months)

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
Brazil, Bairro Lagoa Bonita 2009 (AL Bandeirante)	EC	2	0.015	0.005	53	Kernels	35	< 0.01	M09089-LZF, Matarazzo (2012, LUFEN_094)
Brazil, Colónia Benifica 2009 (30 R 50)	EC	2	0.015	0.005	51	Kernels	35	< 0.01	M09089-DMO, Matarazzo (2012, LUFEN_094)
Brazil, Rodovia Nova Veneza 2009 (Impacto)	EC	2	0.015	0.005	51	Kernels	35	< 0.01	M09089-MFG, Matarazzo (2012, LUFEN_094)
Brazil, Rodavia 2009 (Master)	EC	2	0.015	0.005	69	Kernels	35	< 0.01	M09089-JJB, Matarazzo (2012, LUFEN_094)

DAT=Days after last treatment

BBCH 51=Beginning of tassel emergence: tassel detectable at top of stem

BBCH 53=Tip of tassel visible

BBCH 69=End of flowering: stigmata completely dry

Table 53 Residues of lufenuron following foliar application to soybeans

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
Brazil, Rodovia	EC	4	0.038	0.025	76	Seeds, dry	35	< 0.01	T06014-JJB1, Ribeiro (2008,

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
2007 (Conquista)	EC	4	0.075	0.05	76	Seeds, dry	35	< 0.01	LUFEN_097)
Brazil, Rodovia	EC	4	0.038	0.025	88	Seeds, dry	35	< 0.01	T06014-JJB2, Ribeiro (2008,
2007 (BRS Valiosa)	EC	4	0.075	0.05	88	Seeds, dry	35	< 0.01	LUFEN_097)
Brazil, Ponta Grossa	EC	4	0.038	0.025	75	Seeds, dry	35	< 0.01	T06014-DMO, Ribeiro (2008,
2007 (CD 206)	EC	4	0.075	0.05	75	Seeds, dry	35	< 0.01	LUFEN_097)
Brazil, Rodovia 2009 (NK 9074 RR)	EC	2	0.008	0.004	80	Seeds, dry	35	< 0.01	M09092-JJB, Roncato (2011, LUFEN_098)
Brazil, Rodovia Nova Venecia 2009 (NK 9074)	EC	2	0.008	0.004	79	Seeds, dry	35	< 0.01	M09092-MFG, Roncato (2011, LUFEN_098)
Brazil, Carambei 2009 (BRS 230)	EC	2	0.008	0.004	81	Seeds, dry	35	< 0.01	M09092-DMO1, Roncato (2011, LUFEN_098) months
Brazil, Itaberá 2009 (M 5942)	EC	2	0.008	0.004	81	Seeds, dry	35	< 0.01	M09092-DMO2, Roncato (2011, LUFEN_098)

Ribeiro (2008, LUFEN_097)=Method POPIT MET.015 Rev 01, LOQ: 0.01 mg/kg, 85–106% Recovery (n=10), Storage: 5 months

Roncato (2011, LUFEN_098)=Method POP PAT 004 V00, LOQ: 0.01 mg/kg, 72–104% Recovery (n=10), Storage: 5 months

DAT=Days after last treatment

BBCH 75–79=About 50–100% of pods have reached final length (15–20 mm).

BBCH 81–88=About 10–80% of pods are ripe; beans final colour, dry and hard

Table 54 Residues of lufenuron following foliar application to potatoes (Method POP-PAT-004 v.04, LOQ: 0.01 mg/kg, 71–80% Recovery (n=10), Storage: 3 months)

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
Brazil, Pouso Alegre 2009 (Cupido)	EC	4	0.04	0.005	47	Tubers	7 14 21	< 0.01 < 0.01 < 0.01	M09086-JJB, Matarazzo (2012, LUFEN_093)
Brazil, Piedade 2009 (Agata)	EC	4	0.04	0.005	46	Tubers	7 14 21	< 0.01 < 0.01 < 0.01	M09086-LZF, Matarazzo (2012, LUFEN_093)
Brazil, Curitibanos 2009 (Atlantic)	EC	4	0.04	0.005	44	Tubers	7 14 21	< 0.01 < 0.01 < 0.01	M09086-DMO1, Matarazzo (2012, LUFEN_093)
Brazil, Carambei 2009 (Atlantic)	EC	4	0.04	0.005	44	Tubers	7 14 21	< 0.01 < 0.01 < 0.01	M09086-DMO2, Matarazzo (2012, LUFEN_093)

DAT=Days after last treatment

BBCH 41–47=10–70% of total final tuber mass reached

Table 55 Residues of lufenuron following foliar application to maize (Method POP-PAT-004 v.03, LOQ: 0.01 mg/kg, 94–119% Recovery (n=6), Storage: 7 months)

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
Brazil, Bairro Lagoa Bonita 2009 (AL Bandeirante)	EC	2	0.015	0.005	53	Grain	82	< 0.01	M09089-LZF, Matarazzo (2012, LUFEN_094)
Brazil, Colónia Benifica 2009 (30 R 50)	EC	2	0.015	0.005	51	Grain	66	< 0.01	M09089-DMO, Matarazzo (2012, LUFEN_094)
Brazil, Rodovia Nova	EC	2	0.015	0.005	51	Grain	78	< 0.01	M09089-MFG, Matarazzo

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
Veneza 2009 (Impacto)									(2012, LUFEN_094)
Brazil, Rodavia 2009 (Master)	EC	2	0.015	0.005	69	Grain	56	< 0.01	M09089-JJB, Matarazzo (2012, LUFEN_094)

DAT=Days after last treatment

BBCH 51=Beginning of tassel emergence: tassel detectable at top of stem

BBCH 53=Tip of tassel visible

BBCH 69=End of flowering: stigmata completely dry

Table 56 Residues of lufenuron following foliar application to sugarcane (Method POPIT MET.077, LOQ: 0.01 mg/kg, 83–108% Recovery (n=12), Storage: 3 months)

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
Brazil, Rodovia 2007 (SP 803280)	EC	2	0.025	0.013	45	Sugarcane	7 14 21 28	< 0.01 < 0.01 < 0.01 < 0.01	M08083-LZF1, Marconi (2008, LUFEN_095)
Brazil, Rio das Pedras 2007 (RB 3280)	EC	2	0.025	0.013	49	Sugarcane	7 14 21 28	< 0.01 0.02 < 0.01 < 0.01	M08083-LZF2, Marconi (2008, LUFEN_095)
Brazil, Baneirantes 2007 (RB 415)	EC	2	0.025	0.013	45	Sugarcane	7 14 21 28	< 0.01 0.02 < 0.01 0.02	M08083-LZF3, Marconi (2008, LUFEN_095)
Brazil, Tupaciguara 2007 (SP 832847)	EC	2	0.025	0.013	39	Sugarcane	7 14 21 28	0.01 0.01 0.02 0.02	M08083-JJB, Marconi (2008, LUFEN_095)

DAT=Days after last treatment

BBCH 39 = Maximum stem length or rosette diameter reached

BBCH 45–49=50–100% of harvestable vegetative plant parts or vegetatively propagated, organs have reached final size

Table 57 Residues of lufenuron following foliar application to cotton (unnamed HPLC-UV method, LOQ : 0.05 mg/kg, 84–94% Recovery)

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
China, Changsha 2007 (Xiangmian 15)	EC	1	0.023	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	A7814A, Renbin (2008, LUFEN_096)
		2	0.023	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
		1	0.034	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
		2	0.034	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
China, Zhengzhou 2007	EC	1	0.023	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	A7814A, Renbin (2008, LUFEN_096)

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
(Zhongmian 41)		2	0.023	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
		1	0.034	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
		2	0.034	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
China, Changsha 2008 (Xiangmian 15)	EC	1	0.023	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	A7814A, Renbin (2008, LUFEN_096)
		2	0.023	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
		1	0.034	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
		2	0.034	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
China, Zhengzhou 2008	EC	1	0.023	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	A7814A, Renbin (2008, LUFEN_096)
(Zhongmian 41)		2	0.023	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
		1	0.034	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	
		2	0.034	n/s	n/s	Seeds	10 20 30	< 0.05 < 0.05 < 0.05	

n/s=Not stated

DAT = Days after last treatment

Table 58 Residues of lufenuron following foliar application to coffee (Method POPIT MET.077, LOQ: 0.01 mg/kg, 91–106% Recovery (n=12), Storage: 6 months)

Location,	Application					Residues, mg/kg			Report/Trial No., Reference
Year (variety)	Form.	no	kg ai/ha	kg ai/hL	BBCH	Sample	DAT	lufenuron	
Brazil, Holambra 2006 (Mundo Novo)	EC	2	0.04	0.01	87	Green beans (dry processed)	3 7 10	0.01 0.01 < 0.01	M05035-LZF1, Gois Fatima (2007, LUFEN_099)
Brazil, Santa Amélia 2006 (Obatá)	EC	2	0.04	0.01	87	Green beans (dry processed)	3 7 10	< 0.01 < 0.01 < 0.01	M05035-LZF2, Gois Fatima (2007, LUFEN_099)
Brazil, Monte Carmelo 2006 (Mundo novo)	EC	2	0.04	0.01	89	Green beans (dry processed)	3 7 10	< 0.01 < 0.01 < 0.01	M05035-JJB, Gois Fatima (2007, LUFEN_099)

DAT = Days after last treatment

BBCH 88 = Fruit is fully-ripe color and ready for picking

Fate of residues in storage and processing

Nature of residue during processing

The hydrolysis of lufenuron under processing conditions was investigated by Grout (2003, LUFEN_100). [^{14}C]diflurophenyl and [^{14}C]dichorophenyl labelled lufenuron 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 characterized by fractionation and co-chromatography with authenticated reference compounds using HPLC.

Table 59 Hydrolysis of [^{14}C]dichorophenyl labelled lufenuron under simulated processing conditions

		% applied radioactivity				
Process represented	Sample	Lufenuron	CGA224443	CGA238277	Unknowns	Recovery
PH 4 90 °C 20 mins	1	99.0	0.0	0.0	0.0	100.9
	2	101.5	0.0	0.0	0.0	104.2
PH 5 100 °C 60 mins	1	93.4	6.3	0.5	1.1	103.5
	2	97.3	6.9	0.4	1.1	108.0
PH 6 120 °C 20 mins	1	114.0	0.0	0.0	0.0	115.9
	2	100.4	0.0	0.0	0.0	102.6

Table 60 Hydrolysis of [^{14}C]diflurophenyl labelled lufenuron under simulated processing conditions

		% applied radioactivity				
Process represented	Sample	Lufenuron	CGA149772	CGA149766	Unknowns	Recovery
PH 4 90 °C 20 mins	1	97.2	0.5	0.0	0.0	99.2
	2	100.9	0.6	0.0	0.0	103.3
PH 5 100 °C 60 mins	1	99.7	6.9	0.7	0.4	110.0
	2	99.7	3.8	0.5	0.2	106.2
PH 6 120 °C 20 mins	1	100.3	0.0	0.0	0.0	101.6
	2	102.9	0.0	0.0	0.0	104.9

Residues after processing

The fate of lufenuron during processing of raw agricultural commodity (RAC) was investigated in tomatoes using important processing procedures. As a measure of the transfer of residues into processed products, a processing factor was used, which is defined as:

Processing factor=Residue in processed product (mg/kg) ÷ 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).

Tomato

A study on the behaviour of lufenuron during processing of tomatoes was conducted by Sole (2003, LUFEN_101). Tomatoes grown outdoor in Southern France were treated three times with 0.03 kg lufenuron/ha each at one week intervals. Samples were harvested 8 days after the last application. Tomatoes were used for the production of tomato juice, canned tomato and tomatoes puree. The field sample was split into subsamples processed multiple times for each commodity:

- 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 61 Summary of lufenuron residues in tomato and processed commodities from a trial conducted in Southern France (Sole 2003, LUFEN_101)

Commodity	Lufenuron in mg/kg	Processing factor	Median or best estimate processing factor
Fruits (RAC)	0.029	–	–
Raw juice	< 0.005, 0.005	< 0.17, 0.17	0.17
Pasteurized juice	< 0.005(4)	< 0.17(4)	0.17
Wet pomace	0.23, 0.23, 0.25, 0.28	7.9, 7.9, 8.6, 9.7	8.3
Canned tomato	< 0.005(4)	< 0.17(4)	0.17
Raw paste	0.024, 0.032	0.83, 1.1	0.97
Pasteurized puree	0.023, 0.024, 0.025, 0.026	0.79, 0.83, 0.86.0.9	0.85

RAC=Raw agricultural commodity

Residues in animal commodities

Farm animal feeding studies

For the estimation of residues of lufenuron in animal matrices one lactating cow feeding study and one steer feeding study were submitted to the Meeting.

Lactating cows

In the first study residues in lactating cows were investigated by Tribolet (1995, LUFEN_102). The dose rates were approximately 0, 39, 230 and 415 µg lufenuron/kg body weight/day (equivalent to nominal concentrations of 0, 0.82, 4.3 and 8.6 mg/kg in the daily feed).

The cows in the treatment groups were fed with the lufenuron twice daily with the active ingredient mixed with pelleted feed, for a period of 28–29 days. Milk samples were collected pre-treatment and throughout the dosing period. At Day 29–30 the cows were slaughtered and samples of muscle (tenderloin, round steak), liver, kidney and fat (omental and peri-renal) were taken for analysis.

Milk and tissues were extracted and analysed for lufenuron using method REM 118.04. The LOQ for milk, blood and tissues are 1 µg/L, 10 µg/L and 0.01 mg/kg respectively.

In the control group no detectable residues of lufenuron were found. The findings in milk and tissues are summarized in the following table.

Table 62 Residues of lufenuron in cow tissues and milk following administration of lufenuron at 0.82, 4.3 and 8.6 ppm in the diet

Commodity	Sampling Interval (days)	Maximum Lufenuron Residues (mg/kg)		
		Group 2 (0.82 ppm)	Group 3 (4.3 ppm)	Group 4 (8.6 ppm)
Milk	1	0.005, 0.013, 0.012 (0.01)	0.062, 0.104, 0.16 (0.11)	0.28, 0.13, 0.14 (0.18)
	4	0.062, 0.105, 0.05 (0.072)	0.38, 0.48, 0.84 (0.57)	1.2, 1.2, 0.68 (1.0)
	7	0.076, 0.098, 0.036 (0.07)	0.62, 0.505, 0.565 (0.58)	1.3, 0.82, 0.6 (0.9)
	10	0.095, 0.12, 0.13 (0.12)	0.56, 0.76, 0.96 (0.76)	2.0, 1.4, 1.8 (1.7)

Commodity	Sampling Interval (days)	Maximum Lufenuron Residues (mg/kg)		
		Group 2 (0.82 ppm)	Group 3 (4.3 ppm)	Group 4 (8.6 ppm)
	14	0.136, 0.16, 0.121 (0.14)	0.68, 0.84, 0.96 (0.83)	2.2, 2.2, 1.6 (2.0)
	17	0.118, 0.184, 0.125 (0.14)	0.61, 0.84, 0.96 (0.89)	2.2, 2.1, 1.7 (2.0)
	21	0.15, 0.188, 0.132 (0.16)	0.71, 0.94, 1.15 (0.93)	2.1,2.7, 1.8 (2.2)
	24	0.105, 0.197, 0.122 (0.14)	0.55, 1.23, 1.18 (0.99)	2.3, 2.3, 2.8 (2.5)
	28	0.12, 0.167, 0.168 (0.15)	0.85, 0.99, 0.77 (0.87)	1.6, 1.9, 1.4 (1.6)
Milk—skim milk	28	0.007, 0.004, 0.008 (0.006)	0.023, 0.059, 0.032 (0.038)	0.049, 0.058, 0.057 (0.054)
Milk—cream	28	4.3, 2.3, 2.6 (3.1)	25, 28, 19 (24)	27, 30, 39 (32)
Muscle—tenderloin	29	0.02, 0.04, 0.04 (0.03)	0.09, 0.15, 0.26 (0.17)	0.26, 0.49, 0.54 (0.43)
Muscle—round steak	29	0.01, 0.02, 0.02 (0.02)	0.04, 0.09, 0.12 (0.08)	0.09, 0.16, 0.34 (0.2)
Liver	29	0.05, 0.06, 0.07 (0.06)	0.32, 0.39, 0.39 (0.37)	0.64, 0.67, 0.99 (0.77)
Kidney	29	0.03, 0.03, 0.04 (0.03)	0.19, 0.23, 0.23 (0.22)	0.32, 0.35, 0.42 (0.36)
Fat—peri-renal	29	0.53, 0.56, 0.84 (0.64)	3.9, 4.2, 5.3 (4.5)	6.3, 7.7, 10.1 (8.0)
Fat—omental	29	0.42, 0.57, 1.2 (0.73)	3.5, 3.6, 4.1 (3.7)	6.2, 7.0, 8.9 (7.4)

Steer

Residues of lufenuron in steer were also investigated by Tribolet (2000, LUFEN_103). Three groups of steers, Angus X Hereford, were used in this study, two treated and one control group. One group of three steers was dosed with capsules containing 0.2 mg lufenuron and a further treatment group of 12 steers were dosed with 10 mg of lufenuron. Each treatment group was dosed for 28 consecutive days. The dose rates were equivalent to nominal concentrations of 0.02 and 1 mg/kg in the daily feed (0.0006 and 0.031 mg/kg bw/day).

The lower dose group and three steers from the higher group were sacrificed 20–24 hours after the final dose and a further three steers were sacrificed at two week intervals, i.e. days 42, 56 and 70 after the commencement of dosing. At sacrifice, samples of blood, muscle (tenderloin, round steak), liver, kidney and fat (omental and peri-renal) were taken for analysis.

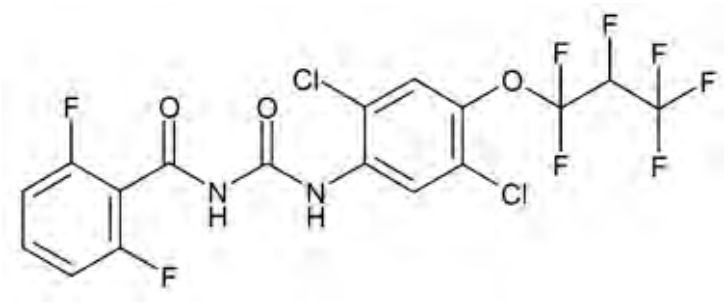
Tissues were extracted and analysed for lufenuron using method REM 118.04. The LOQ for blood and tissues are 2 µg/L, and 0.01 mg/kg respectively.

Table 63 Residues of lufenuron in steer tissues following administration of lufenuron at 0.02 and 1 ppm in the diet

Days	Lufenuron residues in mg/kg					
	Muscle—tenderloin	Muscle—round steak	Liver	Kidney	Fat—peri-renal	Fat—omental
Low dose group (0.02 ppm)						
28	< 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)	0.022, 0.038, 0.035 (0.032)	0.024, 0.038, 0.045 (0.036)
High dose group (1 ppm)						
28	< 0.01, < 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01, < 0.01 (< 0.01)	0.018, 0.027, 0.025 (0.023)	0.032, 0.022, 0.023 (0.026)	0.15, 0.26, 0.27 (0.23)	0.16, 0.24, 0.26 (0.22)
42	< 0.01, < 0.01, < 0.01 (< 0.01)	< 0.01, < 0.01, < 0.01 (< 0.01)	0.01, 0.01, 0.01 (0.01)	0.011, 0.011, 0.014 (0.012)	0.066, 0.081, 0.1 (0.082)	0.071, 0.084, 0.12 (0.092)
56	< 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, 0.011 (0.01)	0.057, 0.072, 0.082 (0.07)	0.061, 0.077, 0.086 (0.075)
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)	< 0.01, < 0.01, 0.013 (0.011)	0.038, 0.038, 0.055 (0.044)	0.039, 0.041, 0.065 (0.048)

APPRAISAL

Lufenuron (ISO common name) is an insect growth inhibitor that is active against larvae of Lepidoptera and Coleoptera. When ingested, lufenuron interferes with chitin synthesis, and prevents larvae from moulting. It was considered for the first time by the 2015 JMPR for toxicology and residues.



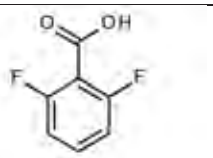
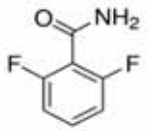
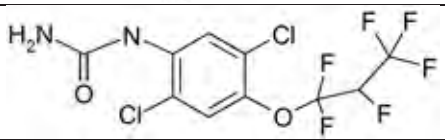
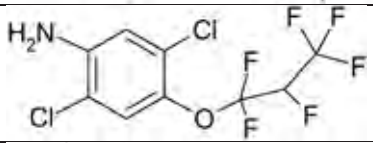
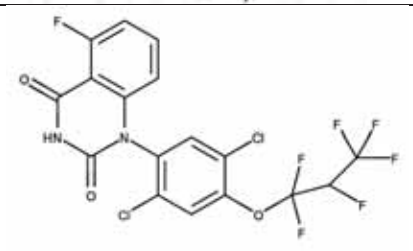
The IUPAC name of lufenuron is (RS)-1-[2,5-dichloro-4-(1,1,2,3,3,3-hexafluoropropoxy)phenyl]-3-(2,6-difluorobenzoyl)urea and the CA name is N-[[[2,5-dichloro-4-(1,1,2,3,3,3-hexafluoropropoxy)phenyl]amino]carbonyl]-2,6-difluorobenzamide.

Lufenuron consists of a pair of enantiomers. A chiral centre exists at the 2-position of the hexafluoropropoxy side-chain. Lufenuron technical active ingredient is manufactured under non-stereospecific conditions giving a racemate (R:S 50:50).

The physical-chemical properties of lufenuron indicate low volatility and no accelerated photochemical degradation in water. The octanol-water partition coefficient, log $P_{ow,}$ is 5.12.

Lufenuron radio-labelled either in the dichlorophenyl- or difluorophenyl-moiety was used in the metabolism and environmental fate studies.

The following abbreviations are used for the metabolites discussed below:

CGA149776	2,6-Difluoro-benzoic acid	
CGA149772	2,6-Difluoro-benzamide	
CGA238277	2,5-Dichloro-4-(1,1,2,3,3,3-hexafluoropropoxy)-phenyl-urea	
CGA224443	N-[2,5-dichloro-4-(1,1,2,3,3,3-hexafluoropropoxy)-benzenamine	
CGA301018	no chemical name submitted	

Environmental fate in soil

The Meeting received information for lufenuron on soil photolysis, aqueous hydrolysis, aerobic soil metabolism and soil degradation.

Soil photolysis using [dichlorophenyl-^{14}C]-lufenuron and [difluorophenyl-^{14}C]-lufenuron revealed no significant degradation (84–99% parent remaining after 17 days of continuous irradiation).

Hydrolysis in aqueous solutions representative of environmental conditions (25 °C) showed virtually no degradation at pH 5, 7 and 9 within 5 days. Under more extreme conditions the parent substance was stable at pH 1 and 70°C, representing more than 90% of the radioactivity. At pH 9 an accelerated degradation was observed at 50 °C and 70 °C with 0–53% of the parent remaining after 1–5 days. Depending on the label the cleavage products CGA224443 and CGA238277 and its counterparts CGA149776 and 2,6-difluorobenzamide (CGA149772) were observed. In addition both labelled compounds produced CGA301018 by loss of fluoride and ring closure.

In the aerobic soil metabolism studies lufenuron was degraded with half-lives of 9–24 days in microbial active soil and 17–83 days in sterilised soil. Cleavage of the parent molecule was the primary degradation step, leaving CGA238277 and CGA224443 for [dichlorophenyl-^{14}C]-lufenuron. For [difluorophenyl-^{14}C]-lufenuron no metabolites were identified. Unextracted residues in soil at the end of the studies were between 25–79% of the AR. Mineralisation ranged up to 59% AR.

2,6-difluorobenzamide (CGA149772), which is a common soil metabolite to other active substances, e.g., diflubenzuron, was investigated separately for its behaviour in soil. Within 120 days it was completely degraded, leaving CGA149776 as its main degradate within the first two weeks. Afterward the radioactivity was further degraded and remained unextracted (up to 41% AR) or was mineralized (up to 65% AR).

The soil degradation of lufenuron and its metabolites CGA238227 and CGA224443 was also investigated on three different soils under laboratory conditions. Following 1st-order kinetic, DT_{50} and DT_{90} values of 13.7 d and 81.1d for lufenuron, 12.8 d and 42.5 d for CGA238277 and of 35.8 d and 119 d for CGA224443 were calculated, respectively.

In summary the Meeting concluded that lufenuron is moderately quickly degraded in soil under laboratory conditions, presumably by microbial activity. To assess the degradation behaviour under field conditions, field dissipation studies would be required. The residue is stable against photolysis and hydrolysis under environmental conditions, however at high temperature and basic conditions cleavage of the parent molecule was observed.

Plant metabolism

The Meeting received plant metabolism studies for lufenuron following foliar application of either [dichlorophenyl-^{14}C]-lufenuron or [difluorophenyl-^{14}C]-lufenuron in cabbage, tomato and cotton.

For cabbage the metabolism of lufenuron was investigated with [dichlorophenyl-^{14}C]-lufenuron only. Greenhouse plants received three spray applications equivalent to 0.02 kg ai/ha each in two week intervals. Samples were taken one hour after the first and last application, and at crop maturity, 28 days after the last application.

In mature cabbage heads TRR levels were 0.195 mg eq/kg (up to 1.8 mg eq/kg in withered leaves). 97.5% of the TRR (0.19 mg eq/kg) was recovered as unchanged lufenuron. In the head cabbage as well as in withered leaves, CGA238277 was identified at estimated levels of 0.6% and 3.3% of the TRR, respectively. The actual amounts were not measured in the TLC system used. No further metabolites were found.

For tomatoes the metabolism of lufenuron was investigated with [dichlorophenyl-^{14}C]-lufenuron only. Fruit bearing plants kept in a protected environment were treated with three sprayings equivalent to 0.03 kg ai/ha per application with one week intervals. Samples were collected directly

after the first application and up to 28 DALA. In parallel 34µg lufenuron was directly injected into single fruits, which were sampled after 18 and 33 days.

Directly after the last foliar application, TRR levels in fruits were 1.2 mg eq/kg, degrading to 0.69 mg eq/kg after 28 days. TRR levels found in additional samples at 28 DAT were 0.47 mg eq/kg for leaves and 0.44 mg eq/kg in mature fruits. Newly developed green fruits had much lower total radioactive residues of 0.03 mg eq/kg. In all fruits receiving a foliar treatment > 89% of the residue was recovered in the surface wash. Unextracted residues were generally low (< 0.6% TRR).

The identification of the radioactivity (combined surface wash and extract) showed unchanged lufenuron as the major residue in fruits and leaves (93–98% TRR). Only in one fruit sample collected 28 DAT, minor amounts of CGA238277 (0.2% TRR, 0.0013 mg eq/kg) were found.

In mature fruits receiving a direct injection of lufenuron, the results from the extracts were comparable to foliar treated fruits. 90–95% of the radioactivity was identified as unchanged lufenuron. CGA238277 was identified in minor amounts up to 2% of the TRR. 5% of the TRR remained unextracted.

For cotton grown under glasshouse conditions the metabolism was investigated in two studies using [dichlorophenyl-^{14}C]-lufenuron or [difluorophenyl-^{14}C]-lufenuron.

For [dichlorophenyl-^{14}C]-lufenuron cotton plants received three foliar sprayings equivalent to 0.03 kg ai/ha each at 14 day interval, beginning at flowering. Sampling of leaves took place 1 hour, 1 day, 3 and 7 days after the first application and 14 days, 28 and 84 days (maturity) after the last application. In addition, four cotton plants received three stem injections (100 µg lufenuron each) made at 14-day intervals.

TRR levels found were up to 4.9 mg eq/kg in the leaves, < 0.001 mg eq/kg in seeds, 0.092 mg eq/kg in hulls and 0.001 mg eq/kg in green bolls. In leaves the amount of radioactivity in the surface wash decreased from 98% TRR after application 1 to 43% TRR at maturity (84 DALA).

The identification of the radioactivity (combined surface wash and extracts) showed 89–100% of the TRR as unchanged lufenuron. No metabolites were identified. In seeds and green bolls TRR levels were too low for further identification. Unextracted residues did not exceed 3.3% of the TRR.

The stem injection showed that most of the applied radioactivity remained at the injection site (81.2% AR). Minor translocation was observed into adjected stalks (13.3% AR) and leaves (1.6–3.9% AR). In all samples the unchanged parent was the only residue identified (~95–98% TRR).

For [difluorophenyl-^{14}C]-lufenuron the use pattern was comparable to the other label, but only the foliar treatment experiment was conducted. Samples of mature plant parts were collected 52 DALA.

TRR levels found were up to 5.95 mg eq/kg in leaves (52 DALA), 0.69 mg eq/kg in hulls and 0.003 mg eq/kg in seeds. In the leaves the surface wash contained most of the residue with 96% TRR directly after treatment and 49–58% TRR at maturity (52 DALA).

The identification again revealed unchanged lufenuron exclusively, representing >92% of the TRR in leaves and 79–83% TRR in other matrices. The TRR found in seeds was too low for identification. No further metabolites were detected.

Two confined rotational crop studies for lufenuron were submitted

In the first study [difluorophenyl-^{14}C]-lufenuron was applied under protected conditions to bare soil at a rate equivalent to 0.15 kg ai/ha. Lettuce, spring wheat, maize and carrots were planted in the treated soil 63 days after test substance application. The transfer of radioactivity into succeeding crops was very limited. In mature lettuce (126 d after treatment) the highest TRR level of 0.047 mg eq/kg was found. 53% of the TRR was identified as unchanged parent (0.025 mg/kg). In other matrices only wheat straw (0.023 mg eq/kg, 0.007 mg lufenuron/kg) and immature carrots roots (0.023 mg eq/kg, no identification conducted) showed total radioactive residues above 0.01 mg eq/kg. No further identification was conducted for these matrices. In soil samples, nearly the entire extracted

radioactivity was attributed to lufenuron. No residue of CGA149772 or CGA149776 could be identified in any sample.

In a second confined study conducted under field conditions [dichlorophenyl-^{14}C]-lufenuron was applied to bare soil once at a rate equivalent to 0.13 kg ai/ha. After different plant-back intervals (PBI) lettuce (PBI 76 d), winter wheat (PBI 126 d), sugar beets (PBI 306 days) and maize (PBI 331 d) were planted/sown and grown to maturity. TRR levels in all plant samples was between < 0.001 mg eq/kg and 0.004 mg eq/kg, which was too low for further identification.

In summary lufenuron is deposited on the plant surface and slowly adsorbed by leaves following direct treatment. On the surface and in plant tissue, the active substance is the only residue present in major amounts. Minor amounts of CGA238277 were identified in cabbage and tomato (up to 3.3% TRR). All plant metabolism studies for lufenuron were conducted under protected conditions. However, since lufenuron is not subject to photolysis the residue pattern in plants grown under field conditions is expected to be similar. Also, two of three studies were conducted with [dichlorophenyl-^{14}C]-lufenuron only. Since nearly the entire applied radioactivity was recovered as unchanged parent compound in these studies, no investigations with a second label are considered necessary.

For rotational crops the transfer of residues into succeeding crops from soil is very limited and mostly resulted in TRR levels too low for identification. In soil and in crop samples subject to identification parent lufenuron was the major residue. No further metabolites were identified.

Animal metabolism

Information was available on metabolism of lufenuron in laboratory animals, lactating goats and laying hens. Studies on rats, mice and dogs were evaluated by the WHO Core Assessment Group.

For lactating goats two studies were conducted involving daily administration of either ^{14}C-difluorophenyl-labelled lufenuron at 5.4 ppm (0.135 mg/kg bw) or ^{14}C-dichlorophenyl-lufenuron at 6.0 ppm (0.15 mg/kg bw) for ten consecutive days. The animals were slaughtered approximately 24h after the last dose.

The total recovery of the administered radioactivity was 95% for both labels. The majority of the radioactivity (73–74%) was found in the faeces. Radioactive residues in the edible tissues were 0.8–1.6% AR in muscle (0.038–0.08 mg eq/kg), 4.2–5.4% AR in fat (0.82–2.4 mg eq/kg), 0.28–0.3% AR in liver (0.37–0.42 mg eq/kg), 0.01–0.02% AR in kidney (0.11–0.12 mg eq/kg) and 5.8–6.8% AR in milk (up to 1.0 mg eq/kg). A plateau in milk was observed after approximately one week.

In tissues and milk unchanged parent was the only residue identified for both radiolabels, representing 73–94% of the TRR. The remaining radioactivity remained unresolved in the TLC-System used (6.6–19% TRR) or was not extracted from the sample (0.6–8.9% TRR).

Also for laying hens two studies were conducted involving daily administration of either ^{14}C-difluorophenyl-labelled lufenuron at 3.4 ppm (2.6 mg/kg bw) or ^{14}C-dichlorophenyl-lufenuron at 5.2 ppm (3.5 mg/kg bw) for fourteen consecutive days. The animals were slaughtered approximately 24h after the last dose.

The total recovery of the administered radioactivity was 75–79%. The majority of the radioactivity (54–62%) was found in the excreta. Radioactive residues in the edible tissues were 0.55–1.2% AR in lean meat (0.1–0.24 mg eq/kg), 5.1–9.9% AR in fat (7.2–13 mg eq/kg), 0.4–0.58% AR in liver (0.83–1.5 mg eq/kg), 0.07–0.09% AR in kidney (0.52–0.74 mg eq/kg) and 8.7–9.6% AR in eggs (up to 0.016 mg eq/kg in egg white and 8.5 mg eq/kg in egg yolk). In eggs a plateau was observed after one week for ^{14}C-difluorophenyl-lufenuron while residues for ^{14}C-dichlorophenyl-lufenuron showed a slight increase until the end of dosing.

In tissues and eggs unchanged parent lufenuron was the predominant residue, representing 79–94% TRR in all matrices except egg white. For the difluorophenyl-label the cleavage product CGA149772 was the only metabolite detected, being present in egg white at 0.001 mg eq/kg (17.3% TRR). For the dichlorophenyl-label its counterpart CGA238277 was found in minor amounts in kidney (0.028 mg eq/kg, 5.3% TRR) and egg white (< 0.001 mg eq/kg, 7.0% TRR). The remaining

radioactivity remained unresolved in the TLC-System used (3–42% TRR) or was not extracted from the sample (2–11% TRR).

In summary the metabolic degradation of lufenuron in livestock animals is very limited, showing parent as the predominant residue in all matrices. Minor amounts of the cleavage products CGA149772 and CGA238277 were found in poultry kidney and egg white.

Methods of residue analysis

The Meeting received analytical methods for the analysis of lufenuron in plant and animal matrices. The basic principle employs extraction by homogenisation with methanol or water and partitioning against hexane: ethyl ether (9:1,v:v). Clean-up is normally achieved by C18 solid-phase extraction. Residues are determined by liquid chromatography (LC) in combination with UV (255 nm) or tandem mass spectroscopy (MS/MS). Mass-transitions are m/z 509.1 → 326 for quantification and m/z 509→ 175 for confirmation. The methods submitted are suitable for measuring residues with a LOQ of 0.01 mg/kg in high water, high oil and high starch matrices while acidic matrices were validated with a LOQ of 0.02 mg/kg.

For animal matrices the analytical methods were comparable, however silica gel SPE was used for clean-up instead. Validated LOQs were 0.001 mg eq/kg for milk, 0.01 mg/kg for liver and kidney, 0.02 mg/kg for meat and 0.1 mg/kg for fat.

The application of multi-residue methods was tested with DFG S19 for both plant and animal matrices. The method was shown suitable with a general LOQ of 0.02 mg/kg for lufenuron.

Stability of residues in stored analytical samples

The Meeting received information on the storage stability of lufenuron in plant and animal matrices stored at -18°C.

In plant matrices with high water, high acid and high oil content parent lufenuron was stable for at least 24 months. High starch matrices were not tested.

In animal matrices (bovine tissues and milk) no significant degradation was observed within 9 months. No storage stability data were provided for poultry matrices and eggs.

Definition of the residue

The fate of lufenuron in plants was investigated after foliar application to tomatoes, cabbage and cotton. In all crop samples investigated unchanged lufenuron was the only major residue present, representing 79–100% TRR. The residue was mainly present as a surface residue. No significant transfer into untreated plant parts was observed.

In confined rotational crop studies the overall uptake of radioactivity was very limited. Only parent lufenuron could be detected in collected plant samples.

The Meeting concluded that lufenuron is the relevant residue in all plant matrices for compliance with MRLs and for dietary intake purposes. Analytical multi-residue methods are capable of measuring lufenuron in all plant matrices.

Livestock animal metabolism studies were conducted on lactating goats (5.4–6.0 ppm) and laying hens (3.4–5.2 ppm).

In both species unchanged parent lufenuron was the only residue identified in major amounts, representing 73–94% of the TRR in all matrices. In goat matrices and milk no other metabolites could be detected. In poultry matrices minor amounts of the cleavage products CGA149772 and CGA238277 were found, representing up to 17% TRR in egg white but at low levels (0.001 mg eq/kg) and 5.3% TRR in kidney (0.028 mg eq/kg). No further metabolites were found in poultry matrices or eggs.

The Meeting concluded that parent lufenuron is the relevant residue in all animal matrices for compliance with MRLs and for dietary intake purposes. Analytical multi-residue methods are capable of measuring lufenuron in all animal matrices.

In all species residue concentrations in fat tissues or egg yolk were at least one order of magnitude higher than in muscle tissues or egg white. The log P_{ow} of lufenuron is 5.12. The Meeting decided that residues of lufenuron are fat soluble.

Definition of the residue for compliance with MRL and for dietary intake for plant and animal commodities: *lufenuron*

The residue is fat-soluble.

Results of supervised residue trials on crops

The Meeting received supervised trial data for applications of lufenuron on various vegetables crops as well as for soya beans, maize, sugarcane, cotton and coffee conducted in Brazil, China and Europe.

Cucumber

Lufenuron is registered in Spain for cucumbers under protected conditions at rates of 2 × 0.1 kg ai/ha with a PHI of 7 days. Supervised field trials from France, Greece and Spain according to this GAP and at rates up to +50% higher were submitted.

In protected cucumbers residues of lufenuron following GAP treatment (±25%) were (n=4): 0.01, 0.02, 0.06, 0.06 mg/kg.

The Meeting concluded that four supervised trials on cucumber approximating GAP are insufficient for an evaluation and decided to explore the proportionality approach using trials at +50% GAP rate. Since some of the trials according to GAP were also conducted at slightly elevated rates, all data are proportionally adjusted to the Spanish GAP rate of 0.1 kg ai/ha:

In protected cucumbers treated with 0.1 kg ai/ha lufenuron residues were (no scaling factor): 0.06 mg/kg.

In protected cucumbers treated with 0.11 kg ai/ha lufenuron residues were (scaling factor 0.91): 0.018 mg/kg (0.91×0.02 mg/kg).

In protected cucumbers treated with 0.12 kg ai/ha lufenuron residues were (scaling factor 0.83): 0.0083 and 0.05 mg/kg (0.83×0.01 mg/kg and 0.83×0.06 mg/kg).

In protected cucumbers treated with 0.15 kg ai/ha lufenuron residues were (scaling factor 0.66): 0.013(3), 0.02(3), 0.026 mg/kg (0.66×0.02 mg/kg(3), 0.66×0.03 mg/kg(3) and 0.66×0.04. mg/kg)

The combined total dataset for lufenuron in protected cucumbers was (n=11): 0.0083, 0.013(3), 0.018, 0.02(3), 0.026, 0.05 and 0.06 mg/kg.

The Meeting estimated a maximum residues level of 0.09 mg/kg and a STMR of 0.02 mg/kg for lufenuron in cucumber.

Melons, except watermelons

Lufenuron is registered in Spain for melons under protected conditions at rates of 3 × 0.1 kg ai/ha with a PHI of 7 days. Supervised field trials from Spain according to this GAP were submitted.

All samples were segmented and in some trials already separated into pulp and peel in the field, which is against the current Codex sampling procedure. However, lufenuron was not metabolized in plant metabolism studies, even after direct injection into tomato fruits. In addition simulated hydrolysis indicated no degradation at pH 7 or lower, which is representive of fruits and vegetables. The Meeting therefore concluded that segmentation of samples in the field did not influence the magnitude of residues. The Meeting also noted that no contamination of melon pulp with peel residues during separation occurred and decided to use the data for its assessment.

Some trials submitted involved a last sampling at 3 DALA which is shorter than the PHI of the Spanish GAP of 7 days. In plant metabolism studies lufenuron was a surface residue not subject to degradation or metabolism. Also, melons near maturity have already finalized their growth and are only subject to ripening. Therefore the Meeting concluded that no different residue populations have to be expected for melons within the last week before harvest when sampled at 3 or 7 DALA and decided to take samples collected after three days also into account for the assessment. This conclusion is supported by several decline studies from 0 to 10 DALA, indicating no constant decrease of the residue concentration but the usual sampling variation within the results.

In protected melons (whole fruits) residues of lufenuron were (n=6): 0.02, 0.03, 0.06, 0.09, 0.13, 0.19 mg/kg.

In the corresponding pulp samples, if measured, residues of lufenuron were (n=4): < 0.02(4) mg/kg.

For melon, except watermelons, the Meeting estimated a maximum residues level of 0.4 mg/kg, based on whole melon fruits, except watermelons and an STMR of 0.02 mg/kg, based on pulp data.

Peppers, sweet

Lufenuron is registered in Spain for sweet peppers under protected conditions at rates of 3 × 0.1 kg ai/ha with a PHI of 7 days. Supervised field trials on sweet peppers from Greece, Italy and Spain according to this GAP were submitted.

In protected sweet peppers residues of lufenuron following GAP treatment (±25%) were (n=6): 0.08, 0.13, 0.13, 0.17, 0.18 and 0.54 mg/kg.

The Meeting estimated a maximum residues level of 0.8 mg/kg and an STMR of 0.15 mg/kg for lufenuron in sweet peppers.

Tomato

Lufenuron is registered in Spain for tomatoes under protected conditions at rates of 3 × 0.1 kg ai/ha with a PHI of 7 days. Supervised field trials on tomatoes from Greece, Spain and Switzerland according to this GAP were submitted.

In protected tomatoes residues of lufenuron following GAP treatment were (n=13): 0.02, 0.04, 0.04, 0.05, 0.06, 0.08(4), 0.09, 0.1, 0.11 and 0.24 mg/kg.

The Meeting estimated a maximum residues level of 0.4 mg/kg and an STMR of 0.08 mg/kg for lufenuron in tomatoes.

Sweet corn

The Meeting received supervised field trail information on sweet corn, however no corresponding GAP was made available to the Meeting and therefore no recommendation was made.

Soya beans

Lufenuron is registered in Brazil for soya beans at maximum rates of 2 × 0.02 kg ai/ha with a PHI of 35 days. Supervised field trials on soya beans from Brazil at exaggerated rates (3.8 times higher) and a higher number of treatments (four instead of two) were submitted.

In soya beans residues of lufenuron after exaggerated treatment were (n=3): < 0.01(3) mg/kg

The Meeting concluded that under consideration of the exaggerated treatment regime involved, the seeds being protected by the pod during applications and the non-systemic properties of the active substance observed in plant metabolism studies, no finite residue following treatment at GAP rate have to be expected. The Meeting estimated a maximum residues level of 0.01* mg/kg and an STMR of 0 mg/kg for lufenuron in soya beans (dry).

Potatoes

Lufenuron is registered in Brazil for potatoes at rates of 4 × 0.04 kg ai/ha with a PHI of 14 days. Supervised field trials from Brazil matching the GAP were submitted.

In potato tubers residues of lufenuron after treatment according to GAP were (n=4): < 0.01(4) mg/kg

Taking into account the non-systemic properties of the active substance, the Meeting concluded that residues in tuber above the LOQ are unlikely to occur and estimated a maximum residues level of 0.01* mg/kg and an STMR of 0.01 mg/kg for lufenuron in potatoes.

Maize

Lufenuron is registered in Brazil for maize at maximum rates of 2 × 0.01 kg ai/ha with a PHI of 35 days. All supervised field trials on maize submitted were sampled at significantly longer DAT intervals than the PHI.

The Meeting concluded that the data submitted for lufenuron in maize is insufficient for a recommendation.

Sugar cane

Lufenuron is registered in Brazil for sugar cane at rates of 2 × 0.02 kg ai/ha with a PHI of 14 days. Supervised field trials from Brazil matching the GAP were submitted.

In sugar cane residues of lufenuron after treatment according to GAP were (n=4): < 0.01 and 0.02(3) mg/kg

The Meeting concluded that the data submitted for lufenuron in sugar cane is insufficient for a recommendation.

Cotton

Lufenuron is registered in China for cotton at rates of 2 × 0.045 kg ai/ha with a PHI of 28 days. Supervised field trials from China according to this GAP were submitted, however the trial description did not included information on the stage of boll opening for cotton plants.

In cotton seeds residues of lufenuron after treatment according to GAP were (n=4): < 0.05(4) mg/kg

The Meeting concluded that the stage of boll opening is a sensitive parameter for residues following foliar application. Without this type of information, a set of four field trials in not considered sufficient for estimating maximum residue levels in cotton seed. Supportive information from plant metabolism studies cannot be taken into account as the active substance was applied before boll opening in these studies.

Coffee

Lufenuron is registered in Brazil for coffee at rates of 2 × 0.04 kg ai/ha with a PHI of 7 days. Supervised field trials from Brazil matching the GAP were submitted.

In coffee beans (dry processed) residues of lufenuron after treatment according to GAP were (n=4): < 0.01(3) and 0.01 mg/kg

The Meeting concluded that the data submitted for lufenuron in coffee is insufficient for a recommendation.

Fate of residues during processing

The Meeting received information on the hydrolysis of radio-labelled lufenuron as well as processing studies using unlabelled material in tomatoes.

In a hydrolysis study using [dichlorophenyl-^{14}C]-lufenuron or [difluorophenyl-^{14}C]-lufenuron, typical processing conditions were simulated (pH 4,5 and 6 with 90°C, 100°C and 120°C for 20, 60 and 20 minutes). No significant degradation of the parent was observed. For pH5 with 100°C for 60min a minor formation of CGA224443 and CGA149772 (up to 6.9% of the applied radioactivity) was observed.

The fate of lufenuron residues has been examined simulating household and commercial processing of tomatoes. Estimated processing factors for the commodities considered at this Meeting are summarized below.

Raw commodity	Processed commodity	Lufenuron		
		Individual processing factors	Mean or best estimate processing factor	STMR-P in mg/kg
Tomato (STMR: 0.08 mg/kg)	Juice, raw	<0.17, 0.17	0.17	0.014
	Puree	0.79, 0.83, 0.86.0.9	0.85	0.068
	Paste	0.83, 1.1	0.97	0.078
	Canned/preserve	<0.17(4)	0.17	0.014
	pomace, wet	7.9, 7.9, 8.6, 9.7	8.3	0.66

Residues in animal commodities

Farm animal feeding studies

The Meeting received feeding studies involving lufenuron on lactating cows and steers.

Three groups of lactating cows were dosed daily at levels of 0.82, 4.3 and 8.6 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 residues of lufenuron were 0.16 mg/kg, 0.99 mg/kg and 2.5 mg/kg for the low, middle and high dose group, respectively. Skim milk and cream were analysed individually, showing residues of 0.006, 0.038 and 0.054 mg/kg for skim milk and 3.1, 24 and 32 mg/kg for cream.

In tissues mean concentrations of lufenuron with increasing dose rate were 0.03, 0.17 and 0.43 mg/kg in muscle, 0.06, 0.37 and 0.77 mg/kg in liver, 0.03, 0.22 and 0.36 mg/kg in kidney and 0.73, 4.5 and 8.0 mg/kg in fat.

In the steer study three groups of Angus steers were dosed 0.02 or 1 ppm in the diet for 28 consecutive days. Animals were sacrificed 24h after the last administrations (day 28).

Mean lufenuron residues in the low and high-dose animals were < 0.01 and < 0.01 mg/kg in muscle, < 0.01 and 0.023 mg/kg in liver, < 0.01 and 0.026 mg/kg in kidney and 0.036 and 0.23 mg/kg in fat, respectively.

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. 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, lufenuron, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	max.	mean	max.	mean	max.	mean	max.	mean
Beef cattle	0.02	0.02	0.34	0.34	0.02	0.02	none	none
Dairy cattle	0.02	0.02	0.34 [a]	0.34 [b]	0.02	0.02	none	none
Poultry - broiler	none	none	0.01	0.01	none	none	none	none
Poultry - layer	none	none	0.01 [c]	0.01 [d]	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 STMR estimates for mammalian meat and milk
[c] Highest maximum broiler or laying hen burden suitable for MRL estimates for poultry products and eggs
[d] Highest mean broiler or laying hen burden suitable for STMR estimates for poultry products and eggs
none - no relevant feed items

Animal commodities maximum residue levels

For beef and dairy cattle a maximum and mean dietary burden of 0.34 ppm was estimated. Two feeding studies on lactating cows and steers were submitted. Since no accumulation of residues in steers compared to dairy cows was observed, the Meeting decided to base its recommendations for mammalian products on the lactating cow feeding study, generally showing higher residues at identical intake levels.

Lufenuron feeding study	Feed level	Total residue				
	(ppm)	(mg/kg) in milk	(mg/kg) in muscle	(mg/kg) in kidney	(mg/kg) in liver	(mg/kg) in fat
Maximum residue level: dairy cattle						
Feeding study (HR for each dose group, except for milk)	0.82	0.16 (cream: 3.1)	0.04	0.04	0.07	1.2
Dietary burden and residue estimate	0.34	0.066 (cream: 1.2)	0.017	0.017	0.029	0.5
STMR dairy cattle						
Feeding study (Mean for each dose group)	0.82	0.16 (cream: 3.1)	0.03	0.03	0.06	0.73
Dietary burden and residue estimate	0.34	0.066 (cream: 1.2)	0.012	0.012	0.025	0.3

The Meeting estimated STMR values of 0.012 mg/kg for muscle, 0.025 mg/kg for edible offal (based on liver) and 0.3 for fat. Corresponding maximum residue levels were estimated at 0.04 mg/kg for edible offal, mammalian (based on liver) and 0.7 mg/kg for meat (based on the fat) and mammalian fat.

For milk, an STMR and a MRL of 0.066 mg/kg and 0.1 mg/kg were estimated, respectively. Based on the data for cream, the Meeting also estimated an STMR and MRL of 1.2 mg/kg and 2 mg/kg for lufenuron in milk fat, respectively.

For poultry a maximum and mean dietary burden of 0.01 ppm was estimated. No farm animal feeding studies were provided for poultry. Therefore the Meeting decided to make its recommendations based on the ^{14}C-difluorophenyl-labelled poultry metabolism study which showed higher residues than the corresponding ^{14}C-dichlorophenyl-labelled experiment.

Lufenuron feeding study	Feed level	Total residue				
	(ppm)	(mg/kg) in eggs	(mg/kg) in muscle	(mg/kg) in kidney	(mg/kg) in liver	(mg/kg) in fat
Mean and maximum residue level: poultry						
^{14}C-difluorophenyl-labelled metabolism study	3.4	2.5 [a]	0.196	0.588	1.34	9.15
Dietary burden and residue estimate	0.01	0.01	0.0006	0.0017	0.004	0.027

[a] In the metabolism study egg white and egg yolk were analysed separately. To estimate residues in whole eggs, an average ratio of 65% egg white and 35% egg yolk was taken into account: 0.65 × 0.003 mg eq/kg in egg white + 0.35 × 7.18 mg eq/kg in egg yolk=2.5 mg eq/kg in whole eggs

The Meeting estimated STMR values of 0.01 mg/kg for eggs, 0.0006 mg/kg for poultry meat, 0.004 mg/kg for poultry edible offal of (based on liver) and 0.027 mg/kg for poultry fat. Corresponding maximum residue levels for lufenuron were estimated at 0.02 mg/kg for eggs, poultry meat and edible offal of and at 0.04 mg/kg for poultry fat.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed in Annex 1 were suitable for estimating maximum residue limits and for IEDI assessment.

Definition of the residue for compliance with MRL and for dietary intake purposes for plant and animal commodities: *Lufenuron*

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
VC 0424	Cucumbers	0.09		0.02	
MO 0105	Edible offal (Mammalian)	0.04		0.025	
PE 0112	Eggs	0.02		0.01	
MF 0100	Mammalian fats	0.7		0.3	
MM 0095	Meat (from mammals other than marine mammals)	0.7 (F)		Muscle: 0.012 Fat: 0.3	
VC 0046	Melon, except watermelons	0.4		0.02 (pulp)	
ML 0106	Milks	0.1		0.066	
FM 0183	Milk fats	2		1.2	
VO 0445	Pepper, sweet	0.8		0.15	
VR 0589	Potato	0.01*		0.01	
PF 0111	Poultry fats	0.04		0.027	
PM 0110	Poultry meat	0.02		0.0006	
PO 0111	Poultry, edible offal of	0.02		0.004	
VD 0541	Soya beans (dry)	0.01*		0	
VO 0448	Tomato	0.4		0.08	
JF 0048	Tomato juice			0.014	
MW 0448	Tomato puree			0.068	
VW 0448	Tomato paste			0.078	
	Tomato preserve			0.014	
	Tomato wet pomace			0.66	

FURTHER WORK OR INFORMATION

- Poultry feeding study

DIETARY RISK ASSESSMENT

Long-term intake

The evaluation of lufenuron 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 this years estimated STMRs, were in the range 0–4% of the maximum ADI of 0.02 mg/kg bw. The results are shown in Annex 3 to the 2015 Report.

The Meeting concluded that the long-term intake of residues of lufenuron from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

For short-term intake, an ARfD was considered unnecessary. The Meeting concluded that the short-term intake of lufenuron residues from uses considered by the Meeting is unlikely to present a public health concern.

REFERENCES

Code	Author	Year	Title, Institute, Report reference
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LUFEN_003	Das, R	1998	Report on general physico-chemical properties, Syngenta Crop Protection AG, Basel, CH, Report No 62939, GLP, not published, Syngenta File No CGA184699/0547
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LUFEN_103	Tribolet, R	2000	Residue of Lufenuron (CGA 184699) in Blood and Tissues (Muscle, Fat, Liver, Kidney) of Beef Cattle (Steers) after Feeding of Lufenuron at Two Dose Levels, Syngenta Crop Protection AG, Basel, CH, Report No 104/99, GLP, not published, Syngenta File No CGA184699/0615

PYRIMETHANIL (226)

The first draft was prepared by Ms Monique Thomas. Pest Management Regulatory Agency, Canada

EXPLANATION

Pyrimethanil, an anilinopyrimidine fungicide was evaluated for the first time by the 2007 JMPR, where an ADI of 0–0.2 mg/kg bw was established and an ARfD was deemed unnecessary. At this Meeting, maximum residue levels were recommended for a limited number of uses where GAP information was available.

The residue definitions for pyrimethanil are:

- For plant products (compliance with MRLs and dietary risk assessment)—*pyrimethanil*
- For milk (compliance with MRLs and dietary risk assessment)—sum of pyrimethanil and 2-anilino-4,6-dimethylpyrimidin-5-ol, expressed as pyrimethanil
- For livestock tissues, excluding poultry (compliance with MRLs and dietary risk assessment)—sum of pyrimethanil and 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine, expressed as pyrimethanil

New GAP information, freezer storage stability studies and supervised residue trials on cane berries, bush berries and greenhouse cucumbers were provided to the current Meeting.

METHODS OF ANALYSIS

Residue trial samples from the EU were analysed using gas chromatography with mass selective detection (GC-MS) method DGM C05/98-0, which was previously evaluated by the JMPR in 2007. The North American trial samples were analysed using a similar method with minor adaptations (LC-MS/MS instead of GC-MS), in order to simplify the clean-up procedure (no hexane partition and no SPE purification step). In the case of cucumbers, an Evolute ABN SPE was used instead of a Silica SPE. The method has a demonstrated LOQ of 0.05 mg/kg.

Stability of pesticide residues in stored analytical samples

The storage stability data from the 2007 JMPR cover a diverse range of crops (apples, grapes, tomatoes, lettuce, carrots, dry peas, peaches, and plums) and demonstrated stability of pyrimethanil for up to 12 months. The samples from the submitted cane berry and bush berry supervised residue trial studies were stored for periods less than 12 months. Therefore, the current Meeting concluded that the available data is sufficient to cover the storage intervals from the berry crop field trials.

Although the stability of residues of pyrimethanil in cucumber is covered by the 12 month storage interval for the high-water content commodity group, as determined during the 2007 JMPR, the current Meeting noted that concurrent storage stability data provided with the cucumber supervised residue trials also demonstrated stability of pyrimethanil residues up to 4.5 months (the period for which the samples were stored) in greenhouse cucumbers.

The 2015 Meeting received freezer storage stability data investigating the stability of pyrimethanil in almond nutmeat and in wheat matrices.

Control samples of almond nutmeat were fortified at 0.50 mg/kg with pyrimethanil and stored in a freezer at –20 °C. Samples from Day 0 were analysed immediately after fortification, followed by time periods of 1, 3, 6 and 12-months. At each time period, a control, two freshly fortified controls, and two aged fortifications were analysed for residues of pyrimethanil.

Control samples of wheat forage, straw and grain were fortified at 0.50 mg/kg with pyrimethanil in glass jars and were stored in a freezer at –20 °C. Samples from day 0 were analysed immediately after fortification, followed by time periods of 1, 3, 6, 12, 18 and 24-months. At each time period, a control, two freshly fortified controls, and two aged fortifications were analysed for residues of pyrimethanil.

The GC-MS method DGM C05/98-0 was used to analyse residues of pyrimethanil in almond and wheat matrices.

Table 1 Stability of pyrimethanil residues in almond nutmeat spiked at 0.5 mg/kg and stored at –20 °C

Storage Interval (months)	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residues (mg/kg)	Remaining (%)	Individual Procedural Recoveries (%)	Mean Procedural Recoveries (%)
0	0.45, 0.47	0.46	100	90.1, 94.0	92.1
1	0.44, 0.45	0.45	97.0	93.0, 81.3	87.2
3	0.44, 0.39	0.42	91.0	90.1, 89.6	89.9
6	0.44, 0.43	0.44	94.8	87.7, 86.5	87.1
12	0.41, 0.46	0.44	95.7	84.2, 90.4	87.3

Table 2 Stability of pyrimethanil residues in wheat straw, forage and grain spiked at 0.5 mg/kg and stored at –20 °C

Storage Interval (months)	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residues (mg/kg)	Remaining (%)	Individual Procedural Recoveries (%)	Mean Procedural Recoveries (%)
Wheat straw					
0	0.50, 0.47	0.486	100	99.8, 94.4	97.1
1	0.33, 0.31	0.319	65.6	70.5, 70.6	70.6
3	0.35, 0.37	0.358	73.7	68.2, 69.6	68.9
6	0.42, 0.36	0.388	79.8	79.1, 83.3	81.2
12	0.36, 0.38	0.367	75.5	75.9, 80.1	78.0
18	0.46, 0.46	0.462	95.1	84.6, 93.8	89.2
24	0.31, 0.27	0.288	59.1	63.4, 64.7	64.1
Wheat forage					
0	0.38, 0.44	0.412	100	76.4, 88.5	82.5
1	0.40, 0.37	0.387	93.9	83.0, 83.4	83.2
3	0.44, 0.37	0.404	98.1	93.2, 95.4	94.3
6	0.42, 0.45	0.438	106.3	89.6, 87.2	88.4
12	0.42, 0.47	0.438	106.3	90.1, 107	98.6
18	0.44, 0.47	0.457	110.9	99.1, 101	100.1
24	0.41, 0.44	0.428	103.9	91.0, 90.5	90.8
Wheat grain					

0	0.40, 0.41	0.404	100	79.7, 82.0	80.9
1	0.30, 0.31	0.309	76.5	79.8, 84.1	82.0
3	0.35, 0.32	0.332	82.2	93.1, 98.9	96.0
6	0.34, 0.31	0.329	81.4	75.6, 71.3	73.5
12	0.38, 0.31	0.346	85.6	98.8, 85.0	91.9
18	0.39, 0.39	0.394	97.5	102, 106	104
24	0.33, 0.30	0.312	77.2	75.2, 93.6	84.4

USE PATTERNS

Crop (Remarks)	Country	Form.	Application				PHI, Days
			Method	Rate, kg ai/ha	Spray Conc. kg ai/hL	No	
Berries and other small fruits							
Blackberries, raspberries	Canada	400SC	Foliar	0.8	0.08	2	0
Raspberries	Poland	300SC	Foliar	0.75	0.075	2	3
Highbush blueberries	Canada	400SC	Foliar	0.8	0.08	2	0
Fruiting vegetables, cucurbits							
Greenhouse cucumbers	Greece, Italy, Spain	400 SC	Foliar	–	0.08	3	3

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received new information on supervised field trials involving foliar applications of pyrimethanil to the following crops.

Crop	Field/Greenhouse	Treatment Type	Countries	Table
Raspberries	Field	foliar (spray)	USA, Germany	3, 4
Blackberries	Field	foliar (spray)	USA	3
Blueberries	Field	foliar (spray)	USA	5
Cucumbers	Greenhouse	foliar (spray)	USA, CAN	6
Cucumbers	Greenhouse	foliar (spray)	France, Italy, Spain, Greece	7

Berries and other small fruits

Results from supervised residue trials on cane berries (blackberries and raspberries), and on bush berries (blueberries) conducted in the USA and raspberries conducted in Germany were provided to the Meeting.

Cane berries (blackberries and raspberries)

Five supervised field trials were conducted in the USA (2007) on cane berries (two trials on raspberries and three trials on blackberries). The blackberries and raspberries analysed in this study were held in frozen storage for a maximum of 11.6 months prior to analysis using the adapted analytical method DGM C05/98-0 by LC/MS/MS. The reported LOQ was 0.05 mg/kg. Berry samples

fortified with 0.05–9 mg/kg pyrimethanil were within the acceptable range of 70–120%, with a relative standard deviation of less than 20%.

Table 3 Pyrimethanil residues in raspberries and blackberries from supervised trials in the USA, involving two foliar applications of pyrimethanil (400 SC formulation)

Location, year (variety)	Application						DALT, days	Pyrimethanil Residues (mg/kg)	Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			
Canada GAP	400 SC	0.80	0.08	1000	2	7–10	0		Report No. RAGMP079 Doc. No. M-307677-01-1
USA, Enigma, GA, 2007 Blackberry (Arapaho)	600 SC	0.80–0.81	0.21–0.23	377–360	2	7	0	1.86 (2.22, 1.50)	
USA, Arkansaw, WI, 2007 Raspberry (Kilarney)	600 SC	0.77–0.82	0.21	363–385	2	7	0	8.38 (8.46, 8.30)	
USA, Jefferson, OR, 2007 Raspberry (Meeker)	600 SC	0.79–0.80	0.22	358–365	2	7	0	2.13 (2.47, 1.78)	
USA, Hillsboro, OR [a], 2007 Blackberry (Katata)	600 SC	0.80	0.21–0.24	337–382	2	7	0	2.62 (2.38, 2.87)	
							3	0.77 (0.70, 0.85)	
							5	0.25 (0.22, 0.27)	
							7	0.15 (0.16, 0.15)	
							10	0.10 (0.10, 0.09)	
USA, Hillsboro, OR [a], 2007 Blackberry (Boysenberry)	600 SC	0.81	0.21–0.24	345–386	2	7	0	1.69 (1.63, 1.76)	

[a] Both treatments were made on the same days, rendering the trials dependent.

Raspberries

Five supervised field trials were conducted in Germany (1999–2000) on raspberries.

The raspberries were held in frozen storage for a maximum of 259 days prior to analysis using the GC/MS Method DGM C05/98-0. The reported LOQ was 0.05 mg/kg. Raspberry samples fortified with 0.05–5 mg/kg pyrimethanil were within the acceptable range of 70–120%, with a relative standard deviation of less than 20%.

Table 4 Pyrimethanil residues in raspberries from supervised residue trials in Germany, involving three foliar applications of pyrimethanil (400 SC formulation)

Location, year (variety)	Application						DALT, days	Pyrimethanil Residues (mg/kg)	Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			
Poland GAP	300 SC	0.75	0.075	1000	3	7	3		
Germany, Neustadt-Geinsheim [a], 1999 (Rumla)	400 SC	0.80	0.13	600	3	10	0	4.65	Report No. ER99ECN274
							3	3.02	
							7	2.33	
							14	1.35	
							0	4.42	
							3	2.4	
							7	1.2	
							14	0.69	
Germany,	400 SC	0.80	0.13	600	3	9–12	0	20.17 [b]	

Location, year (variety)	Application						DALT, days	Pyrimethanil Residues (mg/kg)	Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			
Lumpzig, 1999 (Wilamette)							3	6.95	
							7	2.53	
							14	1.18	
Germany, Neustadt-Geinsheim, 2000 (Autumnbliss)	400 SC	0.80	0.13	600	3	10	0	5.14	Report No. DR 00EUN 674
							1	5.02	
							3	3.37	
Germany, Vechta-Langförden, 2000 (Schönemann)	400 SC	0.80	0.13	600	3	13–15	0	3.92	
							1	1.04	
							3	0.78	

[a] Last applications were made 25 days apart, rendering the trials independent

[b]Application and sampling before the beginning of ripening (BBCH 79). It is not compatible with a DALT = 0 (no marketable fruit available). This value is then excluded.

Bush berries (highbush blueberries)

Eight supervised field trials were conducted in the USA (2007) on highbush blueberries. The highbush blueberries analysed in this study were held in frozen storage for a maximum of 11.4 months prior to analysis using the adapted analytical method DGM C05/98-0 by LC/MS/MS. The reported LOQ was 0.05 mg/kg. Blueberry samples fortified with 0.05–6 mg/kg pyrimethanil were within the acceptable range of 70–120%, with a relative standard deviation of less than 20%.

Table 5 Pyrimethanil residues in highbush blueberries from supervised residue trials in the USA, involving two foliar applications of pyrimethanil (600 SC formulation)

Location, year (variety)	Application						DALT, days	Pyrimethanil Residues (mg/kg)	Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			
Canada GAP	400 SC	0.80	0.080	1000	2	7–10	0		
USA, Hillsboro, OR, 2007 (Bluecrop)	600 SC	0.79–0.81	0.66–0.73	111–119	2	7	0	2.11 (2.17, 2.04)	Report No. RAGMP037
USA, Fennville, MI, 2007 (Jersey)	600 SC	0.80	0.50–0.51	157–161	2	7	0	1.89 (1.80, 1.97)	
USA, Hixton, WI, 2007 (Patriot)	600 SC	0.79–0.80	0.21	371–374	2	7	0	2.14 (1.70–2.59)	
USA, Elizabethtown, NC, 2007 (Reka)	600 SC	0.79–0.81	0.51–0.54	146–158	2	7	0	2.27 (2.37–2.16)	
USA, Covert, MI, 2007 (Jersey)	600 SC	0.80	0.14–0.16	495–580	2	7	0	5.13 (5.76, 4.50)	
USA, Chula, GA, 2007 (Brightwell)	600 SC	0.79–0.80	0.21	378–382	2	7	0	1.40 (1.44, 1.36)	
USA, Ochlocknee, GA, 2007 (Tifblue)	600 SC	0.80–0.82	0.16–0.17	466–508	2	7	0	1.08 (1.05, 1.12)	
							1	1.12 (1.10, 1.15)	
							3	0.64 (0.63, 0.66)	
							7	0.32 (0.32, 0.32)	

							10	0.18 (0.14, 0.22)	
USA, New Tripoli, PA, 2007 (Bluecrop)	600 SC	0.79–0.81	0.17	462–472	2	7	0	2.00 (1.92, 2.08)	

Fruiting vegetables, cucurbits

Greenhouse Cucumbers—North America

Five greenhouse trials were conducted in Canada and the USA (2010–2011) on cucumbers.

The cucumber samples analysed in this study were held in frozen storage for a maximum of 4.6 months prior to analysis using the adapted analytical method DGM C05/98-0 by LC/MS/MS. The reported LOQ was 0.05 mg/kg. With the exception of one recovery of 68%, cucumber samples fortified with 0.05–5 mg/kg pyrimethanil were within the acceptable range of 70–120%, with a relative standard deviation of less than 20%.

Table 6 Pyrimethanil residues in greenhouse cucumbers from supervised trials in the USA and Canada, involving three foliar applications of pyrimethanil (400 SC formulation)

Location, year (variety)	Application						DALT, days	Pyrimethanil Residues (mg/kg)	Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			
USA, Salisbury, MD, 2010 (Samir)	400 SC	0.80	0.07	1132	3	7	1	0.07 (0.07, 0.07)	Report No. AAC10-056R Doc. No. M-477841-01-1
USA, Raleigh NC, 2010 (Jawell F1)	400 SC	0.78–0.80	0.08	988–1016	3	7	1	0.38 (0.38, 0.38)	
USA, Citra FL, 2011 (Jawell F1)	400 SC	0.79–0.81	0.09	926–948	3	13–14	1	0.47 (0.44, 0.49)	
USA, Parlier CA, 2010 (Cumlaude)	400 SC	0.81–0.85	0.07	1133–1191	3	7	1	0.82 (0.83, 0.80)	
Canada, Harrow ON, 2010 (Camaro)	400 SC	0.79–0.80	0.04	1982–2008	3	7–8	0	0.46 (0.50, 0.42)	
							1	0.45 (0.48, 0.42)	
							5	0.33 (0.33, 0.33)	
							11	0.17 (0.16, 0.17)	
							14	0.14 (0.14, 0.14)	

Greenhouse Cucumbers—Southern Europe 1997–1998

Nine greenhouse trials were conducted in the EU (1997–1998) on cucumbers.

The cucumber samples analysed in this study were held in frozen storage for a maximum of 6 months prior to analysis using the validated GC/MS method DGM C05/98-0. The reported LOQ was 0.05 mg/kg. With the exception of one recovery of 65%, cucumber samples fortified with 0.05–0.50 mg/kg pyrimethanil were within the acceptable range of 70–120%, with a relative standard deviation of less than 20%.

Table 7 Pyrimethanil residues in protected cucumbers from supervised residue trials in Southern EU, in 1997–1998 involving three foliar applications of pyrimethanil (400 SC formulation)

Location, year(variety)	Application						DALT, days	Pyrimethanil Residues (mg/kg)	Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			
Southern EU GAP (Greece, Italy, Spain)	400 SC	0.80	0.08	1000	3	10–14	3		
Southern France, Ledenon, 1997 (Girola)	400 SC	0.80	0.08	1000	3	10–14	0 1 3 7	0.09 0.09 <u>0.12</u> 0.12	Report No. ER97ECS 261
Italy, Mantova, 1997 (Darina)	400 SC	0.80	0.08	1000	3	12	0 1 3 7	0.45 0.20 <u>0.16</u> 0.04	
Spain, Alboraya, 1997 (Potomac F1)	400 SC	0.80	0.08	1000	3	12–14	0 1 3 7	0.42 0.50 <u>0.32</u> 0.12	
Greece, Ionia, 1997 (Hitel F1 RS)	400 SC	0.80	0.08	1000	3	10–11	0 1 3 7	0.88 0.45 <u>0.24</u> 0.08	
Spain, Sueca, 1997 (Potomac)	400 SC	0.80	0.08	1000	3	11	0 1 3 7	1.02 0.60 <u>0.25</u> 0.18	
France, Bruges, 1998 (De Ruiter)	400 SC	0.80	0.08	1000	3	10	0 3	0.31 <u>0.37</u>	Report No. ER98ECS 261
Greece, Esovalta, 1998 (Babina)	400 SC	0.80	0.04	2000	3	8–10	0 3	0.50 <u>0.19</u>	
Italy, Molfetta, 1998 (Cetriolo di Polignano)	400 SC	0.80	0.05	1500	3	9–11	0 3	0.49 <u>0.29</u>	
Portugal, Torres Vedras, 1998 (Jazzer)	400 SC	0.80	0.08	1000	3	9–10	0 3	0.51 <u>0.10</u>	

APPRAISAL

Pyrimethanil, an anilinopyrimidine fungicide, was evaluated for the first time by the 2007 JMPR, where an ADI of 0–0.2 mg/kg bw was established and an ARfD was deemed unnecessary. It was listed by the Forty-sixth Session of the CCPR (2014) for the evaluation by the 2015 JMPR for additional MRLs. New GAP information, freezer storage stability studies and supervised residue trials on cane berries, bush berries and greenhouse cucumbers were provided to the current Meeting

Residue definitions are:

- For compliance with the MRL and for dietary intake estimation for plant commodities: pyrimethanil
- For compliance with the MRL and for dietary intake estimation for milk: sum of pyrimethanil and 2-anilino-4,6-dimethylpyrimidin-5-ol, expressed as pyrimethanil
- For compliance with the MRL and for dietary intake estimation for livestock tissues (excluding poultry): sum of pyrimethanil and 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine, expressed as pyrimethanil

The residue is not fat-soluble.

Stability of pesticide residues in stored analytical samples

Based on the storage stability data submitted, the Meeting concluded that no significant dissipation of pyrimethanil residues was observed in almond nutmeat after 12 months of storage or in wheat straw, forage and grain after 24 months of storage.

Results of supervised residue trials on crops

The Meeting received new supervised trial data for foliar applications of pyrimethanil (SC formulations) on cane berries (blackberries and raspberries), bush berries (blueberries), and greenhouse cucumbers.

Berries and other small fruits

Results from supervised field trials on blackberries, raspberries, and blueberries conducted in North America were provided to the Meeting, including raspberry data from Germany.

Cane berries (blackberries and raspberries)

Results from supervised field trials on blackberries and raspberries conducted in the USA and trials on raspberries conducted in Germany were provided to the Meeting.

A total of four independent supervised trials were conducted in the USA on blackberries and raspberries according to the critical GAP of Canada for cane berries (blackberries and raspberries) which allows a maximum of 2 applications of 0.8 kg ai/ha/application, and a PHI of 0 day.

Residues of pyrimethanil matching the Canadian GAP were: 1.86, 2.13, 2.62 and 8.38 mg/kg.

A total of five independent supervised trials were conducted in Germany on raspberries according to the Poland critical GAP for raspberries which allows a maximum of 3 applications of 0.8 kg ai/ha/application, and a PHI of 3 days.

Residues of pyrimethanil in raspberries matching the Poland GAP were: 0.78; 2.40; 3.02; 3.37 and 6.95 mg/kg.

The Meeting agreed to use the data set according to the Canadian GAP and estimated a maximum residue level of 15 mg/kg and an STMR of 3.02 mg/kg from the German trials for cane berries.

Bushberries-Blueberry

Results from supervised field trials on highbush blueberries conducted in the USA were provided to the Meeting.

A total of eight independent supervised trials were conducted in the USA on highbush blueberries according to the critical GAP in Canada for bush berries which allows a maximum of 2 applications of 0.8 kg ai/ha/application, and a PHI of 0 day.

Residues of pyrimethanil in highbush blueberries conducted in North America matching the GAP were: 1.12, 1.40, 1.89, 2.00, 2.11, 2.14, 2.27, and 5.13 mg/kg.

The Meeting estimated a maximum residue level of 8 mg/kg and an STMR 2.06 mg/kg for pyrimethanil on blueberries.

Greenhouse cucumbers

Results from supervised field trials on greenhouse cucumbers conducted in North America and Southern Europe were provided to the Meeting.

In the absence of a North American GAP for greenhouse cucumbers, the Meeting did not consider the USA and Canada trials in estimating a maximum residue level.

A total of nine independent supervised trials were conducted in Southern Europe on greenhouse cucumbers according to the critical GAPs in Greece, Italy, and Spain which allow a maximum of 3 applications of 0.8 kg ai/hL/application, and a PHI of 3 days.

Residues of pyrimethanil in greenhouse cucumbers matching the Southern EU GAP were: 0.10; 0.12; 0.16; 0.19; 0.24; 0.25; 0.29; 0.32; and 0.37 mg/kg.

The Meeting estimated a maximum residue level of 0.70 mg/kg and an STMR of 0.24 mg/kg for residues of pyrimethanil in greenhouse cucumbers.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed in Annex 1 are suitable for establishing maximum residue levels and for IEDI assessment.

Definition of the residue for compliance with the MRL and for the estimation of dietary intake for plant commodities: *pyrimethanil.*

Definition of the residue for compliance with the MRL and for dietary intake estimation for milk: sum of pyrimethanil and 2-anilino-4,6-dimethylpyrimidin-5-ol, expressed as pyrimethanil.

Definition of the residue for compliance with the MRL and for dietary intake estimation for livestock tissues (excluding poultry): sum of pyrimethanil and 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine, expressed as pyrimethanil.

The residue is not fat soluble.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	New		
FB 0264	Blackberries	15		3.0	
FB 0020	Blueberries	8		2.1	
VC 0424	Cucumbers	0.7		0.24	
FB 0272	Raspberries	15		3.0	

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes of Pyrimethanil for the GEMS/Food 17 cluster diets, based on estimated STMRs were 0% of the maximum ADI of 0.2 mg/kg bw. The Meeting concluded that the long-term intake of residues of pyrimethanil from uses considered by the current Meeting is unlikely to contribute to the overall intake and will not present a public health concern.

Short-term intake

The 2007 JMPR determined that no ARfD was considered necessary. Therefore the short-term intake of pyrimethanil residues from uses considered by the current Meeting is unlikely to present a public health concern.

REFERENCES

Code	Author(s)	Year	Title, Institute, Report reference
03RAP1X001	Tauber, R	2005	Frozen storage stability of Pyrimethanil and its metabolite AE C621312 in wheat forage, straw, and grain. Enviro-Test Laboratories, Ontario, Canada, Bayer CropScience Report No.: 03RAP1X001, Date 2005-11-15. GLP, unpublished M-264524-02-1
RAP1Y009	Tauber, R	2005	Frozen storage stability (12 months) of Pyrimethanil (AE B100309) in

Code	Author(s)	Year	Title, Institute, Report reference
			almond nut meat using GC/MS. Enviro-Test Laboratories, Ontario, Canada, Bayer CropScience Report No.: RAP1Y009, Date 2005-11-15, amended 2006-01-31. GLP, unpublished M-269503-03-1
RAGMP079	Dallstream, KA & Fischer, DR	2008	AE C656948 500 SC + pyrimethanil 600 SC—Magnitude of the residue in/on caneberry. Bayer CropScience LP, Environmental Research, Stilwell, KS, USA, Bayer CropScience Report No.: RAGMP079, Date 2008-09-17. GLP, unpublished M-307677-01-1
C013366	Sonder, KH	2001	Decline of residues in raspberries European Union (Northen zone) 2000—Pyrimethanil water miscible suspension concentrate (SC) 37.38 percent w/w (=400 g/L) Code: AE B100309 00 SC37 A404. Aventis CropScience GmbH, Frankfurt am Main, Germany, BASF Report No.: C013366, Date 2001-07-31. GLP, unpublished M-204476-01-1
C008559	Sonder, KH & Peatman, M	2000	Decline of residues in raspberries European Union (Northen zone) 1999—Pyrimethanil water miscible suspension concentrate (SC) 37.38 percent w/w (=400 g/L) Code: AE B100309 00 SC37 A404. Aventis CropScience GmbH, Frankfurt am Main, Germany, BASF Report No.: C008559, Date 20001-11-09. GLP, unpublished M-197582-01-1
RAGMP037	Fischer, DR	2008	AE C656948 500 SC + pyrimethanil 600 SC—Magnitude of the residue in/on bushberry (crop subgroup 13B). Bayer CropScience LP, Environmental Research, Stilwell, KS, USA, Bayer CropScience Report No.: RAGMP037, Date 2008-09-17. GLP, unpublished M-307682-01-1
AAFC10-056R	Ballantine, J	2014	Pyrimethanil: Magnitude of the residue on cucumber, greenhouse. Trace Analytical Laboratory, University of California, USA, Bayer CropScience Report No.: AAFC10-056R, Date 2014-01-27. GLP, unpublished M-477841-01-1
A91283	Old, J, Smith, A & Doran, A	1998	Residue trials in protected cucumbers for establishment of an MRL following three applications in Southern Europe 1997 pyrimethanil suspension concentrate 400 g/L. Inveresk Research Int. Ltd., Tranent, Scotland, Bayer CropScience Report No.: A91283, Date 1998-07-22. GLP, unpublished M-167980-01-1
C003104	Sonder, KH	1999	Residues at harvest in cucumbers, European Union, southern zone, 1998 Pyrimethanil = AE B100309 water miscible suspension concentrate (SC) 37.38 percent w/w (=400 g/L). Hoechst Shering AgrEvo GmbH, Frankfurt am Main, Germany, Bayer CropScience Report No.: C003104, Date 1999-05-05. GLP, unpublished M-185627-01-1

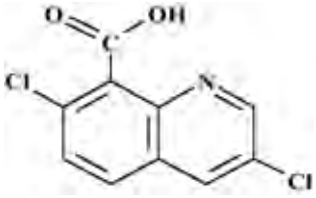
QUINCLORAC (287)

First draft was prepared by Dr Anita Stromberg, National Food Agency, Uppsala, Sweden

EXPLANATION

Quinclorac (ISO common name) is a quinone carboxylic herbicide used to control annual grass and broadleaf weed species in barley, canary seed, rape seed (canola), non-crop areas, pasture, rhubarbs cranberry, rice, sorghum and wheat. The herbicide has an auxin activity similar to that of indolylacetic acid and belongs to the auxin-type class of herbicides that includes the phenoxy-acids, benzoic acids and pyridine compounds.. The use of quinclorac results in the rupture of the cell membranes due to overstimulation of the growth of the plant. Quinclorac is mainly adsorbed via the root system and partly through foliage, mainly for the pre- and post-emergence control of *Echinocloa* spp, but also other weeds like *Aeschynomene* spp., *Sesbania* spp., and *Ipomoea* spp. occurring in direct-seeded and transplanted rice. Quinclorac was scheduled by the 46h session of the CCPR (2014) as a new compound for consideration by the 2015 JMPR.

IDENTITY

ISO common name	Quinclorac
Chemical name, IUPAC	3,7-dichloroquinoline-8-carboxylic acid
Chemical name, CA	3,7-dichloro-8-quinoline carboxylic acid
CIPAC No.	493
CAS No.	84087-01-4
Structural formula	
Molecular formula	$C_{10}H_5Cl_2NO_2$
Molecular mass	242.1 g/mol

PHYSICAL AND CHEMICAL PROPERTIES

Property	Results	Method (test material)	Reference
Appearance	Off-white powder		JMPS, Quinclorac 2002 Evaluation report 493/2002
Melting point	The melting point quinclorac pure (99.8%): 272.4-276.9 °C The melting point of quinclorac technical (purity 98.7) at atmospheric pressure is 279.9°C.	OECD 102	JMPS, Quinclorac 2002 Evaluation report 493/2002 Kroehl, T. 2010 2010/1057264
Boiling point	No boiling point of quinclorac technical (purity 99, 8%) before melting. At the end of melting gas evolution begins.	OECD 102	Daum, A. 1999 1999/11542
Relative density	Quinclorac technical (purity 99.8%): $D_4^{20} = 1.68$	EEC A3, OECD 109	Kästel, R. 2001 2001/1010797
Vapour pressure	Quinclorac technical (purity 98.7%): 4.9×10^{-11} mbar (hPa) at 25ºC 1.9×10^{-11} mbar (hPa) at 20ºC	OPPTS 830.7950	Kroehl, T. 2010, 2010/1057264
Henry´s law constant Coefficient	Henry´s law constant at 20 ºC (calculated) 3.381×10^{-13} kPa m3 / mol	Calculation	Ohnsorge, U, 2001 2001/1014896
Physical state, colour	Quinclorac, pure: white crystals	OECD 102	Daum, A. 1999

Property	Results		Method (test material)	Reference
				1999/11542
Odour	Quinclorac, pure: odourless		OECD 102	Daum, A. 1999 1999/11542
	Quinclorac technical; characteristic odour, free from visible extraneous matter and added modifying agents			JMPS, Quinclorac 2002 Evaluation report 493/2002
Solubility in water at 20ºC including effect of pH	Quinclorac, pure: 80.1 mg/L at pH 3 61.5 mg/L at PH 6.1		OECD 105 EC A.6	Daum, A. 2005 2005/1005667
	Quinclorac, (purity 99.8%) 0.072 g/l at pH 5.5 (deionized water) 75.9 g/l at pH 10.3 (NaOH, 0.1 Mol/l		EEC A8, by extrapolation	JMPS, Quinclorac 2002 Evaluation report 493/2002
Solubility in organic solvents	g/L 20 ºC:		OECD 105 EC A.6.	Daum, A. 2005 2005/1008919
	Methanol	2.7		
	Acetone	2.8		
	Ethyl acetate	0.9		
	dichloromethane	0.5		
	Toluene	0.006		
	n-heptane	0.003		
Dissociation in water	Quinclorac, pure (99.4%): Quinclorac has the character of an acid pKa = 4.34 at 20ºC pKa = 4.35 at 25ºC		OECD 112, titration method	Redeker, DC 1988 88/0137 JMPS, Quinclorac 2002 Evaluation report 493/2002
Partition coefficient n-octanol/water	Quinclorac technical (purity 99.4%): log Pow = 1.78 (at pH 4) log Pow = -0.72 (at pH 7)		OECD 117 (HPLC-method)	Daum, A. 2005 2005/1005668
	Quinclorac technical (purity 99.8%): log POW = 1.76 at 20 °C (at pH 4) log POW = -0.74 at 20 °C (at pH 7) log POW = -3.74 at 20 °C (at pH 10)		EEC A8, by extrapolation	JMPS, Quinclorac 2002 Evaluation report 493/2002
Hydrolysis rate	Half-life > 30 days at 25 °C (at pH 5, pH 7 and pH 9).		US-EPA Assessment guidelines, Subdiv. N, 161-2 (1982)	JMPS, Quinclorac 2002 Evaluation report 493/2002
Photochemical characteristics	In sterile aqueous buffer solution pH 7 using artificial light in the wavelength 300-800 nm at 25ºC. Half-life = approx. 100 days (continuous illumination) Half-life = approx. 43 days (sensitized, = 0.5% acetone),		EPA 161-2	Ellenson, JL. 2001 2001/5000828
	Half life = ca. 100 days (nonsensitized, sterile solution, calculated for continuous illumination) Half life = ca. 43 days (sensitized , sterile solution, calculated for continuous illumination) Experimental setup: solution in water (sterile), pH 7, 25°C, simulated sunlight at 805 w/m², for 660 h over 35 d (15 h light, 9 h dark, illuminated at weekends). Result: Half life > 30 days (dark control solution, non-sensitized, sterile, see hydrolysis) The results were used to extrapolate the half life values above. Half -life > 30 days (dark control solution, non-sensitized sterile)		US-EPA Assessment guidelines, Subdiv. N, 161-2 (1982)	JMPS, Quinclorac 2002 Evaluation report 493/2002

Hydrolysis of quinclorac

A hydrolysis study was carried out by Hassink, J (2005/1016370). Quinclorac at a concentration of 29.9 µg/L was investigated in aqueous solution at pH 4, 5, 7 and 9 at 25 ºC. Samples were taken 0, 2, 7, 9, 11, 14, 21 and 30 days after treatment and analysed using LC/MS.

A summary of the results is presented in the table below.

Table 1 Summary of hydrolysis of quinclorac at pH 4, pH 5, pH 7 and pH 9 at 25ºC

DAT [a]	pH4		pH5		pH7		pH9	
	µg/L	%[b]	µg/L	%[b]	µg/L	%[b]	µg/L	%[b]
0	28.8	100	28.5	100	27.6	100	27.5	100
2	28.6	99.3	27.8	97.5	27.6	100	27.7	100.7
7	29.3	101.7	28.4	99.6	28.0	101.4	28.1	102.2
9	29.1	101.0	28.2	98.9	29.3	106.2	28.7	104.4
11	31.6	109.7	30.6	107.4	29.8	108.0	30.0	109.1
14	29.3	101.7	29.1	102.1	29.0	105.1	29.5	107.3
21	28.0	97.2	29.4	103.2	29.1	105.4	28.6	104.0
30	29.4	102.1	29.3	102.8	28.7	104.0	28.7	104.4

[a] DAT = days after treatment

[b] % of initial applied test item, concentration of day 0 set to 100%

Formulations

Quinclorac is applied as a single active ingredient in different formulations.

Table 2 Available formulations of containing quinclorac as active ingredient

Formulation	Content of active ingredient
WP (wettable powder)	50% w/w
WG (wettable granules)	75% w/w
SC (suspension concentrates)	250 g/L
SL (soluble concentrates)*	180 g/L
FL (liquid flowable)*	40% w/w
DF (dry flowable)*	75% w/w

* Formulation in registered label presented to the 2015 JMPR Meeting

METABOLISM AND ENVIRONMENTAL FATE

Metabolism studies were conducted using 2, 3, 4-[^{14}C]-quinclorac (quinolone-label, Fig. 3) or 3-[^{14}C]-quinclorac (quinolone-label, Fig 2). In strawberry the [^{14}C]-quinclorac (quinolone-label, Fig. 3) was used. The position of the labels for both substances is presented in the following figures:

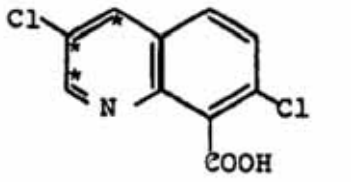


Figure 1 2, 3, 4-[^{14}C]-quinclorac

*location of the radiolabel

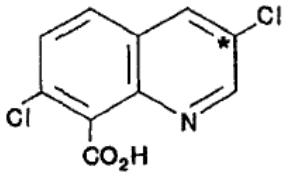


Figure 2 3-[^{14}C]-quinclorac

*location of the radiolabel

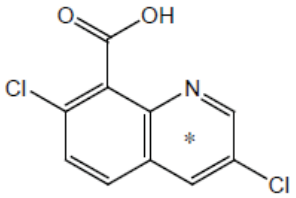
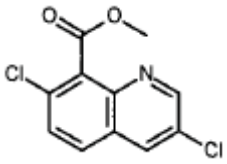
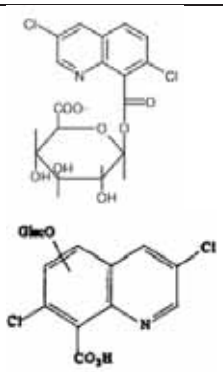
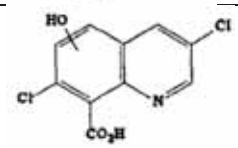


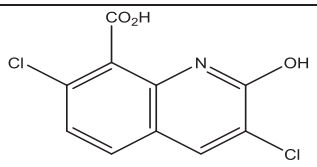
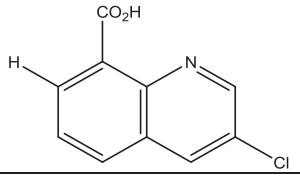
Figure 3 [^{14}C]-quinclorac

*location of the radiolabel

Chemical names, structures and code names of metabolites and degradation products of quinclorac are presented in the table below.

Table 3 Known metabolites of quinclorac from studies provided in animal, plants and soil matrices

Codes	Molecular formula and Nominal mass	Structure	Occurrence
BH 514-Me Reg. No. 161555 Quinclorac methyl ester SES218	$C_{11}H_7Cl_2NO_2$ methyl-3,7-dichloroquinoline-8-carboxylate 255		rats[1] canola sorghum rotational crops (mustard green, turnip, barley) soil (terrestrial aerial metabolism)
BAS 514 H M1 glucuronide (glucuronic acid) conjugate Quinclorac glucose conjugate	419		rat[1] goat hen wheat strawberry
Hydroxy-quinclorac	Hydroxy-quinclorac 257		wheat

Codes	Molecular formula and Nominal mass	Structure	Occurrence
BH 514-2-OH 2 -hydroxyquinclorac	$C_{10}H_5Cl_2NO_2$ 3,7-dichloro-2-hydroxyquinoline-8-carboxylic acid 258		soil (terrestrial metabolism)
BH 514-1 3-chloroquinoline-8-carboxylic acid	$C_{10}H_6ClNO_2$ 3-chloroquinoline-8-carboxylic acid 207.6		soil (aquatic metabolism)

Animal metabolism

The Meeting received metabolism studies on laboratory animals, poultry and lactating goats using the 2, 3, 4-[^{14}C]-quinclorac (quinoline label).

Laboratory animals

Rats

In rats given 2, 3, 4-[^{14}C]-quinoline labelled quinclorac orally absorption was rapid and accounted for at least 85.5% given a single administration of low and high doses (15 mg/kg and 600 mg/kg bw, respectively). The maximum plasma concentration of radioactivity was reached approximately 30 minutes after administration of the low or high dose. The half life in plasma was 3-4 hours for the low dose and 12-13 hours for the high dose. Radioactivity was widely distributed throughout the body. Elimination of radioactivity was mainly via urine (> 91%) for both female and male rates, while faecal excretion ranged from 0.7 to 3.7% of the dose. The bile was a minor route of excretion of the low dose but was found to be a significant route after administration the high dose (600mg/kg). Minor radioactivity was excreted in the faeces of intact rats dosed at this level, indicating that the greater part of the biliary excreted radioactivity was reabsorbed and eliminated via urine.

Biotransformation of quinclorac was minimal. The parent and one metabolite (a glucuronide of quinclorac) was identified in the urine. The metabolism of quinclorac was characterized in the bile where > 18 minor (< 10% TRR) metabolites were identified. The metabolism is characterized by two primary reactions; nucleophilic substitution of the chlorine atom at the isocycle with glutathione and formation of an arene oxide intermediate followed by reactions with glutathione to form S-conjugates and/or by addition of water to form hydroxylated derivatives. Metabolite M1 (glucuronic acid conjugates of quinclorac) was the major metabolite identified in the liver and kidney

Livestock

Lactating goats

The kinetic behaviour and the metabolism of 2, 3, 4-[^{14}C] quinclorac was investigated by Hawkins *et al.* (1986, BASF 86/0434, 1987 BASF 86/0473).

One lactating goat (47 kg) was dosed orally daily for five days with 34 mg radiolabelled quinclorac per kg bodyweight/day (1600 mg/animal/day, equivalent to 800 ppm in the diet). The goat was sacrificed 6 hours after the last dose. Milk, plasma, urine and faeces were collected during the whole dosing period. After sacrifice liver, kidney, fat and loin muscle were sampled.

Analysis of the total radioactive residue (TRR) was carried out using combustion and or liquid scintillation counting (LSC). In total 66.7% of applied radioactivity was recovered in the experiment. The relatively low recovery of applied radioactivity is explained by unabsorbed radioactivity within gastrointestinal tract due to the termination of the sacrifice of the animal relatively early, 6 hours after the final dose. The passage time of material within the GI tract of ruminants can be up to 72 hours. The GI tract was not analysed for TRR. Excretion of radioactivity in

urine, faeces and milk accounted for 63.0%, 3.7% and 0.003%, respectively of the total dose up to 6 hours after the final dose.

The TRR found in organs and tissues were about 0.2% of the applied radioactivity, with levels being highest in kidney (10.3 mg eq/kg) followed by liver (2.13 mg eq/kg), fat (subcutaneous: 0.78 mg eq/kg, omental 0.14 mg eq/kg) and muscle (leg: 0.19 mg eq/kg, loin: 0.16 mg eq/kg). In milk the TRR level increased from 0.034 mg eq/kg directly after the first administration up to 0.055 mg eq/kg after two days, then down to 0.032 mg eq/kg at day three and back to 0.056 mg eq/kg day, a plateau level was thus not reached. The TRR levels found are summarized in Table 4. The radioactivity in milk over time is presented in table 5.

Table 4 TRR in goats milk and tissue after daily oral administration for five days with 2, 3, 4-^{14}C-quinclorac at 34 mg/kg bw/ day (equivalent to 800 ppm in the diet) 6 h after sacrifice.

Matrix	% of total (cumulative)dose administrated	TRR (mg eq/kg)
Liver	0.12	2.13
Kidney	0.10	10.3
Leg muscle	n.r.	0.19
Loin muscle	n.r	0.16
Omental fat	n.r.	0.14
Subcutaneous fat	n.r.	0.78
Total in organs and tissue	0.22	13.7
Milk 0-102h	0.003	see table 5
Urine, 0-120 h*	47.8	
cage washes	15.2	
Faeces, 0-120 h	3.7	
Total excreted in urine, cage washes, faeces	66.7	
Total excreted in organ, tissue milk, urine, cage washes .and faeces	66.92	
Bile**	n.r.	4.7
Plasma**	n.r.	2.09
Whole-blood	n.r.	1.44

* Includes cages washes

** Concentration is expressed as µg equivalents quinclorac/mL

n.r. not reported

Table 5 Concentration of radioactivity in milk during after daily oral administration of 3, 4-^{14}C-quinclorac at 34 mg/kg bw/ day (equivalent to 800 ppm in the diet) to one goat for five days

Time period (hours after first dose)	one goat TRR (mg eq/kg) milk		
	afternoon collection	morning collection*	mean concentration (total 24 h collection)
0-24	0.052	0.025	0.034
24-48	0.088	0.043	0.055
48-72	0.078	0.026	0.038
72-96	0.039		0.032
96-102	0.056		0.056

***** Refers to morning following the afternoon collection immediately prior to the next dose..

Milk (day 2, PM) was mixed with methanol to precipitate the proteins. After centrifugation the methanolic extracts were reduced by evaporation and mixed with 1 M HCl. The extract was fractionated by column chromatography (C-8, octylsilane). The column was washed sequentially with 1 M HCl and hexane followed by elution with ethyl acetate. The ethyl acetate extracts were evaporated to dryness and reconstituted in methanol.

Liver and kidney samples were homogenized and extracted with 1 M HCl/ethyl acetate (1:10, v/v) for 10-20 min followed by centrifugation. The ethyl acetate extracts were evaporated to dryness and reconstituted in methanol.

Fat and muscle samples were extracted with water/ethanol (1:9, v/v) and 1 M NaOH , followed by extraction with methanol. Both extracts were combined, concentrated by evaporation and mixed with 1 M HCl. The extract was fractionated as described for milk.

The extraction efficiency was generally > 90% TRR except in muscle where it was 83% TRR. In muscle tissue 17% TRR (mg eq/kg not given) of the residue was not extracted and attempts to further extract this solid material was not performed. Identification of the radioactivity in extracts and column fractions was done using TLC in five different solvent systems. The reference substance used was the parent quinclorac which was confirmed on TLC plates by the quenching of UV fluorescence at 254 nm.

Quinclorac is not significantly metabolized in the goat. Parent quinclorac was present at levels >80% TRR in milk, liver and kidney. The metabolite M1 (glucuronic acid conjugate of the parent) was present at levels of 4.0% TRR in milk and at 4.7% TRR in kidney. Three other unidentified fractions (R01, R03 and R05) were found at levels of 0.4–5.4% TRR in milk, liver and kidney.

Since the methods used to extract the residues included 1 M HCl, it is not clear whether parent compound represents parent only or includes parent released from conjugates and whether the M1 is the fraction of conjugates that remained uncleaved.

Table 6 Characterization and identification of compounds in milk, tissues and urine of the lactating goat after administration of 2, 3, 4-^{14}C-quinclorac at 34 mg/kg bw/day (800 ppm in the diet)

Compound/fraction	Milk day 2 PM 0.088 mg/kg eq	Loin muscle 0.16 mg/kg eq	subcu taneous fat 0.78 mg/kg eq	Liver 2.13 mg/kg eq	Kidney 10.3 mg/kg eq	Urine %TRR*	
	%TRR*	%TRR*	%TRR*	%TRR*	%TRR*	0-24 h	96-102h
Total identified	91.1			81.3	91.2	98	96.6
- Quinclorac	86.1	***	***	81.3	86.5	95.1	95.4
- M1 (R02)	4.0				4.7	2.9	1.2
Total characterized	6.9			5.6	4.4	0.19	3.4
- R01-1	1.2			1.8**	2.0	0.7	1.5
- R01-3	0.3				0.4	0.5	0.2
- R01-5	5.4			3.8	2.0	0.7	1.7
Total extracted	97.0	82.6	100.0	86.9	95.6	99.9	100
- Post extracted solids	3.7	17.4	-	13.1	4.4	0	0

* Relative % of total sample radioactivity

** Includes more than 2 regions of interest (rf 41-53)

***. not quantified because of the low levels of radioactivity; qualitatively all radioactivity co-chromatographed with parent compound

M1 is glucuronic acid conjugate of parent

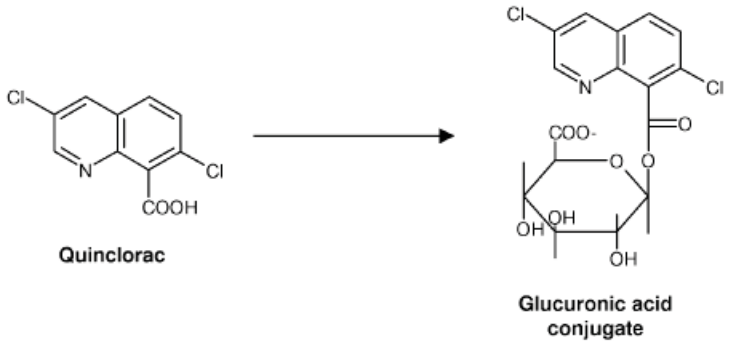


Figure 4 Metabolic pathway from available data in lactating goat

Laying hens

The kinetic behavior and the metabolism of 2, 3, 4-^{14}C-quinclorac in laying hens was investigated by Hawkins et al. (1986, BSF 1986/5003). Seven birds (366-372) were selected from a group of 15 birds based on egg laying records. These hens (1.8–2.4 kg) were orally dosed once for five days with 33–44 mg radiolabelled quinclorac per kg body weight per day (80 mg/bird/day) corresponding to 800 ppm in the diet (based on a food consumption of 100 g/day). Excreta and eggs were collected daily during the dosing period. The number of laid egg varied significantly from hen to hen. For example two birds did not lay any eggs during the study, and for two of the birds all eggs were broken and no eggs collected during the sampling time point. The birds were sacrificed 6 hours after the last dose and liver ,kidney, muscle, skin with underlying fat were collected. Blood samples were collected just prior to sacrifice and separated by centrifugation into plasma and cells.

Total radioactivity was measured in excreta, eggs and tissues using combustion and/or LSC. The TRR levels found in tissues and eggs (in concentration and per eggs) are summarized in Tables 7 and 8. Excretion of radioactivity in excreta was 87.5–95.1% of the applied radioactivity up to 6h after the final dose. The total radioactive residue (TRR) were highest in the kidney (0.77–88.98 mg eq/kg), followed by liver (0.26–10.53 mg eq/kg), plasma (0.14–13.50 mg eq/kg), whole blood (0.09–9.41 mg eq/kg), skin/fat (0.23–7.2 mg eq/kg and leg muscle (0.05–3.95 mg eq/kg). Plasma levels and tissue concentration obtained at sacrifice, 6 hours after the final dose, showed considerable inter-animal variation.

In eggs the TRR levels increased from 0.06 mg eq/kg one day after first administration up to an average plateau of 0.18–0.65 mg eq/kg after four days. Levels showed a wide variation as also found in plasma and tissue. One bird reached a plateau of 1.06 mg eq/kg after three days, while two other birds reached a plateau after two days (1.21 and 0.27 mg eq/kg.

Table 7 TRR in egg and tissue after administration of 2, 3, 4-^{14}C-quinclorac at 33-44 mg/kg bw and day (equivalent to 800 ppm in the diet) 6 h after sacrifice

Matrix	% of total dose administrated	TRR (mg eq/kg)							
		Bird number							
		366	367	368	369	370	371	372	average
Liver	n.r.	0.78	9.39	4.41	0.43	0.38	0.26	10.53	3.74
Kidney	n.r.	5.86	37.44	4.13	1.27	0.92	0.77	88.98	19.91
Breast muscle	n.r.	0.17	3.2	0.20	0.05	0.05	< 0.05	4.22	1.82
Leg muscle	n.r.	0.24	3.37	0.18	0.06	0.05	0.11	3.95	1.14
Skin/fat	n.r.	0.61	5.28	0.62	0.25	0.23	0.17	7.20	2.05
Plasma	n.r.	0.66	11.22	0.65	0.24	0.15	0.14	13.5	3.79
Whole blood	n.r.	0.48	8.38	0.41	0.19	0.12	0.09	9.41	2.73
Total in organs and tissue	-								
Excreta 0-120 h*	92.6 ± 5.6								

Table 8 Time course of total radioactive residues (mg eq/g) in eggs laid by hens, after administration of 2, 3, 4-^{14}C-quinclorac at 33-44 mg per kg body weight, for five days.

Day of collection	Bird number TRR (mg eq/kg)							Mean TRR (mg eq/kg)	**±SD**
	366	**367**	368	369	370	371	**372**		
1	ns	ns	ns	ns	< 0.06	ns	0.06	< 0.06	
2	ns	1.21	0.27	ns	B	ns	0.42	0.63	0.51
3	ns	0.44	0.15	0.20	B	B	1.06	0.46	0.42
4	ns	0.46	0.18	0.65	ns	ns	0.57	0.47	0.21
5	ns	ns	0.15	ns	0.19	ns	0.20, 0.41*	0.24	0.11

Eggs excluding shells

ns no eggs laid during this period

B eggs laid but broken by the bird and included with excreta

* Hen 272 laid two eggs on day 5

Samples of excreta, eggs, liver, breast muscle and skin/fat were further analysed for the composition of the radioactivity. Excreta were mixed with methanol and the methanolic supernatant was collected after centrifugation.

Whole egg homogenates from birds 367 (day 2), 369 (day 4) and 372 (day 3) were mixed with methanol and the protein precipitate removed by centrifugation. The methanol extracts were reduced by evaporation and acidified with 1 M HCl. The extract was fractionated by column chromatography (C-8, octylsilane). The column was washed sequentially with 1 M HCl and hexane followed by elution with ethyl acetate. The ethyl acetate extracts were evaporated to dryness and reconstituted in methanol.

Birds 367 and 372 had the highest tissue concentration of radioactivity and were selected for extraction. Liver samples were extracted with 1M HCl/ethyl acetate (1:10, v/v). After centrifugation, the remaining solids were extracted again with ethyl acetate. Both extracts were combined. Muscle and skin/fat samples were extracted with methanol/water (1:1) with a few drops of 1 M NaOH per 10 mL. After centrifugation, the remaining solids were extracted again with methanol. Both extracts were combined, reduced by evaporation, acidified with 1 M HCl and fractionated by column chromatography as for eggs.

Identification of the radioactivity in all extracts was done using TLC in five different solvent systems. The reference substance used was the parent quinclorac which was confirmed on TLC plates by the quenching of UV fluorescence at 254 nm.

Extraction efficiency varied from bird to bird but were generally around 90% for excreta, liver, breast muscle and skin/fat (Table 9). The unextracted residues in eggs were rather high from 10.5–21.1% of TTR in the egg tissue.

Besides the parent quinclorac (levels > 78% TRR in tested matrices) the only identified metabolite was M1 (glucuronic acid conjugate of the parent). M1 co-chromatographed with the major radioactive component in the bile of rat. In the rat study this metabolite was identified as the glucuronic acid conjugate of parent by enzymatic cleavage, followed by MS analysis. The combined concentration of M1 and fractions R01-1 and R01-3 was a maximum of 3% TRR in the tissues and not detected in eggs. Another fraction R01-5was present at levels from 0.3–3.7% TRR in eggs and tissue. In eggs (excluding shells) 10.5–21.1% of the residue was not extracted.

Since the methods used to extract the residues included 1 M HCl, it is not clear whether parent compound represents parent only or includes parent released from conjugates and whether the M1 is the fraction of conjugates that remained uncleaved.

Table 9 Characterization and identification of compounds in eggs, tissues and excreta of the laying hen after administration of quinoline-2, 3, 4-^{14}C-quinclorac at 33–44 mg/kg bw and day (800 ppm in the diet).

	Eggs [a] 367, 369, 372	Breast muscle 367, 372	Skin/fat 367, 372	Liver 367, 372	Excreta 371, 372	
TRR (mg/kg eq)	1.21; 0.65; 1.06	3.2; 4.22	5.28; 7.20	9.39; 10.53		
	%TRR*	%TRR*	%TRR*	%TRR*	day 1	day 2
Quinclorac	83; 81; 78	87.0; 86.4	86.0; 87.7	91.5; 91.3	88.9-91.2	84.6-85.0
R01-1	ND; ND; 1.0	1.4; 1.2	1.1; 0.5	3.0; 2.7	0.3	0.3
M1 (R02)					1.1-1.7	1.9-4.4
R01-3					0.05-0.07	0.01-0.08
R01-5	2; 0.3; 0.9	1.8; 0.8	1.8; 2.5	3.4; 3.7	0.2-0.4	0.05-0.2
Total identified	≤ 83	≤ 88.8	≤ 90.2	≤ 95.3	≤ 93.67	≤ 96.18
Total characterized	≤ 2	≤ 1.8	≤ 2.5	≤ 3.7	≤ 2.47	≤ 4.98
Total extracted	82-90; 80-84; 79-82	90; 88	89; 91	98; 98	90.8-93.4	87.4-89.9

	Eggs [a] 367, 369, 372	Breast muscle 367, 372	Skin/fat 367, 372	Liver 367, 372	Excreta 371, 372	
TRR (mg/kg eq)	1.21; 0.65; 1.06	3.2; 4.22	5.28; 7.20	9.39; 10.53		
Unextracted	10-18; 16-20; 18-21	9.9; 11.6	11.1; 9.3	2.0; 2.4	6.7-9.2	10.1-12.6

* Relative % of total sample radioactivity

M1 is glucuronic acid conjugate of parent

nd not detected

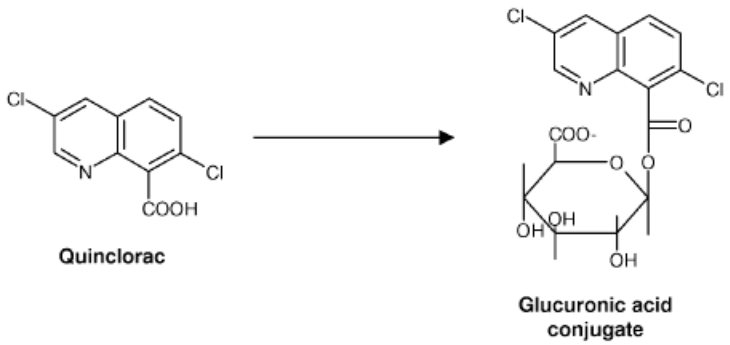


Figure 5 Metabolic pathway from available data in laying hen

Plant metabolism

The Meeting received plant metabolism studies after foliar application of ^{14}C-radiolabelled active substances to rice, wheat, rape seed (canola), sorghum and strawberry. In these studies 2, 3, 4-[^{14}C]-quinclorac (quinoline label) or 3-[^{14}C]-quinclorac (quinoline label) were used.

Rice

In a study reported by Wood (1988, Ref. BASF 88/5059) one foliar application of 2,3,4-^{14}C-quinclorac was made to paddy rice plants. The experiments were performed on plants grown in a growth chamber and in the field. These plants were treated at 1.5 kg ai/ha at the 4 leaf stage (~ BBCH 14) and were grown in pots containing a mixture of loam compost and peat moss. The field plants (variety Starbonnet) were treated at 0.84 kg ai/ha at 3-5 leaf stage (~BBCH 15-16) under unflooded conditions in a sandy loam soil. Seven days later a permanent flood was established for the field plants. Whole plants (forage) were harvested from the field plots 28 days after treatment and mature grain and straw samples were taken from the growth chamber plants (97 days after treatment) and field grown plants (118 days after treatment).

Analysis of the TRR was done using combustion and LSC. An overview of the TRR levels found in collected samples is presented in Tables 10 and 11 below. Only rice grain and straw from the growth chamber and rice forage and grain from the field treatment were analysed further.

Radioactivity in rice straw and forage was easily extracted with organic solvents. Straw samples were homogenized and 92% TRR was extracted with acetone /water (6:4, v/v). The extract was evaporated, acidified with HCl and residues were partitioned overnight between diethyl ether (87% TRR) and water (4.9% TRR). The diethyl ether fraction was analysed by TLC.

Forage samples were homogenized and 92% TRR was extracted with acetone/water (1:1, v/v) and acetone/water (6:4, v/v). The extract was evaporated and acidified with HCl. The residues in the aqueous extract were exhaustively partitioned between dichloromethane (87% TRR), ethyl acetate (2.4% TRR) and water (4.9% TRR). The dichloromethane and ethyl acetate extracts were combined, evaporated to dryness and redissolved in acetone for TLC analysis.

Radioactivity in rice grain was extracted poorly with organic solvents. When the grain was first dissolved in boiling water and then acidified or refluxed with 1 M HCl, the residues could be easily extracted with organic solvents. Grain samples from growth chambers were refluxed with water

for 2 hrs, acidified with HCl and residues were partitioned for 8 hrs into diethyl ether (97% TRR). The diethyl ether extract was partitioned with 1 M NaOH, whereby the oil remained in the diethyl ether and the residues transferred into the aqueous phase. The combined aqueous extracts were acidified with HCl and partitioned with diethyl ether. The diethyl ether extract (94% TRR) was evaporated to near dryness and then diluted with methanol for TLC analysis.

Field grain samples were first extracted with hexane (1.9% TRR) to remove the oils. Remaining solids were air dried and solubilized by reflux with 1 M HCl. Boiling acid was used to avoid frothing and to avoid emulsion problems during extraction. Residues were partitioned between diethyl ether (58% TRR), ethyl acetate (25% TRR) and water (15% TRR). The combined diethyl ether and ethyl acetate extracts were concentrated to near dryness and diluted with methanol for TLC analysis.

Organic fractions were analysed by TLC. An overview of the composition of the residue is presented in Table 11. Nearly all the radioactivity in all parts of the rice plant could be accounted for as unchanged parent up to the final harvest. Metabolites were not further characterized. The identification of the parent was performed by derivatization of quinclorac to its methyl ester and analysis with GC-MS.

Grain samples required boiling water or boiling 1 M HCl to allow extraction of the residues with organic solvents. This suggests that quinclorac is bound to the grain matrix and the quinclorac identified is actually quinclorac released from conjugates. Forage and straw were easily extracted with organic solvents, therefore the quinclorac identified in forage and straw is likely the unchanged parent compound.

Table 10 Radioactive residues in field grown rice treated after foliar application to rice with 2, 3, 4-^{14}C-quinclorac at 0.84 kg ai/ha

Matrix	Days after application	Days before harvest	mg eq/kg
Whole plant	3	115	34.60
Whole plant	14	104	5.42
Whole plant	28	90	0.49
Final harvest straw	118	0	0.10
Final harvest grain	118	0	0.12

Table 11 Extractability/mass balance of radioactivity from treated rice with 2, 3, 4-^{14}C-quinclorac

Sample	Rice straw (growth chamber) 1.5 kg ai/ha DAT 97 12.79 mg eq /kg TRR		Rice forage (field) 0.84 kg ai/ha DAT 28 0.49 mg eq/kg TRR		Rice grain (growth chamber) 1.5 kg ai/ha DAT 97 1.52 mg eq/kg TRR		Rice grain (field) 0.84 kg ai/ha DAT 118 0.12 mg eq/kg TRR	
	% TRR	mg eq/kg TRR	% TRR	mg eq/kg TRR	% TRR	mg eq/kg TRR	% TRR	mg eq/kg TRR
Quinclorac	86 a	10.99	85 a	0.42	94 b	1.43	84 b	0.11
Organosoluble	1.4	0.18	4.2	0.02	0.7	0.01	1.9	0.002
Aqueous soluble	4.9	0.63	3.9	0.02	1.2 3.0	0.02 0.05	14.6	0.02
Post extracted solids	7.6	0.98	8.2	0.04	-	-	-	-
Total	99.9		101.3		98.9		100.5	

* Final fraction containing parent compound

A extracted with acetone/water without boiling or reflux

B extracted with diethyl ether and ethyl acetate after boiling in water or reflux in 1 M HCl

Wheat

In a study reported by Ellenson, J, L (1996a, BASF 1996/5 197) one foliar application of 3-^{14}C quinclorac was applied at 3-5 leaf stage to wheat plants grown in a greenhouse at 1 × 0.125 kg ai/ha

and at 1 × 0.500 kg ai/ha. Plants (variety Katepwa) were grown in pots containing silt loam. Forage was sampled at 37 DAT when plants were in early to late boot stage and mature wheat grain and straw were sampled at 92 DAT.

Analysis of the TRR was carried out using combustion and LSC. The TRRs found in the samples collected amounted to 3.26 and 13.14 mg eq/kg at the two application rates in forage. In the straw 1.86 mg eq/kg was detected at the low application rate and 8.16 mg eq/kg at the high application rate. TRR for the grain were 1.13 mg eq/kg at the low application rate and 3.94 mg eq/kg at the high application rate samples. An overview of the TRR levels found in collected samples is presented in Table 12.

In forage, straw and grain 85–95% TRR was extracted with acetone/water (2:1, v/v). A further 3.3–12% TRR could be released by hydrolysis with 0.1 M NaOH. Identification of residues in the acetone/water fraction in straw was based upon retention time comparison with known standards and/or determination with HPLC-MS. Separation and isolation of specific radioactive residues from the straw samples was accomplished using semi-preparative and analytical HPLC methods coupled with fraction collection. Identification and characterization of residues in forage and grain acetone/water fractions were derived from HPLC retention time comparison with residues isolated from the higher application straw. An aliquot of the acetone/water extract was treated by base hydrolysis (pH 13, 100 °C, 2 hrs) to cleave any conjugates present in the extract and the extract was re-analysed by HPLC-MS.

In forage, parent residues accounted for 24% TRR (0.78 mg eq/kg) in the low application rate and 45% TRR (5.92 mg eq/kg) in the high rate samples. A total of 9.8% TRR (0.32 mg eq /kg) in the low application rate and 6.4% TRR (0.84 mg/kg) in the high application rate forage were associated with hydroxyquinclorac conjugates. Other metabolites identified at levels <5% TRR were quinclorac conjugates and hydroxyquinclorac. Unidentified components were <5% TRR (0.16 mg/kg) for the low application rate or 6.93%TRR (0.91 mg/kg) for the high application rate.

In grain, parent residues accounted for 62% TRR (0.69 mg eq/kg) in the low application rate grain, and 68% TRR (2.68 mg eq/kg) in the high rate samples. A total of 4% TRR (0.14 mg eq/kg) in the high application rate grain was assigned to hydroxyquinclorac conjugates. Unidentified components were none > 2.11% TRR (0.024 mg eq/kg) for the low application rate or 3.47%TRR (0.14 mg eq/kg) for the high application rate.

In straw, parent accounted for 12% TRR (0.22 mg eq/kg) in the low application rate samples and 22% TRR (1.83 mg eq/kg) in the high application rate samples. Additional residues at 13.7% TRR (0.26 mg eq/kg) in the low application rates samples and 12.6% TRR (1.02 mg eq/kg) in the high application rate were assigned to hydroxyquinclorac conjugates. Unidentified compounds were none > 7.07% TRR (0.13 mg eq/kg) for the low application rate samples or 7.71%TRR (0.63 mg eq/kg) for the high application rate.

A more detailed fractionation of the high application rate straw were generally in agreement with the simpler profile determined by HPLC analysis of the soluble residues only. Individual residues identified during analysis of the high application rate straw sample included the parent 23% TRR, a glucose conjugate of hydroxyquinclorac (~6% TRR), and hydroxyquinclorac (~3% TRR). The hydroxyquinclorac was not the 2-OH quinclorac identified in the soil degradation studies. Mass spectral analysis also indicated the presence of small (<1% TRR) amounts of possible malonate esters of parent and/or hydroxyquinclorac. A relatively large portion of TRR (20% or 1.6 mg/kg in the 4× sample) was associated with high molecular weight species that are presumed to be natural products.

Base hydrolysis (pH 13, 100 °C, 2 hrs) of the entire homogenized forage, straw and grain samples did not release any compounds beyond what was already identified.

The results indicate that the residues in wheat primarily consist of unchanged parent compound. Low levels of hydroxyquinclorac are formed by oxidative hydroxylation of the ring structure of the parent compound. Both quinclorac and hydroxyquinclorac can be metabolized to glucose conjugates. A less prevalent metabolic pathway may involve esterification of the parent at the carboxyl group.

Table 12 Extractability from wheat forage, straw and grain treated with 3-^{14}C-quinclorac

Sample	Forage 0.125 kg ai/ha DAT 37 TRR= 3.26 mg/kg eq		Forage 0.50 kg ai/ha DAT 37 TRR = 13.14 mg/kg eq		Straw 0.125 kg ai/ha DAT 92 TRR= 1.86 mg/kg eq		Straw 0.50 kg ai/ha DAT 92 TRR = 8.16 mg/kg eq		Grain 0.125 kg ai/ha DAT 92 TRR = 1.13 mg/kg eq		Grain 0.50 kg ai/ha DAT 92 TRR = 3.94 mg/kg eq	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Acetone/water	87.8	2.86	93.4	12.3	84.6	1.57	87.0	7.09	93.8	1.06	95.3	3.75
- Quinclorac	24.0	0.78	45.1	5.92	11.7	0.22	22.4	1.83	61.6	0.69	68.0	2.68
- Quinclorac glucose conjugates a	4.6	0.15	6.9	0.91	-	-	-	-	-	-	-	-
- Hydroxy quinclorac	3.0	0.098	-	-	-	-	-	-	-	-	-	-
- Hydroxy quinclorac glucose conjugates b	6.8	0.22	6.4	0.84	13.7	0.26	12.6	1.02	4.0	0.05	3.7	0.14
-Unidentified compounds	42.7 c	1.39[c]	38.4 c	5.01 [c]	44.8 d	0.83 d	45.1 d	3.68 d	15.0 e	0.17 e	17.2 e	0.68 e
Unidentified fraction released by 0.1 M NaOH	10.3	0.34	5.6	0.74	12.4	0.23	10.9	0.09	3.34	0.04	3.97	0.16
PES	1.87	0.06	0.43 0.44	0.06 0.06	3.27	0.06	1.02 0.59	0.08 0.05	0.33	0.004	1.10	0.043
Total	100.0		99.9		100.3		99.5		97.5		100.4	

A 419 dalton quinclorac glucose conjugate

B Conjugates which released a hydroxyquinclorac exocon with molecular weight 257, when the acetone/water extract was treated by base hydrolysis (pH 13, 100 °C, 2hrs).

C 23 chromatographic regions, none > 4.96 TRR (0.16 mg/kg) for 1X or 6.93%TRR (0.91 mg/kg) for 4X

d 20 chromatographic regions, none > 7.07 TRR (0.13mg/kg) for 1X or 7.71%TRR (0..63 mg/kg) for 4X

e 19 chromatographic regions, none > 2.11 TRR (0.024mg/kg) for 1X or 3.47%TRR (0.14 mg/kg) for 4X

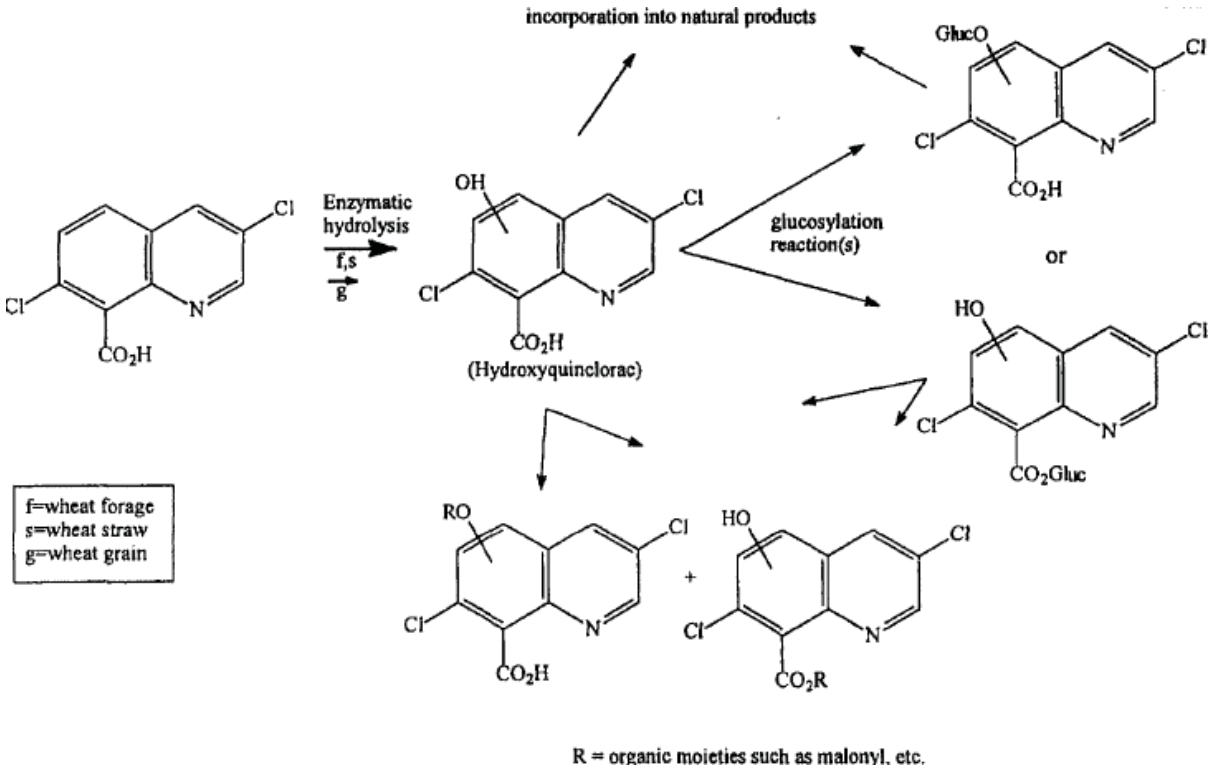


Figure 6 Proposed metabolic pathway of quinclorac in wheat

Sorghum

In a metabolism study reported by Ellenson J, L, (1993, Ref 1993/5088) 3-^{14}C-quinclorac was applied pre-emergent outdoors to the soil followed by a post-emergence treatment when sorghum plants were 15–25 cm tall. The pre-emergence treatment was 0.525 kg ai/ha and the post-emergence was 0.504 kg ai /ha (total 1.03 kg ai/ha) with an interval of 25 days. The plants (variety G820) were grown in a field plot with loamy sand soil in North Carolina, USA.

Analysis of the TRR was carried out using combustion and LSC. Residue analysis was done on forage sampled 25 days after the last treatment and on grain and fodder sampled 95 days after the last treatment. The TRRs for these samples amounted to 4.01 mg eq/kg in forage, 0.87 mg eq/kg in fodder and 0.83 mg eq/kg in grain.

Sorghum forage was extracted with acetone/water and the filtrate was further extracted with ethyl acetate. Sorghum fodder samples were extracted with acetone/water and the filtrate was subsequently extracted with hexane, dichloromethane and ethyl acetate. Sorghum grain samples were extracted with acetone/water and the filtrate was subsequently extracted with hexane, dichloromethane and ethyl acetate and refluxed with HCl.

Remaining solids were subjected to refluxing with NaCl solution (10 g/L) for 2 hr to determine radioactivity incorporated into water soluble polysaccharides, boiling with EDTA (5 g/L) for 1 hr to determine incorporation in peptic polysaccharides, with 1.25 M NaOH (50 g/L) for 6 hr at 80 °C to determine incorporation in hemicellulose I, with sodium chlorite ($NaClO_3$, 10 g/L at room temperature) for 3 hr to determine incorporation in lignin and with 6 M NaOH (240 g/L) for 6 hr to determine incorporation in hemicellulose II.

Extracts were analysed by radio TLC. Quinclorac was confirmed using methylation to its methyl ester and determined by GC-ECD. The presence of quinclorac methyl ester was also separately confirmed by two dimensional TLC.

An overview of the TRR levels found in collected samples and the composition of the residue is presented in Tables 13–15. In all samples unchanged quinclorac was the major residue being present at levels of 73.4% TRR in forage, 21.5% TRR in fodder and 73.5% in grain. This residue included the parent compound that was released from remaining solids (4% in grains to 9% TRR in forage and fodder). The only other metabolite identified was quinclorac methyl present at 3.6% TRR in forage, 5.9% in fodder and 1.7% in grain. In spite of substantial alkaline extraction procedure, a large amount of unidentified residues was present in forage and fodder in organic and aqueous fractions, maximum 18.7% TRR (0.75 mg eq/kg in forage and 52.4% TRR (0.46 mg eq/kg) in fodder. The majority of the released residue from the remaining solids was in the polysaccharide fraction ($NaCl/H_2O$) or the hemicellulose fraction (1 M NaOH).

The results indicate that extraction with acetone/water is very effective for the sorghum forage and grain, but less so for the fodder. An additional 24% TRR could be extracted from the remaining fodder solids after addition of NaOH. The current analytical method incorporates an alkaline extraction step.

Table 13 Fractionation, characterization and identification of radioactive residues in sorghum forage after treatment with 3-^{14}C-quinclorac

Sample	Sorghum forage DALT=25 TRR =4.01 mg/kg eq			quinclorac		quinclorac methyl ester		unidentified			
								organo soluble		aqueous soluble	
Acetone/water extract		%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
	EtOAc, pH 2	71.3	2.86	64.5	2.585	3.6	0.145	3.2	0.130	-	-
86%TRR	aqueous	14.6	0.58					-	-	14.6	0.58
3.44 mg/kg eq	subtotal	85.9	3.44	64.5	2.58	3.6	0.145	3.2	0.13	14.6	0.58
Solids	$NaCl/H_2O$	5.8	0.23								
	EtOAc			4.1	0.163	-	-	0.3	0.012		
14% TRR	Aqueous					-	-			1.1	0.05
0.57 mg/kg eq	EDTA	0.8	0.03								
	EtOAc			0.5	0.020	-	-	1.0	0.040		
	Aqueous					-	-			2.8	0.111
	NaOH	7.6	0.29								
	EtOAc			3.9	0.156	-	-	0.1	0.007		
	Aqueous			-	-	-	-			0.2	0.009
	$NaClO_3$	1.2	0.05								
	EtOAc			0.4	0.014	-	-				
	Aqueous			-	-						
	NaOH	0.3	0.01								
	Residuum	0.1	0.004								
	subtotal	15.8	0.614	8.9	0.353	-			1.4		
	overall total	**102**	**4.10**	**73.4**	**2.93**	**3.6**	**0.145**	**4.7**	**0.188**	**18.7**	**0.750**

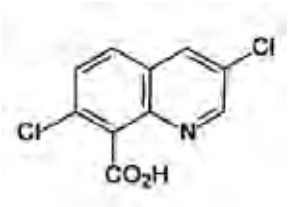
Table 14 Fractionation, characterization and identification of radioactive residues in sorghum fodder after treatment with 3-^{14}C-quinclorac

Sample	Sorghum fodder DALT = 95 0.87 mg/kg eq			quinclorac		quinclorac methyl ester		unidentified			
								organo soluble		aqueous soluble	
Acetone/water extract	Partition	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
	Hexane, pH 8	2.7	0.023	-	-	2.7	0.023	-	-	-	-
52%TRR	DCM, pH 8	3.0	0.026	-	-	3.0	0.026	-	-	-	-
0.45 mg/kg eq	EtOAc, pH 8	1.8	0.016	-	-	-	-	1.8	0.016	-	-
	Hexane, pH 2	0.8	0.007	0.5	0.004	0.2	0.002	0.1	0.001	-	-
	DCM, pH 2	10.7	0.093	7.3	0.063	-	-	3.4	0.030	-	-
	EtOAc, pH 2	9.3	0.081	3.2	0.028	-	-	6.1	0.053	-	-
	Aqueous	23.5	0.204	-	-	-	-	-	-	23.5	0.204
	Subtotal	51.8	0.45	11.0	0.095	5.9	0.051	11.4	0.1	23.5	0.204
Post extracted	$NaCl/H_2O$	18.5	0.160								

Sample	Sorghum fodder DALT = 95 0.87 mg/kg eq			quinclorac		quinclorac methyl ester		unidentified			
								organo soluble		aqueous soluble	
solids	EtOAc			5.2	0.045	-	.	2.2	0.019		
	Aqueous									10.0	0.087
	EDTA	1.6	0.014								
49%TRR	EtOAc			0.4	0.003	-	-	0.4	0.003	-	-
0.42 ppm	Aqueous									0.6	0.005
	NaOH 1	24.3	0.211								
	EtOAc			3.8	0.033	-	-	4.6	0.04	-	-
	Aqueous					-	-	-	-	13.3	0.116
	$NaClO_3$	2.8	0.024								
	EtOAc			0.9	0.008	-	-	0.4	0.004	-	-
	Aqueous					-	-	-	-	4.5	0.039
	NaOH II	1.3	0.011								
	EtOAc			0.2	0.002	-	-	0.4	0.003	-	-
	Aqueous									0.5	0.004
	subtotal	48.5	0.42	10.5	0.09			8.0	0.069	28.9	0.25
	overall total	**100**	**0.08**	**21.5**	**0.186**	**5.9**	**0.051**	**19.4**	**0.169**	**52.4**	**0.455**

Table 15 Fractionation, characterization and identification of radioactive residues in sorghum grain with 3-^{14}C-quinclorac

Sample	Sorghum grain DALT=95 TRR=0.83 mg/kg eq			quinclorac		quinclorac methyl ester		unidentified			
								organo soluble		aqueous soluble	
Acetone/water extract	Partition	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
	Hexane, pH 2	7.7	0.064	5.9	0.049	1.7	0.014	0.1	0.001		-
15% TRR	DCM, pH 2	62.6	0.520	60.7	0.504			1.9	0.016		
0.09 mg/kg eq	EtOAc, pH 2	3.8	0.032	1.0	0.008			2.8	0.023		
	Aqueous	10.6	0.088	2.1	0.017					2.1	0.017
	Subtotal	84.7	0.704	69.7	0.578	1.7	0.014	4.8	0.040	2.1	0.017
Post extracted solids	$NaCl/H_2O$	7.5	0.062								
	EtOAc			3.8	0.032			0.6	0.005		
	Aqueous									2.4	0.020
12% TRR	EDTA	0.6	0.004								
0.1 mg/kg eq	NaOH	4.1	0.034								
	EtOAc							1.2	0.010		
	Aqueous									0.4	0.003
	subtotal	12.2	0.1	3.8	0.032			1.8	0.015	3.4	0.027
	overall total	**96.9**	**0.804**	**73.5**	**0.610**	**1.7**	**0.014**	**6.6**	**0.055**	**5.5**	**0.044**



Quinclorac BAS H (parent compound)

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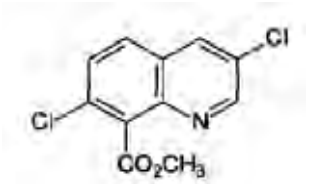


Quinclorac methyl ester BH 514 ME

Figure 7 Proposed metabolic pathway of quinclorac in sorghum

Rape seed (canola)

In a study reported by Parker 1998a (BASF 1998/5180) one foliar application of 3-^{14}C quinclorac was made post emergence (30 days after sowing at 5th true leaf stage) at 0.2 kg ai/ha. The experiment was performed in a growth chamber on a *brassica rapa v*ariety "Horizon" grown on a sandy loam. By simulation of a North American climate during summer it was possible to cultivate oil seed within 90 days. Whole plants were harvested 1 and 29 days after treatment. Seed and straw were harvested 60 days after treatment.

Analysis of the TRR was carried out using combustion and SC. An overview of the TRR levels found in collected samples is presented in Table 16. Forage was not further characterized.

Homogenized samples of oilseed rape seeds or straw were subsequently extracted with acetone/phosphate buffer pH 7 (50:50, v/v). Rape seeds were further extracted with 0.1 M NaOH at room temperature (mild alkaline hydrolysis) and 0.1 M NaOH at 100 °C (harsh alkaline hydrolysis). Organic solvents extracted 84–87% TRR from seed and straw, while an additional 6.9% and 5.2% TRR could be extracted from the seeds by mild and harsh alkaline hydrolysis (Table 17). After centrifugation, each extract was partitioned between an aqueous and organo-soluble fraction (Table 18). Extracts and solids were analysed by (combustion) LSC.

Table 16 Total radioactive residues of quinclorac in rape seed grown in a growth chamber after foliar treatment with 3-^{14}C-quinclorac at 0.2 kg ai /ha.

Matrix	Combustion TRR mg eq/kg	Extraction TRR mg eq/kg
Plants Pre-treatment	0.001	-
Plants, 1 DAT*	9.952	-
Forage, 29 DAT	0.676	-
Straw, 60 DAT	0.645	0.637
Seed, 60 DAT	0.469	0.475

* DAT - Days after treatment

Table 17 Extractability of radioactive residues of quinclorac in rape seed after foliar treatment with 3-^{14}C-quinclorac at 0.2 kg ai /ha

Matrix	TRR [a]		Solvent extraction [b]		Mild hydrolysis [c]		Harsh hydrolysis [d]		Total extracted [e]		PES [f]	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Seed	0.475	100	0.402	84.5	0.033	6.9	0.025	5.2	0.459	96.6	0.013	2.7
Straw	0.637	100	0.560	87.8	-		-		-		0.078	12.2

[a] Combustion TRR

[b] Solvent extraction by 50:50% acetone:pH 7 phosphate buffer

[c] Mild hydrolysis by 0.1 M NaOH at room temperature

[d] Harsh hydrolysis by 0.1 M NaOH at 100°C temperature

[e] Total extracted represents the total extracted residues; i.e. the sum of solvents 1-3

[f] PES represents the residues in the post extracted solids

Table 18 Extractability of radioactive residues of quinclorac in rape seed and straw after foliar treatment with 3-^{14}C-quinclorac at 0.2 kg ai/ha

Commodity	Description	Organo-soluble		Aqueous soluble	
		mg eq/kg	% TRR	mg eq/kg	% TRR
Oilseed rape seed	50:50 acetone:buffer	0.359	75.6 [a]	0.037	7.7
	Mild base hydrolysis	0.030	6.2 [b]	0.005	1.1
	Hard base hydrolysis	0.023	4.8 [c]	0.002	0.4
	Total extracted	0.412	86.6	0.044	9.2
Oilseed rape straw	50:50 acetone:buffer	0.338	53.0 [c]	0.193	30.2

[a] Partitioned into hexane at pH 7 (37.1% TRR quinclorac methyl) and into dichloromethane at pH 2 (38.5%, contains 37.1% TRR quinclorac parent)

[b] Partitioned into ethyl acetate at pH 2

[c] Partitioned into dichloromethane at pH 2

Identification and characterization of residues in rape seed was based upon retention time comparison with standards for parent and quinclorac methyl and by confirmation using HPLC-MS/MS. An overview of the composition of the residues in collected samples is presented in Table 19.

In rape seed a total of 37.1% TRR (0.176 mg eq/kg) was identified as the parent quinclorac and 37.1% TRR (0.176 mg eq/kg) was identified as a methyl ester. Quinclorac methyl, in the acetone/phosphate buffer extract partitioned into hexane at pH 7, while the quinclorac parent partitioned into dichloromethane at pH 2. The remainder of the extracts (17.4% TRR) represents a multitude of minor discrete residues and was characterized as organo soluble or aqueous-soluble. The post extraction solids accounted for 2.7% TRR (0.013 mg eq/kg).

For straw, the organo soluble fractions consisted of two major fractions containing quinclorac and quinclorac methyl ester, and at least two minor fractions. A minor peak at approximately 25 min. corresponded to a minor peak seen in the seed samples. No quantitative data are indicated in the report. The post extraction solids from straw containing 12.2% TRR (0.078 mg eq/kg) were not further analysed as it was concluded that rape seed straw not is a feed item.

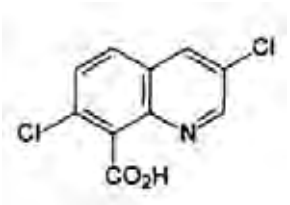
Table 19 Degree of identification/characterization of radioactive residues of quinclorac in rape seed after foliar treatment with 3-^{14}C-quinclorac at 0.2 kg ai /ha.

Degree of identification	Designation	Oilseed rape seeds mg eq/kg	Oilseed rape seeds % TRR
TRR	Total [c]	0.475	
Identified	Quinclorac (parent)	0.176	37.1
	Quinclorac methyl ester	0.176	37.1
	Subtotal	0.352	74.2
Characterized	Aqueous soluble residues	0.042 [a]	8.7 [a]
	Organo soluble residues	0.041 [a]	8.6 [a]
	Subtotal	0.083	17.4
Post-extraction solids	PES	0.013 [b]	2.7 [b]
	Subtotal	0.013	2.7
Total	Total	0.448	94.2

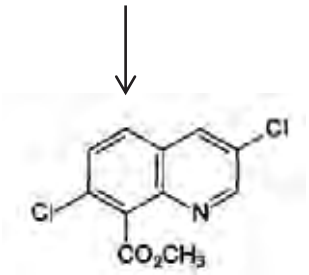
[a] Fractionated after solvent extraction and hydrolysis by 0.1 M NaOH at room temperature and at 100 C.

[b] Remains after hydrolysis

[c] TRR based on sum of extracts



Quinclorac BAS 514 H (parent compound)



Quinclorac methyl ester BH 514 ME

Figure 8 Proposed metabolic pathway of quinclorac in rape seed (canola) following foliar post-emergence application of 3-^{14}C-quinclorac

Strawberry

In a study reported by Walsh, K (2015, BASF/029602-1, report 029602-1) mature strawberry plants were individually treated with one foliar spray application of ^{14}C-quinclorac at 1.120 kg ai/ha at growth stage BBCH 73 61 days prior to the third sampling. The fruits were harvested 21, 37 and 61 days after treatment (DAT). All mature fruit was collected and approximately 2-3 leaves from each plant. At the third and final harvest, all the remaining leaves and immature green strawberry fruits were collected.

Foliage was surfaced washed with ethanol prior to homogenization. Homogenized samples of both fruits and foliage were extracted with ethanol/water. The post extracted solids (PES) were combusted and further extracted using a Soxhlet apparatus. Acid hydrolysis of pooled extracts was done with 12 N hydrochloric acid and 2 hours incubation at 37 °C. Extracts and PES were analysed by LSC, and concentrated extracts were analysed on HPLC to determine the TRR and the nature of major terminal residues. The identity was confirmed by LC-MS/MS. An overview of the residues and fractions found in collected samples is presented in tables below.

In foliage, parent quinclorac accounted for 67.4% TRR (10.43 mg eq/kg) at first harvest 21 DAT and at 57.4% TRR (4.36 mg eq/kg) at the last harvest 61 DAT. Conjugated quinclorac (M1) released by acid hydrolysis was identified from 26.8%TRR (4.19 mg eq/kg) at first harvest and at 28.6%TRR (2.27 mg eq/kg) in the last harvest. As the parent is an acid it is possibly a glucose ester conjugate. A minor polar metabolite was present from 1.8% (0.27 mg eq/kg) at first harvest 21DAT to 5.8% TRR (0.47 mg eq/kg) at last harvest 61 DAT. Post extracted (non-characterized) solids went from 4.5% TRR (0.7 mg eq/kg) at the first harvest to 8.1% TRR (0.67 mg eq/kg) at the last harvest. Quinclorac methyl ester was not detected in the foliage.

In fruit, parent quinclorac accounted for 78.8% TRR (9.13 mg eq/kg) at first harvest and at 50.8% TRR (1.69 mg eq/kg) at third harvest. Conjugated quinclorac (M1) released by acid hydrolysis went from 10.7%TRR (1.26 mg eq/kg) at first harvest to 47.3%TRR (1.57 mg eq/kg) in the last harvest. Quinclorac methyl ester accounted for 9.6% TRR (1.13 mg eq/kg) at first harvest, to 4.9% TRR (0.42 mg eq/kg) at second harvest and was not detected at the last harvest. Level of post extracted (non-characterized) solids was below 10% TRR throughout the study.

Table 20 Characterization of total radioactive residues detected in strawberry fruit after foliar application of ^{14}C-quinclorac

	Quinclorac methyl ester		Quinclorac		Quinclorac conjugates (M1)		PES		Total	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Harvest 1 (21 DAT)										
Solvent extract	1.09	9.3	8.97	76.4	1.22	10.4	-	-	11.29	96.2
Soxhlet extract	0.04	0.3	0.16	1.4	0.04	0.3	-	-	0.24	2.0
PES							*0.22*	*1.8*	*0.22*	*1.8*
Total	1.13	9.6	9.13	77.8	1.26	10.7	0.22	1.8	11.75	100
Harvest 2 (37 DAT)										
Solvent extract	0.39	4.5	6.78	77	1.27	14.5	-	-	8.44	96
Soxhlet extract	0.03	0.4	0.15	1.8	0.03	0.3	-	-	0.21	2.5
PES							*0.14*	*1.6*	*0.14*	*1.6*
Total	0.42	4.9	6.93	78.8	1.3	14.53	0.14	1.6	8.79	100
Harvest 3 (61 DAT)										
Solvent extract	nd	nd	1.53	46	1.57	47.3	-	-	3.10	93.3
Soxhlet extract	nd	nd	0.16	4.8	-	-	-	-	0.16	4.8
PES	-	-	-	-	-	-	*0.06*	*1.9*	*0.06*	*1.9*
Total	nd	nd	1.69	46	1.57	47.3	0.06	1.9	3.32	100

*approximate times, nd not detected

** M1 the portion of the radioactive residue in the conjugated form

PES = post extracted solids

DAT = days after treatment

*** Quinclorac total = quinclorac parent +quinclorac methyl ester

Table 21 Characterization of total radioactive residues detected in strawberry foliage after foliar application of ^{14}C-quinclorac

	4.5 minutes* peak retention minutes		Quinclorac		Quinclorac conjugates (M1)		Post extracted solids (PES)		Total	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Harvest 1 (21 DAT)										
Surface wash	nd	nd	5.33	34.7	0.24	1.5	-	-	5.57	35.7
Solvent extract	nd	nd	4.02	25.8	3.79	24.3	-	-	7.81	50.1
Soxhlet extract	0.27	1.8	1.08	6.9	0.16	1.0	-	-	1.51	9.7
PES							0.7	4.5	0.7	4.5
Total	0.27	1.8	10.43	67.4	4.19	26.8	0.7	4.5	15.59	100
Harvest 2 (37 DAT)										
Surface wash	nd	nd	3.01	17.1	0.15	0.9	-	-	3.16	18.0
Solvent extract	nd	nd	7.67	45.6	1.79	10.7	-	-	9.46	56.3
Soxhlet extract	0.548	3.3	1.38	8.2	0.34	2.0	-	-	2.268	13.5
PES							1.89	11.3	1.89	11.3
Total	0.548	3.3	12.06	70.9	2.28	13.6	1.89	11.3	16.78	99.1
Harvest 3 (61 DAT)										
Surface wash	nd	nd	0.34	8.3	0.08	1.8	-	-	0.42	10.1
Solvent extract	nd	nd	2.86	34.9	2.19	26.8	-	-	5.05	61.7

	4.5 minutes* peak retention minutes		Quinclorac		Quinclorac conjugates (M1)		Post extracted solids (PES)		Total	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Soxhlet extract	0.47	5.8	1.16	14.2	-	-	-	-	1.63	20.0
PES							0.67	8.1	0.67	8.1
Total	0.47	5.8	4.36	57.4	2.27	28.6	0.67	8.1	7.77	99.9

*approximate times, nd not detected

** M1 the portion of the radioactive residue in the conjugated form.

PES = post extracted solids

DAT = days after treatment

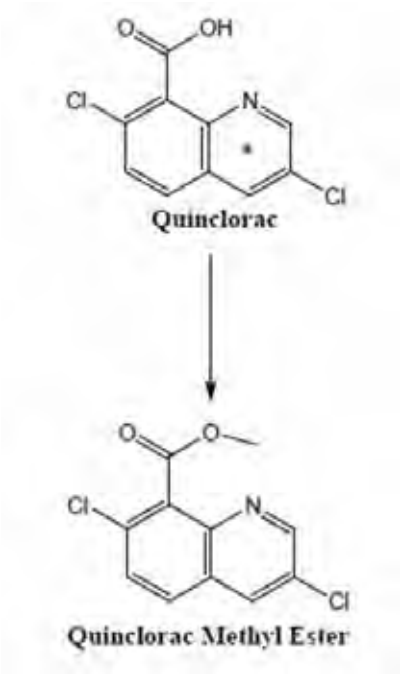


Figure 9 Proposed metabolic pathway of quinclorac in strawberry following foliar post-emergence application of ^{14}C-quinclorac

Environmental fate in soil

For the investigation of the environmental fate of quinclorac the Meeting received one study on hydrolysis, one on photolysis, two on terrestrial and aquatic soil metabolism and one on field dissipation. In the studies, 2, 3, 4-[^{14}C]-quinclorac (quinoline label) or 3-[^{14}C]-quinclorac (quinoline label) were used.

The characteristics of the soils used in the experiments are presented in the table below.

Table 22 Characteristics of soils used for laboratory and field dissipation studies

Study	Clark J.R BASF1987/5040; Clark J.R. BASF 1988/5046 Wood & Winkler BASF 1991/5005	Clark, J R BASF1987/5040 Clark J.R BASF 1988/5046			Goetz A, BASF /1993/5074		Jackson, S et al . BASF 1996/5205
Remark	non-GLP	non-GLP	non-GLP	non-GLP	GLP, Lab	GLP, Lab	GLP, field
Location	Savoy, IL, USA	Greenville,	Davis, CA,	Greenville,	Leland, MS,	Holly	Kingman

Study	Clark J.R BASF1987/5040; Clark J.R. BASF 1988/5046 Wood & Winkler BASF 1991/5005	Clark, J R BASF1987/5040 Clark J.R BASF 1988/5046			Goetz A, BASF /1993/5074		Jackson, S et al . BASF 1996/5205
		MS, USA	USA	MS, USA	USA	Springs, NC, USA	County, KA, USA
Soil name	Soil	Soil	Aquatic/ sediment	Aquatic/ sediment	-	-	-
% sand	14.8	20.8	11.2	9.2	28.5	81.0	84
% silt	65.2	65.2	48.8	48.8	30.0	13.0	10
% clay	20.2	14.0	40.0	42.0	41.5	6.0	6
Texture Class (USDA)	Silt loam*	Silt loam*	Silty Clay Loam	Silty Clay	Clay	Loamy sand	Loamy sand
organic matter %OM	2.5	0.6	2.1	2.5	1.6	1.2	0.7
pH in water	6.4	6.2	7.1	7.1	6.9	6.8	6.7
Cation Exchange Capacity (CEC)	24.8	10.6	33.5	34.2	21.4	3.3	7.3
Water holding capacity at 1/3 bar	29.6	21.3	34.5	21.9	33.0	6.2	7.3

*Soil classification system not stated

Photolysis on soil

The photodegradation of quinclorac was investigated by Wood, N and Winkler, V (1991, BASF 1991/5005) with 3-[^{14}C]-quinclorac in soil (15% sand, 65% silt and 20% clay). Irradiated soil layers were exposed to light from a xenon lamp, filtered to remove light with wavelengths < 290 nm, in a 12 hour light/ 12 hour dark cycle. Test systems were maintained at 26ºC during irradiated periods and 18 ºC during dark periods. The test system was continually aerated with a stream of moistened, CO_2 free, air, and the outgoing air connected to traps for the collection of $^{14}CO_2$. Total recoveries were generally in the range of 93–106% of the % AR.

Under the conditions of the test quinclorac degraded slowly in irradiated samples, with a half-life (DT_{50}) of 162 days; polar metabolites were identified but each to an extent less than 10% AR. In dark control samples the degradation of quinclorac was very slow, with DT_{50} of 529 days.

Soil metabolism

The aerobic soil metabolism was investigated in aquatic and terrestrial soil systems using quinoline [2, 3, 4-^{14}C]- or [3-^{14}C] labelled quinclorac.

In the terrestrial and aquatic soil metabolism study conducted by Clark, J (1987, BASF 1987/5040) [2,3,4-^{14}C]- quinclorac was applied to two silt loam soils from Savoy, IL (USA) and Greenville, MS (USA) and two aquatic water and soil systems from rice fields near Davis, CA, USA and Greenville, MS, USA, see Table 22. The studies were performed prior to GLP being required, but were performed in accordance with US EPA guideline 162-1. Applications were made at a concentration of 0.5 mg/kg, and 5.0 mg/kg corresponding to field application rates of 0.375 kg ai/ha and 3.750 kg ai/ha respectively.

The terrestrial soil test systems were maintained in the dark at 23 ºC, and the moisture adjusted at the start of the study to 40% of the maximum water holding capacity (mWHC). Reaction

flasks with gas outlets to allow CO_2 free air to pass into the bottom and out at the top of the cylinders were used in the experiment. Soil, 500 g for the 0.5 ppm rate and 200 g for the 5 ppm rate. was placed in the flasks and an acetone solution (0.4 mg/g of soil) of quinclorac was added.

The aquatic soil test systems were conducted in a growth chamber in the dark (temperature not given). 250 mL flasks contained approximately 100 g of sediment and 100 mL of water collected from the rice field simultaneously with the sediment. The flasks were connected via tubing to allow the flasks to receive a stream of CO_2 free air and an exit containing scintillation cocktail to capture $^{14}CO_2$. In both the aerobic and the anaerobic experiments, the application of 0.5 mg/kg and 5 mg/kg of quinclorac was added to the water and the water added to each flask containing the sediment.

The individual test systems were continually aerated with a stream of moistened, CO_2 free air, and the outgoing air connected to traps for the collection of $^{14}CO_2$. Single samples for the 0.5 mg/ kg studies were taken after application and at seven further sample times including at the study termination 12 months after application. For the 5 mg/kg studies samples were collected at 5 sample times from 1 month to 12 months after application. In the aerobic aquatic soil studies water was decanted and analysed by LSC. Soil samples were frozen at -20 ºC following collection until they were analysed.

Soil samples were extracted at room temperature with water and 0.1 N NaOH. The aqueous extracts were partitioned with ethyl acetate. The extracted soil was then refluxed with 0.1 N NaOH, neutralized with HCl, and partitioned with ethyl acetate.

All extracts were radio-assayed, with residual soil combusted and analysed by LSC to determine radioactive mass balances. TLC was used for identification and quantification, with confirmation by HPLC using eight synthetized standards.

An overview of radioactive residues extracted from terrestrial and aquatic soil systems are presented in tables below.

Table 23 Recovery of radioactivity (% AR) during aerobic terrestrial degradation of 0.5 mg/kg from 2, 3, 4-^{14}C- quinclorac

Soil	Days after treatment	H_2O wash	0.1N NaOH extract	0.1 N NaOH reflux	Unextracted residues	$^{14}C0_2$ trapped	Total recovery
Savoy, IL, USA Silt loam	14	55.7	39.7	12.1	1.5	-	109.0
	180	49.6	26.0	20.2	3.9	-	95.8
	360	47.1	21.3	15.7	11.3	0.05	95.45
Greenville, MS,USA Silt loam	14	74.8	16.4	9.7	0.7	-	101.8
	180	62	20.4	18.8	2.4	-	113.6
	360	58.8	17.2	22.1	2.5	0.08	100.68

Table 24 Recovery of radioactivity (% AR) during aerobic aquatic degradation of 0.5 and 5 mg/kg from 2, 3, 4-^{14}C- quinclorac

Soil	Days after treatment	H_2O wash	0.1N NaOH extract	0.1 N NaOH reflux	Unextracted residues	$^{14}C0_2$ trapped	Total recovery
Davis, CA USA Silty clay loam 0.5 mg/kg	30	88.4	3.3	5.4	5.1	0.03	102.2
	180	74.4	7.7	8.0	3.6	3.9	97.5
	360	56.7	10.7	12.3	4.8	5.4	89.9
Greenville, MS, USA Silt loam 0.5 mg/kg	30	84.0	11.0	5.6	1.4	0.02	102.0
	180	74.5	13.5	6.8	3.3	6.9	105.0
	360	41.6	15.0	17.9	14.9	8.8	98.2
Greenville,	120	58.0	20.0	9.5	3.5	-	91.0

Soil	Days after treatment	H_2O wash	0.1N NaOH extract	0.1 N NaOH reflux	Unextracted residues	$^{14}C0_2$ trapped	Total recovery
MS, USA Silt loam 5.0 mg/kg	180	30.0	27.0	20.0	12.0	-	89.0
	360	20.2	24.4	23.1	21.5	-	89.2

Table 25 Recovery of radioactivity (% AR) 180 days after treatment in analysis of fractions during aerobic and anaerobic aquatic degradation in a silt loam* of 0.5 mg/kg from 2, 3, 4-^{14}C- quinclorac

Conditions	Supernatant + wash	0.1 N NaOH extract	0.1 N NaOH reflux	Unextracted residues	$^{14}C0_2$ trapped	Total recovery
Anaerobic	85.7	2.5	2.1	2.0	0.04	92.3
Aerobic	74.4	7.6	8.0	3.6	5.1	98.7

* Silt loam soil from Greenville, MS, USA

Table 26 Characterization of total extracted radioactive residues (% AR) in the aerobic aquatic soil degradation in a silt loam after application of 5 mg/kg from 2, 3, 4-^{14}C-quinoline labelled quinclorac

Soil	Days after treatment	Quinclorac	BH 514-1	Unk-2	Unk-3	Unk-4
Greenville, MS, USA Silt loam 5.0 mg/kg	120	55.8	31.7	0.0	0.0	0.0
	180	5.7	55.7	5.0	3.4	0.9
	360	7.6	30.8	5.0	7.6	6.9

*3-chloro-8-quinolilne carboxylic acid

Quantified characterization data of extracted residues is not presented in the reports. It is stated with regard to the terrestrial soil samples that the TLC profiles on total extracted residues in both soils indicated no degradation of quinclorac 120 days after treatment. After one year incubation, traces of metabolite BH 514-1 (3-chloro-8-quinolilne carboxylic acid) was reported to have been observed in the soil.

In the aquatic anaerobic and aerobic systems (Table 25) extraction data 180 days after the application of quinclorac show small differences in the distribution of residues in the different fractions.

In the aerobic aquatic system, with an application rate of 0.5 mg/kg, it is stated in the report that samples displayed mainly unchanged quinclorac after 1 year. In the same system (Table 26) at an application rate (AR) of 5 mg/kg (corresponding to 3.75 kg ai/ha) to sediment and water of a silty loam (rice field) quinclorac degraded slowly to the metabolite BH 514-1 (3-chloro-8-quinolilne carboxylic acid) at a maximum concentration of 55.7% AR 180 days after treatment. Three additional metabolites were detected each present at less than 10% AR and not further characterized. The half-life (DT_{50}) of quinclorac in this system was 4.7 months and for the metabolite BH 514-1 7.4 months. Under anaerobic conditions at the 5 mg/kg level, the same metabolites were formed but at a slower rate, there was a 50% conversion of quinclorac to BH 514-1 within one year.

In an aerobic soil metabolism study conducted by Goetz AJ (1993, BASF 1993/5074) [3-^{14}C]-quinclorac was applied to a clay soil from Holly Springs, NC, USA, and to a loamy sand soil from Leland, MS, USA (Table 22 beginning of chapter). Applications were made at concentrations of 5.3 mg/kg dry soil to the clay soil, and 5.5 mg/kg dry soil to the loamy sand soil, corresponding to field application rates of 3.975 kg ai/ha and 4.125 kg ai/ha respectively. Soil samples from loamy sand and a clay soil were extracted by shaking and/ or refluxing borate buffer and additionally by refluxing in sodium hydroxide if required.

Radioactivity in the extracts was quantified by LSC, with residual soil combusted and analysed by LSC. HPLC was used for characterization and quantification of extracted radioactivity. Analytical standards were used for quinclorac, BH 514-1 (3-chloro-8-quinolilne carboxylic acid), BH 514-2-OH (2-hydroxyquinclorac) and BH 514-Me (quinclorac methyl ester).

The extractability and characterization of the extracted residues in the organic extracts is presented in tables below. Additional residues present in the sodium hydroxide fractions are presented in brackets.

Table 27 Extractability and distribution of radioactive residues after incubation of 5.5 mg/kg (corresponding to 3.98 kg ai/ha) of [3-^{14}C quinclorac dry loamy sand soil (81.0% sand, 13.0% silt, 6.0% clay)

Fraction	Residues (% of applied radioactivity) at days after treatment										
	0	3	7	14	30	61	90	149	210	273	364
Borat buffer	90.9	84.2	83.0	92.8	89.9	76.6	84.6	73.9	77.9	72.4	81.1
Aqueous	0.4	-	-	0.8	1.9	1.0	2.9	2.5	4.6	4.6	5.3
1 N NaOH	-	-	-	-	4.1	-	6.3	-	9.1	13.5	10.7
Humic	-	-	-	-	0.3		0.5		0.8	1.0	1.1
Unextracted residue	1.0	5.6	10.4	1.8	0.4	9.0	0.7	10.8	0.8	1.2	1.2
Volatile organic radioactivity	-	0.1	0.2	0.4	0.5	2.0	3.2	4.2	5.2	5.6	7.5
Total *recovery	97.1	89.9	93.6	101.7	95.7	85.7	90.5	88.9	89.9	89.3	99.9
Identification and characterization											
Quinclorac**	95.7	-	-	94.8	74.5 (3.3)	64.2	60.2 (4.9)	52.9	53.5 (6.3)	45.7 (10.9)	51.5 (6.6)
Met-1	0.0	-	-	1.3	3.9 (0.4)	3.0	4.8 (0.7)	5.0	2.5 (1.2)	4.1 (1.5)	9.1 (0.8)
Met-2	0.2	-	-	0.2	2.2	0.0	2.1	1.7	1.6	1.9	0.0 (1.2)
BH 514-2-OH	0.0	-	-	0.8	3.6	2.6	5.3	5.5	6.7	7.6 (0.4)	8.1 (0.3)
BH 514-Me	0.0	-	-	1.6	4.7	3.9	5.2	5.6	7.1	4.8	7.8
Other	0.0	-	-	1.2	0.0	0.0	0.0	0.0	0.0	0.0	0.3

***** The radioactivity found in the different fraction reported does not always add up to exact TTR

** Additional residues from the extraction with methanol/ammonium hydroxide following reflux in sodium hydroxide are presented in brackets.

Table 28 Extractability and distribution of radioactive residues after incubation of 5.3 mg/kg (corresponding to 4.1 kg ai/ha) [3-^{14}C] quinclorac to dry clay soil (28.5% sand, 30.0% silt, 41.5% clay)

Fraction	Residues (% of applied radioactivity) at days after treatment										
	0	3	7	14	30	61	91	150	210	274	364
Borat buffer	96.7	84.2	80.6	90.2	86.2	61.2	66.0	55.4	61.2	55.6	54.5
Aqueous	0.2	-	0.5	2.5	4.4	9.1	6.6	10.8	13.6	5.8	11.6
1 N NaOH	-	-	-	6.2	10.6	18.0	20.2	22.1	23.1	28.6	26.1
humic	-			1.5	1.6	2.2	2.9	2.3	3.0	6.0	5.5
Unextracted residue	4.4	14.1	16.6	0.9	1.8	3.0	3.7	4.0	4.5	4.7	3.9
Volatile organic radioactivity	-	0.1	0.1	0.2	0.3	1.4	2.6	3.0	4.4	5.4	6.1
Total *recovery	96.8	98.4	93.1	102.6	94.4	82.8	94.2	90.6	94.2	91.7	89.4
Identification and characterization											
Quinclorac**	92.2	-	-	88.9 (4.3)	73.8 (4.7)	43.9 (12.8)	44.1 17.1)	33.7 (13.1)	32.6 (14.2)	25.5 (15.8)	19.2 (11.8)
Met-1	0.0	-	-	1.2	0.0	1.4	1.2	4.4	1.5	3.3	4.2

Fraction	Residues (% of applied radioactivity) at days after treatment										
	0	3	7	14	30	61	91	150	210	274	364
				(0.3)	(0.4)	(1.6)	(2.4)	(3.1)	(3.2)	(2.8)	(3.9)
Met-2	0.0	-	-	0.8 (0.1)	0.0 (0.2)	0.6	0.8 (0.2)	1.4 (0.2)	1.8 (0.9)	2.4 (0.8)	2.2 (0.6)
BH 514-2-OH	0.0	-	-	0.7	3.8 (0.2)	3.4 (0.8)	7.2 (1.2)	8.2 (1.0)	10.0 (0.3)	12.4 (2.7)	12.4 (2.5)
BH 514-Me	0.0	-	-	0.5	2.3	1.7	3.1	4.0	2.3	2.5	3.0
Other	0.0	-	-	0.3 (0.1)	0.0 (0.2)	0.0	0.0	0.0	0.0	0.0	0.0

***** The radioactivity found in the different fraction reported does not always add up to exact TTR

** Additional residues from the extraction with methanol/ammonium hydroxide following reflux in sodium hydroxide are presented in brackets.

The radioactive residues extracted from soil by refluxing with sodium hydroxide solution are considered bound. The residues associated with the humic material increased with time and at 364 days after treatment accounted for 1.1 and 5.5% TRR in the loamy sand and clay soil. The majority of the residues extracted from the humic material by sodium hydroxide were quinclorac.

The residues extracted from the borate buffer solution are considered as available residues**.** Under the condition of the study quinclorac degraded with half-lives (DT_{50}) of 391 days in a loamy sand and 168 days in a clay, forming the metabolites BH 514-2 –OH (2-hydroxyquinclorac) at a maximum concentration of 12% AR and the metabolite BH 514-Me (quinclorac methyl ester) at a maximum of 7.8% AR. Other metabolites were identified at levels well below 10% AR throughout the study and not further characterized.

Field dissipation study

The dissipation of quinclorac in a loamy sand soil was investigated at one site in Kansas USA by Jackson, S *et al* (1997, BASF 1996/5205). Quinclorac was applied with two applications of 2.8 kg ai/ha to bare soil. One application of was made in autumn (October 1994) and the other at summer (June 1995).

Soil samples were taken at day 0 to approximately 540 days after the first application at a maximum depth of 1.22 m. Samples were separated into 15 cm segments (table below) and three samples were analysed per segment. The samples were extracted with 0.1 N NaOH followed by acidification and partitioning with methylene chloride/ ethyl acetate. An aliquot of the organic extract was dried and reconstituted in HPLC mobile phase and analysed by LC/MS/MS. The LOQ for the summed residue was 0.01 mg/kg. Quinclorac methyl ester was extracted from soil with methylene chloride, ethyl acetate and methanol. The extract was dried, re-constituted in HPLC mobile phase, and analysed by LC/MS/MS.

Soil characteristics and residue concentrations of quinclorac and the metabolites BH 514-2-OH and BH 514-Me are presented in tables below.

Table 29: Soil characteristics for soil used for a field dissipation study for quinclorac

Soil characteristic	Soil depth (cm)						
	0-15	15-30	30-45	45-61	61-76	76-91	91-107
% sand	84	84	88	94	94	94	90
90% silt	10	08	04	0	02	02	02
% clay	06	08	08	06	04	04	08
% organic matter	0.7	0.9	0.4	0.5	0.4	0.4	0.2
pH	6.4	6.3	6.6	6.9	7.1	7.2	6.8
% moister 1/3 bar	7.3	8.6	6.9	4.3	4.0	3.5	6.1
CEC (m eq/kg soil)	7.3	9.7	8.6	5.1	5.5	3.6	6.7
texture	loamy soil	loamy soil	loamy soil	sand	sand	sand	sand

Residue values were determined at each sampling interval in (15 cm) segments to a depth of up to 121 cm. Based on the distribution and magnitude of concentrations, ca. 89% of the quinclorac was observed in the topsoil (0–30cm) with 74% in the 0–15 cm segment and 15% in the 15–30 inch segment. At the exaggerated use rate of this study (2 × 2.8 kg ai/ha) some movement to lower depth segments was observed with 7% in the 30-46 cm segment, 2% in the 46-61 cm segment, 1% in the 61-76 cm segment, and < 1% in all lower depths. Metabolite BH 514-2-OH was observed primarily in the topsoil (91%) with 32% in the 0–15 cm segment and 59% in the 15-30 cm segment. The remainder (9%) was observed in the 30–46 cm segment. Metabolite BH 514-Me was only observed in topsoil with ca. 84% in the 0–15 cm segment and 16% in the 15–30 cm segment.

Table 30 below shows total residues from quinclorac during the 540 day study detected from samples taken to a maximum depth of 1.22 m.

Table 30 Total residue concentration of quinclorac, metabolite BH 514-2-OH and BH 514-Me following application of 5.6 kg ai/ha quinclorac to bare soil in Kansas USA

Time	Total residue of all depths (mg/kg)		
Days after treatment (DAT)	quinclorac	BH 514-2 -OH	BH 514-Me
0 Treatment 1	0.836	0	0
1	0.918	0	0
3	0.801	0	0
5	0.816	0	0
7	0.774	0	0
14	0.757	0	0
21	0.617	0	0
30	0.582	0	0
60	0.499	0	0.003
90	0.539	0.017	0.007
120	0.414	0	0
180	0.421	0.020	0.010
240 Treatment 2	0.799	0	0
241 (1 DAT 2)	0.840	0	0
243 (3 DAT 2)	0.769	0	0.007
245 (5 DAT 2)	0.591		0
247 (7 DAT 2)	0.583	0.003	0
254 (14 DAT 2)	0.545	0.003	0
261 (21 DAT 2)	0.330	0.025	0.007
300 (60 DAT 2)	0.004	0.033	0
330 (90 DAT 2)	0	0.013	0
420 (180 DAT 2)	0	0.014	0
540 (300 DAT 2)	0	0.019	0.009

The maximum observed concentrations of the two metabolites were 3.6% for BH 514-2-OH (2-hydroxyquinclorac quinclorac) and 1.1% for BH 514-Me (quinclorac methyl ester). DT_{50} and DT_{90} for quinclorac were 126 days and > 360 days following the first application (winter), and 8 days and 26 days following the second application (summer).

Long-term soil accumulation studies were not submitted to the Meeting.

Confined rotational crop studies

The metabolism of [2, 3, 4-^{14}C]-quinclorac was investigated by Winkler, V and Brown M (1987, BASF 1987/5037) in the rotational crops wheat, mustard greens, turnips, sorghum and soya bean from two consecutive (first (120 days after treatment) and second (360 days after treatment) rotations. A replanting of a crop just after harvest (30 days) was not considered necessary as in good agricultural practice a new crop is never planted 30 days after harvesting of a rice crop. The first (autumn) rotational crops were wheat, mustard green and turnips, the second (annual) rotational crops were sorghum, mustard green, soya beans and turnip.

Quinclorac was applied to flooded and non-flooded rice (primary crop) at a rate of 0.84 kg ai/ha in Mississippi, USA. Rice was planted approximately one month prior to application.

The formulation was applied to one plot under flooded conditions and to another plot under non-flooded conditions; seven days later, permanent flood conditions were established on this plot. After mature harvest of rice, the soil was “worked and prepared” before first rotational crops were planted less than one month after the rice harvest (120 DAT) followed by the second rotational crops 360 DAT. The study was ended 474 days after the application of quinclorac.

Table 31 Physiochemical properties of the soil

Soil type	pH	OM%	Sand %	Silt	Clay	Field moisture	CEC meq/100g
Silty clay	6.5	2.2	9.6	40.4	50.0	21.69	33.18

Soil and plant samples were collected. Soil samples were extracted with distilled water following centrifugation. Residual soil was re-suspended in 0.1N NaOH and refluxed, centrifuged and analysed by combustion to determine $^{14}CO_2$ content. Plant samples were extracted with aqueous acetone, concentrated into the aqueous phase, acidified and extracted with ethyl ether. Soya bean and wheat seeds were defatted with hexane, hydrolysed with 1N HCl and extracted with ethyl ether. Soya bean was also further extracted by 1% NaCl reflux, EDTA reflux, 5% NaOH extraction and finally 1% sodium chlorite treatment for three hours at 80 °C. The extracted residues were derivatized using diazomethane before TLC analysis using authentic standards for parent quinclorac and the metabolite BH 514-1 (3-chloro-8-quinoline carboxylic acid) which was found as a major metabolite (55.7% AR) in the aquatic soil degradation systems.

The non-flooded treatment gave the highest residues in soil and in crops. In the following table the TRRs found in soil samples (silty clay) in the first 1–10 cm from non-flooded treatments are summarized.

Table 32 Total radioactive residues (mg/kg ^{14}C-quinclorac equivalents) in the first 10 cm of a silty clay soil (non-flooded)

Days after treatment	Total TRR	Water extract	0.1N NaOH Extract	Unextracted	Unextracted % TRR	% material balance
0	0.424	0.365	0.094	0.022	5	116
1	0.056	0.031	0.031	0.011	20	107
3	0.029	0.004	0.013	0.013	45	103
4	0.033	0.007	0.015	0.012	36	102
6	0.051	0.003	0.009	0.017	33	58
326	0.123	0.026	0.067	0.034	27	103
385	0.043	0.002	0.010	0.024	65	94
474	0.059	0.005	0.025	0.028	45	99

Water extracted residues is considered as free and available to the plant, while generally sodium hydroxide extracts are ionic and covalent bound residues**.** The total radioactive residues decreased from an initial level of 0.4 ppm to 0.056 ppm one day after application, and remained relatively constant at that level through the 474 days study.

The table below summarizes the uptake of radioactive residues in different rotational crops and their matrixes.

Table 33 Total radioactive residue (mg/kg ^{14}C-quinclorac equivalents) in first (autumn) and second (annual) rotational crops

Crop *In brackets days from application to harvest is stated*	First rotation**,** planted 120 days after treatment		Second rotation**,** planted 360 days after treatment	
	Non-flooded	Flooded	Non-flooded	Flooded
Leafy vegetable				
Mustard top (158)	0.015	0.006	0.014 (0.016,0.012)	0.003 (0.002, 0.003)
Mustard plant (40)	0.028	0.009		
Small grain				

Crop *In brackets days from application to harvest is stated*	First rotation, planted 120 days after treatment		Second rotation, planted 360 days after treatment	
	Non-flooded	Flooded	Non-flooded	Flooded
Wheat seed (205)	0.025	0.013	n.a.*	n.a.*
Wheat straw (205)	0.021	0.016-	n.a.*	n.a.*
Wheat plant (40d)	0.019	0.029	n.a.*	n.a.*
Soya bean seed (82)	n.a.*	n.a.*	0.017	0.009
Soya bean stalk (82)	n.a.*	n.a.*	0.025	0.013
Soya bean plant (50)	n.a.*	n.a.*	0.006 (< 0.002, 0.009)	0.004 (0.005 0.003)
Sorghum seed (171)	n.a.*	n.a.*	< 0.002	0.006
Sorghum heads (171)	n.a.*	n.a.*	< 0.002	0.028
Sorghum tops (23)	n.a.*	n.a.*	0.03 (0.038, 0.023)	0.006
Sorghum plants (65)	n.a.*	n.a.*	-	0.003
Sorghum stalk (171)	n.a.*	n.a.*	0.013	0.006
Root and tuber vegetable				
*Turnip plant**(40-50)*	0.012	0.004	0.008	0.003
*Turnip root** (82-172)*	0.008 (0.013,0.003)	0.006 (0.009, 0.002)	0.02 (0.042,0.005)	0.002
Soil mean values				
0-10 cm	0.041	0.012	0.065	0.017
10-20	0.034	0.016	0.023	0.014
20-30 cm	0.021	0.014	0.023	0.018

*The first (fall) rotational crops were wheat, mustard green and turnips, the second (annual) rotational crops were sorghum, mustard green, soya beans and turnip.

**sampling days for first and second rotational crop

In the first rotational crops as well as the second rotational crops, the residues found were higher from crops grown under non-flooded conditions. Uptake of residues was observed in both first and second rotational crops. For the first rotational crops maximum uptake was 0.028 mg eq/kg for leafy vegetable (mustard plant), for small grain (wheat seed) 0.025 mg eq/kg and for root and tuber vegetable (turnip plant) 0.012 mg eq/kg. For the second rotational crops maximum uptake was 0.014 mg eq/kg for leafy vegetable (mustard top) for small grain (soya bean seed) 0.017 mg eq/kg and for root and tuber vegetable (turnip root) 0.02 mg eq/kg.

The methylated extracted residues were identified as mainly total quinclorac and trace amounts of the metabolite BH 514-1 (3-chloro-8-quinoline carboxylic acid).

The table below quantifies the total radioactive residues found in the different rotational crops and their matrixes.

Table 34 Extraction of ^{14}C-quinclorac from mustard, wheat and soya bean samples

Crop	days after treatment	TRR (quinclorac equivalents)*	
		mg/kg TRR	% TRR
Mustard top	147		
total		0.015	(100)
acidic ether		0.005	31
aqueous acidic		-	0
Marc		0.004	23
Wheat seed	147		
total		0.025	(100)
hexane		-	
acidic ether		0.017	67
aqueous acidic		0.007	42
Marc		0.005	19
Mustard top	303		
total		0.012	(100)
acidic ether		0.003	27
aqueous acidic		-	0
Marc		0.004	32
Soya bean seed	303		
total		0.017	(100)

Crop	days after treatment	TRR (quinclorac equivalents)*	
		mg/kg TRR	% TRR
hexane		0.004	25
acidic ether		0.003	17
aqueous acidic		-	0
Marc		0.010	62
Soya bean hay	303		
Total		0.025	(100)
acetone/water		0.002	7
1N HCL		0.002	6
Marc		0.018	73

Quinclorac was the only major residue (>10% TRR but less than 0.05 mg eq/kg) detected in the examined rotated crops. The metabolism of quinclorac by soya bean was different from mustard and wheat due to that as much as 62% TRR (0.01 mg eq/kg) was found in soya bean seed as insoluble residues in the marc. Further extraction of the marc revealed radioactive residues in protein, carbohydrate and lignin fractions according to table below.

Table 35: Characterization of ^{14}C-quinclorac residues in soya bean

Fraction	soya bean seed	soya bean hay
	% TRR	
Hexane	8	
Pellet (insoluble debris)	37	
Protein	34	
Carbohydrate soluble	35	
Acetone extract		15.8
Polysaccharides water soluble		11.7
Pectic polysaccharides		21.8
Hemicellulose I		36.8
Lignin		11.8
Hemicellulose II		10.5

The metabolism of [3-^{14}C]-quinclorac was investigated by Nelsen, J (1992, BASF 1992/5044) in rotational crops mustard green, turnip and barley from one rotational interval (120 days). 3-^{14}C-quinclorac was applied as a spray pre-emergence and 25 days later post-emergence to sorghum plants. Treatment levels were 0.527 kg ai/ha pre-emergence and 0.504 kg ai/ha post-emergence. Sorghum plants were harvested 95 days after the post-emergence treatment. Approximately 120 days after the post-emergence treatment, mustard, turnip and barley seed were planted. Rotational crops were harvested at maturity. Barely forage was harvest 205 days after treatment.

Crop samples were treated with 0.1N NaOH or 0.1N NH_4OH and then extracted with acetone. The filtrate was subsequently acidified, concentrated, the pH adjusted to 8 and then diluted with water and extracted with dichloromethane. The water layer was further extracted with dichloromethane or refluxed with HCl. All subsamples of marc were subjected to refluxing with sodium chloride to determine radioactivity incorporated into water soluble polysaccharides, with EDTA to determine peptic polysaccharides, with sodium hydroxide to determine hemicellulose I or II and with sodium chlorate to determine lignin.

Total radioactivity was determined by combustion analysis and LSC. The nature of the residues was determined by fractionation and TLC using quinclorac, BH 514-1 (3-chloro-8-quinilone carboxylic acid) and their respective methylated samples as reference standards.

In the table below the extracted and identified radioactivity residue found at harvest in the different rotated crop matrixes is presented.

Table 36: Identification and characterization of radioactive residues in rotational crops (1st rotation, 120 days) following application of [3-^{14}C]-quinclorac to sorghum crop at a total rate of 1.0131 kg ai/ha

Metabolite fraction	Mustard green		Turnip				Barely					
			turnip top		root		grain		straw		forage	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Dichloromethane I (neutral)	4.89	0.008	6.46	0.014	22.22	0.006	5.71	0.008	1.75	0.003	7.58	0.014
Dichloromethane II (acidic)	74.98	0.12	63.07	0.132	44.82	0.011	58.71	0.088	28.31	0.045	64.50	0.116
Dichloromethane III (hydrolysed)	-	-	-	-	2.80	< 0.001	5.33	0.008	6.55	0.01	1.77	0.003
Aqueous	6.01	0.01	8.19	0.017	13.1	0.003	13.29	0.020	27.79	0.044	10.52	0.019
Non-extracted residues (marc)	7.42	0.012	17.34	0.036	9.83	0.002	10.86	0.016	37.02	0.059	14.09	0.025
TRR*	100	0.16	100	0.21	100	0.025	100	0.18	100	0.16	100	0.15
Identification and characterization of TRR												
quinclorac	72.1	0.115	61.3	0.129	40.2	0.010	63.7	0.114	63.7	0.114	58.7	0.088
quinclorac methyl ester	3.82	0.006	4.17	0.009	1.76	< 0.001	< 0.3	< 0.001	< 2.9**	< 0.001	< 5.6**	< 0.008
Total identified	75.92	0.12	65.47	0.138	41.96	0.0011	64.0	0.115	66.6	0.115	64-3	0.097

*The TRR identified in the different fractions does not always sum up to the TRR in the combustion analysis.

** TLC was not performed on these samples

The parent quinclorac was a major residue in all matrices and the metabolite BH 514-Me (quinclorac methyl ester) a minor metabolite (3-4% TRR) in mustard green and turnips.

Field rotational crop study

Magnitude of quinclorac residues in rotational rape seed (canola) was investigated by Barney, WP (1993, BASF 1993/5157) in a field trial in Canada. Rape seed was planted in the same plot as barley which had been grown and treated the previous year with quinclorac in a single broadcast post emergence application at the rate of 0.2 kg ai/ha.

Rape seed samples (four replicates) were harvested at maturity and stored frozen until analysis within 5 months. Samples were analysed for quinclorac with method A8902 using GC/EDC detection. The method was validated to a LOQ of 0.05 mg/kg for oil seed and the method recovery was at fortification level 0.05 mg/kg was 96% (n=1) and at fortification level 0.5 mg/kg 82% (n=1).

Table 37 Residues from quinclorac in rotating seed from plots where barley was grown previous year and treated with 0.2 kg ai/ha

Location	Application			Residues				Trial
Year (variety)	kg ai/ha	no	Growth stage	matrix	PHI	Total quinclorac (mg/kg)	mean mg/kg	Reference
Canada Minto, Manitoba 1991 barley	0.2	1	Not reported	Not sampled	60	-	-	BASF 1993/5157
Canada Minto, Manitoba 1992 Rape seed	-	-	-	grain	-	< 0.05, < 0.05, < 0.05, < 0.05	< 0.05	BASF 1993/5157

ANALYTICAL METHODS

The meeting received analytical method description and validation data for quinclorac and its metabolite quinclorac methyl ester. Most matrices are validated with a LOQ of 0.05 mg/kg for both analytes, however in strawberry and oil seeds the analytes were validated also with a LOQ of 0.01 mg/kg. A summary of the analytical methods for plant and animal commodities is provided below.

Table 38 Overview of analytical methods used for the quantification of quinclorac residues in plant and animal matrices

Method (analytes)	Matrix	Extraction	Clean-up	Detection, LOQ
Method 268 Quinclorac	Animals (eggs, milk, muscle, kidney, fat, liver)	Acetone/sodium solution, acidify and partition with ethyl acetate, and derivatized with diazomethane.	filtration and C-18 SPE column	GLC-ECD SE 54 capillary column at 270-350°C, Quantification by external standards. Quinclorac-Me LOQ, 0.05 mg/kg
Method 268-1 amendment Quinclorac	Animals (eggs, milk, muscle, kidney, fat, liver)	Acetone/sodium solution, acidify and partition with dichloromethane, and derivatized with diazomethane.	filtration and amino SPE column	GLC-ECD SE 54 capillary column at 270-350°C, Quantification by external standards. Quinclorac-Me LOQ, 0.05 mg/kg
Method M829/A Quinclorac	strawberry and high water fruit crops	Acetic acid in acetonitrile in the presence of magnesium sulfate and sodium chloride		LC-MS/MS using a C18 analytical column. Quantification is made using internal standard The quinclorac ion transition 242→224 is used for quantification and the ion transition m/z 244-226 is used for confirmation. LOQ 0.01 mg/kg
Method A8902 Quinclorac	Rice (grain and straw rough rice, rice hulls, brown rice, rice bran, milled rice) Sorghum forage grain and stover.	Acetone /0.1 M NaOH solution acidify and partitioned with dichloromethane, and derivatized with diazomethane.	filtration and by solid phase extraction (silica gel column)	The methylated samples are analysed by GC-EDC at 300°C using a DB17 fused silica column at 200ºC and an electron capture detector (GC-ECD). Quantification by external standards. Quinclorac-Me LOQ 0.05 mg/kg
Method D9708 Quinclorac	Sorghum forage, grain and stover Validation data for	Acetone /0.1 M NaOH acidify and partitioned with dichloromethane, diluted with sodium	filtration and by using quaternary amine SPE column	HPLC-MS/MS using a Betasil C18 column Quantification by

Method (analytes)	Matrix	Extraction	Clean-up	Detection, LOQ
	sorghum stover/fodder was not presented	hydroxide and pH adjusted to 8-11		external standards. The ion transition m/z 240→196 is monitored. Quinclorac LOQ 0.05 mg/kg
Method D9708/1 Quinclorac	Wheat (forage, grain, straw, grain and processed commodities) Rape seed and oil	Plant material: Acetone /sodium solution, acidify and partition with dichloromethane Canola seed: hexane/sodium solution, partition with acetonitrile	filtration and C18 SPE column	HPLC-MS/MS using a Betasil C18. Quantification is performed using external standards. The ion transition 240→196 is used for quantification Quinclorac LOQ 0.05 mg/kg
Method D9806 Quinclorac-Me	Rape seed and oil	Seed: acetone/hexane and partitioned with acetonitrile/water and methanol. Oil: hexane and partitioned acetonitrile/water and methanol.	Filtration and C18 SPE columns	HPLC-MS/MS using a Betasil C18 column Quantification is performed using external standards. Quinclorac methyl ester. The ion transition m/Z 255-224 is used for quantification. LOQ 0.05 mg/kg
method D9708/01 and method D9706 Quinclorac and Quinclorac -Me	Cereal grain and oil seed	Quinclorac extracted as in method A8902. Quinclorac methyl ester was extracted by acetone and the residue diluted in hexane. Partitioned twice acetonitrile/water and methanol.	Filtration and C18 SPE columns	HPLC-MS/MS using a Betasil C18 column Quantification is performed using external standards. Quinclorac. The ion transmission m/Z 240-196 used for quantification Quinclorac methyl ester. The ion transition m/Z 255-224 is used for quantification LOQ 0.05 mg/kg
Method D9708/2 and Method D9806/2 Quinclorac and Quinclorac -Me	wheat grain and oil seed (canola)	The extraction procedure is the same as for method D9708, see above	Filtration and C18 SPE columns	HPLC-MS/MS using an Acquity UPLC HSS T3 column. Quantification is performed using external standards The quinclorac ion transition m/z 242→224 is used for quantification and the ion transition m/z 242→161 is used for

Method (analytes)	Matrix	Extraction	Clean-up	Detection, LOQ
				confirmation. The quinclorac methyl ester ion transition m/z 256→224 is used for quantification and the ion transition m/z 256→161 is used for confirmation LOQ 0.05 mg/kg for both analytes
Method R0036 Quinclorac and Quinclorac methyl ester	rape seed, rape oil	Plant material: *Quinclorac* acetone/sodium solution, acidify and partition with dichloromethane. *Quinclorac methyl ester* acetone. The centrifuged sample is saturated with sodium chloride and extracted with dichloromethane. Oil: *Quinclorac* hexane followed by acetonitrile/sodium hydroxide *Quinclorac methyl ester* hexane and acetonitrile/water and methanol. The centrifuged samples is saturated with sodium chloride, acidified and extracted with dichloromethane.	Not necessary using HPLC-MS/MS and the instruments high degree of specificity	HPLC-MS/MS using an Atlantis T3 column. The quinclorac ion transition m/z 242→224 is used for quantification and the ion transition m/z 242→161 is used for confirmation. The quinclorac methyl ester ion transition m/z 256→224 is used for quantification and the ion transition m/z 256→161 is used for confirmation LOQ 0.01 mg/kg for both analytes.

Animal commodities

For quantification of parent quinclorac in animal commodities method 268 was developed and validated by Mayer, F (1988, BASF 88/0542).

Method 268 (animal matrices)

The analytical method 268 was described and validated for parent quinclorac by Mayer, F (1988 a, BASF 88/0542) for cow and chicken tissues, milk and eggs. Homogenised samples (20g) were extracted with acetone/0.1 N NaOH (15:10, v/v) for 5 min followed by acidification with sulphuric acid. After centrifugation, the remaining solids were extracted again with acetone/0.1 N sulphuric acid (50:50 v/v). Both extracts were combined and the interferences were removed by clean-up on an Extrelut column, followed by $NaHCO_3$/ethyl acetate partition at pH =8. The extract was acidified to pH 2 and quinclorac was partitioned into ethyl acetate. After clean-up with C18 modified silica, quinclorac was derivatized (methylated) with diazomethane and determined by GC-ECD using a derivatized external standard. Confirmation was obtained by HPLC-UV or GC MS at m/z 224, 226, 255, 257. The reported LOQ was 0.05 mg/kg. Validation results are shown in Table 39

Method 268/1, amendment to method 268 (animal matrices)

In the amended method 268/1 by Mayer, F (1989 BASF/10911) the same procedure was followed except that quinclorac was partitioned into dichloromethane, cleaned using amino SPE and eluted with citrate buffer /dichloromethane.

Independent laboratory validation (ILV) studies for the method were not presented to the Meeting.

Table 39 Recovery data for determination of quinclorac in animal matrices

Cow	Fortification level	n	recovery mean	SD	CV	Analyte, reference
muscle	0.05	5	79.7	9.7	12.1	Quinclorac equivalents
	5	5	70.2	2.2	3.2	
Fat	0.05	5	68.7	5.8	8.4	(quantified as quinclorac-ME)
	5	5	66.7	3.4	5.1	
Liver	0.05	5	77.5	8.4	10.8	(BAS 514H)
	5	5	70.4	1.2	1.7	(1988, BASF 88/0542)
Kidney	0.05	5	70.9	7.8	11.0	method 268 original
	5	5	72.9	5.0	6.9	
Milk	0.05	5	76.9	4.6	6.0	
	5	5	69.6	5.1	7.4	
Chicken						
muscle	0.05	5	75.6	11.5	15.2	
	5	5	78.2	3.4	4.4	
skin + fat	0.05	5	75.1	15.7	20.9	
	5	5	77.1	2.3	3	
Liver	0.05	5	69.9	14.4	20.6	
	5	5	90.0	3.6	4.0	
Egg	0.05	5	70.0	2.9	4.1	
	5	5	68.8	2.7	4.0	
cow milk	0.05	5	90.2	1.9	2.1	Quinclorac (BAS F514H)
	5	5	82.6	1.4	1.7	(1989a, BASF 89/5001)
goat muscle	0.05	5	75.2	7.9	10.5	method 268/1
	5	5	85.0	2.1	2.5	
goat liver	0.05	5	81.4	7.5	9.3	
	5	5	63.6	5.6	8.8	

A radiovalidation of the method 268 was conducted by Mayer F (1988 b BASF/10179) with samples from muscle, skin with fat, liver, kidney and eggs from the hen metabolism study. The total radioactive residues were determined by combustion LSC and thereafter analysed in duplicate according to method 268. All fractions containing the parent compound were analysed by LSC. Concurrent recoveries verified on non-radiolabelled control samples ranged between 66–88%. Extraction efficiency in the different hen matrices varied from 91–98% TRR. Quinclorac as quantified by method 268 accounted for 60–81% TRR. This is slightly lower than the amounts found in the metabolism study, where 78–92% TRR could be assigned to parent. Losses mainly occurred during C18 clean-up due to irreversible adsorption to the column.

Table 40 Radiovalidation for hen matrices using method 268

Matrix	TRR mg/kg	Total extracted %TRR [a]	Parent %TRR method 268 (B)	Parent mg/kg method 268	Parent %TRR metabolism study (C)	Trueness ratio B:C	Concur recovery
Hen muscle 367; 372	4.29	91%, 93%	63%; 72%	2.72; 3.10	86-87%	0.78	84%
Hen skin with fat 367	6.41	98%, 98%	60%; 60%	3.85; 3.88;	86-88%	0.69	88%
Hen liver 367	9.28	91%; 95%	78%; 81%;	7.54; 7.20	91-92%	0.88	72%
Hen kidney 366-372	18.4	91%; 93%;	63%; 63%	11.7; 11.7	na	-	88%
Hen eggs	1.24	93%; 96%;	74%; 65%	0.92; 0.80;	78-83%	0.87	66%

367, day 2							

[a] Extraction using acetone/0.1 M NaOH and acetone; 0.1 M sulfuric acid (method 268)

A radiovalidation of the method 268 was conducted by Mayer, F (1989 BASF/5001) with samples from muscle, fat, liver, kidney and milk from the goat metabolism study. The total radioactive residues were determined by combustion LSC and thereafter analysed in duplicate according to method 268 and 268/1. All fractions containing the parent compound were analysed by LSC. Extraction efficiency in the different goat matrices varied from 72 to 104% TRR. Concurrent recoveries verified on non-radiolabelled control samples ranged between 54–77% for method 268. Quinclorac as quantified by method 268 accounted for 42–88% TRR, this is lower than the amounts found in the metabolism study, where 81–96% TRR could be assigned to parent. Losses mainly occur during C18 clean-up due to irreversible adsorption to the column. When the clean-up procedure was changed, as in method 268/1, concurrent recoveries improved to 66–96%. Quinclorac as quantified by method 268 accounted for 61–85% TRR, this is lower than the amounts found in the metabolism study, where 81–96% TRR could be assigned to parent.

Table 41 Radiovalidation for lactating goat matrices using method 268

Matrix	TRR mg/kg	Total extracted %TRR (A)	Parent %TRR method 268 (B)	Parent mg/kg method 268	Parent %TRR metabolism study (C)	Trueness ratio B:C	Concur recovery
Method 268,original method							
goat muscle	0.196	79%; 81%	48%; 57%	0.095; 0.112	na	na	77%
goat omental fat	0.191	73%; 72%	83%; 83%	0.159; 0.159	na	na	60%
goat subcu taneous fat	0.672	93%; 86%	70%; 67%	0.470 0.449	na	na	54%
goat liver	2.216	82%; 85%	42%; 47%	0.926; 1.05	81%	0.55	58%
goat kidney	11.54	102%; 104%	78%; 71%	9.03; 8.23	86%	0.87	66%
goat milk	0.117	73%; 76%	42%; 47%	0.048; 0.055	86%	0.52	77%
method 268/1 modification							
goat liver	2.307	93%; 94%	61%; 64%	1.40; 1.48	81%	0.77	71%; 79%
goat muscle	0.230	96%; 94%	77%; 80%	0.178; 0.183	na	na	66%; 74%
goat milk	0.090	96%; 96%	85%; 82%	0.076; 0.073	86%	0.97	92%; 96%

Plant commodities

Strawberry

The analytical (enforcement) (method M829/A was developed by White, G (2015, J20044) for the determination of quinclorac in strawberry representing a high water content crop. Residues of quinclorac are extracted from plant matrices by sonication with 1% acetic acid in acetonitrile in the presence of magnesium sulfate and sodium chloride. Following centrifugation, samples are diluted with 0.1% formic acid and analysed by LC-MS/MS. The determination of the residues is calculated using matrix matched standards employing triphenyl phosphate (TPP) as the internal standard. The quinclorac ion transition 242→224 is used for quantification and the ion transition m/z 244-226 is used for confirmation. LOQ is 0.01 mg/kg.

The applicability of the method was confirmed in an independent laboratory by Moinuddin, A (2015, JRF/228-2-13-10872). In both laboratories parent quinclorac were analysed with validated LOQ of 0.01 mg/kg in strawberry see Table 46).

Rice

The analytical method A8902 was validated for parent quinclorac by Single, YM (1989 BASF 5007) for residues in rice grain and straw with an LOQ of 0.05 mg/kg. The residues are extracted from 5–10 g plant materials. Samples are soaked in 0.1 N sodium hydroxide for 1 hr prior to extraction with acetone (acetone/0.1 M NaOH, 10:15, v/v). After centrifugation in the presence of Celite, the extract was acidified with sulfuric acid and the acetone was removed by evaporation at 50 °C. The extract was adjusted to pH 8 by $NaCO_3$ and partitioned with dichloromethane to remove matrix impurities. The aqueous phase was acidified and residues were partitioned into dichloromethane. After filtration and cleaning by SPE (silica gel column) quinclorac residues were derivatized (methylated) with diazomethane and determined by GC-ECD. Quantification is performed using a derivatized external standard. Residues are expressed as quinclorac.

Independent laboratory validation (ILV) studies for the method were not presented to the Meeting.

A radiovalidation of the method A8902 was conducted by Single Y (1989 BASF 5006) with samples from rice grain, straw and forage from the rice metabolism study. The total radioactive residues were determined by combustion LSC and thereafter analysed in duplicate according to method A8902. All fractions containing the parent compound were analysed by LSC. Extraction efficiency in the different rice matrices were 88% for the grain, 90% for the straw and 84% for the forage. Average concurrent recoveries verified on non-radiolabelled control samples for rice grain were 87% for method A8902. Quinclorac as quantified by method A8902 accounted for 69–77% TRR, this is lower than the amounts found in the metabolism study, where 85–94% TRR could be assigned to parent.

Table 42 Radiovalidation for rice using method A8902

Matrix	TRR mg/kg	Total extracted %TRR [a]	Parent %TRR method A8902 (B)	Parent mg/kg method A8902	Parent %TRR metabolism study (C)	Trueness ratio B:C	Concur recovery
rice grain (growth chamber)	1.66	88	77	1.28	94	0.82	87
rice straw (growth chamber)	13.5	90	86	11.6	86	1.00	-
rice forage (field)	0.68	84	69	0.40	85	0.81	-

[a] Extraction with 0.1 N NaOH in acetone as for method A8902

Wheat

The analytical method D9708/1 was validated for parent quinclorac in wheat (forage, grain, straw, flour and bran) with an LOQ of 0.05 mg/kg by Guirguis M, and Riley M (1998 BASF/5095**).** The residues were extracted from 5–10 g plant material. Samples were soaked in 0.1 N sodium hydroxide for 1 hr prior to extraction with acetone (acetone/0.1 M NaOH, 10:15, v/v). After centrifugation an aliquot of the extract was acidified with HCl to pH < 2 and evaporated at 50 °C to remove the acetone. The residues in the extract were partitioned into dichloromethane. The dichloromethane was evaporated to dryness and redissolved in 0.0025 M NaOH (pH 9–11). After cleaning using a quaternary amine SPE column, the solution was analysed for quinclorac by HPLC-MS/MS using ion transition 240→196 for quantification and using external standards. Validation data for the method is presented in Table 46

The applicability of method D9708/2 for determination of quinclorac in wheat grain was confirmed in an independent laboratory by Li F and Patel D (2013 a, BASF/7000579). Parent quinclorac was analysed with validated LOQ of 0.05 mg/kg Table 46.

Sorghum

The analytical method D9708 was validated for the determination of parent quinclorac in sorghum commodities by Haughey D, *et.al.* (1998, BASF/5081). The residue was extracted from ≥ 0.9 kg plant material. Further description as for wheat D9708/1.

The analytical method D9708 was validated for the determination of parent quinclorac in forage, grain and fodder by Versoi, P. et al (1996/5136). The residue was extracted from >2 kg plant material. Further description is as for rice A9002.

Rape seed

The analytical method D9806 was validated for rape seed by Guirguis M and Riley M (1998 BASF/5184) for the determination of quinclorac methyl in rape seed and oil (canola). Seed samples (10 g) were extracted with acetone. An aliquot of the extract is evaporated to dryness and redissolved in hexane. Oil samples (2g) are dissolved in hexane. Hexane solutions from seeds or oil are partitioned twice with 95% acetonitrile/water (2:1, v/v)/5% methanol. The samples are cleaned-up using C18 SPE columns. The eluates are evaporated to dryness and redissolved in HPLC mobile phase. The samples are analysed for quinclorac-methyl by HPLC-MS/MS. The ion transition m/z 255→224 is used for quantification. Quantification is performed using external standards.

The analytical method D9708/1 for the determination of quinclorac and analytical method D9806 for determination of quinclorac methyl ester were validated for oilseed rape seed by Guirguis M, (1998/ BASF 5174).

The applicability of the method D9708/2 for determination of parent quinclorac and method D9806/2 for determination quinclorac methyl ester was confirmed in an independent laboratory for wheat grain and rape seed (canola) by Li F, and Patel D 2013a BASF/7000579). Quinclorac and quinclorac methyl ester were analysed with a validated LOQ of 0.05 mg/kg.

Recovery data are presented in table below.

Table 43 Procedural recovery for quinclorac with method D9708/2

Matrix	Fortification level (mg/kg)	n	recovering	average rec	SD	% RSP
wheat grain	Primary quantification (mz 242-mz 224) using LC-MS/MS					
	0.05	5	85, 86, 89, 92, 94	87	3.2	3.6
	5.0	5	87, 887, 79,79 86	84	4.4	5.3
	Confirmatory quantification (mz 256-m/z 161)					
	0.05	5	85, 82, 86, 94, 84	86	4.4	5.1
	5.0	5	87, 86, 79, 77, 85	83	4.6	4.6
canola seed	Primary quantification (mz 242-mz 224) using LC-MS/MS					
	0.05	5	85, 105,84,72,86	86	11.8	13.6
	5.0	5	86,83,90,99,105	93	9.3	10.0
	Confirmatory quantification (mz 256-m/z 161)					
	0.05	5	87, 107, 78, 71, 82	85	13.8	16.2
	5.0	5	86, 83, 86,99,102	91	8.6	9.4

Table 44 Procedural recovery for quinclorac methyl ester (BH 514-Me)

Matrix	Fortification level (mg/kg)	n	recovering	average rec	SD	% RSP
canola seed	Primary quantification (mz 242-mz 224) using LC-MS/MS					
	0.05	5	76, 95, 109, 98, 84,	92	12.8	13.9
	5.0	5	96, 85, 88, 92, 90,	90	4.1	4.5
	Confirmatory quantification (mz 256-m/z 161) using LC-MS/MS					
	0.05	5	67, 84, 100, 89, 73	83	13.1	15.8
	5.0	5	93, 83, 86, 90, 88	88	3.9	4.4

A radiovalidation of the method D9708/1 (quinclorac) was conducted by Parker 1998a (BASF 1998/5180). Analytical method D9708/1 was used to quantify quinclorac in the rape seed from the rape seed metabolism study and 45.3% TRR (0.218 mg/kg) accounted for parent quinclorac. Compared to the metabolism study, where 37.1% TRR (0.176 mg/kg) accounted for parent quinclorac after extraction with acetone/buffer pH 7, the analytical method results in higher residue levels for the parent compound. This can be explained by the partial conversion of quinclorac methyl back to parent as a result of the alkaline extraction conditions (acetone/0.1 M NaOH) used in the analytical method. The conversion percentage was determined by fortifying control samples with 0.5 mg/kg quinclorac methyl and analysing the rapeseed by method D9708/01 (for parent quinclorac). The conversion averaged 25.2% for four samples with a range of 15.8–32.6%. Method D9708/1 is therefore not suitable for determination of parent quinclorac.

A radiovalidation of the method D9806 (quinclorac methyl) was conducted by Parker 1998a (BASF 1998/5180). Analytical method D9806 was used to quantify quinclorac methyl in the rape seed from the rape seed metabolism study and 30.3% TRR (0.144 mg/kg) accounted for quinclorac methyl. Compared to the metabolism study where 37.1% TRR (0.176 g/kg) accounted for quinclorac methyl, the analytical method results are within acceptable levels.

The analytical (enforcement) method R0036 was validated by Malinsky, D, S (2013 BASF 7002468) for the determination of quinclorac and quinclorac methyl ester residues in rape seed and oil. Validation data for the method is presented in Table 45.

Parent quinclorac residues in/on plant samples (5 g each) are extracted using acetone/0.1 N NaOH (3:1, v/v). After centrifugation, residues in an aliquot of sample extract are cleaned up by liquid-liquid partitioning in which residues are diluted with water and saturated NaCl solution, concentrated to remove the acetone, and partitioned against dichloromethane, which is discarded. The residues in the aqueous phase are then acidified (pH ~2–3) with concentrated formic acid, partitioned into dichloromethane, and evaporated to dryness. The residues are re-dissolved in a final volume of acetonitrile:water (10:90, v/v), filtered, and analysed by HPLC-MS/MS.

From oil samples (5 g each), parent quinclorac residues are extracted with a mixture of hexane, acetonitrile:0.1 N NaOH (1:1, v/v), and methanol. After centrifugation, residues in the aqueous acetonitrile layer are diluted to volume with acetonitrile:0.1 M NaOH (1:1, v/v). An aliquot of the extract is concentrated to remove the acetonitrile, and residues in the aqueous remainder are then subjected to extensive liquid-liquid partitioning, finally into dichloromethane, the combined extracts of which are evaporated to dryness. The residues are re-dissolved in acetonitrile:water (10:90, v/v), filtered, and then analysed by HPLC-MS/MS.

Residues of quinclorac methyl ester in/on canola seed samples (5 g each) are extracted with acetone. An aliquot of extract is evaporated to dryness, and the residues are redissolved, and subjected to liquid-liquid partitioning, in saturated NaCl solution and dichloromethane. The residues in an aliquot of the dichloromethane layer are evaporated to dryness, re-dissolved in methanol:water (1:1, v/v), filtered, and then analysed by HPLC-MS/MS.

From oil samples (5 g each), quinclorac methyl ester residues are extracted with a mixture of hexane, acetonitrile:water (2:1, v/v), and methanol. The residues in the aqueous acetonitrile layer are diluted with acetonitrile:water (2:1, v/v), an aliquot is taken, further diluted with methanol-water (1:1, v/v), filtered, and analysed by HPLC-MS/MS.

Quantification is performed using external standards. Quinclorac is quantified using m/z 242→224 for quantification and m/z 242→161 for confirmation. Quinclorac methyl is quantified using m/z 256→224 for quantification and 256→161 for confirmation. LOQ is 0.01 mg/kg for both analytes.

Acceptable linearity was observed within the 0.01–0.25 ng/mL standard range and the two mass transitions for each analyte ($r = \geq 0.9976$). No interfering peaks were found at the retention times for these analytes. Matrix effects on the detector response were less than 20%); therefore, the validation samples were analysed only using solvent-based calibration standard solutions. Further validation results are shown in Table 45.

The applicability of the method was confirmed in an independent laboratory by Schmitt J.L (2013 a, BASF/7002603). In both laboratories parent quinclorac and quinclorac methyl ester were analysed with validated LOQ of 0.01 mg/kg. Validation results are shown in table below.

Table 45 Recovery data for determining quinclorac and quinclorac methyl ester for Method R0036

Matrix	Analyte	No. of tests	Fortification level [mg/kg]	Transition 242 > 224			Transition 242 > 161		
				mean [%]	SD [+/-]	CV [%]	mean [%]	SD [+/-]	CV [%]
Lettuce leaves	Quinclorac	5	0.01	94	8	8	97	8	8
		5	1.0	100	2	2	101	5	5
		10	Overall	97	6	7	99	7	7
Corn grain	Quinclorac	5	0.01	90	4	5	92	4	5
		5	1.0	105	4	4	106	2	2
		10	Overall	98	9	9	99	8	8
Bean, dried seed	Quinclorac	5	0.01	99	4	4	103	1	1
		5	1.0	91	9	10	88	7	8
		10	Overall	95	8	8	96	9	9
Grape, fruit	Quinclorac	5	0.01	105	6	6	110	4	3
		5	1.0	109	4	3	109	7	7
		10	Overall	107	5	5	109	6	5
Canola Seed	Quinclorac	5	0.01	104	2	2	108	2	2
		5	1.0	107	6	6	108	5	5
		10	Overall	106	5	4	108	4	4
Canola Oil	Quinclorac	5	0.01	104	8	8	110	9	8
		5	1.0	99	6	7	100	4	4
		10	Overall	101	7	7	105	8	8
Canola Seed	Quinclorac methyl ester	5	0.01	85	15	17	73	11	15
		5	1.0	95	3	3	91	3	3
		10	Overall	90	11	13	82	12	15
Canola Oil	Quinclorac methyl ester	7	0.01	90	5	6	82	3	4
		7	1.0	86	2	2	86	4	4
		14	Overall	88	4	4	84	4	5

A radiovalidation study showed that extraction with acetone/0.1 M NaOH converts quinclorac-methyl partly into parent compound. For this reason, the parent is overestimated in samples containing quinclorac-methyl ester. Methods D9708/1 (quinclorac) and R0036 (quinclorac) use acetone/0.1 M NaOH and are therefore not suitable for the determination of parent compound in oilseed rape seed and possibly other pulses and oilseeds, where the quinclorac methyl ester can be expected to be present.

Table 46 Overview of recovery data for determination of quinclorac in plant matrices with presented methods

Matrix	Fortification level	n	recovery mean	SD	CV	Analyte, reference, MRM transition
rice grain	0.05	9	93	17	19	Quinclorac (1989 BASF 5007), method A8902
	1.0	6	85	11	13	
	5.0	5	84	9.5	11	
	10.0	1	91	-	-	
rice straw	0.05	9	93	14	15	
	1.0	5	93	18	20	
	5.0	2	101	28	27	
	10.0	2	92	4.2	4.6	
	20.0	3	97	18	18	
rough rice	0.05	2	88	7.2	8.2	Quinclorac (1989a BASF 5004)
	0.5	1	80	-	-	
rice hulls	0.05	2	93	3.7	4.9	
	0.5	1	85	-	-	
	1.0	1	87	-	-	
brown rice	0.05	2	93	8.8	9.4	
	0.5	1	85	-	-	
	1.0	1	87	-	-	

Matrix	Fortification level	n	recovery mean	SD	CV	Analyte, reference, MRM transition
rice bran	0.05	4	76	7.1	9.4	
	0.5	2	83	7.8	9.4	
	1.0	1	64	-	-	
	2.0	1	82	-	-	
milled rice	0.05	2	88	12	14	
	0.5	1	89	-	-	
	1.0	1	90	-	-	
rice straw	0.05	3	77	10	13	
	1.0	1	88	-	-	
	5.0	2	72	11	15	
wheat straw	0.05	4	83	20	24	Quinclorac (1998a BASF 5008
	0.5	4	86	7.2	8.4	
	5.0	4	87	22	26	
wheat grain	0.05	6	89	9.9	11	
	0.5	6	85	25	29	
	5.0	6	99	7.8	7.9	
wheat flour	0.05	4	89	9.0	10	
	0.5	4	95	4.3	4.5	
	5.0	4	92	3.9	4.3	
wheat bran	0.05	4	75	14	19	
	0.5	4	83	21	25	
	5.0	4	86	15	17	
wheat forage	0.05	4	93	26	28	
	0.5	4	100	14	15	
	5.0	4	95	5.0	5.3	
Rape seed	0.05	4	81	19	23	
	0.5	4	85	11	14	
	5.0	4	84	15	18	
Rape oil	0.05	4	84	10	12	
	0.5	4	82	13	16	
	5.0	4	82	7.6	9.3	
Rape seed	0.05	3	76.7	9.0	11.8	Quinclorac (1998 BASF 5174)
	0.5	10	73.7	9.9	13.4	
Strawberry fruit	0.01	5	92.8	2.56	2.76	Quinclorac (2015, J20044)
	0.10	5	92.2	3.8	4.13	
Sorghum forage	0.05	5	74.4	12.0	16.2	Quinclorac (1996/5136)
	1.0	5	83.8	13.7	16.3	
Sorghum grain	0.05	5	79.6	9.9	12.5	
	1.0	5	82.8	6.7	8.1	
Sorghum fodder	0.05	4	80.0	8.5	10.6	
	1.0	3	90.7	5.0	5.6	
Sorghum forage	0.05	1	65	-	-	Quinclorac (1998/5081)
	1.0	2	94	8.5	9.0	
Sorghum grain	0.05	1	87	-	-	
	1.0	2	101	4.2	4.2	
Sorghum fodder	0.05	3	93	20.0	21.5	
Rape seed (canola)			Transition 242 > 224			Transition 242 > 161			Quinclorac (2013/7002468
			recovery mean	SD	CV	recovery mean	**SD**	**CV**	
seed	0.01	5	104	2	2	108	2	2	m/z 242-224 quantification
	1.0	5	107	6	6	108	5	5	
oil	0.01	5	104	8	8	110	9	8	m/z 242-161 confirmation
	1.0	5	99	6	7	100	4	4	

Matrix	Fortification level	n	Recovery mean	SD	CV	Analyte, reference, MRM transition
Rape seed	0.05	3	87.7	7.8	8.9	Quinclorac Methyl Ester
	0.5	6	94.5	16.2	17.1	

Matrix	Fortification level	n	Recovery mean			SD		CV	Analyte, reference, MRM transition
									(1998 BASF 5174)
Rape seed	0.05	4	105			4.8		4.6	Quinclorac Methyl Ester (1998/5184)
	0.5	4	100			1.5		1.5	
	5.0	4	95			8.0		8.5	
Rape oil	0.05	4	95			16		16	
	0.5	4	85			2.5		2.9	
	5.0	4	75			11		15	
			Quantification			Confirmation			
			recovery	mean	SD	recovery	mean	SD	
Rape seed	0.01 1.0	5 5	85 95	15 3	17 3	73 91	11 3	15 3	Quinclorac Methyl Ester ((2013/7002468) m/z 256-224 quantification m/z 256-161 confirmation
Rape oil	0.01 1.0	7 7	90 86	5 2	6 2	82 86	3 4	4 4	

Soil

The method A8903 was validated by Mayer, F et al (1989, BASF 1989/5017) for analysis of quinclorac and its metabolite BH 514-1 (3-chloro-8-quinolinecarboxylicacid) in soil. Residues of quinclorac are extracted from soil (25 g) with sodium hydroxide followed by acetone/aqueous solution and then acidified with concentrated sulphuric acid and extracted with dichloromethane.

The samples are analysed by high performance liquid chromatography with ultra-violet detected (HPLC-UV) at 230 nm, using Nucleosil 100-5-C18 column (50 mm × 4.6 mm −pre-column and 250 mm × 4.6 mm main column) and a waters Guard-Pak Pre-column with gradient elution using mobile phases of acetonitrile/water/acetic acid. Quantification is performed using external standards. Limit of quantification was 0.05 mg/kg for both analytes.

Recovery data generated from samples fortified at the LOQ and from samples fortified at 10 × LOQ are presented in the table below.

Table 47 Recovery data for quinclorac and BH 514-1 (3-chloro-8-quinolinecarboxylicacid) in soil

Test	Analyte	No of tests	mean (%)	SD (±)	CV (%)
87101	Quinclorac	5	85	6	7
	BH 514-1	5	72	10	14
87127	Quinclorac	12	85	11	13
	BH 514-1	12	74	12	16
87125	Quinclorac	6	81	6	7
	BH 514-1	6	70	6	9
87098	Quinclorac	10	76	7	9
	BH 514-1	8	59	4	7

For the analysis of quinclorac and its metabolites BH 514-2-OH (2-hydroxyquinclorac) and (BH 514-ME) (quinclorac methyl ester) in soil the method D9513 was validated by Jordan J (1996, BASF 1996/5149). The extraction of quinclorac and BH 514-2-OH from soil samples (10g) are first extracted with sodium hydroxide acidified and partitioned with 8:2 methylene chloride/ethyl acetate. The metabolite BH 514-ME is converted to parent quinclorac and is analysed as parent equivalents in the method. For the determination of BH 514-ME soil samples are extracted in a mixture of methylene chloride, ethyl acetate and methanol.

The final quantitative determination of quinclorac, BH 514-2-OH and BH 514-ME is made by LC/MS/MS using multiple reaction monitoring. The LOQ for each metabolite is defined as the lowest fortification that was successfully run through the method. For this method it is 10 ppb. The average

recoveries for BAS 514 was 85.3% +/- 3.6 (n=6) for the shake extraction method and 95.3% +/- 7.7% for the reflux extraction. The average recoveries for BH 514-2-OH was 81.7% +/- 4.5% (n=6) for the shake extraction and 83.6% +/- 8.4% (n=13) for the reflux extraction. The average recovery for BH 514-ME is 89.3% +/- 3.3% (n=13).

Stability of residues in stored analytical samples

Plant matrices

Storage stability of quinclorac was investigated in rice (grain and straw) and sorghum (forage, hay, grain, silage and fodder) matrixes up to 38 months by Burkey, J (1994, BASF/5015) in wheat up to 26 months by Burkey, J (1996, BASF/5110) and in cranberry up to 14 months by Barney, WP, Homa K (2010, BASF /7018348).

Homogenized samples of rice, sorghum and wheat were fortified individually at levels of 1 mg/kg for quinclorac and stored frozen. Bulk control matrix was placed into storage simultaneously. At each sampling interval, two fortified samples and control samples were removed from the freezer. Subsequently, two control samples of each sampling material were freshly fortified with quinclorac 1 mg/kg to determine the procedural recovery.

For cranberry triplicate untreated field samples were individually fortified with quinclorac at 0.5 mg/kg (10 × method LOQ). At two time intervals three fortified and one control samples freshly fortified with 0.5 mg/kg were prepared to test procedural recovery.

The analytical method A8902 was used to determine quinclorac total in all matrixes. The samples (10g) were soaked in a 0.1N sodium hydroxide solution and extracted with acetone. Samples were then acidified, extracted with dichloromethane and derivatized with diazomethane. Quantification of samples was done using a calibration curve for quinclorac. The LOQ was 0.05 mg/kg.

The storage stability of quinclorac and the metabolite BH514-Me (quinclorac methyl ester) in rape seed (seed, meal and oil) up to 671 days was investigated by Saha, M (2013, BASF/7000581).

Samples from a field trial of homogenized seed, meal and oil were individually fortified with 1 mg/kg quinclorac and quinclorac methyl ester respectively and stored frozen. At each sampling interval two fortified samples and three control samples were removed from the freezer. Two of the control samples were fortified with 1.0 mg/kg each analyte. The modified versions of analytical methods D9708/1 and D9806 were used to determine quinclorac and quinclorac methyl ester.

Residues of parent quinclorac in/on seed and meal samples (10 g each) were extracted were soaked in a 0.1N sodium hydroxide solution and extracted with acetone. Samples were then acidified, extracted with dichloromethane.

Residues of quinclorac methyl ester in/on seed and meal samples (10 g each) were extracted with acetone partitioned with dichloromethane/methanol and water. Quantification was performed using external standards.

The residues were analysed by LC-MS/MS. The MS/MS detection in the positive ionization mode was used to monitor ion transition from m/z 242-160.8 for quinclorac and m/z 256 to 224 for quinclorac methyl ester. The LOQ was 0.05 mg/kg

In the following tables the recovered residues in stored samples are summarized

Table 48 Storage stability of quinclorac in plant commodities fortified at level of 1 mg/kg

Matrix	Storage period months	Procedural recovery mg/kg**	Residues remaining mg/kg**
Rice grain	0	0.87	0.87
	8	0.79	0.76
	19	0.93	0.79
	38	0.92	0.75
Rice straw	0	0.86	0.86

Matrix	Storage period months	Procedural recovery mg/kg**	Residues remaining mg/kg**
	8	0.91	0.77
	19	1.07	0.98
	38	1.01	0.90
Sorghum forage	0	0.98	0.98
	25	0.75	0.91
	38	0.84	0.85
Sorghum hay	0	1.09	1.09
	25	0.75	0.89
	38	0.92	0.90
Sorghum grain	0	0.91	0.91
	25	0.86	0.94
	38	0.97	0.97
Sorghum silage	0	1.04	1.04
	25	0.95	0.79
	38	0.90	0.84
Sorghum fodder	0	1.01	1.01
	25	0.90	0.80
	38	0.86	0.84
Wheat grain	0		-
	6	0.83	0.82
	13	74	0.86
	26	85	0.9
cranberry fruit	8	73	75
	14	90	93

* Values are the average from duplicate or triplicate analyses

** days

*** only one replicate

Table 49 Storage stability of quinclorac and quinclorac methyl ester in rape seed commodities fortified at level of 0.5 mg/kg

Matrix	Storage period days	Procedural recovery %	Residues remaining %
Quinclorac			
Seed	0	87	86
	31	92	88
	94	76	86
	185	95	92
	377	77*	78
	397	76	96
	669	87	77
Meal	0	84	85
	31	96	94
	94	73	75
	185	84	89
	377	107	92
	398	71	81
	669	70	92
Oil	0	98	93
	34		84
	95	76	69
	186	67	50
	390	69	81
	671	97	98
Quinclorac methyl ester			
Seed	0	89	95
	31	84	73
	94	82	65
	185	86	73
	384	82	74

Matrix	Storage period days	Procedural recovery %	Residues remaining %
	668	72	77
Meal	0	100	94
	31	96	86
	94	95	79
	185	94	86
	384	96	93
	668	89	81
Oil	0		80
	31	74	78
	94	73	71
	185	82	82
	384	89	76
	668	76	75

* Values are the average from duplicate analyses

Animal matrices

For animal matrices no procedural recovery studies (with fortified samples) for storage stability were provided. The maximum storage time for hen was eggs were 90 days and tissue 74 days and for lactating goat milk 31 days, subcutaneous fat 58 days, peritoneal fat 56 days and muscle 51 days.

USE PATTERN

Quinclorac is registered for uses in berries and other small fruits stalk and stem vegetables, cereal grains and rape seed in a number of countries. Information on GAP with supporting labels from Canada and USA was provided to the Meeting. Quinclorac is a systemic herbicide with uptake through roots and foliage and used to control annual grass and broadleaf weeds. Its mode of action is overstimulation of growth resulting in the rupture of the cell membranes.

Table 50 Registered uses quinclorac from labels provided.

Crop	Country	Application details					Comments
		Method	Rate; kg ai/ha min.-max. (max. kg ai/ha /season)	Crop growth stage at last treatment	No (interval in days)	PHI	Restrictions
Berries and other small fruits							
Cranberry	USA* Quinstar 4L	ground spray post emergent	0.24-0.48 (0.48)		1-2 (30)	60	Do not allow livestock to graze in treated areas
Stalk and stem vegetables							
Rhubarb	USA* Quinstar 4L	ground spray	0.35-0.7 (0.7)		1-2 (30)	30	Do not allow livestock to graze in treated areas
Cereal grains							
Rice	USA* Quinstar 4L	aerial or ground spray Soil: to soil surface pre-planting or pre-emergent (dryland rice) Foliar: after 2-leaf stage (but before heading) on	0.29-0.54 (0.54)	Do not apply to rice that is heading Rice must be in at least 2-leaf stage.	1	40	Do not plant any crop other than rice for a period of 309 days following application State-specific restrictions in Arkansas. Do not use in California or

Crop	Country	Application details					Comments
		Method	Rate; kg ai/ha min.-max. (max. kg ai/ha /season)	Crop growth stage at last treatment	No (interval in days)	PHI	Restrictions
		dryland and water seeded/paddy					Florida. Can be used in paddy rice post emergently as long as the water depth is reduced to expose the grass and/or broadleaf weeds.
Wheat (spring and durum)	Canada** Accord DF	ground spray post-emergent	0.135-0.165 (0.165)	1-5 leaf	1	77	Do not graze the treated wheat or barley or cut for hay within 77 days of application
Spring barely	Canada ** Accord DF		0.135	1-4 leaf (prior to tillering)	1	80	
Wheat	USA* Facet L	ground spray, air application in certain states pre-plant	(0.29)	pre-plant	1	-	
Wheat	USA* Quinstar 4L	ground spray, air application in certain states pre-plant application	(0.29)	pre-plant	1	-	Do not feed forages, hay, silage or straw to livestock. Do not apply in ID, MT, NV, OR, UT, WA or WY
Sorghum	USA* Quinstar 4L	aerial or ground spray pre-plant	0.29	pre-plant	1	-	Quinclorac can be applied both pre and post emergently as long as the seasonal maximum amount of 0.78 kg ai/ha is not exceeded.
		post-emergent	0.56	Up to 30 cm tall stage	1		
Sorghum	USA* Facet L	aerial or ground spray pre-plant	0.29	pre-plant	1	-	Quinclorac can be used both pre and post emergently as long as the seasonal maximum amount is not exceeded.
		post-emergent	0.3-0.42	Up to 30 cm tall	1		
Oilseed rape							
Rape seed	Canada** Accord DF	ground spray post-emergent	0.135	2-6 leaf stage	1	60	Only grain and meal can be fed to livestock. Do not graze or feed other portions of the treated rape seed to livestock

*SL (liquid flowable)
** DF (dry flowable)

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on supervised field trials involving quinclorac for the following crops.

Group	Crop commodity	Portion of commodity to which MRL apply	Countries	Table No
FB, Berries and other small fruits	Cranberry	Whole commodity after removal of caps and stems	USA	51
VS, Stalk and stem vegetables	Rhubarb	Whole commodity after removal of obviously decomposed or withered leaves	USA	52
GC, Cereal grain	Rice	Whole commodity	USA	53-55
	Wheat	Whole commodity	Canada, USA	56-58
	Sorghum	Whole commodity	USA	59-60
SO, Rapeseed	Canola	Whole commodity	Canada, USA	61

Conditions of the supervised residue trials were generally well reported in detailed field reports. 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 levels are reported as measured, when residues were not detected they are shown as below the LOQ (e.g. < 0.01 mg/kg). Residue data are recorded unadjusted for % recovery.

Laboratory reports included method validation including batch recoveries with spiking at residue levels similar to those occurring in samples from supervised trials. Data of analysis or duration of residue samples storage were also provided. 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.

Cranberry

To determine magnitude of residue of quinclorac in cranberry five supervised field trials were conducted in USA (Massachusetts Wisconsin and Oregon). Quinclorac was applied as two post-emergence ground broadcast applications each at 0.28 kg ai/ha using a SL formulation. All applications contained crop oil concentrate as spray adjuvant.

Duplicate cranberry fruit samples were collected and stored frozen (< -27 °C) until homogenization. Frozen samples were processed in presence of dry ice. Upon grinding, all samples were subsampled. Samples were analysed for quinclorac using GC/EDC detection. The LOQ was of 0.05 mg/kg and average recovery, 86 ± 14% (n=14).

The method validation recoveries of quinclorac at 0.05, 0.5, 0 5 mg/kg was 86 ± 14% (n=12). Concurrent recoveries ranged from 72% to 104% (average 83 ± 13 (n=8). The limit of storage stability for quinclorac in rhubarb petioles were 385 days. The maximum storage time for samples (from sampling to extraction) was 334 days. The storage period is covered by the storage stability studies (385 days).

Results from residues in cranberry fruit are presented in the table below.

Table 51 Residues of quinclorac residue in cranberry fruit following two post-emergence foliar broadcast applications with an SL formulation.

Location	Application			Residues			Trial
Year, (variety)	Total Rate, (kg ai/ha)	Growth stage	PHI (days)	Matrix	quinclorac (mg/kg)	mean (mg/kg)	Trial comment
USA Plymouth County, MA, 2008 (Stevens) Stevens 1	2 x 0.27	Bloom July 5, Fruit set July 31	59	Mature cranberries	0.50, 0.60	0.55	08000.08-MA01 2010/7018348
USA Wareham, MA 2008 (Early Blacks) Early blacks 1	2 × 0.28	Bloom July 5, Fruit set July 31	59	Mature cranberries	0.16, 0.20	0.18	08000.08-MA03 2010/7018348
USA Warrens, WI 2008 (Stevens) 5	2 × 0.28	Bloom, July 7 Fruiting August 4	57	Mature cranberries	0.17, 0.16	0.17	08000.08-WI01 2010/7018348
USA Warrens, WI 2008 (Ben Lear) 5	2 × 0.28	Bloom, July 7 Fruiting August 4	57	Mature cranberries	0.16, 0.15	0.16	08000.08-WI02 2010/7018348
USA Langlois, OR 2008 (Pilgrims) 12	2 × 0.29	End of bloom, July 1 Green fruit August 6	62	Mature cranberries	0.66, 0.68	0.67	08000.08-OR10 2010/7018348

Rhubarb

To determine magnitude of residue of quinclorac in rhubarb four field trials were conducted in USA (Michigan and Oregon). Quinclorac was applied as two post-emergence ground broadcast applications each at 0.42 kg ai/ha and a ~ 30 days interval. All applications contained crop oil concentrate as spray adjuvant**.**

Duplicate samples of rhubarb petioles were collected and stored frozen (< -15°C) until homogenization. After processing the samples were returned to frozen storage until analysis within 357 days. Samples were analysed for quinclorac according to method A8902 using GC/EDC detection. Method validation recoveries of quinclorac at 0.05, 0.5, 0 5 mg/kg were in 88 ± 12% (n=9). Concurrent recoveries ranged from 80% to 117% (average 98 ± 9 (n 13). The maximum storage time of samples (from sampling to extraction) was 357 days. The storage period is covered by the storage stability studies (385 days).

Results from residues in rhubarb fruit are presented in the table below.

Table 52 Residues of quinclorac in rhubarb following two post-emergence broadcast applications with a SL formulation

Location	Application			Residues			Trial
Trial Identification Year, variety	Total Rate, kg ai/ha	Growth stage	PHI (days)	Matrix	quinclorac (mg/kg)	mean (mg/kg)	report comment
USA Holt, MI 2009 (German wine)	0.42+0.43	Vegetative April 22 Blooming May 26	29	Rhubarb	0.18, 0.23	0.21	10135.09-M108 2010/7018328
USA Hillsboro, OR 2009 (Crimson red)	0.43+0.44	Late dormancy March 18 Vegetative April 15	33	Rhubarb	0.20, 0.15	0.18	10135.09-OR10 2010/7018328
USA Canby, OR 2009 (Red Crimson)	0.43+0.40	Coming out of dormancy March 14 Vegetative April 15	32	Rhubarb	0.05, 0.05, 0.10, 0.13, 0.14, 0.13, 0.14	0.11	10135.09-OR11 2010/7018328
USA Canby, OR 2009 (Red Crimson)	0.46 + 0.43	Spring growth beginning March 19 Vegetative April 17	33	Rhubarb	0.08, 0.06	0.07	10135.09-OR112 2010/7018328

Rice

Results from supervised trials from USA on rice were provided to the Meeting.

To compare aerial and ground application a total of nine field trials were performed during growing season 1988 in USA (California, Texas, Arkansas, Louisiana and Mississippi) using a WP formulation. In all trials except two conducted in California, quinclorac was applied to a non-flooded rice field.

Single rice grain samples were homogenized and straw samples were pre-cut, ground and stored at -5 °C until analysis within 4–5 months. Samples were analysed for quinclorac by method A8902 using GC/ECD detection. The LOQ was 0.05 mg/kg and the average recovery were 88±14% (n=21) for grain and 94±15% (n=21) for the straw.

Table 53 Residues of quinclorac in rice grain and straw following aerial and ground broadcast application with a WP formulation

Location	Application						Residues				Trial no.
Year (variety)	kg ai/hl	water L/ha	kg ai/ha	treatment	no	BBCH	matrix	PHI	quinclorac (mg/kg)	mean (mg/kg)	Reference comment
USA (CA) 1989, (M202)	0.581	96	0.560	aerial	1	41-43 (booting)	grain	77	1.5	-	88045 BASF 1989/5007
							straw	77	2.6	-	
USA (CA) 1989, (M202)	0.565	99	0.560	ground	1	nr	grain	77	1.9	-	88045 BASF 1989/5007 Adjuvant
							straw	77	3.2	-	
USA (CA)	0.594	132	0.784	ground	1	n.r.	grain	77	4.3	-	88045 BASF
							straw	77	11.1	-	

Location	Application						Residues				Trial no.
Year (variety)	kg ai/hl	water L/ha	kg ai/ha	treatment	no	BBCH	matrix	PHI	quinclorac (mg/kg)	mean (mg/kg)	Reference comment
1989, (M202)											1989/5007 Adjuvant
USA (CA) 1989, (KRM2)	0.581	96	0.560	aerial	1	n.r.	grain	77	1.6	-	88046 BASF 1989/5007
							straw	77	4.0	-	
USA (CA) 1989, (KRM2)	0.301	224	0.672	ground	1	n.r.	grain	77	2.2	-	88046 BASF 1989/5007 adjuvant
							straw	77	4.0 6.7	5.4	
USA (TX) 1989 (Gulf Mont)	1.197	47	0.560	aerial	1	n.r.	grain	77	< 0.05	-	88047 BASF 1989/5007
							straw	77	0.120	-	
USA (TX) 1989 (Gulf Mont)	0.454	123	0.560	ground	1	n.r.	grain	77	0.06, 0.07	0.065	88047 BASF 1989/5007 adjuvant
							straw	77	0.10, 0.47	0.285	
USA (TX) 1989 (Lemont)	1.197	47	0.560	aerial	1	n.r.	grain	76 174	0.12 < 0.05	- -	88048 BASF 1989/5007
							straw	76 174	0.18 < 0.05	- -	
USA (TX) 1989 Lemont	0.599	94	0.560	ground	1	n.r.	grain	76 174	0.08, 0.09 < 0.05	0.085 -	88048 BASF 1989/5007 adjuvant
							straw	76 174	0.18, 0.23 < 0.05	0.20 -	
USA (AR) 1989 (Mars)	0.599	94	0.560	aerial	1	n.r.	grain	76	< 0.05	-	88049 BASF 1989/5007
							straw	76	0.14	-	
USA (AR) 1989 (Mars)	0.299	187	0.560	ground	1	n.r.	grain	76	0.09, 0.10	0.095	88049 BASF 1989/5007 adjuvant
							straw	76	0.39, 0.46	0.425	
USA (AR) 1989 (Lemon)	0.599	94	0.560	aerial	1	n.r.	grain	80	0.12	-	88050 BASF 1989/5007
							straw	80	0.23	-	
USA (AR) 1989 (Lemont)	0.599	94	0.560	ground	1	n.r.	grain	80	0.22	-	88050 BASF 1989/5007 adjuvant
							straw	80	0.23, 0.24	0.235	
USA (LA) 1989 (Lemon)	0.599	94	0.560	aerial	1	n.r.	grain	98	< 0.05	-	88051 BASF 1989/5007
							straw	98	0.08	-	

Location	Application						Residues				Trial no.
Year (variety)	kg ai/hl	water L/ha	kg ai/ha	treatment	no	BBCH	matrix	PHI	quinclorac (mg/kg)	mean (mg/kg)	Reference comment
USA (LA), Newelton 1989 (Lemont)	0.599	94	0.560	ground	1	n.r.	grain	98	< 0.05, 0.08	0.065	88051 BASF 1989/5007 adjuvant
							straw	98	0.05, 0.11	0.08	
USA (LA), Midland 1989 (Lemont)	1.197	47	0.560	aerial	1	n.r.	grain	76	0.08	-	88052 BASF 1989/5007
							straw	76	0.03	-	
USA (LA), Midland 1989 (Lemont)	0.440	127	0.560	ground	1	n.r.	grain	76	< 0.05, 0.15	0.1	88052 BASF 1989/5007 adjuvant
							straw	76	0.09, 0.54	0.31	
USA (MS), 1989 (Lemont)	1.197	47	0.560	aerial	1	n.r.	grain	78	< 0.05		88053 BASF 1989/5007
							straw	78	< 0.05		
USA (MS), 1989 (Lemont)	0.599	94	0.560	ground	1	n.r.	grain	78	0.06, 0.16	0.11	88053 BASF 1989/5007 adjuvant
							straw	78	< 0.05, 0.11	0.08	

n.r. = not reported

PHI = Pre-harvest interval

To determine magnitude of residues of quinclorac in rice, field trials were performed during growing season 1996 in USA (Texas, Arkansas, Louisiana, Mississippi, Missouri, and Texas) using a DF formulation. In all trials quinclorac was applied as a single post-emergence ground spray to flooded (paddy field) rice fields. All applications contained crop oil concentrate as spray adjuvant.

Duplicate samples of rice grain and straw were sampled and kept at < -10 °C until they were homogenized at room temperature (grain) and in dry ice (straw) and then returned to frozen storage until analysis within 5 months. The storage period is covered by the storage stability studies (38 months). Samples were analysed for quinclorac by method A8902 using GC/EDC detection. The LOQ was 0.05 mg/kg and the average recoveries were 82±13% (n=26) for grain and 84±14% (n=24) for the straw.

Results from residues in rice grain are presented in Table 54 and from straw in Table 55.

Table 54 Residues of quinclorac in rice grain following broadcast ground application with a DF formulation

Location	Application					Residues				Trial
Year (variety)	kg ai/hl	water L/ha	kg ai/ha	no	Growth stage	matrix	PHI	quinclorac (mg/kg)	mean (mg/kg)	Reference
USA (MS), Washington county 1996 (Lemont)	0.599	94	0.560	1	Booting	grain	34	0.37, 0.44	0.40	96152 BASF/97/5051
						grain	37	0.35, 0.39	0.37	
						grain	40	0.40, 0.37	0.38	
						grain	43	0.35, 0.34	0.35	
						grain	46	0.43, 0.31	0.37	

Location	Application					Residues				Trial
Year (variety)	kg ai/hl	water L/ha	kg ai/ha	no	Growth stage	matrix	PHI	quinclorac (mg/kg)	mean (mg/kg)	Reference
USA (MS) Bolivar county, 1996 (Lemont)	0.570	94	0.549	1	Booting	grain	40	1.7, 1.9	1.8	96154 BASF/97/5051
USA (AR) Crittenden county 1996 (Bengal)	0.593	95	0.560	1	heading	grain	34	4,05, 3.20	3.63	96155 BASF/97/5051
						grain	37	4.24, 4.45	4.35	
						grain	40	3.74, 3.84	3.79	
						grain	43	2.97, 3.58	3.28	
						grain	46	3.67, 4.27	3.97	
USA (AR) Crittenden county 1996 (Bengal)	0.604	96	0.582	1	heading	grain	40	0.325, 0.366	0.346	96156 BASF/97/5051
USA (AR) St Francis county 1996 (Kaybonnet)	0.605	95	0.571	1	Early booting	grain	40	0.480, 0.631	0.556	96157 BASF/97/5051
USA (LA) St Laundry parish 1996 (Cypress)	0.576	97	0.560	1	Early booting	grain	40	0.710, 0.822	0.766	96158 BASF/97/5051
USA (LA) Evangeline parish 1996 Cypress	0.565	99	0.560	1	Early booting	grain	40	0.551, 0.429	0.490	96159 BASF/97/5051
USA (LA) Jeff Davis Parish 1996 (Cypress)	0.571	100	0.571	1	Early booting	grain	41	0.271, 0.252	0.262	96160 BASF/97/5051
USA (LA) St Laundry county 1996 (Bengal)	0.593	94	0.560	1	Early booting	grain	40	0.912, 0.662	0.787	96161 BASF/97/5051
USA (LA) St Laundry county 1996 (Maybell)	0.599	95	0.571	1	Early booting	grain	40	1,07, 1.07	1.07	96162 BASF/97/5051
USA (M0) Permiscot 1996 (Lemont)	0.549	102	0.560	1	Booting	grain	40	0.137, 0.083	0.110	96163 BASF/97/5051
USA (M0) Stoddard county 1996 Cypress	0.604	96	0.582	1	Heading	grain	40	1.96, 1.52	1.74	96164 BASF/97/5051
USA (TX) Walter county 1996 (Cypress)	0.571	102	0.582	1	Full boot stage	grain	40	0.675, 0.743	0.709	96166 BASF/97/5051

PHI = Pre-harvest interval

Trial 96165 is missing

To determine the influence of the formulation on the residues in rice grain five supervised trials were conducted during the growing season 2009 in USA (Arkansas and Louisiana). Each trial consisted of side-by-side tests comparing the dry flowable (DF) and the soluble liquid (SL). The rice was irrigated according to typical commercial practices for paddy-grown rice

Duplicate samples were sampled and maintained frozen until analysis within 7.7 months. Samples were analysed for quinclorac method D9708/1 using LC- MS/MS. The LOQ was 0.05 mg/kg and the average recovery was 94% (n=2).

Table 55 Residues of parent quinclorac in rice grain following ground foliar application with a DF and a SL formulation

Location	Application					Residues				Trial
Year (variety)	formulation	water L/ha	kg ai/ha	no	BBCH	matrix	PHI	quinclorac (mg/kg)	mean (mg/kg)	Reference
USA (LA) Rapides 2009 (Cheniere)	DF	214 217	0.277 0.277	2	88-89	grain	110	< 0.05, < 0.05	< 0.05	RO90420 BASF/2013/7000580
USA (AR) Crittenden 2009 (Wells)	DF	189 190	0.280 0.289	2	88-89	grain	96	0.09, 0.08	0.085	RO90421 BASF/2013/7000580
USA (LA) Rapides 2009 (Cheniere)	SL	217 219	0.281 0.281	2	88-89	grain	110	< 0.05, < 0.05	< 0.05	RO90420 BASF/2013/7000580
USA (AR) Crittenden 2009 (Wells)	SL	189 190	0.279 0.281	2	88-89	grain	96	0.12, 0.11	0.115	RO90421 BASF/2013/7000580

PHI = Pre-harvest interval

Wheat

Results from supervised trials from Canada and USA on wheat were provided to the Meeting.

To determine magnitude of residues of quinclorac in spring wheat field trials were performed during growing season 1998 in Canada (Alberta, Manitoba, Saskatchewan). At each trial four different treatments were applied each including quinclorac. One treatment was only quinclorac and the other three were with quinclorac plus one or more tank mix partner. In all trials quinclorac was applied as a single broadcast post-emergence spray 75 days prior to harvest. Forage was sampled 9 to 16 days after treatment

Duplicate samples of wheat forage, grain and straw were sampled and stored frozen (<-15 °C) until they were homogenized and until analysis within 7 months. The storage period is covered by the storage stability studies (26 months). Samples were analysed for quinclorac by method A8902 using GC/ECD detection. The LOQ was 0.05 mg/kg and the average recovery were 85±14% (n=8) for forage, 80±9% (n=6) for grain and 103±1 7% (n=7) for the straw.

Results from residues in wheat grain are presented in Table 56 and from forage and straw in Tables 64-5.

Table 56 Residues of quinclorac) in spring wheat grain following broadcast foliar application with a DF formulation

Localisation	Treatment			Residues				Trial
Year (variety)	Treatment kg ai/ha	no	BBCH	matrix	PHI	quinclorac	mean	Reference

Localisation	Treatment			Residues				Trial
Year (variety)	Treatment kg ai/ha	no	BBCH	matrix	PHI	quinclorac	mean	Reference
Canada Saskatchewan 1995 (Katepwa)	quinclorac 0.123	1	22-29	grain	75	0.136, 0.252	0.194	95106 BASF/96/5103
Canada Saskatchewan 1995 (Katepwa)	quinclorac; 0.125, imazethabenz; 2,4-D	1	22-29	grain	75	0.10, 0.15	0.125	95106 BASF/96/5103
Canada Saskatchewan 1995 (Katepwa)	quinclorac; 0.125, imazethabenz; Tribenuron/thifensulfuron;	1	22-29	grain	75	0.14, 0.17	0.155	95106 BASF/96/5103
Canada Saskachewan 1995 (Katepwa)	quinclorac 0.125 Bromonynil/MCPA	1	22-29	grain	75	0.18, 0.19	0.185	95106 BASF/96/5103
Canada Nisku, Alberta 1995 (Katepwa)	quinclorac 0.122	1	22-29	grain	75	0.088, 0.096	0.092	95107 BASF/96/5103
Canada Nisku, Alberta 1995 (Katepwa)	quinclorac; 0.125, imazethabenz; 2,4-D	1	22-29	grain	75	0.077 0.049	0.063	95107 BASF/96/5103
Canada Nisku, Alberta 1995 (Katepwa)	quinclorac; 0.125, imazethabenz; Tribenuron/thifensulfuron	1	22-29	grain	75	0.120 0.107	0.114	95107 BASF/96/5103
Canada Nisku, Alberta 1995 (Katepwa)	quinclorac 0.125 Bromonynil/MCPA	1	22-29	grain	75	0.161 0.143	0.152	95107 BASF/96/5103

PHI = Pre-harvest interval

To determine magnitude of residues of quinclorac in spring wheat field trials were performed during growing season 1998 in USA (Minnesota, Montana, North Dakota, Oregon, South Dakota and Washington) using a DF formulation. In all trials quinclorac was applied as a single broadcast post-emergence spray 70–74 days prior to harvest. All applications contained crop oil concentrate as spray adjuvant.

Samples of wheat forage (duplicate), grain (single) and straw (triplicate) were sampled and stored frozen (< -10 °C) until they were homogenized and until analysis within 27 months. Samples were analysed for quinclorac by method A8902 using ECD detection. The LOQ was 0.05 mg/kg and the average recoveries were 78±12% (n=15) for forage, 83±11% (n=12) for grain and 75±7% (n=12) for the straw.

Results from residues in wheat grain are presented in Table 57 and from forage and straw in Tables 64-5.

Table 57 Residues of quinclorac in spring wheat grain following broadcast foliar application with a DF formulation

Location	Application			Residues			Reference
Year (variety)	kg ai/ha	no	Growth stage BBCH	matrix	PHI	quinclorac (mg/kg)	Reference
USA (MN) 1998 Pioneer	0.56	1	20-29 (tillering)	grain	71	0.22	90056 BASF/1998/5104
USA (MN) 1998 Stoa	0.56	1	20-29 (tillering)	grain	70	0.14	90057 BASF/1998/5104
USA Grand Forks (ND) 1998 Marshall	0.56	1	20-29 (tillering)	grain	70	0.26	90058 BASF/1998/5104
USA Steele (ND) 1998 Marshall	0.56	1	20-29 (tillering)	grain	70	0.17	90059 BASF/1998/5104
USA Grand Forks (ND) 1998 Marshall	0.56	1	20-29 (tillering)	grain	73	0.13	90060 BASF/1998/5104
USA* Minehaha (SD) 1998 Guard	0.56	1	20-29 (Tillering)	grain	70	0.49	90061 BASF/1998/5104
USA* Minehaha (SD) 1998 Guard	0.56	1	20-29 (tillering)	grain	71	0.76	90062 BASF/1998/5104
USA (MT) 1998 926	0.56	1	20-29 (tillering)	grain	82	0.80	90063 BASF/1998/5104
USA (MT) 1998 Nevanna	0.56	1	20-29 (tillering)	grain	72	0.53	90064 BASF/1998/5104
USA (ID) 1998 Pondera	0.56	1	20-29 (tillering)	grain	73	0.45	90065 BASF/1998/5104
USA (WA) 1998 Yecora Rojo	0.56	1	51 (beginning of heading)	grain	71	2.86	90066 BASF/1998/5104
USA (OR) 1998 Ovens	0.56	1	20-29 (tillering)	grain	74	0.73	90067 BASF/1998/5104

PHI = pre harvest interval

*Different planting dates, independent from each other

scaling factor = 0.22 (0.125/0.56 = 0.22)

BBCH 51= Inflorescence emergence

To determine magnitude of residues of quinclorac in spring wheat field trials were performed during growing season 1998 in Canada (Alberta, Manitoba, Saskatchewan) using a DF formulation. In all trials quinclorac was applied as a single broadcast post-emergence spray 90-76 days prior to harvest for grain and straw and . All applications contained crop oil concentrate as spray adjuvant.

Samples (four replicates) of wheat forage (duplicate), grain (single) and straw (triplicate) were sampled and stored frozen (< -5 °C) until they were homogenized and analysed within 8 months. Samples were analysed for quinclorac by method A8902 using GC/ECD detection. . The LOQ was 0.05 mg/kg and the average recoveries were 89±15% (n=17) for forage, 88±18% (n=24) for grain and 83±9% (n=18) for the straw.

Results from residues in wheat grain are presented in Table 58 and from forage and straw in Table 63

Table 58 Residues of total quinclorac in spring wheat grain following broadcast foliar application with a DF formulation

Location	Application			Residues				Trial
Year (variety)	kg ai/ha	no	Growth stage BBCH	matrix	PHI	Total quinclorac (mg/kg)	mean mg/kg	Reference
Canada Minto, Manitoba 1994 Katepwa	0.126	1	23 (three tillers)	grain	90	4x < 0.05	< 0.05	94108 BASF/1995-7004167
Canada Aberdeen, Saskatchewan 1994 Katepwa	0.126	1	23 (three tillers)	grain	77	0.14, 0.16, 0.16, 0.17	0.16	94109 BASF/1995-7004167
Canada Portage, Manitoba 1994 Katepwa	0.126	1	23 (three tillers)	grain	76	3 x < 0.05, 0.5	0.05	94110 BASF/1995-7004167
Canada Swift current Saskatchewan 1994 Katepwa	0.126	1	23 (three tillers)	grain	76	0.10, 0.07, 0.07, 0.12	0.09	94111 BASF/1995-7004167

Zadoks 23-25; tillering with 3-5 tillers present

Sorghum

Results from supervised trials from USA on sorghum were provided to the Meeting.

To determine magnitude of residues of quinclorac in sorghum field trials were performed during growing season 1995 in USA (Kansas, Nebraska, Oklahoma and Texas) using a DF formulation. In all trials quinclorac was applied as a single broadcast post-emergence spray. All applications contained crop oil concentrate as spray adjuvant.

Duplicate samples of sorghum grain and straw were sampled and kept at < -10 °C until they were homogenized in room temperature (grain) and in dry ice (straw) and then returned to frozen storage until analysis within 12 months. The storage period is covered by the storage stability studies (38 months). Samples were analysed for quinclorac by method A8902 using GC/ECD detection. The LOQ was 0.05 mg/kg and the average recoveries were for trials 94200-94203; 79±17% (n=10) for forage, 81±10.1% (n=10) for grain and 85±11% (n=7) for the fodder and for trials 9766-97270; 84±21% (n=3) for forage, 96±9.4% (n=3) for grain and 93±21.5% (n=3) for the fodder.

Results from residues in sorghum grain are presented in Table 59 and from forage and straw in Table 66.

Table 59 Residues of quinclorac in sorghum grain following broadcast application with a DF formulation

Location	Application			Residues				Trial
Year (variety)	kg ai/ha	no	Growth stage	matrix	PHI	quinclorac (mg/kg)	mean (mg/kg)	Reference
USA (KS) 1995 (Hoegemeyer S-688)	0.29	1	6-leaf stage	grain	98	0.06, 0.06	0.06	94200 BASF/1996/5136
				grain	103	0.10, 0.08	0.09	
				grain	108	0.07, 0.06	0.065	
				grain	118	0.07, 0.07	0.07	
USA (NE) 1995 (NK 1210)	0.29	1	6-leaf stage	grain	89	< 0.05, < 0.05	< 0.05	94201 BASF/1996/5136
USA (OK) 1995 (Cargill 630)	0.29	1	5-7 leaves, mainly 6	grain	81	0.278, 0.242	0.26	94202 BASF/1996/5136
USA (KS) 1995 (DK 705)	0.29	1	6-leaf stage	grain	93	0.231, 0.234	0.233	94203 BASF/1996/5136
USA (NE) York county 1997 (NK 11210)	0.28	1	6-leaf stage	grain	86	< 0.05, < 0.05	< 0.05	97266 BASF/1998/5081
USA (NE) Hall county 1997 (NK 11210)	0.28	1	6-leaf stage	grain	87	< 0.05, < 0.05	< 0.05	97267 BASF/1998/5081
USA (CO) 1997 (Cargill 577)	0.29	1	6-leaf stage	grain	91	0.08, 0.08	0.08	97268 BASF/1998/5081
USA (NE) 1997 (Pioneer 8699)	0.28	1	6-leaf stage	grain	95	0.31, 0.28	0.30	97269 BASF/1998/5081
USA (NE) 1997 (F270E)	0.28	1	6-leaf stage	grain	93	0.49, 0.51	0.50	97270 BASF/1998/5081

To determine the influence of the formulation on the residues in sorghum grain five supervised trials were conducted during the growing season 2009 in USA (Arkansas and Louisiana). Each trial consisted of side-by-side tests comparing the dry flowable (DF) and the soluble liquid (SL). The rice was irrigated according to typical commercial practices for paddy-grown rice

Duplicate samples were sampled and maintained frozen until analysis within 7.7 months. Samples were analysed for quinclorac using method D9708/1 using LC- MS/MS. LOQ was 0.05 mg/kg and the average recovery was 94% (n=2).

Table 60 Residues of quinclorac in sorghum grain following ground foliar application with a DF and a SL formulation

Location	Application	Residues	Trial

Year	formulation	water L/ha	kg ai/ha	no	BBCH	matrix	PHI	quinclorac (mg/kg)	Reference
USA (LA) Rapid Parish 2009	DF	205	0.413	1	4-5 leaf	grain	97	< 0.05	RO90424 BASF/2013/7000580
USA (ND) Cass 2009	DF	210	0.429	1	BBCH 15	grain	113	< 0.05	RO90425 BASF/2013/7000580
USA (LA) Rapid Parish 2009	SL	205	0.459	1	4-5 leaf	grain	97	< 0.05	RO90424 BASF/2013/7000580
USA (ND) Cass 2009	SL	211	0.477	1	BBCH 15	grain	113	< 0.05	RO90425 BASF/2013/7000580

PHI = Pre-harvest interval

Rape seed

To determine magnitude of residue of quinclorac in rape seed seventeen supervised field trials were conducted in Canada (16) and USA (1). Quinclorac was applied as a single post-emergence broadcast application.

Duplicate rape seed samples were collected and stored frozen (< -10 °C) until homogenization. After homogenization samples were returned to frozen storage until analysis within 6 months for quinclorac and 12 months for quinclorac methyl ester. The storage period is covered by the storage stability studies (22 months) for both analytes. Samples were analysed for quinclorac according to method D9708/1 with LOQ of 0.05 mg/kg and average recovery of 75±13% (n=13) and for quinclorac methyl ester according to method D9806 with LOQ of 0.05 mg/kg and average recovery of 92±15% (n=9).

Results from residues in rapeseed (canola) grain are presented in table below.

Table 61 Residues in rape seed following ground broadcast application with quinclorac (DF formulation)

Location		Application			Residues				Trial
Year (variety)	Total Rate,	no	Growth stage	Matrix	PHI (days)	Quinclorac residues (mg/kg)	Methyl ester residues (mg/kg)	Mean total residues (mg/kg)	Comment
Canada Hines Creek, Alberta, 1997 Reward	(kg ai/ha)	1	6 -leaf stage	seed	60	< 0.05, < 0.05	< 0.05, < 0.05	< 0.10	RCN 97334 1998/5094
Canada Fairview, Alberta, 1997 Reward	0.1	1	6 -leaf stage	seed	53 60 67 74	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.10 < 0.10 < 0.10 < 0.10	RCN 97335 1998/5174
Canada, Lacombe, Alberta, 1997	0.1	1	7 leaves and bolting	seed	60	0.10, 0.09	0.19 0.17	0.28	RCN 97336 1998/5094

Location		Application			Residues				Trial
Year (variety)	Total Rate,	no	Growth stage	Matrix	PHI (days)	Quinclorac residues (mg/kg)	Methyl ester residues (mg/kg)	Mean total residues (mg/kg)	Comment
Quantum									
Canada Stettler, Alberta 1997 Quantum	0.1	1	7-8 leaves and bolting	seed	60	0.18, 0.22	0.09, 0.08	0.29	RCN 97337 1998/5094
Canada Red Deer, Alberta 1997 Hyson 110	0.1	1	5-leaf stage	seed	60	< 0.05, < 0.05	0.12, 0.13	0.18	RCN 97338 1998/5094
Canada, Aberdeen, Saskatchewan 1997 Quantum	0.1	1	3-8 leaves	seed	60	0.14, 0.12	0.06, < 0.05	0.20	RCN 97339 1998/5094
Canada, Rosthern, Saskatchewan 1997 Quantum	0.1	1	3 leaves	seed	60	0.30, 0.18	0.09, 0.09	0.37	RCN 97340 1998/5094
Canada, Wakaw, Saskatchewan 1997 Ebony	0.1	1	43-82 cm tall, 5-10 leaves	seed	60	< 0.05, < 0.05	< 0.05, < 0.05	< 0.10	RCN 97341 1998/5094
Canada, Melfort, Saskatchewa 1997 Quantum	0.1	1	5-10 leaves	seed	60	0.09, 0.08	0.08 0.06	0.16	RCN 97342 1998/5094
Canada, Duck Lake, Saskatchewa 1997 Garrison	0.1	1	6-8 leaves and flowering	seed	60	0.21, 0.25	0.12, 0.10	0.34	RCN 97343 1998/5094
Hague, Saskatchewan Canada, 1997 Garrison	0.1	1	5-7 leaves and flowering	seed	60	0.63, 0.57	0.14, 0.11	0.73	RCN 97344 1998/5094
Canada, Saskatoon, Saskatchewan 1997 Garrison	0.1	1	4-6 leaves	seed	60	0.85 0.86	0.15 0.12	0.99	RCN 97345 1998/5094
Canada, Boussevain, Manitoba 1997 46A05	0.1	1	11 leaves	seed	60	0.24, 0.21	0.23, 0.07	0.38	RCN 97346 1998/5094
Canada Minto, Manitoba 1997 A5471	0.1	1	11 leaves	seed	60	0.15, 0.17	< 0.05 < 0.05	0.21	RCN 97347 1998/5094

Location		Application			Residues				Trial
Year (variety)	Total Rate,	no	Growth stage	Matrix	PHI (days)	Quinclorac residues (mg/kg)	Methyl ester residues (mg/kg)	Mean total residues (mg/kg)	Comment
Canada, Portage La Prairie, Manitoba 1997 46A72	0.1	1	22 leaves and flowering	seed	60	< 0.05, 0.05	0.10 0.10	0.15	RCN 97348 1998/5094
Canada Bagot, Manitoba 1997 Quantum	0.1	1	8-10 leaves, mid flowering	seed	60	0.21, 0.21	0.23, 0.13	0.39	RCN 97349 1998/5094
USA New Rockford (ND,) 1997 Hyola 308	0.1	1	22 leaves, early bloom	seed	53 60 67 74	0.07, < 0.05 < 0.05, 0.06 0.06, 0.05 < 0.05, < 0.05	< 0.05 < 0.05 < 0.05, < 0.05 < 0.05, < 0.05 < 0.05, < 0.05	0.11 0.11 0.11 < 0.10	RCN 97350 1998/5094

RESIDUES IN ANIMAL COMMODITES

Straw, forage, fodder of cereal grains

Table 62 Residues of quinclorac in rice straw following a broadcast ground application with a DF formulation

Year (variety)	kg ai/hl	water L/ha	kg ai/ha	no	Growth stage	matrix	PHI	quinclorac	mean	Reference
USA (MS), 1996 (Lemont)	0.599	94	0.560	1	Booting	straw	34	0.419, 0.258	0.339	96152 BASF/97/5051
						straw	37	0.328, 0.233	0.281	
						straw	40	0.443, 0.275	0.359	
						straw	43	0.357, 0.170	0.259	
						straw	46	0.379, 0.216	0.298	
USA (MS), 1996 (Lemont)	0.570	94	0.549	1	Booting	straw	40	1,74, 1.84	1,79	96154 BASF/97/5051
USA (AR) 1996 (Bengal)	0.593	95	0.560	1	heading	straw	34	1.49, 1.64	1.57	96155 BASF/97/5051
						straw	37	2.37, 1.42	1.90	
						straw	40	1.15, 1.29	1.22	
						straw	43	2.30, 1.74	1.89	
						straw	46	1.25, 1.33	1.29	
USA (AR) 1996 (Bengal)	0.604	96	0.582	1	heading	straw	40	0.107, 0.133	0.120	96156 BASF/97/5051
USA (AR) 1996 (Kaybonnet)	0.605	95	0.571	1	Early booting	straw	40	1.05, 0.865	0.958	96157 BASF/97/5051

Year (variety)	kg ai/hl	water L/ha	kg ai/ha	no	Growth stage	matrix	PHI	quinclorac	mean	Reference
USA (LA) 1996 (Cypress)	0.576	97	0.560	1	Early booting	straw	40	1.23, 1.08	1.16	96158 BASF/97/5051
USA (LA) 1996 (Cypress)	0.565	99	0.560	1	Early booting	straw	40	0.769, 0.622	0.696	96159 BASF/97/5051
USA (LA) 1996 (Cypress)	0.571	100	0.571	1	Early booting	straw	41	1.15, 1.22	1.19	96160 BASF/97/5051
USA (LA) 1996 (Bengal)	0.593	94	0.560	1	Early booting	straw	40	1.56, 0.659	0.11	96161 BASF/97/5051
USA (LA) 1996 (Maybell)	0.599	95	0.571	1	5 cm pancile in the sheat	straw	40	1.35, 1.20	1.28	96162 BASF/97/5051
USA (M0) 1996 (Lemont)	0.549	102	0.560	1	7,6 cm panicle in the sheat	straw	40	0.473, 0.31	0.392	96163 BASF/97/5051
USA (M0) 1996 (Cypress)	0.604	96	0.582	1	heading	straw	40	1.94, 3.54	2.74	96164 BASF/97/5051
USA (TX) 1996 (Cypress)	0.571	102	0.582	1	Full boot stage	straw	40	0.757, 0.927	0.84	96166 BASF/97/5051

PHI = Pre-harvest interval

Table 63 Residues of quinclorac in spring wheat forage and straw following broadcast foliar application with a DF formulation

Location	Application			Residues				Trial
Year (variety)	kg ai/ha	no	Growth stage	matrix	PHI	quinclorac	mean mg/kg	Reference
Canada Minto, Manitoba 1994 Katepwa	0.126	1	23 (three tillers)	forage	21	4x < 0.05	< 0.05	94108 BASF/1995-7004167
				straw	90	4x < 0.05	< 0.05	
Canada Aberdeen, Saskatchewan 1994 Katepwa	0.126	1	23 (three tillers)	forage	24	0.62, 0.51, 0.56, 0.49	0.545	94109 BASF/1995-7004167
				straw	77	0.06, 0.05, 2x< 0.05	0.063	
Canada Portage, Manitoba 1994 Katepwa	0.126	1	22 (two tillers)	forage	15	0.10, 0.13, 0.09, 0.11	0.108	94110 BASF/1995-7004167
				straw	76	4x < 0.05	< 0.05	
Canada Swift current Saskatchewan 1994 Katepwa	0.126	1	24 (four tillers)	forage	23	0.22, 0.19, 0.17, 0.13	0.179	94111 BASF/1995-7004167
				straw	76	4x < 0.05	< 0.05	
Canada Alberta	not done	-	-	no data	no data	no data	no data	94112 BASF/1995-

Location	Application			Residues				Trial
Year (variety)	kg ai/ha	no	Growth stage	matrix	PHI	quinclorac	mean mg/kg	Reference
1994 Katepwa								7004167

Table 64: Residues of quinclorac in spring wheat forage and straw following broadcast foliar application with a DF formulation

Location	Application			Residues				Trial
Year (variety)	Treatment kg ai/ha	no	Growth stage BBCH	matrix	PHI	quinclorac	mean	Reference
Canada Manitoba 1995 Katepwa	quinclorac 0.125	1	21-22 (max two tillers)	forage only	16	0.06, 0.07	0.065	95105 BASF/96/5103
Canada Manitoba 1995 Katepwa	quinclorac; 0.125, imazamethabenz; 2,4-D	1	21-22 (max two tillers)	forage	16	0.06, 0.05	0.06	95105 BASF/96/5103
Canada Manitoba 1995 Katepwa	quinclorac; 0.125, imazamethabenz; Tribenuron/thifensulfuron;	1	21-22 (max two tillers)	forage	16	0.08, 0.07	0.075	95105 BASF/96/5103
Canada Manitoba 1995 Katepwa	quinclorac 0.125 Bromoxynil/MCPA	1	21-22 (max two tillers)	forage	16	0.06, 0.08	0.07	95105 BASF/96/5103
Canada Saskatchewan 1995 Katepwa	quinclorac 0.123	1	Zadock 23-30 20-25 cm high with 4-6 tillers	forage	9	1.8, 1.5	1.7	95106 BASF/96/5103
				straw	75	0.20, 0.17	0.19	
Canada Saskatchewan 1995 Katepwa	quinclorac; 0.125, imazamethabenz; 2,4-D	1	21-22 (max two tillers)	forage	9	1.0, 1.1	1.05	
				straw	75	0.2, 0.17	0.19	
Canada Saskatcoon 1995 Katepwa	quinclorac; 0.125, imazamethabenz; Tribenuron/thifensulfuron;	1	21-22 (max two tillers)	forage	9	1.1, 1.1	1.1	95106 BASF/96/5103
				straw	75	0.14, 0.16	0.15	
Canada Saskatchewan 1995 Katepwa	quinclorac 0.125 Bromoxynil/MCPA	1	21-22 (max two tillers)	forage	9	1.5, 1.6	1.55	95106 BASF/96/5103
				straw	75	0.1, 0.12	0.11	
Canada Nisku, Alberta 1995 Katepwa	quinclorac 0.122	1	21-22 (max two tillers)	forage	9	0.23, 0.23	0.23	95107 BASF/96/5103
				straw	75	< 0.05, < 0.05	≤ 0.05	
Canada Alberta 1995 Katepwa	quinclorac; 0.125, imazamethabenz; 2,4-D	1	21-22 (max two tillers)	forage	9	0.13, 0.14	0.135	95107 BASF/96/5103
				straw	75	< 0.05, < 0.05	< 0.05	
Canada Alberta 1995 Katepwa	quinclorac; 0.125, imazamethabenz; Tribenuron/thifensulfuron	1	21-22 (max two tillers)	forage	9	0.18, 0.19	0.175	95107 BASF/96/5103
				straw	75	< 0.05, < 0.05	< 0.05	
Canada	quinclorac 0.125	1	21-22	forage	9	0.3, 0.29	0.295	95107

Location	Application			Residues				Trial
Year (variety)	Treatment kg ai/ha	no	Growth stage BBCH	matrix	PHI	quinclorac	mean	Reference
Alberta 1995 Katepwa	Bromoxynil/MCPA		(max two tillers)	straw	75	< 0.05, < 0.05	< 0.05	BASF/96/5103

Table 65 Residues of quinclorac in spring wheat forage and straw following broadcast foliar application with a DF formulation

Location	Application			Residues				Trial
Year (variety)	kg ai/ha	no	Growth stage BBCH	matrix	PHI	quinclorac	Scaled quinclorac residues at 0.125 kg ai/ha	Reference
(USA MN) 1998 Pioneer	0.56	1	20-29 (tillering)	forage	22	0.27	0.059	90056 BASF/1998/5104
				straw	71	0.10[a]	0.022	
USA (MN) 1998 Stoa	0.56	1	20-29 (tillering)	forage	15	0.67	0.147	90057 BASF/1998/5104
				straw	70	0.05[a]	0.011	
USA Grand Forks (ND) 1998 Marshall	0.56	1	20-29 (tillering)	forage	22	0.47[b]	0.103	90058 BASF/1998/5104
				straw	70	0.04[a]	0.0088	
USA Steele (ND) 1998 Marshall	0.56	1	20-29 (tillering)	forage	15	0.27	0.059	90059 BASF/1998/5104
				straw	70	0.10	0.022	
USA Grand Forks (ND) 1998 Marshall	0.56	1	20-29 (tillering)	forage	15	0.60	0.132	90060 BASF/1998/5104
				straw	73	0.10	0.022	
USA* Minehaha (SD) 1998 Guard	0.56	1	20-29 (tillering)	forage	15	0.94	0.207	90061 BASF/1998/5104
				straw	70	0.32	0.070	
USA* Minehaha (SD) 1998 Guard	0.56	1	20-29 (tillering)	forage	15	1.55[a]	0.34	90062 BASF/1998/5104
				straw	71	0.74	0,163	
USA (MT) 1998 926	0.56	1	20-29 (tillering)	forage	15	1.1 [a]	0.24	90063 BASF/1998/5104
				straw	82	0.47	0.103	
USA (MT) 1998 Nevanna	0.56	1	20-29 (tillering)	forage	15	3.62[b]	0.796	90064 BASF/1998/5104
				straw	72	0.55[b]	0.121	
USA (ID) 1998 Pondera	0.56	1	20-29 (tillering)	forage	15	1.08[a]	0.234	90065 BASF/1998/5104
				straw	73	0.14	0.031	
USA (WA) 1998 Yecora Rojo	0.56	1	51 (beginning of heading)	forage	15	0.84	0.185	90066 BASF/1998/5104
				straw	71	0.50[b]	0.11	

Location	Application			Residues				Trial
Year (variety)	kg ai/ha	no	Growth stage BBCH	matrix	PHI	quinclorac	Scaled quinclorac residues at 0.125 kg ai/ha	Reference
USA (OR) 1990 Ovens	0.56	1	20-29 (tillering)	forage	15	0.85[b]	0.187	90067 BASF/1998/5104
				straw	74	0.57[b]	0.125	

PHI = pre harvest interval

[a] Value is the average of three analysis

[b] Value is the average of two analysis

n.r. = not reported

scaling factor = 0.22 (0.125/0.56 = 0.22)

Table 66 Residues of quinclorac in sorghum forage and stover following broadcast foliar application with a DF formulation

Location	Application			Residues				Trial
Year (variety)	kg ai/ha	no	Growth stage	matrix	PHI	quinclorac	mean	Reference
USA (NE) Hall county 1997 (NK 11210)	0.28	1	6-leaf stage	forage	64	< 0.05, < 0.05	< 0.05	97267 BASF/1998/5081
				stover	87	< 0.05, < 0.05	< 0.05	
USA (CO) 1997 (Cargill 577)	0.29	1	6-leaf stage	forage	50	0.06, 0.06	0.06	97268 BASF/1998/5081
				stover	91	< 0.05, < 0.05	< 0.05	
USA (NE) 1997 (Pioneer 8699)	0.28	1	6-leaf stage	forage	54	0.15, 0.12	0.14	97269 BASF/1998/5081
				stover	95	< 0.05, < 0.05	< 0.05	
USA (NE) 1997 (F270E)	0.28	1	6-leaf stage	forage	62	0.20, 0.17	0.19	97270 BASF/1998/5081
				stover	93	< 0.05, < 0.05	< 0.05	

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

Storage stability of quinclorac in sorghum starch was investigated up to 20 months by Brukey, J and Stewart J (1997/5046).

Control sorghum starch samples from study 1994/5104 study were fortified with 1.0 mg/kg quinclorac. The fortified samples were stored frozen (<-5 ºC) for a period of 20 months. Duplicate samples were analysed for total quinclorac using Method A8902 at 1 day and then 7, 17 and 20 months after the initial fortification.

Table 67 Storage stability of quinclorac in sorghum starch

Storage periods (months)	Procedural recovery % AR (mean)	Residues remaining % AR mean
0	na*	89, 88 (89)
7	98, 101 (100)	92, 95 (94)
14	72, 77 (75)	69, 80 (75)
20	89, 87 (88)	81, 86 (84)

* Data not available. The 0 day analysis, was extracted the day after fortification.

Nature of residue during processing

The hydrolysis of quinclorac during processing condition was investigated by Kennan, D and Brusky, M (2014 BASF/700909). ^{14}C-quinclorac was applied directly to a target concentration of 30 µg/mL to buffer solutions of pH 4, 5 and 6. Incubation was done at three representative sets of hydrolysis conditions: 90 °C, pH 4 for 20 minutes (pasteurization); 100 °C, pH 5 for 100 minutes (boiling) and 120 °C, pH 6 for 20 minutes (sterilization).

Parent compound and potential hydrolysis products were quantified by LSC and identified by HPLC using a radioactive detector (HPLC-RAD) and three replicates per sample. Quinclorac reference standard was chromatographed at the beginning of each sampling set. Material balance was established for each set of hydrolysis conditions. In the following tables recovered radioactivity is summarized.

Table 68 Hydrolysis of quinclorac under simulated processing conditions expressed as % TRR

Incubation time (minutes)	Hydrolysis conditions	Recovered % AR (average)	Quinclorac (average)	Total other (average)
0	pH 4, 90 °C	98.8, 98.9, 99.6 (99.1)	97.2, 97.1, 98.3 (97.5)	1.7, 1.9, 1.3 (1.6)
20		99.6, 100.2, 99.4 (99.7)	98.1, 98.6, 96.7 (97.8)	1.9, 1.6, 2.7 (2.1)
0	pH 5, 100 °C	100.1, 101, 99.8 (100.3)	98.2, 98.9, 97.2 (98.1)	1.8, 2.1, 2.6 (2.2)
20		100.6, 100.3, 99.8 (100.2)	97.5, 98.7, 97.4 (97.9)	3.1, 1.5, 2.3 (2.3)
0	pH 6, 120 °C	99.3, 98.6, 100 (99.3)	95.4, 96.6, 98.7 (96.9)	3.9, 2.0, 1.3 (2.4)
20		100.8, 100, 100.2 (100.3)	99.1, 97.0,98.8 (98.3)	1.8, 3.0, 1.4 (2.1)

Table 69 Hydrolysis of quinclorac under simulated processing conditions, expressed in concentrations µg/mL

Incubation time (minutes)	Hydrolysis conditions	Recovered µg/mL (average)	Quinclorac µg/mL (average)	Total other µg/mL (average)
0	pH 4, 90 °C	30.9, 29.7, 30.6 (30.4)	30.4, 29.1, 30.2 (29.9)	0.5, 0.6, 0.4 (0.5)
20		29.4, 30.3, 30.2 (29.9)	28.8, 29.8, 29.4 (29.3)	0.6, 0.5, 0.8 (0.6)
0	pH 5, 100 °C	29.9, 29.9, 29.8 (29.9)	29.4, 29.3, 28.9 (29.2)	0.6, 0.6, 0.8 (0.7)
20		29.8, 30.0, 20.8 (29.9)	28.9, 29.6, 29.2 (29.2)	0.6, 0.6, 0.8 (0.7)
0	pH 6, 120 °C	29.8, 29.8, 29.8 (29.8)	28.6, 29.2, 29.4 (29.1)	1.2, 0.6, 0.4 (0.7)
20		30.6, 30.6, 30.5 (30.6)	30.0, 29.7, 30.1 (29.9)	0.5, 0.9, 0.4 (0.6)

Within pH 4 and pH 5 none of the individually degradates exceeded 3.0% applied radioactivity (AR). Within pH 6 individual degradate did not exceed 4% AR. Therefore the products were not further analysed.

Residues after processing

The fate of quinclorac and its metabolite quinclorac methyl ester during processing of raw agricultural commodity (RAC) was investigated in rice, wheat, and sorghum and rape seed. As a measure of the transfer of residues into processed products, a processing factor (PF) was used, this is defined as:

$$PF = \frac{\text{Total residue in processed products (mg/kg)}}{\text{Total residue in raw agriculture commodity (mg/kg)}}$$

If residues in the RAC were below the LOQ, no processing factor could be derived.

Rice

In one field trial conducted in Texas and reported by Single, YH (1989, BASF/5003) rice samples were taken from field plots treated with a single foliar application of 1.68 kg ai/ha (3N GAP rate) and a pre-harvest interval of 79 days. The samples were harvested at normal maturity and then processed into hulls and brown rice which was further processed into bran and white milled rice indicating that it is polished milled rice

The milling process was designed to simulate commercial processing. The rough rice was shelled to remove hulls. The remaining brown rice was milled to remove the bran and to yield white milled rice. The processed fractions were homogenized and stored frozen. Rough rice was analysed 12 months after storage followed by the processed fraction 13 months after harvest.

All samples (10g) were analysed for quinclorac according to method A8902. The method is designed to determine residues of quinclorac expressed as its methyl ester. The LOQ was 0.05 mg/kg. Spiked samples were run concurrently and the overall average recovery was 82±11% (n=23). In the following table the residues found in the processed commodities are summarized.

Table 70 Residues of quinclorac in rice and rice processed products

Commodity	Residues (mg/kg)	Mean residues (mg/kg)	Processing factor
Rice grain	0.43, 0.46	0.45	-
Hulls	0.50, 0.45	0.48	1.07
Brown rice	0.47, 0.45	0.46	1.02
Rice bran	1.4, 1.2, 1.5, 1.3	1.35	3
Milled rice	0.33, 0.35	0.34	0.76

Residues in the hulls have been corrected for the control baseline. None of the other results were corrected for control or recovery values.

Wheat

In two independent field trials conducted in USA and reported by Burkey, JD and Riley, M (1994, BASF/5093) samples of spring wheat were taken from plots treated with three post emergence broadcast applications of quinclorac each at 0.56 kg ai/ha (6× GAP rate). The treatments were made in a sequence within growth stage BBCH 22–49 (from tillering to first awn visible). The samples (control and treated) were harvested at normal maturity (57–58 days after the last application) and then processed.

The spring wheat grain was first dried and cleaned to separate husks and other impurities from the grain. The seed were then conditioned by adding tap water to adjust the moisture content to 16%. The milling process followed. For analysis, samples of whole wheat, bran, middlings, shorts, low grade flour and patent flour was collected.

Samples were analysed for quinclorac according to method A8902. The LOQ was 0.05 mg/kg. Spiked samples were run concurrently and the overall average recovery was 76±9% (n=14). In the following table the residues found in the processed commodities are summarized.

Table 71 Residues of quinclorac in wheat and wheat processed products

Location, year	No	kg ai/ha total	DALT*	Commodity	Residues (mg/kg)	average mg/kg	PF calculated
Minnesota 1990	3	1.68	57-58	Wheat grain	0.21, 0.20	0.21	
				Bran	0.43, 0.44	0.44	2.1
				Middlings	0.17, 0.11	0.14	0.67
				Shorts	0.26, 0.26	0.26	1.24
				Low grade four	0.12, 0.14	0.13	0.62
				Patent flour	0.16, 0.15	0.16	0.76
North Dakota 1990	3	1.68		Wheat grain	1.01, 0.95	0.98	
				Bran	1.59, 1.45	1.52	1.55
				Middlings	0.92, 0.86	0.89	0.91
				Shorts	1.23, 1.28	1.26	1.29
				Low grade four	0.48, 0.58	0.53	0.54
				Patent flour	0.53, 0.57	0.55	0.56

DALT= days after last treatment

PF = processing factor

In one field trial in spring wheat conducted in USA and reported by Versoi, PL (1996, BASF/5208) samples of spring wheat were taken from plots treated with one pre emergence broadcast applications of quinclorac at 1.4 kg ai/ha (5× GAP rate). The treatment was made on bare soil on the date the wheat seed was later planted. Grain samples (control and treated) were collected as raw agricultural commodity at normal maturity 103 days post application and then processed.

The spring wheat grain was first dried and cleaned to separate husks and other impurities from the grain. 50 kg of cleaned grain from untreated control sample and 19 kg of treated grain were removed for germ recovery process. The yield of grain from treated plots was reduced due to the extreme rate of application. The 50 kg sample was divided into 25 kg batches. The entire sample of treated wheat and each batch of untreated control was lightly ground and separated over a 12 mesh screen. The resulting material was screened over a 46 mesh screen. The material that passed through the 46 mesh screen was discarded. The material that remained on the screen contained the germ fraction. The endosperm fragments were separated from the germ fraction by bulk density. The entire wheat germ samples recovered was stored frozen. The original plan for processing is presented below. Due to the low supply of treated grain, residues were only measured in the germ fraction.

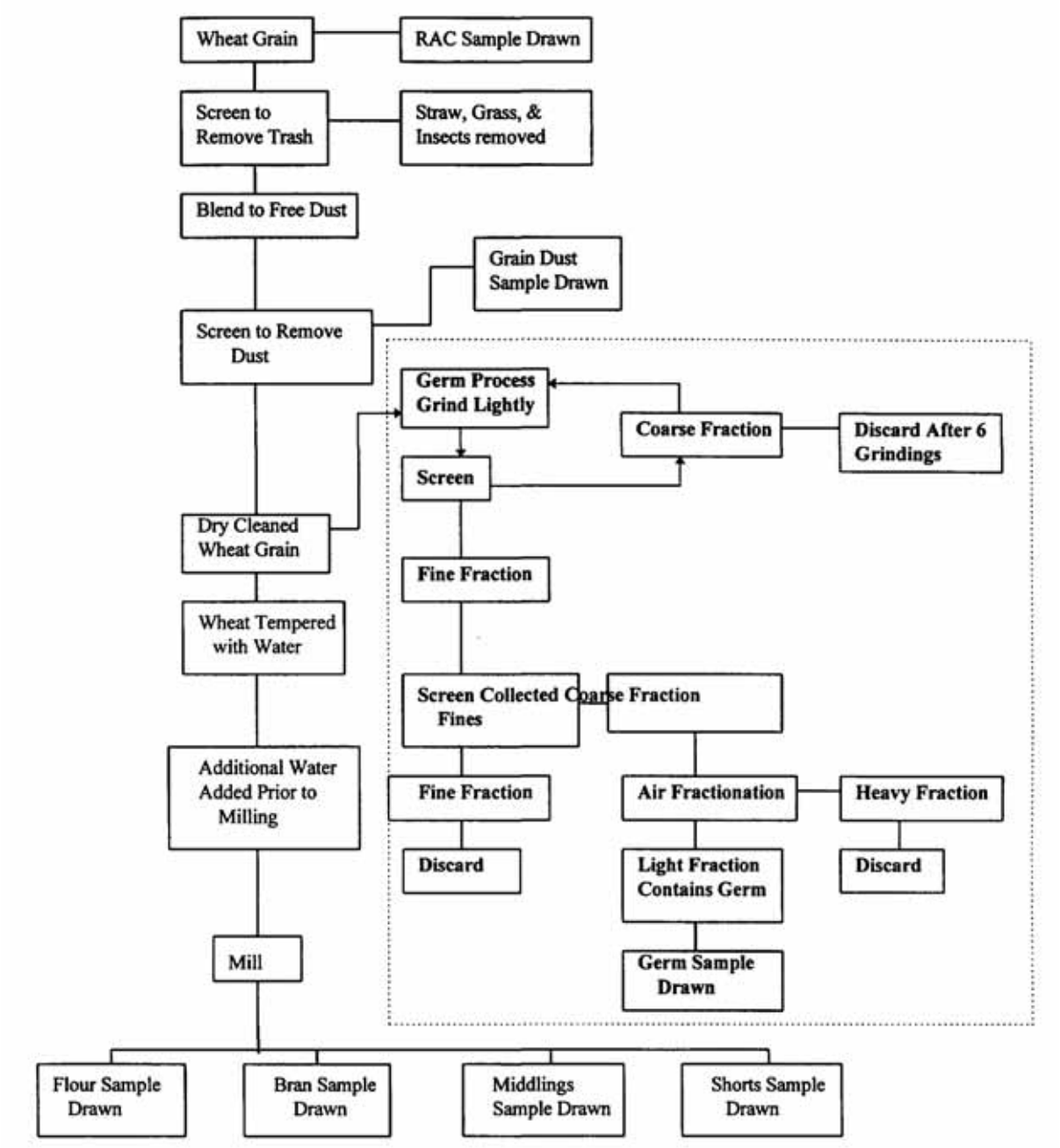


Figure 10 The original plan for processing into wheat germ

Samples were analysed for quinclorac according to method A8902. The LOQ was 0.05 mg/kg. Spiked samples were run concurrently and the overall average recovery was 79±7% (n=5). In the following table the residues found in the wheat germs are summarized.

Table 72 Residues of quinclorac in wheat and wheat germs

Commodity	Residues (mg/kg)	Mean residues (mg/kg)	PF
Wheat grain	0.221, 0.221	0.221	-
Germ	0.544, 0.683	0.614	2.8

PF = processing factor

Rape seed (canola)

In two independent field trials in rape seed one conducted in Canada and the other in USA reported by Guirguis, M (1998, BASF/5093) samples of rape seed were taken from plots treated with 0.1 kg ai/ha (1× GAP rate), 0.5 kg ai/ha and control plots. The application was a broadcast spray made to the crop 60 days, before the rape seed was harvested and then processed.

The rape seed was dried at 54–71 °C to a moisture content of 7–10%. After aspiration separating light impurities, the sample is screened to separate large and small foreign particles (screenings) from the canola. The conditioned and cleaned oil seeds were flaked and pressed yielding crude oil, press cake (meal), refined oil and soap stock.

Whole seed were flaked with a gap setting of 4–5 mm. The flakes were heated to 82–99 °C and pressed to liberate most of the crude oil. Residual crude oil remaining in the solid material (press cake) exiting the expeller was extracted with the solvent hexane.

The press cake was placed in stainless steel batch extractors and submerged in 43–52 °C solvent (hexane). After 30 minutes, the hexane was drained and fresh hexane added to repeat the cycle two more times. After the final draining, warm air was forced through the extracted press cake to remove residual hexane.

The miscella (crude oil and hexane) was passed through a Precision Scientific Recovery unit to separate the crude oil and hexane. The crude oil was heated to 73–90 °C for hexane removal. The crude oil recovered from the expeller and solvent extraction was combined and refined. Before refining the crude oil was pre-treated with phosphoric acid. Refining is performed according to AOCS method Ca9a52. After refining, the refined oil and soap stock are collected.

Residues of quinclorac and quinclorac methyl ester (BH 514 ME) were determined in rape seed, meal and refined oil.

Samples (duplicate) were analysed for quinclorac according to method D9708/1 (LC-MS/MS) and for quinclorac methyl ester by the method D9806 (LC-MS/MS). The LOQ was 0.05 mg/kg for each analyte. Spiked samples were run concurrently for each analyte and the recovery for each of them ranged in rape seed, meal, and oil from 69–110%. In the following table the residues found processed products are summarized.

Table 73 Quinclorac resides in rape seed, meal and refined oil

Location, year	No	kg ai/ha	DALT	Sample	Quinclorac		Quinclorac methyl ester		Total Quinclorac + quinclorac methyl ester	
					mg/kg	PF	mg/kg	PF	mg/kg	PF
USA 1998	1	0.1	60	seed	0.05	-	0.054		0.1	-
		0.5	60	seed	0.19	-	0.30		0.49	-
		0.1	60	meal	< 0.05	<1	< 0.05	< 0.93	< 0.05	< 0.5
		0.5	60	meal	0.07	0.36	0.45	1.5	0.52	1.06
		0.1	60	refined oil	< 0.05	<1	0.055	1.02	0.06	0.6
		0.5	60	refined oil	< 0.05	< 0.26	0.20	0.33	0.25	0.5
Canada 1998	1	0.1	60	seed	0.13		0.24		0.37	-
		0.5	60	seed	0.36		1.0		1.36	-
	1	0.1	60	meal	0.28	2.15	< 0.05	< 0.21	0.33	0.89
		0.5	60	meal	0.58	1.61	0.08	0.08	0.66	0.49
		0.1	60	refined oil	< 0.05	< 0.39	0.29	1.21	0.34	0.92
		0.5	60	refined oil	0.08	0.22	1.36	1.36	1.44	1.06

DALT= days after last treatment

PF = processing factor

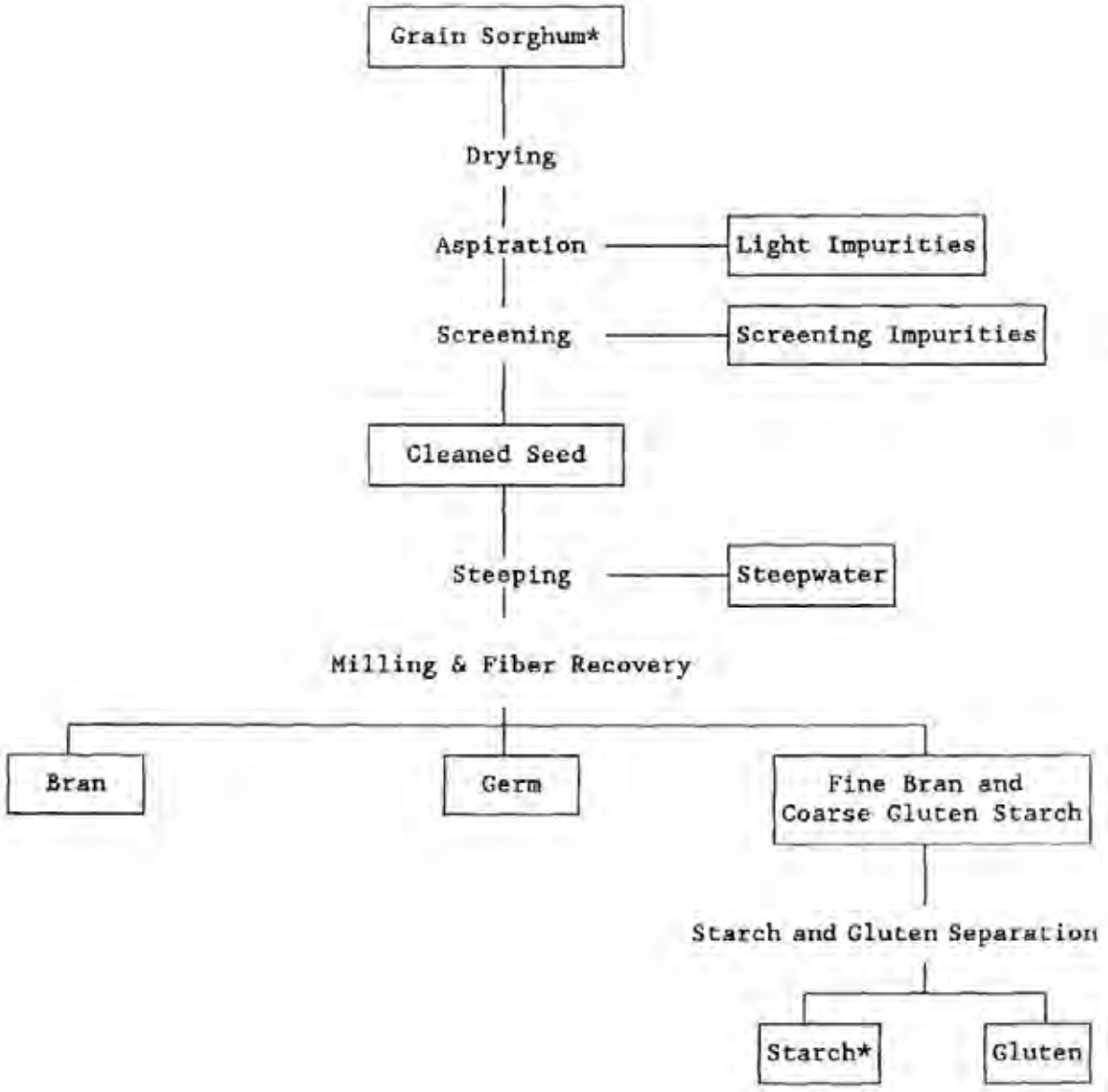
Sorghum

In two independent field trials conducted in USA and reported by Burkey, JD and Riley, M (1994, BASF 1994/5104) samples of sorghum grain were taken from plots treated with a pre-emergence application of 1.12 kg ai/ha followed by two sequential broadcast post emergence applications of 0.84 kg ai/ha. The applications were broadcast spray made when the sorghum was 3 to 5 leaf stage and again at the 8–10 leaf stage. Samples of sorghum grain were taken at normal maturity which was 89 days or 106 days after the last application and then processed.

The sorghum grain was dried until moisture content was 13% or less. After drying the grain was cleaned from light impurities (aspiration) and screened for large impurities (screening).

For dry milling of grain into flour, the hulls were first separated (decorticated) and then ground with a 2 mm screen and passed through a sifter

For wet milling of grain into starch, the cleaned grain was conditioned by steeping in a stainless steel tank with water, sodium bisulfite, and lactic acid at 50 °C. The steeped grain was ground, a majority of the germs were removed, the stock solution was passed over a shaker equipped with a 610µ (0.6 mm) screen. The material collected after screening was passed through a mill with 6 mm screen. The milled product was washed in a shaker equipped with a 43µ (0.043 mm) screen. The remaining process water with gluten and starch fraction was separated by centrifugation. The resulting fractions were starch, gluten and process water.



*fraction analysed

Figure 11 Flow chart of wet milling sorghum grain into flour and starch

Samples (duplicate) were analysed for quinclorac residues according A8902 (derivatized extracts analysed by GC). The LOQ was 0.05 mg/kg. Spiked samples were run concurrently and the average recoveries were 86% (n=2) for the grain, 81% (n=2) for the flour and 79% (n=2) for the starch. In the following table the residues found processed products are summarized.

Table 74 Quinclorac resides) in sorghum grain, flour and starch

Location, year	No	kg ai/ha total	DALT*	Sample	quinclorac		
Nebraska 1990	3	2.8	106		mg/kg	average	pf
				grain	0.33, 0.29	0.31	-
				flour	0.24, 0.28	0.26	0.83
				starch	< 0.05, < 0.05	< 0.05	< 0.16
Kansas 1990			89	grain	1.98, 1.91	1.95	-
				flour	1.67, 2.03	1.85	0.95

Location, year	No	kg ai/ha total	DALT*	Sample	quinclorac		
				starch	0.08, 0.08	0.08	0.04

DALT= days after last treatment

PF = processing factor

Table 75 Summary of quinclorac residues in processed commodities

		Calculated processing factors			
RAC	Commodity	Quinclorac	Quinclorac methyl ester	Total Quinclorac+quinclorac methyl ester	PF median or best estimate
Rice	RAC: grain				
	hulls			1.07	1.07
	brown rice			1.02	1.02
	bran			3	3
	milled			0.76	0.76
Wheat	RAC: grain				
	bran			2.1, 1.55	1.83
	middlings			0.67, 0.91	0.79
	shorts			1.24, 1.29	1.27
	low grade flour			0.62, 0.54	0.58
	patent flour			0.56,0.76	0.66
	germ			2.8	2.8
Rape seed	RAC: seed				
	meal	<1.0, 1.61, 2.15, 0.36,	0.08, < 0.21, < 0.93, 1.5	0.49, <,0.5, 0.89, 1.06	
	refined oil	0.22, < 0.26, < 0.39, <1.0,	0.33, 1.02, 1.21, 1.36	0.5, 0.6, 0.92, 1.06	
Sorghum	RAC: grain				
	flour			0.83, 0.95	0.89
	starch			0.16, 0.04	0.10

RESIDES IN ANIMAL COMMODITES

Farm animal feeding studies

For the estimation of residues of quinclorac in animal matrices laying hen and lactating cow feeding studies was submitted to the Meeting. Storage stability data was not provided in the studies.

Poultry

The magnitude of the residue of quinclorac has been studies in laying hens by Mayer, F (1989, BASF 89/5024)(Method 268). Adult hens (15 birds per diet group divided in 3 subgroups with five birds each, one control with four to three birds) were exposed for 28 consecutive days to levels of 1 ppm (1 × dose group), 10 ppm (10 × dose group) and 100 ppm feed/day (100 × dose group) corresponding to approximately (0.07, 0.7 and 7 mg/kg bw/day

Eggs were collected during the whole dosing period. At sacrifice (day 28) samples of muscles, skin and subcutaneous fat, heart, gizzard, liver and kidney were sampled.

Eggs and tissues were analysed for the parent using method no 268. The LOQ was 0.05 mg/kg for the parent. The limit of detection was (LOD) was 0.01 mg/kg. The maximum storage time under frozen conditions was 90 days for eggs and 74 days for tissues.

In the following table the residues from eggs are summarized. Prior to dosing of quinclorac eggs collected contained no detectable residues of quinclorac. The results for those samples are not presented.

Table 76 Residues of quinclorac in eggs of laying hens after daily administration of quinclorac for 28 days

Days	Residues* in mg quinclorac-equivalents per kg (mean)		
Dose level	1 ppm	10 ppm	100 ppm
-1	< 0.01 (2)	< 0.01 (2)	< 0.01
1	< 0.01 (2)		< 0.01
2	< 0.01 (2)		0.016, 0.013 [0.015]
3	< 0.01 (2)		0.020, 0.023 [0.025]
4	< 0.01 (2)		0.011, 0.019 [0.015]
5	< 0.01 (2)		0.017, < 0.01 [0.09]
6	< 0.01 (2)		0.024, 0.025 [0.025]
7	< 0.01 (2)	< 0.01 (2)	**0.032, 0.033 [0.033**]
10	< 0.01 (2)		0.016, 0.025 [0.021]
12	< 0.01 (2)		0.021, 0.032 [0.027]
14	< 0.01 (2)		0.030, 0.019 [0.025]
18	< 0.01 (2)		0.013, 0.016 [0.015]
21	< 0.01 (2)	< 0.01 (2)	0.015, 0.033 [0.024]
23	< 0.01 (2)		0.013, 0.031 [0.022]
25	< 0.01 (2)		**0.036, 0.041 [0.039]**
28	< 0.01 (2)	< 0.01	0.036, 0.024 [0.03]

*based on limit of detection LOD (0.01 mg/kg)

The bodyweight of the birds were not influenced, however the number of egg laid appeared to be lower in the highest dose group.

Table 77 Number if egg laid per diet group after administration of quinclorac at 0.07, 0.7 or 7 mg/kg bw/day

Days	Control	1 × (1 ppm, 0.07 mg/kg bw)	10 × (10 ppm, 0.7 mg/kg bw)	100 × (100 ppm, 7 mg/kg bw)
-1--7	82	91	66	53
1-7	78	78	90	49
8-14	67	69	85	46
15-21	68	59	73	42
22-28	59	65	65	44

For laying hen tissue residues of quinclorac found in tissue after end of dosing period are presented in the following table

Table 78 Residues of quinclorac in tissues of laying hens after daily administration of quinclorac for 28 days

Tissue	Residues* in mg quinclorac-equivalents per kg (mean)		
Dose level	1 ppm	10 ppm	100 ppm
Skin and fat	0.00, 0.013, 0.018 [0.01]	0.12, 0.13, 0.17 [0.14]	0.122, 0.475, 0.760 [0.452]
Muscle dark	0.005, 0.00, 0.00 [0.002]	0.00, 0.00, 0.00 [0]	0.022, 0.025, 0.045, [0.03]
Muscle light	0.005, 0.00, 0.00 [0.002]	0.002, 0.003, 0.004, [0.003]	0.018 ,0.039, 0.068 [0.042]
kidney	0.002, 0.02, 0.059 [0.027]	0.007, 0.011, 0.015 [0.011]	0.235, 0.456, 0.558 [0.412]
Liver	0,00, 0.009, 0.009 [0.006]	0.009, 0.012, 0.013 [0.011]	0.042, 0.054, 0.128 [0.075]

*based on limit of detection LOD (0.01 mg/kg)

Lactating cows

Residues in lactating cows were investigated by Mayer F (1989 BASF 89/5025)(Method 268). Fifteen lactating Friesian dairy cows, three cows/treatment group, were dosed orally, via capsule, for 28

consecutive days with quinclorac either 0 ppm (control), 1 ppm (1 × dose group), 10 ppm (10 × dose group), 50 ppm (50 × dose group) or 500 ppm (500 × dose group) corresponding to approximately 0.002 mg/kg bw, 0.02 mg/kg bw, 0.09 mg/kg bw and 0.9 mg/kg bw, respectively.

Milk was collected twice daily. On day 29 after the administration of the first dose, the animals were sacrificed and liver, kidney, muscle, omental fat, and subcutaneous fat were collected for analysis. The maximum storage time under frozen conditions was for milk 31 days, subcutaneous fat 58 days, peritoneal fat 56 days and muscle 51 days.

Milk and tissues were analysed for the quinclorac using BASF method no 268. The LOQ was 0.05 mg/kg. The LOD was 0.01 mg/kg.

Quinclorac residues in milk are presented in the following table:

Table 79 Residues* of quinclorac in milk after daily oral administration of quinclorac for 28 days

Days	1 ppm	10 ppm	50 ppm	500 ppm
-1	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
1	< 0.01 (3)			0.01, 0.014, 0.016 **[0.013]**
2	< 0.01 (3)			< 0.01, 0.011 0.035 **[0.019**]
3	< 0.01 (3)			0.016, 0.026, 0.033 **[0.025]**
4	< 0.01 (3)			0.032, 0.027, 0.038 **[0.032]**
5	< 0.01 (3)			0.016, 0.018 0.030, **[0.021**]
6	< 0.01 (3)			0.018, 0.026, < 0.01 **[0.018]**
7	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	0.013, 0.023, 0.024 **[0.02]**
8				
9				
10	< 0.01 (3)			0.012, 0.014, 0.017 **[0.014]**
11				
12	< 0.01 (3)			0.01, 0.016, 0.02 **[0.015]**
13				
14	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (2) 0.013 **[0.011]**
15				
16				
17	< 0.01 (3)			
18				< 0.01, 0.011, 0.019 **[0.013]**
19				
20				
21	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01, (2), 0.01 **[0.01]**
22				
23	< 0.01 (3)			< 0.01, (2), 0.01 **[0.01]**
24				
25	< 0.01 (3)			< 0.01, (3) **[0.01]**
26				
27				
28	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01, (2), 0.012 **[0.011]**

*based on limit of detection LOD (0.01 mg/kg)

For lactating cow residues of quinclorac found in tissue after the end of the dosing period are presented in the table below.

Table 80 Residues of quinclorac in tissues from lactating cows after daily oral administration of quinclorac for 28 days

Tissue	Residues* in mg quinclorac-equivalents per kg (mean)			
	1 ppm	10 ppm	50 ppm	500 ppm
Fat, subcutaneous fat	< 0.01, < 0.01, 0.013 [0.005]	< 0.01 (3)	< 0.01, 0.01 (2)	0.11, 0.122,1.38 [0.537]
Fat, peritoneal	< 0.01 (2), 0.01	< 0.01 (2), 0.023 [0.008]	< 0.01 (2), 0.014, [0.005]	0.195, 0.253, 0.269, [0.239]

Tissue	Residues* in mg quinclorac-equivalents per kg (mean)			
Muscle	< 0.01 (2), 0.01	< 0.01 (3)	< 0.01 (3)	0.010, 0.033, 0.037 [0.027]
kidney	< 0.010, 010, 0.016, [0.06]	0.062, 0.074, 0.082 [0.073]	0.144, 0.174, 0.186 [0.168]	1.188, 1.514, 2.634 [1.779]
Liver	< 0.01 (3)	0.010, 0.014, 0.020 [0.015]	0.022, 0.026, 0.029 , [0.026]	0.188, 0.276, 0.326 [0.263]

*based on limit of detection LOD (0.01 mg/kg)

National residue definitions

Country	MRL-compliance	Dietary intake	Exceptions/comment
Australia	quinclorac	quinclorac	
Canada	quinclorac	quinclorac	For rape seed quinclorac + quinclorac methyl ester
Europe	not registered	not registered	Import tolerance for rice: quinclorac
Korea	quinclorac	quinclorac	Import tolerance for rape seed: quinclorac + quinclorac methyl ester
Japan	quinclorac + quinclorac methyl ester for crops	quinclorac + quinclorac methyl ester for crops	
Japan	quinclorac for terrestrial animal	quinclorac for terrestrial animal	
USA	quinclorac	quinclorac	For rape seed quinclorac +quinclorac methyl ester expressed as quinclorac

APPRAISAL

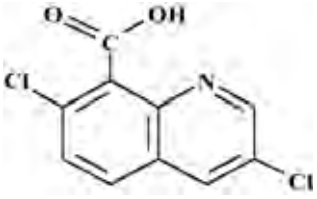
Quinclorac is a systemic herbicide with uptake through roots and foliage and used to control annual grass and broadleaf weeds. Quinclorac mode of action is similar to phenyl herbicides as it imitates the plant growth hormone auxin. The use of quinclorac results in the rupture of the cell membranes due to overstimulation of the growth of the plant.

It was scheduled by the Forty-sixth Session of the CCPR (2014) as a new compound for consideration by the 2015 JMPR. The manufacturer submitted studies on metabolism, analytical methods, supervised trials, processing, storage stability, environmental fate in soil and rotational crop studies.

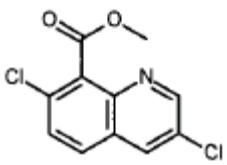
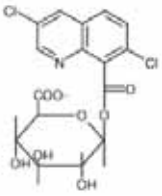
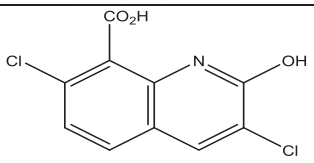
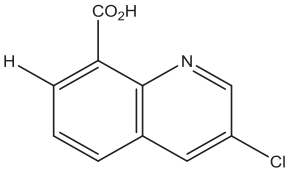
Quinclorac is registered for uses in berries and other small fruits stalk and stem vegetables, cereal grains and rape seed in Australia, Canada, China, Republic of Korea, South America and USA. Information on GAP with supporting labels from Canada and USA was provided to the Meeting.

Chemical name

Quinclorac: 3,7-dichloroquinoline-8-carboxylic acid
Structural formula:



Metabolites referred to in the appraisal with codes:

BH 514-Me Quinclorac methyl ester SES218	 methyl-3,7-dichloroquinoline-8-carboxylate
BAS 514 H M1 glucuronide (glucuronic acid) conjugate	
BH 514-2-OH 2 -hydroxyquinclorac	 3,7-dichloro-2-hydroxyquinoline-8-carboxylic acid
BH 514-1 3-chloroquinoline-8-carboxylic acid	 3-chloroquinoline-8-carboxylic acid

Animal metabolism

The Meeting received metabolism studies on laboratory animals, poultry and lactating goats using 2, 3, 4-[^{14}C]-quinclorac (quinoline label).

In rats quinclorac is widely distributed in the body, with highest concentrations of radiolabel present in the blood, kidney and plasma. The labelled material was excreted primarily via urine (50-90% in 24 hours). Absorbed quinclorac was metabolized to only a limited extent, with unchanged parent compound representing approximately 80% of the excreted radiolabel. The major bio-transformation product was quinclorac glucuronide conjugate at approximate 5% of the administrated dose.

One lactating goat received five daily doses of ^{14}C- quinclorac at a rate equivalent to 800 ppm in the diet (34 mg/kg bw). The animal was sacrificed approximately 6 h after the last dose.

A total 67% of the applied radioactivity was recovered. Excretion of radioactivity in urine and faeces accounted for 63 and 3.7% respectively of the total dose. In milk 0.003%, in liver 0.12% and kidney 0.1% of the administrated dose was recovered. The extraction efficiency using 1 M HCl) was generally > 80% TRR in muscle and liver. In milk and kidney it was above 95% TRR.

In milk the TRR levels reached a plateau after 48 hrs. Residues found in tissues at sacrifice were 0.16 to 0.19 mg eq/kg in muscle, 0.14 and 0.78 mg eq/kg in fat (omental and subcutaneous respectively), 10 mg eq/kg in kidney and 2.1 mg eq/kg in liver. Muscle and fat were not analysed

further. In milk, liver and kidney, parent quinclorac was the major residue at 86, 81 and 86% TRR respectively. Metabolite (M1) identified as the glucuronic acid conjugate of the parent was found at 4.0% TRR in milk and at 4.7% TRR in kidney.

Seven laying hens (1.8–2.4 kg) were orally dosed once daily for five days with 33–44 mg radiolabelled quinclorac per kg body weight per day corresponding to 800 ppm in the diet. The animals were sacrificed after 6 hours after the last dose. The major part of the radioactivity was recovered in the excreta (93%).

Extraction efficiency (including 1 M HCl) was generally above 80% for excreta, liver, breast muscle and skin. In eggs the TRR levels increased from < 0.06 mg eq/kg one day after first administration up to a plateau of 1.2 mg eq/kg after three days; however levels of TRR showed wide variation in eggs. TRR levels in tissues were 1.1–1.8 mg eq/kg in muscle (breast and leg respectively), 2.0 mg eq/kg in skin/fat, 3.7 mg eq/kg in kidney and 20 mg eq/kg in liver. The unextracted residues were from 2.0–21.1% of TTR.

Parent quinclorac was the major residue in poultry tissues and eggs (78–92% of the TRR). The only metabolite identified was M1, present up to 3% TRR in a combined concentration with two other fractions.

In summary from data presented quinclorac is not significantly metabolized in animals. Parent quinclorac is the major residue found in tissues, milk and eggs, making up from 78–92% TRRs, with the only identified metabolite being M1 present at low levels (< 5% TRR) and also identified in the rat. Since the extraction methods used for lactating goat and poultry tissues included 1 M HCl, it is not clear whether parent compound represents parent only or includes parent released from conjugates and whether the M1 is the fraction of conjugates that remained uncleaved.

Plant metabolism

The Meeting received plant metabolism studies for quinclorac following pre- and/or post-emergent foliar application of 2,3,4-^{14}C-quinclorac to rice, or with 3-^{14}C-quinclorac to wheat, rape seed sorghum and strawberry.

Rice plants were treated in the growth chamber with one foliar application at 1.5 kg ai/ha, and with one application at 0.84 kg ai/ha in the field at the 4 and 3-5 leaf stage, respectively. Samples were collected from whole plant (28 days after application), straw (97 days after application) and grain (97 and 118 days after application from growth chamber and field respectively). Total radioactive residues were 0.49 mg eq/kg from whole plant, 13 mg eq/kg from straw and 1.5 mg eq/kg and 0.12 mg eq/kg from grain in growth chamber and field respectively. Extraction rates were in general above 80% TRR.

Quinclorac was the major residue identified (85–94% TRR) in rice straw, whole plant, and grain in growth chamber and the field. Since rice grain was extracted by reflux with 1 M HCl, the quinclorac detected in rice grain might be released from conjugates. Metabolites present at low levels were not identified.

Wheat plants were treated in the greenhouse with one foliar application of 0.125 kg ai/ha or 0.5 kg ai/ha at the 3–5 leaf stage. Samples were collected of forage (early to late boot stage, 37 days before harvest), straw and grain (92 days after application). Total radioactive residues following the low application rate were 3.3 mg eq/kg (forage), 1.9 mg eq/kg (straw) and 1.1 mg eq/kg (grain) and following the high application rate were 13 mg eq/kg (forage), 8.2 mg eq/kg (straw) and 3.9 mg eq/kg in grain. Extraction with acetone/water and subsequent treatment with NaOH for forage, grain and straw in general was above 80% TRR.

In all plant parts parent quinclorac was the major residue identified at 24% and 45% TRR in forage, 12 and 22% TRR in straw and 62 and 68% TRR in grain from low and high application rate respectively. Metabolites characterized as hydroxyquinclorac conjugates were present in forage at 6.8% TRR (0.22 mg/kg) in the low application rate and 6.4% TRR (0.84 mg/kg) in the high application rate, straw at 14% TRR (0.26 mg eq/kg) and 13% TRR (1.83 mg eq/kg), in grain at 4%

TRR (0.05 mg eq/kg) and 4%TRR (0.14 mg eq/kg) in low and high rate application respectively. Other metabolites identified in forage and straw were quinclorac conjugates and hydroxyquinclorac, each < 5% TRR.

Sorghum plants were grown outdoor and treated with a pre-emergence spray application to the soil followed by a foliar treatment (post-emergence) when sorghum plants were 15–25 cm tall. The pre-emergence treatment was 0.525 kg ai/ha and the post-emergence at 0.504 kg ai /ha (total 1.03 kg ai/ha. Residue analysis was done on forage (whole plants) collected at 25 days after the last treatment and on mature fodder and grain collected at 95 days after last treatment.

Extraction with acetone/water and subsequent treatment with HCl were in general above 80% TRR for forage, grain and straw. In all plant samples, unchanged parent quinclorac was the major residue being present at levels of 73% TRR (2.9 mg eq/kg) in forage, 22% TRR (0.19 mg eq/kg) and 74% (0.61 mg eq/kg) in grain. This residue included the quinclorac that was released from remaining solids (4% in grains to 9% TRR in forage and fodder) under hydrolysis conditions. The only other metabolite identified was quinclorac methyl ester present at 3.6% TRR in forage, 5.9% in fodder and 1.7% in grain. A large amount of unidentified residues was present in forage and fodder in organic and aqueous fractions, maximum 19% TRR (0.75 mg eq/kg in forage and 52% TRR (0.46 mg eq/kg) in fodder.

Rape seed plants were grown in a growth chamber and treated with one foliar post emergence application of 0.2 kg ai/ha at 30 days after sowing at 5th true leaf stage. Whole plants were sampled 1 and 29 days after treatment. Seed and straw were sampled 60 days after treatment. Extraction with acetone/ phosphate buffer and subsequent treatment with 0.1M NaOH was above 90% TRR in seed and straw.

Residues in seed were identified as parent quinclorac at 37% TRR (0.18 mg eq/kg) and the quinclorac methyl ester 37% TRR (0.18 mg eq/kg). Metabolites characterized as ‘aqueous soluble’ were present at 8.7% TRR (0.042 mg eq/kg) and those characterized as ‘organo soluble’ were found at 8.6% TRR (0.041 mg eq/kg). Residues in straw (0.64 mg eq/kg) and forage (0.68 mg eq/kg) were not further identified

Strawberry plants were grown outdoor and treated with one foliar post-emergence application at growth stage BBCH 73 (seeds clearly visible). The treatment rate was 1.12 kg ai/ha. Foliage and fruits were sampled at three harvest times 21, 37 and 61 days after treatment.

In foliage, unchanged parent quinclorac accounted for 67% TRR (10 mg eq/kg) at first harvest 21 DAT and at 57% TRR (4.4 mg eq/kg) at the last harvest 61 DAT. Conjugated quinclorac released by acid hydrolysis ranged from 27%TRR (4.2 mg eq/kg) at first harvest to 29%TRR (2.3 mg eq/kg) in the last harvest. Extraction efficiency was above 90% TRR in fruit and foliage.

In fruit, unchanged parent quinclorac accounted for 79% TRR (9.1 mg eq/kg) at first harvest and at 51% TRR (1.7 mg eq/kg) at third harvest 61 DAT. Conjugated quinclorac released by acid hydrolysis increased from 11%TRR (1.3 mg eq/kg) at first harvest to 47%TRR (1.6 mg eq/kg) in the last harvest. Quinclorac methyl ester accounted for 9.6% TRR (1.1 mg eq/kg) at first harvest, to 4.9% TRR (0.42 mg eq/kg) at second harvest and was not detected at the last harvest.

In summary the Meeting concluded that in cereals (rice, wheat and sorghum), and in strawberry quinclorac is not significantly metabolized and parent quinclorac including conjugates is the major residue > 80% TRR in both food and feed matrices. A number of identified quinclorac conjugates were identified in amounts below 5% TRR in cereals and up to 47% TRR in fruit. Quinclorac levels reported in cereal metabolism studies may already include the quinclorac released from conjugates. Other metabolites were not found in tested crop matrices above 10% TRR except quinclorac methyl ester which was found at 37% TRR (0.18 mg eq/kg) in rape seed. Quinclorac methyl ester was found as a minor metabolite in strawberry fruit at a maximum of 9.6% TRR (1.1 mg eq/kg), in sorghum at a maximum of 1.7% TRR (0.014 mg eq/kg) and in forage at 3.6% TRR (0.14 mg eq/kg).

Environmental fate in soil

The Meeting received studies on hydrolysis, photolysis, terrestrial and aquatic soil metabolism and field dissipation for the investigation of the environmental fate.

In the photolysis study it was shown that quinclorac degraded slowly with a half-life of 162 days. The soil hydrolysis study showed that quinclorac was stable during the testing period 30 days and at the temperature 25 °C.

In aerobic soil metabolism studies in silt loam soils under laboratory conditions and an application rate of 0.375 kg ai/ha, quinclorac degraded slowly; no degradation was indicated 120 days after treatment. In another study at an application rate of 3.9 to 4.1 kg/ha, the half-life (DT_{50}) for quinclorac was estimated at 391 days in loamy sand and 168 days in a clay soil. In this study two major soil metabolites were detected; 2-hydroxyquinclorac, at a maximum of 12% AR and quinclorac methyl ester at a maximum of 7.8% AR. Other metabolites were present at levels below 10% AR.

In one tested aerobic aquatic system (rice field) at an application rate 3.75 kg ai/ha, quinclorac degraded to the metabolite 3-chloro-8-quinolilne carboxylic acid (BH 514-1) up to a maximum of 55.7% AR. Three additional fractions were present (not characterized) but present at less than 10% AR. The half-life of quinclorac in this system was 4.7 months and for the metabolite 3-chloroquinoline-8-carboxylic acid, 7.4 months. Under anaerobic conditions at the same application rates the same metabolites were formed but at a slower rate; there was 50% conversion of quinclorac to 3-chloroquinoline-8-carboxylic acid.

In one field dissipation study using a loamy sand soil, quinclorac was applied to bare soil with two applications of 2.8 kg ai/ha. DT_{50} and DT_{90} values for parent quinclorac were 126 days and > 360 days respectively following the first application (autumn), and DT_{50} and DT_{90} of 8 days and 26 days respectively following the second application (summer). The maximum of the two metabolites were less than 5% TRR. The results indicate that quinclorac is tightly bound to the loamy sand soil.

One confined rotational metabolism study from crops rotated after flooded and non-flooded rice grown on silty clay was available. Quinclorac [2, 3, 4-^{14}C] was applied to flooded and non-flooded rice (primary crop) at a rate of 0.84 kg ai/ha in Mississippi, USA. After harvest of mature rice, the first rotational crops (wheat, mustard green and turnips) were planted 120 DAT followed by the second crops (sorghum, mustard green, soya beans and turnip) 360 DAT. The extractable radioactive residues were analysed for quinclorac and the metabolite 3-chloroquinoline-8-carboxylic acid (BH 514-1).

For the first rotational crops, maximum TRRs were 0.028 mg eq/kg for mustard plant, wheat seed, 0.025 mg eq/kg and turnip plant, 0.012 mg eq/kg. For the annual rotational crops, maximum TRRs were 0.014 mg eq/kg for mustard top, soya bean seed 0.017 mg eq/kg and for root and turnip root, 0.02 mg eq/kg. The metabolism of quinclorac by soya bean was qualitatively similar, although up to 62% TRR (0.01 mg eq/kg) was not extractable.

Quinclorac was the only major residue (>10% TRR but less than 0.05 mg eq/kg) detected in the examined rotational crops. Furthermore in the first rotational crops as well as the second rotational crops, TRRs were higher from crops grown under non-flooded conditions.

Another confined rotational metabolism study with one interval (120 days) was also available from crops planted after sorghum. Treatment levels to sorghum plants with 3-^{14}C-quinclorac were 0.53 kg ai/ha pre-emergence and 0.50 kg ai/ha post-emergence giving a total of 1.03 kg ai/ha (2 times GAP). The rotational crops mustard green, turnip and barley were planted 120 days after the last treatment of sorghum. The parent quinclorac was the major (up to 0.1 mg/kg) residue in all matrices. Quinclorac methyl ester was a minor metabolite below 5% in mustard green, turnip roots, and barley.

One field rotational crop study with rape seed planted after barley treated at 0.2 kg ai/ha the previous year was available. The application rate was below -25% critical GAP for cereals (0.29 kg ai/ha, wheat). The residues in rape seed at harvest analysed for parent quinclorac were below the LOQ of 0.05 mg/kg.

In the confined rotational studies, uptake of quinclorac and quinclorac methyl ester was observed in both first and second rotational crops. Residues were no more than 0.01 mg/kg (0.012 mg eq/kg) at the GAP rate.

In summary quinclorac is persistent in some soils and the amount, dependent on the season; residues from quinclorac in rotational crops may be found but generally at levels <.05 mg/kg.

Methods of residue analysis

The Meeting received analytical methods for the analysis of quinclorac residues in plant and animal matrices.

The extraction in lactating goat and laying hen was with acetone/0.1M NaOH. After clean-up, residues of parent quinclorac are determined by GC-ECD. The method is suitable for measuring residues of quinclorac in animal commodities with a LOQ of 0.05 mg/kg. It is not clear whether identified quinclorac represents quinclorac only or also includes quinclorac released from conjugates by the alkaline extraction method used.

The extraction in strawberry was with 1% acetic acid, in rice and wheat with acetone/0.1 M NaOH, in rape seed with acetone. After clean-up, residues of parent quinclorac in wheat, sorghum, rape seed, and strawberry were determined by HPLC-MS/MS or GC-ECD. Methods used for analysis of quinclorac in cereals may hydrolyse any quinclorac conjugates present. The LOQ ranged between 0.01–0.05 mg/kg.

The metabolite quinclorac methyl ester identified as a metabolite in rape seed and sorghum matrices is extracted with acetone and after clean-up determined by HPLC-MS/MS. The LOQ was 0.05 mg/kg.

A radiovalidation study showed that extraction with acetone/0.1 M NaOH converts quinclorac methyl ester partly into parent compound. For this reason, the parent is overestimated in samples containing quinclorac methyl ester. Methods D9708/1 (quinclorac) and R0036 (quinclorac) use acetone/0.1 M NaOH and are therefore not suitable of the determination of parent compound in oilseed rape seed and possibly other pulses and oilseeds, where the quinclorac methyl ester can be expected to be present.

In summary analytical methods are available for determining parent quinclorac in plant (cereals and fruit) and animal (lactating goat and hen) matrices and for the quinclorac methyl ester in plant (fruit and sorghum) matrices. However the methods for animal and cereal commodities use a hydrolysis step; indicating that the quinclorac residues measured may actually include quinclorac released from conjugates. Current analytical methods presented for oil seed rape are likely to overestimate quinclorac residues as the determination of quinclorac may also include some of its methyl ester.

Stability of residues in stored analytical samples

The Meeting received information on the storage stability of quinclorac and quinclorac methyl ester in plant matrices. Quinclorac (> 80% of spiked levels remained) was stable in rice and sorghum matrices for 38 months, in wheat grain for 26 months, and in cranberry fruit for 14 months. For quinclorac and quinclorac methyl ester no significant degradation was observed within 22 months in oilseed meal and oil.

For animal matrices no storage stability studies were provided.

Definition of the residue

In wheat and rice the parent quinclorac is the major residue present (above 80% TRR). Glucose conjugates, hydroxylated conjugates of quinclorac and hydroxyquinclorac were identified as minor metabolites (< 10% TRR) in wheat. In sorghum parent was also the major (> 73% TRR) residue present. The metabolite quinclorac methyl ester was also present (< 6% TRR) in sorghum.

In rape seed besides the parent, the metabolite quinclorac methyl ester was found as a significant metabolite (37%TRR).

In strawberry the parent quinclorac was the major residue present (> 98% TRR). Quinclorac methyl ester accounted for 9.6% TRR in fruit at the first harvest and was not detected in the third harvest

In rotational crop studies including mustard, barley and turnip in first rotation, uptake of residues identified as quinclorac (major) and quinclorac methyl ester (minor) was observed when analysed and resulted in residues near the LOQ at GAP rate.

Thus based on available metabolism data parent quinclorac is the major residue in examined crops. The metabolite quinclorac methyl ester was a significant residue in rape seeds and was a minor residue in other primary and subsequent rotational crops analysed.

Analytical methods are available for determining parent quinclorac in plant (cereals and fruit) and quinclorac methyl ester in fruit and sorghum matrices.

Current analytical methods determining quinclorac and quinclorac methyl ester in rape seed is not suitable as they overestimate the level of parent present.

Taking into account that the methodology measuring quinclorac is also accounting for conjugates derived from hydrolysis during the extraction process, and that quinclorac is the major residue measured in plants, the Meeting decided that the residue definition should be as follows:

Definition of the residue for compliance with MRL for plant commodities: Quinclorac plus quinclorac conjugates

The Meeting noted that quinclorac methyl ester has a toxicological potency up to 10 times that of quinclorac and decided to include it in the residue definition for dietary intake.

Definition of the residue for estimating dietary intake for plant commodities: Quinclorac plus quinclorac conjugate plus quinclorac methyl ester expressed as quinclorac

In calculating residue values for dietary intake estimation the Meeting agreed to use the following formula: residues = (quinclorac +conjugate) + 10 × quinclorac methyl ester.

In lactating goat the major residue was quinclorac and the highest residues were found in liver and kidney with small amounts of other metabolite also found (less than 5% TRR).

For laying hen, the available data show that quinclorac is the only major residue in tissues and eggs.

In both species, measurement of the parent in the metabolism studies probably also includes conjugates of quinclorac as the extraction method used strong acid or alkali. This conclusion is supported by partitioning of residues in the animal feeding studies where quinclorac residues are more than ten times higher in fat tissue compared to muscle tissue.

The Meeting noted however that quinclorac residue was more than ten times higher in fat tissue compared to muscle tissue.

For quinclorac, a log Kow of -0.72 at pH 7 was reported suggesting residues of free quinclorac are water soluble.

The fact that the residue is generally found in the fat suggests that the actual tissue residue is not the parent molecule but may be a fatty acid conjugate of quinclorac.

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

Definition of the residue for compliance with MRL and estimating the dietary intake for animal commodities: Quinclorac plus quinclorac conjugates.

The residue is fat soluble

Results of supervised residue trials on crops

Quinclorac is registered for use as a herbicide in many countries. The Meeting received supervised trial data for foliar application of quinclorac to rice, wheat, rape seed, sorghum, cranberry and rhubarb. The trials were conducted in USA and Canada. Frozen samples from the trials presented are covered by storage stability studies. The residue trials did not measure the methyl ester required for estimating dietary intakes.

The Meeting noted quinclorac methyl ester in oilseed equal level to quinclorac in the rape metabolism study and for cereals and fruit at levels up to10 percent of the parent, and agreed to use to the following formula to estimate levels for use in dietary intake calculations:

Plants except oilseed:

HR/STMR = (quinclorac + conjugate) + 10 x 0.1 (quinclorac + conjugate) = 2 × (quinclorac + conjugate)

Oil seed:

HR/STMR = (quinclorac + conjugate) + 10 x (quinclorac + conjugate) = 11 × (quinclorac + conjugate)

Cranberry

Data from supervised trials on cranberry from USA were presented to the Meeting. The critical GAP in USA is two foliar post-emergent applications of 0.28 kg ai/ha, with a 30 day interval and a PHI of 60 days.

In four independent trials from USA matching the critical GAP residues of quinclorac in cranberry fruit for MRL estimation were (n=4): 0.16, 0.17, 0.18, 0.67 mg/kg. The highest residue of 0.68 mg/kg was measured in an individual cranberry sample.

Residues for dietary intake estimation in cranberry fruit were (n=4): 0.32, 0.34, 0.36 and 1.34 mg/kg

Based on a data set from USA the Meeting estimated a maximum residue level, an STMR value and an HR value for quinclorac in cranberry fruit of 1.5 mg/kg, 0.35 mg/kg and 1.36 mg/kg, respectively.

Rhubarb

Data from supervised trials on rhubarb from USA were presented to the Meeting. The critical GAP in USA is two foliar post-emergence applications of 0.42 kg ai/ha, with a 30 day interval and a PHI of 30 days.

In three independent trials from USA matching the critical GAP residues in rhubarbs for MRL estimation were (n=3) 0.11, 0.18, 0.21 mg/kg. The highest residue of 0.23 mg/kg was measured in an individual rhubarb sample.

Residues for dietary intake estimation in rhubarbs were (n=3): 0.22, 0.36 and 0.42 mg/kg.

Based on a data set from USA the Meeting estimated a maximum residue level, an STMR value and an HR value for quinclorac in rhubarb of 0.5 mg/kg, 0.36 mg/kg and 0.46 mg/kg, respectively.

Rice

Data from supervised trials on rice from USA were presented to the Meeting. The critical GAP in USA is one application of 0.29-0.54 kg ai/ha and a PHI of 40 days. The use can be soil application, pre-planting or pre-emergence (dryland rice) or post-emergence broadcast application after the 2-leaf stage (but before heading) on dryland and water seeded rice. Only six trials matched the GAP and an estimation of maximum residue level was not made.

Wheat

Data from supervised trials on wheat from USA and Canada were presented to the Meeting. The critical GAP in Canada is one post-emergent foliar application of 0.135 kg ai/ha and a PHI of 80 days. Only six trials matched the GAP and an estimation of maximum residue level was not made.

Sorghum grain

Data from supervised trials from USA were presented to the Meeting. The critical GAP is one application pre- and /or post-emergence (at maximum 12 cm height limit) as long as the seasonal maximum amount of 0.7 kg ai/ha is not exceeded. The maximum post-emergent application rate is 0.56 kg ai/ha. The trials did not match the critical GAP and an estimation of maximum residue level was not made.

Rape seed (canola)

A registered use with a supporting label from Canada was presented with one foliar application at 2-6 leaf stage of 0.1 kg ai/ha and a PHI of 60 days. Data from seventeen independent supervised trials from Canada (16) and USA (1) supporting this GAP were presented to the Meeting.

The analytical method used in the trials method D9708/1 for determining quinclorac and method D9806 for determining quinclorac methyl ester (BH514-Me) overestimates the level of the parent. Therefore the trials cannot be used for estimating the maximum residue level.

Animal feeds

Strawberry and rhubarbs are not used as animal feeds.

Fate of residues during processing

The Meeting received information on the fate of incurred residues of quinclorac during the processing of rice, wheat, rape seed and sorghum. Supporting trials with matching GAPs were not available and therefore the studies were not considered by the current Meeting.

Residues in animal commodities

Farm animal feeding studies

The Meeting received feeding studies on residue levels of quinclorac plus quinclorac conjugates in laying hens and lactating cows.

For lactating cows three groups of were dosed daily at levels of 1, 10, 50, or 500 ppm in the diet (0.002, 0.02, 0.09 and 0.9 mg/kg bw) for 28 consecutive days.

In milk residues were only detected in the 500 ppm group. A plateau level was reached in this group after 4 days (mean: 0.032 mg/kg).

In muscle residues were only detected in the 500 ppm group, 0.01–0.037 mg/kg (mean: 0.027 mg/kg).

In fat two different tissues were analysed (peritoneal and subcutaneous fat). The highest residues were found in subcutaneous fat with < 0.01–0.013 (mean: 0.005 mg/kg) for the 1 ppm group, < 0.01 mg/kg for the 10 ppm group. In peritoneal fat with < 0.01–0.01 mg/kg for the 1 ppm group, < 0.01–0.023 mg/kg for the 10 ppm group.

In liver residues were < 0.01–0.01 mg/kg for the 1 ppm group, 0.01-0.02 mg/kg for the 10 ppm group.

In kidney residues were < 0.01–0.016 mg/kg for the 1 ppm group, 0.062-0.082 mg/kg for the 10 ppm group.

For laying hens three groups of animals were dosed with rates of 1, 10 and 100 ppm by dry weight in the feed (0.07, 0.7 and 7 mg/kg bw/day) for 28 consecutive days. Eggs were collected throughout the whole study and tissues were collected on day 29 after the last dose.

In eggs a clear plateau level was not reached in any dosing group. For the 1 and 10 ppm the residues were below 0.01 mg/kg during the whole experiment.

In dark and light muscle residues were 0.0–0.005 mg/kg (max mean: 0.002 mg/kg) for the 1 ppm group.

In skin + fat total residues in fat for the 1 ppm group was 0.0–0.018 mg/kg.

In liver residues were: 0.0–0.009 mg/kg for the 1 ppm group. In kidney residues were 0.002–0.059 mg/kg for the 1 ppm group.

Animal commodities residue levels estimation

Strawberry and rhubarbs are not used as animal feed and therefore estimation of residue levels was not made for animal commodities.

RECOMMENDATIONS

On the basis of the data from supervised residue trials the Meeting concluded that the residue levels listed in Annex 1 are suitable for establishing maximum residue limits and for the IEDI and IESTI assessment.

Definition of the residue for compliance with MRL for plant commodities: quinclorac plus quinclorac conjugates

Definition of the residue for estimating dietary intake: quinclorac plus quinclorac conjugate plus quinclorac methyl ester expressed as quinclorac

Definition of the residue for compliance with MRL and estimating the dietary intake for animal commodities: quinclorac plus quinclorac conjugates

The residue is fat soluble.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FB 0265	Cranberry	1.5		0.35	1.36
VS 0627	Rhubarb	0.5		0.36	0.46

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intake of quinclorac for the 17 GEMS/Food regional diets based on estimated STMRs were 0% of the maximum ADI of 0.4 mg/kg bw for the sum of quinclorac, its conjugates plus 10× quinclorac methyl ester, expressed as quinclorac (see Annex 3 of the 2015 Report). The Meeting concluded that the long-term dietary intake of residues of quinclorac is unlikely to present a public health concern.

Short-term intake

The International Estimated Short Intake (IESTI) for quinclorac was calculated for commodities for which STMRs or HRs were estimated and for which consumption data were available. The results are shown in Annex 4 to the 2015 Report. The ARfD for quinclorac, its conjugates plus 10 × quinclorac

methyl ester, expressed as quinclorac is 2 mg/kg bw and the IESTIs varied from 0–1% of the ARfD for children and the general population.

The Meeting concluded that the short-term intake of residues of quinclorac when used in ways that have been considered by the JMPR is unlikely to present a public health concern.

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1995/7004167	Versoi P.L. McDonell J	1995	Magnitude of the residues of Quinclorac in spring wheat when treated with Quinclorac or Quinclorac plus Difenzoquat: 1994 Canadian field project. Unpublished
1996/5208	Versoi P.L. et al.	1996	The magnitude of Quinclorac residues in the spring wheat processed fraction wheat germ. Unpublished
1996/5136	Versoi P.L.McDonell J	1996	The magnitude of Quinclorac residues in a grain sorghum use pattern for field bindweed control. Unpublished
029602-1	Walsh, K.	2015	Strawberry Metabolism of [^{14}C] Quinclorac, Unpublished
J20044	White G	2015	Validation of GC Laboratories Analytical Method M829/A
1987/5037	Winkler V., Brown M	1987	Confined accumulation study of ^{14}C-BAS 514 H residues in fall and spring rotational crops. Unpublished.

Code	Author	Year	Title, Institute, Report reference
1988/5059	Wood N.F.	1988	Metabolism of BAS 514 in rice. Unpublished
1991/5009	Wood N.F, WinklerV.W	1991	Further identification studies on Quinclorac aerobic soil/sediment metabolites. Unpublished
1997/5051	Zehr R.D.,Riley M.E.	1997	The magnitude of Quinclorac residues in rice treated forty days pre-harvest. Unpublished

SPIROTETRAMAT (234)

The first draft was prepared by Professor Arpad Ambrus, Hungarian Food Chain Safety Office, Budapest Hungary

EXPLANATION

The compound was evaluated by the JMPR for the first time in 2008. The Meeting established an ADI of 0–0.05 mg/kg bw per day and an ARfD of 1 mg/kg/bw and defined the residues as follow:

Residue for enforcement plant commodities: spirotetramat plus spirotetramat enol, expressed as spirotetramat.

Residue for dietary intake plant commodities: *spirotetramat plus the metabolites enol, ketohydroxy, enol glucoside, and monohydroxy, expressed as spirotetramat*.

Residue for enforcement and dietary intake animal commodities: *spirotetramat enol, expressed as spirotetramat*.

The residue is not fat soluble.

The Meeting estimated residue levels for a number of commodities. Additional residue data were evaluated by the 2011 Meeting. Subsequently, the recommendations, including several animal feed commodities, were adopted as Codex MRLs except those for strawberry, avocado and guava.

The manufacturer provided new supervised trial data in avocado, guava and sweet corn and corresponding labels for the evaluation by the 2015 JMPR.

METHODS OF RESIDUE ANALYSIS

Several analytical methods were developed for the residue analysis of spirotetramat in different matrices.

The analytical method 00857 used to measure residues of spirotetramat (STM) and its metabolites, STM-enol, STM-ketohydroxy, STM-mono-hydroxy and STM-enol-Glc was evaluated by the JMPR in 2008. This method was applied with minor modifications for determination of residues in guava, avocado and sweet corn on cob husk removed, sweet corn forage and fodder. The residues were extracted with an acidic acetonitrile/water mixture (4/1,v/v) filtered and quantitated by high performance liquid chromatography/triple stage quadrupole mass spectrometry (LC/MS/MS) using stable isotopically labelled internal standards. The individual analyte derived residues were converted to spirotetramat equivalents and summed up to yield the total residue of BYI08330 calc.1. Additionally the sum of spirotetramat and STM cis-enol was calculated. The limit of quantitation (LOQ) for each analyte was 0.01 mg/kg (expressed as parent equivalents), the LOQ for the total residue was 0.05 mg/kg and was 0.02 mg/kg for the sum of spirotetramat and BYI08330 enol.

The recoveries obtained during the validation of the method and analysis of supervised trial samples are summarized in Tables 1-6.

Table 1 Recoveries for STM, STM-enol, STM-ketohydroxy, STM-mono-hydroxy and STM-enol-Glc in/on guava fruit

Study Trial No. Year	STM, metabolite	n	Spike level (mg/kg)	Recovery (%)				
				Individual recoveries	Min	Max	Mean	RSD
RAFNP042 FN075-07BA-B FN075-07BA-A1 FN075-07BA-A2 FN076-07HA-A1 FN076-07HA-A2 2007/2008	STM	3	0.01	103; 102; 93	93	103	99	5.5
		3	1.0	95; 111; 110	95	111	105	8.5
		6	overall		93	111	102	7.3
	STM cis-enol	3	0.01	102; 104; 98	98	104	101	3.0
		3	1.0	112; 80; 85	80	112	92	18.6
		6	overall		80	112	97	12.5
	STM cis-keto-hydroxy	3	0.01	104; 90; 94	90	104	96	7.5
		3	1.0	105; 115; 113	105	115	111	4.8

Study Trial No. Year	STM, metabolite	n	Spike level (mg/kg)	Recovery (%)				
				Individual recoveries	Min	Max	Mean	RSD
		6	overall		90	115	104	9.6
	STM mono-hydroxy	3	0.01	94; 112; 96	94	112	101	9.8
		3	1.0	103; 110; 110	103	110	108	3.8
		6	overall		94	112	104	7.4
	STM enol-glucoside	3	0.01	78; 74; 75	74	78	76	2.8
		3	1.0	72; 88; 87	72	88	82	10.9
		6	overall		72	88	79	8.7
RAFNL058 FN002-11DB-A FN002-11DB-B FN002-11DB-C FN002-11DB-D FN003-11DA-A FN003-11DA-B FN003-11DA-C FN003-11DA-D 2011	STM	10	0.01	102; 99; 97; 97; 101; 102; 101; 79; 80; 73	73	102	93	12.0
		5	4.0	85; 97; 87; 99; 93	85	99	92	6.6
		15	overall		73	102	93	10.3
	STM cis-enol	10	0.01	103; 93; 92; 102; 103; 117; 96; 97; 111; 73	73	117	99	12.1
		5	4.0	86; 97; 71; 71; 71	71	97	79	15.0
		15	overall		71	117	92	16.2
	STM cis-keto-hydroxy	10	0.010	103; 100; 95; 111; 98; 106; 98; 110; 93; 86	86	111	100	7.8
		5	4.0	108; 108; 93; 118; 106	93	118	107	8.4
		15	overall		86	118	102	8.3
	STM mono-hydroxy	10	0.01	105; 99; 102; 101; 113; 101; 108; 106; 74; 94	74	113	100	10.6
		5	4.0	95; 89; 113; 106; 98	89	113	100	9.4
		15	overall		74	113	100	9.9
	STM enol-glucoside	10	0.01	105; 97; 86; 96; 94; 99; 93; 96; 102; 92	86	105	96	5.6
		5	4.0	90; 86; 85; 84; 91	84	91	87	3.6
		15	overall		84	105	93	6.8

STM: spirotetramat

Table 2 Recoveries for STM, STM-enol, STM-ketohydroxy, STM-mono-hydroxy and STM-enol-Glc in/on avocado fruit

Study Trial No. Year	Spirotetramat, metabolite	n	Spike level (mg/kg)	Recovery (%)				
				Individual recoveries	Min	Max	Mean	RSD
RAFNP042 FN070-07BA-A1 FN070-07BA-A2 FN070-07BA-B FN073-07DA-A1 FN071-07DA-A2 FN072-07HA-A1 FN072-07HA-A2 FN073-07BB-A1 FN073-07BB-A2 FN073-07BB-B FN074-07HA-A1 FN074-07HA-A2 2008	STM	10	0.01	110;101;93;97;91;97;98;111;104;109	91	111	101	7.1
		2	0.10	97;88	88	97	93	
		3	0.50	90;97;96	90	97	94	4.0
		15	overall		88	111	99	7.3
	STM cis-enol	10	0.01	79;78;79;88;76;86; 87;86;86;85	76	88	83	5.4
		2	0.10	90;98	90	98	94	
		3	0.50	77;88;88	77	88	84	7.5
		15	overall		76	98	85	7.0
	STM cis-keto-hydroxy	10	0.01	84;80;86;94;88;82; 109;95;84;100	80	109	90	10.2
		2	0.10	107;91	91	107	99	
		3	0.50	105;101;97	97	105	101	4.0
		15	overall		80	109	94	10.1
	STM mono-hydroxy	10	0.01	103;109;97;96;75;72;69;96;96;78	69	109	89	15.9
		2	0.10	101;96	96	101	99	
		3	0.50	92;101;108	92	108	100	8.0
		15	overall		69	109	93	13.9
	STM enol-glucoside	10	0.01	102;94;90;75;92;79; 73;111;88;107	73	111	91	14.3
		2	0.10	90;89	89	90	90	
		3	0.50	91;87;92	87	92	90	2.9
		15	overall		73	111	91	11.6

STM: spirotetramat

Table 3 Recoveries for STM, STM-enol, STM-ketohydroxy, STM-mono-hydroxy and STM-enol-Glc in/on sweet corn ear without husk

Study Trial No. Year	Spirotetramat, metabolite	n	Spike level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
AAFC09-027R AAFC09-027R-116 AAFC09-027R-117 AAFC09-027R-118 AAFC09-027R-119 AAFC09-027R-120 AAFC09-027R-121 AAFC09-027R-122 AAFC09-027R-123 2009	STM	4	0.010	87.4; 81.5; 91.2; 81.9	81.5	91.2	85.5	5.4
		1	0.10	94.6	94.6	94.6	94.6	
		1	1.0	106.5	106.5	106.5	106.5	
		6	overall		81.5	106.5	90.5	10.3
	STM cis-enol	4	0.010	102.0; 99.5; 101.5; 99.8	99.5	102.0	100.7	1.2
		1	0.10	105.0	105.0	105.0	105.0	
		1	1.0	108.5	108.5	108.5	108.5	
		6	overall		99.5	108.5	102.7	3.4
	STM cis-keto-hydroxy	4	0.010	98.8; 97.4; 91.8; 104.5	91.8	104.5	98.1	5.3
		1	0.10	95.4	95.4	95.4	95.4	
		1	1.0	115	115.0	115.0	115.0	
		6	overall		91.8	115.0	100.5	8.2
	STM mono-hydroxy	4	0.010	91.2; 83.0; 94.8; 73.9	73.9	94.8	85.7	10.9
		1	0.10	98.4	98.4	98.4	98.4	
		1	1.0	98.7	98.7	98.7	98.7	
		6	overall		73.9	98.7	90.0	10.9
	STM enol-glucoside	4	0.010	97.6; 95.5; 98.5; 95.0	95.0	98.5	96.7	1.7
		1	0.10	98.4	98.4	98.4	98.4	
		1	1.0	108.0	108.0	108.0	108.0	
		6	overall		95.0	108.0	98.8	4.8
AAFC09-027R AAFC09-027R-116 AAFC09-027R-117 AAFC09-027R-118 AAFC09-027R-119 AAFC09-027R-120 AAFC09-027R-121 AAFC09-027R-122 AAFC09-027R-123 GLP: yes 2009	STM	4	0.010	84.1; 84.9; 80.3; 86.4	80.3	86.4	83.9	3.1
		2	0.10	86.0; 84.4	84.4	86.0	85.2	
		1	1.0	92.8	92.8	92.8	92.8	
		7	overall		80.3	92.8	85.6	4.4
	STM cis-enol	4	0.010	97.1; 97.3; 95.5; 93.5	93.5	97.3	95.9	1.8
		2	0.10	99.5; 99.8	99.5	99.8	99.7	
		1	1.0	93.3	93.3	93.3	93.3	
		7	overall		93.3	99.8	96.6	2.7
	STM cis-keto-hydroxy	4	0.010	95.8; 91.3; 91.1; 97.9	91.1	97.9	94.0	3.6
		2	0.10	93.5; 92.9	92.9	93.5	93.2	
		1	1.0	92.0	92.0	92.0	92.0	
		7	overall		91.1	97.9	93.5	2.7
	STM mono-hydroxy	4	0.010	82.5; 73.3; 73.4; 74.9	73.3	82.5	76.0	5.8
		2	0.10	95.4; 75.5	75.5	95.4	85.5	
		1	1.0	88.7	88.7	88.7	88.7	
		7	overall		73.3	95.4	80.5	10.8
	STM enol-glucoside	4	0.010	92.4; 99.0; 101.5; 95.0	92.4	101.5	97.0	4.2
		2	0.10	94.4; 94.7	94.4	94.7	94.6	
		1	1.0	93.2	93.2	93.2	93.2	
		7	overall		92.4	101.5	95.7	3.4
BCS-0272 B000-T2 GLP: yes 2008	STM	3	0.02	81;85;100	81	100	89	11.3
		3	1	87;95;85	85	95	89	5.9
		6	overall		81	100	89	8.1
	STM cis-enol	3	0.024	94;97;97	94	97	96	1.8
		3	1.2	96;96;95	95	96	96	0.6
		6	overall		94	97	96	1.2
	STM cis-keto-hydroxy	3	0.024	120;116;106	106	120	114	6.3
		3	1.2	86;90;88	86	90	88	2.3
		6	overall		86	120	101	14.9
	STM mono-hydroxy	3	0.024	99;92;98	92	99	96	3.9
		3	1.2	87;91;93	87	93	90	3.4
		6	overall		87	99	93	4.8
	STM enol-glucoside	3	0.016	100;89;109	89	109	99	10.1
		3	0.8	102;94;93	93	102	96	5.1

Study Trial No. Year	Spirotetramat, metabolite	n	Spike level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
		6	overall		89	109	98	7.4

Table 4 Recoveries for STM, STM-enol, STM-ketohydroxy, STM-mono-hydroxy and STM-enol-Glc in/on sweet corn fodder.

Study Trial No. Year	Spirotetramat, metabolite	n	Spike level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
BCS-0272 B000-T2 GLP: yes 2008	STM	3	0.02	92;92;80	80	92	88	7.9
		3	1.0	86;90;89	86	90	88	2.4
		6	overall		80	92	88	5.2
	STM cis-enol	3	0.024	99;100;90	90	100	96	5.7
		3	1.2	90;83;91	83	91	88	5.0
		6	overall		83	100	92	6.9
	STM cis-keto-hydroxy	3	0.024	86;103;102	86	103	97	9.8
		3	1.2	86;77;83	77	86	82	5.6
		6	overall		77	103	90	11.8
	STM mono-hydroxy	3	0.024	83;97;89	83	97	90	7.8
		3	1.2	87;81;85	81	87	84	3.6
		6	overall		81	97	87	6.5
	STM enol-glucoside	3	0.016	102;106;91	91	106	100	7.8
		3	0.8	93;88;95	88	95	92	3.9
		6	overall		88	106	96	7.2
BCS-0319 C457-T2 C458-T2 C459-T2 2009	STM	3	0.02	90;111;94	90	111	98	11.3
		3	1	94;83;88	83	94	88	6.2
		6	overall		83	111	93	10.3
	STM cis-enol	3	0.024	73;75;84	73	84	77	7.6
		3	1.2	88;87;94	87	94	90	4.2
		6	overall		73	94	84	9.7
	STM cis-keto-hydroxy	3	0.024	114;114;108	108	114	112	3.1
		3	1.2	89;88;93	88	93	90	2.9
		6	overall		88	114	101	12.2
	STM mono-hydroxy	3	0.024	93;84;74	74	93	84	11.4
		3	1.2	84;82;84	82	84	83	1.4
		6	overall		74	93	84	7.3
	STM enol-glucoside	3	0.016	109;102;111	102	111	107	4.4
		3	0.8	84;83;85	83	85	84	1.2
		6	overall		83	111	96	13.7
BCS-0322 AUS-BCS-0322-C471-A AUS-BCS-0322-C472-A AUS-BCS-0322-C473-A 2010	STM	3	0.02	84;85;88	84	88	86	2.4
		3	1.0	98;96;88	88	98	94	5.6
		6	overall		84	98	90	6.5
	STM cis-enol	3	0.024	97;87;81	81	97	88	9.2
		3	1.2	97;95;90	90	97	94	3.8
		6	overall		81	97	91	7.0
	STM cis-keto-hydroxy	2	0.024	109;89	89	109	99	
		3	1.2	94;86;87	86	94	89	4.9
		5	overall		86	109	93	10.2
	STM mono-hydroxy	2	0.024	98;77	77	98	88	
		3	1.2	108;99;95	95	108	101	6.6
		5	overall		77	108	95	11.9
	STM enol-glucoside	3	0.016	99;109;81	81	109	96	14.7
		3	0.8	101;103;95	95	103	100	4.2
		6	overall		81	109	98	9.7

Study Trial No. Year	Spirotetramat, metabolite	n	Spike level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
BCS-0322 AUS-BCS-0322-C471-A AUS-BCS-0322-C472-A AUS-BCS-0322-C473-A 2010	STM	3	0.02	102;87;74	74	102	88	16.0
		3	1.0	85;88;85	85	88	86	2.0
		6	overall		74	102	87	10.3
	STM cis-enol	3	0.024	99;83;71	71	99	84	16.7
		3	1.2	84;98;86	84	98	89	8.5
		6	overall		71	99	87	12.0
	STM cis-keto-hydroxy	3	0.024	92;106;78	78	106	92	15.2
		3	1.2	81;87;86	81	87	85	3.8
		6	overall		78	106	88	11.2
	STM mono-hydroxy	3	0.024	85;108;93	85	108	95	12.2
		3	1.2	94;103;96	94	103	98	4.8
		6	overall		85	108	97	8.4
	STM enol-glucoside	3	0.016	96;99;79	79	99	91	11.8
		3	0.8	96;100;92	92	100	96	4.2
		6	overall		79	100	94	8.2

STM: Spirotetramat

Table 5 Recoveries for STM, STM-enol, STM-ketohydroxy, STM-mono-hydroxy and STM-enol-Glc in/on sweet corn forage

Study Trial No. Year	Spirotetramat, metabolite	n	Spike level (mg/kg)	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
AAFC09-027R AAFC09-027R-116 AAFC09-027R-117 AAFC09-027R-118 AAFC09-027R-119 AAFC09-027R-120 AAFC09-027R-121 AAFC09-027R-122 AAFC09-027R-123 2009	STM	4	0.010	93.3;90.6;81.1;85.0	81.1	93.3	87.5	6.3
		1	0.10	92.9	92.9	92.9	92.9	
		1	0.50	101.5	101.5	101.5	101.5	
		6	overall		81.1	101.5	90.7	7.8
	STM cis-enol	4	0.010	94.8; 94.0; 94.0; 96.5	94.0	96.5	94.8	1.2
		1	0.10	95.7	95.7	95.7	95.7	
		1	0.50	99.3	99.3	99.3	99.3	
		6	overall		94.0	99.3	95.7	2.1
	STM cis-keto-hydroxy	4	0.010	93.0; 93.3; 93.2; 102.5	93.0	102.5	95.5	4.9
		1	0.10	92.8	92.8	92.8	92.8	
		1	0.50	98.6	98.6	98.6	98.6	
		6	overall		92.8	102.5	95.6	4.2
	STM mono-hydroxy	4	0.010	95.0; 98.9; 83.3; 86.7	83.3	98.9	91.0	7.9
		1	0.10	97.3	97.3	97.3	97.3	
		1	0.50	98.2	98.2	98.2	98.2	
		6	overall		83.3	98.9	93.2	7.1
	STM enol-glucoside	4	0.010	96.0; 95.5; 95.3; 94.1	94.1	96.0	95.2	0.8
		1	0.10	93.5	93.5	93.5	93.5	
		1	0.50	98.7	98.7	98.7	98.7	
		6	overall		93.5	98.7	95.5	1.9

Table 6 Recoveries for STM, STM-enol, STM-ketohydroxy, STM-mono-hydroxy and STM-enol-Glc in/on sweet corn stover

Study Trial No. Year	Spirotetramat, metabolite	n	Spike level mg/kg	Recovery (%) Individual recoveries	Min	Max	Mean	RSD
AAFC09-027R AAFC09-027R-116 AAFC09-027R-117	Spirotetramat,	4	0.010	84.1; 84.9; 80.3; 86.4	80.3	86.4	83.9	3.1
		2	0.10	86.0; 84.4	84.4	86.0	85.2	
		1	1.0	92.8	92.8	92.8	92.8	
		7	overall		80.3	92.8	85.6	4.4
	STM cis-enol	4	0.010	97.1; 97.3; 95.5; 93.5	93.5	97.3	95.9	1.8

Study Trial No. Year	Spirotetramat, metabolite	n	Spike level mg/kg	Recovery (%)				
				Individual recoveries	Min	Max	Mean	RSD
AAFC09-027R-118 AAFC09-027R-119 AAFC09-027R-120 AAFC09-027R-121 AAFC09-027R-122 AAFC09-027R-123 2009		2	0.10	99.5; 99.8	99.5	99.8	99.7	
		1	1.0	93.3	93.3	93.3	93.3	
		7	overall		93.3	99.8	96.6	2.7
	STM cis-keto-hydroxy	4	0.010	95.8; 91.3; 91.1; 97.9	91.1	97.9	94.0	3.6
		2	0.10	93.5; 92.9	92.9	93.5	93.2	
		1	1.0	92.0	92.0	92.0	92.0	
		7	overall		91.1	97.9	93.5	2.7
	STM mono-hydroxy	4	0.010	82.5; 73.3; 73.4; 74.9	73.3	82.5	76.0	5.8
		2	0.10	95.4; 75.5	75.5	95.4	85.5	
		1	1.0	88.7	88.7	88.7	88.7	
		7	overall		73.3	95.4	80.5	10.8
	STM enol-glucoside	4	0.010	92.4; 99.0; 101.5; 95.0	92.4	101.5	97.0	4.2
		2	0.10	94.4; 94.7	94.4	94.7	94.6	
		1	1.0	93.2	93.2	93.2	93.2	
		7	overall		92.4	101.5	95.7	3.4

Stability of residues in stored analytical samples

Individual data on storage stability of spirotetramat and its metabolites were evaluated by the JMPR in 2008. No new information was provided.

The 2008 Meeting concluded that spirotetramat, when determined as the sum of spirotetramat and its enol, was stable (≥ 80% remaining) for 2 years in tomato, potato, lettuce, almond nutmeat, climbing French beans and tomato paste on various commodities stored frozen for intervals typical of storage prior to analysis. Considered alone, however, spirotetramat may show significant loss (to spirotetramat enol). Likewise, the metabolites spirotetramat enol, spirotetramat ketohydroxy, spirotetramat monohydroxy, spirotetramat enol Glc (glucoside) are stable.

No new information was provided.

USE PATTERN

The use patterns relevant for the residue data submitted for evaluation by the present meeting are summarized in Table 7. Spirotetramat 150 OD is an oil dispersible (OD) formulation containing 150 g ai/L; Spirotetramat 240 SC is a suspension concentrate (SC) formulation containing 240 g ai/L.

Table 7 Foliar spray application for spirotetramat on avocado, guava and sweet corn

Crop and/ country	Pests or Group of pests controlled	Formulation Type	Application					PHI (days)	Remarks:
			No. min max	Interval (min)	kg ai/hL min/ max	Water L/ha min/ max	kg ai/ha min/ max		
Avocado USA	Aphids Avocado thrips Mealybugs Scales Whiteflies	240SC	3	14		3000 max	0.146-0.179	1	Max. dose per season is 0.440 kg/ha
Avocado Mexico		150 OD	3	14	-	n.a.	0.14 0.171	not given	
Avocado Chile		100SC	2	not given	0.085-0.10	2000-3000		3	
Guava USA	Aphids. Avocado thrips Mealybugs Scales Whiteflies	150 OD/ 240 SC	3	14		3000 max	0.146-0.179	1	max. dose per season is 0.440 kg/ha

Crop and/ country	Pests or Group of pests controlled	Formu lation Type	Application					PHI (days)	Remarks:
			No. min max	Interval (min)	kg ai/hL min/ max	Water L/ha min/ max	kg ai/ha min/ max		
Sweet corn Australia	Corn aphid	240S C	1-2	min. 7		Min.200	0.048-0.072	7[a]	
Sweet corn Canada	Aphids	240 SC	3		0.0438		0.053-0.088	7/50[c]	/[b]

[a]: Do not graze or cut for stock food for 7 days after application

[b]: Max. annual rate 0.264 kg/ha

[c]: PHI is 50 days if the crop is being harvested for silage

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The residue trials were conducted with the two formulations OD 150 (150 g ai/L) and SC 240 (240 g ai/L). 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. Unless stated otherwise, residue data are recorded unadjusted for recovery.

Residues 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 GAP have been used for the estimation of maximum residue levels. Those results used for estimation of maximum residue levels and dietary intake calculations are underlined and double underlined, respectively.

Assorted tropical and sub-tropical fruits – edible peel

Guava

Four supervised field residue trials were conducted with spirotetramat on guava in Mexico in the growing seasons 2007/2008 (2) (Hoag, P.E. and Harbin, A.M. 2009) and 2011 (2) (Hoag, R.E., Fain, J. 2013). Each trial included several plots, where the application parameters varied (spray volume, SC or OD formulation, application rate). In total 13 plots were treated.

Three dilute or concentrated airblast applications of spirotetramat 150 OD were made to guava trees at a target rate of 0.15 kg ai/ha or 0.288 kg ai /ha/application. The actual application rates ranged from 0.147-0.153 and 0.274 to 0.309 g ai/ha/application. Side-by-side bridging trials conducted in 2008 and 2011 received three concentrated airblast applications of spirotetramat 240 SC or spirotetramat OD 150 at the same rate to confirm that the formulation type (OD or SC) does not have any effect on residue behaviour. Adjuvant Dyne Amic or Induce was included in all spray mixtures at a rate of 0.25% or 0.5%, respectively.

Samples of guava fruit were taken 1 and 3 days after last treatment in trials conducted during 2008 growing season. From the 2011 trials samples of guava fruit were taken on day 0, 1, 3, 7 and 12 (14) days after the last treatment. The two parallel samples of guava fruit were analysed using method 00857 with minor modifications. The maximum storage period of deep-frozen samples before analysis was 474 days (5.8 months), which is covered by the storage stability studies. Residues of spirotetramat (STM), STM cis-enol, STM cis-keto-hydroxy, BYI08330 monohydroxy, STM enol-glucoside were determined separately and each expressed as the parent compound. The sum of STM and cis-enol, as well as the sum of residues of STM and 4 metabolites were calculated and expressed as STM.

The full dataset on guava (including the two trials already described in 2010) is presented in Table 8.

Assorted tropical and sub-tropical fruits – inedible peel

Avocado

A total of 5 residue trials are available which were conducted with spirotetramat in avocado in the USA (2) (Hoag, P.E. and Harbin, A.M. 2009.), Chile (2) and Mexico (1) following three broadcast foliar spray applications (either diluted or concentrated spray) of spirotetramat. The nominal application rate per treatment was 0.288 kg ai/ha. Actual application rates for all plots ranged from 0.274 to 0.309 g ai/ha.

Side-by-side bridging plots were included that received three concentrated airblast applications of spirotetramat 240 SC at the rate of 0.288 to 0.272 kg ai/ha/application. The concentrated spray applications were made at spray volumes ranging from 364 to 686 L/ha and the dilute spray applications were made at spray volumes ranging from 1943 to 2839 L/ha. The intervals between applications ranged from 12 to 14 days. For all trials, the first application was made between BBCH 47 and 85. Adjuvant Dyne Amic or Induce was included in all spray mixtures at a rate of 0.25% or 0.5% respectively.

Samples of avocado fruit were taken 1 and 3 days after the last treatment. In one decline trial additional samples were taken on day 0, 5 and 7 days after the last application. The samples were analysed for the parent compound spirotetramat (STM) and its metabolites STM cis-enol, STM cis-keto-hydroxy, STM cis-enol-glucoside and STM 8330 cis-mono-hydroxy using method 00857 with the LOQ of 0.01 mg/kg for each analyte.

The maximum storage period of deep-frozen samples before analysis was 211 days which is covered by the storage stability studies reported by the previous Meeting.

Residue results are presented in Table 9.

Fruiting vegetables – other than cucurbits

Sweet corn

Eight trials on sweet corn were conducted in Canada during the 2009 growing season at about the maximum dose specified on the Canadian label (3 × 0.088 kg ai/ha at 7 days PHI and maximum seasonal rate of 0.264 kg ai/ha) (Lonsbary, S. 2011). Actual application rates ranged from 78 to 95 g as/ha/application, with re-treatment intervals of three to eight days and a PHI of 7 ± 1 days.

Seven trials on sweet corn were conducted in Australia during the growing season 2008 (1), 2009 (3) and 2010 (3) according to Australian label (2 × up to 0.072 kg ai/ha at 7 days interval and 7 day PHI) (Radunz, L. 2009. Radunz, L. 2010.). The actual application rates ranged from 0.015 to 0.08 kg ai/ha/application. The spray intervals between applications ranged from 6 to 9 days. A Hasten adjuvant was included in all spray mixtures at a rate of 0.5–1.0 L/ha.

Ear without husk and fodder samples were collected 6 to 9 days after the final application. Stover samples were collected 33 to 85 days after the last application, according to the normal harvest of stover.

Residues of spirotetramat and its four metabolites STM-enol, STM-ketohydroxy, STM-mono-hydroxy and STM-enol-glucoside (Glc) were analysed using method 00857, including minor modifications. For all analytes the limit of quantitation (LOQ) was determined to be 0.01 mg/kg.

The maximum storage period of deep-frozen samples before analysis was 552 days for Canadian trials and 253 days for Australian trials. These storage periods are covered by the previously reported storage stability studies.

The results are summarized in Tables 10 and 11.

Animal feed

Sweet corn forage, fodder and stover

The trial conditions are described under sweet corn. The results are summarized in Tables 12-14.

Table 8 Results of residue trials conducted with SC 240 and OD 150 formulations on guava in Mexico

Study Trial No.	Plot No. Year	Crop Variety	Appl. Rate [b] (kg ai/ha)	DAT (days)	Residues [mg/kg][a] STM	STM cis-enol	STM cis-keto-hydroxy	STM enol-glucoside	STM mono-hydroxy	Sum of STM and cis-enol	Total residue of STM+4
US GAP: max. 3 x 0.179 at 14 days, PHI of 1 day											
RAFNP042 FN075-07BA Mexico Municipio Juarez	FN075-07BA-B 2008	Guava Media China	240SC 3×[c] 0.274-0.291	1	0.312	0.136	0.042	< 0.01	< 0.01	0.448	0.499
				1	0.188	0.118	0.030	< 0.01	< 0.01	0.306	0.344
				3	0.107	0.082	0.033	< 0.01	< 0.01	0.189	0.226
				3	0.207	0.164	0.036	< 0.01	< 0.01	0.371	0.413
	FN075-07BA-A1 2008	Guava Media China	150OD 3×[c] 0.287-0.297	0	0.187	0.199	0.046	< 0.01	< 0.01	0.386	0.446
				0	0.222	0.148	0.041	< 0.01	< 0.01	0.370	0.426
				1	0.065	0.136	0.029	< 0.01	< 0.01	0.201	0.241
				1	0.091	0.158	0.028	< 0.01	< 0.01	0.249	0.291
				3	0.058	0.115	0.029	< 0.01	0.012	0.173	0.219
				3	0.433	0.107	0.029	< 0.01	< 0.01	0.540	0.580
				5	0.049	0.115	0.042	< 0.01	< 0.01	0.204	0.219
				5	0.041	0.100	0.032	< 0.01	0.013	0.141	0.189
				7	0.024	0.102	0.041	< 0.01	< 0.01	0.126	0.177
				7	0.021	0.083	0.035	< 0.01	0.012	0.104	0.155
	FN075-07BA-A2 2008	Guava Media China	150OD 3×[d] 0.284-0.302	1	0.172	0.215	0.029	< 0.01	< 0.01	0.387	0.423
				1	0.209	0.262	0.042	< 0.01	< 0.01	0.471	0.525
				3	0.056	0.132	0.032	< 0.01	< 0.01	0.188	0.228
				3	0.075	0.122	0.037	< 0.01	< 0.01	0.197	0.241
RAFNP042 FN076-07HA Mexico Calvillo	FN076-07HA-A1 2007	Guava China	150OD 3×[c] 0.283-0.309	1	0.351	0.211	0.020	< 0.01	0.011	0.562	0.600
				1	0.347	0.202	0.018	< 0.01	0.010	0.549	0.585
				3	0.427	0.344	0.023	< 0.01	0.016	0.771	0.815
				3	0.431	0.328	0.030	< 0.01	0.016	0.759	0.810
	FN076-07HA-A2 2007	Guava China	150OD 3×[d] 0.181-0.287	1	0.559	0.338	0.026	< 0.01	0.024	0.897	0.954
				1	0.560	0.355	0.026	< 0.01	0.028	0.915	0.976
				3	0.259	0.169	0.018	< 0.01	< 0.01	0.428	0.457
				3	0.264	0.157	0.016	< 0.01	< 0.01	0.421	0.447
RAFNL058 FN002-11DB Mexico Zitacuaro	FN002-11DB-A 2011	Guava Media China	240SC 3×[c] 0.150-0.152	0	0.246	0.136	0.052	< 0.01	< 0.01	0.382	0.609
				0	0.262	0.152	0.069	< 0.01	< 0.01	0.414	0.616
				1	0.209	0.160	0.038	< 0.01	< 0.01	0.369	0.673
				1	0.185	0.098	0.032	< 0.01	< 0.01	0.283	0.658
				3	0.233	0.141	0.059	< 0.01	< 0.01	0.374	0.533
				3	0.177	0.130	0.047	< 0.01	< 0.01	0.307	0.758
				7	0.126	0.155	0.047	< 0.01	< 0.01	0.281	0.398
				7	0.099	0.152	0.041	< 0.01	< 0.01	0.251	0.575
				14	0.074	0.098	0.065	< 0.01	0.01	0.172	0.211
				14	0.066	0.127	0.066	< 0.01	< 0.01	0.193	0.252
	FN002-11DB-B 2011	Guava Media China	240SC 3×[c] 0.288-0.293	0	0.664	0.178	0.058	< 0.01	< 0.01	0.842	0.910
				0	0.712	0.251	0.099	< 0.01	< 0.01	0.963	1.076
				1	0.362	0.150	0.057	< 0.01	< 0.01	0.512	0.579
				1	0.439	0.231	0.079	< 0.01	< 0.01	0.670	0.761
				3	0.473	0.238	0.076	< 0.01	< 0.01	0.675	0.763
				3	0.404	0.150	0.063	< 0.01	< 0.01	0.554	0.629
				7	0.351	0.279	0.090	< 0.01	< 0.01	0.630	0.734
				7	0.345	0.292	0.092	< 0.01	< 0.01	0.637	0.743
				14	0.219	0.151	0.102	< 0.01	0.014	0.370	0.494
				14	0.163	0.149	0.122	< 0.01	0.011	0.312	0.450

Study Trial No.	Plot No. Year	Crop Variety	Appl. Rate [b] (kg ai/ha)	DAT (days)	Residues [mg/kg][a]						
					STM	STM cis-enol	STM cis-keto-hydroxy	STM enol-glucoside	STM mono-hydroxy	Sum of STM and cis-enol	Total residue of STM+4
Cont. RAFNL058 FN002-11DB	FN002-11DB-C 2011	Guava Media China	150OD 3×[c] 0.147-0.151	0	0.340	0.155	0.080	< 0.01	0.010	0.495	0.591
				0	0.326	0.149	0.061	< 0.01	< 0.01	0.475	0.550
				1	0.229	0.176	0.060	< 0.01	0.010	0.405	0.480
				1	0.185	0.140	0.053	< 0.01	< 0.01	0.325	0.390
				3	0.139	0.156	0.043	< 0.01	0.010	0.295	0.354
				3	0.129	0.136	0.044	< 0.01	< 0.01	0.265	0.323
				7	0.098	0.190	0.071	< 0.01	0.010	0.288	0.374
				7	0.085	0.235	0.078	< 0.01	0.013	0.320	0.419
				14	0.043	0.078	0.082	< 0.01	0.012	0.121	0.223
				14	0.071	0.152	0.090	< 0.01	0.020	0.223	0.341
	FN002-11DB-D 2011	Guava Media China	150OD 3×[c] 0.287-0.291	0	0.529	0.219	0.087	< 0.01	0.014	0.748	0.856
				0	0.466	0.187	0.076	< 0.01	0.013	0.653	0.749
				1	0.449	0.296	0.098	< 0.01	0.015	0.745	0.867
				1	0.375	0.209	0.069	< 0.01	0.013	0.584	0.671
				3	0.305	0.286	0.104	0.011	0.015	0.591	0.720
				3	0.437	0.300	0.097	< 0.01	0.018	0.737	0.862
				7	0.192	0.326	0.137	0.012	0.019	0.518	0.685
				7	0.247	0.310	0.129	0.011	0.016	0.557	0.713
				14	0.166	0.252	0.135	0.012	0.028	0.418	0.593
				14	0.133	0.099	0.138	0.011	0.024	0.232	0.405
RAFNL058 FN003-11DA Mexico Zitacuaro	RAFNL058 FN003-11DA FN003-11DA-A 2011	Guava Calvillo	240SC 3×[c] 0.150-0.153	0	0.369	0.196	0.033	< 0.01	< 0.01	0.565	0.609
				0	0.415	0.161	0.028	< 0.01	< 0.01	0.576	0.616
				1	0.429	0.202	0.032	< 0.01	< 0.01	0.631	0.673
				1	0.424	0.198	0.027	< 0.01	< 0.01	0.622	0.658
				3	0.308	0.181	0.034	< 0.01	< 0.01	0.489	0.533
				3	0.434	0.253	0.059	< 0.01	< 0.01	0.687	0.758
				7	0.205	0.148	0.036	< 0.01	< 0.01	0.353	0.398
				7	0.322	0.194	0.047	< 0.01	< 0.01	0.516	0.575
				12	0.085	0.094	0.024	< 0.01	< 0.01	0.179	0.211
				12	0.085	0.124	0.035	< 0.01	< 0.01	0.209	0.220
Cont. RAFNL058 FN003-11DA	FN003-11DA-B 2011	Guava Calvillo	240SC 3×[c] 0.286-0.291	0	1.14	0.342	0.048	< 0.01	< 0.01	1.482	1.542
				0	0.809	0.299	0.040	< 0.01	< 0.01	1.108	1.159
				1	1.08	0.378	0.062	< 0.01	< 0.01	1.458	1.531
				1	0.785	0.357	0.051	< 0.01	< 0.01	1.142	1.206
				3	0.725	0.339	0.053	< 0.01	< 0.01	1.064	1.128
				3	0.815	0.408	0.053	< 0.01	< 0.01	1.223	1.290
				7	0.627	0.445	0.075	< 0.01	< 0.01	1.072	1.162
				7	0.407	0.335	0.055	< 0.01	< 0.01	0.742	0.808
				12	0.348	0.298	0.058	< 0.01	< 0.01	0.646	0.715
				12	0.409	0.302	0.098	< 0.01	< 0.01	0.711	0.822
	FN003-11DA-C 2011	Guava Calvillo	150OD 3×[c] 0.150-0.153	0	0.324	0.214	0.043	< 0.01	< 0.01	0.538	0.595
				0	0.557	0.387	0.090	0.011	0.015	0.944	1.060
				1	0.411	0.385	0.067	< 0.01	0.010	0.796	0.882
				1	0.396	0.264	0.066	< 0.01	0.012	0.660	0.745
				3	0.321	0.296	0.061	< 0.01	< 0.01	0.617	0.695
				3	0.326	0.316	0.064	< 0.01	< 0.01	0.642	0.722
				7	0.226	0.293	0.093	0.012	0.013	0.519	0.637
				7	0.202	0.323	0.099	0.010	0.014	0.525	0.648
				12	0.110	0.194	0.068	< 0.01	0.011	0.304	0.390
				12	0.087	0.157	0.060	< 0.01	0.011	0.244	0.322

Study Trial No.	Plot No. Year	Crop Variety	Appl. Rate [b] (kg ai/ha)	DAT (days)	Residues [mg/kg][a]						
					STM	STM cis-enol	STM cis-keto-hydroxy	STM enol-glucoside	STM mono-hydroxy	Sum of STM and cis-enol	Total residue of STM+4
	FN003-11DA-D 2011	Guava Calvillo	150OD 3×[c] 0.284-0.293	0	0.914	0.494	0.122	0.015	0.014	1.408	1.560
				0	0.895	0.448	0.107	0.016	0.020	1.343	1.485
				1	0.514	0.310	0.072	0.010	0.012	0.824	0.919
				1	0.636	0.360	0.073	< 0.01	0.012	0.996	1.090
				3	0.433	0.362	0.124	0.011	0.019	0.795	0.951
				3	0.602	0.414	0.093	0.012	0.016	1.016	1.137
				7	0.481	0.455	0.115	0.016	0.020	0.936	1.088
				7	0.465	0.469	0.154	0.015	0.021	0.934	1.122
				12	0.168	0.331	0.121	0.013	0.020	0.499	0.652
				12	0.130	0.219	0.085	0.010	0.013	0.349	0.456

Notes: c: concentrated spray; d: diluted spray;

[1]: The residues were measured in guava fruits.

[b]: The applications were made at growth stages between 77-81.

Calc 1: Residues of STM, STM cis-enol, STM cis-keto-hydroxy, BYI08330 monohydroxy, STM enol-glucoside each expressed as STM. Total residue of STM calc.1 and Sum of STM and STM cis-enol expressed as STM**.**

Table 9 Results of residue trials conducted with spirotetramat on avocado

Study Trial No. Plot No. GLP Year	Crop Variety Year	Appl. rate (kg ai/ha)	DALT (days)	Residues [a]						
				STM (mg/kg)	STM cis-enol (mg/kg)	STM cis-keto-hydroxy (mg/kg)	STM enol-glucoside (mg/kg)	STM mono-hydroxy (mg/kg)	Sum of STM and STM cis-enol (mg/kg)	Total residue of STM calc.1 (mg/kg)
Mexico GAO: 3 times 0.29 kg ai/ha at 14 days intervals and PHI of 1 day										
RAFNP042	Avocado	3×0.288	1	0.082	0.064	0.011	< 0.01	< 0.01	0.146	0.161
FN070-07BA	Haas	(conc.) [c]	1	0.083	0.068	0.010	< 0.01	< 0.01	*0.151*	*0.167*
FN070-07BA-A1	2008		3	0.047	0.052	0.016	< 0.01	< 0.01	0.099	0.121
San Luis Obispo, USA, California			3	0.049	0.054	0.015	< 0.01	< 0.01	0.103	0.128
RAFNP042	Avocado	3×0.288	1	0.101	0.099	0.017	< 0.01	< 0.01	0.200	0.226
FN070-07BA	Haas	(diluted) [c]	1	0.101	0.098	0.016	< 0.01	< 0.01	0.199	0.224
FN070-07BA-A2	2008		3	0.120	0.083	0.021	0.013	< 0.01	*0.203*	*0.240*
San Luis Obispo, USA, California			3	0.120	0.080	0.021	0.011	< 0.01	0.200	0.234
RAFNP042	Avocado	3× 0.288	1	0.120	0.080	0.012	< 0.01	< 0.01	*0.200*	*0.217*
FN070-07BA	Haas	(conc.) [b]	1	0.114	0.075	0.011	< 0.01	< 0.01	0.189	0.204
FN070-07BA-B	2008		3	0.049	0.061	0.014	< 0.01	< 0.01	0.110	0.128
San Luis Obispo, USA California			3	0.048	0.061	0.014	< 0.01	< 0.01	0.109	0.127

Study Trial No. Plot No. GLP Year	Crop Variety Year	Appl. rate (kg ai/ha)	DALT (days)	Residues [a]						
				STM (mg/kg)	STM cis-enol (mg/kg)	STM cis-keto-hydroxy (mg/kg)	STM enol-glucoside (mg/kg)	STM mono-hydroxy (mg/kg)	Sum of STM and STM cis-enol (mg/kg)	Total residue of STM calc.1 (mg/kg)
RAFNP042	Avocado	3×0.288	0	0.031	0.061	0.015	< 0.01	< 0.01	0.092	0.111
FN071-07DA	Hass	(conc.) [c]	0	0.023	0.051	0.011	< 0.01	< 0.01	0.074	0.090
FN073-07DA-A1	2008		1	0.042	0.067	0.012	< 0.01	< 0.01	0.109	0.127
Arroyo Grande, USA California			1	0.031	0.057	< 0.01	< 0.01	< 0.01	0.088	0.101
			3	0.030	0.041	< 0.01	< 0.01	< 0.01	0.071	0.083
			3	0.026	0.032	< 0.01	< 0.01	< 0.01	0.058	0.071
			5	0.031	0.034	< 0.01	< 0.01	< 0.01	0.065	0.076
			5	0.039	0.040	< 0.01	< 0.01	< 0.01	0.079	0.089
			7	0.018	0.045	< 0.01	< 0.01	< 0.01	0.063	0.073
			7	0.050	0.081	0.013	< 0.01	< 0.01	*0.131*	*0.152*
RAFNP042	Avocado	3× 0.288	1	0.036	0.083	0.022	< 0.01	< 0.01	*0.119*	*0.145*
FN071-07DA	Haas	(diluted) [c]	1	0.035	0.062	0.013	< 0.01	< 0.01	0.097	0.114
FN071-07DA-A2	2008		3	0.032	0.057	0.015	< 0.01	< 0.01	0.089	0.110
Arroyo Grande, USA California			3	0.037	0.066	0.012	< 0.01	< 0.01	0.103	0.119
RAFNP042	Avocado	3× 0.288	1	< 0.01	0.011	< 0.01	< 0.01	< 0.01	0.021	0.024
FN072-07HA	Haas	(conc.) [c]	1	0.019	0.050	0.012	< 0.01	< 0.01	*0.069*	*0.087*
FN072-07HA-A1	2008		3	< 0.01	0.018	< 0.01	< 0.01	< 0.01	0.028	0.033
Mexico Nuevo Parangaricutiro			3	< 0.01	0.036	< 0.01	< 0.01	< 0.01	0.046	0.062
RAFNP042	Avocado	3× 0.288	1	< 0.01	0.018	< 0.01	< 0.01	< 0.01	0.028	0.034
FN072-07HA	Haas	(diluted) [c]	1	< 0.01	0.017	< 0.01	< 0.01	< 0.01	0.027	0.034
FN072-07HA-A2	2008		3	< 0.01	0.031	< 0.01	< 0.01	< 0.01	*0.041*	*0.058*
Mexico Nuevo Parangaricutiro			3	< 0.01	0.011	< 0.01	< 0.01	< 0.01	0.021	0.021
RAFNP042	Avocado	3× 0.288	1	0.193	0.080	0.013	< 0.01	< 0.01	0.273	0.291
FN073-07BB	Hass	(conc.) [c]	1	0.224	0.070	0.012	< 0.01	< 0.01	*0.294*	*0.309*
FN073-07BB-A1	2008		3	0.197	0.088	0.012	< 0.01	< 0.01	0.285	0.300
Chile Llay Llay, Valparaiso			3	0.145	0.082	0.011	< 0.01	< 0.01	0.227	0.242
RAFNP042	Avocado	3× 0.288	1	0.144	0.058	0.011	< 0.01	< 0.01	0.202	0.217
FN073-07BB	Hass	(diluted) [c]	1	0.186	0.070	0.016	< 0.01	< 0.01	0.256	0.276
FN073-07BB-A2	2008		3	0.166	0.097	0.017	< 0.01	< 0.01	0.263	0.284
Chile Llay Llay, Valparaiso			3	0.186	0.090	0.019	< 0.01	< 0.01	*0.276*	*0.299*
RAFNP042	Avocado	3× 0.288	1	0.160	0.081	0.011	< 0.01	< 0.01	0.241	0.256
FN073-07BB	Hass	(conc.)[b]	1	0.250	0.098	0.014	< 0.01	< 0.01	*0.348*	0.365
FN073-07BB-B	2008		3	0.224	0.119	0.017	< 0.01	< 0.01	*0.343*	*0.365*
Chile Llay Llay, Valparaiso			3	0.128	0.087	0.012	< 0.01	< 0.01	0.215	0.231
RAFNP042	Avocado	3× 0.288	1	0.059	0.075	0.019	< 0.01	< 0.01	0.134	0.157
FN074-07HA	Hass	(conc.) [c]	1	0.068	0.072	0.016	< 0.01	< 0.01	0.140	0.159
FN074-07HA-A1	2008		3	0.066	0.071	0.020	< 0.01	< 0.01	0.137	0.161
Chile Ocoa, Valparaiso			3	0.079	0.097	0.026	< 0.01	< 0.01	*0.176*	*0.207*

Study Trial No. Plot No. GLP Year	Crop Variety Year	Appl. rate (kg ai/ha)	DALT (days)	Residues [a]						
				STM (mg/kg)	STM cis-enol (mg/kg)	STM cis-keto-hydroxy (mg/kg)	STM enol-glucoside (mg/kg)	STM mono-hydroxy (mg/kg)	Sum of STM and STM cis-enol (mg/kg)	Total residue of STM calc.1 (mg/kg)
RAFNP042 FN074-07HA FN074-07HA-A2 Chile Ocoa, Valparaiso	Avocado Hass 2008	3× 0.288 (diluted) [c]	1 1 3 3	0.087 0.080 0.064 0.068	0.083 0.092 0.073 0.076	0.024 0.025 0.022 0.021	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.170 *0.172* 0.137 0.144	0.200 *0.204* 0.163 0.169

[a]: Residues of STM, STM cis-enol, STM cis-keto-hydroxy, STM enol-glucoside each expressed as STM. Total residue of STM calc.1 and Sum of STM and STM cis-enol expressed as STM. The residues were measured in avocado fruits.

[b] 240 EC;

[c] 150 OD; the applications were made at growth stages between BBCH 47-85.

Table 10 Results of residue trials conducted with an SC 240 formulation in/on sweet corn in Canada

Study Trial No. Plot No. Location Year	Variety Dosage	Dosage Kgai/ha	DALT (days)	Residues [a] [mg/kg]						
				STM	STM cis-enol	STM cis-keto-hydroxy	STM enol-glucoside	STM mono-hydroxy	Sum of STM and STM cis-enol	Total residue of STM calc.1
Canadian max GAP 3 x 0.088 kgai/ha at 7 days with PHI of 7 days.										
AAFC09-027R AAFC09-027R-116 Delhi, 2009	Brocade	3× 0.091-0.093	1 1 3 3 **7** 7 9 9	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.38 0.46 0.39 0.37 0.61 0.46 0.39 0.56	0.12 0.12 0.18 0.19 0.11 0.16 0.15 0.075	< 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	0.390 0.470 0.400 0.380 **0.620** 0.470 0.400 0.570	0.53 0.61 0.59 0.58 **0.75** 0.64 0.57 0.66
AAFC09-027R AAFC09-027R-117 Delhi, 2009	Luscious	3× 0.078-0.091	7 **7**	< 0.01 < 0.01	0.40 0.40	0.12 0.14	< 0.01 < 0.01	< 0.01 < 0.01	0.410 0.410	0.56 0.57
AAFC09-027R AAFC09-027R-118 Harrow, 2009	Fantastic	3 x 0.091-0.095	**7** 7	< 0.01 < 0.01	0.034 0.021	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	0.044 0.031	0.074 0.061
AAFC09-027R AAFC09-027R-119 Harrow, 2009	Awesome	3× 0.089-0.093	6 **6**	< 0.01 < 0.01	0.024 0.035	0.013 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	0.034 **0.045**	0.067 **0.075**
AAFC09-027R AAFC09-027R-120 L'Arcadie, 2009	114E Fleet	3×0.087-0.091	**6** 6	< 0.01 < 0.01	0.25 0.20	0.048 0.063	< 0.01 < 0.01	< 0.01 < 0.01	**0.260** 0.210	**0.33** 0.29

Study Trial No. Plot No. Location Year	Variety Dosage	Dosage Kgai/ha	DALT (days)	Residues [a] [mg/kg] STM	STM cis-enol	STM cis-keto-hydroxy	STM enol-glucoside	STM mono-hydroxy	Sum of STM and STM cis-enol	Total residue of STM calc.1
AAFC09-027R AAFC09-027R-121, L'Arcadie 2009	Hybrid Trinity	3×0.087-0.91	7 **7**	< 0.01 < 0.01	0.074 0.096	0.060 0.059	< 0.01 < 0.01	< 0.01 < 0.01	0.084 0.106	0.16 0.19
AAFC09-027R AAFC09-027R-122 Taber, 2009	King Cobb	3 x 0.084-0.086	**7** 7	< 0.01 < 0.01	0.053 0.049	0.048 0.039	< 0.01 < 0.01	< 0.01 < 0.01	**0.063** 0.059	**0.13** 0.12
AAFC09-027R AAFC09-027R-123, Agassiz 2009	G118K Luscious	3×0.087-0.09	7 **7**	< 0.01 < 0.01	0.47 0.47	0.070 0.13	< 0.01 < 0.01	< 0.01 < 0.01	0.480 **0.480**	0.57 **0.63**

[a] Residues of STM, STM cis-enol, STM cis-keto-hydroxy, STM enol-glucoside each expressed as STM. Total residue of STM calc.1 and Sum of STM and STM cis-enol expressed as STM.

Trials 116-117 are not considered independent. Same location, soil 1 week difference in application with same/similar equipment.

Trials 118-119 are not considered independent. Same location, dates of application equipment and soil

Trials 120-121 are not considered independent. Same location, dates of application equipment and soil.

Table 11 Results of residue trials conducted with an SC 240 formulation in/on sweet corn in Australia

Study Trial No. Plot No. Location Year	Variety Dosage	Dosage kg ai/ha	DALT (days)	Residues [a] [mg/kg] STM (mg/kg)	STM cis-enol	STM cis-keto-hydroxy	STM enol-glucoside	STM mono-hydroxy	Sum of STM and STM cis-enol	Total residue of STM calc.1
Australian max GAP: 2×0.072 kg ai/ha at 7 days interval with PHI of 7 days.										
BCS-0272 B000 B000-T2 4343 Gatton 2008	Golden sweet improved	2×0.015-0.016	0* 0 1 3 **7**	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.096 0.096 0.14 0.23 0.22	0.036 0.072 < 0.024 0.036 < 0.024	< 0.016 < 0.016 < 0.016 < 0.016 < 0.016	< 0.024 < 0.024 < 0.024 < 0.024 < 0.024	0.12 0.12 0.16 0.25 **0.24**	0.19 0.23 0.23 0.32 **0.30**
BCS-0319 C457 C457-T2 4805 Bowen 2009	Golden Sweet	2×0.071	0* 0 1 4 **7** 11 14	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.024 < 0.024 < 0.024 < 0.024 < 0.024 0.036 0.036	< 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024	< 0.016 < 0.016 < 0.016 < 0.016 < 0.016 < 0.016 < 0.016	< 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024	< 0.044 < 0.044 < 0.044 < 0.044 < 0.044 **0.056** 0.056	< 0.11 < 0.11 < 0.11 < 0.11 < 0.11 **0.12** 0.12
BCS-0319 C458 C458-T2 4805 Bowen 2009	Sentinel	2× 0.071	0* 0 1 4 **7** 11 14	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.024 < 0.024 < 0.024 < 0.024 0.024 0.036 < 0.02	< 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024	< 0.016 < 0.016 < 0.016 < 0.016 < 0.016 < 0.016 < 0.016	< 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024	< 0.044 < 0.044 < 0.044 < 0.044 0.044 0.056 < 0.044	< 0.11 < 0.11 < 0.11 < 0.11 0.11 0.12 < 0.11

Study Trial No. Plot No. Location Year	Variety Dosage	Dosage kg ai/ha	DALT (days)	Residues [a] [mg/kg]						
				STM (mg/kg)	STM cis-enol	STM cis-keto-hydroxy	STM enol-glucoside	STM mono-hydroxy	Sum of STM and STM cis-enol	Total residue of STM calc.1
BCS-0319 C459 C459-T2 4341 Laidley 2009	H5	2×0.072-0.075	0* 0 1 3 **7** 10 14	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.024 < 0.024 0.036 0.048 0.036 0.048 0.084	0.036 0.036 0.036 0.060 0.084 0.048 0.036	< 0.016 < 0.016 < 0.016 < 0.016 < 0.016 < 0.016 < 0.016	< 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024	< 0.044 < 0.044 0.056 0.068 0.056 0.068 <u>0.10</u>	0.12 0.12 0.13 0.17 0.18 0.16 <u>0.18</u>
BCS-0322 C471 AUS-BCS-0322-C471-A 3981 Koo Wee Rup 2010	Golden Sweet	2×0.073	0* 0 1 4 **7** 11 14	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.17 0.17 0.16 0.43 0.38 0.35 0.37	0.096 0.28 0.096 0.084 0.18 0.18 0.28	< 0.016 < 0.016 < 0.016 < 0.016 < 0.016 < 0.016 < 0.016	< 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024	0.19 0.19 0.18 0.45 <u>0.40</u> 0.37 0.39	0.32 0.50 0.31 0.58 <u>0.62</u> 0.59 *<u>0.71</u>*
BCS-0322 C472 AUS-BCS-0322-C472-A 4380, Stanthorpe 2010	Spaceship	2×0.068-0.070	0* 0 1 3 **7** 10 13	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.06 0.06 0.072 0.096 0.096 0.084 0.084	< 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024	< 0.016 < 0.016 < 0.016 < 0.016 < 0.016 < 0.016 < 0.016	< 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024	0.08 0.08 0.092 0.12 <u>0.12</u> 0.10 0.10	0.14 0.14 0.16 0.18 <u>**0**.18</u> 0.17 0.17
BCS-0322 C473 AUS-BCS-0322-C473-A 7307 Wesley Vale, 2010	Super Sweet	2×0.078-0.080	0* 0 1 3 **7** 10 14	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.036 0.036 0.048 0.072 0.096 0.096 0.084	< 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024	< 0.016 < 0.016 < 0.016 < 0.016 < 0.016 < 0.016 < 0.016	< 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024	0.056 0.056 0.068 0.092 <u>0.12</u> 0.12 0.10	0.12 0.12 0.13 0.16 <u>0.18</u> 0.18 0.17

[a]: Residues of STM, STM cis-enol, STM cis-keto-hydroxy, STM enol-glucoside each expressed as STM. Total residue of STM calc.1 and Sum of STM and STM cis-enol expressed as STM.

Animal feed

Table 12 Results of residue trials conducted with an SC 240 formulation in/on sweet corn forage in Canada

Study Trial No. Plot No. Location Year	Variety	Dosage	DALT (days)	Residues [mg/kg]						
				STM	STM cis-enol	STM cis-keto-hydroxy	STM enol-glucoside	STM mono-hydroxy	Sum of STM and STM cis-enol	Total residue of STM+4 metabolite
Canadian max GAP 3 × 0.088 kg ai/ha at 7 days with PHI of 7 days.										
AAFC09-027R AAFC09-027R-116 Delhi, 2009	Brocade	3× 0.091-0.093	1 1 3 3 **7** 7 9 9	0.59 0.77 0.047 0.031 0.018 0.015 0.014 0.017	0.45 0.42 0.14 0.18 0.17 0.15 0.12 0.12	0.22 0.21 0.14 0.11 0.15 0.093 0.095 0.12	< 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	1.040 1.190 0.187 0.211 0.188 0.165 0.134 0.137	1.28 1.42 0.34 0.34 0.36 0.27 0.25 0.28
AAFC09-027R AAFC09-027R-117 Delhi, 2009	Luscious	3× 0.078-0.091	**7** 7	0.035 0.021	0.081 0.092	0.15 0.13	< 0.01 < 0.01	< 0.01 < 0.01	0.116 0.113	0.29 0.26
AAFC09-027R AAFC09-027R-118 Harrow, 2009	Fantastic	3× 0.091-0.095	7 **7**	< 0.01 0.010	0.011 0.013	0.011 0.012	< 0.01 < 0.01	< 0.01 < 0.01	0.021 0.023	0.052 0.055
AAFC09-027R AAFC09-027R-119 Harrow, 2009	Awesome	3× 0.089-0.093	6 **6**	0.010 0.010	0.017 0.017	0.013 0.012	< 0.01 < 0.01	< 0.01 < 0.01	0.027 0.027	0.060 0.060
AAFC09-027R AAFC09-027R-120 L'Arcadie 2009	114E Fleet	3× 0.087-0.091	**6** 6	0.097 0.077	0.088 0.091	0.11 0.095	< 0.01 < 0.01	< 0.01 < 0.01	0.185 0.168	0.32 0.28
AAFC09-027R AAFC09-027R-121 L'Arcadie 2009	Hybrid Trinity	3×0.087-0.91	7 **7**	0.14 0.16	0.096 0.096	0.076 0.091	< 0.01 < 0.01	< 0.01 < 0.01	0.236 0.256	0.33 0.37
AAFC09-027R AAFC09-027R-122 Taber, 2009	King Cobb	3× 0.084-0.086	7 **7**	1.3 1.7	0.29 0.29	0.14 0.11	< 0.01 < 0.01	< 0.01 < 0.01	1.590 1.990	1.7 2.1
AAFC09-027R AAFC09-027R-123 Agassiz, 2009	G118K Luscious	3× 0.087-0.09	**7** 7	0.050 0.022	0.12 0.17	0.12 0.066	< 0.01 < 0.01	< 0.01 < 0.01	0.170 0.192	0.31 0.27

Table 13.Results of residue trials conducted with an SC 240 formulation in/on sweet corn stover in Canada

Study Trial No. Plot No.GLP Year	Variety	Dosage	DALT (days)	Residues [mg/kg]						
				STM	STM cis-enol	STM cis-keto-hydroxy	STM enol-glucoside	STM mono-hydroxy	Sum of STM and STM cis-enol	Total residue of STM calc.1
Canadian max GAP 3 x 0.088 kg ai/ha at 7 days with PHI of 7 days.										
AAFC09-027R AAFC09-027R-116 Canada, Delhi 2009	Brocade	3× 0.091-0.093	50 50 **56** 56 64 64 69 69	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.014 0.012 0.011 0.011 0.010 < 0.01	0.027 0.043 0.028 0.043 0.039 0.037 0.065 < 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	< 0.02 < 0.02 0.024 0.022 0.021 0.021 0.020 < 0.02	0.067 0.083 0.071 0.085 0.079 0.078 0.11 < 0.05

Study Trial No. Plot No.GLP Year	Variety	Dosage	DALT (days)	Residues [mg/kg]						
				STM	STM cis-enol	STM cis-keto-hydroxy	STM enol-glucoside	STM mono-hydroxy	Sum of STM and STM cis-enol	Total residue of STM calc.1
AAFC09-027R AAFC09-027R-117 Canada, Delhi 2009	Luscious	3× 0.078-0.091	**56** 56	< 0.01 < 0.01	0.017 < 0.01	0.036 0.036	< 0.01 < 0.01	< 0.01 < 0.01	0.027 < 0.02	0.083 0.076
AAFC09-027R AAFC09-027R-118 Canada , Harrow 2009	Fantastic	3× 0.091-0.095	85 **85**	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.02 < 0.02	< 0.05 < 0.05
AAFC09-027R AAFC09-027R-119 Canada, Harrow 2009	Awesome	3× 0.089-0.093	55 **55**	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.02 < 0.02	< 0.05 < 0.05
AAFC09-027R AAFC09-027R-120 Canada L'Arcadie 2009	114E Fleet	3×0.087-0.091	**55** 55	0.010 0.020	0.020 < 0.01	0.062 0.056	< 0.01 < 0.01	< 0.01 < 0.01	0.030 0.030	0.11 0.11
AAFC09-027R AAFC09-027R-121 Canada L'Arcadie 2009	Hybrid Trinity	3×0.087-0.91	47 **47**	0.021 0.026	0.011 0.16	0.037 0.082	< 0.01 < 0.01	< 0.01 < 0.01	0.032 0.186	0.089 0.14
AAFC09-027R AAFC09-027R-122 Canada, Taber 2009	King Cobb	3 x 0.084-0.086	**47** 47	0.40 0.32	0.059 0.050	0.16 0.13	< 0.01 < 0.01	< 0.01 < 0.01	0.459 0.370	0.64 0.52
AAFC09-027R AAFC09-027R-123 Canada Agassiz 2009	G118K Luscious	3×0.087-0.09	85 **85**	0.020 0.040	< 0.01 0.022	0.022 0.051	< 0.01 < 0.01	< 0.01 < 0.01	0.030 0.062	0.072 0.13

Table 14 Results of residue trials conducted with an SC 240 formulation in/on sweet corn fodder in Australia

Study Trial No. Plot No. Year	Variety	Dosage	DALT (days)	Residues[1] [mg/kg] STM (mg/kg)	STM cis-enol	STM cis-keto-hydroxy	STM enol-glucoside	STM mono-hydroxy	Sum of STM and STM cis-enol	Total residue of STM calc.1
Australian max GAP: 2×0.072 kgai/ha at 7 days interval with PHI of 50 days for stover.										
BCS-0272 B000 B000-T2 Australia 4343 Gatton 2008	Golden sweet improved	2×0.015 -0.016	0* 0 1 3 **7**	0.05 1.94 2.00 0.94 0.10	0.04 1.2 0.70 0.36 0.096	0.11 0.20 0.22 0.29 0.16	< 0.016 < 0.016 < 0.016 < 0.016 < 0.016	< 0.024 < 0.024 < 0.024 < 0.024 < 0.024	0.086 3.14 2.70 1.30 0.20	0.23 3.38 2.95 1.63 0.39
BCS-0319 C457 C457-T2 Australia 4805 Bowen 2009	Golden Sweet	2×0.071	0* 0 1 4 **7** 14	0.30 1.55 1.49 1.31 0.21 0.11	0.096 0.74 0.26 0.46 0.096 0.036	0.096 0.17 0.19 0.58 0.23 0.12	< 0.016 < 0.016 < 0.016 < 0.016 < 0.016 < 0.016	< 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024	0.40 2.29 1.75 1.77 0.31 0.15	0.53 2.50 1.99 2.38 0.57 0.31
BCS-0319 C458 C458-T2 Australia 4805 Bowen 2009	Sentinel	2× 0.071	0* 0 1 4 **7** 14	0.34 1.41 1.35 0.83 0.38 0.15	0.096 0.65 0.24 0.26 0.20 0.060	0.11 0.17 0.20 0.31 0.37 0.22	< 0.016 < 0.016 < 0.016 < 0.016 < 0.016 < 0.016	< 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024	0.44 2.06 1.59 1.09 0.58 0.21	0.58 2.27 1.83 1.45 1.00 0.47
BCS-0319 C459 C459-T2 Australia 4341 Laidley 2009	H5	2×0.072 -0.075	0* 0 1 3 **7** 14	0.05 1.80 0.28 0.27 0.16 0.04	0.036 0.91 0.38 0.16 0.12 < 0.024	0.060 0.18 0.17 0.22 0.31 0.096	< 0.016 < 0.016 < 0.016 < 0.016 < 0.016 < 0.016	< 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024	0.086 2.71 0.66 0.43 **0**.28 0.064	0.19 2.93 0.87 0.68 0.63 0.20
BCS-0322 C471 AUS- BCS- 0322- C471-A 3981 Koo Wee Rup 2010	Golden Sweet	2×0.073	0* 0 1 4 **7** 11 14	0.40 1.16 1.11 0.47 0.25 0.11 0.16	0.14 0.72 0.35 0.26 0.084 0.048 0.072	0.19 0.18 0.22 0.23 0.17 0.12 0.22	< 0.016 < 0.016 < 0.016 < 0.016 < 0.016 < 0.016 < 0.016	< 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024	0.54 1.88 1.46 0.73 0.33 0.16 0.23	0.78 2.10 1.71 1.00 0.54 0.32 0.49
BCS-0322 C472 AUS- BCS- 0322- C472-A Australia 4380 Stanthorpe 2010	Spaceship	2×0.068- 0.070	0* 0 1 3 **7** 10 13	0.59 1.91 1.26 0.47 0.34 0.18 0.12	0.17 0.17 0.18 0.16 0.16 0.11 0.072	0.60 0.36 0.37 0.38 0.83 0.68 0.55	< 0.016 < 0.016 < 0.016 < 0.016 < 0.016 < 0.016 < 0.016	< 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024 < 0.024	0.76 2.08 1.44 0.63 0.50 0.29 0.19	1.40 2.48 1.85 1.05 1.36 1.01 0.78

Study Trial No. Plot No. Year	Variety	Dosage	DALT (days)	Residues[1] [mg/kg] STM (mg/kg)	STM cis-enol	STM cis-keto-hydroxy	STM enol-glucoside	STM mono-hydroxy	Sum of STM and STM cis-enol	Total residue of STM calc.1
BCS-0322 C473 AUS-BCS-0322-C473-A Australia 7307 Wesley Vale 2010	Super Sweet	2×0.078 -0.080	0*	0.40	0.24	0.55	< 0.016	< 0.024	0.64	1.23
			0	1.46	0.52	0.48	< 0.016	< 0.024	1.98	2.50
			1	0.39	0.31	0.42	< 0.016	< 0.024	0.70	1.16
			3	0.16	0.12	0.23	< 0.016	< 0.024	0.28	0.55
			7	0.08	0.036	0.096	< 0.016	< 0.024	0.12	0.25
			10	0.11	0.072	0.25	< 0.016	< 0.024	0.18	0.47
			14	0.06	0.048	0.17	< 0.016	< 0.024	0.11	0.32

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

No data are available from the storage under warehouse conditions.

In processing

The effect of processing on spirotetramat residues have already been evaluated by JMPR in 2008. The meeting concluded that spirotetramat-enol was resistant to hydrolysis under all test conditions. Processing factors have been established for cooked bean (0.46), canned tomato (0.58) and canned cherries (0.47). In all these commodities the reduction of residues was observed. Similarly, it is expected that the residues will not concentrate in sweet corn.

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

Based on a dairy cattle feeding study and poultry metabolism study the 2008 JMR estimated residue levels in animal commodities. No new information was provided.

APPRAISAL

The compound was evaluated by the JMPR for the first time in 2008. The Meeting established an ADI of 0–0.05 mg/kg bw per day and an ARfD of 1 mg/kg/bw and defined the residues as follow:

Residue for enforcement plant commodities: spirotetramat plus spirotetramat enol, expressed as spirotetramat.

Residue for dietary intake plant commodities: *spirotetramat plus the metabolites enol, ketohydroxy, enol glucoside, and monohydroxy, expressed as spirotetramat*.

Residue for enforcement and dietary intake animal commodities: *spirotetramat enol, expressed as spirotetramat*.

The residue is not fat soluble.

Additional residue data were evaluated by the 2011 JMPR.

Spirotetramat was listed by the Forty-sixth Session of CCPR (2014) for the evaluation by the 2015 JMPR for additional MRLs. Supervised trials data were submitted for evaluation on avocado, guava and sweet corn for the evaluation by the 2015 JMPR.

Analytical methods

Analytical methods were evaluated by the 2008 and 2011 Meetings. Recovery data obtained from the analysis of avocado, guava and sweet corn and sweet corn fodder. The limit of quantification was 0.01 mg/kg for individual residues. The residues of individual analyte were expressed as spirotetramat equivalents and summed up to yield the total residue of spirotetramat plus enol (LOQ 0.02 mg/kg) and spirotetramat plus 4 metabolites (LOQ 0.05 mg/kg). The recoveries for individual residue components in the matrices tested 0.01 and 0.1 mg/kg or 1.0 and 10 mg/kg spike level and their relative standard deviations were within acceptable range.

Stability of analytes

Individual data on storage stability of spirotetramat and its metabolites were evaluated by the JMPR in 2008. The Meeting concluded that spirotetramat including its enol metabolite was stable (≥ 80% remaining) for about 2 years in tomato, potato, lettuce, almond nutmeat, climbing French beans and tomato paste. No new information was provided.

Residues resulting from supervised trials in crops

Results of new trials and some of the previously submitted ones on guava, avocado and sweet corn were evaluated by the present meeting. The sum of respective residues was expressed in spirotetramat equivalent.

Assorted tropical and sub-tropical fruits – edible peel

Guava

In 2008 and 2011, four residue trials in guava were conducted (including 13 plots) in Mexico. The trials were performed either with the OD 150 or the SC 240 formulation. The trials were conducted at two different application rates: 3× 0.288 kg ai/ha or 3× 150 kg ai/ha at spray intervals of 14 days. The US GAP permits 3 applications at 0.179 kg ai/ha rate at 14 days intervals with a PHI of 1 day. The results of supervised trials conducted in Mexico are evaluated against the US GAP.

The results indicate that the type of formulation and concentration of the spray solution did not affect the residue level. Therefore, the highest residues were selected from each set of trials.

The sum of residues of spirotetramat and its enol metabolite deriving from the 3 times 0.288 kg ai/ha nominal application rates at 1-3 days after last application were: 0.429, 0.660, 0.906 and 1.30 mg/kg.

Taking into account the nominal application rate of 288 g ai/ha and the USA GAP rate of 179 g ai/ha, the scaling factor is 179/228=0.6215. The residues scaled to match US GAP are in rank order: 0.27, 0.41, 0.56, and 0.81 mg/kg.

The sum of residues of spirotetramat and 4 metabolites are: 0.474, 0.79, 0.965 and 1.37 mg/kg.

The residues scaled to US GAP are: 0.29, 0.49, 0.60, and 0.85 mg/kg.

The Meeting estimated maximum residue level of 2 mg/kg, an HR of 0.85 mg/kg and an STMR residue of 0.55 mg/kg.

Assorted tropical and sub-tropical fruits – inedible peel

Avocado

The uses on avocado and the corresponding residue trials were previously submitted in 2010, but no recommendation could be made at that time. Subsequently, the GAPs of Chile and Mexico have been changed.

The use of spirotetramat in/on avocado is registered in the USA (3 applications of maximum 0.179 kg ai/ha at 14 days interval with a maximum seasonal rate of 0.44 kg ai/ha and PHI of 1 day), Chile (2 applications with a maximum seasonal rate of 0.8 kg ai/ha and PHI of 3 days) and Mexico (1 applications at maximum rate of 0.168 kg ai/ha and PHI of 1 day.

Five trials were conducted in USA, Chile and Mexico with nominal application rates of 0.288 kg ai/ha.

The critical GAP is from USA. The results of trials were evaluated based on the US GAP.

The highest sum of spirotetramat and enol from each replicate plots corresponding to this GAP are: 0.045, 0.11, 0.17, 0.20, 0.29 mg/kg.

Taking into account the targeted application rates of 0.288 and the maximum authorised rate of 0.179, the scaling factor is 0.179/0.288=0.6215.

The scaled residues in avocado fruits were in rank order: 0.028, 0.067, 0.106, 0.125, and 0.183 mg/kg.

For dietary intake assessment the sum of residues of spirotetramat and 4 metabolites was considered. They are in rank order: 0.055, 0.13, 0.20, 0.24, and 0.31 mg/kg.

The scaled residues in rank order are: 0.034, 0.080, 0.126, 0.147, and 0.193 mg/kg.

The highest residue observed in any single sample was 0.23 mg/kg.

The Meeting estimated a maximum residue level an STMR and HR of 0.4 mg/kg, 0.126 mg/kg and 0.23 mg/kg, respectively.

Sweet corn

Seven trials were conducted in Australia between 2008 and 2010 with applications close to Australian maximum GAP (2 times 0.072 kg ai/ha at 7 day intervals with a PHI of 7 days). One sample was taken from each plot.

In Australian trials the sum of spirotetramat and enol in ear without husk were: 0.056, 0.056, 0.1, 0.12, 0.12, 0.24 and 0.40 mg/kg.

For dietary intake assessment the sum of residues of spirotetramat and 4 metabolites was considered. They were in rank order: 0.12, 0.12, 0.18, 0.18, 0.18, 0.3 and 0.62.

Eight trials were conducted in Canada approximating maximum GAP which permits treatments with 3 × 0.088 kg ai/ha at 7 days intervals and a PHI of 7 days. Duplicate samples were taken in each trial.

Some Canadian trials were carried out at the same location, timing, dosage and equipment. The highest sum of spirotetramat and enol in ear without husk from the independent trials were: 0.040, 0.061, 0.235, 0.48 and 0.545 mg/kg.

For dietary intake assessment the sum of residues of spirotetramat and 4 metabolites was considered. They were in rank order: 0.071, 0.125, 0.31, 0.60 and 0.695 mg/kg.

The maximum residue in a single sample was 0.75 mg/kg.

Based on the Canadian trials reflecting maximum GAP, the Meeting estimated a maximum residue level of 1.5 mg/kg, and for dietary risk assessment an STMR residue of 0.31 mg/kg and an HR of 0.75 mg/kg.

Animal feed

Residue data on sweet corn forage and stover derived from supervised trials conducted in Australia and Canada were made available for evaluation. The trial conditions, reflecting maximum GAP are described under sweet corn.

The independent Canadian trials resulted in the following highest average residues:

Sum of spirotetramat and enol:

Sweet corn forage 7 days after last application: 0.027, 0.18, 0.18, 0.25 and 1.8 mg/kg.

Sum of residues of spirotetramat and 4 metabolites:

Sweet corn forage 7 days after last application: 0.06, 0.29, 0.32, 0.35 and 1.9 mg/kg.

The meeting estimated 0.32 mg/kg median and 1.9 mg/kg high residue for animal burden calculation.

In the independent Canadian trials 47–85 days after last application the residues in sweet corn stover were:

Sum of spirotetramat and enol: < 0.02, 0.023, 0.046, 0.11 and 0.41 mg/kg

Sum of residues of spirotetramat and 4 metabolites: < 0.05, 0.078, 0.10, 0.11, and 0.58 mg/kg.

In Australian trials 7 days after last application the sum of residues in/on sweet corn fodder was:

Spirotetramat and enol: 0.18, 0.2, 0.31, 0.28, 0.33, 0.5, 0.58 mg/kg.

Spirotetramat and 4 metabolites: 0.39, 0.47, 0.54, 0.57, 0.63, 1.0, and 1.36 mg/kg,

The Australian trials resulted in higher residues in sweet corn stover and fodder. Based on the Australian trials the Meeting estimated highest and median residues of 1.36 mg/kg and 0.57 mg/kg for sweet corn stover and fodder.

Farm animal feeding studies

Based on a dairy cattle feeding study and poultry metabolism study the 2008 JMPR estimated residue levels in animal commodities. No new information was provided.

Residues in animal commodities

The residues in sweet corn forage and stover do not increase the maximum animal burden that would affect the maximum, HR and median residue values estimated by the 2008 Meeting.

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 intake assessment.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FI 0326	Avocado	0.4		0.126	0.23
FI 0336	Guava	2		0.55	0.85
GC 0447	Sweet corn	1.5		0.31	0.75

DIETARY RISK ASSESSMENT

Long-term intake

The ADI is 0–0.05 mg/kgbw. The long-term intake calculated for the commodities considered by the present meeting is 0% of maximum ADI and did not affect the previously made long-term dietary estimates. Hence, a new risk assessment was not necessary.

Short-term intake

The ARfD is 1 mg/kgbw. The estimated short-term intakes of avocado, guava and sweet corn are up to 1% 2% of ARfD for the general population and children.

The Meeting concluded that the short-term intake of residues of spirotetramat from the uses

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TEBUCONAZOLE (189)

First draft prepared by Professor Eloisa Dutra Caldas, University of Brasilia, Brazil

EXPLANATION

Tebuconazole, a triazole fungicide, was last evaluated for residues in 2011 within the periodic re-evaluation program. It was listed by the 46th Session of CCPR (2014) for the evaluation of 2015 JMPR for additional data on residues. Residue data were submitted on banana and cucumber by the government of China, on ginseng by the government of Korea, and on asparagus, sunflower, onion, bulb and onion, green by the Government of the United States. The government of Korea also submitted storage stability and processing studies on ginseng.

METHOD OF ANALYSIS

The analytical method used to analyse fresh ginseng and processed products (dried and red ginseng and ginseng extracts) involves extraction with acetone, partition with chloromethane, cleaned up in a glass florisil column and quantification by GC-NPD. The method was satisfactorily validated for fresh ginseng at a LOQ of 0.03 mg/kg up to 0.5 mg/kg, and for processed commodities at a LOQ of 0.06 mg/kg up to 1 mg/kg (n=5, recovery in the range of 80–120% and CV< 10%).

Storage stability under frozen conditions

Samples of fresh and processed ginseng fortified with tebuconazole were stored at -20 °C up to 156 days (Kyung, 2014). The results are shown in Table 1.

Table 1 Stability of tebuconazole in samples of ginseng, stored at -20 °C

Matrix	Fortification level, mg/kg	Period of storage, days	Mean % remaining; n=5	Coefficient variation, %
Fresh ginseng	0.3	42	88.7	2.1
		52	74.7	2.5
Dried ginseng	0.6	142	101	2.4
		41	80.0	1.3
Red ginseng	0.6	156	80.4	3.0
		32	91.6	3.6
Water extract of dried ginseng	0.6	121	89.4	6.9
		44	81.4	1.7
Water extract of red ginseng	0.6	96	90.6	1.4
		25	93.2	1.9

USE PATTERNS

Table 2 shows the critical registered uses of tebuconazole in China, Republic of Korea and USA for crops relevant to this submission.

Table 2 Use patterns of tebuconazole in China, Republic of Korea and USA

Crop	Country	Formulation	Application					PHI (days)
			Method	Max. rate, kg ai/ha	kg ai/hL	Number	Interval, days	
Asparagus*	USA		Foliar	0.2		3/season	14	100[a] or 180
Banana	China	ME/WP/WG	Foliar	0.27	0.03	3		42
		EC	Foliar	0.28	0.031	3		35
		EW	Foliar, bagged	0.25	0.025	3		14
Cucumber	China	SC	foliar	0.12		3	7-10	5
Ginseng	Rep. Korea	SC	foliar	0.13		3		21
Onion (dry bulb) and garlic	USA		Over/in furrow at planting	0.65		1/season	---	---
			Foliar	0.2		4/season	10-14	7

Crop	Country	Formulation	Application					PHI (days)
			Method	Max. rate, kg ai/ha	kg ai/hL	Number	Interval, days	
			In furrow plus two foliar [b]	0.65 +0.2		3/season	In furrow then 10-14	7
Onion (green)	USA		Foliar	0.2		4/season	10-14	7
Sunflower	USA		Foliar	0.2		max 0.49 kg ai/ha	14	50

* Apply to the developing ferns after harvest of spears is completed;

[a] in California;

[b] If over/in furrow treatment used, then only two foliar applications are allowed. Otherwise four foliar treatments maybe used.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

A total of 77 foliar supervised trials were conducted in China, Republic of Korea and USA. Trials conducted in Republic of Korea were not at GLP, but the report provided information on the field conditions and analytical method used in the study. Unless indicated, concurrent determinations of residues in untreated crops gave residues <LOQ. Residues of tebuconazole within 25% of GAP are underlined and were considered for estimation of STMR, HR and maximum residue levels. When residues in samples harvested at a later stage were higher than that at the critical PHI, they will be selected for the estimations.

Banana

A total of 22 foliar residue trials were conducted in banana (bagged and ungagged) in China in 2013. The samples were analysed by LC-MS/MS and validated at a LOQ of 0.01 mg/kg. The results are shown in Table 3.

Table 3 Results of residue trials conducted with tebuconazole in/on banana in China in 2013 using 3 applications of an EW formulation

Location	Banana variety	Application			Residues			Trial No.
		Method	kg ai/ha	kg ai/hL	Portion analysed	DAT, days	mg/kg	
Guangzhou, Guangdong	Baxi	Non-bagged	0.25	0.028	Whole fruit	35	0.53	212-FT-01
					Pulp	35	0.07	
					Whole fruit	42	0.31	
					pulp	42	0.05	
	Baxi	bagged	0.25	0.028	Whole fruit	14	<0.01	
					Pulp	14	<0.01	
					Whole fruit	21	<0.01	
					pulp	21	<0.01	
Gaoyao, Guangdong	818 banana	Non-bagged	0.25	0.028	Whole fruit	35	0.10	212-FT-02
					Pulp	35	0.05	
					Whole fruit	42	0.07	
					pulp	42	0.05	
	818 banana	bagged	0.25	0.028	Whole fruit	14	<0.01	
					Pulp	14	<0.01	
					Whole fruit	21	<0.01	
					pulp	21	<0.01	
Nanning, Guangxi	Williams B6	Non-bagged	0.25	0.028	Whole fruit	35	0.21	212-FT-03
					Pulp	35	0.03	
					Whole fruit	42	0.13	
					pulp	42	0.03	
	Williams B6	bagged	0.25	0.028	Whole fruit	14	<0.01	
					Pulp	14	<0.01	
					Whole fruit	21	<0.01	
					pulp	21	<0.01	

Location	Banana variety	Application			Residues			Trial No.
		Method	kg ai/ha	kg ai/hL	Portion analysed	DAT, days	mg/kg	
Fangcheng Guangxi	Williams B6	Non-bagged	0.25	0.028	Whole fruit	35	0.17	212-FT-03
					Pulp	35	0.07	
					Whole fruit	42	0.13	
					pulp	42	0.05	
	Williams B6	bagged	0.25	0.028	Whole fruit	14	<0.01	
					Pulp	14	<0.01	
					Whole fruit	21	<0.01	
					pulp	21	<0.01	
Zhangzhou Fujian	Tinbao	Non-bagged	0.25	0.028	Whole fruit	35	0.37	212-FT-05
					Pulp	35	0.16	
					Whole fruit	42	0.17	
					pulp	42	0.08	
	Tinbao	bagged	0.25	0.028	Whole fruit	14	0.09	
					Pulp	14	0.04	
					Whole fruit	21	0.07	
					pulp	21	0.06	
Kaiyuan, Yunnan	Williams	Non-bagged	0.25	0.028	Whole fruit	35	0.74	212-FT-06
					Pulp	35	0.15	
					Whole fruit	42	0.30	
					pulp	42	0.09	
	Williams	bagged	0.25	0.028	Whole fruit	14	0.42	
					Pulp	14	0.10	
					Whole fruit	21	0.43	
					pulp	21	0.05	
Chengmai Hainan	Baxi	Non-bagged	0.25	0.028	Whole fruit	35	0.13	212-FT-07
					Pulp	35	0.06	
					Whole fruit	42	0.11	
					pulp	42	0.10	
	Baxi	bagged	0.25	0.028	Whole fruit	14	0.03	
					Pulp	14	<0.01	
					Whole fruit	21	0.01	
					pulp	21	<0.01	
Guangzhou, Guangdon	Baxi	Non-bagged	0.28	0.03	Whole fruit	35	0.54	213-FT-01
					Pulp	35	0.09	
					Whole fruit	42	0.47	
					pulp	42	0.11	
	Baxi	bagged	0.28	0.03	Whole fruit	14	0.15	
					Pulp	14	<0.01	
					Whole fruit	21	0.01	
					pulp	21	<0.01	
Zhaoqing, Guangdong	818 banana	Non-bagged	0.25	0.028	Whole fruit	35	0.20	213-FT-02
					Pulp	35	0.07	
					Whole fruit	42	0.16	
					pulp	42	0.04	
	818 banana	bagged	0.25	0.028	Whole fruit	14	0.01	
					Pulp	14	<0.01	
					Whole fruit	21	<0.01	
					pulp	21	<0.01	
Nanning, Guangxi	Williams B6	Non-bagged	0.28	0.03	Whole fruit	35	0.13	213-FT-03
					Pulp	35	0.02	
					Whole fruit	42	0.07	
					pulp	42	0.03	
	Williams B6	bagged	0.28	0.03	Whole fruit	14	<0.01	
					Pulp	14	<0.01	
					Whole fruit	21	0.01	
					pulp	21	<0.01	
Fangchenggang, Guangxi	Williams B6	Non-bagged	0.28	0.03	Whole fruit	35	0.19	213-FT-04
					Pulp	35	0.07	
					Whole fruit	42	0.15	
					pulp	42	0.10	

Location	Banana variety	Application			Residues			Trial No.
		Method	kg ai/ha	kg ai/hL	Portion analysed	DAT, days	mg/kg	
	Williams B6	bagged	0.28	0.03	Whole fruit	14	0.03	
					Pulp	14	<0.01	
					Whole fruit	21	0.01	
					pulp	21	<0.01	

Onion (Dry Bulb)

Seventeen supervised residue trials were conducted in USA in 1999 and 2002. Samples were analysed by a GC-NPD validated method at a LOQ of 0.05 mg/kg. The results from analysis of treated samples are summarized in Table 4.

Table 4 Residues resulting from tebuconazole application to dry bulb onions in USA and Canada (Reports IR-4 PR)

Region, country	Onion variety	Application		DAT	Residue, mg/kg	Report No; trial
		No.	kg ai/ha			
New Jersey, USA	-	4 foliar	0.18	7	<0.05	07196; 07196.99-NJ10
Texas, USA	-	4 foliar	0.18	7	<0.05	07196; 07196.99-TX11
Ohio, USA	-	4 foliar	0.18	7	<0.05	07196; 07196.99-OH*02
Salinas, CA, USA	-	4 foliar	0.18	7	0.06	07196; 07196.99-CA*34
Holtville, CA, USA	-	4 foliar	0.18	6	0.08	07196; 07196.99-CA18
Washington, USA	-	4 foliar	0.18	7	<0.05	07196; 07196.99-WA03
Oregon, USA	-	4 foliar	0.18	7	0.09	07196; 07196.99-OR05
Colorado, USA	-	4 foliar	0.18	7	<0.05	07196; 07196.99-CO02
Texas, USA	Early White	1 furrow + 2 foliar	0.63 + 0.18	7	0.02, 0.03 (0.02)	08365; 08365.02-TX02
Colorado, USA	Vantage	1 furrow + 2 foliar	0.63 + 0.18	6	0.03, 0.04 (0.04)	08365; 08365.02-CO01
Salinas, CA, USA	Ruby	1 furrow + 2 foliar	2 + 0.19	6	0.02, 0.04	08365; 08365.02-CA*04
Holtville, CA, USA	Cebolla	1 furrow + 2 foliar	0.63 + 0.18	6	0.02, 0.10 (0.06)	08365; 08365.02-CA17
Washington, USA	Pinnacle	1 furrow + 2 foliar	0.63 + 0.18	7	<0.02 (2)	08365; 08365.02-WA*01
Lynden, Ontario, CAN	Yellow Sets	1 furrow + 2 foliar	0.63 + 0.21	7	0.03, 0.06 (0.04)	08365; 08365.02-ON05
Lynden, Ontario, CAN	Spanish Sets	1 furrow + 2 foliar	0.63 + 0.18	7	0.03 (2)	08365; 08365.02-ON06
St-Paul-d'Abbotsford, Quebec, CAN	Broedrebe	1 furrow + 2 foliar	0.63 + 0.18	5	0.02 (2)	08365; 08365.02-QC02
St-Paul-d'Abbotsford, Quebec, CAN	Stuttgart	1 furrow + 2 foliar	0.63 + 0.18	5	<0.02 (2)	08365.02-QC03

Onion (Green)

Three supervised residue trials were conducted in USA and Canada in 1999. Samples were analysed by a GC-NPD validated method at a LOQ of 0.05 mg/kg. The results from analysis of treated samples are summarized in Table 5.

Table 5 Residues resulting from tebuconazole application to green onions in USA and Canada (Report: IR-4 PR No. 07245)

Region, country	Application No.	kg ai/ha	DAT	Residue, mg/kg	Trial
New Jersey, USA	4	0.19	8	0.06	07245.99-NJ11
Ontario, CA	4	0.19	7	0.80	07245.99-ON03
Oregon, USA	4	0.19	7	0.10	07245.99-OR11

Cucumber

Eleven residue trials were conducted in cucumber in China in 2014. The samples were analysed by LC-MS/MS and validated at a LOQ of 0.01 mg/kg, and the results are shown in Table 6.

Table 6 Results of residue trials conducted with tebuconazole in/on cucumber in China in 2014 using 3 applications of a SC formulation

Location	Banana variety	Application			Residues		Trial No.
		Method	kg a.i./ha	kg a.i./hL	DAT, days	mg/kg	
Changchun, Jilin	Shengchun	field	0.116	0.013	1	0.03	RP006-13Teb-01
					3	0.03	
					5	0.02	
					7	0.02	
	Shengchun	greenhouse	0.116	0.013	1	0.03	RP006-13Teb-02
					3	0.03	
					5	0.03	
					7	0.02	
Qingdao, Shandong	Huhuang	field	0.116	0.013	1	0.08	RP006-13Teb-04
					3	0.03	
					5	0.03	
					7	0.04	
	Budaojuncheng	greenhouse	0.116	0.013	1	0.06	RP006-13Teb-05
					3	0.07	
					5	0.04	
					7	0.02	
Hangzhou, Zhejiang	Zhexiu 302	field	0.116	0.013	1	0.08	RP006-13Teb-06
					3	0.06	
					5	0.03	
					7	0.02	
Changsha, Hunan	Shuyan 5	field	0.116	0.013	1	0.06	RP006-13Teb-07
					3	0.03	
					5	0.02	
					7	0.02	
Chongming, Yunnan	Bonei 2	field	0.116	0.013	1	0.06	RP006-13Teb-08
					3	0.04	
					5	0.03	
					7	0.02	
Guangzhou, Guangdong	Dadiao	field	0.116	0.013	1	0.17	RP006-13Teb-09
					3	0.12	
					5	0.07	
					7	0.03	
Zhangzhou, Fujian	Jinyou 48	field	0.116	0.013	1	0.21	RP006-13Teb-10
					3	0.19	
					5	0.09	
					7	0.03	
	Jinyou 10	greenhouse	0.116	0.013	1	0.12	RP006-13Teb-11
					3	0.07	
					5	0.06	
					7	0.03	
Hefei, Anhui	Jinyou 1	field	0.116	0.013	1	0.41	RP006-13Teb-13
					3	0.13	

Location	Banana variety	Application			Residues		Trial No.
		Method	kg a.i./ha	kg a.i./hL	DAT, days	mg/kg	
					5	0.11	
					7	0.04	

Ginseng

Nine supervised trials were conducted in Republic of Korea in ginseng in 2013 and 2014. Fresh harvested ginseng was rinsed with tap water to remove soil particle, ground and stored at -20 °C until analysis. The results are shown in Table 7.

Table 7 Residues of tebuconazole resulting from supervised trials on fresh ginseng conducted in Korea using SC formulation (Kyung, 2014)

Location, year	Application			PHI, days	Residues, mg/kg*
	Number	Rate, kg ai/ha	Water (L/ha)		
Yeongju, 2013	3	0.13	1900-2000	21	0.06 (3)
Geumsan, 2013	3	0.13	1900-2000	21	0.08 (3)
Jeungpyeong, 2013	3	0.13	1900-2000	21	0.06 (3)
Yeongju, 2014	3	0.13	1900-2000	21	0.03 (3)
Geumsan, 2014	3	0.13	1900-2000	21	0.04 (3)
Jeungpyeong, 2014	3	0.13	1900-2000	21	0.03 (3)
Yeongju, 2013-2014	6	0.13	1900-2000	21	0.03 (3)
Geumsan, 2013-2014	6	0.13	1900-2000	21	0.04 (3)
Jeungpyeong, 2013-2014	6	0.13	1900-2000	21	0.03 (3)

*Three replicate plots in each field. All samples were analysed in triplicates.

Asparagus

Eight supervised residue trials were conducted in USA in 2001. Tebuconazole was applied to the developing ferns after harvest of spears is completed. Samples were analysed by a LC-MS/MS validated method at a LOQ of 0.02 mg/kg. The results from analysis of treated samples are summarized in Table 8.

Table 8 Residues resulting from tebuconazole application to asparagus in USA (IR-4 PR No. 07991)

Region	Asparagus variety	Application		DAT	Residue, mg/kg	Trial
		No.	kg ai/ha			
Gonzales, CA	UC157	3	0.19	125	<0.02 (2)	07991.01-CA*54
Soledade, CA	UC157	3	0.19	125	<0.02 (2)	07991.01-CA*90
Stockton, CA	UC157	3	0.19	100	<0.02 (2)	07991.01-CA102
Holt, MI	Jersey Knight	3	0.19	184	<0.02 (2)	07991.01-MI14
East Lansing, MI	Jersey Giant	3	0.19	184	<0.02 (2)	07991.01-MI15
New Jersey	Jersey Giant	3	0.19	186	<0.02 (2)	07991.01-NJ16
Prosser, WA	Jersey Giant	3	0.19	199	<0.02 (2))	07991.01-WA23
Prosser, WA	Jersey Giant	3	0.19	197	<0.02 (2)	07991.01-WA24

Sunflower

Seven supervised residue trials were conducted in USA in 1997 and 1998. Samples were analysed by a GC-NPD validated method at a LOQ of 0.04 mg/kg. The results from analysis of treated samples are summarized in Table 9.

Table 9 Residues resulting from tebuconazole application to sunflowers in USA (IR-4 PR No. 06414)

Region, year	Application No.	kg ai/ha	DAT	Residue, mg/kg	Trial
Courtland, KS 1997	2	0.25	55	<0.04 (2)	06414.97-KS01
	2	1.3	55	<0.04, 0.04	
Prosper, ND 1997	2	0.25	58	<0.04 (2)	06414.97-ND08
	2	1.3	58	0.06 (2)	
Amenia, ND 1998	2	0.25	57	<0.04 (2)	06414.98-ND19
	2	1.3	57	0.04, 0.06	
Carrington, ND 1998	2	0.25	48	<0.04 (2)	06414.98-ND20
	2	1.3	48	<0.04 (2)	
Minot ND 1998	2	0.25	56	<0.04, 0.04 (0.04)	06414.98-ND21
	2	1.3	56	0.05, 0.09	
Scandia, KS 1998	2	0.25	55	<0.04 (2)	06414.98-KS01
	2	1.3	55	0.09, 0.10	
Belleville, KS	2	0.25	55	<0.04	06414.98-KS02
	2	1.3	55	0.125, 0.128	

Processing studies

After rinsing with tap water, fresh ginseng was dried in hot air at 60 °C reaching a moisture content under 14% to yield dried ginseng. Washed fresh ginseng was steamed for 3 hours at 98 °C, dried at 65 °C to a moisture content of 50~55% and ground to yield red ginseng. Dried ginseng or red ginseng was cut into about 1 cm length and extracted three times in a refluxing extractor with water at 85 °C for about 18 hours. The water was evaporated reaching 72 °Brix to yield water extract of dried or red ginseng (Kyung, 2014). Residues in fresh and processed ginseng and the respective processing factors are shown in Table 10.

Table 10 Residues of tebuconazole in ginseng processed commodities and calculated processed factors

Matrix	Residues, mg/kg*	Processing factors	Residues, mg/kg*	Processing factors	Residues, mg/kg*	PF	Processing factors, best estimate underlined
Fresh ginseng	0.06 0.08 0.06	-	0.03 0.04 0.03	-	0.03 0.04 0.03	-	
Dried ginseng	0.12 0.15 0.12	2 1.88 2	0.08 0.10 0.09	2.67 2.5 3	0.08 0.10 0.07	2.67 2.5 2.33	1.88, 2 (2), 2.33, 2.5 (2), 2.67 (2) and 3
Red ginseng	<0.06 0.08 <0.06	<1 1 <1	<0.06 <0.06 <0.06	<2 <2 <2	<0.06 <0.06 <0.06	<2 <1.5 <2	<1 (2), 1, <1.5 and <2 (5)
Water extract of dried ginseng	0.20 0.26 0.19	3.33 3.35 3.17	0.12 0.15 0.10	4 3.75 3.33	0.13 0.16 0.11	4.3 4 3.7	3.17, 3.33 (3), 3.35, 3.7, 3.75, 4 and 4.3
Water extract of red ginseng	0.08 0.15 0.06	1.33 1.87 1	0.08 0.06 <0.06	2.67 1.5 <2	0.08 0.09 <0.06	2.67 2.25 <2	1, 1.33, 1.5, 1.87, 2.25, 2.67 (2), <2 (2)

*Three replicate plots in each field. All samples were analysed in triplicates.

APPRAISAL

Tebuconazole a triazole fungicide was last evaluated for residues in 2011 within the periodic re-review programme. It was listed by the Forty-sixth Session of CCPR (2014) for the evaluation in the 2015 JMPR for additional data on residues. Data was submitted for banana, cucumber, ginseng, asparagus, sunflower, onion bulb; and onion, green. The residue definition for plant commodities for enforcement and risk assessment purposes is tebuconazole. The ADI for tebuconazole is 0-0.03 mg/kg bw and the ARfD is 0.3 mg/kg bw.

Method of analysis and stability of residues

A GC-NPD analytical method was satisfactorily validated for the analysis of tebuconazole in fresh ginseng at a LOQ of 0.03 mg/kg up to 0.5 mg/kg and for processed commodities at a LOQ of 0.06 mg/kg up to 1 mg/kg.

Tebuconazole residues were shown to be stable under frozen conditions (at -20 °C) in fresh ginseng for at least 52 days; in dried ginseng for at least 142 days; in red ginseng for at least 96 days; and in ginseng water extracts for at least 121 days.

The sample storage period used in the trials for ginseng and other commodities evaluated by the present Meeting was within the storage period that guaranteed that the residues in the samples were not degraded.

Residues resulting from supervised trials

Banana

In China, the critical GAPs for tebuconazole in banana is 3 × 0.28 kg ai/ha and 35 days PHI for unbagged banana and 3 × 0.25 kg ai/ha and 14 days PHI for bagged banana.

In eleven trials conducted with unbagged banana in China according to GAP, residues in the whole fruit were 0.10, 0.13 (2), 0.17, 0.19, 0.20, 0.21, 0.37, 0.53, 0.54 and 0.74 mg/kg. Residues in the pulp were 0.02, 0.03, 0.05, 0.06, 0.07 (4), 0.09, 0.15 and 0.16 mg/kg.

In eleven trials conducted with bagged banana according to GAP, residues in the whole fruit were < 0.01 (5), 0.01, 0.03 (2), 0.09, 0.15 and 0.42 mg/kg. Residues in the pulp were < 0.01 (9), 0.04 and 0.10 mg/kg.

Residues from trials conducted with unbagged banana gave the highest residues. The Meeting estimated a maximum residue level of 1.5 mg/kg, a STMR of 0.07 mg/kg and a HR of 0.16 mg/kg for tebuconazole in banana. These estimates replace the previous recommendations for tebuconazole in banana.

Onion, bulb and shallots

In the USA tebuconazole can be applied in onion and shallots at 4 foliar applications at 0.19 kg ai/ha or one furrow at 0.65 kg ai/ha plus 2 foliar at 0.19 kg ai/ha. The PHI is 7 days for both. In eight trials conducted in USA at the foliar GAP, residues were < 0.05 (5). 0.06. 0.08 and 0.09 mg/kg. In five trials conducted using furrow plus foliar application, residues were < 0.02, 0.02, 0.04 (2), and 0.06 mg/kg.

The Meeting agreed that the foliar only trials gave the highest residues and estimated a maximum residue level of 0.15 mg/kg, an STMR of 0.055 mg/kg and an HR of 0.09 mg/kg for tebuconazole in onion, bulb. These estimates replace the previous recommendation for tebuconazole in onion bulb.

The Meeting agreed to extrapolate this estimate to shallots.

Spring onion (Onion, green)

In the USA, tebuconazole can be applied in onions, green witht 4 foliar applications at 0.19 kg ai/ha and a 7 day PHI. In three trials conducted in the USA and Canada in 1999 at GAP, residues were 0.06, 0.10 and 0.80 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg, a STMR of 0.10 mg/kg and an HR of 0.8 mg/kg for tebuconazole in spring onion.

Cucumber

GAP for tebuconazole in cucumber in China is 3 × 0.12 kg ai/ha and 5 days PHI. In eight field trials conducted in the country according to GAP, residues were 0.02 (2), 0.03, 0.04, 0.06, 0.07, 0.09 and 0.11 mg/kg. In three protected trials residues were 0.03, 0.04 and 0.06 mg/kg.

Based on the residue data from field trials, the Meeting estimated a maximum residue level of 0.2 mg/kg, an STMR of 0.05 mg/kg and an HR of 0.11 mg/kg for tebuconazole in cucumber. These estimates replace the previous recommendations for tebuconazole in cucumber.

Ginseng

Six trials were conducted with tebuconazole in ginseng in Korea according to GAP (3 x 0.13 kg ai/ha; 21 days PHI). giving residues in fresh ginseng of 0.03 (2), 0.04, 0.06 (2) and 0.08 mg/kg. Three other trials conducted with 6 applications gave similar results.

The Meeting estimated a maximum residue level of 0.15 mg/kg, an STMR of 0.05 mg/kg and an HR of 0.08 mg/kg for tebuconazole in ginseng.

Asparagus

In the USA the critical GAP for tebuconazole in asparagus is to apply up to 3 × 0.19 kg ai/ha to the developing ferns after harvest of spears is completed; the PHI is 100 days. In three trials conducted in USA at GAP gave residues of < 0.02 (3) mg/kg.

The Meeting estimated a maximum residue level of 0.02* mg/kg and an STMR and HR of 0.02 mg/kg for tebuconazole in asparagus.

Sunflower

In the USA tebuconazole can be applied to sunflowers at a maximum rate of 0.49 kg ai/ha with a 50 day PHI. In seven trials conducted in the USA at GAP residues were < 0.04 (6) and 0.04 mg/kg.

The Meeting estimated a maximum residue level of 0.1 mg/kg and an STMR of 0.04 mg/kg for tebuconazole in sunflower seed.

Fate of residues in processing

Nine processing studies were conducted with ginseng yielding dried ginseng (≤ 14% water content). red ginseng (50–55% water content) and water extracts of dried and red ginseng. Median processing factors were 2.5 for dried ginseng, 1.0 for red ginseng, 3.35 for dried ginseng extract and 1.87 for red ginseng extract.

Using the estimated maximum residue level and STMR for ginseng (0.15 and 0.05 mg/kg, respectively) and the processing factor for dried ginseng (2.5), the Meeting estimated a maximum residue level of 0.4 mg/kg and an STMR of 0.125 mg/kg for ginseng, dried including red ginseng.

Using the processing factor for water extract of dried ginseng (3.35), the Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.17 mg/kg for ginseng extracts.

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 intake assessment.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
VS 0621	Asparagus	0.02*		0.02	0.02
FI 0327	Banana	1.5	0.05	0.07	0.16
VC 0424	Cucumber	0.2	0.15	0.05	0.11
VR 0604	Ginseng	0.15		0.05	0.08
DM 0604	Ginseng, extracts	0.5		0.17	
DV 0604	Ginseng, dried including red ginseng	0.4		0.125	
VA 0385	Onion, Bulb	0.15	0.1	0.055	0.09
VA 0388	Shallot	0.15		0.055	0.09
VA 389	Spring onion	2		0.1	0.8
SO 0702	Sunflower seed	0.1		0.04	

DIETARY RISK ASSESSMENT

Long-term intake

The IEDI of tebuconazole based on the STMRs estimated by this and previous Meetings for the 17 GEMS/Food regional diets were 2–9% of the maximum ADI of 0.03 mg/kg bw (see Annex 3 of the 2015 Report). The Meeting concluded that the long-term dietary intake of residues of tebuconazole is unlikely to present a public health concern.

Short-term intake

An ARfD for tebuconazole is 0.3 mg/kg bw. The Meeting estimated 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. The IESTI represented a maximum of 5% of the ARfD. The Meeting concluded that the short-term intake of tebuconazole residues from uses considered by the current Meeting was unlikely to present a public health concern.

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Dong, F., 2014	RP006-13Teb-08	Determination of the residues of tebuconazole on cucumber after spraying of 430g/L tebuconazole SC in Chongming, Yunnan Province, P.R. China. Institute of Plant Protection, Chinese Academy Agricultural Science. GLP, not published

Dong, F., 2014	RP006-13Teb-09	Determination of the residues of tebuconazole on cucumber after spraying of 430g/L tebuconazole SC in Guangzhou, Guangdong Province, P.R. China.. Institute of Plant Protection, Chinese Academy Agricultural Sciences. GLP, not published
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Dong, F., 2014	RP006-13Teb-13	Determination of the residues of tebuconazole on cucumber after spraying of 430g/L tebuconazole SC in Hefei, Anhui Province, P.R. China. Institute of Plant Protection, Chinese Academy Agricultural Sciences. GLP, not published
Kyung, K. S., 2014	MFDS 2013-17889	Magnitude of tebuconazole residues in ginseng and its processing products in Korea. Chungbuk National University. Non - GLP. Unpublished
Liu, Y, 2013	212-FT-01	Determination of the residues of tebuconazole on banana after spraying of 250g/L tebuconazole EW in Panyu, Guangzhou, Guangdong Province, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture. P. R. China.. GLP, not published
Liu, Y, 2013	212-FT-02	Determination of the residues of tebuconazole on banana after spraying of 250g/L tebuconazole EW in Gaoyao,Zhaoqing, Guangdong Province, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture. P. R. China. GLP, not published
Liu, Y, 2013	212-FT-03	Determination of the residues of tebuconazole on banana after spraying of 250g/L tebuconazole EW in Nanning, Guangxi, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture. P. R. China. GLP, not published
Liu, Y, 2013	212-FT-04.	Determination of the residues of tebuconazole on banana after spraying of 250g/L tebuconazole EW in Fangchenggang, Guangxi, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture. P. R. China. GLP, not published
Liu, Y, 2013	212-FT-05.	Determination of the residues of tebuconazole on banana after spraying of 250g/L tebuconazole EW in Zhangzhou in Fujian Province, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture. P. R. China. GLP, not published
Liu, Y, 2013	212-FT-06	Determination of the residues of tebuconazole on banana after spraying of 250g/L tebuconazole EW in Kaiyuan ,Yunnan, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture. P. R. China. GLP, not published
Liu, Y, 2013	212-FT-07	Determination of the residues of tebuconazole on banana after spraying of 250g/L tebuconazole EW Chengmai , Hainan, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture. P. R. China. GLP, not published
Liu, Y, 2013	213-FT-01	Determination of the residues of tebuconazole on banana after spraying of 250g/L tebuconazole EC in Panyu,Guangzhou, Guangdong Province, P.R. China.. Institute for Control of Agrochemicals, Ministry of Agriculture. P. R. China. GLP, not published
Liu, Y, 2013	213-FT-02	Determination of the residues of tebuconazole on banana after spraying of 250g/L tebuconazole EC in Gaoyao, Zhaoqing, Guangdong Province, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture. P. R. China. GLP, not published
Liu, Y, 2013	213-FT-03	Determination of the residues of tebuconazole on bananar after spraying of 250g/L tebuconazole EC in Nanning, Guangxi, P.R. China. Institute for Control of Agrochemicals, Ministry of Agriculture. P. R. China. GLP, not published
Liu, Y, 2013	213-FT-04	Determination of the residues of tebuconazole on bananar after spraying of 250g/L tebuconazole EC in Fangchenggang, Guangxi, P.R. ChinaInstitute for Control of Agrochemicals, Ministry of Agriculture. P. R. China. GLP, not published
Thompson, D., 2005		Tebuconazole: Magnitude of the Residue on Onion (Dry); Author: David Thompson, IR-4 Project HQ, Rutgers, The State University of New Jersey, Princeton, NJ 08540; dated: 28 April 2005. GLP, not published
Thompson, D., 2006		Tebuconazole: Magnitude of the Residue on Onion (Dry); Author: David Thompson, IR-4 Project HQ, Rutgers, The State University of New Jersey, Princeton, NJ 08540; dated: 15 February 2006 GLP, not published
Thompson, D., 2006		Tebuconazole: Magnitude of the Residue on Onion (Green); Author: David Thompson, IR-4 Project HQ, Rutgers, The State University of New Jersey, Princeton, NJ 08540; dated: 19 July 2006. GLP, not published
Corley, J., 2000		Tebuconazole: Magnitude of the Residue on Sunflower; Author: Johannes Corley, IR-4 Project HQ, Rutgers, The State University of New Jersey, Princeton, NJ 08540; dated: 17 April 2000. GLP, not published

TRIFLOXYSTROBIN (213)

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EXPLANATION

Trifloxystrobin is a broad-spectrum contact fungicide for foliar application. It was first evaluated by the JMPR in 2004 (T, R) and 2012 (R). The 2004 Meeting established an ADI of 0–0.04 mg/kg bw and decided that ARfD was not necessary. The Meeting agreed that the residue definition for enforcement purposes for plant commodities should be trifloxystrobin per se, for animal commodities and dietary intake assessment the residue definition should be parent compound and CGA 321113 (expressed as trifloxystrobin equivalents).

Data on identity, formulations, physical and chemical properties, metabolism and environmental fate of trifloxystrobin were submitted to the JMPR in 2004. No new information was made available. Supervised trials data were submitted for a number of commodities for evaluation by the 2004, and 2012 JMPR.

Trifloxystrobin was listed by the 46th Session of CCPR (2014) for the evaluation by the 2015 JMPR for additional MRLs. Additional supervised trial data were submitted for evaluation on dry soya beans, lentils, chick peas and peas together with new data, which were not available in the first evaluation in 2004.

METHODS OF RESIDUE ANALYSIS

Analytical methods

The Meeting received descriptions and validation data for analytical methods for residues of trifloxystrobin (CGA 321113) and several other metabolites in different plant matrices.

The DFG method S19, evaluated in 2004, is suitable for enforcement. Analytical methods used in residue trials evaluated by the present Meeting are summarized below.

Method 00742 (Nuesslein, F 2002)

The method was developed and validated for the determination of residues of parent trifloxystrobin and CGA 321113 (metabolite) in/on sample materials of carrots, Brussels sprouts, cabbages, tomatoes, red peppers and lettuce. Both analytes were extracted from plant materials using a mixture of acetonitrile/water. After filtration and concentration to the aqueous remainder, the acidified crude extract was purified by liquid-liquid partition on a ChemElut cartridge, thereby partitioning the analytes in a mixture of cyclohexane / ethyl acetate. The residues were quantified by reverse-phase HPLC with Turbo-Ionspray MS/MS-detection. The limit of quantitation (LOQ) was 0.02 mg/kg in all matrices. Recoveries for trifloxystrobin ranged from 72 to 99% with mean values between 81 and 93% and relative standard deviations between 0.7 and 10.4%. In the case of CGA 321113, recoveries were between 71 and 103% with mean values between 83 and 100% and relative standard deviations between 0.6 and 8.1%. The repeatability was tested with carrots and tomatoes.

Supplement E001 for method 00742 (Nuesslein, F 2003)

The analytical method 00742 was validated for the determination of trifloxystrobin and CGA 321113 in additional plant materials. Recovery tests were performed at fortification levels of 0.02 mg/kg, 0.20 mg/kg and 2.0 mg/kg with the sample materials beans (beans with pods), broccoli (head), cauliflower (head), cherries (fruit), cucumbers (fruit), currants (fruit), leeks (shoots), melons (fruit, peel), plums (fruit) and strawberries (fruit, jam and preserves). The LOQ was 0.02 mg/kg in all matrices. Individual recovery rates of trifloxystrobin ranged from 68 to 103% with overall standard deviations (RSD) between 1.1 and 9.3%. In the case of CGA 321113, recoveries were between 81 and

101% with overall standard deviations (RSD) between 1.4 and 5.4%. The repeatability was tested successfully with cauliflower and strawberries.

Method 00765 (Sur, R 2003.)

The method was developed and validated to determine trifloxystrobin, its metabolite CGA 321113, and cyproconazole in/on cucumbers, green peppers, melons and tomatoes. The active substances and the metabolite were extracted twice from the sample material with an acetonitrile/water mixture. After filtration and dilution the extract solution was subjected to HPLC-MS/MS analysis for quantitation. For all three analytes two transitions are recorded. The LOQ for all analytes was 0.01 mg/kg. The method was validated by spiking control samples with the analytes at fortification levels of 0.01 and 0.1 mg/kg. The overall mean recovery for the quantifier transition was 99% (RSD 3.8%, n=32) for trifloxystrobin and 100% (RSD 2.7%, n=32) for CGA 321113. For the qualifier transition the overall mean recovery was 100% (RSD 4%, n=32) for trifloxystrobin and 96% (RSD 6.2%, n = 32) for CGA 321113. The repeatability was tested with melons and tomatoes.

Method 01013 (Brumhard, B and Stuke, S 2007.)

The method was developed for the determination of residues of trifloxystrobin and metabolite CGA 321113, and other actives substances and their metabolites in/on plant material (citrus fruit, pea green seed, wheat grain, rape seed, and corn green material). All analytes were extracted from plant materials using a mixture of acetonitrile/water. After filtration of the extract, the stable isotopically labelled analytes were added. The solution was made up to volume, diluted and subjected to reversed phase HPLC-MS/MS without a further clean-up step.

The LOQ for the determination of trifloxystrobin and CGA 321113 was 0.01 mg/kg in all matrices tested. The method was validated by spiking control samples with the analytes at fortification levels of 0.01 and 0.1 mg/kg. Mean recoveries for each fortification level and the overall mean recovery were within the 70–110% range for all matrices. The correlation between the injected amount of substance and the detector response was linear for solvent standards ranging from 0.005 to 50 µg/L. Possible matrix effects were eliminated by the internal standard procedure using isotopically stable labelled standards.

Apparent residues in control samples were below 0.3 × LOQ. Two MRM transitions were monitored and calculated for each analyte and each matrix tested. The HPLC-MS/MS method is highly specific and an additional confirmatory method is not necessary. The repeatability was tested successfully with all matrices. Relative standard deviations were below 20% for all analytes and sample materials. The method 01013 allows determination of trifloxystrobin-derived residues in crops with a LOQ of 0.01 mg/kg.

Method 01313 (Stuke, S 2013.)

The method was developed for the determination of residues of trifloxystrobin (CGA 279202) and metabolite CGA 321113, and their isomers CGA357262, CGA357261, CGA331409, and CGA373466 in/on corn green material, bean dry seed, wheat grain, rape seed, dried hops and orange fruit. All analytes were extracted from plant materials using a mixture of acetonitrile/water. After filtration by using celite and addition of ammonium acetate solution to adjust the pH, the extract was made up to volume, diluted and subjected to HPLC-MS/MS measurement. The LOQ for the determination of trifloxystrobin and CGA 321113 was 0.01 mg/kg in all matrices tested. The method was validated by spiking control samples with the analytes at fortification levels of 0.01 and 0.1 mg/kg (0.05 and 0.5 mg/kg in hops). Mean recoveries for each fortification level and the overall mean recovery were within the 70–110% range for trifloxystrobin and CGA 321113. The mean and the overall mean relative standard deviations at each fortification level were below 20%.

The results of the method validation were confirmed using a second and a third MRM transition for confirmation. The LOQ was 0.01 mg/kg for trifloxystrobin-derived residues.

Method 200177 (de Haan, RA 2002.)

The method was developed for the determination of trifloxystrobin and CGA 321113 in plant materials. The residues of trifloxystrobin and CGA 321113 were extracted from homogenised plant samples with acetonitrile/water (4 vol. + 1 vol.) in a blender. The suspension was vacuum filtered through a paper filter. The remaining solids were blended a second time with fresh solvent and filtered. The filtrates were combined and deuterated internal standard was added. The total volume was adjusted to 50 mL with acetonitrile/water (4 vol. + 1 vol.). A solid phase extraction was performed on a SPE column under slight vacuum. The column was rinsed with acetonitrile/water (4 vol. + 1 vol.) and the analytes were eluted with acetonitrile. The solution was evaporated to dryness, the dry residue was subsequently dissolved in acetonitrile/water (4 vol. + 1 vol.). The final determination was done with LC/MS/MS in the positive ion mode. The LOQ was 0.01 mg/kg.

The recovery values obtained during the validation of the above methods are summarized in Table 1.

Table 1 Recovery of trifloxystrobin and CGA321113 from different plant materials

Sample /method	Analyte	Spike level [mg/kg)	No of tests	Mean recovery [%]	RSD [%]
Method 00742 trifloxystrobin 409.2 →186.3 (145.2) amu; CGA 321113: 395.1→186.1 (145.2).					
Carrot	Trifloxystrobin	0.02, 0.2	10	85	5.9
	CGA321113		10	83	5.6
Brussels sprout	Trifloxystrobin	0.02, 0.2, 2.0	9	86	5.2
	CGA321113			90	5.6
Cabbage, head	Trifloxystrobin	0.02, 0.2, 2.0	9	84	5.3
	CGA321113		9	91	2.5
Lettuce, head	Trifloxystrobin	0.02, 0.2, 2.0	9	88	7.5
	CGA321113		9	95	3.0
Pepper	Trifloxystrobin	0.02, 0.2, 2.0	9	90	4.5
	CGA321113		9	96	3.3
Tomato	Trifloxystrobin	0.02, 0.2	10	89	9.5
	CGA321113		10	96	5.2
Method 00742/Supplement E001					
Bean	Trifloxystrobin	0.02, 0.2, 2.0	9	91	5.3
	CGA321113			91	5.1
Broccoli, head	Trifloxystrobin	0.02, 0.2, 2.0	9	93	3.6
	CGA321113			96	4.1
Cauliflower	Trifloxystrobin	0.02, 0.2	10	92	3.6
	CGA321113			93	4.1
Cherry, fruit	Trifloxystrobin	0.02, 0.2, 2.0	9	95	2.7
	CGA321113			94	2.9
Cucumber	Trifloxystrobin	0.02, 0.2, 2.0	9	94	3.5
	CGA321113			90	4.7
Currant	Trifloxystrobin	0.02, 0.2, 2.0	9	92	4.0
	CGA321113			95	4.3
Leek	Trifloxystrobin	0.02, 0.2, 2.0	9	93	1.9
	CGA321113			94	1.8
Melon, fruit	Trifloxystrobin	0.02, 0.2, 2.0	9	94	2.3
	CGA321113			94	2.6
Melon, peel	Trifloxystrobin	0.02, 0.2, 2.0	9	93	1.1
	CGA321113			94	1.4
Plum, fruit	Trifloxystrobin	0.02, 0.2, 2.0	9	94	1.3
	CGA321113			96	1.4
Strawberry, fruit	Trifloxystrobin	0.02, 0.2	10	86	9.3
	CGA321113			89	3.8
Strawberry, jam	Trifloxystrobin	0.02, 0.2, 2.0	9	94	5.3
	CGA321113			95	4.7
Strawberry, preserve	Trifloxystrobin	0.02, 0.2, 2.0	9	95	5.4
	CGA321113			95	5.4
Method 00765 trifloxystrobin 409.2 →186.3 (145.2) amu; CGA 321113: 395.1→186.1 (145.2).					
Cucumber	Trifloxystrobin	0.01, 0.1	6	94	2.9

Sample /method	Analyte	Spike level [mg/kg)	No of tests	Mean recovery [%]	RSD [%]
	CGA321113			99	3.0
Green pepper	Trifloxystrobin	0.01, 0.1	6	101	3.4
	CGA321113			102	3.3
Melon	Trifloxystrobin	0.01, 0.1	10	97	1.9
	CGA321113			98	107
Tomato	Trifloxystrobin	0.01, 0.1	10	101	1.9
	CGA321113			100	1.9
Method 01013 trifloxystrobin 409.2 →186.3 (145.2) amu; CGA 321113: 395.1→186.1 (145.2).					
Citrus fruit	Trifloxystrobin	0.01, 0.1	10	99	3.7
	CGA321113			102	8.8
Peas	Trifloxystrobin	0.01, 0.1	10	100	3.1
	CGA321113			101	4.7
Rape seed	Trifloxystrobin	0.01, 0.1	10	99	3.6
	CGA321113			103	5.6
Wheat grain	Trifloxystrobin	0.01, 0.1	10	98	3.1
	CGA321113			88	13.2
Corn green material	Trifloxystrobin	0.01, 0.1	10	104	5.6
	CGA321113			94	12.8
Method 200177 trifloxystrobin 409.2 →186.3 (145.2) amu; CGA 321113: 395.1→186.1 (145.2). TFS-d_3 412 →186; CGA 321113-d_3 398 →186					
Pepper, tomato	Trifloxystrobin	0.01	16	93	11.2
	CGA321113			91	13.6
Soya beans	Trifloxystrobin	0.01, 0.05, 0.2		86	6.4
	CGA321113			91	19

Stability of residues in stored analytical samples

Individual data on storage stability were evaluated by the 2004 JMPR. The results indicated that the residues of trifloxystrobin and CGA 321113 are stable under freezer storage conditions for at least 24 months (grapes, cucumbers, potato, and wheat grain, straw and whole plant) or 18 months (apple fruit, apple wet pomace, peanut nutmeat, peanut oil, and grape juice). No new information was provided.

USE PATTERNS

Various formulations of trifloxystrobin are registered for application in chickpeas, dry peas, lentils and soya (Table 2) for the control of various fungus diseases of Chickpea (Ch), dry peas (Dp), Lentils (L) and soya (S): *Alternaria* spp. (S), *Ascochyta lentis* (L), *Ascochyta pisi* (Dp), *Ascochyta rabiei* (Ch), *Botrytis cinerea* (CH, L), *Cercospora kikuchii* (S), *Cercospora sojina* (S), *Colletotrichum truncatum* (Ch,L,S), *Corynespora cassiicola* (S), *Diaporthe phaseolorum* (S), *Erysiphe diffusa* (S), *Macrophomina phaseolina* (S), *Microsphaera diffusa* (S), *Mycosphaerella pinodes* (Dp), *Phakopsora pachyrhizi* (S), *Phomopsis longicolla* (S), *Rhizoctonia solani* (S), *Sclerotinia sclerotiorum* (Ch, L, S), and *Septoria glycines* (S).

Table 2 Composition of trifloxystrobin formulations

Formulation	Active ingredient content
SC 500	375 g/L trifloxystrobin + 125 g/L prothionazole
SC 535	375 g/L trifloxystrobin + 160 g/L cyproconazole
EC 267.5	187.5 g/L trifloxystrobin + 80 g/L cyproconazole
SC 325	150 g/L trifloxystrobin + 175 g/L prothionazole
EC 250	125 g/L trifloxystrobin + 125 g/L propiconazole
SC 300	100 g/L trifloxystrobin + 200 g/L tebuconazole

Table 3 Registered uses for foliar application of trifloxystrobin in peas, lentils and soya beans

Crop	Country	Formulation	Application			PHI
			Rate, [kg ai/ha]		No.	(days)
Chickpea	Canada	SC 325		0.132	1–2	30 (seed) [a]

Crop	Country	Formulation	Application			PHI
			Rate, [kg ai/ha]		No.	(days)
Chickpea	USA	SC 325		0.12	1–2	30 (seed) [a]
Dry peas	Canada	SC 325		0.132	1–2	30 (seed) [a]
Dry peas	USA	SC 325		0.12	1–2	30 (seed) [a]
Lentils	Canada	SC 325		0.132	1–2	30 (seed) [a]
Lentils	USA	SC 325		0.12	1–2	30 (seed) [a]
Soya bean	Brazil	EC 267.5	0.0563	0.075	2	30
		SC 325	0.045–	0.060	2	30
		SC 300	0.04–	0.06	1–4	30
		SC 535	0.0563–	0.075	2	30
		EC 250		0.05	2	30
Soya bean	Canada	EC 250		0.0625	max. 2	20 [b]
		SC 325		0.0858	max. 2	20 [b]
Soya bean	USA	EC 250		0.0913	max. 3	21 [b]
Soya bean	USA	SC 500	0.1095	0.1271	max. 3	21 [b]

[a] Do not apply within 7 days of cutting or swathing of the crop for forage

[b] Do not graze or feed soya bean forage or hay

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Numerous residue trials were performed according to GLP with different mixture formulations of trifloxystrobin.

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 averages of detected residues, used for estimation of residue levels, are double underlined.

Residues have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. However, the calculations were made with Excel utilising all digits. Residue values from the trials conducted according to GAP have been used for the estimation of maximum residue levels. Those results included in the evaluation are underlined.

Pulses

Soya beans

Four field trials were conducted in Canada (Ardiel, KD 2007.) with application rates of two times 0.0625 kg trifloxystrobin/ha and sampling 19 or 21 days after the last application.

In 2003 a total of twenty residue trials were performed in the USA. Trifloxystrobin was applied three times at application rates of 0.086 to 0.095 kg trifloxystrobin/ha. Samples of soya bean (seed) were taken at days 19 to 24 after the last application. In 2005 an additional twenty residue trials were conducted in the USA. Trifloxystrobin was applied three times at application rates of 0.122 to 0.134 kg ai/ha. Samples of soya bean (seed) were taken at days 19 to 23 after the last application.

The residues of trifloxystrobin and CGA 321113 were determined according to method 200177. The LOQ was 0.01 mg/kg.

Altogether 16 trials were reported from Brazil according to the Brazilian use patterns with two applications up to 0.075 or four applications up to 0.06 kg trifloxystrobin/ha and a PHI of about 30 days. The LOQ was 0.01–0.02 mg/kg (Anon. 2010, Anon. 2012a, 2012b, Resende, G 2011, Santiago, L 2012a, 2012b, Galhiane, MS and de Sousa, SL 2006a, Galhiane, MS and de

Sousa, SL 2006b, Galhiane, MS and de Sousa, SL 2006c, Galhiane, MS and de Sousa, SL 2006d, Galhiane, MS and de Sousa, SL 2006e, Galhiane, MS and de Sousa, SL 2006f.).

The results of the trials are summarized in Tables 4–6.

Table 4 Results of residue trials conducted with 250 EC trifloxystrobin (TFS) in/on soya bean seed in Canada

Study Trial No. Plot No. Year			Application					Residues [mg/kg] [a]		
	Crop Variety	Country	No	kg/ha (as)	kg/hL (as)	GS	DAT	TFS	CGA	
Canadian GAP: 2 time 0.0625 kg ai/ha with PHI of 30 days.										
06BCS-14 05BCS06-01-05D CND-05BCS06-01-05D, 2005	DeKalb, DKBOO-99	Canada Rock-wood	2	0.0625	0.0179–0.0182	87	9 9 14 14 21 21 24 24 30 30	0.012 < 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
06BCS-14 05BCS06-02-05H CND-05BCS06-02-05H, 2005	Pioneer 90B73	Canada Green-field	2	0.0625	0.0166–0.0182	86	19 19	< 0.01 < 0.01	< 0.01 < 0.01	
06BCS-14 05BCS06-03-05H CND-05BCS06-03-05H, 2005	SeCan Raptor	Canada Breslau	2	0.0625	0.0168–0.0185	85	19 19	< 0.01 < 0.01	< 0.01 < 0.01	
06BCS-14 05BCS06-04-05H CND-05BCS06-04-05H, 2005	Herbic. Inc. 26-02R	Canada St-Pie America, North	2	0.0625	0.0230–0.0231	77	21 21	< 0.01 < 0.01	< 0.01 < 0.01	

FL=Formulation

No=number of applications

GS=growth stage at last application

DAT=days after last treatment

TFS= trfloxystrobin

[a] Residues were measured in dry seeds.

Table 5 Results of residue trials conducted with trifloxystrobin in/on soya bean seed in Brazil

Study Trial No. Plot No. Year	Crop Variety	Country Location	Application					DDAT	Residues [mg/kg] [a]		
			FL	No	kg/ha (as)	kg/hL (as)	GS		TFS	CGA 321113	Sum
Brazil GAP: SC325 max 2×75 g/ha PHI=30 days											
F11-035 F11-035-01 2011	Soya bean TMG 7161 RR	Brazil Paulinia	325 SC	2	0.06000.0650	0.0300-0.0325	75	25 30 35	< 0.01 < 0.01 < 0.01	0.01 < 0.01 < 0.01	< 0.01
F11-035 F11-035-02 2012	Soya bean Monsoy 7808 RR	Brazil Ribeirão Preto	325 SC	2	0.06080.0615	0.0304-0.0308	71	25 30 35	< 0.01 < 0.01 < 0.01	0.02 0.02 0.01	0.03
F11-035 F11-035-03 2012	Soya bean BRS GO 7560	Brazil Uber-lândia	325 SC	2	0.05970.0608	0.0298-0.0304	79	25 30 35	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01
F11-035 F11-035-04 2012	Soya bean ANTA 82	Brazil Trindade	325 SC	2	0.0600	0.0300	75	30	< 0.01	0.01	< 0.01
F11-035 F11-035-05 2011	Soya bean CD206	Brazil Castro	325 SC	2	0.06000.0653	0.0300-0.0327	65	32	< 0.01	0.01	< 0.01
F11-036 F11-036-01 2011	Soya bean TMG 7161 RR	Brazil Paulinia	325 SC	2	0.0766	0.0383	75	25 30 35	< 0.01 < 0.01 < 0.01	< 0.01 0.01 0.01	0.02

Study Trial No. Plot No. Year	Crop Variety	Country Location	Application					DDAT	Residues [mg/kg] [a]		
			FL	No	kg/ha (as)	kg/hL (as)	GS		TFS	CGA 321113	Sum
F11-036 F11-036-02 2012	Soya bean Monsoy 7808 RR	Brazil Ribeirão Preto	325 SC	2	0.07500.0759	0.0375- 0.0380	71	25 30 35	< 0.01 < 0.01 < 0.01	0.02 0.02 0.02	0.03
F11-036 F11-036-03 2012	Soya bean M 7908 RR	Brazil Uber- lândia	325 SC	2	0.07140.0774	0.0357- 0.0387	75	25 30 35	< 0.01 < 0.01 < 0.01	< 0.01 0.01 0.01	0.02
F11-036 F11-036-04 2012	Soya bean ANTA 82	Brazil Trindade	325 SC	2	0.07500.0762	0.0375- 0.0381	75	30	< 0.01	< 0.01	< 0.01
F11-036 F11-036-05 2011	Soya bean CD 206	Brazil Castro	325 SC	2	0.07660.0776	0.0383- 0.0388	65	32	< 0.01	0.01	
Brazilian GAP: SC 300: 0.05 kg ai/ha 4 times; 0.06 kg ai/ha 2 times, PHI 30 days											
F09-022 F09-022-02 2010	Soya bean M7908 RR	Uberlan- dia / MG	300 SC	4	0.073-0.080	0.037- 0.040	83	25	< 0.01	< 0.01	< 0.01
F09-022 F09-022-03 2010	Soya bean NK 8350	Ponta Grossa / Parana	300 SC	4	0.07250.0849	0.0363- 0.0425	60	28	< 0.01 (n.d.)	< 0.01	< 0.01
F09-022 F09-022-04 2010	Soya bean Valiosa	Goiania / GO	300 SC	4	0.075-0.077	0.038- 0.039	85	29	< 0.01	< 0.01 (n.d.)	< 0.01
UNESP RA- 992/06 FR05BRA001 BRA- FR05BRA001- P1-A, 2005	Soya bean CD 205	EAE- Paulinia/ SP	75 WG	4	0.050	0.025	86	30	< 0.02	< 0.01 (n.d.)	< 0.02
UNESP RA- 993/06 FR05BRA001 BRA- FR05BRA001- P2-A,2005	Soya bean CD 201	Brazil Londrina - PR	75 WG	4	0.050	0.025	85	30	< 0.02	< 0.01 (n.d.)	< 0.02
UNESP RA- 994/06 FR05BRA001 BRA- FR05BRA001- P3-A,2005	Soya bean Xingu	Brazil Rondono- polis - MT	75 WG	4	0.050	0.025	83	30	< 0.01 (n.d.)	< 0.01 (n.d.)	< 0.01

FL=Formulation

No=number of applications

GS=growth stage at last application

DAT=days after last treatment

TFS: trifloxystrobin

n.d.=residues below limit of detection

[a] Residues were measured in dry seeds,

Table 6. Results of residue trials conducted with trifloxystrobin in/on soya bean in the USA.

Study Trial No. Plot No. Year			Application						Residues [mg/kg] [a]		
	Variety	Country	FL	No	kg/ha (as)	kg/hL (as)	GS	DAT	TFS	CGA 321113	Sum
USA GAP 250EC, 0.09125 kg/ha max 3 times with PHI of 21 days											
RCTFY004 FL079-03H USA-FL079-03H-B, 2003	Hartz Seed H6686RR	USA Tifton, Georgia	250 EC	3	0.092	0.064	86	20 20	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RCTFY004 FL080-03H USA-FL080-03H-B, 2003	NK S73-Z5	USA Molino, Florida	250 EC	3	0.084–0.092	0.043–0.044	79	24 24	< 0.01 < 0.01	< 0.01 < 0.01	< 0,.01
RCTFY004 FL081-03H USA-FL081-03H-B, 2003	Hornbeck 5588RR	USA Proctor, Arkansas	250 EC	3	0.091–0.092	0.063–0.066	91	21 21	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RCTFY004 FL082-03H USA-FL082-03H-B, 2003	Delta King 5661 RR	USA Newport, Arkansas	250 EC	3	0.092–0.093	0.049–0.049	79	20 20	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RCTFY004 FL083-03D USA-FL083-03D-B 2003	S56-D7	USA Leland, Mississippi	250 EC	3	0.087–0.092	0.068–0.078	77	18 18 21 21 26 26 27 27 32 32	0.055 0.020 0.018 0.035 <u>0.265</u> 0.012 < 0.01 0.030 0.019 0.014 0.015	0.022 < 0.01 < 0.01 < 0.01 <u>≤ 0.01</u> < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.265
RCTFY004 FL084-03D USA-FL084-03D-B : yes 2003	FS HT322 STS	USA Seymour, Illinois	250 EC	3	0.092–0.093	0.063–0.068	85	18 18 21 21 24 24 27 27 33 33	< 0.01 < 0.01 <u>< 0.01</u> <u>< 0.01</u> < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 <u>< 0.01</u> <u>< 0.01</u> < 0.01 < 0.01 0.017 0.014 < 0.01 < 0.01	
RCTFY004 FL085-03H USA-FL085-03H-B, 2003	NK S26 C9	USA Springfield, Nebraska	250 EC	3	0.092	0.063–0.064		21 21	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RCTFY004 FL086-03H USA-FL086-03H-B, 2003	Patriot Round-up Ready	USA Stilwell, Kansas	250 EC	3	0.087–0.095	0.060–0.066	85	22 22	0.041 0.040 0.041	0.016 0.016 0.016	0.057
RCTFY004 FL087-03H USA-FL087-03H-B, 2003	Becks 323RR	USA Oxford, Indiana	250 EC	3	0.091–0.093	0.048–0.053	79	20 20	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RCTFY004 FL088-03H USA-FL088-03H-B, 2003	92B94	USA Bagley, Iowa	250 EC	3	0.090–0.094	0.040–0.041	77	20 20	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RCTFY004 FL089-03H USA-FL089-03H-B, 2003	BT-402	USA Carlyle, Illinois	250 EC	3	0.090–0.094	0.048–0.061	79	19 19	0.010 < 0.01 0.01	0.013 0.020 0.017	0.027

Study Trial No. Plot No. Year			Application						Residues [mg/kg] [a]		
	Variety	Country	FL	No	kg/ha (as)	kg/hL (as)	GS	DAT	TFS	CGA 321113	Sum
RCTFY004 FL090-03H USA-FL090-03H-B, 2003	GL2301 RR	USA Saginaw, Michigan	250 EC	3	0.092	0.047–0.048	81	20 20	< 0.01 < 0.01 < 0.01	0.013 < 0.01 0.013	0.023
RCTFY004 FL091-03H USA-FL091-03H-B, 2003	Mycogen 44150	USA Gardner, North Dakota	250 EC	3	0.091–0.093	0.031–0.031	79	20 20	< 0.01 < 0.01 < 0.01	0.013 < 0.01 0.013	0.023
RCTFY004 FL092-03H USA-FL092-03H-B, 2003	SC 9373	USA New Holland, Ohio	250 EC	3	0.089–0.093	0.060–0.061	77	19 19	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RCTFY004 FL093-03H USA-FL093-03H-B, 2003	Dekalb 06-51	USA Campbell, Minnesota	250 EC	3	0.091–0.092	0.032–0.033	93	20 20	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RCTFY004 FL094-03H USA-FL094-03H-B, 2003	Pioneer91m50	USA Geneva, Minnesota	250 EC	3	0.091–0.094	0.062–0.063	95	20 20	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RCTFY004 FL095-03H USA-FL095-03H-B, 2003	Dekalb 3151	USA Sheridan, Indiana	250 EC	3	0.091–0.094	0.055–0.061	81	21 21	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RCTFY004 FL096-03H USA-FL096-03H-B, 2003	Rough Rider	USA Northwood North Dakota	250 EC	3	0.091–0.094	0.033–0.033	95	21 21	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RCTFY004 FL097-03H USA-FL097-03H-B, 2003	Pioneer 93B86	USA Richland, Iowa	250 EC	3	0.090–0.092	0.043–0.059	91	19 19	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RCTFY004 FL098-03H USA-FL098-03H-B, 2003	Brunner BR-1500 RR	USA Arkansaw, Wisconsin	250 EC	3	0.095	0.033–0.033	81	20 20	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
US GAP 500 SC: 0.1095–0.1271 max 3 times, PHI 21 days											
RATFY011 TF001-05H USA-TF001-05H-B, 2005	S73-Z5	USA Tifton	500 SC	3	0.128	0.0977–0.100	87	21 21	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RATFY011 TF002-05H USA-TF002-05H-B, 2005	Pioneer 95B96	USA Molino	500 SC	3	0.123–0.129	0.107–0.110	88	19 19	< 0.01 0.029	< 0.01 < 0.01	0.02
RATFY011 TF003-05H USA-TF003-05H-B, 2005	AG4403 RR	USA Proctor	500 SC	3	0.128–0.129	0.0895–0.0902	92	20 20	0.030 0.022 0.026	0.017 0.016 0.0165	0.043
RATFY011 TF004-05H USA-TF004-05H-B, 2005	DPL 5806 RR	USA Cheneyville	500 SC	3	0.128–0.129	0.0848–0.0896	80	21 21	0.011 0.013 0.012	< 0.01 < 0.01 < 0.01	0.012

Study Trial No. Plot No. Year			Application						Residues [mg/kg] [a]		
	Variety	Country	FL	No	kg/ha (as)	kg/hL (as)	GS	DAT	TFS	CGA 321113	Sum
RATFY011 TF005-05D USA-TF005-05D-B, 2005	Soya bean Pioneer 9492 RR	USA Leland	500 SC	3	0.130–0.132	0.0872–0.0898	83	17 17 21 21 23 23 27 27 31 31	< 0.01 < 0.01 0.014 < 0.01 0.014 < 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	0.012
RATFY011 TF006-05D USA-TF006-05D-B, 2005	RG 200 RR	USA Sabin	500 SC	3	0.128–0.133	0.0757–0.0847	70	16 16 21 21 24 24 27 27 31 31	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.014 0.014 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 0.011 < 0.01 0.010 0.01 < 0.01 0.010 < 0.01 < 0.01	0.021
RATFY011 TF007-05H USA-TF007-05H-B, 2005	Taylor 427 RR	USA Stilwell	500 SC	3	0.128–0.133	0.0908–0.0937	83	20 20	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RATFY011 TF008-05H USA-TF008-05H-B, 2005	Nk 32G5	USA Spring-field	500 SC	3	0.127–0.130	0.104–0.106	79	19 19	0.013 0.015 0.014	0.010 < 0.01 0.01	0.0244
RATFY011 TF009-05H USA-TF009-05H-B, 2005	HS3236	USA Monti-cello	500 SC	3	0.127–0.133	0.0901–0.0943	79	20 20	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RATFY011 TF010-05H USA-TF010-05H-B, 2005	Taylor 427 RR	USA Stilwell	500 SC	3	0.126–0.130	0.0906–0.0935	87	20 20	0.012 < 0.01 0.012	< 0.01 < 0.01 < 0.01	0.012
RATFY011 TF011-05H USA-TF011-05H-B, 2005	Asgrow 2801	USA Earlham	500 SC	3	0.128–0.129	0.104–0.106	79	19 19	< 0.01 0.018 0.014	0.010 < 0.01 0.01	0.0254
RATFY011 TF012-05H USA-TF012-05H-B, 2005	92M70	USA Bagley	500 SC	3	0.128–0.129	0.102–0.105	79	21 21	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RATFY011 TF013-05H USA-TF013-05H-B. 2005	Mycogen 0941731	USA Gardner	500 SC	3	0.130–0.132	0.0788–0.0910	81	21 21	< 0.01 < 0.01 < 0.01	0.011 < 0.01 0.011	0.021
RATFY011 TF014-05H USA-TF014-05H-B, 2005	SC 9374	USA New Holland	500 SC	3	0.128–0.133	0.0883–0.0899	95	19 19	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RATFY011 TF015-05H USA-TF015-05H-B, 2005	Pioneer 92M80	USA York	500 SC	3	0.127–0.129	0.0686–0.0690	77	21 21	0.015 0.017 0.16	0.038 0.043 0.042	0.058
RATFY011 TF016-05H USA-TF016-05H-B, 2005	NK 43-B1	USA Carlyle	500 SC	3	0.127–0.129	0.0696–0.0759	79	20 20	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01

Study Trial No. Plot No. Year			Application						Residues [mg/kg] [a]		
	Variety	Country	FL	No	kg/ha (as)	kg/hL (as)	GS	DAT	TFS	CGA 321113	Sum
RATFY011 TF017-05H USA-TF017-05H-B, 2005	Asgrow AG1603	USA Arkansaw	500 SC	3	0.129–0.130	0.0733–0.0734	79	21 21	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RATFY011 TF018-05H USA-TF018-05H-B, 2005	Dairyland 3410	USA Sheridan	500 SC	3	0.126–0.127	0.0608–0.0692	97	21 21	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RATFY011 TF019-05H USA-TF019-05H-B, 2005	Soya bean Asgrow 3802	USA Kiowa	500 SC	3	0.129–0.131	0.0872–0.108	95	23 23	0.015 < 0.01 0.012	< 0.01 < 0.01 < 0.01	< 0.012
RATFY011 TF020-05H USA-TF020-05H-B, 2005	Soya bean Pioneer 93B85	USA St. John	500 SC	3	0.126–0.132	0.0751–0.0781	79	19 19	0.030 0.011 0.02	0.018 0.017 0.018	0.039

FL=Formulation

No=number of applications

GS=growth stage at last application

DAT=days after last treatment

TFS=Trifloxystrobin

[a] Residues were measured in dry seeds

Beans and Peas (dry)Green beans

Nine field trials were conducted in Canada in 2012 with trifloxystrobin in/on dry beans (Milo, J and Harbin, A 2013a.) Two applications at 0.129 to 0.137 kg ai/ha were done with a spray interval of 10 to 14 days and a pre-harvest interval of 28 to 32 days.

Nine field trials were conducted in Canada in 2012 with trifloxystrobin in/on peas (Milo, J and Harbin, A 2013b.). Two applications at 0.108 to 0.135 kg ai/ha were done with a spray interval of 11 to 14 days and a pre-harvest interval of 29 to 31 days.

The residues of trifloxystrobin and CGA 321113 were quantified according to methods 00742, 00742/M001 and 01313 at a LOQ of 0.01 mg/kg.

The results are summarized in Tables 7 and 8.

Table 7 Results of residue trials conducted with 325 SC trifloxystrobin in/on kidney beans in Canada

Study Trial No. Plot No. Year			Application					Residues [mg/kg] [a]		
	Crop Variety	Country	No	kg/ha (as)	kg/hL (as)	GS	DAT	TFS	CGA 321113	SUM
Canadian GAP SC325> 0.132 kg ai/ha 1–2 times, PHI 30 days										
RAJAN003 RAJAN003-01-12H, 2012	Zorro Black Bean	Canada Arthur	2	0.13430.1375	0.0959–0.0982	77	29 29	< 0.01 < 0.01 < 0.01	0.011 0.011 0.011	< 0.021
RAJAN003 RAJAN003-02-12H, 2012	Red Hawk (red Kidney)	Canada Rock-wood	2	0.13570.1362	0.0969–0.0973	73	32 32	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RAJAN003 RAJAN003-03-12H, 2012	Zorro Black Bean	Canada Breslau	2	0.1292	0.0923	75	29 29	< 0.01 < 0.01 < 0.01	0.010 0.012 0.011	0.021

Study Trial No. Plot No. Year			Application					Residues [mg/kg] [a]		
	Crop Variety	Country	No	kg/ha (as)	kg/hL (as)	GS	DAT	TFS	CGA 321113	SUM
RAJAN003 RAJAN003-04-12H, 2012	Pinto	Canada Whitecap	2	0.13060.1319	0.0933– 0.0942	66	28 28	< 0.01 < 0.01 < 0.01	0.012 0.013 0.012	0.023
RAJAN003 RAJAN003-05-12H, 2012	Pintos	Canada Outlook	2	0.13060.1315	0.0933– 0.0939	65	28 28	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RAJAN003 RAJAN003-06-12H, 2012	Pinto	Canada Kenaston	2	0.13040.1343	0.0931– 0.0959	66	28 28	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RAJAN003 RAJAN003-07-12H, 2012	Viva Pink	Canada Taber	2	0.12940.135	0.0924– 0.0964	75	32 32	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RAJAN003 RAJAN003-07-12H, 2012	Viva Pink	Canada Taber	2	0.12940.135	0.0924– 0.0964	75	32 32	< 0.01 < 0.01	< 0.01 < 0.01	
RAJAN003 RAJAN003-08-12D, 2012	Pinto	Canada Rosthern	2	0.12940.1314	0.0924– 0.0939	71	21 21 25 25 29 29 36 36 40 40	0.011 0.018 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.015 0.017 0.014 0.013 0.012 0.011 0.012 0.012 < 0.01 0.011 0.011	0.022
RAJAN003 RAJAN003-09-12H, 2012	Bean, Kidney Pintos	Canada Alvena	2	0.1315- 0.1327	0.0939– 0.0948	71	31 31	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01

FL=Formulation

No=number of applications

GS=growth stage at last application

DAT=days after last treatment

TFS=trifloxystrobin

[a] Residues were measured in dry seeds,

Table 8 Results of residue trials conducted with 325 SC trifloxystrobin in/on pea in 2012

Study Trial No. Plot No.			Application					Residues [mg/kg] [a]		
	Crop Variety	Country	No	kg/ha (as)	kg/hL (as)	GS	DAT	TFS	CGA 321113	Sum
Canadian GAP: SC325, 0.132 kg ai/ha 1–2 times with PHI of 30 (seed)										
RAJAN004 RAJAN004-01-12H	Pea, field Meadow	Canada Whitecap	2	0.1301– 0.1305	0.0929– 0.0932	72	31 31	< 0.01 < 0.01 < 0.01	0.021 0.023 0.022	0.033
RAJAN004 RAJAN004-02-12H	Pea, field Meadow	Canada Outlook	2	0.1312– 0.1314	0.0937– 0.0939	71	31 31	< 0.01 < 0.01 < 0.01	0.011 0.014 0.012	0.023
RAJAN004 RAJAN004-03-12H	Pea, field Admiral	USA Carring-ton	2	0.1308– 0.1357	0.0934– 0.0969	73	30 30	< 0.01 < 0.01	0.016 0.016	0.027
RAJAN004 RAJAN004-04-12H	Pea, field Meadow	Canada Kenaston	2	0.1303– 0.1311	0.0931– 0.0936	73	29 29	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01
RAJAN004 RAJAN004-05-12H	Pea, field Meadow	Canada Waldheim	2	0.1326– 0.133	0.0947– 0.0950	71	29 29	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01

Study Trial No. Plot No.			Application					Residues [mg/kg] [a]		
	Crop Variety	Country	No	kg/ha (as)	kg/hL (as)	GS	DAT	TFS	CGA 321113	Sum
RAJAN004 RAJAN004-06-12H	Pea, field Meadow	Canada Alvena	2	0.1329	0.0949	72	29 29	< 0.01 < 0.01	0.011 0.011	0.021
RAJAN004 RAJAN004-07-12H	Pea, field Meadow	Canada Wakaw	2	0.1309–0.1339	0.0935–0.0956	71	29 29	< 0.01 < 0.01	0.011 0.013	0.022
RAJAN004 RAJAN004-08-12H	Pea, field Thunder-bird	Canada Joseph-burg	2	0.1266–0.1341	0.0904–0.0958	75	30 30	< 0.01 < 0.01	0.010 0.011	0.021
RAJAN004 RAJAN004-09-12D	Pea, field Meadow	Canada Rosthern	2	0.108–0.1082	0.0771–0.0773	69	20 20 25 25 31 31 34 34 40 40	0.012 0.012 0.016 0.019 < 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

FL=Formulation; No=number of applications; GS=growth stage at last application; DAT=days after last treatment; TFS: trifloxystrobin; [1]. Residues were measured in dry seeds,

Animal feeds

The conditions of supervised trials are described under the respective commodities. Only the residues in relevant animal commodities are summarized.

Table 9 Residues of trifloxystrobin in/on soya bean forage derived from trials conducted in the USA

Study Trial No. Plot No. Year			Application						Residues [mg/kg] [a]	
	Variety	Country	FL	No	kg/ha (as)	kg/hL (as)	GS	DAT	TFS	CGA 321113
USA GAP 250EC, 0.09125 kg/ha max 3 times with PHI of 21 days Do not graze or feed soya bean forage or hay.										
RCTFY004 FL079-03H USA-FL079-03H-A, 2003	Hartz Seed H6686RR	USA Tifton, Georgia	250 EC	3	0.092	0.052–0.064	67	0	1.53 6.07	0.106 0.395
RCTFY004 FL080-03H USA-FL080-03H-A, 2003	NK S73-Z5	USA Molino, Florida	250 EC	3	0.086–0.094	0.043–0.046	74	0	0.81 1.21	0.075 0.096
RCTFY004 FL081-03H USA-FL081-03H-A, 2003	Horn-beck 5588RR	USA Proctor, Arkansas	250 EC	3	0.092	0.063–0.066	71	0	2.92 4.65	0.137 0.219
RCTFY004 FL082-03H USA-FL082-03H-A, 2003	Delta King 5661 RR	USA Newport, Arkansas	250 EC	3	0.092–0.094	0.049–0.049	75	0	3.12 3.48	0.168 0.176
RCTFY004 FL083-03D USA-FL083-03D-A,2003	S56-D7	USA Leland, Mississi-ppi	250 EC	3	0.091–0.094	0.074–0.076	70	0 0 3 3 5 5 7 7 10 10	3.00 2.90 2.11 0.828 0.590 0.978 1.27 0.685 0.630 0.388	0.226 0.295 0.215 0.158 0.138 0.230 0.154 0.122 0.091 0.081

Study Trial No. Plot No. Year			Application						Residues [mg/kg] [a]	
	Variety	Country	FL	No	kg/ha (as)	kg/hL (as)	GS	DAT	TFS	CGA 321113
RCTFY004 FL084-03D USA-FL084-03D-A, 2003	FS HT322 STS	USA Seymour, Illinois	250 EC	3	0.093	0.061–0.062	72	0 0 3 3 6 6 8 8 10 10	2.80 2.72 1.30 1.25 0.822 0.895 0.752 0.902 0.705 0.728	0.121 0.135 0.186 0.195 0.160 0.166 0.151 0.160 0.131 0.133
RCTFY004 FL085-03H USA-FL085-03H-A, 2003	NK S26 C9	USA Spring-field, Nebraska	250 EC	3	0.092–0.183	0.061–0.129	65	0	2.30 1.58	0.095 0.083
RCTFY004 FL086-03H USA-FL086-03H-A, 2003	Patriot Round-up Ready	USA Stilwell, Kansas	250 EC	3	0.093–0.187	0.063–0.132	79	0	4.45 6.90	0.178 0.225
RCTFY004 FL087-03H USA-FL087-03H-A, 2003	Becks 323RR	USA Oxford, Indiana	250 EC	3	0.094–0.095	0.051–0.059	69	0	3.45 3.55	0.208 0.196
RCTFY004 FL088-03H USA-FL088-03H-A, 2003	92B94	USA Bagley, Iowa	250 EC	3	0.089–0.092	0.036–0.042	67	0	3.50 5.00	0.164 0.240
RCTFY004 FL089-03H USA-FL089-03H-A, 2003	BT-402	USA Carlyle, Illinois	250 EC	3	0.089–0.093	0.060–0.065	69	0	3.18 3.75	0.213 0.222
RCTFY004 FL090-03H USA-FL090-03H-A, 2003	GL2301RR	USA Saginaw, Michigan	250 EC	3	0.091–0.092	0.047–0.048	69	0	1.54 9.87	0.121 0.948
RCTFY004 FL091-03H USA-FL091-03H-A, 2003	Mycogen 44150	USA Gardner, North Dakota	250 EC	3	0.092–0.093	0.030–0.037	81	0	4.85 /0.027 [b] 6.98	0.485 0.508
RCTFY004 FL092-03H USA-FL092-03H-A, 2003	SC 9373	USA New Holland, Ohio	250 EC	3	0.091–0.093	0.062–0.066	69	0	2.48 2.32	0.154 0.140
RCTFY004 FL093-03H USA-FL093-03H-A, 2003	Dekalb 06-51	USA Campbell, Minnesota	250 EC	3	0.091–0.092	0.032–0.033	70	0	2.75 3.15	0.224 0.258
RCTFY004 FL094-03H USA-FL094-03H-A, 2003	Pioneer91m50	USA Geneva, Minnesota	250 EC	3	0.091–0.092	0.058–0.061	69	0	5.28 4.92	0.365 0.338
RCTFY004 FL095-03H USA-FL095-03H-A, 2003	Dekalb 3151	USA Sheridan, Indiana	250 EC	3	0.091–0.092	0.057–0.058	70	0	3.28 2.46	0.244 0.199
RCTFY004 FL096-03H USA-FL096-03H-A, 2003	Rough Rider	USA Northwood, North Dakota	250 EC	3	0.089–0.094	0.032–0.033	69	0	2.70 2.95	0.255 0.270
RCTFY004 FL097-03H USA-FL097-03H-A, 2003	Pioneer 93B86	USA Richland, Iowa	250 EC	3	0.091–0.094	0.050–0.067	67	0	3.18 3.30	0.115 0.117

Study Trial No. Plot No. Year			Application						Residues [mg/kg] [a]	
	Variety	Country	FL	No	kg/ha (as)	kg/hL (as)	GS	DAT	TFS	CGA 321113
RCTFY004 FL098-03H USA-FL098-03H-A, 2003	Brunner BR-1500-RR	USA Arkansaw, Wisconsin	250 EC	3	0.093–0.096	0.033–0.033	69	0	3.95 3.38	0.148 0.115
US GAP 500 SC: 0.1095–0.1271 max 3 times, PHI 21 days Do not graze or feed soya bean forage or hay.										
RATFY011 TF001-05H USA-TF001-05H-A, 2005	S73-Z5	USA Tifton	500 SC	3	0.128	0.0762–0.102	67	0	6.065 6.843	0.228 0.210
RATFY011 TF002-05H USA-TF002-05H-A, 2005	Pioneer 95B96	USA Molino	500 SC	3	0.123–0.132	0.0971–0.110	70	0	6.771 6.365	0.186 0.199
RATFY011 TF003-05H USA-TF003-05H-A, 2005	AG4403 RR	USA Proctor	500 SC	3	0.128–0.129	0.0902–0.0928	69	0	21.80 23.86	0.262 0.257
RATFY011 TF004-05H USA-TF004-05H-A, 2005	DPL 5806 RR	USA Cheney-ville	500 SC	3	0.122–0.127	0.0738–0.0871	69	0	10.22 9.059	0.171 0.150
RATFY011 TF005-05D USA-TF005-05D-A, 2005	Pioneer 9492 RR	USA Leland	500 SC	3	0.130–0.132	0.107–0.112	66	0 0 3 3 5 5 7 7 11 11	9.389 10.47 7.858 8.267 5.482 5.094 3.728 3.512 2.783 2.658	0.228 0.257 0.382 0.379 0.335 0.305 0.299 0.297 0.261 0.213
RATFY011 TF006-05D USA-TF006-05D-A, 2005	RG 200 RR	USA Sabin	500 SC	3	0.125–0.130	0.0771–0.0839		0 0 3 3 5 5 7 7 10 10	11.53 11.14 2.455 2.393 1.287 1.554 1.288 1.144 1.567 0.676	0.285 0.267 0.296 0.319 0.214 0.246 0.175 0.169 0.263 0.125
RATFY011 TF007-05H USA-TF007-05H-A, 2005	Taylor 427 RR	USA Stilwell	500 SC	3	0.128–0.132	0.0898–0.0934	75	0	13.75 17.36	0.243 0.316
RATFY011 TF008-05H USA-TF008-05H-A, 2005	Nk 32G5	USA Spring-field	500 SC	3	0.129	0.102–0.106	67	0	11.07 9.589	0.274 0.259
RATFY011 TF009-05H USA-TF009-05H-A, 2005	HS3236	USA Monti-cello	500 SC	3	0.124–0.132	0.0917–0.0921	70	0	14.49 14.18	0.254 0.230
RATFY011 TF010-05H USA-TF010-05H-A, 2005	Taylor 427 RR	USA Stilwell	500 SC	3	0.128–0.134	0.0895–0.0937	77	0	5.984 6.586	0.138 0.147

Study Trial No. Plot No. Year			Application						Residues [mg/kg] [a]	
	Variety	Country	FL	No	kg/ha (as)	kg/hL (as)	GS	DAT	TFS	CGA 321113
RATFY011 TF011-05H USA-TF011-05H-A, 2005	Asgrow 2801	USA Earlham	500 SC	3	0.128–0.130	0.101–0.106	67	0	8.649 8.747	0.343 0.318
RATFY011 TF012-05H USA-TF012-05H-A, 2005	92M70	USA Bagley	500 SC	3	0.124–0.128	0.0992–0.102	66	0	9.009 5.698	0.249 0.182
RATFY011 TF013-05H USA-TF013-05H-A, 2005	Myco-gen 0941731	USA Gardner	500 SC	3	0.131–0.133	0.0887–0.0963	71	0	16.11 17.30	0.307 0.297
RATFY011 TF014–05H USA-TF014-05H-A, 2005	SC 9374	USA New Holland	500 SC	3	0.126–0.131	0.0879–0.0894	70	0	11.41 10.09	0.203 0.199
RATFY011 TF015-05H USA-TF015-05H-A, 2005	Pioneer 92M80	USA York	500 SC	3	0.127–0.129	0.0686–0.0690	67	0	12.73 8.950	0.274 0.257
RATFY011 TF016-05H USA-TF016-05H-A, 2005	NK 43-B1	USA Carlyle	500 SC	3	0.127–0.128	0.0743–0.0934	66	0	13.58 12.19	0.358 0.353
RATFY011 TF017-05H USA-TF017-05H-A, 2005	Asgrow AG1603	USA Arkansaw	500 SC	3	0.129	0.0729–0.0733	69	0	12.46 13.44	0.276 0.277
RATFY011 TF018-05H USA-TF018-05H-A, 2005	Dairy-land 3410	USA Sheridan	500 SC	3	0.124–0.130	0.0667–0.0723	69	0	6.096 5.792	0.129 0.120
RATFY011 TF019-05H USA-TF019-05H-A, 2005	Asgrow 3802	USA Kiowa	500 SC	3	0.127–0.129	0.101–0.106	69	0	15.26 16.67	0.343 0.332
RATFY011 TF020-05H USA-TF020-05H-A, 2005	Pioneer 93B85	USA St. John	500 SC	3	0.126–0.129	0.0736–0.0759	73	0	10.43 10.66	0.369 0.418

FL=Formulation

No=number of applications

GS=growth stage at last application

DAT=days after last treatment

[a] Residues were measured in forage samples

[b] residues in control

TFS=trifloxystrobin;

Table 10 Residues of trifloxystrobin in/on soya bean hay derived from trials conducted in the USA

Study Trial No. Plot No. Year			Application					Residues [a] [mg/kg]	
	Variety	Country	FL	No	kg/ha (as)	kg/hL (as)	GS	TFS	CGA 321113
USA GAP 250EC, 0.09125 kg/ha max 3 times with PHI of 21 days Do not graze or feed soya bean forage or hay.									
RCTFY004 FL079-03H USA-FL079-03H-A,2003	Hartz Seed H6686RR	USA Tifton, Georgia	250 EC	3	0.092	0.052–0.064	67	9.62 8.50	0.908 0.840
RCTFY004 FL080-03H USA-FL080-03H-A, 2003	NK S73-Z5	USA Molino, Florida	250 EC	3	0.086–0.094	0.043–0.046	74	4.00 3.58	0.535 0.518
RCTFY004 FL081-03H USA-FL081-03H-A, 2003	Horn-beck 5588RR	USA Proctor, Arkansas	250 EC	3	0.092	0.063–0.066	71	5.50 5.55	0.602 0.562
RCTFY004 FL082-03H USA-FL082-03H-A, 2003	Delta King 5661 RR	USA Newport, Arkansas	250 EC	3	0.092–0.094	0.049–0.049	75	9.18 2.22	0.540 0.129
RCTFY004 FL083-03D USA-FL083-03D-A, 2003	S56-D7	USA Leland, Mississippi	250 EC	3	0.091–0.094	0.074–0.076	70	6.30 6.55	0.788 0.730
RCTFY004 FL084-03D USA-FL084-03D-A, 2003	FS HT322 STS	USA Seymour, Illinois	250 EC	3	0.093	0.061–0.062	72	10.4 9.82	0.90 0.0.88
RCTFY004 FL085-03H USA-FL085-03H-A, 2003	NK S26 C9	USA Springfield, Nebraska	250 EC	3	0.092–0.183	0.061–0.129	65	6.25 [c] /5.55 [b] 3.80	0.858 /1.04 [b] 0.570
RCTFY004 FL086-03H USA-FL086-03H-A, 2003	Patriot Round-up Ready	USA Stilwell, Kansas	250 EC	3	0.093–0.187	0.063–0.132	79	9.98 10.4	0.902 0.930
RCTFY004 FL087-03H USA-FL087-03H-A, 2003	Becks 323RR	USA Oxford, Indiana	250 EC	3	0.094–0.095	0.051–0.059	69	10.4 12.3	1.20 1.36
RCTFY004 FL088-03H USA-FL088-03H-A, 2003	92B94	USA Bagley, Iowa	250 EC	3	0.089–0.092	0.036–0.042	67	8.38 10.6	1.11 1.25
RCTFY004 FL089-03H USA-FL089-03H-A, 2003	BT-402	USA Carlyle, Illinois	250 EC	3	0.089–0.093	0.060–0.065	69	7.92 10.2	4.12 4.45
RCTFY004 FL090-03H USA-FL090-03H-A, 2003	GL2301RR	USA Saginaw, Michigan	250 EC	3	0.091–0.092	0.047–0.048	69	14.6 12.1	2.00 1.35
RCTFY004 FL091-03H USA-FL091-03H-A, 2003	Myco-gen 44150	USA Gardner, North Dakota	250 EC	3	0.092–0.093	0.030–0.037	81	15.4 13.2	2.52 2.58
RCTFY004 FL092-03H USA-FL092-03H-A, 2003	SC 9373	USA New Holland, Ohio	250 EC	3	0.091–0.093	0.062–0.066	69	4.92 7.05	0.732 1.07

Study Trial No. Plot No. Year			Application					Residues [a] [mg/kg]	
	Variety	Country	FL	No	kg/ha (as)	kg/hL (as)	GS	TFS	CGA 321113
RCTFY004 FL093-03H USA-FL093-03H-A, 2003	Dekalb 06-51	USA Campbell, Minnesota	250 EC	3	0.091–0.092	0.032–0.033	70	8.20 7.40	1.04 1.02
RCTFY004 FL094-03H USA-FL094-03H-A, 2003	Pioneer91m50	USA Geneva, Minnesota	250 EC	3	0.091–0.092	0.058–0.061	69	4.28 5.00	0.690 0.812
RCTFY004 FL095-03H USA-FL095-03H-A, 2003	Dekalb 3151	USA Sheridan, Indiana	250 EC	3	0.091–0.092	0.057–0.058	70	1.66 5.19	0.278 0.638
RCTFY004 FL096-03H USA-FL096-03H-A, 2003	Rough Rider	USA Northwood, North Dakota	250 EC	3	0.089–0.094	0.032–0.033	69	10.1 7.00	1.96 1.46
RCTFY004 FL097-03H USA-FL097-03H-A, 2003	Pioneer 93B86	USA Richland, Iowa	250 EC	3	0.091–0.094	0.050–0.067	67	4.02 5.30	0.362 0.475
RCTFY004 FL098-03H USA-FL098-03H-A, 2003	Brunner BR-1500-RR	USA Arkansaw, Wisconsin	250 EC	3	0.093–0.096	0.033–0.033	69	11.8 9.88	0.638 0.515
US GAP 500 SC: 0.1095–0.1271 max 3 times, PHI 21 days Do not graze or feed soya bean forage or hay.									
RATFY011 TF001-05H USA-TF001-05H-A, 2005	S73-Z5	USA Tifton	500 SC	3	0.128	0.0762–0.102	67	8.374 10.37	0.884 1.191
RATFY011 TF002-05H USA-TF002-05H-A, 2005	Pioneer 95B96	USA Molino	500 SC	3	0.123–0.132	0.0971–0.110	70	19.44 25.34	0.906 1.149
RATFY011 TF003-05H USA-TF003-05H-A, 2005	AG4403 RR	USA Proctor	500 SC	3	0.128–0.129	0.0902–0.0928	69	60.81 70.90	1.089 1.404
RATFY011 TF004-05H USA-TF004-05H-A, 2005	DPL 5806 RR	USA Cheneyville	500 SC	3	0.122–0.127	0.0738–0.0871	69	38.99 30.51	0.793 0.883
RATFY011 TF005-05D USA-TF005-05D-A, 2005	Pioneer 9492 RR	USA Leland	500 SC	3	0.130–0.132	0.107–0.112	66	30.78 /[b] 0.0127 28.24	1.218 1.846 0.827
RATFY011 TF006-05D USA-TF006-05D-A, 2005	RG 200 RR	USA Sabin	500 SC	3	0.125–0.130	0.0771–0.0839	67	31.47 30.13	2.026 1.675
RATFY011 TF007-05H USA-TF007-05H-A, 2005	Taylor 427 RR	USA Stilwell	500 SC	3	0.128–0.132	0.0898–0.0934	75	41.21 /0.0960 [b] 44.00	0.956 /0.0349 [b] 0.732
RATFY011 TF008-05H USA-TF008-05H-A, 2005	Nk 32G5	USA Springfield	500 SC	3	0.129	0.102–0.106	67	39.46 /0.0295 [b] 40.51	1.293 1.559

Study Trial No. Plot No. Year			Application					Residues [a] [mg/kg]	
	Variety	Country	FL	No	kg/ha (as)	kg/hL (as)	GS	TFS	CGA 321113
RATFY011 TF009-05H USA-TF009-05H-A, 2005	HS3236	USA Monti-cello	500 SC	3	0.124–0.132	0.0917–0.0921	70	47.32 /0.0200 [b] 46.71	1.264 1.543
RATFY011 TF010-05H USA-TF010-05H-A, 2005	Taylor 427 RR	USA Stilwell	500 SC	3	0.128–0.134	0.0895–0.0937	77	21.51 /0.0205 [b] 11.16	0.639 0.455
RATFY011 TF011-05H USA-TF011-05H-A, 2005	Asgrow 2801	USA Earlham	500 SC	3	0.128–0.130	0.101–0.106	67	26.98 /0.0187 [b] 33.67	0.955 1.361
RATFY011 TF012-05H USA-TF012-05H-A, 2005	92M70	USA Bagley	500 SC	3	0.124–0.128	0.0992–0.102	66	21.61 /0.0158 [b] 21.46	1.470 1.373
RATFY011 TF013-05H USA-TF013-05H-A, 2005	Myco-gen 0941731	USA Gardner	500 SC	3	0.131–0.133	0.0887–0.0963	71	42.98 45.69	1.518 1.611
RATFY011 TF014-05H USA-TF014-05H-A, 2005	SC 9374	USA New Holland	500 SC	3	0.126–0.131	0.0879–0.0894	70	15.90 18.71	1.465 1.120
RATFY011 TF015-05H USA-TF015-05H-A, 2005	Pioneer 92M80	USA York	500 SC	3	0.127–0.129	0.0686–0.0690	67	22.60 27.57	0.821 1.175
RATFY011 TF016-05H USA-TF016-05H-A, 2005	NK 43-B1	USA Carlyle	500 SC	3	0.127–0.128	0.0743–0.0934	66	40.04 37.37	5.460 5.743
RATFY011 TF017-05H USA-TF017-05H-A, 2005	Asgrow AG1603	USA Arkansaw	500 SC	3	0.129	0.0729–0.0733	69	32.68 /0.0158 [b] 31.00	1.647 1.749
RATFY011 TF018-05H USA-TF018-05H-A, 2005	Dairy-land 3410	USA Sheridan	500 SC	3	0.124–0.130	0.0667–0.0723	69	8.100 8.446	0.337 0.346
RATFY011 TF019-05H USA-TF019-05H-A, 2005	Asgrow 3802	USA Kiowa	500 SC	3	0.127–0.129	0.101–0.106	69	30.74 36.92	1.202 1.372
RATFY011 TF020-05H USA-TF020-05H-A, 2005	Pioneer 93B85	USA St. John	500 SC	3	0.126–0.129	0.0736–0.0759	73	32.58 /0.0113 [b] 34.38	2.032 1.878

FL=Formulation

No=number of applications

GS=growth stage at last application

DAT=days after last treatment

TFS: trifloxystrobin

[a] Samples were taken 0–3 days after last application

[b] Residues in control

Table 11 Residues in green parts of pea derived from trials conducted with 325 SC trifloxystrobin in Canada

Study Trial No. Plot No. Year			Application					Residues [a] [mg/kg]		
	Crop Variety	Country	No	kg/ha (as)	kg/hL (as)	GS	DAT	TFS	CGA 321113	Sum
RAJAN004 RAJAN004-01- 12H, 2012	Pea, field Meadow	Canada Whitecap	2	0.1301– 0.1305	0.0929– 0.0932	72	6 6	0.81 1.0 0.90	0.039 0.038 0.038	0.945
RAJAN004 RAJAN004-02- 12H, 2012	Pea, field Meadow	Canada Outlook	2	0.13120.1314	0.0937– 0.0939	71	6 6	1.0 1.1 1.0	0.039 0.040 0.04	1.09
RAJAN004 RAJAN004-03- 12H, 2012	Pea, field Admiral	USA Carring- ton	2	0.13080.1357	0.0934– 0.0969	73	7 7	1.3 1.3 1.3	0.055 0.048 0.052	1.35
RAJAN004 RAJAN004-04- 12H, 2012	Pea, field Meadow	Canada Kenaston	2	0.13030.1311	0.0931– 0.0936	73	8 8	0.67 0.55 0.61	0.027 0.025 0.026	0.637
RAJAN004 RAJAN004-05- 12H, 2012	Pea, field Meadow	Canada Waldheim	2	0.1326–0.133	0.0947– 0.0950	71	6 6	0.79 0.77 0.78	0.033 0.031 0.032	0.813
RAJAN004 RAJAN004-06- 12H, 2012	Pea, field Meadow	Canada Alvena	2	0.1329	0.0949	72	6 6	1.6 1.4 1.5	0.039 0.038 0.385	1.54
RAJAN004 RAJAN004-07- 12H, 2012	Pea, field Meadow	Canada Wakaw	2	0.13090.1339	0.0935– 0.0956	71	6 6	0.73 1.1 0.915	0.035 0.038 0.037	0.953
RAJAN004 RAJAN004-08- 12H, 2012	Pea, field Thunder- bird	Canada Joseph- burg	2	0.12660.1341	0.0904– 0.0958	75	7 7	1.2 1.0 1.1	0.051 0.041 0.046	1.15
RAJAN004 RAJAN004-09- 12D, 2012	Pea, field Meadow	Canada Rosthern	2	0.108–0.1082	0.0771– 0.0773	69	0 0 3 3 7 7 13 13	2.4 3.1 1.7 1.6 2.3 1.6 1.95 0.67 0.73	0.013 0.011 0.030 0.032 0.035 0.023 0.029 0.032 0.030	1.98

FL=Formulation

No=number of applications

GS=growth stage at last application

DAT=Days after last treatment

TFS: trifloxystrobin

[a].Residues were measured in green materials.

Table 12 Residue in/on pea hay derived from trials conducted with 325 SC trifloxystrobin in/on pea in Canada

Study Trial No. Plot No. Year			Application					Residues [a] [mg/kg]		
	Crop Variety	Country	No	kg/ha (as)	kg/hL (as)	GS	DAT	TFS	CGA 321113	Sum
RAJAN004 RAJAN004-01- 12H, 2012	Pea, field Meadow	Canada Whitecap	2	0.1301– 0.1305	0.0929– 0.0932	72	6 6	2.2 6.2 4.2	0.18 0.43 0.305	4.51
RAJAN004 RAJAN004-02- 12H, 2012	Pea, field Meadow	Canada Outlook	2	0.1312– 0.1314	0.0937– 0.0939	71	6 6	5.4 6.6 6.0	0.29 0.35 0.32	6.33

Study Trial No. Plot No. Year			Application					Residues [a] [mg/kg]		
	Crop Variety	Country	No	kg/ha (as)	kg/hL (as)	GS	DAT	TFS	CGA 321113	Sum
RAJAN004 RAJAN004-03- 12H, 2012	Pea, field Admiral	USA Carrington	2	0.1308– 0.1357	0.0934– 0.0969	73	7 7	2.1 2.1 2.1	0.13 0.041 0.086	2.19
RAJAN004 RAJAN004-04- 12H, 2012	Pea, field Meadow	Canada Kenaston	2	0.1303– 0.1311	0.0931– 0.0936	73	8 8	3.1 3.1 3.1	0.15 0.18 0.165	3.27
RAJAN004 RAJAN004-05- 12H, 2012	Pea, field Meadow	Canada Waldheim	2	0.1326– 0.133	0.0947– 0.0950	71	6 6	3.5 3.1 3.3	0.30 0.25 0.275	3.58
RAJAN004 RAJAN004-06- 12H, 2012	Pea, field Meadow	Canada Alvena	2	0.1329	0.0949	72	6 6	6.6 6.8 6.7	0.31 0.49 0.40	7.11
RAJAN004 RAJAN004-07- 12H, 2012	Pea, field Meadow	Canada Wakaw	2	0.1309– 0.1339	0.0935– 0.0956	71	6 6	6.0 4.6 5.3	0.24 0.26 0.25	5.56
RAJAN004 RAJAN004-08- 12H, 2012	Pea, field Thunder- bird	Canada Joseph- burg	2	0.1266– 0.1341	0.0904– 0.0958	75	7 7	8.2 5.3 6.75	0.23 0.15 1.19	6.95
RAJAN004 RAJAN004-09- 12D,2012	Pea, field Meadow	Canada Rosthern	2	0.108– 0.1082	0.0771– 0.0773	69	0 0 3 3 7 7 13 13	15 13 7.2 6.6 3.1 2.9 3.0 3.0 1.6	0.37 0.35 0.21 0.22 0.17 0.16 0.165 0.25 0.33	3.17

FL=Formulation

No=number of applications

GS=growth stage at last application

DAT=days after last treatment

TFS: trifloxystrobin

[a] Residues were measured in hay,

Fate of residues in storage and processing

The effect of processing on trifloxystrobin residues was investigated in soya beans in the USA. In one trial, three foliar spray applications at rates of 0.446–0.471 kg trifloxystrobin/ha were made to soya beans with a 8 to 9-day interval between applications. Soya beans were harvested at normal maturity at a 19-day after last application (Beedle, EC and Harbin, AM 2005b.). Subsamples of the soya bean seed were removed for analysis. The remainder of the soya bean seed was used to generate aspirated grain fractions and then processed into hulls, meal, and refined oil. Processing was performed using batch procedures that simulated commercial processing practices. The residues of trifloxystrobin and CGA 321113 were determined according to method 200177. The individual analyte residues were summed to give a total trifloxystrobin residue. The limit of quantitation (LOQ) for total trifloxystrobin residue was 0.01 mg/kg in soya bean seed, hulls, meal, and refined oil, and 0.10 mg/kg in soya bean aspirated grain fractions.

Table 13 Results of processing soya beans treated with trifloxystrobin

Crop Variety	Application			Portion analysed	Residues [mg/kg]			
	FL	No	kg/ha (as)		TFS	CGA 321113	Total	Pf
S56-D7	250 EC	3	0.223- 0.235	seed	0.223	0.038	0.261	–
				hull	0.116	< 0.01	0.124	0.48
				meal	< 0.01	< 0.01	< 0.01	< 0.04
				oil, refined	0.034	< 0.01	0.034	0.13

Crop Variety	Application			Portion analysed	Residues [mg/kg]			
	FL	No	kg/ha (as)		TFS	CGA 321113	Total	Pf
				aspirated grain fractions	16.1	2.08	18.2	69.7

Residues in animal commodities

Dairy and poultry feeding studies were submitted for the 2004 JMPR review.

APPRAISAL

Trifloxystrobin was first evaluated by the JMPR in 2004 (T, R) and in 2012 (R). The 2004 Meeting established an ADI of 0–0.04 mg/kg bw and decided that ARfD was not necessary. The Meeting agreed that the residue definition for enforcement purposes for plant commodities should be trifloxystrobin per se, for animal commodities and dietary intake assessment the residue definition should be parent compound and CGA 321113 (expressed as trifloxystrobin equivalents) for plant and animal commodities.

Trifloxystrobin was listed by the Forty-sixth Session of CCPR (2014) for the evaluation by the 2015 JMPR for additional MRLs. Supervised trials data were submitted for evaluation on dry soya bean, lentil, chick pea and pea.

Analytical methods used for supervised trials were also provided.

Analytical methods

The Meeting received descriptions and validation data for analytical methods for residues of trifloxystrobin, CGA 321113 and several other metabolites in different plant matrices.

The plant materials are generally extracted with a mixture of acetonitrile/water. After filtration and concentration to the aqueous remainder, the acidified crude extract is purified, where necessary, by liquid-liquid partition. The residues are quantified by reverse-phase HPLC with MS/MS-detection. The average recoveries of trifloxystrobin and CGA 321113 and their relative standard deviations from test portions spiked at 0.01–2 mg/kg levels were for peas (100–101%, 3.1–4.7%) and soya beans (86–91%, 6.4, 19%). The limits of quantification ranged between 0.01–0.02 mg/kg.

The DFG method S19, evaluated in 2004, is suitable for enforcement.

Residues resulting from supervised trials on crops

The sum of trifloxystrobin and CGA 321113 was calculated and expressed as trifloxystrobin on the basis of the relative molecular masses. A conversion factor of 1.036 is required to express CGA 321113 as trifloxystrobin. As CGA 321113 does not generally constitute a significant proportion of the residue in crops, when the levels of trifloxystrobin or CGA 321113 were below the LOQ, their sum was calculated according to the method used by the 2004 JMPR.

Trifloxystrobin (mg/kg)	CGA 321113 (mg/kg)	Total (expressed as trifloxystrobin) (mg/kg)
< 0.01	< 0.01	< 0.01
< 0.01	0.011	0.021
0.10	< 0.02	0.10
0.92	0.16	1.1

In field trials duplicate samples were taken from each treated plot. Of the duplicate results the non-detected residues were disregarded in the calculation of average residue. As a conservative

approach, if the residues measured were 0.015 and < 0.01, the calculated average was taken as 0.015 mg/kg.

Pulses

Soya bean

The GAP in Canada allows maximum 2 times 0.0625 kg/ha treatment with a 20 day PHI. In 4 trials conducted according to GAP the residues in soya bean seeds were < 0.01 mg/kg (4).

The Brazilian GAP permits up to 4 treatments with 0.060 kg/ai/ha or 2 treatments with 0.075 kg ai/ha with a PHI of 20 days. Following treatment according to GAP the trifloxystrobin residues were below the LOQ (< 0.01 or < 0.02 mg/kg). CGA 321113 residues occurred in seven samples at 0.01–0.02 mg/kg level.

The US GAP permits 3 applications at rates between 0.0913–0.127 kg ai/ha and a PHI of 21 days. In 2003 a total of 20 trials were conducted in the USA applying trifloxystrobin three times at rates of 0.086–0.095 kg ai/ha. In addition, another 20 trials were performed in 2005 with application rates of 0.13 kg ai/ha and samples were taken at 21 days. Duplicate samples were taken from each site.

The US use patterns represent the critical GAP. The nominal application rates in US trials are within ± 25% of the GAP. The residues of parent compound in rank order were: < 0.01 (28), 0.01 (4), 0.012, 0.014, 0.016 (2), 0.021, 0.027, and 0.041mg/kg.

The sum of residues were in rank order: < 0.01 (24), 0.012 (4), 0.021 (2), 0.023 (2), 0.024, 0.025, .026, 0.027, 0.039, 0.043, 0.057 and 0.058 mg/kg.

The Meeting estimated a maximum residue level of 0.05 mg/kg for trifloxystrobin in soya beans, and an STMR residue of 0.01 mg/kg for the sum of trifloxystrobin and CGA 321113.

Beans and peas, dry

The use of trifloxystrobin in/on dry pea, chickpea and lentil is registered in Canada and the USA.

Nine trials were conducted on dry peas and nine trials on dry beans according the GAP in Canada (1-2 application with 0.132 kg ai/ha, the PHI is 30 days). Duplicate samples were taken at each sampling interval.

In beans, the average residues of trifloxystrobin at about 30 days were < 0.01 mg/kg in all (9) samples.

The sum of trifloxystrobin and CGA 321113 residues expressed as trifloxystrobin were in rank order: < 0.01 (5), 0.021 (2), 0.022, and 0.023 mg/kg.

In peas, the residues of trifloxystrobin at about 30 days were all < 0.01 mg/kg in all (9) samples.

The sum of residues of trifloxystrobin and CGA 321113 expressed as trifloxystrobin (mg/kg) at about 30 days were: < 0.01 (3), 0.021 (2), 0.022, 0.023, 0.027 and 0.033 mg/kg.

The use pattern is the same for beans and peas and the residues are not different. Consequently the residue datasets can be combined for mutual support.

The residues of trifloxystrobin in dry bean and pea seeds were < 0.01 mg/kg.

The sum of residues in beans and peas in rank order were: < 0.01 (8), 0.021 (4), 0.022, 0.023 (2), 0.025, 0.027 and 0.033 mg/kg.

As the use pattern for lentils is the same as for beans and peas, the Meeting decided that the database is sufficient for making recommendation for these three commodities.

The Meeting estimated a maximum residue level of 0.01* mg/kg and an STMR residue of 0.021 mg/kg for dry beans, lentils, and pea.

Animal feed

Soya bean forage and hay

Altogether 40 trials were conducted in USA in accordance with registered use patterns. Residues in forage and hay were measured and reported. However, grazing animals on soya bean fields or using forage and hay as animal feed are not permitted, therefore the results of trials were not evaluated.

Pea forage and hay

The average residues of trifloxystrobin and CGA 321113 measured in pea green materials (pea vine) obtained from trials conducted according to Canadian GAP are listed below.

Trifloxystrobin residues: 0.61, 0.78, 0.91, 0.92, 1.05, 1.10, 1.30, 1.50 and 1.95 mg/kg.

The sum of trifloxystrobin and CGA 321113 residues: 0.64, 0.81, 0.94, 0.95, 1.09, 1.15, 1.35, 1.54 and 1.98 mg/kg.

The Meeting estimated highest residue of 2 mg/kg and median residue of 1.1 mg/kg for the sum of trifloxystrobin and CGA321113 in pea vine for animal burden calculations.

The residues of trifloxystrobin and CGA 321113 (TFSA) measured in pea hay obtained from trials conducted according to Canadian GAP are listed below. Trifloxystrobin residues: 2.1, 3.0, 3.1, 3.3, 4.2, 5.3, 6.0, 6.7 and 6.8 mg/kg.

The sum of residues were in rank order: 2.2, 3.2, 3.3, 3.6, 4.5, 5.6, 6.3, 6.9 and 7.1 mg/kg

The Meeting estimated a maximum residue level of 17 mg/kg (dry weight) for peanut hay.

The Meeting estimated highest residue of 7.1 mg/kg and median residue of 4.5 mg/kg for the sum of trifloxystrobin and CGA321113 in pea hay for animal burden calculation.

Fate of Residues in Storage and Processing

Soya bean was treated with trifloxystrobin three times at a rate of 0.446–0.471 kg/ha and harvested 19 days after last application. The average total trifloxystrobin residue was 0.26 mg/kg in soya bean seed (raw agricultural commodity (RAC)), 18.2 mg/kg in soya bean aspirated grain fractions, 0.12 mg/kg in hulls, < 0.01 mg/kg in meal, and 0.03 mg/kg in refined oil. Concentration of the total trifloxystrobin residue was seen only in the soya bean aspirated grain fractions (processing factor about 70). No concentration of the total trifloxystrobin residue was seen in soya bean hulls, meal, or refined oil.

For the purpose of animal burden calculation, the Meeting estimated median residue of 0.7 mg/kg for aspirated grain fraction, 0.01 mg/kg for hull and < 0.0008 mg/kg for meal of soya bean.

Residues in animal commodities

Animal feeding studies were evaluated by the 2004 Meeting. Dairy cows were dosed with trifloxystrobin in capsules at the equivalent of 2, 5.9 or 21 ppm in the diet for 28–30 days. The residues measured in various samples are summarized below:

Sample	Day	Maximum trifloxystrobin residues (mg/kg)								
		Dose 2 ppm			Dose 5.9 ppm			Dose 21 ppm		
		Parent	321113	Total	Parent	321113	Total	Parent	321113	Total
Milk	26	-	-	-	-	-	-	< 0.01	< 0.01	< 0.02
Liver	28-30	< 0.02	< 0.02	< 0.04	< 0.02	< 0.02	< 0.04	< 0.02	0.09	0.11
Kidney	28-30	< 0.02	< 0.02	< 0.04	< 0.02	< 0.02	< 0.04	< 0.02	0.02	0.04
Perirenal fat	28-30	< 0.02	< 0.02	< 0.04				0,06	< 0.02	0.08
Omental fat	28-30	< 0.02	< 0.02	< 0.04	< 0.02	< 0.02	< 0.04	0.05	< 0.02	0.07
Round	28-30	-	-	-	-	-	-	< 0.02	< 0.02	< 0.04
Tenderloin	28-30	-	-	-	-	-	-	< 0.02	< 0.02	< 0.04

Laying hens were dosed at 1.5, 4.5 and 15 ppm level for 29 days. At the highest treatment level no residues (< 0.02 mg/kg) were detected in composite tissue samples of breast plus thigh, skin plus attached fat, peritoneal fat, liver and eggs.

The Meeting estimated the dietary burden of trifloxystrobin in farm animals on the basis of the diets listed in Annex 6 of the 2009 JMPR Report and using the estimated residues in livestock feed commodities evaluated by the present and previous Meetings.

	Trifloxystrobin animal dietary burden, ppm, of dry matter diet							
	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	2.17	1.15	26.6[a]	6.97[b]	8.24	5.00	4.53	0.84
Dairy cattle	2.79	1.27	23.2[c]	6.37[d]	8.21	4.11	2.11	0.43
Poultry - broiler	0.11	0.11	0.069	0.069	0.15	0.15	0.03	0.03
Poultry – layer	0.11	0.11	1.83[e]	0.78[f]	0.15	0.15	0.079	0.079

[a] Suitable for estimation maximum residue levels in meat

[b] Suitable for estimation of median residues in meat

[c] Suitable for estimation maximum residue levels in milk

[d] Suitable for estimation median residue levels in milk

[e] Suitable for estimation maximum residue levels in poultry meat and edible offal

[f] Suitable for estimation median residue levels in poultry meat and edible offal

The maximum dietary burden of beef cattle and dairy cattle is about 30% higher than the maximum feeding level of 21 ppm. The Meeting concluded that the residues observed at the highest feeding level can still be used as a basis for estimation of maximum residues in meat, offal and milk.

The Meeting concluded that the current Codex limits cover the residues derived from the uses of trifloxystrobin and maintains its previous recommendations.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed in Annex 1 to the Report were suitable for establishing maximum residue limits and for IEDI assessment.

CCN	Commodity Name	MRL, mg/kg proposed	previous	STMR or STMR-P mg/kg
VD0071	Beans, dry	0.01*		0.021
VD0533	Lentils	0.01*		0.021
VD4511	Pea, dry	0.01*		0.021
VD0541	Soya bean	0.05		0.01

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of trifloxystrobin were calculated for the 17 GEMS/Food cluster diets using STMRs and STMR-Ps estimated by the JMPR in 2004, 2012 and the current meeting. The results are shown in Annex 3 to the 2015 Report.

The ADI is 0–0.04 mg/kg bw and the calculated IEDIs were 1–4% of the maximum ADI. The Meeting concluded that the long-term intake of residues of trifloxystrobin from the uses considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The 2004 JMPR decided that it was unnecessary to establish an ARfD. The present Meeting therefore concluded that the short-term intake of trifloxystrobin residues is unlikely to present a public health.

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UNESP RA-994/06	Galhiane, MS & de Sousa, SL	2006e	Tebuconazol & Trifloxystrobin; 75 WG; soya; Brazil; BBA. Universidade Estadual Paulista (UNESP), Bauru, Brazil. Bayer CropScience AG, Report No.: UNESP RA-994/06, Edition Number: M-276638-01-2, includes FR05BRA001-P3. Unpublished. Translation of Galhiane, MS & de Sousa, SL, 2006f.

PESTICIDES RESIDUES IN SPICES

The first draft was prepared by Professor Arpad Ambrus, Hungarian Food Chain Safety Office, Budapest Hungary

EXPLANATION

Establishing of maximum residue limits for spices was discussed by the CCPR at several occasions.

The 36th Session of CCPR decided (Alinorm 04/24A) to schedule the JMPR to review the monitoring data available for the elaboration of MRLs on spices for pesticides already in the Codex system. The Committee also recommended that governments and the spice trade industry continue to collect monitoring data for pesticides on spices on a regular basis, following agreed criteria and other JMPR guidelines on the conduct of selective surveys, in order to keep the database updated for future review.

Subsequently the 2004 JMPR developed the general principles for evaluation of monitoring data for recommending maximum residue levels, median and high residues depending on the number of residue data available for a given pesticide residue and commodity combination.

The Meeting recommended among others that:

- when no sample contained detectable residues the highest reported LOQ value was used as the maximum residue level and the high residue value. The median residue value was calculated from the reported LOQ values.
- when > 120 samples contained detectable residues, the sample size was sufficiently large to calculate the upper 95% one-tailed confidence limit of the 95th percentile of the population of residues, which should be used as maximum residue level.
- monitoring results should not be used for estimating maximum residue levels that reflect post-harvest use.

Detailed guidance on submission of monitoring data and designing selective field surveys for obtaining residue data in/on spices are given in the FAO Manual Chapter 3.6.

Based on the elaborated principles, the 2004 JMPR recommended maximum residue, median and high residue levels for roots and rhizomes (HS01193) and fruits and berries (HS0191) groups for a number of pesticides. Based on the monitoring data submitted by Thailand, the 2010 JMPR recommended additional maximum, median and high residue levels for a number of pesticide residues in/on fruit, berry, root and rhizome spices.

In accordance with the decision of the 46th Session of the CCPR, India submitted monitoring data from 2009-2014 for acetamiprid, imidacloprid, carbofuran, cypermethrin, lambda-cyhalothrin, phorate, profenofos and triazophos residues in fruit/berry (cardamom, black pepper) spices, and seed spices (cumin, fennel and coriander).

METHODS OF RESIDUE ANALYSIS

Cardamom and pepper

Blend cardamom/pepper sample (250 g) into a coarse powder. Water is added to a representative test portion and mixed. After addition of acetonitrile and mixing, the material was placed in the freezer at -18 ºC for 20 minutes. The sample was then treated with NaCl, shaken and then centrifuged. The supernatant organic layer was treated with Na_2SO_4, vortexed and centrifuged. The subsequent supernatant was then treated with PSA sorbent and anhydrous $MgSO_4$, vortexed and centrifuged. The supernatant was divided into two, one for LC-MS/MS and the other for GC analysis. The pesticide residues detected in GC are confirmed by GC-MS.

Seed spices

Modified QuEChERS multiresidue method was adopted for the extraction and clean-up of various pesticide residues from seed spices. A representative portion ground seed (20 g) was moistened with water followed by addition of acetonitrile. The extract was treated with sodium chloride for separation of acetonitrile layer which was then subjected to dispersive SPE clean-up using PSA, $MgSO_4$ and C_{18}. The residues were determined using GC-MS/MS and/or LC-MS/MS.

The recovery and limit of quantification (LOQ) of pesticides on spices are given in Table-1.

Table 1 Recovery and limit of quantification (LOQ) of pesticides in spices

Compound	Commodity	Spike level (mg/kg)	Recovery range (%)	LOQ (mg/kg)
Acetamiprid	Cardamom	0.1-1.0	84 -103	0.1
	Pepper	0.1-1.0	88-100	0.1
	Cumin, Coriander, Fennel	0.1-1.0	88-112	0.1
Imidacloprid	Cardamom	0.1-1.0	71-83	0.1
	Pepper	0.1-1.0	81-93	0.1
	Cumin, Coriander, Fennel	0.1-1.0	93-109	0.1
Carbofuran	Cardamom	0.1-1.0	88-94	0.1
	Pepper	0.1-1.0	80-94	0.1
	Cumin, Coriander, Fennel	0.1-1.0	94-99	0.1
Cypermethrin	Cardamom	0.1-1.0	89-100	0.1
	Pepper	0.1-1.0	84-98	0.1
Lambda-cyhalothrin	Cardamom	0.1-1.0	91-102	0.1
	Pepper	0.1-1.0	95-107	0.1
Profenofos	Cardamom	0.1-1.0	93-105	0.1
	Pepper	0.1-1.0	94-108	0.1
	Cumin, Coriander, Fennel	0.1-1.0	90-110	0.1
Phorate	Cumin, Coriander, Fennel	0.1-1.0	88-102	0.1
Triazophos	Cardamom	0.1-1.0	92-99	0.1
	Pepper	0.1-1.0	88-107	0.1

Description of agricultural practices for growing spice producing plants

Cardamom, *Elettaria cardamomum* L. Maton, is mostly cultivated in the evergreen forests of Western Ghats of India. The crop is prone to infestation by diverse group of insect pests and diseases. Thrips and capsule borers are the major pests. On an average, farmers often apply pesticides every 15 to 18 days resulting in 18 to 25 sprays per year as against the recommended use of seven to eight treatments.

Pepper, *Piper nigrum* L is a native of South India. It is grown in the tropical regions. Pollu beetle, fungal Pollu and wilt disease are the limiting factors of pepper production in all the growing regions.

Cumin, coriander, and fennel are minor crops which are mainly cultivated in southern and western part of India. They are highly infested by aphid, thrips, cutworm, tobacco caterpillar and root knot nematodes. For the control of sucking pests like aphid and thrips, various pesticides are used as a foliar application. For the control of cutworm and tobacco caterpillar, profenofos is used, while phorate is used for the effective control of root knot nematode.

Information regarding harvesting, processing and storage of the spices

The cardamom is obtained by plucking the fruit or berry in the form of capsules from the spice crop. The sun dried or artificially dried capsules are then polished, graded and stored in polythene lined gunny bags or in wooden boxes in moisture free conditions.

Black pepper is produced from the still-green unripe drupes of the pepper plant. The drupes are boiled briefly in hot water, both to clean them and to prepare them for drying. The drupes are

dried in the sun or by machine for several days, during which the pepper around the seed shrinks and darkens into a thin, wrinkled black layer. The capsule of cardamom and unripe drupes for pepper are harvested up to 6 to 8 times a year.

Cumin and coriander are being harvested only once by separating the seeds from the dried spice-crop by using physical techniques. After removal of the physical impurities, the separated seeds are stored in gunny bags at room temperature. Aluminium phosphide is used for post-harvest protection.

The fennel seeds are obtained by drying matured inflorescence of the spice-crop under shade which are then stored in gunny bags at room temperature. The fennel is harvested up to 3 to 4 times a year.

RESULTS OF MONITORING PESTICIDE RESIDUES

Monitoring data were submitted from the period of 2009-2014. Cumin, fennel and coriander samples (250-500 g) were collected from the retail outlets. No information was provided on sampling of cardamom and black pepper.

Black pepper (HS 0790

Of the 284 samples analysed none of them contained residues at or above the 0.1 mg/kg limit of quantification.

Cardamom (HS 0775)

The residues detected are summarized in Table 2.

Table 2 Number of samples analysed and residues of various pesticides detected in cardamom

Compound	No.	Residues detected [mg/kg]
Acetamiprid	487	< 0.1
Carbofuran	487	< 0.1
Cypermethrin	487	0.10, 0.11 (3), 0.12, 0.13, 0.14 (4), 0.16, 0.18 (2), 0.19 (3), 0.20 (3), 0.21, 0.22 (2), 0.23 (3), 0.24 (2), 0.25, 0.26 (4), 0.27, 0.28 (2), 0.29 (2), 0.30 (2), 0.31 (2), 0.32 (6), 0.34 (5), 0.35(4), 0.36, 0.37 (3), 0.38, 0.39 (2), 0.41 (2), 0.43 (2), 0.44 (4), 0.45 (2), 0.46, 0.47, 0.49, 0.50 (2), 0.52, 0.53 (2), 0.54 (2), 0.55 (2),0.56, 0.58 (2), 0.59 (2), 0.60, 0.63, 0.64, 0.65, 0.66, 0.69 (2), 0.70 (3), 0.71 (2), 0.73, 0.75 (2), 0.76, 0.77, 0.79, 0.81, 0.86, 0.87(2), 0.91,0.92, 0.93, 0.99, 1.03, 1.12, 1.16, 1.34, 1.41, 1.54, 1.62, 1.65, 1.67, 1.76, 1.85, 1.94, 1.98, 2.00, 2.24, 2.97(2)
Lambda- cyhalothrin		0.10 (5), 0.11 (4), 0.12 (7), 0.13 (5), 0.14, 0.15 (4), 0.16 (3), 0.18 (3), 0.19 (7), 0.20 (6), 0.21 (5), 0.22, 0.23 (4), 0.24 (6), 0.25 (2), 0.26 (5), 0.27 (3), 0.28 (4), 0.29, 0.31 (2), 0.32 (3), 0.34 (5), 0.35 (3), 0.36 (2), 0.37 (3), 0.38, 0.40 (2), 0.41 (2), 0.42 (3), 0.43, 0.44, 0.45, 0.46, 0.49 (2), 0.50 (2), 0.51, 0.52 (3), 0.53, 0.54, 0.55, 0.57, 0.58 (2), 0.59, 0.61, 0.62 (2), 0.63, 0.67, 0.68, 0.69, 0.71, 0.73, 0.74 (2), 0.79, 0.82 (2), 0.86, 0.96, 0.99, 1.02, 1.04, 1.06, 1.20, 1.33, 1.87, 1.94, 3.06
Imidacloprid	487	0.10 (4), 0.11 (5), 0.12, 0.13 (2), 0.14 (2), 0.15 (2), 0.16, 0.17 (5), 0.18 (2), 0.20 (3), 0.21 (3), 0.22, 0.25, 0.27, 0.28, 0.30, 0.31, 0.32, 0.35, 0.38, 0.42, 0.47, 0.50, 0.51, 0.71, 0.80, 0.85
Profenofos	487	0.10 (3), 0.11 (5), 0.12 (3), 0.13 (2), 0.14 (5), 0.16, 0.17, 0.19 (2), 0.21, 0.22 (3), 0.24 (2), 0.25, 0.27, 0.28, 0.29, 0.30 (2), 0.3, 0.31, 0.32 (2), 0.34 (2), 0.36, 0.38, 0.39, 0.42 (2), 0.43, 0.44, 0.47 (2), 0.50 (2), 0.53 (2), 0.55, 0.63, 0.65, 0.66, 0.78, 0.79, 0.82, 0.91, 1.08, 1.19, 1.26, 1.54, 1.76, 1.9, 3.06
Triazophos	487	0.10, 0.11(2), 0.12(2), 0.14,0.15,0.16, 0.17 (4),0.19(2), 0.21(5), 0.22, 0.23 (2), 0.25, 0.26, 0.28, 0.29 (3), 0.32, 0.33, 0.34, 0.37(2), 0.39(2), 0.40 (2), 0.43, 0.45 (3), 0.46, 0.47, 0.48, 0.49, 0.5 (2), 0.53 (2), 0.55 (2), 0.58, 0.59, 0.6, 0.61, 0.62, 0.63, 0.64, 0.69, 0.77, 0.82 (2), 0.84, 0.85 (2), 0.86, 1.06, 1.09, 1.11, 1.13, 1.34, 1.38,1.42, 1.49, 1.67, 1.68, 1.71, 2.30, 3.64

Cumin (HS 0780)

The residues detected are summarized in Table 3.

Table 3 Number of samples analysed and residues of various pesticides detected in cumin

Compound	No.	Residues detected
Acetamiprid	447	0.12(2), 0.13(2), 0.15, 0.16, 0.17, 0.18, 0.19, 0.20(2), 0.21, 0.23, 0.25, 0.27, 0.39, 0.40, 0.44, 0.46, 0.48, 0.58, 0.59, 0.65, 0.69, 0.76, 0.81, 1.35, 1.42, 1.43, 1.55, 2.04, 2.38, 2.93
Carbofuran	447	0.11(3), 0.12, 0.13, 0.15 (2), 0.21, 0.24, 0.27, 0.28, 1.35
Imidacloprid	447	0.14 (2), 0.25, 0.27, 0.36, 0.40, 0.45, 0.46
Phorate	447	0.11, 0.15 (2), 0.24, 0.26, 0.34, 0.76
Profenofos	447	0.10 (2), 0.11 (2), 0.12, 0.13 (4), 0.14 (2), 0.15 (2), 0.16 (2), 0.17, 0.18, 0.19(2), 0.20, 0.22 (2), 0.24 (2), 0.25, 0.27, 0.31 (2), 0.32, 0.34, 0.38, 0.39, 0.41, 0.42, 0.44, 0.47, 0.56, 0.63, 0.64, 0.65 (2), 0.66, 0.68 (2), 0.73, 0.77, 0.80, 0.82, 0.85, 0.86, 0.94, 0.95, 0.99, 1.03, 1.05, 1.07, 1.10, 1.21, 1.22 (2), 1.26(2), 1.30, 1.38, 1.51, 1.52, 1.61, 1.85, 1.98, 2.11, 2.32, 2.47, 2.69, 2.90, 3.83, 4.12

Coriander (HS0779)

Altogether 223 samples were analysed (positive results in brackets) for acetamiprid (0.02 mg/kg), imidacloprid (0), profenofos (0), phorate (0) and triazophos (0)

Fennel (HS 0731)

Altogether 255 samples were analysed (positive results in brackets) for acetamiprid (0.023, 0.03 mg/kg), carbofuran (0), imidacloprid (0.32), profenofos (0), phorate (0). and triazophos (0).

APPRAISAL

The Thirty-sixth Session of CCPR decided (Alinorm 04/24A) to schedule by the JMPR the review of the monitoring data available for the elaboration of MRLs on spices for pesticides already in the Codex system.

Subsequently the 2004 JMPR developed the general principles for evaluation of monitoring data for recommending maximum residue levels, median and high residues depending on the number of residue data available for a given pesticide residue and commodity combination.

In accordance with the decision of the Forty-sixth Session of the CCPR, India submitted monitoring data from 2009-2014 for several pesticide residues in cardamom, black pepper, cumin, fennel and coriander for review by the 2015 JMPR.

Sampling and analytical methods

Cumin, fennel and coriander seed samples (250–500 g) were collected from the retail outlets. No information was provided on sampling of cardamom and black pepper.

The residues in/on cardamom and black pepper were extracted with a mixture of acetonitrile/water. The dried extract was purified with a primary secondary amine (PSA) adsorbent in the presence of $MgSO_4$, and the residues were identified and quantified by GC-MS/MS or LC-MS/MS.

Seed spices were extracted with the mixture of acetonitrile/water and further determined with a modified QuEChERS multiresidue method using GC-MS/MS and/or LC-MS/MS.

For both methods, the recoveries were within the acceptable range, and reported LOQ was 0.1 mg/kg for all pesticide residue commodity combinations.

Agricultural practices for growing spice producing plants

Cumin, cardamom, coriander, pepper and fennel are minor crops which are mainly cultivated in the southern and western parts of India. The spices need to be protected against several pests and diseases which require repeated application of pesticides around the year.

The capsule of cardamom and unripe drupes for pepper are harvested up to 6 to 8 times a year. Cumin and coriander are harvested only once and fennel, up to 3 to 4 times.

No information was available on registered or approved uses or application conditions of the pesticides.

Principles of evaluation of residues derived from monitoring programmes

Principles for evaluation of monitoring data elaborated by the 2004 JMPR were followed:

- It is assumed that the laboratories reported only valid results. Therefore, all residue data are taken into account without excluding any value as an outlier.
- When residue values were reported as<LOQ, it does not necessarily mean that the sampled commodity was not treated with or exposed to the pesticide. While, it is unlikely that all the sampled commodities were treated with the pesticides looked for with the multi residue procedure, it cannot be assumed to be a 'nil' residue situation.
- When no sample contained detectable residues, the highest reported LOQ value is used as the maximum residue level. When justified based on the consumption, the high and median residue value are taken from the reported LOQ values.
- Distribution-free statistics are used in estimating the maximum residue level, covering the 95th percentile of the residue population at the 95% confidence level. Thus, the estimated maximum residue level encompasses at least 95% of the residues with 95% probability (in 95% of cases). To satisfy this requirement, a minimum of 58–59 samples is required. In such cases the uncertainty derived from the limited number of data points are taken into account in recommending maximum residue levels.
- When > 120 samples contain detected residues, the sample size is sufficiently large to calculate the upper 95% one-tailed confidence limit of the 95^{th} percentile of the population of residues, which should be used for estimation of maximum residue level after rounding up to the next value of the scale of expressing residues according to the OECD MRL calculator.
- Monitoring results are not used for estimating maximum residue levels that reflect post-harvest use.

Furthermore, the Meeting decided that:

Maximum residue levels would only be estimated for those pesticide residues which were determined according to the definition of residues for enforcement purposes. Consequently, the reported residues of carbofuran and imidacloprid were not considered.

Residues resulting from monitoring programmes

Black pepper

Of the 284 samples analysed for acetamiprid, cypermethrin, lambda-cyhalothrin, profenofos, and triazophos, none were found to contain residues at or above the LOQ of 0.1 mg/kg.

The Meeting concluded that the reported LOQ values are higher than those which can be obtained with current analytical methods. Consequently, the Meeting agreed there was no reason to revise its previous recommendations for maximum residue levels for cypermethrin, lambda cyhalothrin, profenofos and triazophos.

The Meeting estimated a maximum residue level and median residue of 0.1 mg/kg for acetamiprid.

Cardamom seed

Results of analyses of 487 samples were reported for acetamiprid, cypermethrin, lambda-cyhalothrin, imidacloprid, profenofos and triazophos.

No residues (< 0.1 mg/kg) of acetamiprid were detected.

Based on the results, the Meeting estimated a maximum residue and median residue of 0.1 mg/kg for acetamiprid.

Out of 487 samples 133 contained cypermethrin residues which were in rank order: 0.10, 0.11 (3), 0.12, 0.13, 0.14 (4), 0.16, 0.18 (2), 0.19 (3), 0.20 (3), 0.21, 0.22 (2), 0.23 (3), 0.24 (2), 0.25, 0.26 (4), 0.27, 0.28 (2), 0.29 (2), 0.30 (2), 0.31 (2), 0.32 (6), 0.34 (5), 0.35(4), 0.36, 0.37 (3), 0.38, 0.39 (2), 0.41 (2), 0.43 (2), 0.44 (4), 0.45 (2), 0.46, 0.47, 0.49, 0.50 (2), 0.52, 0.53 (2), 0.54 (2), 0.55 (2),0.56, 0.58 (2), 0.59 (2), 0.60, 0.63, 0.64, 0.65, 0.66, 0.69 (2), 0.70 (3), 0.71 (2), 0.73, 0.75 (2), 0.76, 0.77, 0.79, 0.81, 0.86, 0.87(2), 0.91,0.92, 0.93, 0.99, 1.03, 1.12, 1.16, 1.34, 1.41, 1.54, 1.62, 1.65, 1.67, 1.76, 1.85, 1.94, 1.98, 2.00, 2.24, and 2.97(2) mg/kg.

The upper 95% confidence limit of the detected residues is 2.24 mg/kg.

The Meeting estimated a maximum residue level of 3 mg/kg and a median residue of 0.43 mg/kg for cypermethrin which replaces its previous recommendations.

Out of 487 samples 146 contained lambda cyhalothrin residues which were in rank order: 0.10 (5), 0.11 (4), 0.12 (7), 0.13 (5), 0.14, 0.15 (4), 0.16 (3), 0.18 (3), 0.19 (7), 0.20 (6), 0.21 (5), 0.22, 0.23 (4), 0.24 (6), 0.25 (2), 0.26 (5), 0.27 (3), 0.28 (4), 0.29, 0.31 (2), 0.32 (3), 0.34 (5), 0.35 (3), 0.36 (2), 0.37 (3), 0.38, 0.40 (2), 0.41 (2), 0.42 (3), 0.43, 0.44, 0.45, 0.46, 0.49 (2), 0.50 (2), 0.51, 0.52 (3), 0.53, 0.54, 0.55, 0.57, 0.58 (2), 0.59, 0.61, 0.62 (2), 0.63, 0.67, 0.68, 0.69, 0.71, 0.73, 0.74 (2), 0.79, 0.82 (2), 0.86, 0.96, 0.99, 1.02, 1.04, 1.06, 1.20, 1.33, 1.87, 1.94, and 3.06 mg/kg.

The upper 95% confidence limit of the residues is 1.87 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg and a median residue of 0.28 mg/kg for cyhalothrin, which replaces its previous recommendations.

Out of 487 samples 68 contained profenofos residues which were in rank order: 0.10 (3), 0.11 (5), 0.12 (3), 0.13 (2), 0.14 (5), 0.16, 0.17, 0.19 (2), 0.21, 0.22 (3), 0.24 (2), 0.25, 0.27, 0.28, 0.29, 0.30 (3), 0.31, 0.32 (2), 0.34 (2), 0.36, 0.38, 0.39, 0.42 (2), 0.43, 0.44, 0.47 (2), 0.50 (2), 0.53 (2), 0.55, 0.63, 0.65, 0.66, 0.78, 0.79, 0.82, 0.91, 1.08, 1.19, 1.26, 1.54, 1.76, 1.9, and 3.06 mg/kg.

The 95th percentile of the residues is 1.4 mg/kg. The database is insufficient for calculation of the upper confidence limit.

Taking into account the limited database, the Meeting estimated a maximum residue level of 3 mg/kg and a median residue of 0.3 mg/kg for profenofos which replaces its previous recommendations.

Out of 487 samples 79 contained triazophos residues which were in rank order: 0.10, 0.11(2), 0.12(2), 0.14,0.15,0.16, 0.17 (4),0.19 (2), 0.21(5), 0.22, 0.23 (2), 0.25, 0.26, 0.28, 0.29 (3), 0.32, 0.33, 0.34, 0.37(2), 0.39(2), 0.40 (2), 0.43, 0.45 (3), 0.46, 0.47, 0.48, 0.49, 0.5 (2), 0.53 (2), 0.55 (2), 0.58, 0.59, 0.6, 0.61, 0.62, 0.63, 0.64, 0.69, 0.77, 0.82 (2), 0.84, 0.85 (2), 0.86, 1.06, 1.09, 1.11, 1.13, 1.34, 1.38,1.42, 1.49, 1.67, 1.68, 1.71, 2.30, and 3.64 mg/kg.

The 95th percentile of the residues is 1.7 mg/kg. The database is insufficient for calculation of the upper confidence limit.

Taking into account the limited database, the Meeting estimated a maximum residue level of 4 mg/kg and a median residue of 0.45 mg/kg for triazophos, which replaces the previous recommendations.

Coriander seed

Altogether 223 samples were analysed (positive results in brackets) for acetamiprid (0.02 mg/kg), profenofos (0), phorate (0) and triazophos (0). The reported LOQ was 0.1 mg/kg.

The residue data was not sufficient to estimate a maximum residue level for acetamiprid.

The Meeting estimated maximum and median residue levels of 0.1 mg/kg for profenofos, phorate and triazophos in coriander seed.

Cumin seed

The results of analyses of 447 samples were reported for acetamiprid, phorate and profenofos.

Out of 447 samples acetamiprid (33) and phorate (7) residues were detected above the LOQ of 0.1 mg/kg.

As the number of detected residues is lower than the minimum required (58), no recommendations could be made for maximum residue levels for acetamiprid and phorate.

Out of 447 samples 76 contained profenofos residues which were in rank order: 0.10 (2), 0.11 (2), 0.12, 0.13 (4), 0.14 (2), 0.15 (2), 0.16 (2), 0.17, 0.18, 0.19(2), 0.20, 0.22 (2), 0.24 (2), 0.25, 0.27, 0.31 (2), 0.32, 0.34, 0.38, 0.39, 0.41, 0.42, 0.44, 0.47, 0.56, 0.63, 0.64, 0.65 (2), 0.66, 0.68 (2), 0.73, 0.77, 0.80, 0.82, 0.85, 0.86, 0.94, 0.95, 0.99, 1.03, 1.05, 1.07, 1.10, 1.21, 1.22 (2), 1.26(2), 1.30, 1.38, 1.51, 1.52, 1.61, 1.85, 1.98, 2.11, 2.32, 2.47, 2.69, 2.90, 3.83, and 4.12 mg/kg.

The 95th percentile of the residues is 2.52 mg/kg. The database is insufficient for calculation of the upper confidence limit. Taking into account the limited database, the Meeting estimated a maximum residue level of 5 mg/kg and median residue of 0.635 mg/kg for profenofos.

Fennel, seed

Altogether 255 samples were analysed (positive results in brackets) for acetamiprid (0.023, 0.03 mg/kg), profenofos (0), phorate (0) and triazophos (0).

The Meeting estimated maximum and median residue levels 0.1 mg/kg for profenofos, phorate and triazophos.

Maximum residue level recommendations for Spices

Pesticide	CCN	Commodity	MRL mg/kg		Median mg/kg
			New	Previous	
Acetamiprid (246)	HS 0790	Pepper, Black; White	0.1		0.1
	HS 0775	Cardamom	0.1		0.1
Cypermethrin (118)	HS 0775	Cardamom	3		0.43
Lambda-cyhalothrin (146)	HS 0775	Cardamom	2	0.03	0.28
	HS 0191	Spices, Fruits and Berries (except Cardamom)	0.03		
Phorate (112)	HS 0779	Coriander, seed	0.1	0.5	0.1
	HS 0731	Fennel, seed	0.1	0.5	0.1
	HS 0190	Spices, Seeds (except Coriander seed and Fennel seed)	0.5		
Profenofos (171)	HS 0775	Cardamom	3	0.07	0.3
	HS 0779	Coriander, seed	0.1		0.1
	HS 0780	Cumin seed	5		0.635
	HS 0731	Fennel, seed	0.1		0.1
	HS 0191	Spices, Fruits and Berries (except Cardamom)	0.07		
Triazophos (143)	HS 0775	Cardamom	4	0.07	0.45
	HS 0779	Coriander, seed	0.1		0.1
	HS 0731	Fennel, seed	0.1		0.1
	HS 0191	Spices, Fruits and Berries (except Cardamom)	0.07		

DIETARY RISK ASSESSMENT

Long-term intake

The contribution of residues present in the pepper, black white to the long-term-intake of acetamiprid and lambda-cyhalothrin was addressed in the evaluation of these compounds. No consumption data is available for cardamom, coriander, cumin and fennel seeds in the 17 GEMS/Food Cluster diets to estimate the contribution of the residues present in these spices to the long-term-intake of acetamiprid, cypermethrin, lambda-cyhalothrin, profenofos, phorate and triazophos.

Short-term intake

The International Estimated Short-Term Intake (IESTI) of acetamiprid and lambda-cyhalothrin from the consumption of pepper, black white and cardamom seed was addressed in the evaluation of these compounds.

The IESTIs for profenofos, phorate and triazophos from the consumption of the spices considered by the current Meeting were estimated. The results are shown in Annex 4 to the 2015 Report. The IESTI represented 0% of the ARfD of cypermethrin and profenofos, a maximum of 10% of the ARfD of phorate and a maximum of 7% of the ARfD of triazophos. The Meeting concluded that the short-term intake of cypermethrin, profenofos, phorate and triazophos residues from the uses considered by the current Meeting was unlikely to present a public health concern.

CORRIGENDA

Pesticide Residues in Food 2014. Evaluations Part 1 - Residues. FAO Plant Production and Protection Paper 222, 2015

Changes are shown in bold

Fenpropathrin (185)

Page 700, paragraph 11 should read:

The Meeting estimated a maximum residue value of 2mg/kg and, based on the processing factor of 0.065, HR of **0.078 mg/kg** and STMR values 0.02 mg/kg for citrus fruit group.

Page 701, paragraph 11 should read:

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, **0.67 mg/kg** and 0.25 mg/kg; and cherries 7 mg/kg, 3.53, and 1.85 mg/kg, respectively

Page 702, paragraph 11 should read:

The Meeting noted that the trials were not conducted at maximum GAP. For multiple treatments proportionality could **not** be applied. As a result no recommendations could be made.

Page 706, the table should read:

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	**0.014**
Wet pomace				9.9	**~~9.8~~**	9.8	**1.88**
Dry pomace				46	**~~45.0~~**	**46**	**8.74**
Tomato paste				0.78	**~~0.75~~**	**0.78**	**0.148**
Tomato juice				0.12	**~~0.1~~**	**0.12**	0.023

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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 Geneva, Switzerland, from 15 to 24 September 2015. The FAO Panel of Experts had met in preparatory sessions from 10 to 14 September 2015. 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.

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